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Supplement to
Appendix F

of the
Project Scoping Plan for
Performing a CERCLA RI/FS at Building 804 and Associated Radioactive Waste Burial Sites (SEAD-12)

Chemical Data Acquisition Plan

General Engineering Laboratories
Quality Assurance Plan and Radiochemistry SOPs

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General Engineering Laboratories
Quality Assurance Plan

GENERAL ENGINEERING LABORATORIES, INC.

QUALITY ASSURANCE PLAN

(GL-QS-B-001 Revision 11)

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Prepared by:

General Engineering Laboratories, Inc.
P.O. Box 30712
Charleston, SC 29417

Quality Assurance Plan

Approval Signatures

Robert L Pullano — June 9, 1999 (Date)

Quality Systems Manager

James M. Stelling — June 9, 1999 (Date)

Chief Operating Officer

TABLE OF CONTENTS

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SECTION 1
INTRODUCTION

Section 1: Introduction

General Engineering Laboratories, Inc. (GEL), a privately owned environmental consulting and analytical firm, is dedicated to providing personalized services of the highest quality. Our mission is to be the "Analytical, Environmental and Engineering Firm of First Choice."

GEL was established as a full service analytical testing laboratory in 1981. The analytical divisions use state of the art equipment and methods to provide a comprehensive array of organic, inorganic, and radiochemical analyses and related support services to meet the needs of our clients.

This manual provides an overview of our analytical services quality assurance program. We have implemented this program to ensure that we are consistently working toward the fulfillment of our mission. Outlined in this manual are the responsibilities, policies and operational processes essential to maintaining client satisfaction and our high quality of performance.

The intent of this manual is to provide the quality assurance requirements specific to GEL by describing a minimum set of quality management criteria necessary to provide our clients with services that meet the highest quality standards. Project-specific requirements work in conjunction with the quality plan outlined in this manual.

All GEL personnel should understand the policies, objectives and procedures described in this manual so that they fully understand GEL's commitment to quality as well as their roles and responsibilities with respect to our quality assurance program. In addition, all subcontractors employed by GEL should adhere to the quality assurance requirements delineated in this manual.

This Quality Assurance Plan (QAP) has been prepared according to the standards and requirements of the United States Environmental Protection Agency (EPA) National Environmental Laboratory Accreditation Conference (NELAC), Quality Systems Revision 9, July 2, 1998.

1.1 Quality Policy

GEL's quality policy is clearly stated in our corporate objective "to provide high quality, personalized consulting and analytical services that enable our clients to meet their environmental needs cost effectively."

We define quality as "consistently meeting the needs and exceeding the expectations of our clients." Every individual at GEL is responsible and accountable for providing high quality services and data in the most efficient manner possible.

In order to fall within this definition, our our analytical and consulting services must:

- meet or exceed client and/or regulatory requirements
- be technically correct and accurate
- be defensible within contract specifications
- be provided in a cost-effective, timely and efficient manner

Quality is emphasized at every level of the company—from the president and Chief Operating Officer (CEO) to the individual employees. Our ongoing commitment to quality is demonstrated by senior management's dedication of personnel and resources to continuously develop, implement, assess, and improve our technical and management operations.

GEL's quality assurance program complies with the guidelines and specifications outlined in the following:

- ASME/NQA-1
- ISO 9001/Q91
- ISO/IEC Guide 25
- US EPA Quality Assurance Staff (EPA QAMS-005/80) Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans
- Department of Energy Order 5700.6C
- Current U.S. EPA CLP statements of work for inorganic and organic analyses
- Quality Assurance for Bioassay Laboratories
- NELAC

Our corporate quality policy is further articulated in GL-OS-A-001 General Engineering Laboratories, Inc. Quality Manual.

1.2 Quality Goals

The two primary goals of our quality assurance plan are to:

- Ensure that all measurement data generated is scientifically and legally defensible, of known and acceptable quality per the data quality objectives (DQOs) and thoroughly documented so that it provides sound support for environmental decisions

- Ensure compliance with all contractual requirements and environmental standards and regulations established by local, state and federal authorities.

Our additional quality goals are to:

- Provide a uniform framework for the generation of physical and chemical data

- Operate under a comprehensive quality assurance program that ensures the timely and effective completion of each measurement effort by focusing on preventive maintenance.

- Instill a commitment to quality assurance and excellence at all levels of the organization.
- Assist in the early detection of anomalies and nonconformances that might adversely affect data quality.
- Establish the quality assurance objectives for the measurement systems and to assess and monitor analytical data quality in terms of accuracy, precision, completeness, and comparability through the use of proven methods
- Through the use of process control charts and other means, establish procedures to demonstrate that analytical systems are in a state of statistical control.
- Enable personnel responsible for the production of data to identify and implement corrective actions necessary to ensure data integrity.
- Ensure that appropriate type and degree of quality control is applied during sample processing and analysis.
- Ensure adequate document control
- Reduce data entry errors by utilizing comprehensive automated data handling procedures.
- Develop and implement good laboratory and standard operating procedures (SOPs)
- Provide sufficient flexibility for customized quality assurance procedures that meet a client's specific requirements for data quality.
- Establish guidelines for adequate control of instrument, services, and chemical procurement.
- Ensure tracking of samples and analytical data by an automated laboratory data management system.
- Ensure that computer hardware and software used in producing analytical data are independently validated and documented according to their intended use.

1.3 Key Quality Elements

The formation and implementation of a sound quality assurance program are essential to providing data and services that meet GEL's high standards of integrity and commitment to the client.

Elements of our quality assurance plan that significantly contribute to meeting these standards include:

- Independent quality assurance (QA) validation and Quality Systems Department.

- Formal quality policy and QAP.
- Data quality objectives
- Comprehensive employee training program
- Internal audits and self-evaluations.
- Closed-loop corrective actions program.
- Implementation of good laboratory practices.
- State-of-art facilities and instrumentation.
- Adherence to standard operating procedures.
- EPA/NIST traceable reference materials.
- Document control.
- Internal sample chain of custody and electronic sample tracking
- Interlaboratory comparison programs.
- Formal laboratory accreditations.
- Independent QA validation of computer software.
- Evaluation of subcontractor laboratories.
- Statistical evaluation for analytical precision and accuracy.
- Replicate, method blank, matrix spike, tracer yield, internal standards, and surrogate measurements.
- Preventive maintenance of instrumentation and equipment.
- Independently prepared blind standard reference materials.
- Multi-level review process.
- Client satisfaction questionnaires.
- Internal tracking of commitments to clients.
- Independent trend analysis of nonconforming items.

1.4 Supporting Documents

GEL analytical data quality and laboratory operations are in compliance with specifications described in the documents listed in Appendix A.

1.5 Definitions

Relevant definitions are listed in Appendix B.

SECTION 2
ORGANIZATION, MANAGEMENT, AND PERSONNEL

Section 2: Organization, Management, and Personnel

The corporate organizational structure of GEL is shown Figure 1. The chain of command and flow of responsibility depicted in this figure are designed to ensure the overall quality and cost efficiency of our company's analytical products and services.

GEL's structure is more horizontal than the typical vertical alignment. The structure is based on customer-focused divisions that follow a project from the point of initial contact to the final invoicing of work. These divisions include expertise in project management, sample receipt and custody, sample preparation and analysis, data review and data packaging. An independent Quality Department monitors the adherence of these divisions to the GEL Quality Assurance Program.

The general responsibilities associated with the following position levels are discussed in this section:

- Chief Executive Officer (CEO)
- President
- Chief Operating and Financial Officer
- Quality Systems Manager
- Laboratory Division Directors
- Project Managers
- Group Leaders
- Laboratory and Technical Staff
- Information Systems Manager
- Director of Facilities and Sample Management
- Controller
- Consulting Division Director

An overview of the GEL employee training protocol is also provided.

2.1 CEO and President

Joint responsibility for the overall operations of GEL rests with George C. Greene and Molly F. Greene who serve respectively as Chief Executive Officer and President. As the company's founders and owners, it is their philosophical approach to quality and customer service that has resulted in our unique company structure.

George C. Greene, CEO, is a licensed Professional Engineer with a Ph.D. in Chemical Engineering. He and GEL President Molly F. Greene share oversight responsibility for all of GEL's operations. Together they have established and are part of a Leadership Team that works to create a workplace environment that can attract and retain highly qualified professionals.

As CEO, Dr. Greene also has primary responsibility for the development and technical administration of our analytical testing and environmental consulting

services. He leads the management team in implementing total quality initiatives that ensure that the quality of GEL's analytical services meet the most stringent criteria of excellence.

As President, Ms. Greene is also involved in the daily management of community and government affairs, client relations, business development, and human resources. She holds a Bachelor of Liberal Arts degree and speaks Spanish fluently.

2.2 Chief Operating and Financial Officer

James M. Stelling is our Chief Operating Officer (COO) and Chief Financial Officer (CFO.) He holds a Bachelor of Science in Commerce from the University of Virginia.

As COO/CFO, Mr. Stelling has both operational and financial responsibility for all aspects of our business operations. He has delegated authority from the CEO and President to manage and operate the laboratory in accordance with corporate policies and directives and within applicable licenses and regulations.

Mr. Stelling reports directly to the CEO and President. He has specific additional responsibility for:

- Ensuring that the individuals who staff our technical and quality positions have the necessary education, training and experience to competently perform their jobs.
- Ensuring that all staff members receive ancillary training, as needed, to enhance performance in assigned positions.
- Budgeting, staffing, managing and equipping the laboratory to meet current and future analytical program requirements.
- Overseeing the implementation and overall effectiveness of our Quality Assurance Plan, health and safety initiatives, and environmental programs.
- Managing production and cost control activities.
- Ensuring development of capabilities in response to new or revised regulations, instrumentation systems and procedures, and quality assurance initiatives.
- Designating necessary operational authorities in times of absence to one or more appropriately knowledgeable individuals.

Together, the CEO, President, and COO form GEL's Executive Committee.

2.3 Quality Systems Manager

GEL's Quality Systems Manager (QSM) reports directly to our COO/CFO. The QSM manages the design, implementation and maintenance of our quality systems in a timely, accurate, and consistent manner.

In addition to having responsibility for the initiation or recommendation of corrective actions, the Quality Systems Manager is responsible for:

- Establishing, documenting and maintaining comprehensive and effective quality systems.
- Developing and evaluating quality assurance policies and procedures pertinent to our laboratory functions, and communicating these with the division directors and managers.
- Ensuring that the operations of our laboratory are in conformance with the Quality Assurance Plan and meet the quality requirements specific to each analytical method.
- Ensuring that all laboratory activities are in compliance with local, state and federal environmental laws and regulations.
- Reviewing project-specific quality assurance plans.
- Ensuring that quality control limits are established and followed for critical points in all measurement processes, and that they are based on EPA guidance or on sound statistical methods.
- Initiating internal performance audits using certified, high-purity standard reference materials that have been purchased commercially.
- Performing independent quality assurance reviews of randomly selected data reports.
- Conducting periodic audits to ensure method compliance.
- Conducting or arranging periodic technical system evaluations of laboratory facilities, instruments and operations.
- Overseeing nonconformance and corrective actions and monitoring their progress.
- Communicating system breakdowns or deficiencies, recommending corrective action to improve the data generation system, and defining the validity of data generated during out of control situations.
- Preparing and updating quality assurance related documents and monthly reports to our management.
- Coordinating interlaboratory reviews and comparison studies.
- Initiating and overseeing **Stop Work Orders** in out of control situations.
- Administering accreditation and licensing activities.
- Administering our document control system.
- Providing guidance and training to laboratory staff as requested.
- Evaluating subcontractors and vendors that provide analytical and calibration services.
- Designating necessary quality systems authorities in times of absence to one or more appropriately knowledgeable individuals.

2.4 Laboratory Division Directors

To enhance responsiveness to clients through dedicated expertise and teamwork, our laboratory has been divided into Commercial, Federal and Radiochemistry Divisions, each with its own director.

Division Directors report directly to the COO and are ultimately responsible for the technical content and quality of work performed within their divisions. Division Directors are also responsible for strategic planning, profitability and

growth, personnel management and business development. Other principal responsibilities include:

- Monitoring and meeting profitability and growth objectives of the division.
- Establishing and implementing short and long range objectives and policies that support GEL's goals.
- Defining the minimum level of qualification, experience, and skills necessary for all positions in their respective division.
- Establishing and implementing policies and procedures that support our quality standards.
- Ensuring that all technical laboratory staff demonstrate initial and ongoing proficiency in the activities for which they are responsible.
- Documenting all analytical and operational activities of the laboratory.
- Supervising all personnel employed in the division.
- Ensuring that all sample acceptance criteria are verified and that samples are logged into the sample tracking system and properly labeled and stored.
- Documenting the quality of all data reported by the division.
- Developing internal mechanisms and measurements to improve efficiency.
- Overseeing activities designed to ensure compliance with laboratory health and safety requirements.
- Allocating the resources necessary to support an effective, ongoing and comprehensive quality assurance program.
- Representing the company to the public and to established and potential clients.
- Ensuring the appropriate delegation of authorities and assignments during periods of absence.

2.5 Project Managers

Each laboratory division has a number of Project Managers who serve as direct and primary liaisons to our clients. Project Managers report to the Division Directors and are responsible for managing the company's interaction with their assigned clients.

Project Managers are expected to promote the company's best image and strive for "**Total Client Satisfaction**" Additional responsibilities include:

- Retaining clients and soliciting new work.
- Managing multiple sample delivery orders and preparing quotes.
- Working with clients to define analytical methodologies, quality assurance requirements, reports and deliverables, and pricing.
- Overseeing sample receipt and informing the laboratory staff of the anticipated arrival of samples for analysis.
- Assisting in development of project specific quality assurance documents.
- Conducting a final technical review of all documents (quotes, hard copy deliverables, invoices, routine and specialized reports.)

- Working with the accounting team on invoicing and collection issues.
- Working with the Division Directors to predict potential workloads and determine acceptable scheduling.

2.6 Group Leaders

Group Leaders are the critical link between Division Directors, laboratory personnel and support staff. They support our Division Directors in the planning and scheduling analytical work and oversee and direct the workflow within their respective groups on a daily basis.

Group Leaders report directly to the Division Directors. They are primarily responsible for:

- Planning, coordinating and directing the operations of their groups to meet client expectations.
- Scheduling sample preparation and analysis in accordance with holding times, quality assurance and quality control criteria, and client due dates.
- Ensuring the technical review of 100% of the data generated by their groups.
- Coordinating nonconformances and corrective actions in conjunction with the Quality Systems Management team.
- Serving as a technical resource to their groups, including data review and validation.
- Managing special projects, reviewing new work proposals, and overseeing the successful implementation new methodologies.
- Monitoring and controlling expenses incurred within their groups such as overtime and consumables.
- Providing performance and career development feedback to their group members.

2.7 Laboratory and Technical Staff - General Requirements

GEL's commitment to providing clients with quality services and technical documents that meet or exceed their expectations is evident in the laboratory and technical staff. Every effort is made to ensure that the laboratory is sufficiently staffed with personnel who have the training, education, skill. and technical knowledge to perform their assigned jobs competently.

All laboratory personnel are responsible for:

- Complying with quality assurance and quality control requirements that pertain to their organizational and/or technical function.
- Demonstrating a specific knowledge of their particular function.
- Possessing a general knowledge of laboratory operations.
- Understanding analytical test methods and standard operating procedures, which are applicable to their job function.
- Documenting their activities and sample interactions as directed by their respective analytical methods and/or standard operating procedures.

- Implementing the quality assurance program as it pertains to their respective job functions.
- Identifying potential sources of error and reporting any observed substandard conditions or practices to the group leaders or Quality Systems Management Team.
- Identification and correcting any problems affecting the quality of the analytical data.

2.8 Information Systems Manager

The Information Systems Manager reports directly to the COO/CFO. The responsibilities of this position include management of the Information Systems Team and our computerized laboratory information management system.

The combined responsibilities of the Information Systems Team, performing under the Information Systems Manager leadership, include:

- Development and maintenance of all software and hardware needs
- Translation and interpretation of routines for special projects
- Interpretation of general data and quality control routines
- Optimization of processes through better software and hardware utilization
- Customization, development, testing and modification of new and existing data base applications.
- Maintain and modify our computer modeling, bar coding, CAD, statistical process control, project management, and data packaging systems.
- Establish, develop, and implement client or internal electronic data deliverables.
- Ensuring and documenting that all software used in processing analytical data have been validated and verified.

2.9 Director of Facilities and Sample Management

The Director of Facilities oversees the physical facility, sample management, and laboratory instrumentation. This position reports to the COO/CFO, and is responsible for the management and supervision of the functions and staff assigned to these areas. Position responsibilities include:

- Planning, evaluating and making recommendations for facility maintenance, additions and renovations.
- Overseeing building renovations and new construction activities.
- Installing, maintaining, repairing and modifying analytical instrumentation .
- Providing technical expertise and training in instrumentation operation, calibration and maintenance.
- Managing sample and waste flow through the laboratory from receipt to disposal.
- Monitoring and ensuring regulatory compliance for waste management operations and off-site disposal.

2.10 Controller

The Controller reports directly to the COO/CFO and oversees the functions of invoicing, purchasing, payroll, accounts payable, accounts receivable, inventory control, property control, contracts administration and financial forecasting.

2.11 Consulting Division Director

The Director of the Consulting Services Division reports to the COO. It is this Director's responsibility to organize and operate the Consulting Division in a manner that ensures that the planning and execution of consulting projects, including subcontracted work, achieves established quality, production, and financial objectives.

He reinforces the company's policy of continuous improvement in the areas of quality, safety, training, environmental compliance, and housekeeping in the Consulting Services Division. He also ensures that all consulting projects are done in accordance with applicable federal, state, and client specific program requirements.

2.12 Employee Training

In order to ensure that our clients receive the highest quality services possible, all employees are thoroughly trained in all aspects of their respective position. This training is conducted, stored, and documented in accordance with GL-HR-E-001 for Employee Training.

All new employees receive employee orientation, quality system, and chemical/safety training. Additional training is provided on an ongoing basis and includes:

- Company-wide training provided on site
- Training courses or workshops on specific equipment and analytical techniques conducted on site
- University courses
- Professional and trade association conferences, seminars, and courses conducted off-site
- Internal training to standard operating procedures (SOPs) conducted by a certified trainer. Certified trainers are employees who have documented evidence of qualification and proficiency in the process for which they are providing training.

As detailed in GL-HR-E-001, training is documented on the GEL Qualification and Verification of Proficiency Form and includes:

- Evidence that an employee has read and understood SOPs that relate the respective job responsibilities as defined by his respective group or division leader.
- Evidence that an employee has demonstrated an observable level of proficiency
- Successful analysis or preparation of a proficiency sample containing a known quantity of analytes

Documentation of training should be updated annually for all personnel. If a SOP is revised during the course of that year, training to the revised SOP must be documented.

Analytical staff must have documentation of continued proficiency for each laboratory test method applicable to their area of responsibly by at least one of the following once per year:

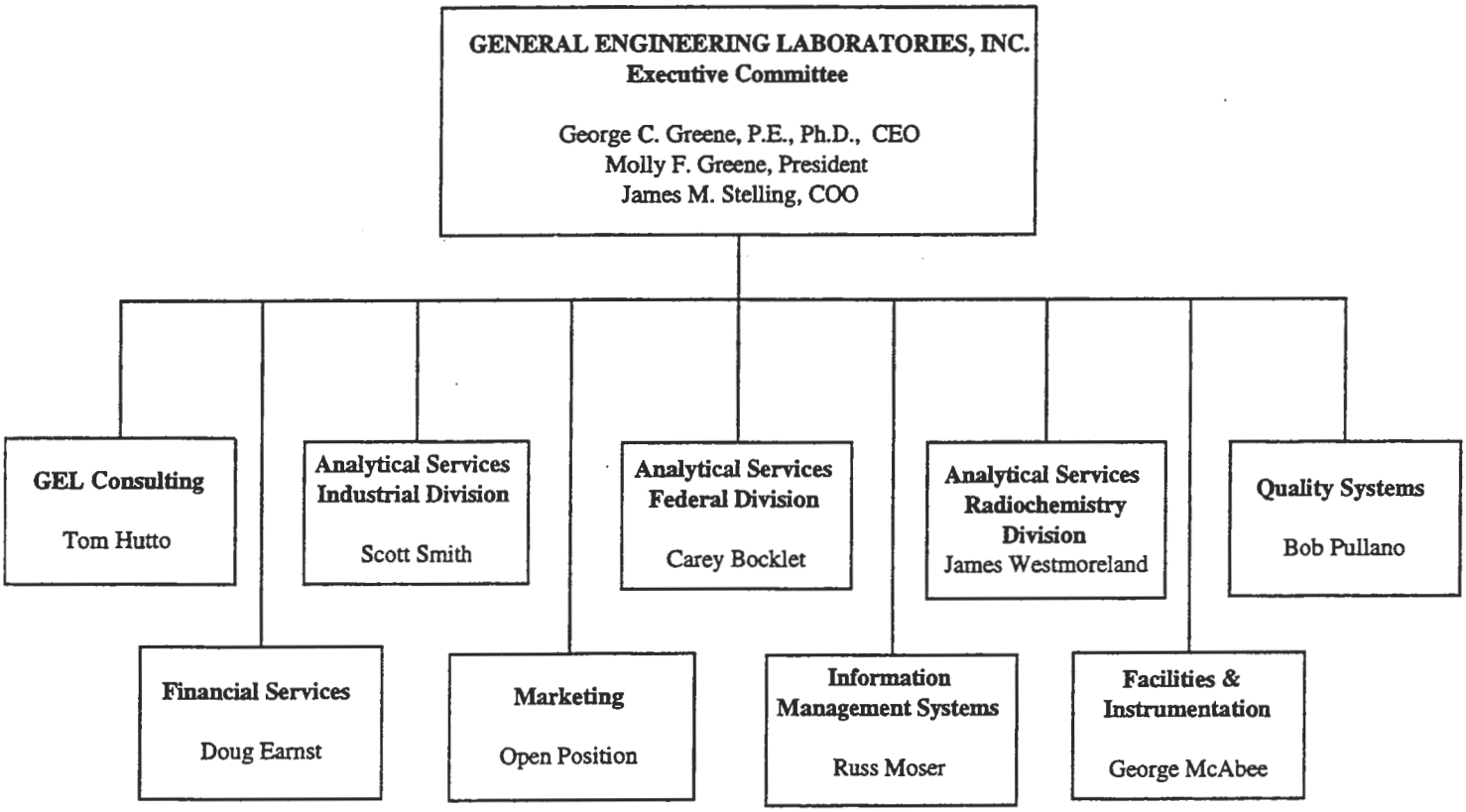
- Acceptable performance of a single or double blind sample
- Another initial demonstration of method performance

Successful analysis of a blind proficiency sample on a similar method using the same technology

- A minimum of four consecutive laboratory control samples with acceptable levels of precision and accuracy
- If the above items cannot be performed, analysis of authentic samples that have been analyzed by another trained analyst with statistically identical results.

Documentation of relevant qualifications, training, skills, and demonstrated proficiency are maintained in the Human Resources Department and on a network-computerized database.

Figure 1
Corporate Organization



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SECTION 3
QUALITY SYSTEMS

Section 3: Quality Systems

Quality systems include all quality assurance (QA) policies and quality control procedures (QC) necessary for planning, implementing, and assessing work performed by an organization.

The GEL comprehensive QA Program establishes a quality management system (QMS) that governs all activities of our organization and is not, therefore, limited to analytical operations.

The GEL quality management system is designed to conform to the requirements in the following standards:

- ASME/NQA-1
- ISO 9001/Q91
- ISO/IEC Guide 25
- US EPA Quality Assurance Staff (EPA QAMS-005/80) Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans
- Department of Energy Order 5700.6C
- Current U.S. EPA CLP statements of work for inorganic and organic analyses
- ANSI 42.2, Section 5, Measurement Quality Assurance for Radioassay Laboratories
- NELAC
- Specialized state and federal requirements.

Essential elements of our quality management system are described in this section.

3.1 Quality Management Systems Team

The quality management systems team is responsible for the management of GEL's QA Program. This team functions independently of the systems it monitors and is comprised of the Quality Systems Manager, QA officers, Quality System Specialists, and Document Control Officer.

The information below summarizes the responsibilities of each position.

3.1.1 Quality Systems Manager

- Reports directly to the Chief Executive Officer
- Serves as the management representative for quality
- Responsible for the implementation and maintenance of the QMS
- Supervises all other members of the Quality Management Team and their functions
- Initiate and recommend solutions to quality problems
- Implement appropriate actions to ensure that quality problems are controlled until solutions are implemented and verified to be effective

- Verify that solutions are implemented and that they are effective
- Thorough knowledge of the Quality System defined under NELAC

3.1.2 Quality Assurance Officers

- Report to the Quality Systems Manager
- Able to evaluate data objectively without outside influence
- Have documented training and/or experience in QA/QC procedures and knowledge of the Quality system as defined under NELAC
- Have a general knowledge of the analytical methods for which data review is performed
- Conduct internal and supplier audits under the direction of the QA Manager
- Monitor and respond to client - identified nonconformances or technical inquiries
- Coordinate external and internal performance evaluation samples
- Coordinate monitoring of deionized water system and volatile coolers
- Write or review Quality documents and/or standard operating procedures under the direction of the QA Manager
- Provide quality systems and good laboratory practices training to the organization.

3.1.3 Quality Systems Specialists

- Report to the Quality Systems Manger
- Coordinates responses to client and regulatory audits
- Acts as the point of contact for client regulatory audits
- Applies for and conducts correspondences vital to state certifications
- Responsible for the administrations of the correction action and nonconformance system
- Maintains statistical process control (SPC) system
- Collects and evaluates data for determination of capacity and amount of reanalyzes
- Monitors balances, weights, and temperature regulation of ovens, waterbaths, and refrigerators

3.1.4 Document Control Officer

- Reports to the Quality Systems Manager
- Responsible for implementing completed DIRRs to standard operating procedures (SOPs)
- Responsible for issuing all controlled documents including but not limited to QAP, QA Manual, and SOPs
- Maintains a revision log, which describes the current revisions number and revision date for all distributed documents and manuals.
- Manages the scanning and electronic storage of a instrument data

3.2 Quality Documents

The Quality Management System is planned and documented in the Quality Manual and other supporting documents, Documenting the quality system in this

manner ensures that the services provided to clients conform to specified quality criteria.
Management approves all quality documents. Departures from policies or planned activities, which affect quality and are described in the quality documents will be approved by management before they occur.

Quality documents include but are not limited to: Quality Manual, QA Plan, QA project or program plans, and standard operating procedures.

3.2.1 Quality Manual

- The Quality Manual contains eighteen quality policies covering all the requirements of the previously listed standards.
- The Quality Manual will be understood and implemented throughout the company.
- This manual is maintained and revised by the Quality Systems Manager. Revisions may be made by replacing individual policies or the entire document.

3.2.2 Quality Assurance Plan

- The Quality Assurance Plan (QAP) encompasses all of the elements of the major quality standards as they relate to laboratory operations.
- The QAP is to be understood and implemented throughout the operation to which it applies.
- This manual is maintained and revised under the direction of the Quality Systems Manager.
- Revisions may be made by replacing individual sections or the entire document.

3.2.3 Quality Assurance Program or Project Plans

- Program or project-specific criteria are specified in the QA Program Plans or QA Project plans respectively.
- These documents will be understood and implemented throughout the operations to which they apply.
- These plans are maintained by the designated manager.
- Revisions made by replacing individual sections or the entire plan.

3.2.4 Standard Operating Procedures

- Activities that affect quality are described in standard operating procedures (SOPs.)
- SOPs are prepared, authorized, and revised in accordance with SOP GL-ADM-E-001 for the Preparation, Authorization, Change and Release of Standard Operating Procedures.
- SOPs will be understood and implemented throughout the operations to which they apply.

3.3 Document Control

The preparation, revision, distribution, and identification of quality documents is critical to the effective implementation of the GEL Quality Program. As part of the GEL Quality system, this process is strictly controlled and defined in the Standard Operating Procedure for Document Control GL-DC-E-001.

As directed in this SOP, the responsibilities for document control are divided between the department managers and the Document Control Officer.

Department managers are responsible for:

- Assure and support development, distribution and maintenance of controlled documents, which are applicable to the department affiliate managed.
- Submitting all originals to the Document Control Officer and that all working copies are destroyed to prevent inadvertent use of an unauthorized document.
- Review all quality documents annually for continued validity.
- Maintain s system that verified that all affected employees are aware of revisions to documents or manuals.

The document control officer is responsible for:

- Maintaining the originals for all controlled documents.
- Maintains a revisions log which describes current revision number and revisions date for all distributed documents and manuals.
- Maintains an obsolete documents file containing a hard copy and diskette of all obsolete documents.
- Maintains a system to identify the nature of document revisions.

Each Quality Document undergoes a multi-layer review process before it is approved. The levels of review for the Quality documents identified in 3.2 are listed below:

- Quality Manual: This manual is reviewed and approved by the Chief Executive officer and the Quality Systems Manager.
- Quality Assurance Plan:

In addition to the Quality Documents mentioned in section 3.3, this SOP also applies to generation and revisions of forms.

3.4 Quality Records

Quality records are records that provide evidence that specified QA requirements have been met and provide the documentation to support results and conclusions. They are generated in accordance with the specifications of applicable procedures, programs, and contracts.

Quality records include but are not limited to:

- Observations
- Calculations
- Calibration data
- Certificates of analysis
- Certification records
- Chains of custody
- External, supplier, and internal audits
- Run logs
- Instrument data and analytical logbooks
- Instrument, equipment and building maintenance logs
- Material requisition forms
- Monitoring logs

- Nonconformance reports
- Corrective actions
- Method development and start-up procedures including method detection limit studies
- Training records
- Waste management records
- Standard logs
- Software validation
- SOPs
- Sample collection and field data

As specified by our quality policy, quality records will be:

- Generated in accordance with the specifications of applicable procedures, programs, and contracts.
- Documented in a neat legible manner.
- Stored in a manner, which protects them from loss, damage, and unauthorized alterations.
- Indexed and files in a manner which allows them to be readily retrieved.
- Accessible to the client for whom the record was generated.
- Retained for an identified time period and disposed of when this time has expired.

The generation, validation, indexing, storage, retrieval, and disposition of quality records are detailed in GL-QS-E-008 for Quality Record Management and Disposition. Quality records of subcontractor services are required to meet the conditions established in this SOP.

3.5 Internal and Supplier Quality Audits

Internal audits are conducted to verify that our operations continue to comply with the requirements of our QA program and those of our clients. Supplier audits are employed to ensure that our suppliers or subcontractors are meeting the requirements of the GEL QA program and our clients. Both internal and supplier audits are conducted in accordance with GL-QS-E-002 for the Conduct of Quality Audits.

3.5.1 Audit Frequency

Comprehensive internal audits are conducted at least annually and are scheduled by the QA team. Initial supplier audits may be conducted prior to the use of a supplier or subcontractor. Suppliers and subcontractors are reevaluated at least once every three years.

Additional internal and supplier audits may be scheduled if deemed necessary. Reasons for scheduling additional audits may include:

- Ensure that client specifications of a new contract are met.
- Requested by management due to an impending client audit.
- Significant changes have been made in the QA program.
- Deficiencies in the QA program may jeopardize the quality of an item or product.
- Declining trend is noted in the quality performance of another or organization.
- Response to client complaints
- Systemic and independent assessment of quality and/or program effectiveness is deemed necessary.

Notification of an internal audit is provided to the appropriate manager seven days prior to audit while suppliers receive a minimum thirty-day notice.

3.5.2 Audit Team Responsibilities

Internal and supplier audits are conducted by qualified auditors under the direction of the Quality Systems Manager or designee. A qualified auditor is any employee that has been trained in accordance with and meets the specification identified in the GL-QS-E-003 for Training and Qualifying Quality Assurance Personnel.

Qualified auditors will not be allowed to audit activities for which they are responsible or in which they are directly involved. It is the responsibility of the lead auditor to ensure that such conflicts of interest are avoided when he assembles the audit team. Additional responsibilities of the lead auditor are specified in GL-QS-E-001 and include:

- Preparation and distribution of the audit plan
- Formation of the audit team
- Preparation and distribution of the audit checklists and notification
- Control of audit activity
- Determination of opportunities for improvement (OFI)
- Preparation and distribution of the audit report
- Review and approve (along with the QA Manager) of OFI corrective action plans.

In addition to working the lead auditor as identified above, the QA Manager or designee is also responsible for:

- Preparation, distribution, and review of the annual audit schedule
- Review and approval of audit report and OFI tracking report.

The senior leadership team has a significant role in the internal audit process, which includes:

- Providing a team of audit personnel
- Review and approval the annual audit schedule submitted by the QA Manager
- Ensure that the auditors have sufficient authority to make the audit effective
- Develop and implement timely corrective action plans for OFIs.

3.5.4 Identification and verification of OFI

An OFI is a condition that identified a condition having a significant adverse effect on the quality of a product of service and is supported by several examples of objective evidence. The OFI could be classified as a finding, an observation, and/or a recommendation.

The lead auditor may initiate a Corrective Action Request and Report referencing the OFI. The OFI is put into the formal GEL corrective action system as described in GL-QS-E-002 for Conducting Corrective Actions.

The audit team may verify the implementation of OFI corrective action

plans by several methods depending on the root cause (NCR) of the OFI. Verifications may be include:

- Re-auditing of the deficient area
- If the OFI involved documentation, revised or new documents may be submitted to the audit team
- If the OFI does not warrant immediate action, the corrective action may be verified on the next scheduled audit.

3.6 Managerial and Audit Review

The GEL leadership team reviews the quality systems internal and supplier audit process at least once a year. This ensures the programs continuing suitability and effectiveness as well as providing an opportunity to introduce any necessary changes or improvements.

All additional review findings as well as any resulting corrective actions will be documented. The quality systems manager or quality assurance officers will be responsible for ensuring that the necessary corrective actions are implemented within the agreed time frame.

3.7 Nonconformances

Processes, items, or services that do not meet specifications or requirements are defined as nonconforming. This may include items purchased from vendors or developed in-house, samples received from clients, work in progress, and client reports.

GEL has implemented a nonconformance (NCR) reporting system to prevent the incorporation of defective goods or services into our processes and to prevent the release of nonconforming products or services to our clients.

The NCR system provides a means for documenting the disposition on nonconforming items that do not meet contractual requirements and specifications as well as communicating the occurrence of nonconformances to all persons involved in the process affected by the condition adverse to quality.

Nonconformances are documented as described in GL-QS-E-004 for Documentation of Nonconformance Reporting and Disposition and Control of Nonconforming Items.

Standard operating procedures, client complaints, and quality records including completed NCRs are reviewed regularly to promptly identify conditions, which result in or have the potential to result in situations or services do not conform to specified quality requirements.

Nonconformances are processed through the Quality Systems, categorized, and trended. Information concerning the trends is provided to the leadership team and/or group leaders of the affected areas.

3.8 Corrective Actions

There are two major forms of corrective actions at GEL. The first consists of corrective actions are implemented at the

analytical and data validation level to decrease the affect of quality control measures that do not meet the acceptance criteria. The second is the formal corrective action that is documented by the Quality Systems Department. A formal corrective action can be initiated, if, in any area of GEL, a nonconformance reoccurs or is so significant that permanent elimination of the problem is required.

Corrective actions initiated in the laboratory by the analyst or data validator in response to an immediate problem affecting an analytical batch's quality control are governed by the steps described the applicable SOP. Each analytical SOP includes information on how to handle nonconforming situations, which include but are not limited to:

- Identification of the individual(s) responsible for assessing each quality control measure and initiating and/or recommending corrective actions
- Instructions on how to assess each type of quality control.
- Descriptions on how the analyst should treat the data for an analytical batch where one or more of the quality control measures were not met. This description is specific to the affect of each type of quality control failure.
- Specifications on how to document out-of control situations and the resulting corrective actions.

This information is included in the SOPs, to ensure that data is reported only if all quality control criteria are met or all quality control measures that did not met the acceptance criteria are clearly documented.

Formal corrective actions are implemented in accordance with GL-QS-E-002 for Conducting Corrective Action. As stated in the SOP, such corrective actions provide guidance for solving problems, which cause significant or reoccurring conditions adverse to quality. Corrective actions may result from internal or external assessments. These corrective actions are documented in the Quality department on Form GEL Corrective Action Request and Report (CARR) Form 001. These forms are maintained as quality records.

The responsibilities for corrective actions are delineated in GL-QS-E-002 for team leaders and the QA Team.

Team leaders are responsible for the following:

- Ensuring that effective correction action are taken in response to internal and external performance and system quality audits and assessments, valid customer complaints, and GEL employees.
- Review CARRs originated within the department and/or division, evaluate and approve corrective action
- Authorize, request, and participate in the formation of a problem solving team to implement the necessary corrections.

- Ensure that individuals participating in the corrective action team have the knowledge needed to effectively participate on the team.

- Ensure that the corrective action and problem analysis are properly documented using CARR.

The QA Team is responsible for:

- Approving corrective action requests

- Initiates corrective actions as needed in response to internal and external performance audits, systems audits, and valid customer complaints.

- Effects change as appropriate in the GEL quality management system as a result of corrective actions.

- Approves the implementation of corrective actions.

- Verifies the completion and adequacy of corrective actions implemented through follow-up analysis

Any employee at GEL can identify and report a nonconformance and request that a corrective action be taken. All GEL employees can participate on corrective action teams as requested by the QA team or team leaders.

The procedure for conducting a corrective action as outlined in GL-QS-E-002 includes the following steps, which are discussed in detail in this SOP.

Step 1: Identify that a corrective action is needed
Step 2: Form a team to solve the problem tough corrective action
Step 3: Describe and define the problem
Step 4: Begin documenting the problem analysis and resolution process
Step 5: If necessary disposition a nonconformance as described in 3.7.
Step 6: Define and verify the root cause of the problem.
Step 7: Select likely causes and identify alternate solutions
Step 8: Verify corrective actions before making them permanent
Step 9: Implement permanent corrective actions
Step 10: Prevent reoccurrence.

3.9 Performance Audits

In addition to internal and client audits, our laboratory is participates in performance audits. Performance audits ensure the quality of the analytical activities by implementing checks to monitor the quality of results.

GEL participates in the following types of performance audits:

- Participation in proficiency testing and other interlaboratory comparisons.

- Adherence to performance requirements necessary to retain certifications listed in Appendix D.

- Use of certified reference material and/or in-house quality control using secondary reference materials and the evaluation of the recoveries of

these materials against statistical process control data.

- Replicate testing using the same analytical methods and the evaluation of the relative percent difference between measurements through the use of statistical process control data.

- Implementation of quality control procedures that use statistical process control data.

- Evaluation of results for different parameters to ensure that, where possible, all data is in agreement.

We participate in a number of proficiency testing programs including those for federal, state, and others required by contract. Prior to the elimination of the EPA WS and WP proficiency testing programs, we participated in both twice a year. We continue to analyze both drinking water and wastewater proficiency samples ate least twice a year by participation in the State of New York Department of Health Potable Water Proficiency Testing Program and the State of New York Non-Potable Water and Hazardous Waste Proficiency Program.

In addition to the State of New York Proficiency Programs, we participate in all programs necessary to maintain the certifications listed in Appendix D and fulfill contractual obligations. This participation includes but is not limited to the following proficiency programs:

- United States Environmental Protection Agency Discharge Monitoring Report, Quality Assurance Program (DMR-QA)
- Department of Energy Mixed Analyte Performance Evaluation Program (MAPEP)
- The United Stated Army Corps of Engineers Laboratory Performance Evaluation Program
- National Institute for Occupational Safety and Health (NIOSH) Proficiency Testing (PAT)
- Analytical Standards Inc. Proficiency Testing
- State of North Carolina Department of the Environment, Health and Natural Resources Laboratory Certification Performance Evaluation Program.
- State of California Hazardous Waste Proficiency Testing
- ERA's INterLab RadCheM Proficiency Testing Program for radiological analyses.

In addition to proficiency testing, we evaluate our analytical performance on a daily basis through the use of statistical process control acceptance criteria. Where feasible, this criteria is applied to both measures of precession and accuracy and is specific to sample matrix. GEL establishes process control limits at least annually. Radiochemical analyses evaluate quality control based on static limits rather than those that are statistically derived.

The statistically derived process control limits are listed in Appendix E.

Precision is measured through the use of matrix duplicates and/or matrix spike duplicates.

The upper and lower control limits (UCL and LCL respectively) for precision are plus or minus three times the standard deviations from the mean of a series of relative percent differences. The static precision criteria for radiochemical analyses is 0 -20% for activity levels exceeding the contract reporting detection limit (CRDL.)

Accuracy is measured through the use of laboratory control samples and/or matrix spikes as well as surrogates and internal standards.

The UCL and LCL for accuracy are plus and minus three times the standard derivation from the mean of a series of recoveries. The static limits for radiochemical analyses is 75 - 125%.

If the recovery of a LCS, surrogate, or matrix spike ifs found to fall outside the acceptance criteria, the process is said to be out of control. This is also true if the RPD is outside the limits for precision.

Instructions for handling out-of control situations are provided in the applicable analytical SOP.

3.10 Essential Quality Control Measures

The quality control measures required to ensure the reporting of accurate data is to some degree method-specific. There are, however, quality control measures whose implementation is essential to our quality system. Essential quality measures for chemical testing, microbiology, and radiochemical testing are described in Appendix F. The quality control measures essential to our quality system include but are not limited to:

- Monitoring of negative and positive controls.
- Defining the variability and reproducibility through the use of duplicates
- Ensuring the accuracy of the test data including calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples, or other measures
- Evaluating test performance using method detection limits and quantitation limits or range of applicability such as linearity;
- Selecting the appropriate method of data reduction
- Selecting and using reagents and standards of appropriate quality;
- Assuring the selectivity of the test for its intended purpose
- Assuring constant and consistent test conditions
- Assessing and evaluating quality control measures on an on-going basis
- Using quality control acceptance limits to determine the usability of the data
- Developing procedures for acceptance criteria
- Ensuring that quality control protocols specified in our SOPs are implemented.

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SECTION 4
FACILITIES

Section 4: Facilities

GEL laboratories are designed with the full-service approach to handling environmental needs. The layout is structured to provide dedicated space for radiochemical analyses, organic extractions, semivolatile organic analyses, volatile organic analyses, metals analyses, general chemistry analyses, and air analyses.

The laboratories and support offices occupy approximately 73,000 square feet specifically designed to meet the stringent quality control and utility requirements of the modern environmental laboratory. Records are stored in a separate, climate-controlled building on-site. The diagrams in Appendix G depict the layout of the laboratories.

Discussed in this section are:

- Facility security
- Utility services and deionized water
- Prevention of contamination
- Assessment of contamination

4.1 Facility Security

GEL features secured laboratory and storage areas. Restricted entry ensures the maintenance of sample integrity and client confidentiality which are critical to meeting the needs of our clients. Entry is restricted to authorized personnel and requires the scanning of a coded identity card. Visitors must be escorted through the laboratory by authorized GEL personnel. A s second level of security is in place through the use of a sample custodian to deliver individual samples and a bar code internal chain-of-custody system.

4.2 Utility Services

The following utilities are provided in each laboratory:

- Cold Water
- Hot Water
- Deionized Water
- Compressed Air
- Natural Gas
- Vacuum
- 110 Volt AC
- 208 Volt AC (at selected stations)
- Specialty gases - as required

4.2.1 Deionized Water

GEL has two independent, but identical, deionized water (DI) systems. One system serves radiochemistry and the other serves the remaining analytical laboratory areas. Each system is capable of distributing water throughout their respective areas through schedule 40 PVC pipe. DI water is prepared from city water flowing through a deionization water system, capable of producing 5 gallons per minute of Type II laboratory water.

The minimum requirements for Type I and Type II DI water are listed in the tables below.

Table 1: ASTM Type I DI Water

Quality Parameter	Limits
Bacteria, CFU/mL	<10
pH	not specified
Resistivity, min. MΩ-cm at 25C	>16.67
Conductivity, max. µmho/cm at 25C	≤0.06
Trace Metals, Single (Cd,Cr,Cu,Ni,Pb, Zn)	< 0.05 mg/L
Trace Metals, Total	< 0.1 mg/L
Free Chlorine	not specified
Ammonia/Organic Nitrogen	not specified
TOC	not specified
Organic contaminants	Activated carbon

Table 2: ASTM Type II DI Water

Quality Parameter	Limits	Frequency
Bacteria, CFU/mL	< 1000	monthly
pH	not specified	monthly
Resistivity, min. MΩ-cm at 25C	> 1.0	daily
Conductivity, max. µmho/cm at 25C	≤1.0	daily
Trace Metals, Single (Cd,Cr,Cu,Ni,Pb, Zn)	< 0.1	quarterly
Trace Metals, Total	not specified	quarterly
Free Chlorine	< 0.1 mg/L	monthly
Ammonia/Organic Nitrogen	< 0.1 mg/L	monthly
TOC	< 1.0 mg/L	monthly
Organic contaminants	not specified	annually

Compliance with the above limits is monitored as specified in GL-LB-E-016 for Collection and Monitoring the DI Water Systems. This monitoring includes:

- Daily conductivity checks of DI water collected from designated sites in the laboratory.
- Twice daily measurements of Type I system resistivity
- Monthly, quarterly and annual monitoring of Type II DI water systems' compliance to quality parameters. The measurement frequency as indicated in Table 2.

4.3 Prevention of Contamination

Work areas that are free of sample contaminants and constituents or measurement interferences are imperative to the generation of quality data. GEL's laboratories are designed to prevent contamination and the implementation of good laboratory practices reinforces this design.

In addition to keeping work areas clean and free of dust and dirt accumulations, policies and features that prevent or minimize contamination include:

- Air conditioning system that controls the environment of individual laboratories to insure optimum performance of sensitive instrument and to eliminate potential cross contamination.

- Segregation of volatile and semivolatile laboratories to minimize potential contamination associated with the use of common required solvents.

- Negative and positive pressure air locks to isolate selected laboratories to prevent entry of airborne contaminants.

- Fume hoods to remove fumes and to reduce risk of aerosol and airborne contaminants and of personnel safety hazards. The performance of our fume hoods is routinely monitored in accordance with GL-FC-E-003 for Fume Hood Face Velocity Performance Checks.

- Restricted access to volatiles laboratory to authorized personnel only.

- Designated area for glassware preparation where all glassware used in sample preparation and analysis is cleaned to the specifications of the particular analysis method. Glassware is cleaned in accordance with GL-LB-E-003 for Glassware Preparation.

- Segregated storage areas for volatile and radioactive samples as discussed in Section

- Production and use of Type I and Type II DI water. The quality of the DI water is monitored in accordance with GL-LB-E-06 for Collection and Analysis of DI Water Systems.

4.4 Assessment of Contamination Levels

Contamination resulting from the following sources is evaluated on the basis of quality assurance and quality control data derived from the analytical method and holding or method blanks.

- Sample containers
- Reagent water
- Reagents and solvents
- Sample storage
- Chemical and physical interference
- Constituent carryover during analysis

If there is evidence of contamination, actions are taken to identify and eliminate the source.

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SECTION 5
EQUIPMENT and REFERENCE MATERIALS

Section 5: Equipment and Reference Materials

Our ability to efficiently generate data that is reproducible, accurate, and legally defensible is dependent upon the documented use of high-quality instrumentation, equipment, and reference materials.

Provided in this section are:

- General policies governing GEL's instruments, equipment, and reference materials
- Identification of instrumentation and support equipment
- Procurement protocol

5.1 General Policies

It is GEL's policy to only purchase state-of-the art instrumentation and equipment and high-quality reference materials that meet or exceed the method and regulatory requirements of all analyses for which we are accredited. If the needed arises for instrumentation or equipment not under our permanent control to be used, GEL ensures that it also meet these requirements and standards.

Instrumentation and equipment will be placed into service on the basis of its ability or meet method or regulatory specified operating conditions such as range and accuracy.

All laboratory instrumentation and testing equipment will be maintained in accordance with the standard operating procedures (SOPs) to confirm that they are operating properly.

Instrumentation and equipment will be used in a manner that will ensure that measurement uncertainty is known and consistent with specified quality requirements.

Any instrument or equipment shall be taken out of service and labeled accordingly under the following conditions:

- Mishandling and/or overloading of the equipment
- Produces results that are suspect
- Has been shown by verification or otherwise to be defective

The affected instrument or equipment will remain out of service until it has been repaired and shown by calibration, verification, or test to perform satisfactory. Instruments that are in service and normally calibrated prior to and during use will not be tagged.

Each item of equipment including reference materials shall, if appropriate, be labeled, marked or otherwise

identified to indicate its calibration status.

Records are maintained for each major item of equipment, instrumentation, and all reference material significant to the performance of analysis. These records may be in the form of maintenance logs which are kept in accordance with GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other record Keeping Devices.

Documentation included in these records includes but is not limited to:

- Equipment name
- Manufacturer's name
- Type identification
- Serial number or other unique identification
- Date received and date placed in service (if available)
- Current location
- Condition when received (if available
- Manufacturer's instruction, where available
- Dates and results of calibrations and or verifications
- Date of the next calibration and/or verification
- Details of maintenance carried out to date and planned for the future
- History of any damage, malfunction, modification or repair

5.2 Instrumentation and Support Equipment

A list of the instruments used for analysis of environmental samples at GEL is provided in Appendix I. Where feasible, instruments are equipped with autosamplers that improve efficiency and facilitate consistent sample introduction to the sample detector as well as being connected to a local area network to facilitate data transfer.

Devices that may not be the actual test instrument but are necessary to support laboratory operations are referred to as support equipment. It is crucial that this equipment be maintained in proper working order.

Support equipment utilized at GEL includes:

- balances
- ovens
- refrigerators
- freezers
- incubators
- water baths
- temperature measuring devices
- volumetric dispensing devices
- muffle furnaces
- distillation apparatus
- grinders and homgenizers
- hot plates and heating mantles
- ultraviolet sterilizers.

Guidelines for the required calibration and evaluations of this equipment are discussed in Section 7.

5.3 Procurement and Control of Purchased Items

Materials and services that affect the quality of our services are designated as Quality Materials and Services and will only be purchased from approved suppliers. Suppliers are approved as described in GL-QS-E-001 for Conduct of Quality Audits.

Records of specific quality requirements for Quality Materials and Services as well as the evaluation of approved suppliers are established and maintained.

Such specifications, which document the quality of a product or service, can include:

- certificates of tractability
- verifications of chemical quality
- certificates of analysis
- inspections of equipment or materials
- verifications or inspections of vendor product specifications

Our procedures for requisitioning supplies, instruments, equipment and other common use material are described in GL-RC-E-002 for Material Requisition Form Procedure. The requests are submitted on Material Requisitions Form gelf031.

Information on a completed requisition form includes:

- Name of person or persons requesting materials
- Date of requisition
- Account, department, and project number to which the material is to be billed
- Recommended supplier or vendor
- Additional information necessary to expedite this purchase request
- Specifications that could affect the quality of Gel's product
- Vendor's material part number
- Amount of material needed
- Description of material
- Cost per unit
- Person or persons authorizing the purchase
- Time frame in which the material is needed

All purchased items including equipment, instruments, and reference materials are inspected upon receipt at GEL in accordance with GL-RC-E-001 for the Receipt and Inspection of Material and Services. This inspection is to verify that procured items meet the acceptance criteria defined in the procurement documentation.

The initial inspection and receipt of items is the responsibility of the Receiving Department and include:

- Opening and inspecting all items for damage
- Comparing the items with the GEL-issued purchase order or contract for catalog or part number, description or procurement specification, quality, requirement, and acceptance criteria.
- Labeling items with a limited shelf life with the date received.

- Determining if the items conform to the specifications agreed to by the vendor.

The individual responsible for technical acceptance of the item is responsible for ensuring that the Purchasing Department receives and provides the proper acceptance documentation.

Items found not to conform to these standards are returned to the supplier, identified as nonconformances, or disposed according to established procedures in GL-QS-E-004 for Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items.

SECTION 6
HEALTH and SAFETY

Section 6: Health and Safety

GEL is committed to the maintaining a safe work environment and promoting healthy work practices. GEL's corporate Safety, Health and Chemical Hygiene Plan was developed by our resident certified industrial hygienist. Procedures outlined in this plan are consistent with Occupational Safety and Health Administration, the National Institution of Occupational Safety and Health Regulations, CERCLA, Environmental Protection Agency, and SCDHEC. All employees receive training in safety practices applicable to their specific job functions. This training is conducted in accordance with GL-HR-E-002 for Employee Training.

Discussed in the section are:

- Fire safety and fire safety equipment
- Other safety equipment
- Safety equipment and procedures related to handling radioactive samples

6.1 Fire Safety

GEL is equipped with a fire system designed to detect sample in certain high-risk areas. These areas are quipped with a bell and buzzer warning system. An automatic halon extinguisher system is located in the cold and ambient storage areas, organic sample preparation laboratory, hazardous waste laboratory, and solvent storage.

Fire blankets and dry chemical fire extinguishers are located at strategic points throughout the laboratory. These extinguishers are routinely inspected in accordance with GL-FC-E-004 for Inspection of Fire Extinguishers. All laboratory personnel are trained in the proper use and selection of fire extinguishers.

In order to decrease the risk of fire, special cabinets have been installed for the storage of acids and solvents that are utilized daily. A concrete storage room located next to the stock room is used for bulk storage of solvents.

6.2 Safety Equipment

Safety equipment is available to all employees as needed including safety glassed, lab coats, safety goggles, protective gloves, hard hats, and coveralls. Respirators are provided on an as needed basis to those who have completed training in the use of this specialized equipment.

Eye wash stations and overhead showers are located throughout the laboratory. Both are routinely inspected as directed in GL-FC-E-002 for Testing of Emergency eyewash and Shower Equipment.

6.3 Radiation Safety

Since GEL specializes in the handling of radioactive material, health physics procedures have been implemented to ensure the safe handling of radioactive material in the facility.

While laboratory personnel encounter insignificant levels of occupational radiation exposure, each employee in the radiochemistry division and selected laboratory and field personnel wear a thermo luminescent dosimeter (TLD) during work hours. These dosimeters are exchanged quarterly to TMA Eberline in Santa Fe, New Mexico, and exposure records are maintained by the Radiation Safety Officer.

Exposure limits protocol for handling excess exposure and instructions for the proper use of dosimeters is addressed in GEL-EPI-E-S009 for Dosimetry Procedures.

Eighteen area dosimeters have been placed in key locations throughout the facility to track area exposures. These dosimeters are exchanged quarterly.

Upon receipt, we take special precautions to ensure samples are safely processed. Environmental samples suspected of containing low level radioactive contamination, including any sample from a Department of Energy facility, are monitored by trained personnel using Geiger-Mueller survey to determine dose rate. Protocols for surveying suspected or known radioactive samples and the receipt of radioactive samples are detailed in GL-EPI-E-S001 for Radiation Survey Procedures and GL-EPI-E-S007 for Receiving Radioactive Samples. Specific features of this process are addressed in Section 9.

Upon leaving any radiological control area all personnel check their hands and feet for potential contamination. This is performed with a Geiger-Mueller type pancake probe and standard survey meter following the protocol identified in GL-EPI-E-S001 for Radiation Survey Procedures.

Several key areas throughout the facility are surveyed on the following frequencies:

- Radiochemistry Labs - Monthly smears
- Radioactive Sample Storage Areas - Monthly smears and dose rate
- Login - Monthly smears and dose rate

Source material is controlled by storage in a secured area. Total source activities are tracked and decay corrected through a data base management program. Two reports are readily generated for review. One report gives the total activity of standards in house. The second report inventories standards that have been consumed through analytical operations.

SECTION 7
MEASUREMENT, TRACEABILITY, and CALIBRATION

Section 7: Measurement Traceability and Calibration

The traceability of measurements and the calibration of testing equipment are imperative to the production of accurate and legally defensible data. Recognizing this fact, GEL has established and implemented procedures that ensure equipment calibration and measurement verification are traceable to national recognized standards.

Traceability to national standards of measurement is provided, where possible, by calibration certificates. Calibration certificates provide the measurement results and associated uncertainty of measurement and/or a statement of compliance with identified specification. Records of calibration certifications are maintained in the affected area of the laboratory and the quality department.

There are situations where traceability to national standards is not applicable. In these cases, verification of measurements are provided by participation in an appropriate program of interlaboratory comparisons, proficiency testing, or independent analyses.

Measurement and traceability calibration policies and procedures are described in this section for the following:

- Calibration criteria for support equipment
- General requirements
- Balances
- Temperature sensitive devices and temperature monitoring
- Air displacement pipets
- Calibration criteria for instruments
- Calibration verification
- Initial calibration verification
- Continuing calibration verification

7.1 Calibration Criteria for Support Equipment

The calibration protocols of specific support equipment including balances, temperature-sensitive equipment, and air displacement pipets are addressed in this section. There are, however, general criteria that apply to the calibration of support equipment.

General specifications affecting support equipment are listed below.

- Equipment should be maintained in proper working order. The records of all maintenance activities including service calls shall be kept.

- Calibrations or verifications over the entire range of use, using NIST traceable references when available, should be conducted annually.

- If the results of calibration and verification are not within the specifications required by the equipment's application then:

  1. The equipment will be removed from service until repaired

  2. Under certain conditions, a deviation curve will be prepared. All measurements will be corrected for the deviation, recorded and maintained

- Prior to use on each day, balances, ovens, refrigerators, freezers, incubators and water baths will be checked with NIST traceable references (where possible) in the expected use range.

- If prescribed by the test method, additional monitoring shall be performed for any device that is used in a critical test (such as incubators or water baths).

- The support equipment is used only if the reference standards specifications (as provided by the supplier or described in the analytical method) are met.

- Reference standards of measurement such as Class S or equivalent weights or traceable thermometers will be used for calibration only unless it can be demonstrated that their performance as reference standards will not be invalidated.

- Reference standards of measurement will be calibrated by a body that can provide, where possible, traceability to a national standard of measurement.

- Reference standards and measuring and testing equipment are, where relevant, subjected to in-service checks between calibrations and verifications.

- Reference materials will, where possible, be traceable to national or international standards of measurement, or to national or international standard reference materials.

- Mechanical volumetric dispensing devices (except Class A glassware) shall be checked for accuracy on monthly.

7.1.1 Balances

Our balances are under a service contract for annual calibration, maintenance and cleaning. Each balance is labeled with a serial number, service date, date of next scheduled service, and signature of service technician.

Balances are set-up, calibrated, and operated in accordance with GL-LB-E-002 for Balances in the range required by the analytical procedure. Prior to the use of a balance, the analyst is responsible for checking the calibration of this equipment.

The calibration and calibration verification are performed using weights that are or have been calibrated against Class S weights. Our set of Class S weights is traceable to NIST and is calibrated annually by the State of South Carolina Department of Agriculture or another independent agency.

Calibration and calibration verification are recorded in the balance's calibration logbook. If the calibration or calibration verification does not meet the acceptance criteria specified in each logbook, the balance is recalibrated. If the calibration criteria are still not met, the balance is removed from service and tagged accordingly.

7.1.2 Refrigerators, Freezers, Ovens, Incubators, Water Baths and Other Similar Devices

Careful control of temperature is often central to the production of acceptable data. Temperature excursions beyond established limits may invalidate the procedure and associated data. GEL ensures that regulatory and/or method temperature requirements are met by constant monitoring in accordance with GL-LB-004 for Temperature Monitoring and Documentation for Refrigerators, Freezers, Ovens, Incubators, and Other Similar Devices.

Temperatures are measured using thermometers that are calibrated annually against NIST traceable thermometer. The NIST traceable thermometers are independently calibrated at least once per year. The protocol for thermometer calibration is described in GL-QS-E-007 for Thermometer Calibration. The temperature of the following equipment is monitored as described in GL-LB-004:

- Refrigerators and freezers used to store samples, standards, and other temperature sensitive materials
- Incubators
- Ovens
- Water Baths
- Autoclaves

Temperatures of refrigerators and freezers are monitored at least daily at an interval of no less than 4 hours.

Temperatures of ovens, water baths, and other devices used as part of an analytical process must be monitored prior to, during, and immediately after use.

Incubators or other devices used for microbiological or other specialized analytical methods may require more frequent monitoring as specified in the appropriate SOP.

Temperature measurements are documented on temperature logs specific to each piece of equipment. Temperature logs are posted on each refrigerator, freezer, waterbath, oven or other temperature control device.

Each temperature log includes the following information:

- Date and time of each measurement
- Initials of person making the measurement

- Acceptance limits for device being monitored
- Whether or not the device was found to conform with specifications, at time of measurement
- Name, location and number of device being monitored
- Name and telephone number of person to contact in event of device failure
- Notation of any out of control condition

The sterilization pressure of each autoclave run must be documented in addition to the sterilization temperature.

Whenever the process to maintain and document temperatures within acceptance limits does not conform to specifications, a GEL nonconformance report (NCR) will be issued. Appropriate action will be taken to dispositioning the nonconformance according to SOP GL-QS-E-004 for Nonconformance Identification Control, Documentation, Reporting, and Dispositioning.

Nonconformance that should be documented include:

- Failure to maintain process temperature within the acceptance limits
- Failure of measurement device to achieve calibration
- Total failure of temperature control device
- Failure to monitor the temperature as required

7.1.3 Air Displacement Pipets

Air displacement pipets offer a level of precision and accuracy exceeded only by Class A transfer pipets. In addition, these pipets eliminate the possibility of cross-contamination since they use disposable tips.

Air displacement pipets are calibrated monthly using five replicate measurements of a frequently used volume setting in accordance with procedures described in GL-LB-E-010 for Maintenance and Use of Air Displacement Pipets. As specified in this SOP, the calibration of an air displacement pipet is verified daily prior to its' use. This verification is based on a single point measurement.

The acceptance criteria for each measurement are based on the standard deviation of the five calibration measurements. Tolerance limits for commonly used verification volumes and accuracy and precision checks are included in the pipet calibration logbook.

Calibration and daily calibration verifications are traceable to a specific pipet using the pipets' unique identification found on its label.

If a pipet does not meet the calibration tolerance limits, its is removed from service until it can again demonstrate compliance after being cleaned and/or repaired.

All analysts whose job functions may require air displacement pipets are

rained in their proper use and calibration prior to use.

7.2 Instrument Calibrations

In order to ensure that the data generated by an instrument is accurate, it is necessary to calibrate the instrument using standards containing known concentrations of target analytes. GEL verifies the accuracy of the calibration standards by analyzing an additional standard containing the target analytes. This initial calibration verification standard (ICV) originates from a second source. The stability of the instrument over the calibration range is verified by the analysis of a continuing calibration verification standard (CCV.)

The traceability of all calibration, calibration verification, and other quality control standards to the recognized standards is documented as described in GL-LB-E-007 for Laboratory Standards Documentation. This GEL protocol involves assigning individual identification numbers to each source standard as well as each subsequent intermediate and working standard prepared.

The identification number makes it possible to trace a standard to any parent standard and ultimately to the source standard. The date each standard is prepared, the recipe used in the standard preparation, the person preparing the standard, and the standards expiration date are all documented in the appropriate standards log. All of this information is accessible by the standard identification number. This number is recorded on the instrument run logs, analytical logbooks, sample preparation logs, and instrument raw data. As a result, any standards used in the analysis of a particular sample or group of samples can be traced to NIST, US EPA, or other nationally recognized standards.

The calibration of specific instruments for defined methods of analysis are described in the applicable analytical SOP. There are, however, several general guidelines that govern instrument calibration and include:

- Initial calibrations are verified with a standard obtained from a second source unless one is not available.

- Verification standards (ICV and CCV) are analyzed with each initial calibration and shall be within 15% of the true value unless historical data has demonstrated that wider limits are applicable.

- Calibration curves are prepared as specified in the analytical reference method.

- If a test method does not address the preparation of a calibration curve, the appropriate number of standards to be used in the initial calibration is based on the percent relative standard deviation (%RSD.)

  The %RSD is determined from either seven replicate measurements of a standard whose concentration is close to the lowest anticipated calibration standard or by performing a calibration linearity test. The

linearity test, such as response or calibration facts, should be based on at least three standards having concentrations that cover the expected calibration range.

The minimum number of standards to be used in the initial calibration is dependent on the resulting %RSD. The number of calibration standards to be used in conjunction with a blank is indicated in Table 1. If the resulting curve is nonlinear, additional standards should be used.

If the %RSD is less than 2, it is GEL's policy to still use at least three calibration standards.

Table 1: % RSD and Calibration Standards

%RSD	Number of Calibration Standards
2 - < 10	3
10 - < 25	5
> 25	7

- Calibration curves are subjected to a calibration linearity test, such as a linear regression or percent RSD of response factors (internal standard calibration) or calibration factors (external standard calibration). The additional requirements are dependent upon the analytical method.

- If, over the calibration range, the RSD or response factor is less than 15% or the RSD of calibration factors is less than 30%, linearity through the origin can be assumed. In these cases, an average relative response factor may be used. If these conditions are not met, the complete calibration curve should be used.

- If a linear regression is used, the correlation coefficient shall be no less than 0.99.

- For results to be reported as quantitative, they must be bracketed by calibration or calibration verification standards. Results not meeting this condition must be reported as having a lower confidence level.

7.3 Calibration Verification

Unless otherwise specified by the analytical test method or demonstrated through historical data, the recovery of target analyte(s) in initial and continuing calibration verification standards shall be between 85 - 115%. Additional requirements of initial and continuing calibration verifications are discussed below.

7.3.1 Initial Calibration Verification (ICV)

- If an initial calibration curve is not established on the day of analysis, the integrity of this curve should be verified on each day of use or every 24-hour period. This verification

requires that a blank and standard from a second source be analyzed initially. The standard concentration should be at the method defined level. If not specified by the method, a standard at a mid-level concentration may be used.

- If the initial calibration verification does not meet the acceptance criteria, the analysis procedure is stopped, evaluated, and appropriate corrective measures taken. The initial calibration verification must be acceptable before any samples are analyzed.

7.3.2 Continuing Calibration Verification (CCV)

Additional standards shall be analyzed after the initial calibration curve or the integrity of the initial calibration curve has been accepted. These standards are designated as continuing calibration verification standards (CCVs.)

CCVs shall be analyzed at a frequency of 5% or every 12 hours whichever is more frequent. If the instrument consistently drifts outside acceptance criteria before the next calibration, this frequency is increased.

CCVs may be the from the same source as the calibration standards or a second source. The concentration of CCVs shall be determined by the anticipated or known concentration of the samples and/or method specified levels. At least one CCV shall be at a low level concentration.

To the extent possible, the samples in each interval (i.e. every 20 samples or every 12 hours) are bracketed with CCV concentrations closely representing the lower and upper range of reported sample concentrations. If this is not possible, the standard calibration checks should vary in concentration throughout the range of the data being acquired.

If the recovery of a CCV does not meet the acceptance criteria and routine corrective action procedures fail to produce a second consecutive check within acceptance criteria, a new initial calibration curve should be constructed.

If the CCV acceptance criteria are exceeded high and there are non-detects for the corresponding analyte in all environmental samples associated with the CCV, then those non-detects may be reported.

If samples are found to contain target analytes that exceeded the associated quantitation limits and the CCV recovery does not meet the acceptance criteria, the affected samples shall be reanalyzed. This reanalysis occurs after a new calibration curve has been established, evaluated and accepted.

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SECTION 8
ANALYTICAL METHODS and STANDARD OPERATING PROCEDURES

Section 8: Analytical Methods and Standard Operating Procedures (SOPs)

The laboratory services provided by GEL include the determination of a wide array of parameters including volatile organics, extractable organics, metals, general inorganic/wet chemistry, radiochemistry, and limited microbiology. The procedures used to determine these parameters are consistently executed due to our extensive system of standard operating procedures (SOPs) and dedication to the training of analytical personnel to perform analyses as directed in the respective SOPs.

A list of our standard operating procedures and their corresponding analytical methods where applicable is provided in Appendix I.

Discussed in this section are:

- Selection of analytical methods
- Standard operating procedures
- Method validation and initial demonstration of capability
- Sample aliquots
- Data verifications
- Standard and reagent documentation and labeling (Refer to Section 10.1)
- Computers and electronic data requirements

8.1 Selection of Analytical Method

Methods used in sample preparation or analysis are selected to meet the specific needs and requirements of the client while providing the data that is scientifically valid. When the use of specific test methods are mandated, only those methods will be used. If an analysis cannot be performed by a client-requested method, the client will be notified. Method substitutions will not performed without the client's consent.

If method modifications must be implemented, the client will be notified of the necessary procedural changes. It is recommended that clients submitting data to regulatory agencies obtain the respective agency's approval of method modifications.

8.2 Standard Operating Procedures (SOPs)

Each parameter is determined by protocol detailed in its corresponding SOP. The defined protocol originates from the analytical method or methods referenced in the SOP and may incorporate regulatory and client requirements.

Descriptions of analytical methods employed by GEL can be found in the applicable reference listed below:

- EPA SW846 Third Edition ,Revision III

- EPA/600/479/020
- Official Methods of Analysis of the Association of Official Analytical Chemists (AOAC)
- American Society for Testing and Materials (ASTM)
- Standard Methods for the Examination of Water and Wastewater (SM)
- South Carolina Department of Health and Environmental Control (SCDHEC)
- Code of Federal Regulations (CFR) Titles 40 and 49
- Department of Energy Environmental Measurements Laboratory (EML)
- Los Alamos Health and Environmental Chemistry (LAHEC)
- DOE
- HASL
- EPA CLP

In addition to the above listed references, many radiochemistry procedures were developed in conjunction with Florida Sate University (FSU) under the guidance of Dr. Bill Burnett.

To ensure and document that each operational system and analytical procedure is performed in standard and uniform manner, manuals containing SOPs are made available to each section of the laboratory. These laboratory method manuals contain SOPs pertaining to a specific section of the laboratory and are controlled as described in GL-DC-E-001 for Document Control.

SOPs are developed and written in accordance with GL-ADM-E-001 for Preparation, Authorization, Change and Release of Standard Operating Procedures. Prior to implementation, SOPs receive both a technical and quality review.

The technical review may be conducted by a senior analyst, group leader, or data validator and ensures that the procedures included in the SOP are technically sound and in accordance with the referenced method(s.)

Performed by members of the Quality Systems Management team, the quality review is independent of the laboratory. This review ensures that all quality requirements of the method, regulatory agencies, and GEL are adequately and accurately identified in the SOP.

Once implemented, any SOP procedure may be modified. Modifications generally occur when:

- There is a change in instrumentation or equipment
- Error identified in the original SOP
- Improvements in technology and/or reagents need to be incorporated
- The reference method has been revised or discontinued in favor of another method.

Proposed revisions are submitted for review on Documentation Initiation and Revision Request (DIRR) forms. No change is implemented without both a technical and quality review. If

requested and specified by contract, clients may review the revisions before they are implemented in the analysis of their samples.

All standard operating procedures are reviewed annually and revised as necessary.

Analytical SOPs in the laboratory method manuals contain or reference SOPs that contain:

- reference method
- applicable matrix or matrices
- method detection limit
- scope and application including parameters to be analyzed
- method summary
- definitions
- interferences and limitations
- specific safety requirements
- required equipment and supplies
- reagents and standards
- sample collection, preservation, shipment, and storage
- quality control
- calibration and standardization
- procedure
- calculations
- method performance
- pollution prevention
- data assessment and acceptance criteria for quality control measures
- corrective actions for out of control or unacceptable data
- waste management
- references
- any table, diagrams, flowcharts, and validation data
- identification of any modifications we have made to the published procedure

8.3 Method Validation and Initial Demonstration of Capability

GEL requires that an initial demonstration of method performance is required prior to the implementation of any new analytical method and any time that there is a significant change in instrumentation or methodology.

Exempted from this requirement are microbiological analyses and any tests for which spiking solutions are not available. Analyses that are exempted include but are not limited to those for the determination of:

- total dissolved, total suspended, total volatile, and total solids
- pH
- odor
- color
- free liquids
- temperature
- dissolved oxygen
- turbidity.

This initial demonstration is conducted as described below in 8.3.1. The Quality Systems Management Team in accordance with GEL-QS-E-008 maintains records and supporting data pertaining to the demonstration for Quality Records Management and Disposition. The records are available upon request.

After initially demonstrating our ability to perform a specific analysis, we continue demonstrate method performance by the analysis of laboratory control samples and performance evaluation samples.

8.3.1 Procedure for Initial Demonstration of Capability

The initial demonstration of capability for mandated analytical or EPA reference test method is conducted following the procedure outlined below. This procedure is adapted from EPA test method published in 40CFR part 136, Appendix A.

Certification statements acknowledging this capability are maintained in the Quality Department and placed in the personnel records of each affected employee.

Step 1: A quality control sample is obtained from an outside source if possible. If one is not available, this sample may be prepared internally using stock standards that are prepared independently from those used in instrument calibration.

Step 2: The quality control sample is diluted in a volume of clean matrix to a concentration approximately 10 times the method-stated or method detection limit determined in accordance with GL-LB-E-001 for Determination of Method Detection Limits. Sufficient volume of this diluted quality control sample is prepared so that at least four aliquots of the required method volume

Step 3: Four aliquots of the diluted quality control sample are prepared and analyzed according to the analytical test method. This may occur either concurrently or over a period of days.

Step 4: Using the results obtained from the analysis of the diluted quality control sample, the average recovery (x) in the appropriate reporting units (such as ug/L) and the standard deviation of the population sample (n-1) (in the same units) is calculated for each parameter of interest.

Step 5: For each parameter, the standard deviation (s) and the average recovery (x) are compared to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory-generated acceptance criteria (if a non-standard method).

If s and x for all parameters meet the acceptance criteria, the analysis of actual samples may begin. If any one of the parameters exceed the acceptance range, the performance is unacceptable for that parameter.

Step 6: When one or more of the tested parameters fail at least one of the acceptance criteria:

1. Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with c) above.
2. Beginning with c) above, repeat the test for all parameters that failed to meet criteria. Repeated

failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with c).

8.4 Sample Aliquots

When obtaining aliquots from a sample, it is imperative that the subsamples be representative of the parent sample. This ensures that results obtained from the analysis of these aliquots are representative of the entire parent sample - not just the subsample. Several techniques are employed in obtaining subsamples.

Representative aliquots of soil samples for the determination of metals can be obtained through the process of quartering. This technique involves repeatedly quartering the sample until the resulting quarter is equivalent to the amount of sample needed for analysis. Quartering, however, may not be an appropriate technique to obtain subsamples for volatiles or other analyses where there is concern over potential contamination or loss of target analyses.

Water samples are often inverted several times prior to the collection of a subsample. This process ensures that the sample is mixed thoroughly and is absolutely required for the accurate determination of analytes such as total and total suspended solids.

The appropriate techniques for obtaining sample aliquots for a designated analysis are discussed in the applicable SOPs. The techniques identified in these SOPs are those that are properly employed by our analysts.

8.5 Data Verification

All data included in the final reports to our clients has undergone extensive data verification. GEL has implemented a multi-level review process in all areas of the laboratory beginning with sample login. The review process and responsibilities of each level of review are delineated in GL-LB-E-005 for Data Review/Validation. This information is summarized below.

8.5.1 Sample Login:

Samples are analyzed by the methods and for the target analytes identified when the samples are logged in to our database. If there is an error in this entry and it is not promptly identified, the incorrect analytical method may be used or certain requested analytes not determined.

In order to prevent this from happening, the person entering the information into the database generates a report containing all the information entered for a particular sample. This report is reviewed against the client confirmation letter and/or chain of custody first by the person who generated it and then by a

peer. If any errors are identified, they are immediately corrected.

Any uncertainties are addressed to the project manager. The project manager or project manager assistants are responsible for providing a third and final review of this information.

8.5.2 Data Validation in the Laboratory

The multi-level review process implemented in our laboratory includes an initial review by the analyst, a second review by a peer, and a final review by a group leader or data validator. Where deemed adequate based on personnel and client needs, the industrial division only institutes the two levels of review.

Data review checklists designed to ensure that the analyst meets the quality control and quality assurance requirements are employed throughout the laboratory. These checklists incorporate a review of the analytical and sample preparation data as well as the necessary elements for hard copy documentation.

Reviewers of analytical data are responsible for:

- Ensuring the analytical procedures used are in compliance with current SOPs
- Ensuring that quality control samples were analyzed at the frequency specified in the SOP or to previously agreed to client specifications.
- Ensuring that the following meet the acceptance criteria for quality control samples: recoveries of all matrix spikes and laboratory control samples; the relative percent difference for matrix duplicates, matrix spike duplicates, and laboratory control sample duplicated and concentrations of target analytes in the method blank .
- Reviewing instrument data, run logs, and logbooks to ensure that all method quality control criteria were met. This includes calibration, initial calibration verifications, and continuing calibration verifications
- Ensuring that the documentation is sufficient to reconstruct the analytical procedures performed.
- Ensuring that all data is maintained in accordance with GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Record Keeping Devices.
- Verifying that raw data is in agreement with the computer generated batch sheets and data reports.
- Verifying calculations, dilution factors, concentration reported, and nominal concentrations.
- Verifying at least one software-generated calculation per analytical batch.

- Ensuring the documentation of comments, qualifiers, or nonconformance reports for noncompliant or questionable data

- Identifying and preventing the reporting of data generated when the analytical process appears to be out of statistical control.

8.5.3 Validation of Data Reports and Packages

Prior to data being reported to the client, the requested data report or package is reviewed for accuracy, completeness, and against client-specifications. Responsibilities for this review are dependent upon the type of report or package being generated. (Refer to Section 11 for Data Report Formats)

If a client is only receiving a certificate of analysis or certificate of analysis and Quality Control Summary Report, the project manager (PM) or project manager assistant (PMA) reviews the information for accuracy and completeness. This includes ensuring that any pertinent comments made by the laboratory about the analysis or sample are added to the certificate of analysis. The PM or PMA reviews data for consistency be analyte determinations as described in GL-QS-E-009 for Sample Report Preparation and Review.

If case narratives are requested, data validators review the analyst-prepared case narrative for accuracy and ensure that it is consistent with the information included on the certificate of analysis and Quality Control Summary Report.

If a client has requested any level of data package up to and including CLP-like data packages, the review of each laboratory fraction of the data package is conducted by that fraction's data validator. The reviewed fractions are then complied into a final data package which is reviewed by the PM or PMA against the client specifications.

8.6 Standard and Reagent Documentation and Labeling

The documentation and labeling of standards and reagents is addressed Section 10.1 for Records Keeping and Design.

8.7 Computer and Electronic Data Related Requirements

Our software Quality Assurance Plan GL-QC-001 describes the way in which we manage our software programs and hardware systems. The control of software development and modification activities are described in GL-IMS-E-001. All development and revision activities are validated, verified, and controlled with revision software prior to production use.

Analytical software that is purchased from a vendor is validated and verified in accordance with GL-IMS-E-003 "Vendor supplied Analytical Equipment Software."

Documentation requirements for assurance of validation and verification are described in GL-IMS-E-004 "The Verification and Validation of Software."

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SECTION 9
SAMPLE HANDLING, ACCEPTANCE, RECEIPT and INTERNAL CHAIN OF CUSTODY

Section 9: Sample Handling, Acceptance, Receipt, and Internal Chain of Custody

The receipt and acceptance of a sample and the handling of that sample while it is in our possession are critical to providing clients with data that is of the highest quality and legally defensible. GEL has established strict policies that govern the initial acceptance of a sample, the receipt of that sample by the laboratory, handling of the sample in order to ensure its integrity, maintenance of internal chain of custody, and storage of the sample upon completion of the required analyses. Described in this section are the policies and practices that govern these activities.

Covered in this section are the following:

- Agreements between client and GEL to perform analysis
- Proper labeling of submitted samples and completed chains of custody
- Sample receipt procedures
- Sample receipt procedures for radioactive samples
- Sample tracking
- Internal chain of custody
- Sample Storage
- Sample Disposal

9.1 Agreement to Perform Analysis

Prior to submitting samples for analysis, an agreement should be made between the client and GEL concerning the methods of analysis to be used, number of samples to be submitted, price of analysis, date by which the client must receive results, and format of the results. In addition, any special needs the clients may have such as non-routine methods and reporting limits should be communicated and an agreement reached prior to the samples being submitted for analysis.

The agreement to perform analysis may be in one of three forms as described in the GEL Analytical Services Reference Manual and SOPs GL-CO-E-002 for the Delegated Authorization to Commit the Company and GL-CO-E-003 for Request for Proposal (RFP)/Contract Review:

- Client confirmation letter (CCL) which is an agreement between the client and project manager for a specific group of samples. This letter includes the cost, turn-around time, requested analysis, sample matrix, number of samples, and type of client report.
- Acceptance by the project manager of sample(s) from an established client based on previously agreed to

conditions and confirmed by the clients submission of the sample(s.)

- Contract between client and GEL to perform analysis over a designated time period and/or project(s) that meet agreed to specifications.

9.2 Sample Labels and Chain of Custody Forms

Once an agreement to accept samples for analysis has been established, it is the joint responsibility of the client and GEL to ensure that samples submitted for analysis are properly labeled and accompanied by full and complete documentation which includes chain of custody and, where possible, material safety data sheets. The laboratory may refuse samples that are submitted without proper documentation.

Information include on sample labels should include:

- client sample identification
- location, date, and time of collection
- collector's name
- chemical preservatives used
- constituents of interest (if space permits)

Upon request from the client, sample containers containing appropriate preservatives and having a standard label may be shipped to the client to be used during sample collection. The containers are prepared and shipped in accordance with GL-RC-E-003 for Sample bottle Preparation and Shipment. Each shipment includes a chain of custody form.

The use of these containers offers several advantages including:

- Dedication of appropriate type sample container for the intended analyte or analytical method.
- Proper sample preservation for analysis
- Traceability of bottle lot number used for a client's sample aliquot to the manufacturer's certification that the containers are clean and show no signs of contamination.

Chain of custody forms are initiated at the time of sample collection and should include the following information when a sample is submitted to the laboratory:

- client name and address
- client sample identification
- date and time of sample collection
- sample matrix
- location description of sampling site
- number of containers
- chemical and physical constituents and methods for which analyses are conducted
- preservatives
- signature of person who collected the sample and date of signature
- signature of person relinquishing sample to the laboratory and date of transfer.

Standard GEL chain of custody forms are available to all clients and are included with each shipment of pre-labeled and preserved containers. All clients should use the GEL chain of

custody forms unless otherwise agreed by contract.

If the samples are being collected by GEL Field Services personnel, the standard GEL chain of custody form and certified containers are automatically used during sample collection.

9.3 Sample Conditions

In addition to proper documentation on the sample container labels and the sample chain of custody form, samples should meet established requirements for analysis. This is extremely critical for samples that are being analyzed to meet regulatory requirements.

Samples should be collected in the appropriate type of container, preserved as directed, and stored in the conditions specified in the analytical method or established regulatory guidelines. In addition, samples should be submitted with sufficient time to conduct the specified analysis within the regulatory or method holding time. Sample aliquots should be of sufficient volume to perform the requested analyses. A summary of these conditions and holding times for routine analyses can be found in Appendix J.

9.4 Sample Receipt

Samples are received at GEL in a central sample receiving area by an employee designated as the laboratory sample custodian or log-in clerk. Upon receipt, all samples are subjected to the protocols established in GL-SR-E-001 for Sample Receipt, Login, and Storage Receipt of a sample is acknowledged by the laboratory sample custodian signing the chain of custody and recording the date and time custody was transferred from the client or client representative to the laboratory. The date, time, and person receiving the sample are also recorded on the standard or client-specific Sample Receipt and Review form.

It is the responsibility of the sample custodian to note the conditions of a sample upon its arrival at GEL. This information is recorded on both the sample chain of custody and the Sample Review and Receipt form.

As detailed in GL-SR-E-001, the laboratory sample custodian should:

- Inspect all sample containers for integrity.
- Document any unusual physical damage or signs of tampering of custody seals
- Place any samples that appear to be leaking or have unusual odor under the fume hood and notifying the responsible project manager.
- Follow the screening procedure described S-007 for the Receiving of Radioactive Samples if the client identifies the sample as radioactive or if the sample is from a Department of Energy site.
- Review the chain of custody submitted by the client for completeness

- Compare the descriptions and other information on the sample container labels to that listed on the chain of custody

- Verify that the sample is within the regulatory holding time for requested analyses

- Measure and record the temperature of sample aliquots that are to be used for analyses requiring thermal preservation

- Measure and record the pH of all sample aliquots submitted for analyses that require chemical preservation to a specific pH

- Verify that adequate sample aliquots were submitted for the requested analyses.

- Verify that the appropriate sample containers were used for the analyses requested.

If departures from standard conditions or abnormalities are noted by the sample custodian, the project manager is immediately informed. The project manager notifies the client as quickly as possible so that a decision can be made to proceed with analysis or submit another sample or additional sample aliquots.

Abnormalities or departures from standard conditions include:

- Sample containers with obvious signs of damage, leaking, or tampering such as damaged custody seals.

- Chain of custody having incomplete information or not submitted with the sample

NOTE: If a nonradioactive sample is submitted without a chain of custody, the sample custodian should initiate one. This initiation is to be documented by printing "INITIATED ON RECEIPT" on the chain of custody.

- Information included on the chain of custody does not agree with that identified on the sample container labels

- Sample exceeds regulatory or method holding time.

- Sample not preserved to the method or regulatory-required pH

- Sample submitted in a container that does not method or regulatory criteria

- Sample temperature exceeds or fall below the thermal preservation regulatory or method requirement by more than 2C

NOTE: If a sample is hand delivered to the laboratory immediately after collection and there is evidence that the chilling process has begun such as arrival on ice, this sample shall be considered acceptable.

- Sample found to contain radioactivity that exceeds that allowed by our radioactive license. The handling of radioactive samples is discussed in 9.5

A sample that is not appropriate for the type of analyses requested, do not conform the method or regulatory requirements, or where the test is not fully specified requires:

- That all correspondence and/or records of conversations concerning the final disposition of the sample be retained
- Complete documentation concerning any decision to proceed with the analysis of the sample including the nonconforming condition on the chain of custody and Sample Receipt and Review form.
- Documentation that the analysis is qualified appropriately on the final report.

9.5 Receipt of Radioactive Samples

Radioactive samples received at GEL are subjected to the same monitoring identified in 9.4 if their levels of radioactivity do not exceed that permitted by our license. Special procedures governing the receipt of radioactive samples are described in the SOP E-007 for the Receiving of Radioactive Samples. It is critical that these procedures be implemented to prevent the inadvertent spread of radioactive contamination throughout the laboratory.

It is imperative that clients notify GEL of impending shipments of radioactive samples and their activities. We cannot exceed the limits of our radioactive license. GEL reserves the right to refuse and return to the client any radioactive sample where the radioactivity:

- Exceeds our permitted level by itself or in combination with other samples already on site or
- Exceeds our administrative level of 25mR/hr.

Special requirement for the receipt of radioactive samples by sample custodians include:

- Only designated personnel who have been trained in the proper handling of radioactive materials handle radioactive samples.
- Samples labeled as radioactive shall be opened by two sample custodians.
- If a sample is labeled as "Radioactive II", the sample custodian is not to open the sample and is to inform the radiation safety officer (RSO) immediately.
- Using a survey meter that is calibrated in mR/hr, measure the radioactivity of the sample by scanning the exterior surface of the cooler. The proper calibration and use of the survey meter is described in GL-EPI-E-001 for Radiation Survey Procedure.
- If the radioactive level of the exterior of the sample cooler exceeds 2 mR/hr, notify the RSO before opening the cooler.
- If the radioactivity level of a sample or group of samples is found to exceed 25mR/hr, the RSO should be notified immediately. The client will be contacted and arrangements will

be made to return the sample(s) or reduce the per sample exposure.

- If a chain of custody is not submitted with a sample, it is placed on hold until a chain of custody is submitted.

- Survey the inside of the cooler to ensure that no leakage or contamination has occurred.

- Survey each sample container and document the highest reading on the Radioactive Shipment Inventory Sheet.

The receipt of samples from DOE sites requires that the samples be monitored for radioactivity.

Each cooler containing DOE samples is surveyed. If the level of radioactivity is determined to be less than 0.5 mR/hr, the samples contained in that cooler can be treated as non-radioactive.

If this level is exceeded, each sample container in the cooler must be scanned in order to determine which samples are elevating the reading. The samples should be handled is accordance with GL-EPI-S-004 for Radioactive Handling Procedures.
If samples are determined to be radioactive, the project manager and RSO are to be notified. The samples will be placed on hold until it is determined if the samples will be accepted.

9.6 Sample Tracking

A sample received by the laboratory is tracked by a unique laboratory identification number. This number is assigned as the information pertaining to the sample is entered into our database.

Pursuant to SOP GL-SR-E-001, the following information is entered into our electronic database for each sample received:

- client and/or project code
- client sample ID
- sample matrix
- equivalent laboratory sample matrix
- type of report format requested by the client
- date and time of collection
- date received
- initials of person making entries
- number of containers submitted for the sample.
- requested analyses
- any pertinent observations or comments affecting the sample analysis or rejection

When this information is uploaded, the computer system automatically assigns a unique number to the sample and each of its containers. This number is used to track the location of a sample container and provides a link to any subsamples and subsequent digestates and/or extracts.

The unique laboratory identification number is printed on a durable barcode

label that also contains the client identification, sample date and time. Once labeled, the sample container's specific identification number is uploaded into the electronic database by scanning the barcode. Information included in the database at the time of sample scanning is the container's storage location, bottle type and volume, physical characteristics of the bottle, preservative, and the initials of the person entering this information.

Entering of this information into the database is an important part of initiating the electronic internal chain of custody.

9.7 Internal Chain of Custody

Chain of custody procedures provide assurance of traceability and integrity of samples. Our legal and evidentiary chain of custody protocol establishes an intact, continuous record of the physical possession, storage, and disposal of sample containers, collected samples and aliquots, and sample digestates or extracts.

The internal chain of custody is based on scanning a container's barcode label into an electronic database while identifying the location of the sample and the person having custody, or placing the sample in a secured storage area. If we supply sample containers, this chain of custody may begin at the time sample containers are provided to the client.

For the purposes of the internal chain of custody, a sample is defined as being in someone's custody if:

- It is in one's actual physical possession
- It is in one's view after being in one's physical possession
- It is in one's possession and then is locked up so that no tampering may occur
- It is kept in a secured area which is restricted to authorized personnel only

The protocol for ensuring sample integrity using the internal chain of custody is detailed in GL-LB-E-012 for Verifying the Maintenance of Sample Integrity.

The electronic internal chain of custody works in conjunction with the chain of custody submitted by the client with a sample to:

- Account for all time periods associated with a sample, its subsamples, and extracts or digestates from the time the sample is received at GEL to its disposal.
- Identify all individuals who physically handled the sample
- Provide evidence that the sample was stored in accordance with method and regulatory protocols

The electronic internal chain of custody is linked to our main database so that information demonstrating the proper maintenance of custody can be provided to the client on the data reports or electronic data deliverables.

9.8 Sample Storage

In order to ensure the maintenance of sample integrity, all aliquots are stored in secured areas designated for sample storage. The storage location of each sample aliquot can be tracked using the internal chain of custody. Areas designated for sample storage include:

- Main cooler where most samples requiring maintenance at a temperature range of 2° - 6° C are stored.
- Volatile cooler for samples to be analyzed for volatile contaminants
- Radioactive cooler for segregation of radioactive sample aliquots requiring refrigeration
- Ambient storage for non-radioactive samples not requiring refrigeration
- Ambient storage for radioactive samples
- Refrigerators for the storage of samples requiring bacteriological analysis and temporary storage for those requiring the determination of biochemical oxygen demand.

The temperature of each storage unit is monitored at least twice a day and documented in accordance with GL-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators Freezers, Ovens Incubators, and Other Similar Devices. In addition, the main and radioactive coolers are monitored twenty-four hours a day by temperature sensors that are connected to our main security system. If the temperatures exceed the required range, an alarm is sounded and the security system notified the facilities manager or his designee immediately. This allows corrective actions to be initiated promptly.

Prior to and immediately after analysis, samples and their respective digestates and extracts are stored in compliance with the requirements of the requested analytical methods and GL-SR-E-001 for Sample Receipt, Login, and Storage. If a single aliquot is supplied for analyses by several methods, the most stringent analytical storage requirements are applied to the sample.

If samples are to be analyzed for volatile organic compounds, they are stored in a designated volatile cooler that is maintained at a temperature range of 2° - 6° C. No sample aliquots are stored in this refrigerator unless they are to be analyzed for volatiles. This storage unit is monitored on a weekly basis for contamination by the analysis of a volatile cooler storage blank.

At the beginning of each month, eight 40 mL vials are filled with treated deionized water that is used for volatile method blanks and placed in the volatiles cooler. Each week one or two vials are analyzed by EPA 8260B and the data is reported to the quality department. If the analysis reveals any evidence of potential contamination, appropriate corrective actions are immediately implemented.
Sample aliquots for non-volatile analysis, which also should be maintained in the temperature range of- 2° - 6° C, are stored in the main cooler adjacent to our sample receipt area

unless they are radioactive. In order to reduce the chance of contamination, radioactive samples are stored in a designated cooler.

Sample aliquots designated for the determination of total coliform bacteria , fecal coliform bacteria, or total plate count are delivered to the bacteriology laboratory and stored in the designated refrigerator at a temperature range of 2° - 6° C. This allows easy access for the analyst ensuring that the short regulatory holding times are met. After analysis is complete, the remaining sample aliquot is disposed of in accordance with GL-LB-E-011 for Laboratory Sample and Waste Disposal and Emergency Instructions.

Samples aliquots to be analyzed for biochemical oxygen demand (BOD) are also delivered to the bacteriology laboratory and stored in the designated BOD cooler. This cooler is also maintained at 2° - 6° C. After initiation of this analysis, the sample aliquots are returned to the main cooler.

Sample aliquots that are to be analyzed for metals are stored in the ambient storage area. Prior to analysis, the samples' digestates are stored in the appropriate area in the TRACE/ICPMS and inorganic preparation laboratories. Sample digestates are then transferred to the sample archive area where they are maintained until disposal.
After all analyses are complete and results are submitted to the client, sample aliquots are transferred to the sample archive area. Samples are stored in this area in accordance to GL-LB-E-011 for Laboratory Sample and Waste Disposal until they are disposed. Radioactive and no-radioactive samples remain segregated while archived to reduce the chance of contamination.

9.9 Sample Disposal

GEL's policies concerning sample disposal can be divided into two sections - those governing the disposal of sample laboratory waste and those directing the disposal of remaining sample aliquots after the completion of all analyses.

9.9.1 Sample laboratory waste

Unless otherwise requested by contract, laboratory sample waste is collected throughout the laboratory in designated satellite containers found in sample collection and accumulation areas. The location and type of each of these satellite containers is described in Appendix 3 of GL-LB-E-011 for Laboratory Sample and Waste Disposal and Emergency Instructions.

As indicated in this appendix, laboratory sample wastes are segregated based on the type of analysis by which they were generated, by matrix, and radioactivity. This contains certain process contaminants thus decreasing the amount of waste material that may be labeled hazardous. It also ensures that solid and aqueous wastes are not mixed.

The satellite collection containers are regularly emptied by the Laboratory Waste Manager or designated personnel into labeled 55-gallon drums in the waste staging areas. A log is contained is this staging area in which the following information is recorded:

container identification, satellite station source, date transferred to 55-gallon drum, volume transferred, and initials of the person transferring the material. There are separate radioactive and nonradioactive staging areas.

The composited sample wastes then undergo hazardous water characterization. The analyses requested are specified in GL-LB-E-011 and differ depending upon sample matrix. Aqueous sample waste composites are typically analyzed for metals, base neutrals and acids, pesticides, PCBs, pH, cyanide, and volatile compounds. Solid sample waste composites are analyzed for the TCLP parameters, BTEX, TPH, total lead, and water content. Specific target analytes are identified in Appendixes 5 and 6 of GL-LB-E-011.

The sample waste is disposed as indicated below depending upon the results of the analyses performed and matrix.

- If all of the parameters are determined to all below the limits specified for discharge and the waste is aqueous, it is discharged into the sewage system.
- If aqueous waste is found to exceed the limits for heavy metals, the pH is adjusted to fall between 7.0 and 9.0 resulting in metal precipitation. The precipitate is allowed to settle and the clarified water is discharged.
- If aqueous sample waste was found to contain volatiles in excess of the limits is vigorously aerated for at least four hours prior to discharge.
- If the pH of aqueous waste is not within the range of 6.0 to 9.0, it is adjusted to fall within this range prior to discharge.
- If the aqueous waste ifs found to contain semivolatile compounds in concentrations exceeding the limits, the waste is passed through two drums of activated carbon placed in series prior to discharge.
- If the waste matrix is solid, it is sent for incineration at an approved facility.
- If sample waste is determined to contain mercury at concentrations exceeding the specified limits, it is incineration at an approved facility.
- Sample waste determined to be hazardous waste is incinerate at an appropriate facility is the concentration of PCB is less than 50 ppm.
- Sample waste determined to be hazardous waste is incinerated at an appropriate facility is the concentration of PCB is less than 50 ppm.
- Radioactive sample waste not returned to the client is disposed of at an appropriate facility for radioactive waste.

Records are maintained documenting the final disposition of all sample waste and all analytical testing supporting the method of disposal.

9.9.2 Remaining Sample Aliquots

Sample that is not consumed during the sample preparation or analytical procedures either returned to the client in accordance with GL-SR-E-002 for Return of Samples or disposed in accordance with GL-LB-E- 011 for Laboratory Sample and Waste Disposal and Emergency Instructions.

All radioactive samples are returned to the client unless otherwise specified by contract. Nonradioactive samples are returned to a client under the conditions and terms agreed to by contract. A chain of custody listing the laboratory waste technician as the relinquishing party is enclosed with each set of samples being returned to a client. Unless otherwise specified by he client, all nonradioactive samples are shipped by UPS. If the samples are radioactive, the procedure for shipment is delineated in S-008 for the Shipment of Radioactive Samples.

It is GEL's policy to hold samples for a minimum of thirty days after invoicing before disposal unless otherwise specified by contract or if the sample is part of litigation. If the sample is part of litigation, disposal of the physical sample shall occur only with concurrence of the affected legal authority, sample data user, and/or submitted of the sample.

When all analyses on a sample are complete and regulatory and/or contractual holding times have expired, samples are moved from their respective storage locations to either radioactive archives or nonradioactive archives. Samples that are to be either returned to the client or must be held for an extended time period are segregated from the other samples. Radioactive and nonradioactive samples remain segregated during this process.

When internal or client-specified storage time has expired, samples with like matrices are composited into 55-gallon drums. The composites are then subjected to the same treatment and disposal protocol as described in 9.9.1. In addition to the log documenting which samples are composted in which drum, the barcode labels for each disposed sample are scanned into our data base and assigned the status of disposed.

The empty containers from nonradioactive samples are shredded and crushed and disposed of with the general refuse. Sample containers from radioactive samples are disposed of as radioactive waste.

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SECTION 10
RECORDS

Section 10: Records

Quality records provide the documentation to support analytical results and conclusions. Documented evidence that quality assurance and quality control requirements have been met is critical to providing data that fulfills specifications of applicable procedures, programs, and contracts.

As described in Section 3 of this Quality Assurance Plan (QAP), GEL quality records include but are not limited to:

- Observations
- Calculations
- Calibration data
- Certificates of analysis
- Certification records
- Chains of custody
- External, supplier, and internal audits
- Run logs
- Instrument data and analytical logbooks
- Instrument, equipment and building maintenance logs
- Material requisition forms
- Monitoring logs
- Nonconformance reports
- Corrective actions
- Method development and start-up procedures including method detection limit studies
- Training records
- Waste management records
- Standard logs
- Software validation
- Standard operating procedures (SOPs)
- Sample collection and field data

Our procedures to provide a legal and evidentiary chain of custody are described in Section 9 of this QAP and are not, therefore, addressed in detail in this section. Described in this section are:

- Records keeping system and design
- Records management and storage
- Sample handling records
- Records of support activities
- Analytical records
- Administrative records

10.1 Records Keeping System and Design

GEL quality records are maintained, managed, and stored in accordance with the Quality Records policy defined in Section 16 of our Quality Manual and our SOP GL-QS-E-008 for Quality Records Management and Disposal.

The protocols established in these documents work in conjunction with those for specific types of records addressed in other SOPs to govern our records keeping system. GEL's record keeping system allows the historical reconstruction of all laboratory activities that produced analytical data.

We facilitate historical reconstruction by maintaining the following records and information, from the time the sample is received until it is disposed.

- A master list of all employee signatures and initials is maintained in the Human Resources department. This allows the identification of any GEL personnel who accept, handle, analyze, prepare, review, store, or dispose of a sample, its subsamples, associated data and data reports, and other related documentation including calibrations and nonconformance reports.
- If provided to the client or sampling personnel by GEL, records of the bottles and containers used for sample collection are kept in accordance with GL-RC-E-003 for Sample Bottle Preparation and Shipment. These electronic and paper records include:
    - Supplier and lot numbers of containers and/or bottles provided
    - Certifications that the containers are free of contaminates that may bias the analyses
    - Addition of preservatives and identity of person responsible for this preservation.
    - Barcode of containers supplied to a particular client or for a specific field sampling event.

The person or agency responsible for collecting a sample is documented on the sample's chain of custody and entered into our database when the sample information in uploaded by the sample custodian as described Section 9 and in GL-SR-E-001 for Sample Receipt, Login, and Storage.

Other records concerning the acceptance of a sample as discussed in Section 9 and maintained in accordance with GL-SR-E-001 for Sample Receipt, Login, and Storage include:

- Date and time of sample receipt
- Person accepting sample
- Conditions of sample upon receipt
- Client-confirmation letter and/or sample quote
- Client chain of custody
- Electronically generated sample identification numbers that are specific to each sample aliquot and linked through our database to the client's sample description, sample collection and receipt information, and analyses to be performed. These identification numbers are produced in accordance with GL-QS-SR-001.
- Identification of each person who has custody of a sample, its subsamples, extracts, or digestates. (This is provided through the internal chain of custody procedures described in Section 9 and in GL-LB-E-012 for Verifying the Maintenance of Sample Integrity.)

Documentation that materials purchased for use in the analysis or preparation of samples meet specifications is maintained in accordance with GL-RC-E-001 for Receipt and Inspection of Material and Services.

Records of equipment calibration are maintained and are traceable by date and the equipment's identification to a specific analysis. These records include but are not limited to: the initials and/or signatures of outside service technician contracted to calibrate and/or service equipment and items such as balances, class S weights, and NIST thermometers are documented on the provided certifications of calibration and/or labels on the item itself. These certifications are maintained by the Quality Assurance Team.

Thermometers are calibrated against the NIST traceable thermometer and records of this calibration are maintained as described in GL-QS-E-007 for Thermometer Calibration.

Records of daily and monthly calibration verifications of analytical balances include the initials of the responsible party as described in GL-LB-E-002 for Balances.

The calibration of air-displacement pipets used to transfer sample aliquots is maintained in pipet calibration logs specific to each pipet. These calibration records are maintained n accordance with GL-LB-E-010 for Maintenance and Use of Air Displacement Pipets.

If methods and/or regulations specify that samples and their subsamples, extracts, and/or digestates be stored at designated temperatures or if the method, itself, has temperature sensitive steps, the temperatures are documented on monitoring logs at the frequency defined in the SOPs. The specific storage location of a sample can be traced using the internal electronic chain of custody.

Initials of all personnel responsible for monitoring temperatures are recorded on the temperature monitoring logs in accordance with GL-LB-E-004 for Temperature Monitoring and Documentation Requirements for Refrigerators, Freezers, Ovens, Incubators, and Other Similar Devices. These monitoring logs are reviewed for completeness by Quality System Specialists in accordance with GL-QS-E-005 for the Review of Monitoring Devices.

Documentation of the instruments and equipment used for the analysis of samples are documented on the run logs, in laboratory logbooks, instrument data, and/or sample preparation logs. Routine or corrective maintenance performed on equipment or instruments are recorded in maintenance logs specific to each instrument. These records are documented in accordance with GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Forms and Other Record Keeping Devices.

Standards containing known quantities of target analytes that are used in instrument calibration, calibration verification, or as quality control samples such as matrix spikes and laboratory control samples are documented in accordance with GL-LB-E-007 for Standards Documentation. These records allow each standard to be traced to the source standard or standards from which it was prepared. In addition to source standard

traceability, the following information is also recorded:

- Recipe by which each standard was prepared
- Traceability of each child standard to its parent.
- Date each standard was prepared
- Initials of person preparing the standard
- Expiration dates
- Concentration of each standard

This information allows us to document that the standards used were prepared in accordance with the established protocol, were produced using source standards that meet the method and regulatory criteria, and used prior to their expiration date.

If required, reagents used in the preparation, dilutions, and analysis of samples are verified to be free of interferences or target analytes. Verifications are recorded in the reagent logs in accordance with GL-LB-E-008. Reagent logs are maintained for solvents used in the organic preparation laboratory, acids used in the inorganic preparation laboratory, redistilled freon utilized for oil and grease determinations, and other areas as deemed necessary to ensure that method and regulatory requirements are met.

Records of the analytical and sample preparation methods applied to each sample aliquot are documented using the internal chain of custody, method information entered in to the database, and information recorded in laboratory notebooks, sample preparation logs, run logs, or instrument data.

The laboratory protocol that is employed at the time a sample is analyzed is provided by the SOP that was in effect at the time a sample was analyzed or prepared by a specific method.

The preparation and analysis of samples and the associated instrument calibrations are documented on run logs, laboratory notebooks, instrument data, and sample preparation logs. This information is recorded in accordance with GL-LB-E-009 for Run Logs and GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Record Keeping Devices.

As indicated in these SOPs, preparation and analytical records not generated by automated data collection systems should be:

- Legible
- Recorded in permanent black ink
- Corrected if a mistake is made by one line marked through the error and the correction dated and initialed
- Signed or initialed by the responsible party

Electronic and hard records are maintained for each analytical batch. These records include the identification numbers of each client and quality control sample prepared and/or analyzed together, the method of analysis and sample preparation to which the batch

was subjected, and the matrix of the samples included in the batch.

Through our electronic statistical process control system (SPC), the acceptance criteria applied for all quality control samples are stored and maintained in accordance with GL-LB-E-019 for Statistical Process Control. Acceptance limits for the target analytes are method, matrix, and time-period specific. This allows us to regenerate the criteria applied to quality control samples associated with identified client samples.

Records of nonconformances and corrective actions associated with specific samples, analytical batches, and processes are maintained by the Quality Systems Management Team. These records are maintained in accordance with GL-QS-E-004 for Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items and GL-QS-E-002 for Conducting a Corrective Action.

Electronic records of data are maintained in a secured database, designed to protect the integrity of the data:

Data that is uploaded directly from instruments as well as that manually entered is backed up by a second system.

Permanent records of the transmittal of electronic disk data deliverables are also maintained. Records of the review of analytical data and sample preparation data are maintained. This documentation includes the initials of the reviewer and date of the review. Records of the data reported to clients are maintained in a manner that ensures client confidentiality. These records include copies of certificates of analysis, quality control summary reports, case narratives, CLP forms, and any other information that may have been provided to the client. The copies can be either paper copies or electronic copies of the scanned document. The majority of the data packages submitted to Federal Division clients are scanned into our system prior to being submitted to the client.

Records of the return of samples to the clients and/or the disposal of samples are documented in accordance to GL-SR-E-002 for Return of Samples and GL-LB-E-011 for Laboratory Sample and Waste Disposal and Emergency Instructions. These records include the date samples were returned and/or disposed of , destinations of samples, and name of person responsible for transfer of sample.

10.2 Record Storage

Quality records are stored in compliance with GL-QS-E-008 for Quality Records Management and Disposition. Records are required to be:

- Be stored in a secured area so that client confidentiality data integrity is maintained
- Kept in areas where they are protected from fire loss, environmental deterioration, vermin and, in the case of electronic records, electronic or magnetic sources.
- Indexed and filed in a manner which allows them to be readily retrieved.
- Accessible to the client for whom the record was generated.

- Retained for an identified time period that exceeds or equals five years and disposed of when this time has expired.

In addition to hard copies of quality records, GEL keeps electronic copies of the data. The electronic copies are created by scanning the records into a records database using Adobe. The records are also stored on compact disks.

All hardware and software necessary for the historical reconstruction of data is also maintained. Records that are stored or generated by computers or personal computers have hard copy or write-protected backup copies.

10.3 Sample Handling Policy

We maintain records of all procedures applicable to samples in our possession. These records include documentation pertaining to:

- Preservation, including appropriate sample container and compliance with holding time requirement
- Sample identification, receipt, acceptance or rejection and login
- Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records
- Sample preparation including cleanup and separation protocols, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents
- Sample analysis
- Standard and reagent origin, receipt, preparation, and use
- Equipment receipt, use, specification, operating conditions and preventative maintenance
- Calibration criteria, frequency and acceptance criteria
- Data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions
- Method performance criteria including expected quality control requirements
- Quality control protocols and assessment
- Electronic data security, software documentation and verification, software and hardware audits, backups and records of any changes to automated data entries
- All automated sample handling systems
- Disposal of hazardous samples including the date of sample or subsample disposal and name of the responsible person.

10.4 Records of Laboratory Support Activities

In addition to our sample handling records, we maintain the following:

- All original raw data, both hard copy and electronic, for calibrations, samples and quality control measures, including work sheets and data output records (chromatograms, strip charts, and other instrument response readout records)

- A written description or reference to the specific method used which includes a description of the specific computational steps used to translate parameters observation into a reportable analytical value
- Copies of final reports
- Archived standard operating procedures
- Correspondence relating to laboratory activities for a specific project
- All corrective action reports, audits and audit responses
- Proficiency test results and raw data
- Data review and cross checking

10.5 Analytical Records

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs are to be maintained and to contain information as requested in GL-LB-E-008 for Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbooks, Forms, and Other Record Keeping Devices and GL-LB-E-009 for Run Logs.

Information documented in analytical records includes but is not limited to:

- Laboratory sample ID code
- Date and time of analysis
- Instrumentation identification and instrument operating conditions/parameter (or reference to such data)
- Method of analysis
- All calculations
- Dilutions
- Analyst's or operator's initials' signature
- Units of measurement

It is our policy that analytical records are:

- Accurate
- Reviewed, verified, and corrected if necessary. Any corrections must be documented appropriately.
- Legible and understandable
- Traceable and authentic to their source
- Maintained in a contemporary manner with data entry and information recorded as it is obtained

10.6 Administrative Records

Several pertinent types of records are maintained in our Human Resources department. These records include:

- Personnel qualifications and experience
- Training records that are maintained in accordance with GL-HR-E-002 for Employee Training. These records include the initial demonstration of efficiency for each analytical procedure an analyst or technician performs.
- A log of names, initials and signatures for all individuals who are responsible for signing or initialing any laboratory record.

Continuing demonstration of proficiency is monitored though performance audits. Records of performance audits are maintained in the Quality Department.

SECTION 11
LABORATORY REPORT FORMAT and CONTENTS

Section 11: Laboratory Report Format and Contents

Accurate data is of no benefit to a client unless it is reported in a format that is easy to interpret and provides all pertinent information relating to the analysis of a sample.

GEL has developed certificate of analysis report formats that meet the varied needs of our clients while providing all information necessary to satisfy regulatory requirements and to allow for the interpretation of the data. Each format provides data that is reported accurately, clearly, unambiguously, and objectively.

In addition to certificates of analysis, clients may request and will be provided with an extended data package. This package may include any of the following elements: certificates of analysis; summaries of quality control; case narratives; instrument data; sample preparation data; measurement traceability and calibration information; and electronic data deliverables.
If clients require the reporting of data following the established contract laboratory protocol (CLP), we can provide a CLP-like data package that will meet their needs.

It is important that the certificate of analysis format and need for and type of data package be discussed with the client prior to our acceptance of the samples.

Project managers and our contracting department are responsible for establishing an agreement with the client concerning data reporting and potential cost to the client for data packages and/or specialized reporting.

The submission, storage, and transmittal of all laboratory reports and data packages is conducted in a manner to ensure client confidentiality. Reports and supporting data packages are not released to persons or organizations outside GEL without the expressed consent of the client. If directed by regulatory agencies or subpoenaed to submit documents to a court of law, the client will be notified as to the requester and the records that were released.

The following report formats and data package elements descried in this section are:

- certificates of analysis (C of A)
- quality control summary reports (QCSR)
- analytical case narratives
- electronic data deliverables (EDDs)
- types of data packages and reporting formats
- review of data packages and reports

11.1 Certificates of Analysis

GEL has two primary C of A report formats, Level 1 and Level 2. Both formats contain the following information:

- Title
- Our address and phone number
- Name of the project manager or other person who serves as the primary client contact
- Barcode identification of the C of A
- Number of each page and the total number of pages
- Name and address of client, where appropriate
- Project name or code if applicable
- Client-provided sample description
- Unique laboratory identification number for the sample
- Sample matrix
- Characterization and condition of the sample where relevant
- Date of receipt of sample
- Date and time of sample collection if provided
- Date and time of sample analysis, reanalysis, and /or sample preparation
- Initials of analyst and, if applicable, person responsible for sample preparation
- Analytical batch number
- Sample analysis and preparation methods (or unambiguous description of any non-standard method used)
- Reference to sampling procedure if relevant
- Deviations from, additions to or exclusions from the test method, and any other information relevant to a specific test, such as environmental conditions including the use of relevant data qualifiers and their meaning
- Any nonconformances that may affect the data
- Where relevant, a description of the transformations, calculations, or operations performed on the data
- Identification of whether date is calculated on a dry weight or wet weight basis where applicable
- Identification of the reporting units such as ug/1 or mg/kg
- A statement of the estimated uncertainty of the test result
- Signature and title of the person(s) accepting responsibility for the content of the C of A
- Date C of A was issued
- If deemed necessary, fact that the results relate only to the items tested or to the sample as received
- A statement, when applicable, that the certificate or report shall not be reproduced except in full, without the written approval of GEL
- Clear identification of all data provided by outside sources, such as air temperature or ambient water temperature
- Identification of the reporting detection limit (RDL) or practical quantitation limit (PQL) for each analyte. The RDL is either that based on the lowest calibration standard corrected for sample dilutions and preparation, or client-specified limits that exceed the laboratory determined RDL.

If any portion of the sample analysis is subcontracted, the C of A clearly identifies all data determined by the subcontracting laboratory and the subcontractors name or applicable accreditation number.

Certificates of analysis issued in the level 2 format also contain the following additional information:

- Dilution factors
- Method detection limits
- Surrogate recoveries and the respective acceptance criteria for all organic analyses
- Estimated concentrations determined for nondetects and appropriate "U" and "J" qualifiers for nondetects and concentrations that fall between the MDL and PQL respectively

Once issued, a C of A is not altered unless the subsequent C of A is identified as a revised report.

11.2 Quality Control Summary Report (QCSR)

Samples are prepared and analyzed in groups not exceeding twenty samples. The quality control data that demonstrates the efficiency of the batches' sample preparation and/or sample analysis is summarized on a QCSR. The data included on the QCSR can be limited to only that pertaining to a sample delivery group contained in that batch or may include all batch quality control.

Information on the QCSR includes:

- Quality control sample identification number
- Type of quality control sample
- Concentrations determined, where applicable, for method blanks, matrix spikes, matrix spike duplicates, matrix duplicates, laboratory control samples. serial dilutions, and laboratory control sample duplicates
- Acceptance criteria for reported matrix spikes, matrix spike duplicates, matrix duplicates, laboratory control samples, and laboratory control sample duplicates
- Nominal concentrations of matrix spikes, matrix spike duplicates, laboratory control samples, and laboratory control sample duplicates
- Concentration of parent sample for the matrix spikes, matrix spike duplicates, or sample duplicates
- Percent recoveries for laboratory control samples and matrix spikes
- Relative percent differences for the matrix spike duplicates, matrix duplicates, and laboratory control sample duplicates
- Analytical batch number with which the quality control data is associated
- Parent sample numbers for matrix spikes, matrix duplicates, and matrix spike duplicates
- Sample or sample delivery group identification number
- Project code
- Date issued
- Page numbers and total number of pages
- Identification of recoveries or relative percent differences that do not meet the acceptance criteria

11.3 Analytical Case Narratives

Analytical case narratives are written by the analyst or data validate to describe the overall conditions affecting the

analysis of a batch of samples or a specific sample contained within the batch.

Case narratives include but are not limited to:

- Sample delivery group identification number
- Analytical batch number
- Methods of sample preparation and analysis
- Sample matrix
- Initial of person preparation and/or reviewing the narrative
- Specific sample identification numbers
- Identification and description of batch quality control samples including parent sample identification
- Affirmation that all sample preparation conditions specified by the method or regulatory agencies were met or identification of specific deviations
- Affirmation that all sample analysis criteria specified by the method or regulatory agencies were met or identification of specific deviations
- Instrumentation employed where applicable and verification of its calibration
- Summary of batch quality control as compared to acceptance criteria
- Identification of any nonconformances
- Pertinent comments and/or observations of other factors that may affect sample data quality

11.4 Electronic Data Deliverables (EDDs)

Electronic disk deliverables are generated according to the client's contractual needs. EDDs are generated using programs supplied by the client or created internally by the GEL EDD team to client specifications. Internally generated EDDs are based on Pearl or Microsoft Excel.

11.5 Types of Data Packages and Reports

We offer three levels of data reports and the ability to design packages to meet the needs of our clients. The levels of data reports are summarized in Table 1.

Table 1: Data Report Formats

Level	Contents
1	Level 1 C of A
2	Level 2 C of A
3	Level 2 C of A plus QCSR

If requested by the client, any of the above level of reports can be accompanied by EDDs, case narratives, copies of any associated nonconformance reports, and other support documentation including sample preparation and analytical data, instrument data, calibration data, and measurement traceability.

A client's specific data package needs are communicated to the laboratory and data validators by through our data

package tracking system. The requirements of a particular client are listed on a tracker, which also includes the sample delivery group to which the information pertains. The laboratory is responsible for providing the appropriate information to the data packaging team. This team compiles the final package from the fractions provided by each laboratory team.

If the client has requested and we have agreed to provide CLP-like data packages, they are produced and contain the information described in GL-LB-E-013 for the Generation and Assembly of CLP Data Packages.

If clients do not request a full CLP-like data package but do request data to be provided on CLP forms generated using Target (organic data) or Banford (metals data), the applicable procedures in GL-LB-E-013 should be followed.

11.6 Review of Data Reports, EDDs, and Data Packages

Level 1, Level 2, and Level 3 data reports are reviewed for accuracy and completeness by the project manager and/or project manager assistant in accordance with GL-ADM-E-002 for Process, Review, and Distribution of Certificates of Analysis and COA packages and GL-QS-E-009 for Sample Report Preparation and Review.

Data packages are reviewed in the laboratory by the appropriate data validator. The validator is responsible for reviewing all portions of the data package for consistency, accuracy, and completeness.

No data package fraction will be provided to the data packaging team without the approval of the appropriate data validator. Data validators are also responsible for overseeing the review of any associated EDDs and to ensure that the EDD is in agreement with the other portions of the package.

Project managers are responsible for reviewing the complete data a package to ensure that all of the client needs are met and to notify the client of any nonconformances or inability to provide requested information prior to the submission of the package.

CLP-like data packages are reviewed following the sample basic protocol. Any specific requirements are described in GL-LB-013 for the Generation and Review of CLP Data Packages

SECTION 12
SUBCONTRACTING ANALYTICAL SAMPLES and OUTSIDE SUPPORT SERVICES

Section 12: Subcontracting Analytical Samples and Outside Support Services

GEL provides a full array of organic, inorganic, and radiochemical analyses. The subcontracting of analytical samples to other facilities is, therefore, a very infrequent occurrence.

Samples may be considered for subcontracting to other laboratories if:

- The client has requested an analysis for which we are either not certified or do not offer as a routine product.
- The regulatory or method holding times and/or client due dates are in danger of not being met as the result of a critical instrument malfunction or unexpected influx of a large group of samples.

No samples are subcontracted without the client's consent. The client will be notified in writing of the intent to subcontract any portion of the requested analyses.

Laboratories selected to receive subcontracted samples should meet the following criteria:

- Demonstrated technical capabilities to provide data that meets and conforms to our quality standards
- Established certification, if available, for the requested analyses.
- Commitment to meet the client's time requirements for delivery of results.
- Agreement to provide all documentation requested in conjunction with the analysis.
- NELAP accreditation for the analysis if it is covered under this program.

GEL audits potential subcontractor laboratories for technical and administrative compliance as directed in GL-QS-E-001 for Conduct of Quality Audits. Following the initial audit, laboratories selected to receive subcontracted samples are re-evaluated every three years. This evaluation may be in the form of a records audit instead of an on-site audit.

If there is evidence of a technical, administrative, or quality deterioration, the laboratory will be removed from the list of approved subcontractor laboratories pending further evaluation, which may include on on-site audit.

Once the laboratory has again demonstrated compliance with GEL's standards, it will be reclassified as an approved subcontractor laboratory.

GEL has a multi-faceted staff of environmental consultants, field services

personnel, data validators, and environmental analysts. There are occasions, however, when it becomes necessary to obtain the services of professionals outside GEL. This may be due to such things as sample workload, introduction of a new instrument or method requiring special knowledge, or employee leaves of absence.

Any outside support services or service personnel are subjected to the same scrutiny as subcontracting laboratories. If any service is found to not met GEL standards for excellence, is promptly notified. If immediate corrections are not implemented and the service is not of adequate quality to maintain confidence in our laboratory's results, the service contract is canceled.

SECTION 13
CLIENT COMPLAINTS

Section 13: Client Complaints

Meeting the needs and expectations of our clients is essential to meeting GEL's commitment to be the environmental laboratory of first choice. An important part of meeting this commitment involves receiving and addressing client concerns and complaints.

Client complaints concerning the quality of the laboratory data or data deliverable received are directed to the Quality Department. As needed, the concerns are responded with input from the laboratory, EDD team, and data packaging group are responded to directly by a member of the quality team.

The types of complaints, area or areas affected, and the impact of any error that was identified in responding to the complaint are trended using in-house developed software. Reports concerning client concerns are available though this database for each member of the senior leadership team, division heads and group leader.

This data base is also used to ensure that client's receive prompt responses. Each complaint or concern is entered into this database upon receipt and assigned a internal and external due date. The external due date is often established by contract. The internal due date allows the Quality Department time to review the response for accuracy and to submit it to the client on or before the due date.

If a trend is noticed that will significantly affect data quality, a corrective action is initiated in accordance with GL-QS-E-002 for Conducting Corrective Actions. The completion of the corrective action ensures that a permanent solution has been implemented.

The Quality team will promptly audit any areas of activity or responsibility if there is a complaint or other concern about our compliance with GEL's policies and procedures or the quality of calibrations or analyses.

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APPENDIX A
REFERENCES

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- Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, American National Standard ANSI/ASQC E4-1994.
- Standard Methods for the Analysis of Water and Wastewater, 18th Edition.
- Standard Methods for the Analysis of Water and Wastewater, 19th Edition.
- State of Florida Department of Environmental Regulation Guidelines for Preparing Quality Assurance Plans, DER-QA-001/85.
- Test methods for Evaluating Solid Wastes, Physical/Chemical Methods, U.S. Environmental Protection Agency, SW-846, 1992.
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APPENDIX B
DEFINITIONS

The following definitions are used throughout the text of our Quality Systems Plan. These definitions were reprinted from "Definitions for Quality Systems," NELAC (July 2, 1998. The original source of each definition is provided.

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in the requirement documents. (ASQC)

Accreditation: the process by which an agency or organization evaluates and recognizes a program of study or an institution as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (Glossary of Quality Assurance Terms, QMAS, 8/31/92)

Analytical Detection Limit: the smallest amount of an analyte that can be distinguished in a sample by a given measurement procedure throughout a given (e.g., 0.95) confidence interval. (Applicable only to radiochemistry)

Analytical Reagent (AR) Grade: designation for the high purity of certain chemical reagents and solvents given by the American Chemical Society. (Quality Systems)

Batch: environmental samples, which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (Quality Systems)

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subject to the usual analytical and measurement process to establish a zero baseline or

background value and is sometimes used to adjust or correct routine analytical results. (ASQC, Definitions of environmental Quality Assurance Terms, 1996)

Blind Sample: a subsample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Calibrate: to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter or other device, or the correct value for each setting of a control knob. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

Calibration: the set of operations which establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system, or values represented by a material measure, and the corresponding known values of a measurement. (VIM - 6.13)

Calibration Curve: the graphical relationship between the known values, such as concentrations, of a series of calibration standards and their analytical response.

Calibration Standard: a solution prepared from the primary dilution standard solution or stock standard solutions and the internal standards and surrogate analytes. The calibration solutions are used to calibrate the instrument response with respect to analyte concentration. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Certified Reference Material (CRM): a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30 - 2.2)

Chain of Custody: an unbroken trail of accountability that documents the physical security of samples, data and records.

Confirmation: verification of the presence of a component through the use of an analytical technique that differs from the original test method. These may include:

Second column confirmation
Alternate wavelength
Derivatization
Mass spectral interpretation
Alternative detectors or
Additional cleanup procedures.

Corrective Action: action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useful form.

Detection Limit: the lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated degree of confidence. See Method Detection Limit.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC, Definitions of Environmental Quality Assurance Terms, 1996)

Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

Environmental Detection Limit (EDL): the smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC, Radioanalysis Subcommittee)

Holding Times (Maximum Allowable Holding Times): the maximum times that samples may be held prior to analysis and still be considered valid. (40 CFR Part 136)

Initial Demonstration of Capability: procedure to establish the ability of the laboratory to generate acceptable accuracy and precision which is included in many of the EPA's analytical test methods. In general, the procedure includes the addition of a specified concentration of each analyte (using a QC check sample) in each of four separate aliquots of laboratory pure water. These are carried through the entire analytical procedure and

the percentage recovery and the standard deviation are determined and compared to specified limits. (40 CFR Part 136)

nternal Standard: a known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method.

Laboratory: body that calibrates and/or tests.

NOTES:

1. In cases where a laboratory forms part of an organization that carries out other activities besides calibration and testing, the term "laboratory" refers only to those parts of that organization that are involved in the calibration and testing process.
2. As used herein, the term "laboratory" refers to a body that carries out calibration or testing
   - ◊ at or from a permanent location
   - ◊ at or from a temporary facility, or
   - ◊ in or from a mobile laboratory. (ISO 25)

Laboratory Control Sample (however named, such as laboratory fortified blank or spiked blank): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias to assess the performance of all or a portion of the measurement system. (NELAC)

Laboratory Duplicate: aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.

Legal Chain of Custody (COC): an unbroken trail of accountability that ensures the physical security of samples, data and records. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Limit of Detection (LOD): the lowest concentration level that can be determined by a single analysis and with a defined level of confidence to be statistically different from a blank. (Analytical Chemistry, 55, p.2217, Dec. 1983, modified)(See also Method Detection Limit.)

Matrix: the component or substrate which contains the analyte of interest. For purposes of batch determination, the following matrix types shall be used:

- ◊ Aqueous: any aqueous sample excluded from the definition of a drinking water matrix or saline/estuarine source. Includes surface water, groundwater and effluents.

- ◊ Drinking Water: any aqueous sample that has been designated a potable or potential potable water source.
- ◊ Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
- ◊ Non-aqueous liquid: any organic liquid with <15% settleable solids.
- ◊ Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- ◊ Solids: includes soils, sediments, sludges and other matrices with ≥15% settleable solids.
- ◊ Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.
- ◊ Air Samples: media used to retain the analyte of interest from an air sample such as sorbent tubes or summa canisters. Each medium shall be considered as a distinct matrix. (Quality Systems)

Matrix Spike (spiked sample, fortified sample): prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Matrix Spike Duplicate (spiked sample/fortified sample duplicate): a second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

May: permitted, but not required. (TRADE)

Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples containing an analyte of interest through all steps of the analytical procedures. (NELAC)

Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater that zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136 Appendix B)

Must: denotes a requirement that must be met. (Random House College Dictionary)

Negative Control: measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.

NELAC: National Environmental Laboratory Accreditation Conference. A voluntary organization of state and federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of National Environmental Laboratory Accreditation Program. (NELAC)

Performance Audit: the routine comparison of independently obtained quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Performance Based Measurement System (PBMS): a set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.

Positive Control: measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

Preservation: refrigeration and or reagents added at the time of sample collection to maintain the chemical and or biological integrity of the sample.

Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Proficiency Testing: determination of the laboratory calibration or testing performance by means of interlaboratory comparisons. (ISO/IEC Guide 2 - 12.6, amended)

Proficiency Testing Program: the aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results in comparison to peer laboratories and the collective demographics and results summary of all participating laboratories.

Protocol: a detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) which must be strictly followed.

Pure Reagent Water: shall be water in which no target analytes or interferences are present at a concentration which would impact the results when using a particular analytical test method.

Quality Assurance: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality within a stated level of confidence. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the need of users. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Quality Manual: a document stating the quality policy, quality system and quality practices of an organization. This may also be called a Quality Assurance Plan or a Quality Plan.

NOTE: the quality manual may call up other documentation relating to the laboratory's quality arrangements.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC. (ANSI/ASQC E-41994)

Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. For organic and general chemistry

Range: the difference between the minimum and the maximum set of values.

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes, which have been transcribed verbatim, dated and verified accurate by signature), the exact copy or exact transcript may be submitted.

Reagent Blank (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the

appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30 -2.1)

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM - 6.08)

Requirement: a translation of the needs into a set of individual quantified or descriptive specifications for the characteristics of an entity in order to enable its realization and examination.

Selectivity: (Analytical chemistry) the capability of a test method or instrument to respond to a target substance or constituent in the presence of nontarget substances.

Sensitivity: the capability of a test method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.

Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there will be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification so long as the requirement is fulfilled. (*Style Manual for Preparation of Proposed American National Standards*, American National Standards Institute, eighth edition, March 1991P)

Should: denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (*Style Manual for Preparation of Proposed American National Standards*, American National Standards Institute, eighth edition, March 1991P)

Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Spike: a known mass of target analyte added to a blank sample or subsample; used to determine recovery efficiency or for other quality control purposes.

Standard Reference Material (SRM): a certified reference material produced by the U.S. National Institute of Standards and Technology and characterized for absolute content, independent of analytical test method.

Surrogate: a substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (Glossary of Quality Assurance Terms, QAMS, 8/31/92)

Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material equipment, organism, physical phenomenon, process or service according to a specified procedure.

NOTE: the result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 - 12.4)

Test Method: defined technical procedure for performing a test.

Tolerance Chart: a chart in which the plotted quality control data is assessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level judged acceptable to meet overall quality/data use requirements instead of a statistical acceptance criteria (e.g. +/-3 sigma). (ANSI N42.23-1995, Measurement and Associated Instrument Quality Assurance for Radiochemistry Laboratories)

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

Verification: confirmation by examination and provision of evidence that specified requirements have been met.

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Validation: the process of substantiating specified performance criteria.

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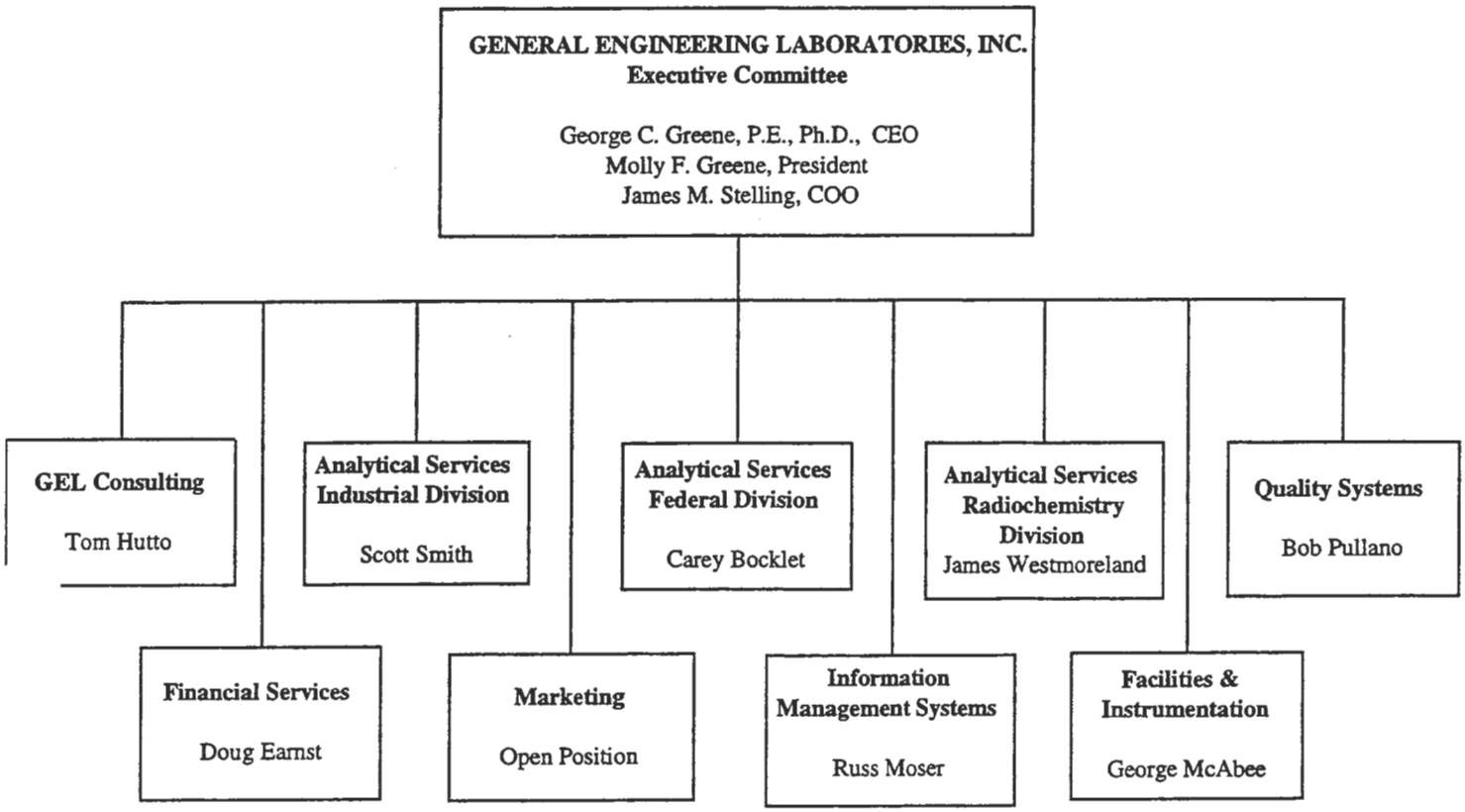
APPENDIX C
ORGANIZATIONAL CHART



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APPENDIX D
CERTIFICATIONS

- **U.S. Army Corps of Engineers** (USACE) - Validation by the Hazardous, Toxic and Radioactive Waste (HTRW) Center of Expertise..
- **U.S. Navy** approval for Naval Facilities Command Southern Division Remedial Action Contract
- **U.S. Department of Agriculture** Foreign soil importation permit # S-38709
- **Alabama** Department of Environmental Management, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program for Safe Drinking Water , Radiologicals and Inorganics (GEL ID: 41040)
- **California** Environmental Laboratory Accreditation Program Certification - Radiochemistry. (GEL ID: I-2089)
- **California** Environmental Laboratory Accreditation Program Certification -Non-Radiochemistry (GEL ID: I-2089)
- **Colorado** Department of Public Health and Environment, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program for Safe Drinking Water Chemistry and Radiochemistry
- **Connecticut** Department of Public Health - Potable Water, Waste Water and/or Trade Waste, Sewage and/or Effluent, Soil and Radiochemistry Certification. (GEL ID: PH-0169)
- **Delaware** Division of Public Health, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program for Safe Drinking Water (GEL ID: SC012)
- **Florida** Department of Health - Office of Laboratory Services, Safe Drinking Water and Environmental Water Certification. (Safe Drinking Water ID: 87294, Environmental Water ID: E87156)
- **Florida** Department of Health - Bureau of Radiation Control, Safe Drinking Water and Environmental Water Certification. (Safe Drinking Water ID: 87294, Environmental Water ID: E87156)
- **Georgia** Department of Natural Resources, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program for Safe Drinking Water (inorganics) (GEL ID: 938)*Expiration date: March 26, 1998)*
- **Idaho** Department of Health and Welfare - Bureau of Laboratories, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program for Safe Drinking Water - Inorganics and Radiologicals

- **Maryland** Department of Health and Mental Hygiene, Laboratories Administration, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program for Safe Drinking Water -Radiochemistry (EPI ID: 270)

- **Michigan** Department of Environmental Quality, Reciprocal Certification to SC DHEC. Drinking Water & Radiological Protection Division Certification for Inorganic Chemistry

- **Mississippi** Department of Health, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program for Safe Drinking Water (GEL ID: 29417).

- **Mississippi** Department of Health - Division of Radiological Health, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program for Safe Drinking Water (GEL ID: 29417)

- **Nevada** Department of Conservation and Natural Resources, Division of Environmental Protection, Clean Water Act Laboratory Certification

- **Nevada** Department of Human Resources, Health Division, Bureau of Licensure and Certification, Radiologicals

- **New Jersey** Department of Environmental Protection, Safe Drinking Water, Solid and Hazardous Waste, and Water Pollution Certification (GEL ID: 79002)

- **New York** Department of Health, Environmental Laboratory Approval Program Certification, Potable Water, Non-potable Waters and Solids/Hazardous Wastes (GEL ID: 11501)

- **North Carolina** Division of Environmental Management Lab Certification Program, Waste Waters/Ground Waters. (GEL ID: 233)

- **North Dakota** Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program, Safe Drinking Water, Clean Water, Solid/Hazardous Waste (GEL ID: R-158)

- **Oklahoma** Department of Environmental Quality, General Water Quality/Sludge Testing Laboratory Dual Certification (GEL ID: 9904)

- **Pennsylvania** Department of Environmental Protection - Bureau of Laboratories, Safe Drinking Water Certification (GEL ID: 68-485)

- **Rhode Island** Department of Health, Analytical Laboratory License for Potable Water, Non-Potable and Waste Water (GEL ID: 135)

- **Rhode Island** Department of Health, Analytical Laboratory License for Food, Surface Water, Air, Waste Water, Potable Water, Sewage - Radiochemistry (EPI ID: 138)

- **South Carolina** Department of Health and Environmental Control - Environmental Laboratory Certification Program, Clean Water, Safe Drinking Water and Solid/Hazardous Wastes (GEL ID: 10120)

- **South Carolina** Department of Health and Environmental Control (DHEC) Radioactive Material License (License #362)
- **Tennessee** Department of Health - Division of Laboratory Services, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program, Safe Drinking Water-Radiochemistry and Non-radiochemistry (GEL ID: 02934)
- **Tennessee** Department of Environment and Conservation - Division of Underground Storage Tanks, UST Approved Lab List
- **Texas** Department of Health - Bureau of Laboratories, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program, Safe Drinking Water (GEL ID: TX 213)
- **Texas** Department of Health - Bureau of Laboratories, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program, Safe Drinking Water (EPI ID: TX 214)
- **Utah** Department of Health, Division of Epidemiology and Laboratory Services, Services, Safe Drinking Water, Clean Water and Resource and Conservation and Recovery Act Certifications (Customer ID: GEL)
- **Virginia** Department of General Services - Division of Consolidated Laboratory Services, Safe Drinking Water Certification (GEL ID: 00151)
- **Virginia** Department of General Services - Division of Consolidated Laboratory Services, Safe Drinking Water Certification (EPI ID: 00111)
- **Washington** Department of Ecology, Non-Potable Water Certification (GEL ID: C223)
- **Wisconsin** Department of Natural Resources, Reciprocal Certification to SC DHEC Environmental Laboratory Certification Program, Safe Drinking Water, Clean Water, Solid/Hazardous Waste (GEL ID: 999887790)
- **West Virginia** Division of Environmental Protection, Office of Water Resources, Certification (GEL ID: 236)

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APPENDIX E
PRECISION AND ACCURACY CONTROL LIMITS

TABLE E1
PRECISION AND ACCURACY
SEMI-VOLATILES: AQUEOUS FOR BNA
SW846 Method 8270C and EPA 625

Analyte	Duplicate	MS	MSD	LCS	LCSD
1,2,4-Trichlorobenzene	N/A	54.1 - 99.6	0 - 24.4	45.7 - 97.7	0 - 30
1,3-Dichlorobenzene	N/A	70 - 130	10 - 30	34.7 - 91.8	0 - 30
1,4-Dichlorobenzene	N/A	58.5 - 92.1	0 - 23.6	34.6 -96.9	0 - 30
1,4-Dioxane	N/A	70 - 130	10 - 30	70 - 130	0 - 30
2,4,5-Trichlorophenol	N/A	70 - 130	10 - 30	63.4 - 95.9	0 - 30
2,4,6-Tribromophenol	0 - 30	10 - 123	10 - 30	10 -123	10 - 123
2,4,6-Trichlorophenol	N/A	70 - 130	10 - 30	62.1 - 94.2	0 - 30
2,4-Dichlorophenol	N/A	70 - 130	10 - 30	55.8 - 95.9	0 - 30
2,4-Dimethylphenol	N/A	70 - 130	10 - 30	14.7 - 123.1	0 - 30
2,4-Dinitrophenol	N/A	70 - 130	10 - 30	25.1 - 150.2	0 - 30
2,4-Dinitrotoluene	N/A	54.9 - 108.5	10 - 30	58.5 - 111	0 - 30
2,6-Dinitrotoluene	N/A	70 - 130	0 - 18.8	64.5 - 106.7	0 - 30
2-Chloronaphthalene	N/A	70 - 130	10 - 30	53.8 - 96.9	0 - 30
2-Chlorophenol	N/A	56.7 - 96.1	10 - 30	36.9 - 94.1	0 - 30
2-Fluorobiphenyl	0 - 30	51 - 114	10 - 30	51 - 113.8	51 - 114
2-Fluorophenol	0 - 30	21 - 100	10 - 30	21 - 100	21 - 100
2-methyl-4,6-dinitrophenol	N/A	70 - 130	10 - 30	70 - 130	0 - 30
2-Methylnaphthalene	N/A	70 - 130	10 - 30	48.2 - 97.8	0 - 30
2-Nitrophenol	N/A	70 - 130	10 - 30	49.4 - 101.2	0 - 30
3,3'-Dichlorobenzidine	N/A	70 - 130	10 - 30	70 - 130	0 - 30
4-Bromophenyl phenyl ether	N/A	70 - 130	10 - 30	65.6 - 103.6	0 - 30
4-chloro-3-methyl phenol	N/A	44.3 - 117.4	0 - 22.2	17.3 - 125.8	0 - 30
4-Chloroaniline	N/A	70 - 130	10 - 30	8.5 - 122.5	0 - 30
4-Chlorophenyl phenyl ether	N/A	70 - 130	10 - 30	64.9 - 103.8	0 - 30
4-Nitrophenol	N/A	.2 - 86.4	0 - 18.6	2.66 - 55.6	0 - 30
Acenaphthene	N/A	56.4 - 105.8	0 - 19.2	53 - 100	0 - 30
Acenaphthylene	N/A	70 - 130	10 - 30	57.4 - 100.1	0 - 30
Anthracene	N/A	70 - 130	10 - 30	67.6 - 104.5	0 - 30
Benzo(a)anthracene	N/A	70 - 130	10 - 30	61.7 - 105.1	0 - 30
Benzo(a)pyrene	N/A	70 - 130	10 - 30	67.1 - 105.4	0 - 30

Analyte	Duplicate	MS	MSD	LCS	LCSD
Benzo(b)fluoranthene	N/A	70 - 130	10 - 30	62 - 105.4	0 - 30
Benzo(g,h,i)perylene	N/A	70 - 130	10 - 30	42.3 - 129.6	0 - 30
Benzo(k)fluoranthene	N/A	70 - 130	10 - 30	65.8 - 109.5	0 - 30
Benzoic Acid	N/A	70 - 130	10 - 30	70 - 130	0 - 30
bis(2-Chloroethoxy)methane	N/A	70 - 130	10 - 30	47.5 - 101.2	0 - 30
bis(2-Chloroethyl) ether	N/A	70 - 130	10 - 30	39.7 - 93.5	0 - 30
bis(2-Chloroisopropyl)ether	N/A	70 - 130	10 - 30	13.7 - 119.3	0 - 30
bis(2-Ethylhexyl)phthalate	N/A	70 - 130	10 - 30	35.2 - 137.7	0 - 30
Butyl benzyl phthalate	N/A	70 - 130	10 - 30	39.8 - 117.3	0 - 30
Chrysene	N/A	70 - 130	10 - 30	56.5 - 110.5	0 - 30
Di-n-butyl phthalate	N/A	70 - 130	10 - 30	57.8 - 115.4	0 - 30
Di-n-octyl phthalate	N/A	70 - 130	10 - 30	49 - 126.1	0 - 30
Dibenzo(a,h)anthracene	N/A	70 - 130	10 - 30	58.6 - 117.7	0 - 30
Dibenzofuran	N/A	70 - 130	10 - 30	60 - 109.2	0 - 30
Diethyl phthalate	N/A	70 - 130	10 - 30	17.8 - 144.4	0 - 30
Dimethyl phthalate	N/A	70 - 130	10 - 30	70 - 130	0 - 30
Fluoranthene	N/A	70 - 130	10 - 30	67.6 - 104.4	0 - 30
Fluorene	N/A	70 - 130	10 - 30	62.4 - 57.7	0 - 30
Hexachlorobenzene	N/A	70 - 130	10 - 30	66.9 - 107.1	0 - 30
Hexachlorobutadiene	N/A	70 - 130	10 - 30	40.1 - 101.9	0 - 30
Hexachlorocyclopentadiene	N/A	70 - 130	10 - 30	12.4 - 95.3	0 - 30
Hexachloroethane	N/A	70 - 130	10 - 30	25.4 - 100.5	0 - 30
Indeno (1,2,3-c,d) pyrene	N/A	70 - 130	10 - 30	59.6 - 130.2	0 - 30
Isophorone	N/A	70 - 130	10 - 30	48 - 109.5	0 - 30
m,p-Cresol	N/A	70 - 130	10 - 30	11.2 - 108.2	0 - 30
m-Nitroaniline	N/A	70 - 130	10 - 30	70 - 130	0 - 30
N-Nitrosodiphenylamine	N/A	70 - 130	10 - 30	59.2 - 114.4	0 - 30
N-Nitrosodipropylamine	N/A	45.3 - 118.7	10 - 30	52.1 - 103.9	0 - 30
Naphthalene	N/A	70 - 130	10 - 30	55.9 - 94.9	0 - 30
Nitrobenzene	N/A	70 - 130	10 - 30	56.3 - 100.3	0 - 30
Nitrobenzene-d5	0 - 30	35 - 114	10 - 30	35 - 114	43 - 114
o-Cresol	N/A	70 - 130	10 - 30	17.7 - 100.9	0 - 30
o-Nitroaniline	N/A	70 - 130	10 - 30	70 - 130	0 - 30
p-Nitroaniline	N/A	70 - 130	10 - 30	70 - 130	0 - 30
p-Terphenyl-d14	0 - 30	33 - 141	10 - 30	33 - 141	33 - 141
Pentachlorophenol	N/A	54.1 - 117.5	0 - 18.9	49.8 - 119.5	0 - 30
Phenanthrene	N/A	70 - 130	10 - 30	67.7 - 104	0 - 30

Phenol	N/A	11.7 - 68.5	0 - 29.2	1.3 - 70.1	0 - 30
Phenol-d6	0 - 30	10 - 80.9	N/A	10.1 - 80.9	10 - 80.9
Pyrene	N/A	52.6 - 125.3	10 - 30	45.4 - 108.7	0 - 30

Surrogate Acceptance Criteria	Lower	Upper
Nitrobenzene-d5	35.3	108
2-Fluorobiphenyl	41.2	107
p-Terphenyl-d4	36.6	110
phenol-d6	10.9	54.6
2-Fluorophenol	23.6	75.9
2,4,6-Tribromophenol	41	122

TABLE E1A
PRECISION AND ACCURACY
SEMI-VOLATILES: NON-AQUEOUS FOR BNA
SW846 Method 8270C

Analyte	Duplicate	MS	MSD	LCS	LCSD
1,2,4-Trichlorobenzene	N/A	46.3-101.9	0-18.9	38.2-109.8	0 – 30
1,3-Dichlorobenzene	N/A	70 – 130	10 - 30	70-130	0 – 30
1,4-Dichlorobenzene	N/A	39-100.6	0-19.5	41.8-103	0 – 30
1,4-Dioxane	N/A	70 – 130	10 - 30	70 – 130	0 – 30
2,4,5-Trichlorophenol	N/A	70 – 130	10 - 30	58.4-99.3	0 – 30
2,4,6-Tribromophenol	0 - 30	10 – 123	10 - 30	52.7-97.7	10 – 123
2,4,6-Trichlorophenol	N/A	70 – 130	10 - 30	62.1 - 94.2	0 – 30
2,4-Dichlorophenol	N/A	70 – 130	10 - 30	50.8-98.2	0 – 30
2,4-Dimethylphenol	N/A	70 – 130	10 - 30	53.3-92.2	0 – 30
2,4-Dinitrophenol	N/A	70 – 130	10 - 30	21.1-113.4	0 – 30
2,4-Dinitrotoluene	N/A	41-111.1	0-21.6	56.5-119.2	0 – 30
2,6-Dinitrotoluene	N/A	70 – 130	0 - 18.8	59.6-98.9	0 – 30
2-Chloronaphthalene	N/A	70 – 130	10 - 30	45.7-106.6	0 – 30
2-Chlorophenol	N/A	50.1-99.8	0-19.7	45.5-106.6	0 – 30
2-Fluorobiphenyl	0 - 30	51 – 114	10 - 30	51 - 113.8	51 – 114
2-Fluorophenol	0 - 30	21 – 100	10 - 30	21 - 100	21 – 100
2-methyl-4,6-dinitrophenol	N/A	70 – 130	10 - 30	70 - 130	0 – 30
2-Methylnaphthalene	N/A	70 – 130	10 - 30	48.2 - 97.8	0 – 30
2-Nitrophenol	N/A	70 – 130	10 - 30	44.9-99.8	0 – 30
3,3'-Dichlorobenzidine	N/A	70 – 130	10 - 30	34.5-129.6	0 – 30
4-Bromophenyl phenyl ether	N/A	70 – 130	10 - 30	58.7-113.3	0 – 30
4-chloro-3-methyl phenol	N/A	50.5-109.8	0 – 21.7	57.5-100.9	0 – 30
4-Chloroaniline	N/A	70 – 130	10 - 30	8.8-76.2	0 – 30
4-Chlorophenyl phenyl ether	N/A	70 – 130	10 – 30	52-112.9	0 – 30
4-Nitrophenol	N/A	42.6-118.5	0-23.3	30.4-135.9	0 – 30
Acenaphthene	N/A	54.9-104.9	0-18.9	48.2-107.6	0 – 30
Acenaphthylene	N/A	70 – 130	10 - 30	42.7-112.2	0 – 30
Anthracene	N/A	70 – 130	10 - 30	61.1-106.8	0 – 30
Benzo(a)anthracene	N/A	70 – 130	10 - 30	45.3-111.9	0 – 30
Benzo(a)pyrene	N/A	70 – 130	10 - 30	65.4-105	0 – 30

Analyte	Duplicate	MS	MSD	LCS	LCSD
Benzo(b)fluoranthene	N/A	70 – 130	10 - 30	53.3-116.8	0 – 30
Benzo(g,h,i)perylene	N/A	70 – 130	10 - 30	46.1-125.4	0 – 30
Benzo(k)fluoranthene	N/A	70 – 130	10 - 30	50.2-121.9	0 – 30
Benzoic Acid	N/A	70 – 130	10 - 30	70 - 130	0 – 30
bis(2-Chloroethoxy)methane	N/A	70 – 130	10 - 30	36.2-108	0 – 30
bis(2-Chloroethyl) ether	N/A	70 – 130	10 - 30	28-102.9	0 – 30
bis(2-Chloroisopropyl)ether	N/A	70 – 130	10 - 30	70-130	0 – 30
bis(2-Ethylhexyl)phthalate	N/A	70 – 130	10 - 30	45.2-136.7	0 – 30
Butyl benzyl phthalate	N/A	70 – 130	10 - 30	49.7-110.7	0 – 30
Chrysene	N/A	70 – 130	10 - 30	22.2-144.9	0 – 30
Di-n-butyl phthalate	N/A	70 – 130	10 - 30	66.9-111.8	0 – 30
Di-n-octyl phthalate	N/A	70 – 130	10 - 30	61.4-119.3	0 – 30
Dibenzo(a,h)anthracene	N/A	70 – 130	10 - 30	52.3-120.1	0 – 30
Dibenzofuran	N/A	70 – 130	10 - 30	47.6-113.2	0 – 30
Diethyl phthalate	N/A	70 – 130	10 - 30	62.2-106.7	0 – 30
Dimethyl phthalate	N/A	70 – 130	10 - 30	58.1-100.6	0 – 30
Fluoranthene	N/A	70 – 130	10 - 30	63.4-104.9	0 – 30
Fluorene	N/A	70 – 130	10 - 30	49.7-113.5	0 – 30
Hexachlorobenzene	N/A	70 – 130	10 - 30	57.8-112.5	0 – 30
Hexachlorobutadiene	N/A	70 – 130	10 - 30	40.9-111	0 – 30
Hexachlorocyclopentadiene	N/A	70 – 130	10 - 30	21.2-104.2	0 – 30
Hexachloroethane	N/A	70 – 130	10 - 30	70-130	0 – 30
Indeno (1,2,3-c,d) pyrene	N/A	70 – 130	10 - 30	34.1-135.5	0 – 30
Isophorone	N/A	70 – 130	10 - 30	28.4-115.4	0 – 30
m,p-Cresol	N/A	70 – 130	10 - 30	70-130	0 – 30
m-Nitroaniline	N/A	70 – 130	10 - 30	70 – 130	0 – 30
N-Nitrosodiphenylamine	N/A	70 – 130	10 - 30	71.4-109.5	0 – 30
N-Nitrosodipropylamine	N/A	0-20.4	10 - 30	14.9-116.2	0 – 30
Naphthalene	N/A	70 – 130	10 - 30	38.2-113	0 – 30
Nitrobenzene	N/A	70 – 130	10 - 30	37.4-109.8	0 – 30
Nitrobenzene-d5	0 - 30	35 – 114	10 - 30	35 – 114	43 – 114
o-Cresol	N/A	70 – 130	10 - 30	70-130	0 – 30
o-Nitroaniline	N/A	70 – 130	10 - 30	70 – 130	0 – 30
p-Nitroaniline	N/A	70 – 130	10 - 30	70 – 130	0 – 30
p-Terphenyl-d14	0 – 30	33 – 141	10 - 30	33 – 141	33 – 141
Pentachlorophenol	N/A	49.1-122.6	0-24.1	45.4-102.6	0 – 30
Phenanthrene	N/A	70 – 130	10 - 30	58.2-110.6	0 – 30

Analyte	Duplicate	MS	MSD	LCS	LCSD
Phenol	N/A	0-19.4	0 - 29.2	36.2-99.7	0 – 30
Phenol-d6	0 – 30	10-80.9	N/A	10.1 - 80.9	10 - 80.9
Pyrene	N/A	57.2-123.4	0-21.4	50.7-110.4	0 – 30

Surrogate Acceptance Criteria	Lower	Upper
Nitrobenzene-d5	35.3	108
2-Fluorobiphenyl	41.2	107
p-Terphenyl-d4	36.6	110
phenol-d6	10.9	54.6
2-Fluorophenol	23.6	75.9
2,4,6-Tribromophenol	41	122

TABLE E2
PRECISION AND ACCURACY
SEMI-VOLATILE: AQUEOUS FOR EXPLOSIVES
SW846 Method 8330

Analyte	Duplicate	MS	MSD	LCS	LCSD
2,4,6-Trinitrotoluene	0 - 30	66.2 - 126.7	0 - 16	61.3 - 130.3	0 - 30
2,4-Dinitrotoluene	0 - 30	70.1 - 127.4	0 - 13.3	60.1 - 132.2	0 - 30
2,6-Dinitrotoluene	0 - 30	62.8 - 134.2	0 - 19.3	64.4 - 127.5	0 - 30
2-Amino-4,6-Dinitrotoluene	0 - 30	58.7 - 134.1	0 - 15.8	58.6 - 133.3	0 - 30
4-Amino-2,6-Dinitrotoluene	0 - 30	56.3 - 144.9	0 - 12.7	58.9 - 137.3	0 - 30
HMX	0 - 30	63.8 - 145.2	0 - 14.4	65.8 - 146.8	0 - 30
Nitrobenzene	0 - 30	57.6 - 118.6	0 - 20.4	56.6 - 113.5	0 - 30
PETN	N/A	70 - 130	0 - 30	43.6 - 139	0 - 30
RDX	0 - 30	64.9 - 133.2	0 - 15.9	69.7 - 129.7	0 - 30
TETRYL	0 - 30	68 - 133.4	0 - 13.4	66 - 134.2	0 - 30
m-Dinitrobenzene	0 - 30	70.8 - 124.9	0 - 15	66.2 - 126.5	0 - 30
m-Nitrotoluene	0 - 30	56.5 - 120.8	0 - 22.8	56.9 - 115.6	0 - 30
o-Nitrotoluene	0 - 30	55.4 - 120.9	0 - 23.1	56.7 - 114.9	0 - 30
p-Nitrotoluene	0 - 30	63.8 - 112.9	0 - 23.1	54.6 - 113.6	0 - 30
sym-Trinitrobenzene	0 - 30	67.7 - 113.1	0 - 13.2	66 - 113.2	0 - 30

TABLE E2A
PRECISION AND ACCURACY
SEMI-VOLATILE: NON-AQUEOUS FOR EXPLOSIVES
SW846 Method 8330

Analyte	Duplicate	MS	MSD	LCS	LCSD
2,4,6-Trinitrotoluene	0 - 30	64.9 - 164.7	0 - 30	60.2 - 134.9	0 - 30
2,4-Dinitrotoluene	0 - 30	65.8 - 160.8	0 - 30	59.7 - 134.5	0 - 30
2,6-Dinitrotoluene	0 - 30	59.7 - 152.9	0 - 30	59.9 - 123.9	0 - 30
2-Amino-4,6-Dinitrotoluene	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
4-Amino-2,6-Dinitrotoluene	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
HMX	0 - 30	54.9 - 156.7	0 - 30	54.3 - 151.5	0 - 30
Nitrobenzene	0 - 30	66.4 - 156.7	0 - 30	61.6 - 124.3	0 - 30
PETN	N/A	44.5 - 131.8	0 - 30	44.5 - 142.7	0 - 30
RDX	0 - 30	61.1 - 155	0 - 30	56.7 - 139.3	0 - 30
TETRYL	0 - 30	55.9 - 147.3	0 - 30	63.3 - 133.6	0 - 30
m-Dinitrobenzene	0 - 30	65.5 - 162	0 - 30	59.6 - 130.9	0 - 30
m-Nitrotoluene	0 - 30	64.1 - 153.4	0 - 30	61.9 - 118.5	0 - 30
o-Nitrotoluene	0 - 30	63.8 - 154.5	0 - 30	62.6 - 120.4	0 - 30
p-Nitrotoluene	0 - 30	63.5 - 155.1	0 - 30	62.6 - 121	0 - 30
sym-Trinitrobenzene	0 - 30	57.5 - 149.4	0 - 30	67.1 - 109	0 - 30

TABLE E3
PRECISION AND ACCURACY
VOLATILE/SEMI-VOLATILES: AQUEOUS FOR FID
SW846 Method 8015B

Analyte	Duplicate	MS	MSD	LCS	LCSD
1,1,1-Trichloroethane	N/A	70 - 130	0 - 20	70 - 130	0 - 20
1,1,2,2-Tetrachlorethane	N/A	70 - 130	0 - 20	70 - 130	0 - 20
1,2-Dichloroethane	N/A	70 - 130	0 - 20	70 - 130	0 - 20
1,3-Dioxalane	N/A	70 - 130	0 - 20	70 - 130	0 - 20
1,4-Dioxane	N/A	70 - 130	0 - 20	70 - 130	0 - 20
2-Butanone	N/A	70 - 130	0 - 20	70 - 130	0 - 20
2-Chloroethanol	N/A	70 - 130	0 - 20	70 - 130	0 - 20
2-Propyn-1-o1	N/A	70 - 130	0 - 20	70 - 130	0 - 20
4-Methyl-2-pentanone	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Acetone	N/A	70 - 130	0 - 20	68.2 - 140	0 - 20
Acrylonitrile	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Benzene	N/A	70 - 130	0 - 20	74.7 - 148	0 - 20
Butyl Acetate	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Butyl Cellosolve	0 - 30	70 - 130	0 - 20	70 - 130	0 - 20
Carbon Disulfide	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Carbon Tetrachloride	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Chloroform	N/A	70 - 130	0 - 20	63.5 - 162	0 - 20
Creosote	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Cyclohexane	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Diesel Range Organics	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Diethylene Glycol	0 - 30	70 - 130	0 - 20	70 - 130	0 - 20
Ethanol	0 - 30	70 - 130	0 - 20	29 - 158	0 - 20
Ethyl Acetate	N/A	70 - 130	0 - 20	52.2 - 163	0 - 20
Ethyl Ether	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Ethylbenzene	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Ethylene Glycol	0 - 30	70 - 130	0 - 20	70 - 130	0 - 20
Ethylene Methylether	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Ethylene Monoethylether	0 - 30	70 - 130	0 - 20	70 - 130	0 - 20
Freon	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Fuel Oil #6	N/A	70 - 130	0 - 20	70 - 130	0 - 20

Analyte	Duplicate	MS	MSD	LCS	LCSD
Heptane	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Hexane	N/A	70 - 130	0 - 20	67.2 - 160	0 - 20
Isobutyl Alcohol	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Isopropyl Alcohol	0 - 30	70 - 130	0 - 20	70 - 130	0 - 20
Isopropyl Ether	N/A	70 - 130	0 - 20	70 - 130	0 - 20
JP4	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Kerosene	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Methyl Alcohol	0 - 30	70 - 130	0 - 20	35.4 - 172	0 - 20
Methylene Chloride	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Mineral Spirits	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Naphthalene	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Pentane	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Propylene	N/A	50 - 130	N/A	50 - 130	N/A
Propylene Glycol	0 - 30	70 - 130	0 - 20	70 - 130	0 - 20
TPH - Nonvolatile Fraction	0 - 30	70 - 130	0 - 20	70 - 130	0 - 20
TPH - Volatile Fraction	0 - 30	70 - 130	0 - 20	50 - 124	0 - 20
Tetrachloroethylene	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Tetraehtylene Glycol	0 - 30	70 - 130	0 - 20	70 - 130	0 - 20
Toluene	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Trichloroethylene	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Triethylene Glycol	0 - 30	70 - 130	0 - 20	70 - 130	0 - 20
Varsal	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Waste Oil Screen	N/A	70 - 130	0 - 20	70 - 130	0 - 20
Xylenes (Total)	N/A	70 - 130	0 - 20	67.5 - 130	0 - 20
meta-Xylenes	N/A	70 - 130	0 - 20	70 - 130	0 - 20
n-Butyl Alcohol	N/A	70 - 130	0 - 20	70 - 130	0 - 20
n-Propyl Alcohol	N/A	70 - 130	0 - 20	70 - 130	0 - 20
o-Terphenyl	N/A	70 - 130	0 - 20	70 - 130	0 - 20
o-Xylene	N/A	70 - 130	0 - 20	70 - 130	0 - 20
p-Xylene	N/A	70 - 130	0 - 20	70 - 130	0 - 20

TABLE E4
PRECISION AND ACCURACY
VOLATILE/SEMI-VOLATILES: NON-AQUEOUS FOR FID
SW846 Method 8015B

Analyte	Duplicate	MS	MSD	LCS	LCSD
1,1,1-Trichloroethane	N/A	70 - 130	0 - 50	0 - 130	0 - 20
1,1,2,2-Tetrachlorethane	N/A	70 - 130	0 - 50	0 - 130	0 - 20
1,2-Dichloroethane	N/A	70 - 130	0 - 50	0 - 130	0 - 20
1,3-Dioxalane	N/A	70 - 130	0 - 50	0 - 130	0 - 20
1,4-Dioxane	N/A	70 - 130	0 - 50	0 - 130	0 - 20
2-Butanone	N/A	70 - 130	0 - 50	0 - 130	0 - 20
2-Chloroethanol	N/A	70 - 130	0 - 50	0 - 130	0 - 20
2-Propyn-1-o1	N/A	70 - 130	0 - 50	0 - 130	0 - 20
4-Methyl-2-pentanone	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Acetone	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Acrylonitrile	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Benzene	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Butyl Acetate	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Butyl Cellosolve	0 - 30	70 - 130	0 - 50	0 - 130	0 - 20
Carbon Disulfide	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Carbon Tetrachloride	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Chloroform	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Creosote	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Cyclohexane	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Diesel Range Organics	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Diethylene Glycol	0 - 30	70 - 130	0 - 50	0 - 130	0 - 20
Ethanol	0 - 30	70 - 130	0 - 50	0 - 130	0 - 20
Ethyl Acetate	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Ethyl Ether	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Ethylbenzene	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Ethylene Glycol	0 - 30	70 - 130	0 - 50	0 - 130	0 - 20
Ethylene Methylether	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Ethylene Monoethylether	0 - 30	70 - 130	0 - 50	0 - 130	0 - 20
Freon	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Fuel Oil #6	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Heptane	N/A	70 - 130	0 - 50	0 - 130	0 - 20

Analyte	Duplicate	MS	MSD	LCS	LCSD
Hexane	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Isobutyl Alcohol	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Isopropyl Alcohol	0 - 30	70 - 130	0 - 50	0 - 130	0 - 20
Isopropyl Ether	N/A	70 - 130	0 - 50	0 - 130	0 - 20
JP4	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Methanol	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Kerosene	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Methyl Alcohol	0 - 30	70 - 130	0 - 50	0 - 130	0 - 20
Methylene Chloride	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Mineral Spirits	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Naphthalene	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Pentane	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Propylene Glycol	0 - 30	70 - 130	0 - 50	0 - 130	0 - 20
TPH - Nonvolatile Fraction	0 - 30	52.9 - 119.7	0 - 50	45.6 - 145.5	0 - 20
TPH - Volatile Fraction	0 - 30	70 - 130	0 - 50	0 - 130	0 - 20
Tetrachloroethylene	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Tetraehtylene Glycol	0 - 30	70 - 130	0 - 50	0 - 130	0 - 20
Toluene	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Trichloroethylene	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Triethylene Glycol	0 - 30	70 - 130	0 - 50	0 - 130	0 - 20
Varsal	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Waste Oil Screen	N/A	70 - 130	0 - 50	0 - 130	0 - 20
Xylenes (Total)	N/A	70 - 130	0 - 50	0 - 130	0 - 20
meta-Xylenes	N/A	70 - 130	0 - 50	0 - 130	0 - 20
n-Butyl Alcohol	N/A	70 - 130	0 - 50	0 - 130	0 - 20
n-Propyl Alcohol	N/A	70 - 130	0 - 50	0 - 130	0 - 20
o-Terphenyl	N/A	70 - 130	0 - 50	0 - 130	0 - 20
o-Xylene	N/A	70 - 130	0 - 50	0 - 130	0 - 20
p-Xylene	N/A	70 - 130	0 - 50	0 - 130	0 - 20

PRECISION AND ACCURACY
SEMI-VOLATILE: AQUEOUS FOR HERBICIDES
SW846 Method 8051B

Analyte	Duplicate	MS	MSD	LCS	LCSD
2,4,5-T	0 - 30	73.2 - 132.6	0 - 32.3	49.9 - 123.4	0 - 25.16
2,4,5-TP	N/A	62.3 - 113.1	0 - 39.8	48.7 - 119.9	0 - 26.14
2,4-D	N/A	55.6 - 133.9	0 - 36.2	48.6 - 121.3	0 - 27.18
4-(2,4-Dichlorophenoxy) Butyric	N/A	52.9 - 139.2	0 - 30	52.9 - 139.2	0 - 30
Dicamba	N/A	50 - 150	0 - 30	50 - 150	N/A

TABLE E5
PRECISION AND ACCURACY
SEMI-VOLATILE: NON-AQUEOUS FOR HERBICIDES
SW846 Method 8051B

Analyte	Duplicate	MS	MSD	LCS	LCSD
2,4,5-T	0 - 30	50 - 150	50 - 150	50 - 150	0 - 30
2,4,5-TP	N/A	50 - 150	50 - 150	33 - 73	0 - 30
2,4-D	N/A	50 - 150	50 - 150	50 - 150	0 - 30
4-(2,4-Dichlorophenoxy) Butyric	N/A	50 - 150	50 - 150	50 - 150	N/A
Dicamba	N/A	50 - 150	50 - 150	50 - 150	N/A

TABLE E6

SEMI-VOLATILE: NON-AQUEOUS FOR HPLC: SW846 Method 8310

Analyte	Duplicate	MS	MSD	LCS	LCSD
Acenapthene	0 - 66.2	41.4 - 108.9	0 - 30.6	65.5 - 110.9	0 - 30
Acenapthylene	0 - 47.2	70.4 - 130.9	0 - 19.6	66.5 - 114.8	0 - 30
Anthracene	0 - 45.5	44.7 - 127.4	0 - 22.4	61.6 - 130.7	0 - 30
Benzo (a) anthracene	0 - 36	53.3 - 130	0 - 15.3	66.1 - 116.2	0 - 30
Benzo (a) pyrene	0 - 52	37.5 - 134.1	0 - 24.9	56.9 - 121.5	0 - 30
Benzo (b) flouranthene	0 - 38	70 - 131.7	0 - 33.5	65.4 - 116.6	0 - 30
Benzo (ghi) perylene	0 - 68	71.4 - 136.4	0 - 22.1	62.9 - 119.4	0 - 30
Benzo (k) flouranthene	0 - 89	62.3 - 139.9	0 - 8.5	54.6 - 125.5	0 - 30
Chrysene	0 - 44	64 - 125.1	0 - 30.2	66.4 - 122.7	0 - 30
Decaflourobiphenyl	50 - 150	63.9 - 119.7	0 - 33.6	68 - 111.3	50 - 150
Dibenzo (a,b) anthracene	0 - 48	54.8 - 127.2	0 - 29.3	67.8 - 121.2	0 - 30
Flouranthene	0 - 35	50.2 - 118.6	0 - 18.5	59.9 - 121.1	0 - 30
Flourene	0 - 56.5	52.8 - 110.1	0 - 25.6	67.6 - 114.7	0 - 30
Indeno(1,2,2-c,d)pyrene	0 - 43	51.7 - 131.4	0 - 21.1	67.2 - 113.2	0 - 30
Napthalene	0 - 48.4	37.4 - 115.2	0 - 29.6	67.5 - 113.3	0 - 30
Phenanthrene	0 - 44.5	51.9 - 117.7	0 - 24.1	65.6 - 116.5	0 - 30
Pyrene	0 - 42	47.9 - 113.9	0 - 23.8	63.9 - 115.2	0 - 30

TABLE E6A

SEMI-VOLATILE: AQUEOUS FOR HPLC: SW846 Method 8310

Analyte	Duplicate	MS	MSD	LCS	LCSD
Acenapthene	0 - 66.2	41.4 - 108.9	0 - 30.6	65.5 - 110.9	0 - 30
Acenapthylene	0 - 47.2	70.4 - 130.9	0 - 19.6	66.5 - 114.8	0 - 30
Anthracene	0 - 45.5	44.7 - 127.4	0 - 22.4	61.6 - 130.7	0 - 30
Benzo (a) anthracene	0 - 36	53.3 - 130	0 - 15.3	66.1 - 116.2	0 - 30
Benzo (a) pyrene	0 - 52	37.5 - 134.1	0 - 24.9	56.9 - 121.5	0 - 30
Benzo (b) flouranthene	0 - 38	70 - 131.7	0 - 33.5	65.4 - 116.6	0 - 30
Benzo (ghi) perylene	0 - 68	71.4 - 136.4	0 - 22.1	62.9 - 119.4	0 - 30
Benzo (k) flouranthene	0 - 89	62.3 - 139.9	0 - 8.5	54.6 - 125.5	0 - 30
Chrysene	0 - 44	64 - 125.1	0 - 30.2	66.4 - 122.7	0 - 30
Decaflourobiphenyl	50 - 150	63.9 - 119.7	0 - 33.6	68 - 111.3	50 - 150
Dibenzo (a,b) anthracene	0 - 48	54.8 - 127.2	0 - 29.3	67.8 - 121.2	0 - 30
Flouranthene	0 - 35	50.2 - 118.6	0 - 18.5	59.9 - 121.1	0 - 30
Flourene	0 - 56.5	52.8 - 110.1	0 - 25.6	67.6 - 114.7	0 - 30
Indeno(1,2,2-c,d)pyrene	0 - 43	51.7 - 131.4	0 - 21.1	67.2 - 113.2	0 - 30
Napthalene	0 - 48.4	37.4 - 115.2	0 - 29.6	67.5 - 113.3	0 - 30
Phenanthrene	0 - 44.5	51.9 - 117.7	0 - 24.1	65.6 - 116.5	0 - 30
Pyrene	0 - 42	47.9 - 113.9	0 - 23.8	63.9 - 115.2	0 - 30

TABLE E7

PRECISION AND ACCURACY
SEMI-VOLATILE: NON-AQUEOUS FOR PCB
SW846 Method 8082

Analyte	Duplicate	MS	MSD	LCS	LCSD
4CMX (surrogate)	N/A	25 – 110	N/A	N/A	N/A
Decachlorobiphenyl (surr.)	N/A	46 - 131	N/A	N/A	N/A
PCB-1260	N/A	31.5 - 159.2	0 - 26.2	53.6 - 136.8	0 - 36

TABLE E7A

PRECISION AND ACCURACY
SEMI-VOLATILE: AQUEOUS FOR PCB
SW846 Method 8082

Analyte	Duplicate	MS	MSD	LCS	LCSD
4CMX (surrogate)	N/A	31 - 126	N/A	N/A	N/A
Decachlorobiphenyl (surr.)	N/A	39 - 133	N/A	N/A	N/A
PCB-1260	N/A	31.5 - 159.2	0 - 26.2	53.6 - 136.8	0 - 36

TABLE E8
PRECISION AND ACCURACY
SEMI-VOLATILE: AQUEOUS FOR PESTICIDES SW846 Method 8081A and EPA 608

Analyte	Duplicate	MS	MSD	LCS	LCSD
4,4'-DDD		70 - 130	0 - 40	70 - 130	0 - 30
4,4'-DDE		70 - 130	0 - 40	70 - 130	0 - 30
4,4'-DDT		50.9 - 133.8	0 - 40	49.2 - 134	0 - 30
Aldrin		53 - 126	0 - 27.3	52 - 134.5	0 - 30
Alpha BHC		70 - 130	0 - 40	70 - 130	0 - 30
Beta BHC		70 - 130	0 - 40	70 - 130	0 - 30
Delta BHC		70 - 130	0 - 40	70 - 130	0 - 30
Dieldrin		50.3 - 126.3	0 - 26.4	62.6 - 123.8	0 - 30
Endosulfan I		70 - 130	0 - 40	70 - 130	0 - 30
Endosulfan II		70 - 130	0 - 40	70 - 130	0 - 30
Endosulfan Sulfate		70 - 130	0 - 40	70 - 130	0 - 30
Endrin		49.3 - 137.6	0 - 25.2	52.6 - 141.6	0 - 30
Endrin Aldehyde		70 - 130	0 - 40	70 - 130	0 - 30
Gamma BHC		50.5 - 132.4	0 - 25.6	55.4 - 131.5	0 - 30
Heptachlor		49.6 - 130.9	0 - 30.1	58.6 - 137.3	0 - 30
Heptachlor epoxide		70 - 130	0 - 40	70 - 130	0 - 30
Methoxychlor		70 - 130	0 - 40	70 - 130	0 - 30

Surrogates:	Lower Limit	Upper Limit
4-CMX	46	125
Decachlorobiphenyl	42	143

TABLE E9
PRECISION AND ACCURACY
SEMI-VOLATILE: NON-AQUEOUS FOR PESTICIDES SW846 Method 8081A

Analyte	Duplicate	MS	MSD	LCS	LCSD
4,4'-DDD		70 - 130	0 - 40	70 - 130	0 - 30
4,4'-DDE		70 - 130	0 - 40	70 - 130	0 - 30
4,4'-DDT		51.8 - 126.4	0 - 30.3	59.3 - 121.1	0 - 30
Aldrin		50.4 - 122.7	0 - 22.5	53.5 - 127.5	0 - 30
Alpha BHC		70 - 130	0 - 40	70 - 130	0 - 30
Beta BHC		70 - 130	0 - 40	70 - 130	0 - 30
Analyte	**Duplicate**	**MS**	**MSD**	**LCS**	**LCSD**
Delta BHC		70 - 130	0 - 40	70 - 130	0 - 30
Dieldrin		52.1 - 108.3	0 - 21.9	51.6 - 125.4	0 - 30
Endosulfan I		70 - 130	0 - 40	70 - 130	0 - 30
Endosulfan II		70 - 130	0 - 40	70 - 130	0 - 30
Endosulfan Sulfate		70 - 130	0 - 40	70 - 130	0 - 30
Endrin		47.2 - 137.4	0 - 19.1	48.2 - 132.5	0 - 30
Endrin Aldehyde		70 - 130	0 - 40	70 - 130	0 - 30
Gamma BHC		56.2 - 118.1	0 - 26.2	56.9 - 119	0 - 30
Heptachlor		53.3 - 131.7	0 - 20.8	56.5 - 128.4	0 - 30
Heptachlor epoxide		70 - 130	0 - 40	70 - 130	0 - 30
Methoxychlor		70 - 130	0 - 40	70 - 130	0 - 30

Surrogates:	Lower Limit	Upper Limit
4-CMX	36.5	131
Decachlorobiphenyl	50.7	135

TABLE E10

PRECISION AND ACCURACY

VOLATILES: AQUEOUS FOR EPA524.2, 624 AND SW846 Method 8260B

Analyte	Duplicate	MS	MSD	LCS	LCSD
1,1,1,2-Tetrachloroethane	N/A	70 - 130	0 - 30	79 - 121	0 - 30
1,1,1-Trichloroethane	N/A	70 - 130	0 - 30	73 - 127	0 - 30
1,1,2,2-Tetrachloroethane	N/A	70 - 130	0 - 30	65 - 134	0 - 30
1,1,2-Trichloroethane	N/A	70 - 130	0 - 30	71 - 124	0 - 30
1,1-Dichloroethane	N/A	70 - 130	0 - 30	70 - 131	0 - 30
1,1-Dichloroethylene	N/A	82 - 136	0 - 30	70 - 144	0 - 33
1,1-Dichloropropene	N/A	70 - 130	0 - 30	72 - 136	0 - 30
1,2,3-Trichlorobenzene	N/A	70 - 130	0 - 30	67 - 130	0 - 30
1,2,3-Trichloropropane	N/A	70 - 130	0 - 30	67 - 130	0 - 30
1,2,4-Trichlorobenzene	N/A	70 - 130	0 - 30	62 - 134	0 - 30
1,2,4-Trimethylbenzene	N/A	70 - 130	0 - 30	59 - 127	0 - 30
1,2-Dibromo-3-chloropropane	N/A	70 - 130	0 - 30	60 - 135	0 - 30
1,2-Dibromoethane	N/A	70 - 130	0 - 30	74 - 128	0 - 30
1,2-Dichlorobenzene	N/A	70 - 130	0 - 30	73 - 111	0 - 30
1,2-Dichloroethane	N/A	70 - 130	0 - 30	74 - 123	0 - 30
1,2-Dichloropropane	N/A	70 - 130	0 - 30	73 - 133	0 - 30
1,3,5-Trimethylbenzene	N/A	70 - 130	0 - 30	53 - 123	0 - 30
1,3-Dichlorobenzene	N/A	70 - 130	0 - 30	67 - 127	0 - 30
1,3-Dichloropropane	N/A	70 - 130	0 - 30	62 - 134	0 - 30
1,4-Dichlorobenzene	N/A	70 - 130	0 - 30	66 - 127	0 - 30
2,2-Dichloropropane	N/A	70 - 130	0 - 30	71 - 135	0 - 30
2-Butanone	N/A	70 - 130	0 - 30	70 - 130	0 - 30
2-Chlorotoluene	N/A	70 - 130	0 - 30	69 - 119	0 - 30
2-Hexanone	N/A	70 - 130	0 - 30	92 - 154	0 - 30
4-Chlorotoluene	N/A	70 - 130	0 - 30	65 - 117	0 - 30
4-Isopropyltoluene	N/A	70 - 130	0 - 30	62 - 127	0 - 30
4-Methyl-2-pentanone	N/A	70 - 130	0 - 30	83 - 138	0 - 30
Acetone	N/A	70 - 130	0 - 30	70 - 130	0 - 30
Benzene	N/A	85 - 126	0 - 30	74 - 133	0 - 29
Benzyl chloride	N/A	70 - 130	0 - 30	55 - 159	0 - 30

Analyte	Duplicate	MS	MSD	LCS	LCSD
Bromobenzene	N/A	70 - 130	0 - 30	71 - 119	0 - 30
Bromochloromethane	N/A	70 - 130	0 - 30	67 - 142	0 - 30
Bromodichloromethane	N/A	70 - 130	0 - 30	73 - 120	0 - 30
Bromoform	N/A	70 - 130	0 - 30	66 - 135	0 - 30
Carbon tetrachloride	N/A	70 - 130	0 - 30	69 - 133	0 - 30
Chlorobenzene	N/A	70 - 130	0 - 30	78 - 118	7.5 - 15
Chloroethane	N/A	70 - 130	0 - 30	62 - 125	0 - 30
Chloroform	N/A	70 - 130	0 - 30	71 - 118	0 - 30
cis-1,3-Dichloropropene	N/A	70 - 130	0 - 30	59 - 138	0 - 30
Dibromochloromethane	N/A	70 - 130	0 - 30	73 - 116	0 - 30
Dichlorodifluoromethane	N/A	70 - 130	0 - 30	49 - 88	0 - 30
Ethylbenzene	N/A	70 - 130	0 - 30	73 - 118	0 - 30
Hexachlorobutadiene	N/A	70 - 130	0 - 30	58 - 114	0 - 30
Isopropylbenzene	N/A	70 - 130	0 - 30	67 - 125	0 - 30
Methylene chloride	N/A	70 - 130	0 - 30	52 - 152	0 - 30
n-Butylbenzene	N/A	70 - 130	0 - 30	56 - 139	0 - 30
n-Propylbenzene	N/A	70 - 130	0 - 30	63 - 120	0 - 30
Naphthalene	N/A	70 - 130	0 - 30	53 - 137	0 - 30
o-Xylene	N/A	70 - 130	0 - 30	70 - 122	0 - 30
sec-Butylbenzene	N/A	70 - 130	0 - 30	64 - 126	0 - 30
Styrene	N/A	70 - 130	0 - 30	74 - 123	0 - 30
tert-Butylbenzene	N/A	70 - 130	0 - 30	67 - 116	0 - 30
Tetrachloroethene	N/A	70 - 130	0 - 30	70 - 130	0 - 30
Toluene	N/A	70 - 130	0 - 30	79 - 129	0 - 21
trans-1,3-Dichloropropene	N/A	N/A	0 - 30	60 - 129	0 - 30
Trichloroethene	N/A	70 - 130	0 - 30	69 - 127	0 - 26
Trichlorofluoromethane	N/A	70 - 130	0 - 30	54 - 111	0 - 30
Vinylchloride	N/A	70 - 130	0 - 30	64 - 111	0 - 30

Surrogates	Lower	Upper
dichlorodifluoromethane	66	117
toluene-d8	73	122
bromofluorobenzene	73	129

TABLE E11

PRECISION AND ACCURACY

VOLATILES: NON-AQUEOUS FOR SW846 Method 8260B

Analyte	Duplicate	MS	MSD	LCS	LCSD
1,1,1,2-Tetrachloroethane	N/A	70 - 130	0 - 30	79 - 121	N/A
1,1,1-Trichloroethane	N/A	70 - 130	0 - 30	73 - 127	N/A
1,1,2,2-Tetrachloroethane	N/A	70 - 130	0 - 30	65 - 134	N/A
1,1,2-Trichloroethane	N/A	70 - 130	0 - 30	71 - 124	N/A
1,1-Dichloroethane	N/A	70 - 130	0 - 30	70 - 131	N/A
1,1-Dichloroethene	N/A	82 - 136	0 - 30	70 - 144	0 - 25
1,1-Dichloropropene	N/A	70 - 130	0 - 30	72 - 136	N/A
1,2,3-Trichlorobenzene	N/A	70 - 130	0 - 30	67 - 130	N/A
1,2,3-Trichloropropane	N/A	70 - 130	0 - 30	67 - 130	N/A
1,2,4-Trichlorobenzene	N/A	70 - 130	0 - 30	62 - 134	N/A
1,2,4-Trimethylbenzene	N/A	70 - 130	0 - 30	59 - 127	N/A
1,2-Dibromo-3-chloropropane	N/A	70 - 130	0 - 30	60 - 135	N/A
1,2-Dibromoethane	N/A	70 - 130	0 - 30	74 - 128	N/A
1,2-Dichlorobenzene	N/A	70 - 130	0 - 30	73 - 111	N/A
1,2-Dichloroethane	N/A	70 - 130	0 - 30	74 - 123	N/A
1,2-Dichloropropane	N/A	70 - 130	0 - 30	73 - 133	N/A
1,3,5-Trimethylbenzene	N/A	70 - 130	0 - 30	53 - 123	N/A
1,3-Dichlorobenzene	N/A	70 - 130	0 - 30	67 - 127	N/A
1,3-Dichloropropane	N/A	70 - 130	0 - 30	62 - 134	N/A
1,4-Dichlorobenzene	N/A	70 - 130	0 - 30	66 - 127	N/A
2,2-Dichloropropane	N/A	70 - 130	0 - 30	71 - 135	N/A
2-Butanone	N/A	70 - 130	0 - 30	70 - 130	N/A
2-Chlorotoluene	N/A	70 - 130	0 - 30	69 - 119	N/A
2-Hexanone	N/A	70 - 130	0 - 30	92 - 154	N/A
4-Chlorotoluene	N/A	70 - 130	0 - 30	65 - 117	N/A
4-Isopropyltoluene	N/A	70 - 130	0 - 30	62 - 127	N/A
4-Methyl-2-pentanone	N/A	70 - 130	0 - 30	83 - 138	N/A
Acetone	N/A	70 - 130	0 - 30	70 - 130	N/A
Benzene	N/A	85 - 126	0 - 30	74 - 133	0 - 21
Benzyl chloride	N/A	70 - 130	0 - 30	55 - 159	N/A

Analyte	Duplicate	MS	MSD	LCS	LCSD
Bromobenzene	N/A	70 - 130	0 - 30	71 - 119	N/A
Bromochloromethane	N/A	70 - 130	0 - 30	67 - 142	N/A
Bromodichloromethane	N/A	70 - 130	0 - 30	73 - 120	N/A
Bromoform	N/A	70 - 130	0 - 30	66 - 135	N/A
Carbon Tetrachloride	N/A	70 - 130	0 - 30	69 - 133	N/A
Chlorobenzene	N/A	70 - 115	0 - 30	78 - 118	0 - 15
Chloroethane	N/A	70 - 130	0 - 30	62 - 125	N/A
Chloroform	N/A	70 - 130	0 - 30	71 - 118	N/A
cis-1,3-Dichloropropene	N/A	70 - 130	0 - 30	59 - 138	N/A
Dibromochloromethane	N/A	70 - 130	0 - 30	73 - 116	N/A
Dichlorodifluoromethane	N/A	70 - 130	0 - 30	49 - 88	N/A
Ethylbenzene	N/A	70 - 130	0 - 30	73 - 118	0 - 30
Hexachlorobutadiene	N/A	70 - 130	0 - 30	58 - 114	N/A
Isopropylbenzene	N/A	70 - 130	0 - 30	67 - 125	N/A
m,p-Xylenes	N/A	70 - 130	0 - 30	74 - 122	N/A
Methylene chloride	N/A	70 - 130	0 - 30	52 - 152	N/A
n-Butylbenzene	N/A	70 - 130	0 - 30	56 - 139	N/A
n-Propylbenzene	N/A	70 - 130	0 - 30	63 - 120	N/A
Naphthalene	N/A	70 - 130	0 - 30	53 - 137	N/A
o-Xylene	N/A	70 - 130	0 - 30	70 - 122	N/A
sec-Butylbenzene	N/A	70 - 130	0 - 30	64 - 126	N/A
Styrene	N/A	70 - 130	0 - 30	74 - 123	N/A
tert-Butylbenzene	N/A	70 - 130	0 - 30	67 - 116	N/A
Tetrachloroethene	N/A	70 - 130	0 - 30	70 - 130	N/A
Toluene	N/A	73 - 117	0 - 30	79 - 129	0 - 15
trans-1,3-Dichloropropene	N/A	70 - 130	0 - 30	60 - 129	N/A
Trichloroethene	N/A	70 - 130	0 - 30	69 - 127	0 - 18
Trichlorofluoromethane	N/A	70 - 130	0 - 30	54 - 111	N/A
Vinyl chloride	N/A	70 - 130	0 - 30	64 - 111	N/A

Surrogates	Lower	Upper
Dibromofluoromethane	66	117
toluene-d8	73	122
bromofluorobenzene	73	129

TABLE E12

PRECISION AND ACCURACY
VOLATILES: NON- AQUEOUS FOR GASOLINE RANGE ORGANICS
SW846 Method 8051B

Analyte	MS	MSD	LCS	LCSD
TPH - Volatile Fraction	36.3 - 126.12	0 - 25	70 - 130	0 - 30
Bromofluorobenzene (surrogate)			54 - 126	
n-Proylbenzene (surrogate)			50 - 135	

TABLE E13

PRECISION AND ACCURACY
VOLATILES: AQUEOUS FOR GASOLINE RANGE ORGANICS
SW846 Method 8051B

Analyte	MS	MSD	LCS	LCSD
TPH - Volatile Fraction	43.61 - 143.99	0 - 25	70 - 130	0 - 25
n-Proylbenzene (surrogate)			60 - 127	

TABLE E14
PRECISION AND ACCURACY
METALS: NON-AQUEOUS FOR ICP
SW846 Method 6010B

Analyte	Duplicate	MS	MSD	LCS	LCSD
Aluminum	0 - 25.9	70 - 130	0 - 20	75.2 - 126.4	0 - 15.1
Antimony	0 - 23.5	70 - 130	0 - 28.4	75.2 - 199.1	0 - 24.4
Arsenic	0 - 22.3	71.5 - 114	0 - 16.3	84.6 - 132.5	0 - 10.3
Barium	0 - 21.4	65.7 - 127.3	0 - 23.2	89.7 - 154	0 - 16
Beryllium	0 - 23.6	74.4 - 118.5	0 - 16.5	76.9 - 128.4	0 - 10.1
Boron	0 - 20	70 - 130	0 - 20	33.3 - 74.7	0 - 20
Cadmium	0 - 14.3	76 - 117.7	0 - 10.3	77.5 - 115.7	0 - 8.89
Calcium	0 - 21.6	67.7 - 125.2	0 - 24.8	95.4 - 139	0 - 9.19
Chromium	0 - 21.1	74 - 122.2	0 - 19.3	73 - 150	0 - 22.8
Cobalt	0 - 19.9	70.9 - 122.8	0 - 12.4	88.1 - 142.3	0 - 10.5
Copper	0 - 23.3	73.3 - 124	0 - 17.1	81.3 - 149	0 - 16.8
Iron	0 - 19.2	70 - 130	0 - 20	68.9 - 243.2	0 - 40.6
Lead	0 - 20.1	70.6 - 123.2	0 - 20.3	80.4 - 117.1	0 -9.47
Lithium	0 - 20	70 - 130	0 - 20	70 - 120	0 - 20
Magnesium	0 - 25.8	70 - 130	0 - 20	72.8 - 109	0 - 9.33
Manganese	0 - 23.3	70 - 130	0 - 20	83.8 - 151.6	0 - 22.2
Molybdenum	0 - 20	70 - 130	0 - 20	92.2 - 140.7	0 - 20
Nickel	0 - 24.5	74.2 - 121.1	0 14.3	85.3 - 148.6	0 - 19.96
Phosphorus	0 - 21.8	70 - 130	0 - 20	70 - 130	0 - 20
Potassium	0 - 20	65.3 - 130.2	0 - 25.6	71.5 - 163.9	0 - 21.9
Selenium	0 - 22.4	67.4 - 112.6	0 - 17	86.6 - 122.1	0 - 8.86
Silica	0 - 20	70 - 130	0 - 20	70 - 130	0 - 20
Silicon	0 - 20	70 - 130	0 - 20	70 - 130	0 - 20
Silver	0 - 18.5	75.9 - 124.2	0 - 14.7	93.2 - 130.2	0 - 8.65
Sodium	0 - 22.7	86.3 - 122.6	0 - 11.1	70 - 130	0 - 12.4
Strontium	0 - 20	70 - 130	0 - 20	85.9 - 124.8	0 - 20
Sulfur	0 - 20	70 - 130	0 - 20	70 - 130	0 - 20
Thallium	0 - 17.9	70.7 - 117.4	0 - 10	57.8 - 122.8	0 - 10.3
Tin	0 - 20	70 - 130	0 - 20	70 - 130	0 - 20
Titanium	0 - 20	70 - 130	0 - 20	27.6 - 249.9	0 - 20
Uranium	0 - 20	70 - 130	0 - 20	5.5 - 26.4	0 - 20
Vanadium	0 - 19.3	66.8 - 129.6	0 - 20.6	78.3 - 124	0 - 10.6
Zinc	0 - 21.2	70.2 - 124.3	0 - 25.3	78.1 - 115	0 - 14.8

TABLE E15
PRECISION AND ACCURACY
METALS: AQUEOUS FOR ICP
SW846 Method 6010B and EPA 200.7

Analyte	Duplicate	MS	MSD	LCS	LCSD
Aluminum	0 - 16.3	87.7 - 119.2	0 - 10.9	89.5 - 115.6	0 - 20
Antimony	0 - 5.2	75.5 - 123.5	0 - 7.4	89.8 - 111.4	0 - 20
Arsenic	0 - 6.1	84.8 - 118.2	0 - 7.2	89.5 - 111.6	0 - 20
Barium	0 - 8.1	90.3 - 112.6	0 - 6.4	90.7 - 110.8	0 - 20
Beryllium	0 - 11.7	88 - 116	0 - 6.5	90.2 - 113	0 - 20
Boron	0 - 20	70 - 130	0 - 20	80 - 120	0 - 20
Cadmium	0 - 6.9	89.4 - 115.9	0 - 6.8	90.7 - 114.6	0 - 20
Calcium	0 - 8.5	83.2 - 125.1	0 - 10.7	89.7 - 115.4	0 - 20
Chromium	0 - 15.3	88.3 - 112.2	0 - 7.4	90 - 11.7	0 - 20
Cobalt	0 - 6.5	88.5 - 115.3	0 - 6.1	91.1 - 113.4	0 - 20
Copper	0 - 13.2	87.7 - 117	0 - 6.3	88.2 - 114.6	0 - 20
Iron	0 - 20.4	90.7 - 117.3	0 - 11.6	91.8 - 113.4	0 - 20
Lead	0 - 14.1	84.6 - 117.6	0 - 7	89.3 - 113.6	0 - 20
Lithium	0 - 20	70 - 130	0 - 20	80 - 120	0 - 20
Magnesium	0 - 17.7	60.7 - 138.1	0 - 14.3	86.6 - 112.8	0 - 20
Manganese	0 - 19.1	84.5 - 115.2	0 - 8.9	87.9 - 111.5	0 - 20
Molybdenum	0 - 20	70 - 130	0 - 20	80 - 120	0 - 20
Nickel	0 - 10.7	89.9 - 117.5	0 - 6.4	91.3 - 115.3	0 - 20
Phosphorus		70 - 130	0 - 12.66	80 - 120	0 - 20
Potassium	0 - 20	80.7 - 118.1	0 - 9	85.3 - 114.2	0 - 20
Selenium	0 - 10.6	84.3 - 112.3	0 - 6.7	87.2 - 109.4	0 - 20
Silica	0 - 20	70 - 130	0 - 20	80 - 120	0 - 20
Silicon	0 - 20	70 - 130	0 - 20	80 - 120	0 - 20
Silver	0 - 17.1	89.3 - 119.8	0 - 7.3	90.9 - 116.1	0 - 20
Sodium	0 - 11.6	74.9 - 130.3	0 - 19.6	91.5 - 117.7	0 - 20
Strontium	0 - 20	70 - 130	0 - 20	80 - 120	0 - 20
Sulfur	0 - 20	70 - 130	0 - 20	80 - 120	0 - 20
Thallium	0 - 5	87.2 - 115.3	0 - 7.9	90.4 - 112.9	0 - 20
Tin	0 - 20	70 - 130	0 - 20	80 - 120	0 - 20
Titanium	0 - 20	70 - 130	0 - 20	80 - 120	0 - 20
Uranium	0 - 20	70 - 130	0 - 20	80 - 120	0 - 20
Vanadium	0 - 20	89.4 - 114.3	0 - 6.6	89.8 - 113	0 - 20
Zinc	0 - 20	91.4 - 113.5	0 - 7.6	92.4 - 112.9	0 - 20

TABLE E16
PRECISION AND ACCURACY
METALS: NON-AQUEOUS FOR ICP-MASS SPEC
SW846 Method 6020

Analyte	Duplicate	MS	MSD	LCS	LCSD
Aluminum	N/A	70 - 130	0 - 20	63.1 - 136.9	0 - 30
Antimony	N/A	70 - 130	0 - 20	66.9 - 126.3	0 - 30
Arsenic	N/A	51.1 - 122.6	0 - 13.2	75.7 - 118.6	0 - 30
Barium	N/A	71 - 128.6	0 - 23.6	70 - 130	0 - 30
Beryllium	N/A	70 - 130	0 - 14.2	75.2 - 121.2	0 - 30
Boron	N/A	70 - 130	0 - 20	70 - 130	0 - 30
Cadmium	N/A	79.6 - 117.4	0 - 13.3	80.6 - 110.8	0 - 30
Calcium	N/A	70 - 130	0 - 20	92.1 - 139.4	0 - 30
Chromium	N/A	63.9 - 125.7	0 - 20.8	61.1 - 143.2	0 - 30
Cobalt	N/A	70 - 130	0 - 20	86.5 - 120.1	0 - 30
Copper	N/A	70 - 130	0 - 22.9	84.3 - 128.6	0 - 30
Iron	N/A	70 - 130	0 - 20	23.3 - 192.5	0 - 30
Lead	N/A	67.3 - 122.4	0 - 23.2	70 - 130	0 - 30
Lithium	N/A	70 - 130	0 - 20	40.9 - 189	0 - 30
Magnesium	N/A	70 - 130	0 - 20	58.9 - 121.7	0 - 30
Manganese	N/A	70 - 130	0 - 20	60.2 - 138.6	0 - 30
Molybdenum	N/A	70 - 130	0 - 20	95.4 - 134.6	0 - 30
Nickel	N/A	70 - 130	0 - 20	78.1 - 125.7	0 - 30
Phosphorus	N/A	70 - 130	0 - 20	70 - 130	0 - 30
Potassium	N/A	70 - 130	0 - 20	25.6 - 201	0 - 30
Selenium	N/A	58.7 - 114.8	0 - 16.2	60.4 - 129.5	0 - 30
Silica	N/A	70 - 130	0 - 20	70 - 130	0 - 30
Silicon	N/A	70 - 130	0 - 20	70 - 130	0 - 30
Silver	N/A	83.8 - 121.6	0 - 11.8	12.5 - 141.6	0 - 30
Sodium	N/A	70 - 130	0 - 20	14.5 - 191	0 - 30
Strontium	N/A	70 - 130	0 - 20	40.9 - 172.1	0 - 30
Sulfur	N/A	70 - 130	0 - 20	70 - 130	0 - 30
Thallium	N/A	70 - 130	0 - 20	28.5 - 157.9	0 - 30
Tin	N/A	70 - 130	0 - 20	70 - 130	0 - 30
Titanium	N/A	70 - 130	0 - 20	70 - 130	0 - 30
Uranium	N/A	70 - 130	0 - 20	70 - 130	0 - 30
Vanadium	N/A	70 - 130	0 - 20	63.9 - 136.2	0 - 30
Zinc	N/A	70 - 130	0 - 20	74.8 - 103.5	0 - 30

TABLE E17
PRECISION AND ACCURACY
METALS: AQUEOUS FOR ICP-MASS SPEC
SW846 Method 6020 and EPA 200.8

Analyte	Duplicate	MS	MSD	LCS	LCSD
Aluminum	N/A	73.1 - 133	0 - 20	73.1 - 133	0 - 20
Antimony	N/A	87.7 - 115.2	0 - 6.9	69.9 - 126.3	0 - 20
Arsenic	N/A	84.9 - 120.6	0 - 13.9	78.2 - 118.6	0 - 20
Barium	N/A	88.4 - 113.2	0 - 13.5	90.7 - 110.9	0 - 20
Beryllium	N/A	82.6 - 119.8	0 - 10.3	74.5 - 120.6	0 - 20
Boron	N/A	70 - 130	0 - 20	70 - 120	0 - 20
Cadmium	N/A	86.9 - 115.3	0 - 13	87.1 - 110.4	0 - 20
Calcium	N/A	91.7 - 142.5	0 - 20	91.7 - 142.5	0 - 20
Chromium	N/A	83.1 - 120.6	0 - 13.3	74.8 - 120.4	0 - 20
Cobalt	N/A	80.1 - 123.3	0 - 9.5	81.6 - 114	0 - 20
Copper	N/A	80.9 - 126.4	0 - 9	82.1 - 128.3	0 - 20
Iron	N/A	62.8 - 157.6	0 - 20	62.8 - 157.6	0 - 20
Lead	N/A	84.8 - 116.4	0 - 11.2	80 - 120	0 - 20
Lithium	N/A	47.5 - 141.4	0 - 20	47.5 -141.4	0 - 20
Magnesium	N/A	83.2 - 119.6	0 - 20	83.2 - 119.6	0 - 20
Manganese	N/A	79.5 - 117.7	0 - 20	79.5 - 117.7	0 - 20
Molybdenum	N/A	89 - 110.5	0 - 20	89 - 110.5	0 - 20
Nickel	N/A	73.6 - 127.5	0 - 10.1	85.9 - 118	0 - 20
Phosphorus	N/A	70 - 130	0 - 20	80 - 120	0 - 20
Potassium	N/A	71 - 125.4	0 - 20	71 - 125.4	0 - 20
Selenium	N/A	81.9 - 117.9	0 - 13.4	66.4 - 129.5	0 - 20
Silica	N/A	70 - 130	0 - 20	80 - 120	0 - 20
Silicon	N/A	70 - 130	0 - 20	80 - 120	0 - 20
Silver	N/A	80.7 - 126.3	0 - 8.9	46.5 - 157.6	0 - 20
Sodium	N/A	70 - 130	0 - 20	80 - 120	0 - 20
Strontium	N/A	94.6 - 110.3	0 - 20	94.6 - 110.3	0 - 20
Sulfur	N/A	70 - 130	0 - 20	80 - 120	0 - 20
Thallium	N/A	84.7 - 117.3	0 - 7.1	28.5 - 157.9	0 - 20
Tin	N/A	56.1 - 150.1	0 - 20	56.1 - 150.1	0 - 20
Titanium	N/A	70 - 130	0 - 20	80 - 120	0 - 20
Uranium	N/A	70 - 130	0 - 20	80 - 120	0 - 20
Vanadium	N/A	83.2 - 124.7	0 - 9	79 - 116.3	0 - 20
Zinc	N/A	74.2 - 128.7	0 - 11.5	62.2 - 150.8	0 - 20
Ziconium	N/A	70 - 130	0 - 20	80 - 120	N/A

TABLE E18
PRECISION AND ACCURACY
METALS: NON-AQUEOUS FOR GRAPHTE FURNACE AND MERCURY
SW846 Method 7000 Series

Analyte	Duplicate	MS	MSD	LCS	LCSD
Antimony	N/A	70 - 130	0 - 30	24.6 - 176	0 - 30.19
Arsenic	N/A	70 - 130	0 - 30	70.2 - 129.8	0 - 11.61
Lead	N/A	70 - 130	0 - 30	69 - 130	0 - 11.52
Selenium	N/A	70 - 130	0 - 30	70 - 130	0 - 11.05
Thallium	N/A	70 - 130	0 - 30	47.46 - 152	0 - 12.22
Mercury	N/A	74.3 - 131.7	0 - 16.3	57.9 - 134.3	0 - 15.6

TABLE E19
PRECISION AND ACCURACY
METALS: AQUEOUS FOR GRAPHITE FURNACE AND MERCURY
SW846 Method 7000 Series and EPA 200 Series

Analyte	Duplicate	MS	MSD	LCS	LCSD
Antimony	0 - 20	70 - 130	0 - 20	70 - 120	0 - 20
Arsenic	0 - 20	60.5 - 123.1	0 - 20	77.2 - 120.8	0 - 20
Lead	0 - 20	62.7 - 130.3	0 - 20	75.6 - 118.5	0 - 20
Selenium	0 - 20	58.4 - 127.2	0 - 20	78 - 117.5	0 - 20
Thallium	0 - 20	70 - 130	0 - 20	70 - 120	0 - 20
Zinc	0 - 20	70 - 130	0 - 20	70 - 120	0 - 20
Mercury	N/A	74.3 - 131.7	0 - 16.3	81.5 - 124.3	0 - 16.3

TABLE E20

PRECISION AND ACCURACY

GENERAL CHEMISTRY: NON-AQUEOUS

Analyte	Duplicate	MS	MSD	LCS	LCSD
Ash, Percent (@ 550C)	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Ash, Percent (@ 750C)	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Biochemical Oxygen Demand - 5 day (soluble)	0 - 34	84.6 - 115.4	0 - 30	84.6 - 115.4	0 - 30
Bromide	0 - 30	70 - 130	0 - 30	80 - 120	0 - 30
Bromine	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Cation Exchange Capacity	0 - 30	N/A - N/A	N/A - N/A	70 - 130	0 - 30
Chemical Oxygen Demand (soluble)	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Chlorides	0 - 30	70 - 130	0 - 30	80 - 120	0 - 30
Chlorine	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Chromium, Hexavalent	0 - 30	70 - 130	0 - 30	76 - 122	0 - 30
Color	0 - 30	N/A - N/A	N/A - N/A	70 - 130	0 - 30
Corrosivity (pH<2 or >12)	0 - 5	N/A - N/A	N/A - N/A	95 - 105	0 - 20
Cyanide Amenable to Chlorination	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
CN-CLP	0 - 30	70 - 130	0 - 30	60 - 125	0 - 30
Cyanide, Releasable	0 - 30	N/A - N/A	N/A - N/A	0.01 - 10.6	0 - 30
Cyanide, Total	0 - 30	70 - 130	0 - 30	60 - 125	0 - 30
Cyanide, Weak Acid Dissociable	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Density of Material	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Evaporative Loss @ 105C	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Extractable Organic Halides	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Fecal Coliform - 5 tube dilution (MPN)	0 - 200	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Fecal Coliform - Membrane Filter	0 - 200	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Flashpoint, Closed Cup (>140 F)	0 - 20	N/A - N/A	N/A - N/A	90 - 110	0 - 10
Flashpoint, Closed Cup (>200 F)	0 - 20	N/A - N/A	N/A - N/A	90 - 110	0 - 10
Flashpoint, Setaflash (>140 F)	0 - 20	N/A - N/A	N/A - N/A	90 - 110	0 - 10
Flashpoint, Setaflash (>200 F)	0 - 20	N/A - N/A	N/A - N/A	90 - 110	0 - 10
Fluoride	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Fluorine	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Formaldehyde, Total	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Hardness as CaCO3 by Titration	0 - 30	70 - 130	0 - 30	80 - 120	0 - 30
Heating Value (Btu/lb)	0 - 30	N/A - N/A	N/A - N/A	80 - 120	0 - 30
Hydroxide Alkalinity as CaCO3	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A

Analyte	Duplicate	MS	MSD	LCS	LCSD
Inhibitory Residue on Glassware	Same Result	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Iodide	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Iodine	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Moisture by Karl Fischer	0 - 30	N/A - N/A	N/A - N/A	95 - 105	0 - 10
Nitrogen, Ammonia	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Nitrogen, Nitrate	0 - 33	67 - 125	0 - 30	80 - 120	0 - 30
Nitrogen, Nitrate/Nitrite	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Nitrogen, Nitrite	0 - 30	70 - 130	0 - 30	80 - 120	0 - 30
Nitrogen, Total Kjeldahl	0 - 19	70 - 130	0 - 30	70 - 130	0 - 30
Nitrogen, Total Organic	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Nitrogen, Total	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Oil & Grease	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
pH	0 - 5	N/A - N/A		70 - 130	0 - 30
Phenols, Total	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Phosphorus, Ortho Spec	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Phosphorus, Total (as PO4)	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Solids, Volatile	0 - 20	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Specific Gravity	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Sulfates	0 - 30	70 - 130	0 - 30	80 - 120	0 - 30
Sulfides, Releasable	0 - 30	70 - 130	0 - 30	0 - 55	0 - 30
Sulfides by 9030	0 - 30	60 - 140	0 - 30	60 - 140	0 - 30
Sulfur	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Surfactants (Methylene Blue Active Sub.)	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Total Alkalinity as CaCO3	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Total Carbon	0 - 29	73 - 129	0 - 30	88 - 130	0 - 30
Total Coliform - 5 tube dilution (MPN)	0 - 200	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Total Coliform - Membrane Filter	0 - 200	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Total Halogens	0 - 30	N/A - N/A	N/A - N/A	70 - 130	0 - 30
Total Inorganic Carbon	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Total Organic Carbon	0 - 29	73 - 129	0 - 30	88 - 130	0 - 20
Total Petroleum Hydrocarbons by IR	0 - 27	70 - 130	0 - 30	70 - 130	0 - 30
Total Petroleum Hydrocarbons	0 - 30	70 - 130	0 - 30	70 - 116	0 - 30

TABLE E21
PRECISION AND ACCURACY
GENERAL CHEMISTRY: AQUEOUS

Analyte	Duplicate	MS	MSD	LCS	LCSD
Acidity	0 - 20	N/A - N/A		N/A - N/A	N/A - N/A
Ash, Percent (@ 550C)	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Ash, Percent (@ 750C)	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Bicarbonate Alkalinity as CaCO3	0 - 11	80 - 120	0 - 30	90 - 110	0 - 20
Biochemical Oxygen Demand - 5 day (soluble)	0 - 34	N/A - N/A	N/A - N/A	84.6 - 115.4	0 - 30
Boiling Point	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Bromide	0 - 20	80 - 120	0 - 30	90 - 110	0 - 20
Bromine	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Carbonate Alkalinity as CaCO3	0 - 11	80 - 120	0 - 30	90 - 110	0 - 20
Chemical Oxygen Demand (soluble)	0 - 25	70 - 30	0 - 30	82.8 - 111	0 - 20
Chlorides	0 - 13.1	80 - 120	0 - 30	90 - 110	0 - 20
Chlorine, Residual	0 - 13	70 - 130	0 - 30	88.3 - 119	0 - 20
Chlorphyll A	0 - 20	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Chromium, Hexavalent	0 - 13	85 - 115	0 - 30	83.8 - 116	0 - 20
Color	0 - 20	N/A - N/A	N/A - N/A	80 - 20	0 - 20
Corrosivity (pH<2 or >12)	0 - 5	N/A - N/A	N/A - N/A	95 - 105	0 - 20
Corrosivity of SAE Steel	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Cyanide Amenable to Chlorination	0 - 20	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
CN-CLP	0 - 20	75 - 125	0 - 30	80 - 120	0 - 20
Cyanide, Free	0 - 20	75 - 125	0 - 30	70 - 130	0 - 20
Cyanide, Releasable	0 - 20	75 - 125	0 - 30	0.01 - 10.6	0 - 20
Cyanide, Total	0 - 20	75 - 125	0 - 30	75 - 132	0 - 20
Cyanide, Weak Acid Dissociable	0 - 20	75 - 125	0 - 30	70 - 130	0 - 20
Density of Material	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Dissolved Organic Carbon	0 - 25	77 - 127	0 - 30	85 - 115	0 - 30
Dissolved Oxygen	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Fecal Coliform - 5 tube dilution (MPN)	0 - 200	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Fecal Coliform - Membrane Filter	0 - 200	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Flashpoint, Closed Cup (>140 F)	0 - 20	N/A - N/A	N/A - N/A	90 - 110	0 - 10
Flashpoint, Closed Cup (>200 F)	0 - 20	N/A - N/A	N/A - N/A	90 - 110	0 - 10
Flashpoint, Setaflash (>140 F)	0 - 20	N/A - N/A	N/A - N/A	90 - 110	0 - 10
Flashpoint, Setaflash (>200 F)	0 - 20	N/A - N/A	N/A - N/A	90 - 110	0 - 10

Analyte	Duplicate	MS	MSD	LCS	LCSD
Fluoride by EPA 340.2	0 - 20	86 - 126	0 - 30	80 - 120	0 - 20
Fluoride by IC	0 - 14.8	80 - 120	0 - 30	90 - 110	0 - 20
Fluorine	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Formaldehyde, Total	0 - 18	70 - 130	0 - 30	83.8 - 113	0 - 20
Hardness as CaCO3 by Titration	0 - 17	89 - 125	0 - 30	87.4 - 114	0 - 20
Heating Value (Btu/lb)	0 - 20	N/A - N/A	N/A - N/A	80 - 120	0 - 20
Hydrazine	0 - 20	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Hydroxide Alkalinity as CaCO3	0 - 11	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Inhibitory Residue on Glassware	Same Result	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Iodide	0 - 20	70 - 130	0 - 30	70 - 130	0 - 20
Iodine	0 - 20	70 - 130	0 - 30	80 - 120	0 - 20
Moisture by Karl Fischer	0 - 30	N/A - N/A	N/A - N/A	95 - 105	0 - 10
Nitrogen, Ammonia	0 - 20	65 - 121	0 - 30	63 - 121	0 - 20
Nitrogen, Nitrate	0 - 12.5	80 - 120	0 - 30	90 - 110	0 - 20
Nitrogen, Nitrate/Nitrite	0 - 20	70 - 130	0 - 30	79 - 122	0 - 20
Nitrogen, Nitrite	0 - 16.9	80 - 120	0 - 30	90 - 110	0 - 20
Nitrogen, Total Kjeldahl	0 - 20	70 - 130	0 - 30	67 - 119	0 - 20
Nitrogen, Total Organic	0 - 20	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Nitrogen, Total	0 - 20	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Oil & Grease	0 - 30	70 - 130	0 - 30	79.6 - 114	0 - 30
pH	0 - 5	N/A - N/A	N/A - N/A	95 - 105	0 - 10
Phenols, Total	0 - 20	70 - 130	0 - 30	72 - 120	0 - 20
Phosphorus, Ortho	0 - 20	80 - 120	0 - 30	90 - 110	0 - 20
Phosphorus, Ortho Spec	0 - 16	70 - 130	0 - 30	85.5 - 121	0 - 20
Phosphorus, Total (as PO4)	0 - 20	70 - 130	0 - 30	66 - 125	0 - 20
Salinity	0 - 20	N/A - N/A	N/A - N/A	80 - 120	0 - 20
Solids, Settleable (waste matrices)	0 - 20	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Solids, Total	0 - 20	N/A - N/A	N/A - N/A	80 - 120	0 - 20
Solids Total Dissolved	0 - 20	N/A - N/A	N/A - N/A	84 - 115	0 - 20
Solids, Total Suspended (waste matrices)	0 - 20	N/A - N/A	N/A - N/A	95 - 105	0 - 20
Solids, Volatile	0 - 20	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Solids, Volatile Suspended (waste matrices)	0 - 20	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Specific Conductance of water-soluble ions	0 - 20	N/A - N/A	N/A - N/A	80 - 120	0 - 20
Specific Gravity	0 - 30	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A

Analyte	Duplicate	MS	MSD	LCS	LCSD
Sulfates	0 - 14.5	80 - 120	0 - 30	90 - 110	0 - 20
Sulfides, Releasable	0 - 30	70 - 130	0 - 30	0 - 55	0 - 30
Sulfides by 376.2	0 - 14.5	80 - 120	0 - 30	90 - 110	0 - 20
Sulfides by 9030A	0 - 30	60 - 140	0 - 30	60 - 140	0 - 30
Sulfite	0 - 20	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Sulfur	0 - 30	70 - 130	0 - 30	70 - 130	0 - 30
Surfactants (Methylene Blue Active Sub.)	0 - 20	70 - 130	0 - 30	80 - 120	0 - 30
Total Alkalinity as CaCO3	0 - 11	80 - 120	0 - 30	90 - 110	0 - 20
Total Carbon	0 - 25	77 - 127	0 - 30	85 - 115	0 - 20
Total Coliform - 5 tube dilution (MPN)	0 - 200	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Total Coliform - Membrane Filter	0 - 200	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Total Halogens	0 - 20	N/A - N/A	N/A - N/A	70 - 130	0 - 20
Total Inorganic Carbon	0 - 20	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Total Organic Carbon	0 - 25	77 - 127	0 - 30	85 - 115	0 - 20
Total Organic Halides	0 - 26	70.8 - 127	0 - 30	63 - 121	0 - 20
Total Petroleum Hydrocarbons (client matrix = sludge)	0 - 30	N/A - N/A	N/A - N/A	64.3 - 121	0 - 30
Total Petroleum Hydrocarbons by IR	0 - 30	N/A - N/A	N/A - N/A	70 - 130	0 - 20
Toxicity of Lab Water	0 - 200	N/A - N/A	N/A - N/A	N/A - N/A	N/A - N/A
Turbidity	0 - 18	N/A - N/A	N/A - N/A	82.5 - 112	0 - 20
Viscosity, Absolute (cPs)	0 - 20	N/A - N/A	N/A - N/A	80 - 120	0 - 20
Viscosity, Kinematic (cSt)	0 - 20	N/A - N/A	N/A - N/A	80 - 120	0 - 20
Viscosity, S.U.S.	0 - 20	N/A - N/A	N/A - N/A	80 - 120	0 - 20

APPENDIX F
ESSENTIAL QUALITY CONTROL REQUIREMENTS

Appendix F - Essential Quality Control Requirements

GEL enforces strict adherence to quality control measures. Quality control measures for each type of analysis are delineated in the associated standard operating procedure and include those specified in the identified analytical method. Client requests for additional quality control and agreed to by GEL will be communicated to the laboratory by the project manager and performed accordingly.

All quality control measures are assessed and evaluated on an on-going basis and used to establish statistically-derived quality control acceptance criteria. The acceptance criteria are used to evaluate if the analytical process is in control and assist in establishing the validity of the data. Procedures for handling out of control situations can be found in the analytical standard operating procedure.

As stated above, specific method quality measures can be found the appropriate standard operating procedure. General essential quality control requirements are summarized in the sections below for chemical testing including both inorganic and organic analyses, microbiological analyses, and radiochemical testing.

Section F1: Chemical Testing

The quality control requirements in this section include those for both inorganic and organic analyses. Discussed in this section are:

- Negative controls
- Positive controls
- Analytical variability and reproducibility
- Method evaluation
- Method detection limits
- Data reduction
- Quality of standards and reagents
- Selectivity
- Constant and consistent test condition

F1.1 Negative controls

A negative control is to be implemented at least once per analytical batch consisting of samples of the same matrix and where, if applicable, the same extraction or preparation method is employed.

The negative control is a method blank and is used to determine the presence of contamination.

The source of contamination must be investigated and measures taken to correct, minimize or eliminate its source under two conditions.

- If the concentration of target analyte exceeds the established practical quantitation limit and exceeds a concentration greater than 1/10 of the measured concentration of any sample in the associated analytical batch

- If the concentration of a target analyte in the method blank exceeds that present in the samples and is greater than 1/10 of the specified regulatory limit.

If analysis of the method blank is indicative of contamination, each sample in the affected batch must be assessed against the above criteria to determine if its data is acceptable. Any sample associated with a contaminated method blank shall be reprocessed for analysis or the results reported with appropriate data qualifying codes.

F1.2 Positive Controls

Positive control consist of laboratory control samples, matrix spikes, and surrogates.

F1.2.1 Laboratory control sample (LCS)

A LCS is analyzed at a minimum of one per analytical batch consisting of twenty or less samples per matrix type and, where applicable, sample extraction or preparation method. The recoveries of target analytes in the LCS are used to determine batch acceptance.

This requirement is not applied if spiking solutions are not either not available or inappropriate for the determination of specific analytes such as total volatile solids, pH, color, odor, temperature, or dissolved oxygen or turbidity. The applicability of a LCS to a particular analysis is included in the associated analytical standard operating procedure.

F1.2.2 Matrix spikes (MS)

A MS is analyzed at least once for every twenty samples per matrix type or sample extraction or preparation method.

The samples selected for use as matrix spikes are rotated among samples from different clients so that various matrix problems may be noted and/or addressed.

Recoveries for a matrix spike that do not fall within the acceptance criteria may indicate a problem with the sample composition, This information is typically communicated to the client whose sample was used for the spike.

This requirement is not applied if spiking solutions are not either not available or inappropriate for the determination of specific analytes such as total volatile solids, pH,

color, odor, temperature, or dissolved oxygen or turbidity. As discussed above, this information can be found in the applicable standard operating procedure.

If the test method does not specify the spiking compounds that are to be used in either the LCS or MS, we spike both with all reportable components. A representative number (at least 10%) of the method listed components may be used to evaluate the control of the process under the following conditions:

- If the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs by EPA 608)
- The test method has an extremely long list of components or components are incompatible.

The selected components of each spiking mix shall represent all chemistries, elution patterns and masses and will include permit specified analytes and other client requested components. All reported components are used in the spike mixture within a two-year time period with no one component or components dominate the spike mixture.

F1.2.3 Surrogates

Surrogates are applicable only to organic analyses. Surrogate compounds are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The number of surrogates used for the determination of the different classes of analytes is listed in the table below.

Analyte suite	Number of surrogates
Base Neutrals	3
Acids	3
Pesticides	2
Herbicides	2
PCB	2
Volatiles	3
TPH-volatile	1
TPH-nonvolatile	1

Poor surrogate recovery may indicate a problem with the sample's composition and is reported to the client. If sufficient aliquots are provided by the client, samples with surrogate recoveries that do not fall within the acceptance criteria are extracted again and/or Reanalyzed to confirm the affects of matrix interference on these recoveries.

F1.3 Analytical Variability/Reproducibility

Matrix spike duplicates (MSDs) or laboratory duplicates are analyzed a minimum of once per twenty samples for each matrix type and/or sample extraction or preparation method. We analyze either a matrix spike duplicate or sample duplicate based on guidance provided by the analytical method and incorporated into the corresponding standard operating procedure.

Sample(s) analyzed in duplicate are rotated among client samples so that various matrix problems may be noted and/or addressed.

If the relative percent difference (RPD) between the sample and it duplicate exceed the quality control criteria, the analyst proceeds as describes in the appropriate analytic standard operating procedure.

For analytical procedures that do not require either a matrix spike or LCS, the RPD is used to evaluate batch acceptability. If the RPD does not fall within the current acceptance limits for such parameters, the batch is reanalyzed.

Poor performance in matrix duplicates or matrix spike duplicates may indicate a problem with the sample matrix and shall be reported to the client whose sample was used for the duplicate.

F1.4 Method Evaluation

The following procedures, as described in the other sections of the QAP, are in place in order to ensure the accuracy of the reported result:

- Procedure for initial demonstration of analytical capability performed initially (prior to the analysis of any samples) and if there is a significant change in instrument type, personnel, matrix or test method. Refer to Section 8.

- Procedures for initial and continuing calibration protocols as specified in Section 7.

- Procedures for utilizing proficiency test samples to evaluate the ability of a procedure and/or analyst laboratory to produce accurate data as specified in Section 3.

F1.5 Method Detection Limits

Method detection limits (MDLs) are determined as descried in GL-LB-E-001 for the Determination of Method Detection Limits. This procedure is based on that established in 40 CFR Part 136, Appendix B.

As specified in this standard operating procedure, method detection limits are determined at a minimum of once per year and prior to the implementation of new methodology. MDL studies are performed twice per year for the determination of metals by EPA 6020, EPA 6010B, EPA 7470, and EPA 7471A.

Where possible, MDL studies are conducted for both aqueous and solid matrices using a clean matrix appropriate to the test method (such as laboratory pure reagent water or Ottawa sand.)

MDL studies for the majority of routine parameters are conducted by:

- analyzing seven replicates of the lowest calibration standard

- determining the standard deviation of the seven replicates
- multiplying the standard deviation by 3.143 (based on six degrees of freedom and representing a 99% confidence level) to obtain the calculated MDL.

If the MDL study is being conducted for a new method or target analyte, the following step are taken:

- the MDL is estimated based on information provided in the method or analytical experience
- a standard with a concentration three to five times the estimated MDL is prepared and analyzed seven times
- the MDL is calculated as above based on the standard deviation and degreases of freedom
- the MDL is evaluated for reasonableness by:

MDL studies are not performed for any target analyte for which spiking solutions are not available such total volatile solids, pH, color, odor, temperature dissolved oxygen or turbidity.

Practical quantitation limits (PQLs) are determined by either multiplying the MDL by 5 TO 10 or are equal to that of the lowest calibration standard. Concentrations of a target analyte determined to be greater that its PQL are defined as quantitative results. All quantitative reported results are bracketed by calibration or calibration verification standards.

All MDL studies conducted by the laboratory are submitted to the Quality team for an independent review. Upon acceptance of the MDL study, the MDLs reported to clients via our computer system are updated unless otherwise specified by contract. PQLs are also updated as directed by the new MDLS or changes to procedures.

All data pertaining to the study and the calculation of the MD(s) is stored on compact discs after being scanned using ADOBE software. The compact discs are maintained as quality records in the Quality department.

F1.6 Data Reduction

The procedures for data reduction, such as use of linear regression, are documented in the individual analytical standard operating procedures. GEL's policy governing the manual integration of chromatographic data is detailed in GL-LB-E-017 for Procedure and Policy for Manual Integration. Understanding of the procedures used for data reduction is an important part of an analyst demonstrating proficiency in an analytical procedure. All analysts who may potentially perform manual integrations of chromatographic data are also trained to GL-LB-E-017.

Manual integrations of chromatographic peaks can only be performed in accordance with this GL-LB-E-017. This ensures that the integrations are done in a consistent and technically justifiable manner while meeting the requirements set forth under the Good Automated Laboratory Practices.

F1.7 Quality of Standards and Reagents

The quality of standards used in instrument calibration or quality control samples and reagents used in sample preparation and/or analysis must meet the criteria described in Section 7. In methods where the purity is not specified, analytical grade reagents are used. Reagents of lesser purity than those specified by the test method are never used. Upon receipt and prior to use the labels on the container are checked to verify that the purity of the reagents meets the documented requirements of the particular test method.

The quality of water sources is monitored and documented as described Section 4. The quality of water used in sample preparation or analysis meets the method-specified requirements. The type of water available in the laboratory is described in Section 4.

F1.8 Selectivity

Absolute and relative retention times aid in the identification of components in chromatographic analyses and to evaluate the effectiveness of a column in separating constituents. The procedures governing retention time widows are documented in the applicable analytical SOP and meet all regulatory and method requirements.

In addition to retention time windows, the acceptance criteria for mass spectral training is also documented in the appropriate analytical SOP. In all cases, the acceptance criteria meet or exceed those specified in the analytical methods.

Unless stipulated in writing by the client, confirmations are performed to verify the compound identification of positive results detected on a sample from a location that has not been previously tested by our laboratory. Such confirmations are performed on a second columns for organic tests such as pesticides, herbicides, or acid extractable or when recommended by the analytical test method except when the analysis involves the use of a mass spectrometer. All conformation is documented.

F1.9 Constant and Consistent Test Conditions

GEL's implementation of standard operating procedures that specify quality criteria including initial and continuing calibrations assures that our test instruments consistently operate within the specifications required of the application for which the equipment is used.

In addition to the specifications applied to instrumentation, glassware used for sample preparation or analyses is cleaned in a manner that reduced the potential for positive or negative interferences. Glassware is prepared in accordance with GL-LB-E-003 for Glassware Preparation.

This SOP details the procedures used to clean the following groups of glassware:

- That used for the determination of metals with a special section for bottles to be used for the determination of mercury by either EPA 7470 or 7471A.

- Reusable bottles and plasticware
- Bottles sued for the determination of biochemical oxygen demand
- Glassware used in the determination of organic compounds
- That used for the determination of methylene blue active substances
- Glassware used in the determination of total organic halides
- Glassware used in the analyses of samples for total kjeldahl nitrogen and total phosphorous
- Generic glassware used in all other analyses

  If the method specifies that the glassware be stored in a particular manner, this requirement is documented in the appropriate analytical SOP.

Section F2: Microbiology

The quality control elements included in this section apply to microbiological analyses performed at GEL. The analyses include the determination of both total and fecal coliforms and standard plate counts.

Discussed in this section are:
- Negative controls
- Positive controls
- Test variability and reproducibility
- Method evaluation
- Test performance
- Data reduction
- Quality of standards, reagents, and media
- Selectivity
- Test conditions

F2.1 Negative Controls

We demonstrate that the cultured samples have not been contaminated during sampling handling and analysis or environmental exposure by the use of negative controls. These negative controls include both sterility checks of media and method blanks.

All blanks and non-inoculated controls specified by the test methods are prepared and analyzed at the frequency stated in the method and in the corresponding standard operating procedure.

A minimum of one non-inoculated control is prepared and analyzed is analyzed with analytical batches containing only one sample. If the analytical batch contains multiple samples, a series of method blanks is prepared. This series includes least one beginning and ending negative control with additional controls inserted after every 10 samples.

If the method blanks show evidence of contamination, the data obtained for the associated samples is not reported and the client is advised that resampling will be necessary.

Prior to initial use, each lot of media is subjected to a sterility check by analyzing an aliquot of sterile buffer water. If there is any evidence of contamination, the media is not utilized for the analysis of samples and is either returned to the supplier or disposed of in accordance with GL-LB-E-011 for Laboratory Sample and Waste Disposal and Emergency Instructions.

F2.2 Positive Controls

Positive controls are used to demonstrate that the medium can support the growth of the target organism and that it produces the specified or expected reaction to that organism.

Prior to the initial and then on a monthly basis each lot of media is tested using least one pure culture of with a known positive reaction. If the positive reaction does not occur, the media is not used for sample analysis and is either returned to the supplier or disposed of in accordance with GL-LB-E-011.

F2.3 Test Variability and Reproducibility

We demonstrate reproducibility of our data by analyzing sample duplicates for least 5% of the suspected positive samples. Each analyst performing microbiological analyses makes parallel analyses on at least one positive sample per month.

For analysis requiring sample volumes of less than 100mL or where the clients submit duplicate sample aliquots, a sample duplicates is analyzed with each analytical batch.

F2.4 Method Evaluation

Our ability to perform a specified analysis successfully for its intended purpose, is demonstrated and documented in meeting at a minimum the acceptance criteria specified by the method, by the EPA, and by state programs under which we are certified. The acceptance criteria demonstrate that the test method as performed at GEL provides correct and expected results with respect to specified detection capabilities, selectivity, and reproducibility.

Proficiency of the analysis is demonstrated prior to the test method through the use of positive and negative controls. The validation of microbiological test methods is conducted under the same conditions as those for routine sample analysis.

All validation data is record in the analytical logbook specified in the appropriate SOP. We maintain this data as long as the analysis is being conducted and for a minimum of five years after this discontinuation of an analytical method.

F2.5 Test Performance

Test performance is demonstrated for all growth and recovery media used by the appropriate growth and reaction of target organisms to the test media through the use of positive controls as discussed in F2.2.

F2.6 Data Reduction

All data is calculated and subjected to data reduction and statistical interpretations as specified by each test method's SOP. These specifications incorporate those found in the associated analytical method.

For test methods specifying colony counts, such as membrane filter or colony counting, then the ability of individual analysts to count colonies is verified at least once per month. This verification includes having two or more analysts count colonies from the same plate.

F2.7 Quality of Standards, Reagents and Media

In addition to the performance of positive and negative controls, we ensure that the quality of the reagents and media meets or exceeds the requirements specified in the analytical methods.

The commercially dehydrated powders used to prepare certain culture media as well as the media that is purchased ready for use are both subjected to positive and negative controls.

In addition, all reagents, commercial dehydrated powders and media are used within the shelf-life of the product as documented in Section 8.

We retain all manufacturer supplied "quality specification statements" which may contain such information as shelf life of the product, storage conditions, sampling regimen/rate, sterility check including acceptability criteria, performance checks including the organism used, their culture collection reference and acceptability criteria, date of issue of specification, or statements assuring that the relevant product batch meets the product specifications.

All media and buffers are prepared using deionized water that has been demonstrated to be free from bacterial contamination. The deionized water used for microbiological analyses and the monitoring of the deionized water is discussed in Section 4.

Media, solutions and reagents are prepared, used and stored in accordance with appropriate SOP.

As described in 2.2, all laboratory media are be evaluated at least monthly to ensure they support the growth of specific microbial cultures. In addition, selective media are checked to ensure they suppress the growth of non-target organisms.

The laboratory detergent is be checked by use of the inhibitory residue test to ensure that its residues do not inhibit or promote growth of microorganisms.

F2.8 Selectivity

We perform all confirmation and verifications tests specified by the test method according to the procedures outlined in our SOPs.

In order to demonstrate traceability and selectivity, we use reference cultures of microorganisms obtained from a recognized national collection. We do not subculture bacterial working stocks. The storage and maintenance of all working and reference stocks are specified in the applicable analytical SOP.

F2.9 Test Conditions

We monitor the levels of by the use of method blanks and other negative controls. The acceptable background counts for each analyses and how to deal with situations in which these levels are exceeded are specified in the applicable SOP.

Walls, floors, ceilings and work surfaces of our microbiological laboratory are non-absorbent and easy to clean and disinfect. Measures are taken to avoid accumulation of dust by the provision of sufficient storage space and daily cleaning of exposed surfaces.

the temperature measuring devices such as liquid-in-glass thermometers used in incubators, autoclaves and other equipment are of the appropriate quality to achieve the specification in the test method.

The graduation of the temperature measuring devices are be appropriate for the required accuracy of measurement. Each device is calibrated at least annually to national or international standards for temperature in accordance with GL-QS-E-007 for Thermometer Calibration.

The temperatures of incubators, refrigerators, autoclaves, and waterbaths are monitored and documented in accordance with GL-LB-E-004 for Temperature Monitoring And Documentation Requirements for Refrigerators, Freezers, Ovens, Incubators, and Other Similar Devices. While in use, each piece of equipment is maintained in the temperature range specified by the applicable SOP and test method.

Records of autoclave operations including temperature and time are maintained for every cycle.

Volumetric equipment such as automatic dispensers, air displacement pipets and disposal pipets are all used in the microbiology laboratory. This equipment is routinely checked for accuracy as discussed in Section 7.

Conductivity meters, pH meters, and other similar measurement instruments are calibrated according to the methods specified requirements detailed in the SOP.

Mechanical timers are checked regularly against electronic timing devices to ensure accuracy.

Section F3: Radiochemical Analysis

This section describes the general quality control applied to radiochemical analysis. The specific quality control criteria applied to each analysis are delineated in the corresponding SOP.

Discussed in this section are:

- Negative controls
- Positive controls
- Test variability/reproducibility
- Tracers and carriers
- Method evaluation
- Radiation measurement system calibration
- Data reduction
- Quality of standards and Reagents
- Test Conditions

F3.1 Negative Controls

Method blanks serve as the primary negative controls providing a means of assessing the existence and magnitude of contamination introduced via the analytical scheme. A method blank is analyzed at a frequency of one per preparation or analytical batch and is one of the quality control measures to be used to assess batch acceptance.

The activity level determined for each target in the method blank is assessed against the specific acceptance criteria specified in the applicable SOP. These criteria are based on a designated sample aliquot size and include appropriate calculations to compare the blank to activity levels determined for different sizes of sample aliquots.

The activity level of any target analyte in the method blank should be less than or equal to the contract required detection limit. The method blank may exceed this limit if the activity is less than 5% that of the lowest sample activity in the batch.

If the method blank acceptance criteria is not met, the specified corrective action and contingencies delineated in the SOPs are followed. Any failures of method blanks to met the acceptance criteria are documented in the laboratory report and through GEL's nonconformance reporting system specified in GL-QS-E-004 for the Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items.

The activity levels determined for method blanks are not subtracted from those obtained for the samples in the associated preparation or analytical batch. Correction factors such as instrument background and analyte presence in the tracer may, however, be applied to all analyzed samples including both client samples and internal quality control samples.

F3. 2 Positive Controls

Positive controls routinely employed in radiochemical analyses include both laboratory control samples (LCS) and matrix spikes (MS.)

The laboratory standards used to prepare LCS and MS are from a different source than those used in instrument calibration. The activity levels of target analytes in the LCS and MS exceed ten times the priori detection limit and are less than one hundred times this detection limit.

If a radiochemical method, however, has more than one reportable analyte isotope, the LCS and MS need to only include one of the analyte isotopes.

Gamma spectroscopy is the exception to this guideline requiring the LCS and MS to contain isotopes representing the low, medium, and high energy range of the analyzed gamma spectra.

F3.2.1 Laboratory Control Sample (LCS)

Laboratory control samples are analyzed at a minimum of once per preparation or analytical batch containing twenty or less samples.

The recovery of target analytes in the LCS is compared to the acceptance criteria (75% - 125%) specified in the applicable analytical SOP. If the recovery of the LCS does not fall within the acceptance range, the corrective actions and contingency steps specified in the SOP are implemented. These steps include the completion of an internal nonconformance report in accordance with GL-QS-E-004 and noting the failure on the laboratory report.

F3.2.2 Matrix Spike (MS)

Matrix spikes are analyzed at a minimum of once per preparation or analytical batch containing twenty samples or less under the following conditions:

- The analytical method does not utilize an internal standard or carrier
- There is a physical or chemical separation process
- There is sufficient sample volume provided for the analysis.

The target analyte recoveries are one of the quality control measures used to assess batch acceptance. The recovery of target analytes in the MS is compared to the acceptance criteria (75% - 125%) specified in the applicable analytical SOP. If the recovery of the MS does not fall within the acceptance range, the data associated with that matrix spike is qualified accordingly.

F3.3 Test Variability/Reproducibility

The reproducibility of measurements is evaluated by the use of matrix duplicates. Matrix duplicates are analyzed once per preparation or analytical batch of twenty samples. The relative percent difference (RPD) obtained between the activity levels obtained for the sample and its duplicate are evaluated against the range in the SOP. This range is 0%- 20% for activities greater than the contract reporting limit. If the RPD exceeds this criteria, the corrective actions addressed in the SOP are implemented .

F3.4 Tracers and Carriers

Two additional quality control measures specific to radiochemical analysis are tracers and carriers. If the analytical method requires a tracer or carrier, each sample result will be associated with a tracer recovery that is calculated and reported. The tracer or carrier recovery should fall within the acceptance criteria specified in the applicable SOP. The acceptance range for tracers is 15 -125% and that for carriers is 25 -125%. If it does not, the corrective actions delineated in the SOP are implemented.

F3.5 Method Evaluation

GEL evaluates the radiochemical preparation and analytical methods to ensure the accuracy of the reported result. This evaluation includes initial demonstrations of capability as described in Section 8 and the analysis of proficiency test samples as described in Section 3. The suppliers of proficiency test samples conform to the requirements of ANSI N42.22

F3.6 Radiation Measurement System Calibration

It is not generally necessary or practical to calibrate radiochemical instrumentation each day of use due to the its stability and the time-consuming nature of some of the measurements. There are, therefore, significant differences in the calibration requirements for radiochemical instrumentation from that used for chemical analyses.

Calibration differences include but are not limited to the following:

- The requirement in Section 7 for the determination of the appropriate number of standards for initial calibration is not applicable to radiochemical methods. If the radiochemical method requires multiple standards for initial calibration, the number of standards is included in the applicable SOP.

- If linear regression or non-linear regression is used to fit standard response or calibration standard results to a calibration curve, the correlation coefficient is determined. This differs from Section 7.

- The requirement identified in Section 7 for the bracketing of quantitative results by calibration or calibration verification standards is not applicable to radiochemical analyses due to the non-correlated event nature of decay counting instrumentation.

- As indicated in Section 7, the LCS may fill the requirements for the performance of an initial calibration and continuing calibration verification standard. The calibration verification acceptance criteria are same as specified for the LCS (75 -125%)

- Background calibration measurements are made on a regular basis and monitored using control charts. These values are subtracted from the total measured activity in the determination of the sample activity. The frequency of these measurements is indicated in the table below.

Instrument type	Minimum Frequency
Gamma spectroscopy	Monthly
Alpha spectroscopy	Monthly
Gas-proportional	Day of use
Scintillation counters	Day of use

- Instrument calibration shall be performed with reference standards as defined in Section F3.8.

- The frequency of calibration shall be addressed in the governing SOPs

F3.7 Data Reduction

All sources of method uncertainties and their propagation must be traceable to reported results. This is performed under the guidance of the ISO "Guide to the Expression of Uncertainty in Measurement" and the NIST Technical Note 1297 on "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results" .

F3.8 Quality of Standards and Reagents

The reference standards we use are obtained from the National Institute of Standards and Technology (NIST), EPA, or suppliers providing NIST standards. Reference standards should be accompanied by a certificate of calibration whose content is described in ANSI N42.22 - 1995, Section 8, Certificates.

All reagents used shall be analytical reagent grade or better.

F3.9 Test Conditions

GEL adheres to written procedures that minimize the possibility of cross contamination between samples. This prevents incorrect analysis results from the cross contamination. Procedures are in place, for example, to separate known radioactive and nonradioactive samples from the time of sample receipt to analysis and sample disposal.

Instrument performance checks are performed on a regular basis and monitored with control charts. This ensures that the instrument is operating properly and that the calibration has not changed. The same check source used in the preparation of the control chart at the time of calibration is used in the performance checks of the instrument. The sources must provide adequate counting statistics for a relatively short count time and should be sealed or encapsulated to provide loss of activity and contamination of the instrument and laboratory personnel.

Instrument performance checks include checks on the counting efficiency and the relationship between channel number and alpha or gamma ray energy. These check are performed at the frequency indicated in the table below.

Instrument	Frequency of Counting Efficiency	Frequency of Channel # and Alpha and Gamma Ray Energy
Gamma Spectroscopy	Day of use	Day of use
Alpha Spectroscopy	Monthly	Day of use
Gas proportional	Day of use	Day of use
Scintillation Counters	Day of use	Day of use

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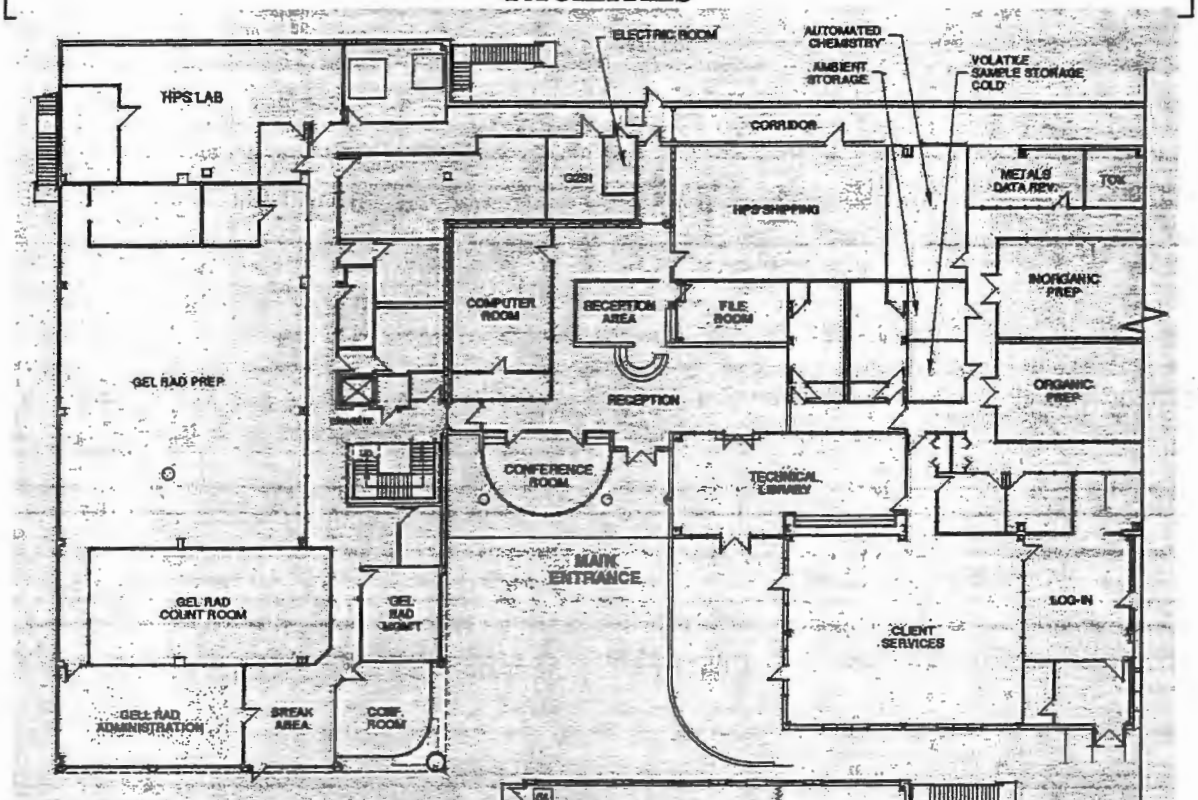
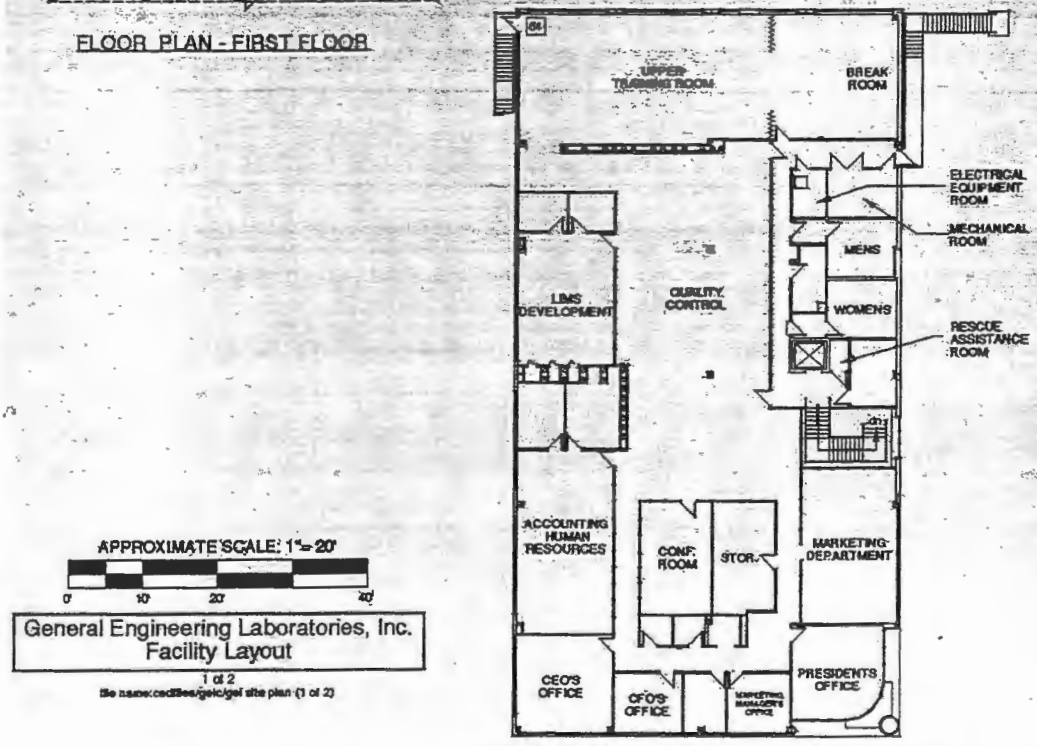
APPENDIX G
FACILITIES



FLOOR PLAN - FIRST FLOOR



FLOOR PLAN - SECOND FLOOR NORTH WING

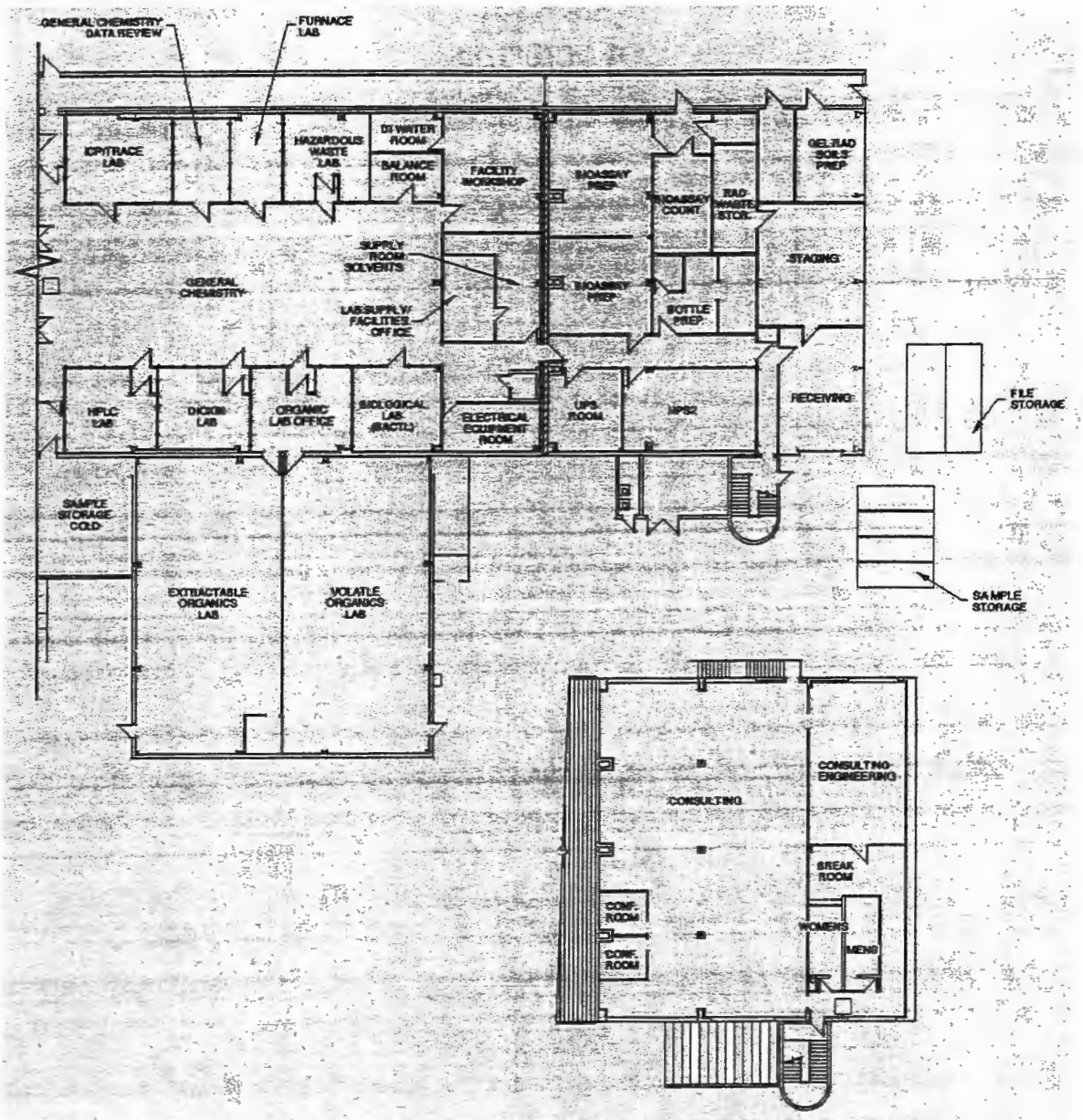
APPROXIMATE SCALE: 1"= 20'

0' 10' 20' 40'

General Engineering Laboratories, Inc.
Facility Layout

1 of 2

file name:cad\ees\gelc\gel site plan (1 of 2)



FLOOR PLAN - SECOND FLOOR SOUTH WING

APPENDIX H
EQUIPMENT LIST

ORGANIC EXTRACTIONS

#	Equipment	Model #	Purch. Date	I.D. #/Serial #
1	Dionex Solvent Extraction	ACE 200	1/97	97070827
2	Tekman Sonic Distribution	600		
1	Analytical Bio-Chemistry Laboratories G.P.C. System	AP-1000	02/93	9231-SI 9176CO 9156/122UVD 1216SCR
3	Ten-place Organomation Continuous Extractors (modified to 5 soxhlet extractors)	13310 13310 13310	06/07/90 06/07/90 06/07/90	1024 1025 1026
2	N-Evaps Organomation	115 1205	06/01/93 06/01/95	5673 15076
4	Zymark Turbovap	Turborvap II	05/96	TV9612N6726 TV9631N6975 TV9628N6939 TV9627N6926

SEMI-VOLATILE ORGANIC ANALYSES

#	Equipment	Model #	Purch. Date	I.D. #/Serial #
1	Tracor Gas Chromatographs, each with Leap Autosamplers and Dual Electron Capture Detectors	540	05/14/91	911864
1	Hewlett-Packard Gas Chromatograph with Dual Electron Capture Detectors and Dual Autosampler	5890	03/94	3336A51011
2	Hewlett-Packard Gas Chromatograph/Mass Spectrometers, each with Autosamplers	5970 5970	07/10/89 10/03/91	2905A11951 3114A13052
2	Hewlett-Packard HPLC Chemstation	1050	10/92	3319A00022 3423A00442
5	Hewlett-Packard 5973 Gas Chromatograph/Mass Spectrometer	5973	5/1/97	US70810371 US72010604 US82311233 US82311481 US82311417
1	Hewlett-Packard Gas Chromatograph with Flow CTC Autosampler	5890	03/92	3203A41418
2	Dionex Ion Chromatograph	Series 4500i	06/01/89 03/01/93	873450 930613
2	Hewlett-Packard Gas Chromatograph-FID	5890	02/91 8/98	3033A33351
1	Head Space Autosampler	CTC-HS500	06/94	30362
4	Hewlett-Packard Gas Chromatograph-ECD	6890	8/97 11/97 3/98 7/98	US00010134 US00009591 US00023402 US00023068

VOLATILE ORGANIC ANALYSES

#	Equipment	Model #	Purch. Date	I.D. #/Serial #
2	Hewlett-Packard Gas Chromatograph/Mass Spectrometer with Tekmar Purge and Arcon Autosampler	5970	07/91	3034A12910 3034A12909
1	Hewlett-Packard Gas Chromatograph/Mass Spectrometer w/DPM-16 Autosampler	5973	11/98	US82311236
1	Hewlett-Packard Gas Chromatograph/Mass Spectrometer Chemstation with OI 4560/Arcon Autosampler	5972	05/93	3341A00976
1	Hewlett-Packard Gas Chromatograph/Mass Spectrometer Chemstation with O.I. 4560/Archon Autosampler	5972	06/93	3251A00145
1	Hewlett-Packard Gas Chromatograph/Mass Spectrometer Chemstation with Tekmar 3000 and Precept II	5973	01/98	US72010562
2	Tracor Gas Chromatograph with one Photoionization Detector and one Flame Ionization Detector and Autosampler 2000/2016	540	11/90	891691 851320
1	Hewlett-Packard Gas Chromatograph/Mass Spectrometer Chemstation with Tekmar Purge and Precept II	5972	02/94	3418A01517
1	Nutech Air Cryogenic Concentrator with Autosampler	3550A		JK-0048-322

1	Entech 8 Place Summa Can Cleaner			
2	Hewlett-Packard Apollo 9000 Series 735 Platforms running: Ingress 6.3, Target 3.12 HPUX 9.05, and Envision 3.20		02/94	6406A00294 6511A00079
5	PE Nelson 2600 Series Gas Chromatography Software System Operating on 386 PC Computers	NEC 386 SX/2	11/90	333572879 333572885 333572881 112573508 333572882

METALS ANALYSES

#	Equipment	Model #	Purch. Date	I.D. #/Serial #
1	Perkin Elmer Mercury Analyzer	Fims 400	11/97	4179
1	Hewlett Packard Inductively Coupled Plasma Mass Spectrometer	4500	08/95	3424J00133
1	Perkin Elmer Graphite Furnace Atomic Absorption Spectrophotometers, each with an Autosampler	5100	07/89	136462
1	Leeman Automatic Mercury Analyzer	PS200	09/93	HG3063
2	Thermo-Jarrell Ash Simultaneous Inductively Coupled Plasma Trace Analyzer with Autosampler and Ultrasonic Nebulizer	61E Trace	01/95	350490 489890

GENERAL CHEMISTRY

#	Equipment	Model #	Purch. Date	I.D. #/Serial #
1	Dohrman Total Organic Carbon Analyzer (with Autosampler)	DC80 (ASM-1)	11/82 (11/82)	9604 (ASHI3087)
1	Dohrman Total Organic Carbon Analyzer	DC190	05/93	9302211
2	Mitsubishi Total Organic Halogen Analyzers	TOX-10-C TOX-10-C	07/84 01/90	43R00334 43R31429
1	Bran & Luebbe/Technicon Automated Chemistry Segmented Flow Autoanalyzer	TRACCS 800	03/90	165-A011-02
1	Alpkem Automated Continuous Flow Autoanalyzer with 510 Detector	RFA300	01/84	7877
1	Alpkem Automated Continuous Flow Solution System Flow Solution with 510 Detector	RFA500	09/90	540
1	Dohrman DX 2000 TOX/EOX	DX2000	02/94	9309876
1	Fourier Transform Infrared Spectrophotometer by Perkin Elmer	1600 FTIR	11/88	134908
1	EM Science Karl Fisher Moisture Analyzer	EV-5	01/86	83109-01
1	Tecator Kjeltec System with (Distiller) and (Block Digestor)	1026 1015	01/93 01/93	10002767
2	Bran & Lubbe Block Digester	BD-20/40	03/90	GG0869033 GG0619005
1	Midi VAP Cyan-Ten Midi Cyanide Distillation	MC-100	07/93	MCVA1390797

2	Sequoia-Turner Spectrophotometer	340	10/93	004966TF 007611TF
2	HACH COD Reactor	COD Reactor	01/94	911005731C 9807000017919
1	Orion Conductivity Meter	160	01/94	32241041
1	Expandable Ionanalyzer	EA940	01/90	2060
1	Setaflash Flashpoint Analyzer	01SF	12/93	2779
1	Parr 1261 Calorimeter	Parr 1261	01/89	289
1	Sartorius Balance	GEL #B005		3410156
2	Sartorius Analytical Balance	GEL #B-010 GEL #B-012		30505030 40245216

AIR ANALYSES

#	Equipment	Model #	Purch. Date	I.D. #/Serial #
3	Nutech Modular Isokinetic Stack Sampling System		01/92	80491
1	Nutech Modified Method 5 Stack Sampling System			
2	Nutech Midget Impinger Stack Sampling System			
1	Nutech Volatile Organic Sampling Train		01/92	8250
1	JUM Total Hydrocarbon Analyzer		02/92	10620192
1	Shimadzu Gas Chromatograph with two Flame Ionization Detectors and One Flame Photometric Detector		01/92	C10552911986
2	Western Research SO2 Analyzer		01/92	91-721AT2-7857

2	ThermoEnvironmental Instruments NOX Analyzer		01/92	10S-35093-251
1	20-Foot Mobile Laboratory Mounted on Diesel Truck Bed		01/92	VX16084096M31798
3	Olympus Phase Contract Microscopes (PCM #1, #2, #3) Green Filter and Walton-Beckett Graticule	CHS w/IF 550	01/92	9F0062 9F0010 307222
1	Tekmar Head Space Autosampler	14-4401-000		91168002
1	Olympus Stereo Zoom Microscope	SZO-60	01/92	SZ4045

RADIOCHEMISTRY

#	Equipment	Model #	Purch. Date	I.D. #/Serial #
2	Tennelec LB-5100 Proportional Counters	LB5100 LB5100W	01/91 08/92	62804 14740
3	Tennelec LB-4100 Proportional Counter with 48 Detectors	LB4100	03/93 06/93 12/98	18483 21938
1	Canberra High Throughput Gas Flow Proportional Alpha/Beta Counter with 8 Detectors	HT1000D	11/91	10912771
4	Beckman Liquid Scintillation Counters	LS6500	06/93 06/93 04/94 11/98	7065155 7067083 7067404 7069123
1	Canberra Scintillation Detector (Nal)	GO470 Relative Efficiency 100%	3/99	
2	Wallac Liquid Scintillation Counters	Guardian Quantallus	3/97 12/98	4040127 2200082

2	PGT Germanium Detectors for Gamma Spectroscopy System	NIGC3019 Relative efficiency 20%	5/97 5/97	2211 2245
4	Canberra Germanium Detectors for Gamma Spectroscopy System	GC3019 Relative efficiency 30%-40%	11/97 11/97 5/97 5/97	1922864 2461 2605 2464
7	Canberra Germanium Detectors for Gamma Spectroscopy System	GC3519 Relative efficiency 35%	12/91 12/91 01/93 01/94 11/97 5/97 5/97	5933088 11912863 12922955 1943199 1943234 7933154 11912876
3	Canberra & Ortec High Efficiency Germanium Detectors for Gamma Spectroscopy System	GC4018 Relative efficiency 40-45%	5/97 11/98 11/98	1933074 30-TN10348 37-TN11260A
2	Canberra & Ortec High Efficiency Germanium Detector for Gamma Spectroscopy System	GC8021 90210-P Relative efficiency 80-90%	08/94 11/98	8943324 30-TP30546A
1	Canberra GX 3519 Extended Range High Efficiency Germanium Detector for Gamma Spectroscopy System	GR3520	08/93	8932581
1	Canberra GCW 3522 Germanium Well Detector for Gamma Spectroscopy System	GCW3523	04/94	3941466
3	Canberra Low Energy Germanium Detector for X-Ray Spectroscopy System	GL2020/S	02/95 01/95 3/98	129 22782 195 4119 3984452
1	Canberra Alpha/Gamma Data Management System	XG3100B	02/92	G-4770
1	Digital Vax Station 4000/90 Computer System for Alpha/Gamma Data Management System	VS49-K-AA	12/94	AB43500 OWN
1	DEC Alpha Work Stations for Alpha/Gamma Data Management System	8544A-R3	11/98	N183806280

66	Canberra PA45020 Alpha Spectrometers for Alpha Spectroscopy System (Environmental)	PA45020	(18ea)02/92 (12ea)10/93 (6ea)06/93 (12ea)3/95 (8ea)5/95	13271, 13911, 13272, 13273, 13258, 13259, 13265, 13268, 13266, 13261, 13296, 13260, 17940, 13912, 13270, 13269, 13274, 13275. 17024, 16318, 16320, 13323, 16323, 16326.
55	Canberra PA45020 Alpha PIPS Detectors for Alpha Spectroscopy System (Bioassay)	PA45020	10/93 03/95 04/95	18915, 18916, 18917, 18918, 18937, 18938, 18939, 18941, 18942, 18943, 19310, 19311. 22846, 22847, 22848, 22849, 22850, 22851, 22838, 22839, 22840, 22841, 22842, 22843. 21063, 21084, 21085, 21086, 21087, 21088, 21090, 13922.
64	Ortec Alpha spectroscopy System	Octete	11/98	
4	Ludlum Scalers for Radium 226 Analysis/Lucas Cells	200	05/05/92 06/93 10/93	92101846 86493 86494 104617
2	Laser Kinetic Phosphorimeter	KPA-11	03/94	9445050064
2	Laser Kinetic Phosphorimeter	KPA-10	04/95	89-0505-0035

UNIVERSAL POWER SUPPLY

#	Equipment	Model #	Purch. Date	I.D. #/Serial #
	International Power Machines Durable Power 300			

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APPENDIX I
STANDARD OPERATIONAL PROCEDURES and ANALYTICAL METHODS

Procedure ID	Title	Associated Method
GL-ADM-E-001	Preparation, Authorization, Change, and Release of SOPs	Not applicable
GL-ADM-E-002	Process, Review, and Distribution of CofAs and COA Packages	Not applicable
GL-ADM-E-003	Courier Service	Not applicable
GL-ADM-E-004	Main Reception Telephone System	Not applicable
GL-ADM-E-005	Outgoing Mail Service	Not applicable
GL-ADM-E-006	Faxing Services	Not applicable
GL-ADM-E-007	MITA 8585 Copier	Not applicable
GL-AP-E-001	Invoicing Analytical Lab Numbers	Not applicable
GL-AP-E-002	Petty Cash	Not applicable
GL-AP-E-003	Establishing Client Project Codes	Not applicable
GL-AP-E-004	Project Manager Billing	Not applicable
GL-CO-E-001	Revising General Engineering Laboratories Catalog of Analytical Services	Not applicable
GL-CO-E-002	Delegated Authorization to Commit the Company	Not applicable
GL-CO-E-003	Request for Proposal (RFP)/Contract Review	Not applicable
GL-CS-E-001	Data File Generation and Shipment of Deliverables for Westinghouse Savannah River Company	Not applicable
GL-CS-E-002	Internal Review of Contractually Required Quality Criteria for Client Package Delivery	Not applicable
GL-DC-E-001	Document Control	Not applicable
GL-EPI-A-001	Determination of Gross Alpha And Gross Non-Volatile Beta in Water	EPI-AOO1 and EPA 900.0
GL-EPI-A-001B	Determination of Gross Alpha And Gross Non-Volatile Beta in Soil	EPI-A001B and EPA 900.0
GL-EPI-A-001C	Determination of Gross Alpha in Water by Coprecipation	EPI-AOO1C and EPA EERF 00-02-1
GL-EPI-A-002	Determination of Tritium	EPI-A002, EPI-AOO2b and EPA 906.0
GL-EPI-A-003	Determination of Carbon-14 in Water	EPI-AOO3 and EPA EERF C-01
GL-EPI-A-003B	Determination of Carbon-14 in Soil, Vegetation, and Other Solid Matrices	EPA EERF C
GL-EPI-A-004	Determination of Strontium 89/90 in Water and Soil	EPI-AOO4 and EPA 905.0
GL-EPI-A-005	Determination of Technitium-99	EPI-AOO5 and DOE EML HASL 300

GL-EPI-A-006	Determination of Iodine-129	EPI-AOO6, EPA 901.1, LANL EM-9 and DOE EML HASL 300
GL-EPI-A-007	Determination of Radon-222 in Water	EPI-A007 and EPA EERF Appendix B
GL-EPI-A-007B	Determination of Radon-222 in Soil	EPI-A007B and EPA EERF Appendix B
GL-EPI-A-008	Determination of Radium-226	EPA 903.1 and EPI-A008
GL-EPI-A-009	Determination of Radium-228 in Water	EPI-A009 and EPA 904.0
GL-EPI-A-009B	Determination of Total Alpha Emitting Radium and Radium-228 in Water	DOE EML HASL 300 and EPI-A009b
GL-EPI-A-010	Total Alpha Radium Isotopes in Water	EPI-A010, EPA 900.0, and DOE EML HASL 300
GL-EPI-A-010B	Total Alpha Radium Isotopes in Soil	EPI-A010b and DOE EML HASL 300
GL-EPI-A-011	Isotopic Determination of Americium, Curium, Plutonium, and Uranium	DOE EML HASL 300 and EPI-A011
GL-EPI-A-012	Isotopic Determination of Thorium	EPI-A012 and DOE EML HASL 300
GL-EPI-A-013	Determination of Gamma Isotopes in Water and Soil	EPA 901.1, EPI-A013, and DOE EML HASL 300
GL-EPI-A-014	Determination of Total Radioactivity in Contact Waste	EPI-A014
GL-EPI-A-015	Microwave Digestion for Soils and Sand	EPI-A015
GL-EPI-A-016	Determination of Polonium in Water	EPI-A016 and DOE EML HASL 300
GL-EPI-A-016B	Determination of Polonium in Soil	EPI-A016b and DOE EML HASL 300
GL-EPI-A-017	Determination of Iodine-131 in Water	EPA 902.0, and DOE EML HASL 300
GL-EPI-A-018	Determination of lead-210 in Water	EPI-A013 and DOE EML HASL 300
GL-EPI-A-019	Determination of Phosphorus-32 in Soil and Water	DOE RESL P1-1 and EPI-A019
GL-EPI-A-020	Determination of Promethium-147 in Soil and Water	EPA EERF PM-01-1 and EPI-A020
GL-EPI-A-021	Soil Sample Preparation for the Determination of Radionuclides	EPI-A021 and ASTM
GL-EPI-A-021B	Soil Sample Ashing for the Determination of Radionuclides	EPI-A021b
GL-EPI-A-022	Determination of NI-59 and NI-63 in Soil and Water	DOE RESL Ni-1 and EPI-A022
GL-EPI-A-023	Total Uranium in Water, Soil, and Urine in Kinetic Phosphorescence	ASTM D5174 and EPI-A023
GL-EPI-A-024	Determination of Radium-223 and Radium-224 Isotopes in Water	DOE RESL A-3-1,DOE EML HASL 300, and EPI-A013b

GL-EPI-A-025	Determination of Iron-55 in Soil and Water	DOE RESL Fe-1 and EPI-A025
GL-EPI-A-026	Preparation of Special Matrices for the Determination of Radionuclides	EPI-A026 and DOE EML HASL 300
GL-EPI-A-027	Uranium in Drinking Water by EPA Method 908.0	EPA 908.0
GL-EPI-A-028	Radium-226 in Drinking Water by EPA Method 903.1	EPA 903.1
GL-EPI-A-029	Determination of Strontium-89/90 in Drinking Water by EPA Method 905.0	EPA 905.0
GL-EPI-A-030	Determination of Radium-228 in Drinking Water by EPA Method 904.0	EPA 904.0
GL-EPI-A-031	Determination of Selenium and Tellurium	EPI-A028 and NERC ORD
GL-EPI-A-032	Isotopic Determination of Neptunium	DOE EML HASL 300 and EPI-A012
GL-EPI-A-034	Isotopic Plutonium in Soil	EPI-A012 and DOE EML HASL 300
GL-EPI-B-001	Sequential Determination of Isotopic Uranium, Americium, Plutonium, and Neptunium in Bioassay Samples	EPI-A012 and DOE EML HASL 300
GL-EPI-B-002	Determination of Polonium-210, Radium-226, and Radium-228 in Urine	EPI-A016 and DOE EML HASL 300
GL-EPI-B-003	Determination of Isotopic Thorium in Bioassay Samples	EPI-A012 and DOE EML HASL 300
GL-EPI-B-004	Determination of Lead-210 in Bioassay Samples	EPI-A013 and DOE EML HASL 300
GL-EPI-B-005	Determination of Blank Populations	EPI-B005
GL-EPI-B-008	Determination of Gross Alpha Activity in Nasal Swipes	EPI-A001 and EPA 900.0
GL-EPI-B-009	Bioassay Countroom Instrumentation Maintenance, Performance Checks and Operation	EPI-B009
GL-EPI-D-001	Standard Operating Training Procedure	Not applicable
GL-EPI-D-002	Analyst and Analytical Methods Validation	Not applicable
GL-EPI-D-003	Data Review, Validation, and Data Package Assembly	Not applicable
GL-EPI-D-004	Standard Operating Supply Maintenance Procedure	Not applicable
GL-EPI-E-S001	Radiation Survey Procedures	Not applicable
GL-EPI-E-S002	Radiation Related Emergency Procedures	Not applicable
GL-EPI-E-S003	Inventory and Tracking of Radioactive Material	Not applicable
GL-EPI-E-S004	Radioactive Material Handling Procedure	Not applicable
GL-EPI-E-S006	Radiation Worker Training	Not applicable
GL-EPI-E-S007	Receiving of Radioactive Samples	Not applicable
GL-EPI-E-S008	Shipping of Radioactive Samples and Sample Residues	Not applicable
GL-EPI-E-S009	Dosimetry Procedures	Not applicable

GL-EPI-E-S010	Handling of Biological Materials	Not applicable
GL-EPI-E-S011	EPI Laboratory Waste Disposal and Emergency Instructions	Not applicable
GL-EPI-I-001	Gamma Spectroscopy System Operating Procedures	Not applicable
GL-EPI-I-002	HT1000 Gross Alpha/Beta Counter Operating Instructions	Not applicable
GL-EPI-I-003	LB5100 Gross Alpha/Beta Counter Operating Instructions	Not applicable
GL-EPI-I-004	Beckman LS-6000/6500 Operating Procedures	Not applicable
GL-EPI-I-005	LB5100W Gross Alpha/Beta Counter Operating Instructions	Not applicable
GL-EPI-I-006	LB4100 Gross Alpha/Beta Counter Operating Instructions	Not applicable
GL-EPI-I-007	Ludlum Model 2000 Lucas Cell Counter Operating Instructions	Not applicable
GL-EPI-I-008	VAX/VMS Quality Control Software Program	Not applicable
GL-EPI-I-009	The Alpha Spectrocopy System	Not applicable
GL-EPI-I-010	Counting Room Instrumentation Maintenance and Performance Checks	Not applicable
GL-EPI-I-011	Operation of the Chemchek Kinetic Laser Phosphorimeter	Not applicable
GL-EPI-I-012	Managing Statistical Data in the Radiochemistry Laboratory	Not applicable
GL-EPI-M-001	Preparation of Radioactive Standards	Not applicable
GL-EPI-M-002	Verification of Customer Work Order and Billing Procedures	Not applicable
GL-EPI-M-003	Magnetic Backup of Hard Drives	Not applicable
GL-EPI-M-006	Validation and Verification of Software	Not applicable
GL-EPI-M-007	Segregation of Radioactive Samples	Not applicable
GL-EPI-M-008	Generation and Shipment of Electronic Disk Deliverables	Not applicable
GL-FC-E-001	Facility Security	Not applicable
GL-FC-E-002	Testing Emergency Eyewash and Shower Equipment	Not applicable
GL-FC-E-003	Fume Hood Face Velocity Performance Checks	Not applicable
GL-FC-E-004	Inspection of Fire Extinguishers	Not applicable
GL-GC-E-001	Total Dissolved Solids	EPA 160.1 and Standard Methods 18th ed. 2540C
GL-GC-E-002	Fluoride Determination by Ion Selective Electrode	EPA 340.2 and Standard Methods 18th ed. 4500 F
GL-GC-E-003	Iodine	Standard Methods 18th ed. 4500 I (B)
GL-GC-E-004	Standards - Definitions and Preparation	Not applicable
GL-GC-E-005	Colilert	South Carolina DHEC

GL-GC-E-006	Total Inorganic and Total Organic Carbon (TOC) Using the Dohrmann DC-80 TOC Analyzer	SW846 9060 and EPA 415.1
GL-GC-E-007	Total Organic Halogen (TOX) on Liquid Samples Using the Mitsubishi TOX-10 Analyzer	SW846 9020B
GL-GC-E-008	pH	SW846 9040B, SW846 9041A, SW846 9045C and EPA 150.1
GL-GC-E-009	Conductivity	SW846 9050, Standard Methods 18th ed. 2510B and EPA 120.1
GL-GC-E-010	Paint Filter Test	SW846 9095A
GL-GC-E-011	Total Solids	EPA 160.3 and Standard Methods 18th ed. 2540 B
GL-GC-E-012	Total Suspended Solids	EPA 160.2 and Standard Methods 18th ed. 2540 D
GL-GC-E-013	Total Phenol Sample Preparation and Analysis by Alpkem RFA	SW846 9066 and EPA 420.2
GL-GC-E-014	Total and Amenable Cyanide Analysis by Alpkem RFA	SW846 9012A, EPA 335.3/335.4, and 335.2 CLP-Modified
GL-GC-E-015	Chlorides by Silver Nitrate	Standard Methods 18th ed. 4500CL(-) B
GL-GC-E-016	Release of Cyanide and Sulfide	HACH
GL-GC-E-017	Gravimetric Total Petroleum Hydro Carbons (TPH) in Liquids	SW846 9070A (Modified) and Standard Methods 18th ed. 5520F
GL-GC-E-018	Determination of Oil and Grease and Total Recoverable Petroleum Hydrocarbons (TPH) in Solid Matrices	SW846 9071A, Standard Methods 18th ed. 5520 E (Modified)/5520F
GL-GC-E-019	Ammonia Determination by Traacs 800 Methodology	EPA 350.1
GL-GC-E-020	Gravimetric Determination of Oil and Grease in Liquid Samples	SW846 9070, Standard Methods 18th ed. 5520B and EPA 413.1
GL-GC-E-021	Determination of Total Petroleum Hydrocarbons Solid by Infrared Spectrophotometry	EPA 418.1 (Modified)
GL-GC-E-022	Determination of Total Petroleum Hydrocarbons Liquid by Infrared Spectrophotometry	EPA 418.1
GL-GC-E-023	Determination of Corrosivity	Standard Methods 16th ed. 203
GL-GC-E-024	General Chemistry: Data Entry and Data Review Procedures	Not applicable
GL-GC-E-025	Hardness by Calculation	Standard Methods 18th ed. 2340B
GL-GC-E-026	Total Phosphorus	EPA 365.4
GL-GC-E-027	Pensky-Martens Closed Cup Flashpoint	SW846 1010

GL-GC-E-028	Carbonaceous Biochemical Oxygen Demand (CBOD)	Standard Methods 18th ed. 5210 and EPA 405.1 (Modified)
GL-GC-E-029	Corrosivity Toward Steel	SW846 1110 (Modified)
GL-GC-E-030	Flammability Potential Screening Analysis of Waste	ASTM D4982-89
GL-GC-E-031	Fecal Coliform by Membrane Filter	Standard Methods 18th ed. 9222D
GL-GC-E-032	Carbon Dioxide (Total and Free) by Calculation	Standard Methods 18th ed. 4500-CO2-D and EPA 310.1 (Modified)
GL-GC-E-033	Alkalinity - Total, Bicarbonate Carbonate, Hydroxide, and Phenolphthalein	Standard Methods 18th ed. 2320B and EPA 310.1
GL-GC-E-034	Fecal Coliform Most Probable Number (5 Tube Dilution)	Standard Methods 18th ed. 9221-E1 and Standard Metohds 18th ed. 9221-E2
GL-GC-E-035	Volatile Suspended Solids	Standard Methods 18th ed. 2540 E and EPA160.2/160.4 (Modified)
GL-GC-E-036	Color Determination by Visual Comparison	Standard Methods 18th ed. 2120B and EPA 110.2
GL-GC-E-037	Turbidity	EPA 180.1
GL-GC-E-038	Inhibitory Residue Analysis	Standard Methods 18th ed. M 9020, Part 3.2a
GL-GC-E-039	Laboratory Water Toxicity	South Carolina DHEC
GL-GC-E-040	Cyanide Amenable to Chlorination	SW846 9012 and EPA 335.1(Modified)/335.3 (Modified)
GL-GC-E-041	Nitrate/Nitrite Sample Preparation and Analysis Using the Traacs 800 Autoanalyzer	EPA 353.1
GL-GC-E-042	Cation Exchange Capacity of Soils (Ammonium Acetate)	SW846 9080(Modified)
GL-GC-E-043	Cation Exchange Capacity of Soils (Sodium Acetate)	SW846 9081
GL-GC-E-044	Colorimetric Determination of Chromium, Hexavalent	SW846 7196A
GL-GC-E-045	Biochemical Oxygen Demand (BOD)	Standard Methods 18th ed. 5210 and EPA 405.1
GL-GC-E-046	Orthophosphate	EPA 365.2 and Standard Methods 18th ed. 4500-P (E)
GL-GC-E-047	Methylene Blue Active Substance	EPA 425.1
GL-GC-E-048	Heating Value Determination by Bomb Calorimeter	ASTM D240-87(Modified), ASTM D2382-88(Modified) and ASTM E711-87(Modified)

GL-GC-E-049	Formaldehyde	Association of Analytical Chemists 931.08 (Modified)
GL-GC-E-050	Threshold Odor, Consistent Series EPA 140.1	EPA 140.1
GL-GC-E-051	Argentometric, Alpha 210C	Standard Methods 18th ed. 2520B
GL-GC-E-052	Sulfide (Methylene Blue Method)	EPA 376.2 (Modified)
GL-GC-E-053	Heterotrophic Plate Count (Standard Plate Count)	Standard Methods 18th ed. 9215
GL-GC-E-054	Total Coliform by Membrane Filter	Standard Methods 18th ed. 9222B (Modified)
GL-GC-E-055	Total Kjeldahl Nitrogen (TKN) Analysis Using the Traacs 800 Autoanalyzer	EPA 351.2
GL-GC-E-056	Sulfide	Standard Methods 18th ed. 4500 SO3 (2-) B
GL-GC-E-057	Volatile Solids and % Ash-550-Procedure for Water Samples	Standard Methods 18th ed. 2540 E
GL-GC-E-058	Volatile Solids and % Ash-550-Procedure for Solid and Semi-Solid Samples	Standard Methods 18th ed. 2540 G
GL-GC-E-059	Dissolved Oxygen Analysis by Membrane Electrode Method	Standard Methods 18th ed. 4500 O (G)
GL-GC-E-060	Extractable Organic Halides	SW846 9023
GL-GC-E-061	Chemical Oxygen Demand (COD) - Digestion Reactor Method	HACH 8000
GL-GC-E-062	Total Carbon and Total Organic Carbon Analysis Using the Dohrmann DC-190 Boat Sampler	SW846 9060 (Modified)
GL-GC-E-063	Total Coliform by Most Probable Number (5 Tube Dilution)	Standard Methods 9221B (Modified)
GL-GC-E-064	Density	ASTM D5057
GL-GC-E-065	Specific Gravity	ASTM D5057
GL-GC-E-066	Flashpoint by Setaflash	SW846 1020A
GL-GC-E-067	Total Cyanide Sample Preparation	SW846 9012, SW846 9013, EPA 335.3, EPA 335.4, and EPA 335.2 CLP
GL-GC-E-068	Viscosity	Manufacturer's method
GL-GC-E-069	Reactive Cyanide and Sulfide	SW846 Chapter 7 Section 3
GL-GC-E-070	Determination of Kinematic and Dynamic Viscosity Using a Glass Capillary Viscometer	ASTM D445-82
GL-GC-E-071	Total Phosphorous Sample Preparation	EPA 365.4
GL-GC-E-072	Ammonia Sample Preparation	EPA 350.1
GL-GC-E-073	Free Cyanide Analysis by Microdiffusion	ASTM D4282
GL-GC-E-074	Extractable Organic Halides (EOX) Using the Dohrmann DX-2000 Analyzer	SW846 9023

GL-GC-E-075	Total Organic Halogen (TOX) Analysis on Liquid Samples Using the Dohrmann DX-2000 Analyzer	SW846 9020B
GL-GC-E-076	Total Residue Chlorine	EPA 330.5
GL-GC-E-077	Cyanide Weak Acid Dissociable Sample Preparation and Analysis	ASTM D2036
GL-GC-E-078	Total Kjeldahl Nitrogen Preparation Using the Traacs 800 Autoanalyzer	EPA 351.2
GL-GC-E-079	Bomb Preparation Method for Solid Waste	SW846 5050
GL-GC-E-080	Total Coliform Membrane Filter Analysis by SW-846 Method	SW846 9132
GL-GC-E-081	Total Coliform Most Probable Number (5 Tube Dilution) Analysis by SW-846 Method	SW846 9131
GL-GC-E-082	Acid-Soluble Sulfides	SW846 9030B
GL-GC-E-083	Chlorophyll A and Pheophytin A by Spectrophotometer	Standard Methods 18th ed.10200 H(2)
GL-GC-E-084	Dissolved Oxygen Analysis by the Azide Modification of the Winkler Titration Method	Standard Methods 18th ed. 4500 O (C)
GL-GC-E-085	Ammonia (NH3-N) by Selective Electrode Method	Standard Methods 18th ed. 4500 NH3 (F)
GL-GC-E-086	Ion Chromatography	Standard Methods 18th ed. 4110 B, EPA 300.0 and SW846 9056
GL-GC-E-087	Percent Water by Karl Fischer Titration	ASTM E203-96
GL-GC-E-090	Acidity	Standard Methods 18th. ed. 2310B and EPA 305.1/305.2
GL-HR-E-002	Employee Training	Not applicable
GL-IMS-E-001	Software Modification	Not applicable
GL-IMS-E-002	Computer Software Development and Maintenance	Not applicable
GL-IMS-E-003	Vendor Supplied Analytical Equipment Software	Not applicable
GL-LB-E-001	Determination of Method Detection Limits	Not applicable
GL-LB-E-002	Balance	Not applicable
GL-LB-E-003	Glassware Preparation	Not applicable
GL-LB-E-004	Temperature Monitoring and Documentation Requirements for Refrigerators, Ovens, Incubators, and Other Similar Devices	Not applicable
GL-LB-E-005	Data Review/Validation	Not applicable
GL-LB-E-006	Toxicity Characteristic Leaching Procedure Preparation	Not applicable
GL-LB-E-007	Laboratory Standards Documentation	Not applicable
GL-LB-E-008	Basic Requirements for the Use and Maintenance of Laboratory Notebooks, Logbook, Forms and Other Record Keeping Devices	Not applicable
GL-LB-E-009	Run Logs	Not applicable
GL-LB-E-010	Maintenance and Use of Air Displacement Pipets	Not applicable

GL-LB-E-011	Laboratory Sample and Waste Disposal and Emergency Instructions	Not applicable
GL-LB-E-012	Verifying the Maintenance of Sample Integrity	Not applicable
GL-LB-E-013	Generation and Assembly of CLP Data Packages	Not applicable
GL-LB-E-014	Operation of the Plastic Granulator	Not applicable
GL-LB-E-015	Control of Laboratory Standards	Not applicable
GL-LB-E-016	DI Water System	Not applicable
GL-LB-E-017	Procedure and Policy for Manual Integration	Not applicable
GL-LB-E-018	Instrument Clock Verification	Not applicable
GL-LB-E-019	Statistical Process Control	Not applicable
GL-LB-E-020	Tuning of High Intensity Ultra-Sonic Processor	Not applicable
GL-LB-E-021	Prep for Oily Wastes by Method 1330A	SW846 1330A
GL-MA-E-001	Determination of Mercury by Cold Vapor Atomic Absorption Spectrometry	SW846 7040A, SW846 7471A, EPA 245.1, EPA 245.5, Standard Methods 18th ed. 3112B
GL-MA-E-002	Determination of Metals by the Thermo Jarrell Ash 1CAP-61E Spectrometer	SW846 6010B, EPA 200.7 and CLP 200.7
GL-MA-E-003	Perkin-Elmer Zeeman 5100 Graphite Furnace Atomic Absorption Spectrometer	SW846 7041, SW846 7060A, SW846 7421, SW846 7740, SW846 7841, EPA 204.2, CLP 204.2, EPA 206.2 , CLP 206.2, EPA 239.2, CLP 239.2, EPA 270.2, CLP 270.2, EPA 279.2, and CLP 279.2
GL-MA-E-004	Metals Standards	Not applicable
GL-MA-E-005	Thermo Jarrell Ash Auto AA Furnace	SW 846 7041, SW846 7060, SW846 7741, SW846 7841, EPA 206.2, EPA 239.2, EPA 270.2, and EPA 279.2
GL-MA-E-006	Acid Digestion of Total Recoverable or Dissolved Metals in Surface and Groundwater Samples for Analysis by FLAA, ICP, or ICP-MS	SW846 3005A and EPA 200 Series
GL-MA-E-007	Acid Digestion of Aqueous Samples and Extracts for Analysis by GFAA or ICP-MS	SW846 3020A and EPA 200 Series
GL-MA-E-008	Acid Digestion of Total Metals in Aqueous Samples and Extracts for Analysis by FLAA, ICP, or ICP-MS	SW846 3010A and EPA 200 Series
GL-MA-E-009	Acid Digestion of Sediments, Sludges, and Soils	SW846 3050A and EPA 200 Series
GL-MA-E-010	Mercury Analysis Using the Leeman PS200 Automated Mercury Analyzer	SW846 7470A, SW846 7471A, Standrd Methods 18th ed. 3112B, EPA 245.1, CLP 245.1, EPA 245.5 and CLP 245.5

GL-MA-E-011	Determination of Metals by ICP with the Thermo Jarrell Ash Enviro II Spectrometer	SW846 6010B, EPA 200.7 and CLP 200.7
GL-MA-E-012	Inorganic CLP Sample Digestions	ILMO 3.0
GL-MA-E-013	Determination of Metals by ICP with the Thermo Jarrell Ash ICAP-61E Trace Spectrometer	SW846 6010B and EPA 200.7 and CLP 200.7
GL-MA-E-014	Determination of Metals by ICP-MS with the Hewlett-Packard Model 4500 Spectrometer	SW846 6020 and EPA 200.8
GL-MA-E-015	Microwave Based Atmosphere Pressure Acid Digestion of Total Recoverable and Dissolved Metals	SW846 200 Series
GL-OA-E-001	Establishing Retention Time Windows for Gas Chromotographic Analysis	SW846 8000
GL-OA-E-002	Organic Standards Preparation and Traceability	Not applicable
GL-OA-E-003	Non-Volatile Total Petroleum Hydrocarbons by Flame Ionization Detector	SW846 8000B, SW846 8015A, SW846 3510B, SW846 3550A and California Method
GL-OA-E-004	Volatile Total Petroleum Hydrocarbons by Flame Ionization Detector	SW846 8000B, SW846 8015B, and SW846 5030B
GL-OA-E-007	Dioxins and Furans	SW846 8280
GL-OA-E-008	Volatile Organic Compounds (VOC) by Gas Chromatograph/Mass Spectrometer (GC/MS)	SW846 8240B and EPA 624
GL-OA-E-009	Semivolatile Analysis by Gas Chromatograph/Mass Spectrometer	SW846 8270C and EPA 625
GL-OA-E-010	Extraction of Semivolatile and Nonvolatile Organic Compounds from Soil, Sludge, and Other Miscellaneous Samples	SW846 8015B, SW846 8081A, SW846 8082, SW846 8270C, and SW846 8310
GL-OA-E-011	Chlorophenoxy Acid Herbicides	SW846 8151A and Standard Methods 18th ed. 6640 B
GL-OA-E-012	Determination of Trace Amounts of Otto Fuel II in Water and Soil Samples by GC/ECD	FID
GL-OA-E-013	Extraction of Semivolatile and Nonvolatile Organic Compounds from Groundwater, Wastewater, and Other Aqueous Samples	SW846 8015B, SW846 8081A, SW846 8082, SW846 8270C, SW846 8310, EPA 608 and EPA 625
GL-OA-E-014	Volatile Organic Hydrocarbons by GC/ELCD	SW846 8010 and EPA 601
GL-OA-E-015	Extraction of Herbicides from Groundwater, Wastewater, and other Aqueous Samples	SW846 8151
GL-OA-E-017	Polychlorinated Biphenyls	SW846 8000, SW846 8081, and EPA 608
GL-OA-E-018	Organochlorine Pesticides, PCBs, and Chlorinated Hydrocarbons	SW846 8000, SW846 8081 and EPA 608
GL-OA-E-020	Percent Moisture	SW846 3550

GL-OA-E-021	Volatile Organic Aromatic Compounds by Gas Chromatograph and Photoionization Detector	SW846 8020 and EPA 602
GL-OA-E-022	Volatile Organic Compounds by Gas Chromatograph/Mass Spectrometer	EPA 524.2
GL-OA-E-023	Volatile Organic Aromatics by Gas Chromatograph Using Photoionization and Flame Ionization Detectors	SW846 8015A (Modified), SW846 8020A and EPA 602
GL-OA-E-024	Cleanup and Extraction of Polyurethane Foam Cartridge (PUF) for Semivolatile Analysis	SW846 8270 (Modified), TO-4, EPA 608 (Modified) and TO-13
GL-OA-E-026	Volatile Organic Compounds (VOC) by Gas Chromatograph/Mass Spectrometer	SW846 8260A and EPA 624
GL-OA-E-027	Extraction of Herbicides from Soil and Sludge Samples	SW846 8151A
GL-OA-E-028	Determination of Volatile Organic Compounds (VOC) in Ambient Air Using Summa Passivated Canister Sampling and GC/MS Analysis	TO-14
GL-OA-E-030	Polynuclear Aromatic Hydrocarbons	SW846 8000B and SW846 8310
GL-OA-E-032	Extraction of Herbicides from Groundwater, Wastewater, and Other Aqueous Samples Esterified Using Boron Trifluoride	Standard Methods 18th ed. 6640B
GL-OA-E-033	Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC)	SW846 8000A and SW846 8330
GL-OA-E-034	Volatile Organic Hydrocarbons by Gas Chromatograph (GC)/Electrolytic Conductivity Detector (ELCD)/Photoionization Detector (PID)	SW846 8021A
GL-OA-E-035	Extraction of Herbicides from Soil, Sludge, and Other Solid Samples Esterified Using Boron Trifluoride	SW846 8151(Modified) and Standard Methods 18th ed. 6640B
GL-OA-E-036	Florosil Cleanup of Organochlorine Pesticide Solvent Extracts	SW846 3550B, SW846 3610C and SW846 8081A
GL-OA-E-037	Sulfuric Acid/Permanganate Cleanup of PCB Solvent Extract	SW846 3550B, SW846 3610C and SW846 8082
GL-OA-E-038	Volatile Organic Compounds (VOC) by GC/MS by Methods 8260B/5030B/5035	SW846 5030B, SW846 5035 and SW846 8260B
GL-OA-E-039	Volatile Organics in Soil and Water Samples by Method 5035	SW846 5035
GL-OA-E-040	Polychlorinated Biphenyls by Method 8082	SW846 8000B and SW846 8082
GL-OA-E-041	Organochlorine Pesticides and Chlorinated Hydrocarbons by Methods 8081A/8121	SW846 8000B, SW846 8080, SW846 8081, SW846 8081A, SW846 8121 and EPA 608

GL-OA-E-042	Gas Chromatograph (GC) Using Photoionization (PID) and Flame Ionization Detectors (FID) by Method 8021B	SW846 5030B, SW846 5035 and SW846 8021B
GL-QS-E-001	Conduct of Quality Audits	Not applicable
GL-QS-E-002	Conducting Corrective Action	Not applicable
GL-QS-E-003	Training, Qualifying, and Certifying Quality Assurance Audit Personnel	Not applicable
GL-QS-E-004	Documentation of Nonconformance Reporting and Dispositioning and Control of Nonconforming Items	Not applicable
GL-QS-E-005	Review of Monitoring Device Logs	Not applicable
GL-QS-E-007	Thermometer Calibration	Not applicable
GL-QS-E-008	Quality Records Management and Disposition	Not applicable
GL-QS-E-009	Sample Report Preparation and Review	Not applicable
GL-RC-E-001	Receipt and Inspection of Material and Services	Not applicable
GL-RC-E-002	Material Requisition Form Procedure	Not applicable
GL-RC-E-003	Sample Bottle Preparation and Shipment	Not applicable
GL-SR-E-001	Sample Receipt, Login, and Storage	Not applicable
GL-SR-E-002	Return of Samples	Not applicable

APPENDIX J
SAMPLE STORAGE AND PRESERVATION REQUIREMENTS

Parameter	Container[1]	eservation	Holding Time[2]
Inorganics			
Acidity	P,G	4°C	14 days
Alkalinity	P,G	4°C	14 days
Demand (BOD)	P,G	4°C	48 hours
Bromide	P,G	None	28 days
Chemical Oxygen Demand (COD)	P,G	4°C, H_2SO_4 to pH<2	28 days
Chlorine by Bomb	P,G	None	None
Chloride	P,G	None	28 days
Color	P,G	4°C	48 hours
Conductivity	P,G	4°C	28 days
Corrosivity by pH	P	None	Immediate
Corrosivity to Steel	P	None	None
Cyanide amenable to chlorination	P,G	4°C, NaOH to pH>12, 0.6g ascorbic acid [3]	14 days [4]
Cyanide, total	P,G	4°C, NaOH to ph>12, 0.6g ascorbic acid [3]	14 days [4]
Dissolved Oxygen	G (bottle and tap	None	Immediate
Fixed and Volatile Solids	P,G	4°C	7 days
Flashpoint	P,G	None	None
Fluoride	P	None	28 days
Hardness	P,G	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months
Heating Value	P	None	None
Hydrazine	G	HC1 to pH<2	Immediate
Percent (%) Moisture	P	4°C	None
Ammonia Nitrogen	P,G	4°C, H_2SO_4 to pH<2	28 days
Nitrate/Nitrite	P,G	4°C, H_2SO_4 to pH<2	28 days
Total Kjeldahl and Organic Nitrogen	P,G	4°C, H_2SO_4 to pH<2	28 days
Odor	G	4°C, Zero headspace	Immediate
Oil and Grease	G	4°C, HC1 or H_2SO_4 to pH<2	28 days
Orthophosphate	P,G	Filter immediately, 4°C	48 hours
Total Phenols	G	4°C, H_2SO_4 to pH<2	28 days

pH	P,G	None	Immediate
Total Phosphorus	P,G	4°C, H_2SO_4 to pH<2	28 days
Residual Chlorine	P,G	None	Immediate
Salinity	P	None	28 days
Specific Gravity	P	4°C	7 days
Sulfate	P,G	4°C	28 days
Sulfide	P,G	4°C, add ZNAce and NaOH to pH>9	7 days
Sulfite	P,G	None	Immediate
Sulfur by Bomb	G	None	None
Surfactants	P,G	4°C	48 hours
Settleable Solid	P,G	4°C	48 hours
Total Dissolved Solid	P,G	4°C	7 days
Total Solid	P,G	4°C	7 days
Total Suspended Solid	P,G	4°C	7 days
Volatile Solid	P,G	4°C	7 days
Total Organic Carbon	P,G	4°C,HCl or H_2SO_4 to pH<2	28 days
Total Organic Halides	G	4°C, H_2SO_4 to pH<2	28 days
Total Petroleum Hydrocarbons	G	4°C, H_2SO_4 to pH<2	28 days
Turbidity	P,G	4°C	48 hours
Metals (except chromium VI and mercury)	P	HNO_3 to pH<2	6 months
Chromium VI - Aqueous	P	4°C	24 hours
Chromium VI - Solids	P	4°C	7 days for extraction
Mercury - Wastewater and Drinking water	P,G	HNO_3 to pH<2	28 days
Mercury - Others	G	HNO_3 to pH<2	38 days
Bacteriology			
Coliform, fecal	P,G	4, 0.008% $Na_2S_2O_3$ [3]	6 hours
Standard Plate Count	P,G	4°C, 0.008% $Na_2S_2O_3$	24 hours
Coliform, total - Wastewater	P,G	4°C, 0.008% $Na_2S_2O_3$	6 hours
Coliform, total - Groundwater	P,G	4°C, 0.008% $Na_2S_2O_3$	24 hours
Organics			
Base/Neutral and Acid Extractables - Water	G, teflon-lined cap	4°C	7 days for extraction 40 ays after extraction for nalysis
Base/Neutral and Acid Extractables - Solid and Waste	G, teflon-lined cap	4°C	14 days for extraction 40 ys after extraction for alysis
Base/Neutral and Acid Extractables -	G, teflon-lined cap	None	7 days for extraction 40

Concentrated Waste			days after extraction for analysis
BTEX - Solid and sludge	G, teflon-lined septum	4°C	14 days
BTEX - Water	G, teflon-lined septum	4°C, 0.008% $Na_2S_2O_3$, zero headspace	14 days
Chlorinated Herbicides - Water	G, teflon- lined cap	4°C	7 days for extraction 40 days after extraction for analysis
Chlorinated Herbicides - Solid and Waste	G, teflon-lined cap	4°C	14 days for extraction 40 days after extraction
Volatiles - Drinking Water	G, teflon-lined cap	4°C, 0.008% $Na_2S_2O_3$[3], zero headspace	14 days
Volatiles (excluding 2-chloroethylvinylether) - Wastewater	Encore Sampler	4°C, zero head-space, HC1 to pH 2	14 days
Volatiles - Wastewater	G, teflon-lined cap	4°C, 0.008% $Na_2S_2O_3$[3], zero headspace	7 days
Volatiles - Solid and Sludge -	Encore Sampler	4°C	14 days
Volatiles - Concentrated Waste	G, teflon-lined septum	None	14 days
Industrial Solvents	G, teflon-lined septum	4°C	None
Organochlorine Pesticides and PCBs	G, teflon-lined cap	4°C	7 days for extraction 40 days after extraction for analysis
PCBs in Oil	G, teflon-lined cap	None	7 days for extraction 40 days after extraction for analysis
Dioxin	G, teflon-lined cap	4°C	7 days after extraction 40 days after extraction for analysis
Total Petroleum Hydrocarbon	G, teflon-lined septum	4°C	14 days
Coliform, total - Drinking water	P,G	4°C, 0.008% $Na_2S_2O_3$	30 hours

Radiochemistry			
Carbon-14 - Water and Soil	P	4°C	6 months
Gamma Isotopes - Water	P	HNO_3 to pH-2	6 months
Gamma Isotopes - Soil	P	None	6 months
Gross Alpha and Beta - Water	P	HNO_3 to pH-2	6 months
Gross Alpha and Beta - Soil	P	None	6 months
Iodine-129 - Water and Soil	P	None	6 months
Iodine -131 - Water	P	None	6 months
Neptunium - Water	P	HNO_3 to pH-2	6 months
Neptunium - Soil, Vegetation, and Air	P	None	6 months

Filters			
Plutonium - Water	P	HNO_3 to pH-2	6 months
Plutonium - Soil, Vegetation, and Air Filters	P	None	6 months
Thorium - Water	P	HNO_3 to pH-2	6 months
Thorium - Soil, Vegetation, and Air Filters	P	None	6 months
Uranium - Water	P	HNO_3 to pH-2	6 months
Uranium - Soil, Vegetation, and Air Filters	P	None	6 months
Americium - Water	P	HNO_3 to pH-2	6 months
Americium - Soil, Vegetation, and Air Filters	P	None	6 months
Curium - Water	P	HNO_3 to pH-2	6 months
Curium - Soil, Vegetation, and Air Filters	P	None	6 months
Lead-210 - Water	P	HNO_3 to pH-2	6 months
Nickel-59 - Water and Soil	P	None	6 months
Nickel-63 - Water and Soil	P	None	6 months
Phosphorus-32 -Water	P	HNO_3 to pH-2	6 months
Phosphorus-32 -Soil	P	None	6 months
Polonium -Water	P	HNO_3 to pH-2	6 months
Polonium -Soil	P	None	6 months
Promethium-147 -Water	P	HNO_3 to pH-2	6 months
Promethium-147 -Soil	P	None	6 months
Radium-223 - Water	P	None	6 months
Radium-224 - Water	P	None	6 months
Radium-226 - Water	P	HNO_3 to pH-2	6 months
Radium-228 - Water	P	HNO_3 to pH-2	6 months
Radon-222 - Water	40ml volatile bottle	4^oC, Zero headspace	7 days
Radon-222 - Soil	P	4^oC	6 months
Strontium-89/90 -Water	P	HNO_3 to pH-2	6 months
Strontium-89/90 -Soil	P	None	6 months
Technetium-99 -Water	P	HNO_3 to pH-2	6 months
Technetium-99 -Soil	P	None	6 months
Total Alpha Radium -Water	P	HNO_3 to pH-2	6 months
Total Alpha Radium -Soil	P	None	6 months
Total Uranium -Water	P	HNO_3 to pH-2	6 months
Tritium - Water, Soil, Vegetation, and Air Filters	P	4^oC	6 months
Iron 55 -Water	P	HNO_3 to pH-2	6 months
Iron 55 -Soil	P	None	6 months
Total Uranium -Soil	P	None	6 months

[1] P = Polyethylene; G = Glass
[2] Samples should be analyzed as soon as possible after collection. The holding times listed are maximum times that samples may be held before analysis and be considered valid.
[3] Used only in the presence of residual chlorine.
[4] Maximum holding time is 24 hours when sulfide is present. All samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If present, remove by adding cadmium nitrate powder until a negative spot test is obtained. Filter sample and add NaOH to pH12.

General Engineering Laboratories
Radiochemistry SOPs Volume I

VOLUME 1
TABLE OF CONTENTS

VOLUME 1 UNCONTROLLED DOCUMENT

TABLE OF CONTENTS

1.0 Analyst and Analytical Methods Validation

Process Owner:	Signature:	Wilbur B. Sigmon, III
Technical Review by:	Signature:	James B. Westmoreland
Quality Review by:	Signature:	Robert L. Pullano
Approved & Authorized by:	Signature:	Heyward H. Coleman

SET

2.0 Method Purpose and Applicability

2.1) To describe the manner in which analytical methods and analysts are validated.

3.0 Discussion

3.1) Prior to conducting and reporting radioanalytical measurements on actual client samples, the proposed method must be validated. The intent is to provide confidence to the analyst, EPI laboratory management and EPI's clients that the method can produce valid data. Methods are validated by achieving acceptable recoveries of target analytes from Laboratory Control Samples (LCSs) or Performance Evaluation (PE) samples. Also, yields of isotopic tracers are evaluated to ensure satisfactory recoveries.

3.2) Analyst proposing to use the method must be trained and certified prior to analyzing actual samples. Analyst validation is the approval process used to document that an analyst can successfully conduct the procedure. The intent is to provide assurance to the analyst and to his or her manager that the analyst can utilize the method to generate valid data on actual samples.

4.0 Procedures

4.1) New Methods. Prior to using a method to conduct actual sample analyses, the method performance must be documented. Method performance is defined as the precision, accuracy and sensitivity (or Minimum Detectable Activity) achievable by the method as it is conducted according to its Standard Operating Procedure.

4.1.1) Method Precision - Replicate analyses are conducted on standard solutions containing the analyte of interest. Typically, seven measurements are made during a 120 hour period, or less. The precision is calculated in terms of standard deviation (SD) or relative standard deviation (RSD). Calculated values are compared with the method requirements as described in the method SOP. Client specifications may also be used. The analysis is repeated until the method precision specification is achieved. If acceptable precision cannot be achieved, a root-cause problem analysis is conducted on the analytical process to identify and resolve problem(s).

4.1.2) Method accuracy - The proposed method is used to analyze

replicate LCSs, Standard Reference Materials (SRMs), PEs or spiked samples. The source material should be independent of the calibration source material. The concentration of the target analyte(s) should approximate the mid-point of the method calibration curve. The recovery of the target analyte(s) are calculated and compared with the requirements specified in the method SOP. The analysis steps used to determine precision and accuracy may be combined to save time. If acceptable accuracy cannot be achieved, the analysis method is repeated or combined with root-cause analysis to complete problem resolution.

4.1.3) Method Sensitivity or Minimum Detectable Activity (MDA). In order to establish realistic reporting limits and to determine if client requirements can be met, the MDA is determined. Replicate method background measurements, typically seven, are made using the proposed method. The average background cpm is inserted into the equation proposed by L. Currie for MDA.

$$MDA = \frac{2.71 + 4.66\sqrt{cpm_{bkg} * Tc}}{2.22 * E * Ab * g * T * Tc}$$

Where: cpm_{bkg}= average detector background counts per minute
Tc=sample counting time
Ab=isotopic abundance
g= mass of sample (typically grams or liters)
T=tracer recovery
E=detector efficiency

If the required MDA is not achieved, the process is evaluated to determine what changes in the method is required.

4.1.4) Method Background: Instrument Versus Method - For each method, ten reagent blanks shall be run. The blanks will then be counted to determine if they contribute to the method background above the instrument background. If the method blanks do not contribute counts above the instrument background then the instrument background shall be used for the method background. If the method reagent blanks do contribute counts above the instrument background then the average of the ten blanks shall be used for the method background. If the reagent blanks are used for the method background then ten new reagent blanks shall be prepared and run in conjunction with the six month calibration to determine a new method background, or the method "reagent" blanks shall be run under the detector as the weekly background.

4.1.5) Chemical yield - Replicate analyses are conducted using an appropriate isotopic tracer. The yield of the analyses are determined using the method described in the SOP. The calculated values are compared with acceptable yields established for the method. Corrective action for the process is conducted if acceptable yields cannot be achieved.

4.1.6) Records - The Radiochemistry Laboratory group leader will maintain records for method precision, accuracy, MDL and yield.

4.2) Methods in Use

4.2.1) Method performance characteristic should be determined at least annually, for methods currently in use.

4.3) Analyst Validation

4.3.1) The analyst must be validated for each method prior to conducting analysis of actual client samples. Please refer to EPI D-001 for specific analyst training requirements.

4.3.2) The analyst validation is conducted by replicate measurements of suitable materials. PE samples, LCSs, SRMs or other suitable material may be utilized for this purpose. The activity of validating the method may be combined with that of validating the analyst.

4.3.3) The relative percent difference (RFD) between two measured samples is calculated. The recovery of samples spiked with the analyte of interest is calculated. The recovery of the analyte from an unknown sample is conducted.

4.3.4) In order for the analyst to be considered qualified to conduct the analysis, the method specifications for precision, accuracy and MDL must be achieved. Until the analyst achieves acceptable method performance, he or she cannot conduct the analysis on actual client samples.

4.3.5) Records of acceptable performance are maintained by the group leader for each analyst.

5.0 Safety, Health and Environmental Hazards

5.1) The user of this SOP is directed to the appropriate analytical Sop's for

information on safety and health.

6.0 Records Management

6.1) Records for analyst and method validation will be maintained by the group leader, Radiochemistry Laboratory.

7.0 References

7.1) L.A. Currie, "Limits for Qualitative Detection and Quantitative Determination" Anal. Chem. 40, No 3 588-693 (1968).

STANDARD OPERATING PROCEDURE

UNCONTROLLED DOCUMENT

FOR

DATA REVIEW, VALIDATION AND DATA PACKAGE ASSEMBLY

(GL-EPI-D-003 Revision 6)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Kerry Campos 7/30/99	Process Owner (sign & date)	Kerry Campos	(print name)
Erica L. Elias 7/30/99	Technical Review (sign & date)	Erica L. Elias	(print name)
Dani D Bost 8/3/99	Quality Review (sign & date)	Dani G. Bost	(print name)
James B. Westmoreland 8/2/99	Approval and Authorization (sign & date)	James B. Westmoreland	(print name)

SET ID.

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR DATA REVIEW, VALIDATION AND DATA PACKAGE ASSEMBLY.

2.0 PURPOSE

This operating procedure is specific to data review and validation of analytical results and assembling data packages.

3.0 DISCUSSION

3.1 Data review and validation can be broken down into two levels of review for laboratory analysis. The first level of review (Data Review) is conducted by the analyst. The second review (Data Review, Validation and Technical Review) is conducted by the Data Validator or Report Specialist.

3.2 Data packages are assembled as required by the client. A checklist is maintained for each client that requires a data package. The package is then reviewed for completeness by the Report Specialist.

4.0 DEFINITIONS

4.1 Batch: A group of samples which behave similarly with respect to the procedures being employed and which are processed as a unit.

4.2 Duplicate: Intralaboratory split sample which is used to document the precision of a method in a given sample matrix.

4.3 Matrix: A component or substrate (e.g. surface water, drinking water, soil) which contains the analyte of interest.

4.4 Method Blank: A matrix to which all reagents are added in the same volumes or proportions as used in sample processing.

4.5 Review: A documented assessment of an item, activity, or work performed that is conducted by one or more individuals who are regarded as technically expert by the scientific/technical community in the subject area being assessed.

4.6 Validation: An activity that demonstrates that a process, item, data set, or service satisfies the requirements defined by the user.

4.7 Que Sheets: Que Sheets contain information about the samples run in a batch (e.g., sample volumes, sample weights, detector identification numbers, spiking standards, etc).

4.8 Batch List: Batch Lists contain the samples in the batch and their quality control samples, their descriptions, tests analyzed, analyst's name, due dates, and any important comments on the sample.

4.9 LIMS: Laboratory Information Management System

5.0 PROCEDURES

5.1 LEVEL I - Analyst Review:

The analyst conducting the procedure reviews 100% of the raw data and QC data for each test once the analyst has completed his/her analyses and enters the data into the Laboratory Information Management System (LIMS). The analyst reviews the following:

5.1.1 Standardization has been conducted according to the specific standard operating procedure for the analysis.

5.1.2 Analytical blanks that have values less than the reported limits of detection or is less than 5% of the lowest activity.

5.1.3 Duplicate analyses are within 20% Relative Percent Difference (RPD) if greater than five times the Required Detection Limit (RDL) and 100% if less than five times the RDL.

5.1.4 Matrix Spikes (MS) and Laboratory Control Samples (LCS) have recoveries between 75% - 125% of the expected value.

5.1.5 Transcription of the raw data has been entered correctly into LIMS.

5.1.6 Comments have been entered into LIMS for unusual dilutions, non-routine handling of samples, and explanations on non-conformances. Non-conformances that have been attributed to sample matrix problems can only be considered after an evaluation by the Group Leader. The non-conformances must be documented, initialed and dated by the analyst and the Group Leader.

5.1.7 The data has been initialed and dated.

5.1.8 The required detection limit has been met.

NOTE: There may be additional requirements or different limits than stated above depending on the client's needs. The analyst

can check for special client requirements on the special client requirements page that prints out with their que sheet. The special requirements page database is kept current by the Data Validator.

NOTE: If any of the following criteria have not been met, the data will be considered out of control and corrective action must be taken before the data can be sent to the next level of review.

5.2 LEVEL II - Data Review, Validation, and Technical Review:

This review is conducted by the Report Specialist or Data Validator

5.2.1 Standardization was conducted according to the specific standard operating procedure for the analysis.

5.2.2 Analytical blanks have values less than the reported limits of detection or is less than 5% of the lowest activity.

5.2.3 Duplicate analyses are within 20% Relative Percent Difference if greater than five times the RDL or 100% if less than five times the RDL.

5.2.4 Matrix Spikes and Laboratory Control Samples have recoveries between 75% - 125% of the expected value.

5.2.5 Transcription of the raw data has been entered correctly into LIMS.

5.2.6 Comments have been entered into LIMS for unusual dilutions, non-routine handling of samples, explanations on non-conformances, etc. Non-conformances that have been attributed to sample matrix problems can only be considered after an evaluation by the Group Leader. The non-conformances must be documented, initialed and dated by the analyst and the Group Leader.

5.2.7 Contract required detection limits have been met.

5.2.8 The analyst has initialed and dated the analytical data package.

NOTE: There may be additional requirements or different limits than stated above depending on the client's needs. The Data Review Specialist will check for special client requirements on the special client requirements page that is included with the batch. This special client requirements database is kept

current by the Data Validator.

5.2.9 If any of the following criteria have not been met, the data is considered out of control and corrective actions must be taken before the data is approved for completion.

5.2.10 The Data Validator or Report Specialist then initials and dates the data package verifying that the data has met all criteria.

5.3.2 When the Data Validator or Report Specialist changes status to 'DONE', the status of the analytical batch data is automatically updated to QCSR (Quality Control Sample Review). When all the parameters for one specific sample have been updated to this status, a QCSR report is automatically generated by LIMS on the sample for review.

5.3.3 The QCSR that was generated is then reviewed for completeness. Certificates of Analysis are processed and a data package is assembled for release to the client. Data packages and Certificates of Analysis have specific checklists used as part of the review process.

6.0 LIMS RAW DATA CHANGES

6.1 If any changes are made to the LIMS raw data after the results have been reported to a client, a Client Nonconformance must be completed (see Appendix 1).

6.2 The data change form is reviewed for completion by the Data Validator and sent to the Quality Group.

6.3 The client is notified and new Certificates of Analysis, electronic deliverables, and data packages are processed as required.

6.4 A record of the changes will be kept in the Quality Management Systems department.

7.0 DATA PACKAGES

7.1 A data package is generated if the client requests additional information on the analysis of their sample. A data package contains the additional information based on the client's requirements and needs. The following are provided in a typical data package:

7.1.1 Raw data

7.1.2 Que sheet

7.1.3 Batch list

7.1.4 Quality control charts

7.1.5 Method spiking sources

7.1.6 Spectrums containing regions of interest (Alpha Spectroscopy)

7.1.7 Method calibrations

7.1.8 Daily and background instrument checks

7.1.9 Run logs

7.2 A checklist is made for each client's requirements.

7.3 After the data package is assembled, it is reviewed by the Report Specialist for completeness. The Certificates of Analysis are reviewed for technical mass balance (Gross alpha vs actinides, Radium-226 vs Total Radium, etc.). The package is then numbered and a copy is made for our records.

7.4 Some data packages are scanned and then printed out with page numbers. A copy is kept on file.

8.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

No potential safety, health or environmental hazards exist for the activity described in this SOP.

9.0. RECORDS MANAGEMENT

Records generated as a result of this SOP and associated management activities are as follows:

9.1 . GL-EPI-D-003

9.1.1 Document Description: GL-EPI-D-003, STANDARD OPERATING PROCEDURE FOR DATA REVIEW, VALIDATION, AND DATA PACKAGE ASSEMBLY.

9.1.2 Form Number: Not Applicable

9.1.3 Records Status/Vital Records: Quality Lifetime Record

9.1.4 Filing Location/Distribution: GEL central Document Control files, GL-EPI-D-003; holders of designated controlled copies of Radiochemistry SOP Manual Volume 1

9.1.5 Disposition Authority: Radiochemistry Laboratory Manager

9.1.6 Retention Period: Radiochemistry SOP Manual holders destroy controlled photocopies of this SOP when superseded. The original document, together with all changes, revisions and DIRRs must be retained indefinitely by GEL Document Control.

10.0 REFERENCES

10.1 ANSI/ASQC-E4-1994, American National Standard: Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, American Society for Quality Control (ASQC), Energy Division, Environmental Waste Management Committee, Milwaukee, 1994.

APPENDIX 1

GENERAL ENGINEERING LABORATORIES, INC.
Client NCR Form- XXX
(Rev. 9\98)

NCR Report No.:_____

CLIENT NONCONFORMANCE/CORRECTIVE ACTION REPORT

Workdef:

Problems/Observations/Findings/Recommendations: Date:

Corrective Action/Response: (Date implemented, completion date, etc.)

Quality Review: _____ **Date:** _____ **Mgmt. Review:** _____ **Date:** _____

NC EVENT TYPES: (Circle)

1. HOLDING TIME 2. DETECTION LIMITS 3. QC FAILURES 4. DATA MISSING 5. PACKAGE ERROR

6. T.O.TIME 7. LOG-IN ERROR 8. EDD NOT DELIVERED 9. DATA ERROR 10. EDD ERROR 11. OTHER

APPENDIX 2

Category: Gamma Spectroscopy

Metric	Acceptance Criteria	Corrective Action*
Method Blank	<CRDL or < 2 X decision level	Recount to confirm out of control condition. If the problem persists, clean the detector and shield and redo the method blank. If samples have positive detections for the isotope failing to meet blank requirement, redo the batch. Qualify the data if required.
Replicate	RPD 0-20% RPD. If result is >5 X the CRDL.	Recount to confirm out of control condition. Redo replicates if the problem persists. Determine and correct the problem. Qualify the data if required.
LCS	75-125% Recovery	Recount to confirm out of control condition. If problem persists, redo the LCS. If the second count fails recalibrate the detector. Qualify the data if required.
MS	N/A	N/A
Gain	+ 3 KeV	Recount to confirm out of control condition. If problem persists, adjust the instrument gain as required and confirm the energy calibration. Qualify the data if required
FWHM	< 3 KeV	Recount to confirm out of control condition. If problem persists, remove the instrument from service and perform a thermal cycle. If the problem persists send the detector for repair. Qualify the data if required

Category: Gas Flow and Liquid Scintillation Counting

Metric	Acceptance Criteria	Corrective Action*
Method Blank	<CRDL or < 2 X decision level	Recount to confirm out of control condition, redo the method blank if the problem persists. If samples have positive detections for the parameter failing to meet the blank requirement redo the batch. Qualify the data if required.
Replicate	RPD 0-20% RPD. If result is >5 X the CRDL.	Recount to confirm out of control condition. Redo replicates if the problem persists. Determine and correct the problem. Qualify the data if required
LCS	75-125% Recovery	Recount to confirm out of control condition. If problem persists and no internal tracer is used to verify sample recovery, repeat the batch. If MS passes the LCS may be rerun instead of the entire batch. Qualify the data if required
MS	75-125% or not applicable if sample concentration is >10 X spike concentration.	Recount to confirm out of control condition. If problem persists and the LCS is also failing redo the batch. Qualify the data if required.
Internal Tracer Yield	25-125% Recovery	Recount to confirm out of control condition. If problem persists repeat sample to achieve acceptablerecovery. Qualify the data if required.

Category: Alpha Spectroscopy

Metric	Acceptance Criteria	Corrective Action*
Method Blank	<CRDL or < 2 X decision level	Recount to confirm out of control condition, redo method blank if the problem persists. If samples have a positive detection for the isotope failing to meet the blank requirement, re-aliquot and repeat the batch. Check and clean the detector and chamber if required. Qualify the data if required.
Replicate	RPD 0-20% RPD. If result is >5 X the CRDL.	Recount to confirm out of control, redo replicates if the problem persists. Qualify the data if required.
LCS	75-125% Recovery	Recount to confirm condition if problem persists, redo LCS and redo batch. If MS passes the LCS may be rerun instead of the entire batch. Qualify the data if required.
MS	75-125% or not applicable if sample concentration is >10 X spike concentration	Recount to confirm if problem persists, redo MS, replicate and sample. Qualify the data if required.
Internal Tracer Yield	15-125% Recovery	Recount to confirm if problem persists, repeat sample to achieve acceptable recovery. Qualify the data if required
FWHM	< 50 KeV on the daily pulser check	Recount to confirm, if problem persists remove the detector from service and repair or replace the detector. Qualify the data if required.
Gain	+ 50 KeV on the daily pulser check.	Recount to confirm out of control condition. If problem persists, adjust the instrument gain as required and confirm the energy calibration. Qualify the data if required.

* Any corrective action that continues to fail after the second attempt must be brought to the attention of the supervisor before proceeding

1.0 Standard Operating Supply Maintenance Procedure

Process Owner:	Signature:	Brian T. Morris
Technical Review by:	Signature:	James B. Westmoreland
Quality Review by:	Signature:	Robert L. Pullano
Approved & Authorized:	Signature:	Heyward H. Coleman

SET 1.1

2.0 Method Objective and Applicability

2.1) This procedure is provided as guidance to ensure goods and services employed by Environmental Physics, Inc. (EPI) are kept in ready supply and are of acceptable quality for their intended use.

2.2) The main focus of this document is to provide guidance for the procurement, inspection, and documentation of goods and services employed by Environmental Physics, Inc.

3.0 Discussion

3.1) EPI is committed to assuring quality and continuous improvement of services to all of it's clients. EPI realizes that the quality of the materials and services we use directly affects the quality of our products. In order to optimize the quality of our products, we must closely monitor the quality of those products we use. The EPI Supply Manager provides the one point of contact for supply procurement and receipt.

4.0 Procedures

4.1) General

4.1.1) Chemicals used as reagents for analytical methods shall be of ACS grade or better. If ACS grade of the chemical is not available, the chemical must be tested for purity upon receipt.

4.1.2) Materials which are used as mounting or holding devices for radioactivity counting should be the same composition and geometry as those which were originally used for the method calibration. If the material changes in either respect, a new calibration may be required.

4.1.3) Durable goods used as instrumentation or sample preparation equipment must be ordered with strict attention to specifications and tolerances. Analytical errors due to poor performance of this type of equipment cannot be tolerated due to the abundance of high quality equipment and numerous vendors.

4.1.4) Consumables and small accessory equipment which do not directly affect EPI product quality can be ordered more liberally, with greater emphasis on lower cost.

4.1.5) This procedure also allows for EPI to perform audits of the vendors which supply goods or services to EPI, as necessary.

4.2) Supply Manager Responsibilities

4.2.1) Route the appropriate procurement documentation through the approval chain then to the purchasing agent for General Engineering Laboratories (GEL). The parent company, GEL, provides the associated accounting , billing and delivery of most items to EPI.

4.2.2) Maintain an adequate inventory of consumables.

4.2.3) Maintain a database of the supply program. The database is used for cost analyses, trend analyses, comparisons, and overall tracking of the entire procurement process.

4.3) Procurement Approval

4.3.1) Single vendor purchases under $500 require the lab manager's approval.

4.3.2) Single vendor purchases over $500 require both the lab manager's and president's approval.

4.4) Procurement documentation

4.4.1) The end users, analysts, are generally the first to notice when a certain item is needed or becoming low in inventory. To provide the most rapid means of responding to this need, a "Supplies in Need" form (attachment 1) is posted in the laboratory.

4.4.2) The "Supplies in Need" form is routinely checked by the Supply Manager. The Supply Manager will enter the items listed on the form into the Supply Database.

4.4.3) From the supply database, a "Requisition Form" (attachment 2) is generated and properly submitted.

4.4.4) The original approved requisition form is sent to the GEL Purchasing Agent and a copy is maintained for EPI records.

4.4.5) Once the items are received , they are inspected as specified in section 4.6, and the supply database is updated.

4.5) The Supply Database contains the following information:

4.5.1) Item description, catalog number, units, cost/unit, quantity ordered, supplier information, date ordered and date received.

4.5.2) Data is maintained in two foundational tables.

4.5.2.1) The main table: an expanding list of every item ever been ordered. This data is used for subsequent ordering. The fields must be periodically cross-checked with the vendor catalog to ensure that changes are updated.

4.5.2.2) The ordered table: an expanding list of every item ever ordered. This data is used for cost and trend analysis.

4.5.3) Due to the historical importance of the data contained in the foundational tables, it should be periodically archived to avoid data loss in the event of file corruption.

4.6) Inspection of Goods and Services Supplied to EPI:

4.6.1) The Supply Manager shall inspect all goods as soon as possible after delivery.

4.6.2) The inspection should include the following checks:

4.6.2.1) Broken, damaged or non-functional problems.

4.6.2.2) Comparison of the material which was ordered with that delivered, including model number, part number, quantity, and size.

4.6.2.3) Shelf life, storage requirements, and MSDS specifications.

4.6.2.4) Assembly manuals, technical data, parts lists, service numbers, handling, operational guides, and templates.

4.6.3) If the inspection is satisfactory, the item can be placed in use. Reagents and standards are then logged in and labeled. Reagents are given a shelf life of two years, from the time of receipt unless more restrictive limits are stated on the container or MSDS.

4.6.4) If the inspection is unsatisfactory, a nonconformance will be issued for the items or services. Materials nonconforming shall be tagged, segregated and corrective action initiated. Refer to Standard Operation Procedures No. GL-QS-E-004, Nonconformance Identification Control, Documentation, Reporting and Disposition.

5.0 References

5.1) General Engineering Laboratories (GEL) "Standard Operating Procedure for the Receipt and Inspection of Materiel and Services" GL-RC-E-001 Rev. #0 DIRR #0 Company Wide Standard Operating Procedures Manual 2/15/93.

Attachment 1

Environmental Physics, Inc.

Supplies in need:

(Please, include description and your initials for odd items.)

1. _____
2. _____
3. _____
4. _____
5. _____
6. _____
7. _____
8. _____
9. _____
10. _____
11. _____
12. _____
13. _____
14. _____
15. _____

Attachment 2

REQUISITION FORM

Environmental Physics Inc. Date: 10/27/93

Submitted By: BTM

SUPPLIER: BAXTER

Urgency: Regular

CATALOG #	ITEM	PRICE / UNIT	UNITS	QTY	EXTENDED PRICE
B2660-250	BEAKER, 250ML LOW FORM	$96.48	CS, 48	1	$96.48
B2660-400	BEAKER, 400ML LOW FORM	$114.72	CS, 48	1	$114.72
2704-7X6	NITRIC ACID, HNO3	$122.70	CS, 6	3	$368.10

PO#_____ TOTAL: $579.30

Authorized Signature:_____

STANDARD OPERATING PROCEDURE

FOR

RADIATION SURVEY PROCEDURES

Process Owner(s):	Wendy K. Foltz, Technician II
Technical Review by:	James B. Westmoreland, EPI Laboratory Manager
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	Robert L. Pullano, Quality Systems Manager
	Heyward H. Coleman, EPI President

1.0 STANDARD OPERATING PROCEDURE FOR RADIATION SURVEY PROCEDURES

2.0 PURPOSE

This operating procedure is designed to outline radiation survey procedures.

3.0 DISCUSSION

Surveys are required to verify the presence or absense of radioactive contamination. Radiation surveys are divided into 4 categories:

3.1 Geiger Mueller (GM) contamination survey

3.2 Geiger Mueller (GM) dose rate survey

3.3 Removable contamination (smear) survey

3.4 Exit monitoring survey.

4.0 DEFINITIONS

4.1 Radioactive material. According to SCDHEC, "There are no established levels at which a sample cam be classified as radioactive or non-radioactive." See EPI SOP "Inventory and Tracking of Radioactive Material" (GL-EPI-E-S003), Appendix 5 for the specific information notice dated June 6, 1994. Therefore, any material received under the authority of our radioactive materials license will be considered "radioactive."

4.2 Geiger-Mueller (GM) survey meter. A portable instrument with a radiation detection probe. An Eberline E-120 instrument with a HP-260 probe (or equivalent) is used for contamination surveys. An Eberline E-120 instrument with a HP-270 (or equivalent) probe is used for dose rate surveys. A Bicron "Frisk Tech" meter with a "PGM" pancake probe is used for exit monitoring surveys.

4.3 Radiation Safety Officer (RSO). Primary individual responsible for the safe use and handling of radioactive material at the facility.

4.4 Radiation control area (RCA). Area designated by the RSO where upon leaving, any individual must monitor to check for radioactive contamination.

5.0 PROCEDURES

5.1 Geiger Mueller (GM) contamination survey

5.1.1 Obtain the E-120 survey meter with HP-260 probe.

5.1.2 Divert the switch to the times 0.1 setting and slowly (1-2 inches per second) pass the meter within 0.25 inch of the area to be surveyed.

5.1.3 Any readings above background should be verified. If elevated readings persist the area will be decontaminated and rechecked as outlined in EPI SOP "Radiation Related Emergencies" (GL-EPI-E-S002).

5.2 Geiger Mueller (GM) dose rate survey

5.2.1 Obtain the E-120 survey meter with HP-270 probe.

5.2.2 Divert the switch to the times 0.1 setting and slowly (1-2 inches per second) pass the meter within 0.25 inch of the area to be surveyed.

5.2.3 Record the observed dose rate in milliroentgen per hour on the applicable survey form.

5.3 Removeable Contamination Survey

5.3.1 Obtain an adequate number of Rad-Wipe Smears from the laboratory.

5.3.2 Label the smear with the current date and time, the sample or area being monitored, and the initials of the analyst performing the survey.

5.3.3 Open the smear so the absorbent material will come in contact with the area to be monitored.

5.3.4 Smear the area in a single "S" pattern for a length of 18 inches. This will approximate 100 square centimeters.

5.3.5 Count the smear for 2 minutes in a gas flow proportional counter at the alpha and beta operating voltage.

5.3.6 Calculate the dpm per 100 square centimeters by the following equations:

alpha dpm/100 cm^2= observed cpm / 0.2

beta dpm/100 cm^2= observed cpm / 0.4

5.3.7 The smear area is considered free of contamination if the alpha dpm is less than 20 dpm/100cm^2 and the beta dpm is less than 100 dpm/100 cm^2.

5.4. Attach the labeled smear cover to the raw data from the count. Initial and date the raw data and submit to the RSO or designee for review and archival.

5.5 **Exit monitoring survey**

5.5.1 The Frisk Tech survey meter with pancake probe should be located at the point of exit monitoring.

5.5.2 Divert the switch to the times 1 setting and slowly (1-2 inches per second) pass the meter within 0.25 inch of the hands and feet.

5.5.3 Any readings above 130 cpm should be verified. If elevated readings persist, do not leave the area, contact the RSO or designee. Record the elevated reading in the instrument log with initials and date. Clean the contaminated area as outlined in EPI SOP " Radiation Related Emergencies" (GL-EPI-E-S002).

5.6 **Monthly Report**

5.6.1 Removable contamination and general area radiation surveys are performed throughout the laboratory once a month to ensure that no contamination problems exist.

5.6.2 Obtain maps of the radiochemistry laboratory, the inner radiation storage cooler, the outer radiation storage cooler, the soil preparation room, the receiving area, and the radioactive waste and sample storage area.

5.6.3 Obtain Rad-Wipe Smears from the laboratory. At least 105 smears will be needed.

5.6.4 Label the maps with the spots that will be swiped. At least 70 swipes should be taken in the laboratory, 5 in the soil preparation room, 5 in each of the coolers and the receiving room, and 15 in the radioactive waste and sample storage area. (See Appendix 1).

5.6.4.1 The smears taken in the laboratory should include all hoods and sinks, the centrifuge and balance areas, the canopy vent, the perchloric hood, the muffle furnace, the drying oven, and representative samples of the counters and floor.

5.6.4.2 The smears taken in the soil preparation room should include the sink, an oven, the balance, and representative samples of the counter and the floor.

5.6.4.3 The smears taken in the inner and outer radiation storage coolers should include representative samples of the shelves and the floor.

5.6.4.4 The smears taken in receiving should include the hood, the center table, the desk, and representative samples of the floor.

5.6.4.5 The smears taken in the radioactive waste and sample storage area should include the compactor, the desks, and representative samples of the shelves, the counters, and the floor.

5.6.4.6 In addition to the specified areas, any area that is suspected of being contaminated should be swiped.

5.6.5 Label the smears with an identifying number corresponding to the number on the maps. The smears taken in the laboratory and the soil preparation area are labeled EPI-#. All other smears are labeled GEL-#. In addition to the identification number, the date, time, room, location, and initials should be filled in. (See Appendix 2).

5.6.6 Perform the smears in accordance with the procedure above.

5.6.7 In addition to the smears, a general area radiation survey should be performed in the radioactive waste and sample storage areas in accordance with the standard operating procedure above.

5.6.8 Attach the raw data to the maps. The raw data and maps from the radiation laboratory and the soil preparation room are attached together and labeled as EPI, and the remaining raw data and maps are attached togehter and labeled as GEL.

5.6.9 The completed paperwork should then be turned into the RSO or designee for review.

6.0 QUALITY CONTROL

6.1 Each day the instrument is verified with the following performance checks:

6.1.1 Battery check. Divert the switch to the battery check setting to ensure the batteries are charged. Replace the batteries when the reading falls below the acceptable range as bracketed on the face of the meter.

6.1.2 Source check. The meter should be source checked on a daily basis prior to use.

6.1.2.1 The Frisk Tech survey meter: Divert the switch to the times 10 setting and pass the Cs-137 source on the probe. The detector should respond to the gamma field by reading 5000 cpm or greater, and setting off the alarm.

6.1.2.2 The Eberline E-120 meter with an HP-260 (or equivalent) probe: Divert the switch to the times 1 setting and place the Cs-137 source on the probe. The detector should respond to the gamma field by reading 5000 cpm or greater.

6.1.2.3 The Eberline E-120 meter with an HP-270 (or equivalent) probe: (The source check should be performed with a closed window.) Divert the switch to the times 1 setting and place the Cs-137 source on the probe. The detector should respond to the gamma field by reading between 1 and 3 mR/hr.

6.1.3 After performing the battery and source checks, complete the appropriate logbook.

6.1.4 If a detector does not respond to the source correctly, notify the RSO or designee and discontinue using the meter.

6.2 Calibration. The instrument will be calibrated on a 12 month frequency by returning to Eberline Instrument Company or an acceptable alternate.

7.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

The following of this SOP should pose no threat to safety, health or the environment.

8.0 RECORDS MANAGEMENT

8.1 The Radiation Safety Officer or designee will maintain records of radioactive surveys.

8.2 The survey meter will be sent to a qualified center for calibration such as Eberline. The calibration will be performed on a 12 month frequency, and calibration records will be maintained by the RSO or designee.

9.0 REFERENCES

9.1 CRC Handbook of Laboratory Safety 2nd Ed. CRC Press.

9.2 South Carolina DHEC Radioactive Materials Regulation 61-63 Title A.

9.3 Technical Manual, Model: FRISK-TECH Ratemeter/Monitor with alarm.

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APPENDIX 1

DIAGRAM OF MAIN RADIOACHEMISTRY LABORATORY

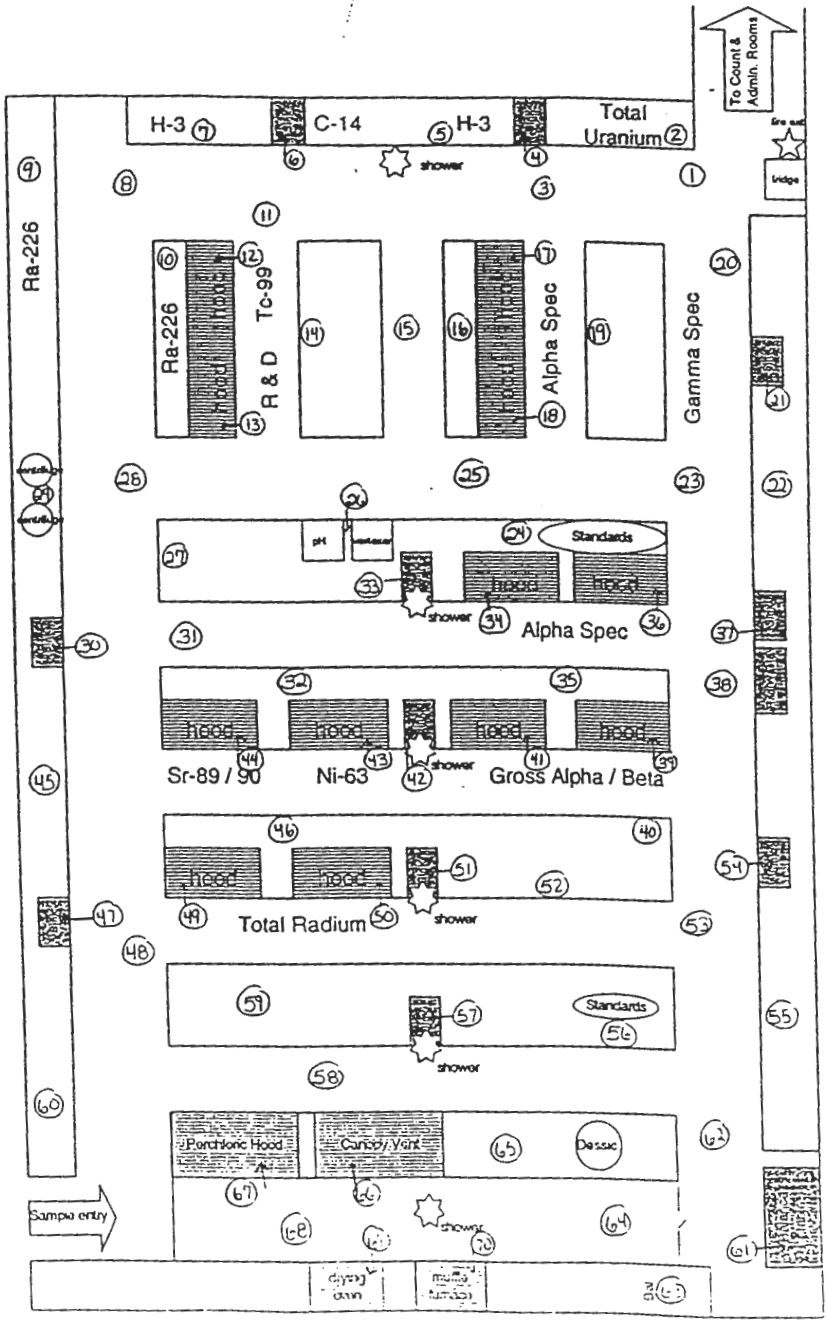


DIAGRAM OF SOIL PREPARATION ROOM

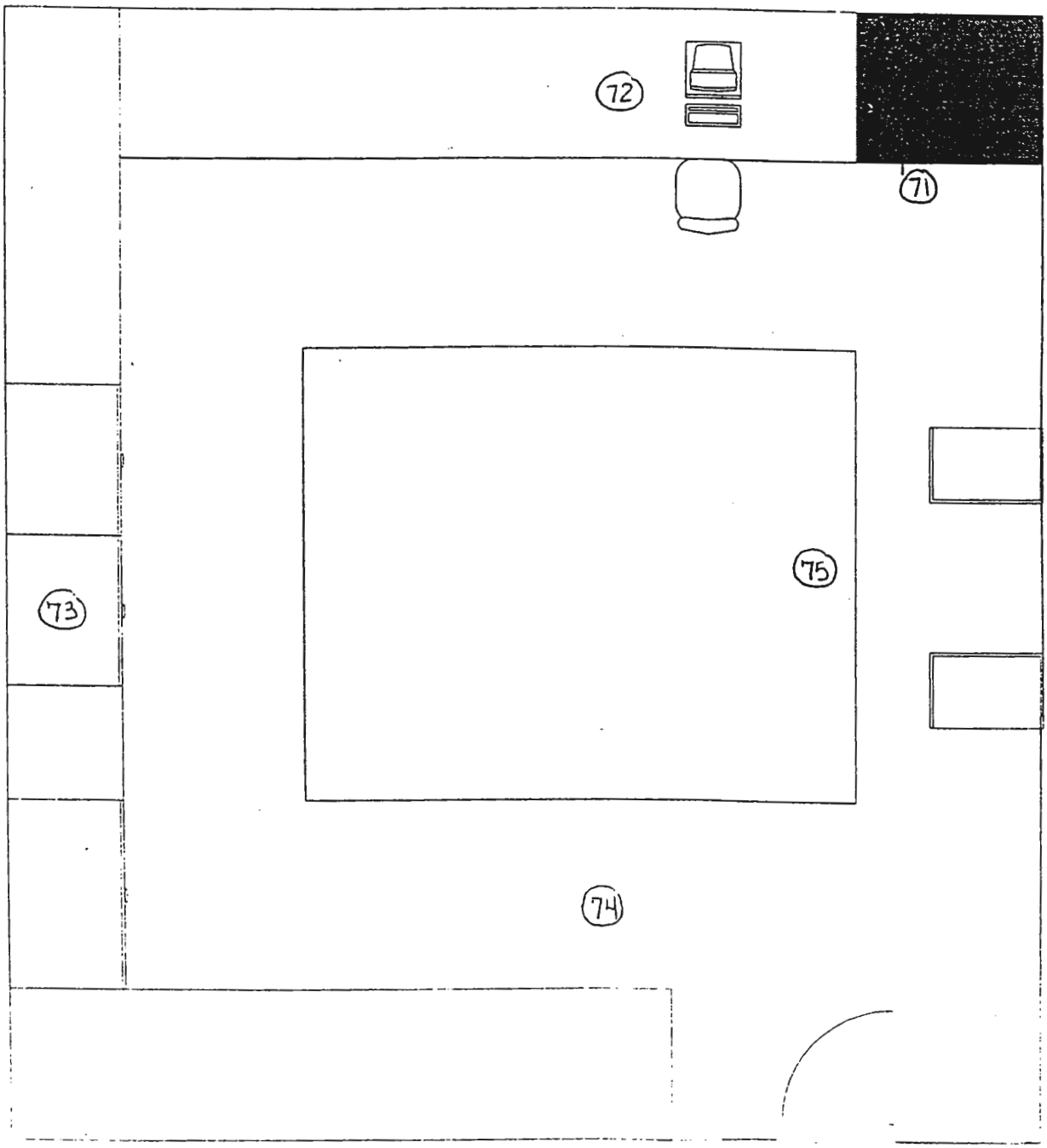


DIAGRAM OF INNER OR OUTER RADIATION STORAGE COOLER

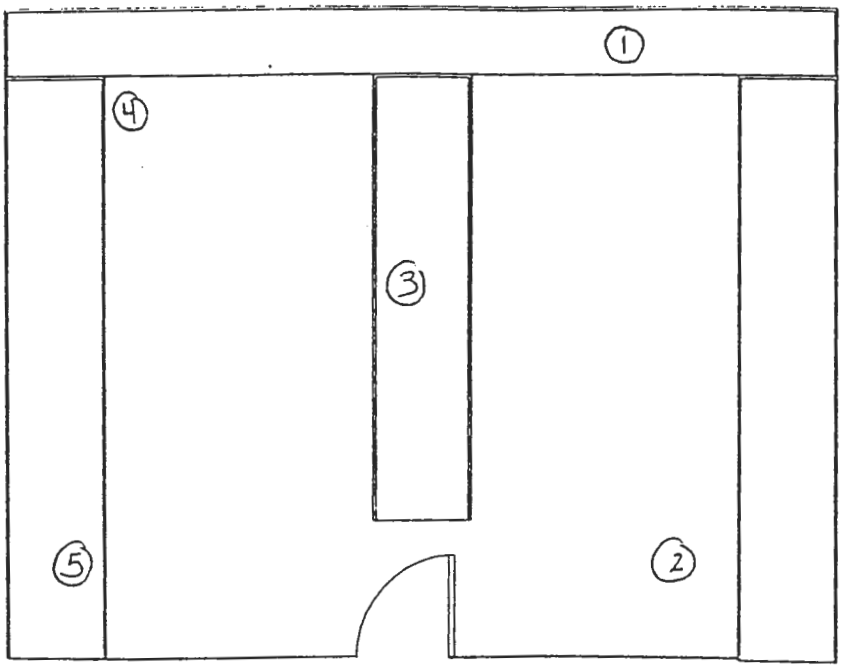


DIAGRAM OF SAMPLE RECEIVING AREA

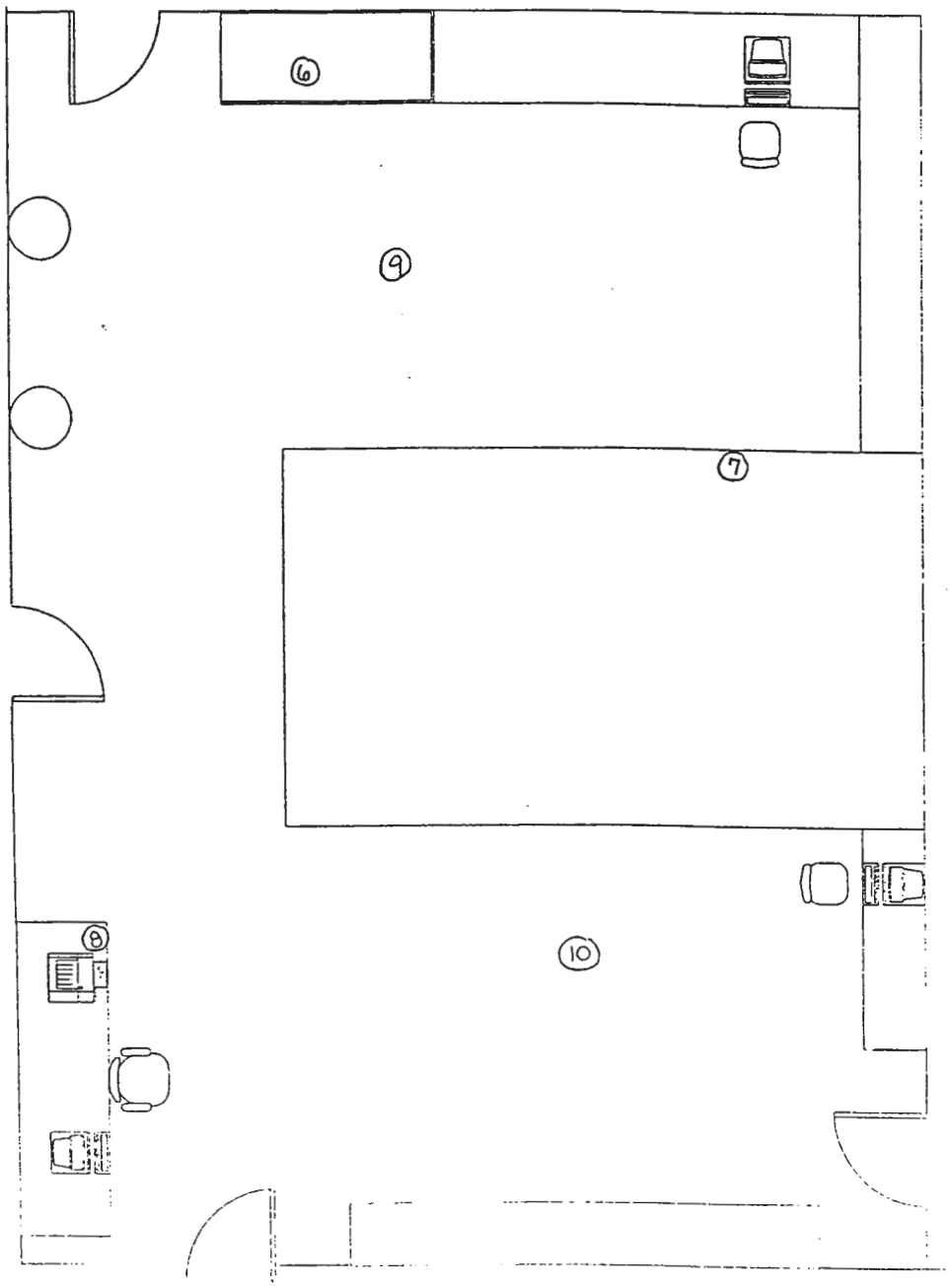
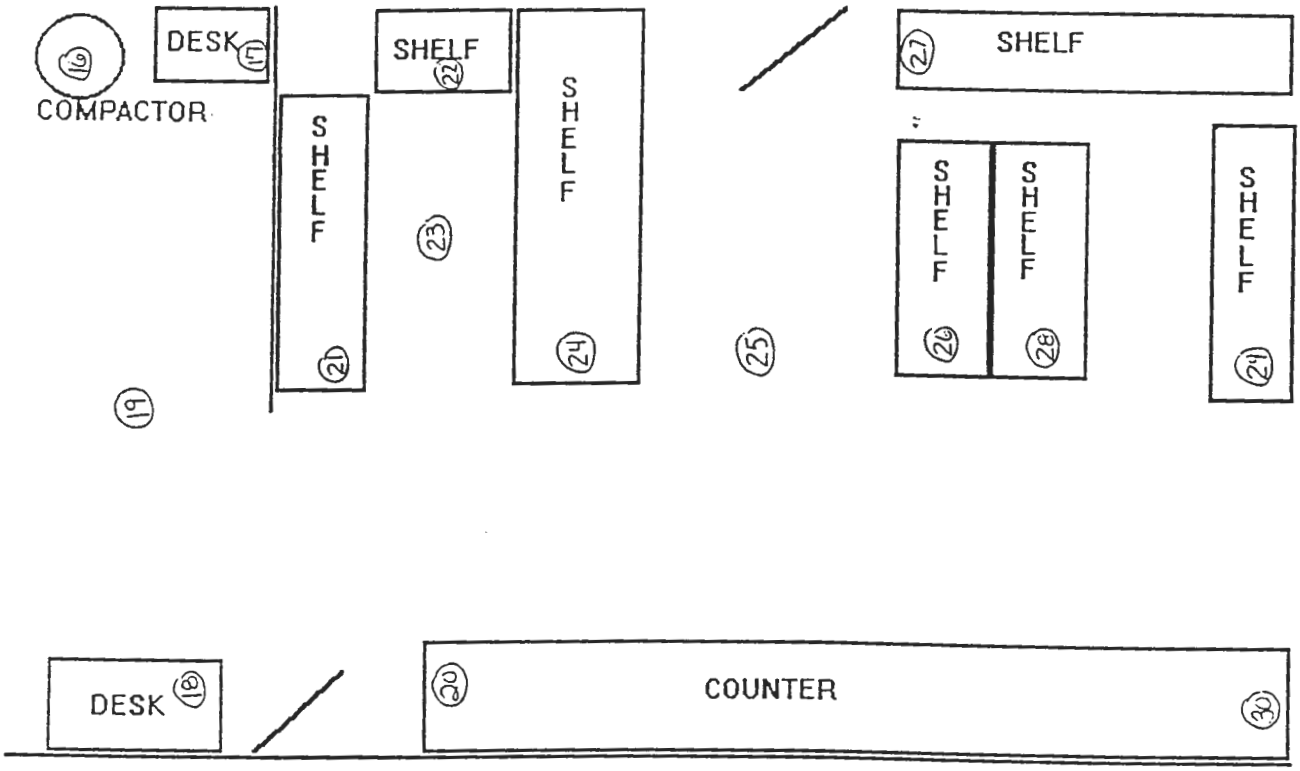


DIAGRAM OF RADIOACTIVE WASTE AND SAMPLE STORAGE AREA



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APPENDIX 2

RAD-WIPE SMEAR LABEL

RAD-WIPE SMEAR #_____

DATE:_____ TIME:_____

SAMPLE I.D.:_____

_____

LOCATION:_____

TAKEN BY:_____

_____ALPHA-_____

COUNT FOR: _____BETA-_____

_____GAMMA-_____

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Radiation Related Emergency Procedures

UNCONTROLLED DOCUMENT

Prepared by:	Signature:	James B. Westmoreland
Technical Review by:	Signature:	Robert J. Pullano
Sr. Management Review by:	Signature:	James T. Chambers
Approved & Authorized by:	Signature:	Heyward H. Coleman

SET

1. Purpose

This operating procedure is designed to outline emergency procedures specific to radiation safety.

2. Discussion

Emergency Procedures are divided into distinct categories:

A) Spill Of Radioactive Material
B) Uptake Of Radioactive Material via inhalation, ingestion, and/or absorption

3. Definitions

3.1) Radioactive material. Any material meeting the following criteria:

3.1.1) Sources used in calibrating methods and/or instruments with activity greater than the exempt quantities listed in RHA 2.24.

3.1.2) Samples received for testing that are labeled or shipped as radioactive material. Samples will be considered radioactive until testing shows the levels are less than the limits for concentrations in air and water above natural background listed in RHA 3.21.

3.1.3) Material that exhibits greater than 0.5 mR/hr on contact.

3.2) Radiation Protection Officer. Primary individual responsible for the safe use and handling of radioactive material at the facility.

4. Procedures

4.1) Spill of Radioactive Material.

4.1.1) The analyst responsible should first notify others in the area that a spill has occurred.

4.1.2) The analyst should take measures to prevent the spread, ingestion or inhalation of the material. This would include removal of lab jacket and/or clothing that is saturated with a radioactive material.

4.1.3) The personnel assisting in cleanup will wear disposable gloves, labcoat, safety glasses and shoe covers.

4.1.4) The spill will be cleaned with appropriate absorbent material and decontamination foam. All potentially contaminated articles will be placed in a plastic bag which will be labelled as radioactive.

4.1.5) The spill area will initially be monitored with a pancake probe GM survey meter. Further monitoring will include smears for gross alpha and beta activity. The area is deemed free of contamination when smear results are less than 100 dpm/100cm2 gross beta and less than 20 dpm/100cm2 gross alpha.

4.2) Ingestion of Radioactive Material.

4.2.1) Any analyst who suspects uptake of a radioactive material should immediately notify the Radiation Protection Officer or designee.

4.2.2) The analyst should attempt to eliminate as much of the contaminant still remaining in the mouth, eyes, or wound. This is generally done with copious amounts of water. In the case of small cuts bleeding should be encouraged to rinse contaminants from the wound.

4.2.3) As quickly as possible the analyst should be taken to a medical facility for care.

5. Safety, Health and Environmental Hazards

The following of this SOP should pose no threat to safety, health or the environment.

6. Records Management

6.1) The Radiation Protection Officer or designee will maintain records of radioactive spills and accidents involving uptake of radioactive materials.

6.2) Accidents involving personnel injury will be maintained by the Human Resource Department.

7. References

7.1) CRC Handbook of Laboratory Safety 2nd Ed. CRC Press.

7.2) South Carolina DHEC Radioactive Materials Regulation 61-63 Title A.

Process Owner(s):	Wendy K. Foltz, Technician III
Technical Review by:	James B. Westmoreland, EPI Laboratory Manager
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	Heyward H. Coleman, President

1.0 STANDARD OPERATING PROCEDURE FOR INVENTORY AND TRACKING OF RADIOACTIVE MATERIAL

2.0 PURPOSE

This SOP, formerly titled "Radioactive Material Procurement Procedures," is intended to define the process of tracking the amounts of radioactive material at the laboratories. The material received may be in the form of samples for analytical testing, sources in instrumentation or standards for calibration. This procedure is followed after standard receiving procedures are completed as outlined in EPI SOP "Radioactive Material Receiving" (S-007).

3.0 DISCUSSION

3.1 Two licenses are currently maintained at the Charleston, SC facility. One is used by Environmental Physics, Inc. (EPI) SC-492, and General Engineering Laboratories (GEL) SC-362 uses the other. Both licenses have similar limits for radioactive material as specified in Table 1 below. The current licenses are divided into categories by the atomic number of the isotope present. Each category has a cumulative activity in millicuries that are permissible in the facility at a given time. An exception to this categorizing is with special nuclear material which is tracked by the grams present for the specific isotope.

SET

3.2 For accurate accountability of radioactive material at the facility it is important to correctly assign the activity to its category. The process of adding radioactive material to the license can be quite simple when the client has a clear knowledge of the isotopes present and the amounts. Some difficulties arise when the client is not aware of the isotopes or concentrations present. This SOP attempts to give guidance regarding the process of assigning activity to the various categories and the process of transferring activity between licenses and eventually off the license.

TABLE 1:

Atomic #	Cumulative Activity
1 (Tritium)	1 curie
2-83	100 millicuries
84-98	10 millicuries
99-100	10 millicuries
Special Nuclear Material	20 g U-235 2 g U-233 2 g Plutonium

4.0 DEFINITIONS

4.1. Radioactive material. According to SCDHEC, "there are no established levels at which a sample can be classified as radioactive or nonradioactive." See Appendix 5 for the specific information notice dated June 6, 1994. Therefore any material received under the authority of our radioactive materials license will be considered "radioactive."

4.2. Radiation Safety Officer (RSO): Individual responsible for the safe use and handling of radioactive material at the facility.

5.0 PROCEDURES

5.1. Material is received as outlined in EPI SOP "Radioactive Material Receiving Procedure" (S-007). Login personnel complete the radioactive material receiving paperwork and attach any radioactive material paperwork. Paperwork and swipes are submitted to EPI for counting.

5.2. If the material is received under the authority of our license as radioactive material, the radioactive material is placed in a secured area with a warning sign indicating the presence of radioactivity. Typically the sign reads "Caution Radioactive Material."

5.3. Once swipe counting is completed, the packet is submitted to the RSO or designee for addition to the license.

5.4. Currently two licenses are maintained for radioactive material at the facility. One is used by Environmental Physics, Inc. (EPI) [SC-492] and General Engineering Laboratories (GEL) [SC-362] uses the other. The RSO or designee will make the decision as to which license inventory the radioactive material will be placed. This decision is based on which company has the primary contract with the sender of the radioactive material.

5.5. The following conditions may exist when paperwork is submitted:

5.5.1. The client has declared the isotopic composition and activity. This case is the simplest, proceed directly to Step 5.6.

5.5.2. The client has declared the sample "Radioactive," but is unsure of the isotopic composition and activity. This case is more troublesome; therefore the following investigation must be completed:

5.5.2.1. If the sample is being submitted for radiological quantification, the paper work is held pending the results of analysis.

5.5.2.2. Once analysis is completed the activities can be assigned to the material balance table. Proceed to Step 5.6.

5.5.2.3. If no radiological testing is requested, the sample is submitted for an activity level screen. The activity level screen is performed using liquid scintillation counting as outlined in EPI SOP "EPI Laboratory Waste Disposal and Emergency Instructions" (S-011).

5.5.2.4. The scintillation screen covers all potentially received radioactive materials and divides them into three energy regions. A low energy region will detect isotopes such as Tritium, a mid energy region will pickup isotopes such as Carbon-14, and a high energy region will detect alpha emitting isotopes and strong beta emitting isotopes such as Strontium-90.

5.5.2.5. Since the activity level screen is a gross screening measurement, there will be times when historical or process knowledge is used to declare the specific isotope. Proceed to Step 5.6.

5.6. The Microsoft Access™ database "epirad_b.mdb" is opened and the macro "RADIOACTIVE RECEIPT STEP 1" is run.

5.7. To facilitate entry into the database, the macro initiates a form which is used to enter key information into the database. The reference number and the total uCi per isotope per sample are needed before using this form. Note: this macro will add activity to the GEL license. If the material should be on the EPI license, it can be cut from the GEL table and pasted into the EPI table after completion of the macro.

5.8. The highest concentration isotope with the longest half-life is chosen for initial entry on the entry form. This will allow the program to decay correct to the most prevalent isotope. After the macro is completed the isotopic information may be adjusted if other isotopes are present. See Appendix 1 for an example of the material balance table.

5.9. The activity is tracked in uCi by atomic number. The atomic numbers of isotopes are available in many reference texts; this information is also available in the database table "NUCLIDES". See Appendix 2 for an example of the NUCLIDES table.

5.10. The structure of the materials balance table is designed so that any entry into the transfer date or transfer ID causes the activity to be removed from the license. The following will cause material to be removed from the Material Balance table:

5.10.1. The isotope received has gone through 10 half-lives

5.10.2. The material has been disposed of according to EPI SOP "EPI Laboratory Waste Disposal and Emergency Instructions" (S-011).

5.10.3. The material has been transferred to another license

5.11. There are times when samples are transferred between the two licenses for analysis. The database must account for this transfer of activity to be in compliance with license conditions. Because each sample is tracked through the facility by bar code label, the LIMS system has a record of each transfer performed on a given sample. The two licenses are adjusted based on this transfer using the macro "Transfer Step 1".

The transfer macro is run to account for internal transfer of radioactive material between the two licenses. A report (Appendix 4) is printed showing the transfers for a given day.

5.12 The total current inventory is calculated using the macro "Grand Total EPI (or GEL)". This macro totals all activity that has no transfer date or ID for each category and prints a summary report that gives the total activity and the percentage of the license that is currently used. See Appendix 3 for an example of the inventory report.

6.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

6.1 The following of this SOP should pose no threat to safety, health or the environment.

7.0 RECORDS MANAGEMENT

7.1 The RSO or designee will maintain the records of total inventory and internal transfers.

7.2 The database is backed up incrementally each night with a weekly complete backup to prevent loss of critical data.

8.0 REFERENCES

8.1 CRC Handbook of Laboratory Safety, 2nd Ed. CRC Press.

8.2 South Carolina DHEC Radioactive Materials Regulation 61-63 Title A.

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Appendix 1.

Materials Balance Table

Reference #	Receive Date	Client Vendor	Source Code Client ID	Lab ID	Radionuclide(s)	H-3 Activity (uCi)
96072	7/25/96	EPI	030297-02	9607478-01.01	H-3/U-238	0.5
96072	7/25/96	EPI	030297-02b	9607478-01.02	H-3/U-238	0.5

Continuation of table

Tc-99m Activity (uCi)	Z=2-83 Activity (uCi)	Z=84-98 Activity (uCi)	Z=99-100 Activity (uCi)	Half Life (yr.)	Reference Date	Volume (mL)	Transfer Date	Transfer ID
0	0	0.02	0	12.28	7/25/96	0		
0	0	0.02	0	12.28	7/25/96	0		

Reference #	Sequential number that is applicable to the entire shipment. For example 96072 denotes that the shipment was the seventy-second in 1996.
Receive date	Calendar date that the sample was received at the laboratory.
Client/Vendor	Normally a four-letter code that is unique for each client.
Source Code/Client ID	Sample ID declared by the client.
Lab ID	Sample number assigned at sample receiving.
Radionuclide(s)	The isotopes declared or determined in order of prevalence.
H-3 (uCi)	The total tritium activity in microcuries assigned to the sample bottle.
Tc-99m (uCi)	The total Tc-99m activity in microcuries assigned to the sample bottle.
Z=2-83 (uCi)	The total activity attributable to Helium (Z=2) through Bismuth (Z=83) assigned to the sample bottle.
Z=84-98 (uCi)	The total activity attributable to Polonium (Z=84) through Californium (Z=98) assigned to the sample bottle.
Z=99-100 (uCi)	The total activity attributable to Einsteinium (Z=99) through Fermium (Z=100) assigned to the sample bottle.
Half Life (yr.)	The half-life in years of the most prevalent isotope in the sample bottle.
Volume (mls)	This number is used to track the volume of a radioactive standard in mL.
Transfer Date	Date that the material was removed from the license.
Transfer ID	Unique ID assigned to the sample upon transfer or disposal

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APPENDIX 2

Nuclides Table (subset shown for illustration)

Isotope_Name	Radionuclide(s)	Z	Weight	Half Life	Daugh 1
Americium-241	Am-241	95	241	432.2	Np-237
Americium-243	Am-243	95	243	7380	Np-239

APPENDIX 3

License Inventory Report

General Engineering Laboratories License Inventory SC-362

31-Oct-96

Category:	Total uCi Z=1	Total uCi Z=2-83	Total uCi Z=84-98	Total uCi Z=99-100
Current Inventory:	249,319.54	3,456.06	2,006.46	0.00
License uCi Limit:	1,000,000 uCi	100,000 uCi	10,000 uCi	10,000
% of Allowable	24.93%	3.46%	20.06%	0.00%

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APPENDIX 4

License Transfer Summary Report

INTERNAL TRANSFERS SUMMARY

27-Mar-95

From : **GEL** To: **EPI**

CONTAINER ID	H-3 (uCi)	Tc-99m	Z=2-83	Z=84-98	Z=99-100
9503320-01.01	23	0	0	0	0
9503320-02.01	23	0	0	0	0
9503320-03.01	23	0	0	0	0
9503387-14.03	5.55	0	0	0	0
TOTALS :	**74.55**	**0**	**0**	**0**	**0**

Materials have been transferred in appropriate containers, and the proper radiological control measures have been taken.

Initials:_____ **Date:**_____

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APPENDIX 5

South Carolina Information Notice

South Carolina
DHEC
Department of Health and Environmental Control
2600 Bull Street, Columbia, SC 29201

Commissioner: Douglas E. Bryant

Board: Richard E. Jabbour, DDS, Chairman
Robert J. Stripling, Jr., Vice Chairman
Secretary, Secretary

Promoting Health, Protecting the Environment

William L. Applegate, III
John H. Burriss
Tony Graham, Jr., MD
John B. Pate, MD

June 6, 1994

Bureau of Radiological Health

INFORMATION NOTICE

TO: S.C. Radioactive Material Licensees Authorized to Receive Samples for Analysis and Testing

FROM: James K. Paterson, Director
Division of Radioactive Material
Licensing and Compliance
Bureau of Radiological Health

SUBJECT: Disposal of Radioactive Wastes

This notice is intended to serve as a reminder to all licensees who receive samples (possibly containing radioactive material) for analysis, testing, research, etc. All samples received under the authority of your license should be disposed of by either (1) return to the sample generator, or (2) transfer to a facility specifically licensed to receive the waste. There are no other disposal options. Samples received under the authority of your license should not be stored or stockpiled at your facility.

You may not dispose of these samples via the sanitary sewer system, decay-in-storage, release to unrestricted areas, or by any other method except those mentioned in the above paragraph. Disposing of these samples at your facility in any manner would require you to obtain a waste disposal license from the Department. This type of license requires a substantial annual licensing fee.

Also, there are no established levels at which a sample can be classified as radioactive or non-radioactive. Therefore ALL samples received under the authority of your radioactive material license should be returned to the generator or transferred to a licensed disposal facility. Contact wastes may be disposed of under the authority of your specific license.

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1.0 Radioactive Material Handling Procedures

Process Owners:	Signature:	Wilbur B. Sigmon, III
	Signature:	Russell L. Moser
Technical Review by:	Signature:	James B. Westmoreland
Approved & Authorized by:	Signature:	Heyward H. Coleman

SET 1.1

2.0 Purpose

2.1) This SOP will be used as a guideline for the safe handling of radioactive material in the laboratory.

3.0 Discussion

3.1) Radioactive material handled in the laboratory is normally activity from contaminated environmental samples. The majority of samples are of more concern from chemical hazards than radiological hazards. However, radioactive material handling procedures are critical to preventing uptakes of radionuclides and cross contamination in the laboratory. The goal of this radiation safety program is to keep exposure to radiation to a minimum.

4.0 Definitions

4.1) Radioactive material. Any material meeting the following criteria:

4.1.1) Sources used in calibrating methods and/or instruments with activity greater than the exempt quantities listed in RHA 2.24.

4.1.2) Samples received for testing that are labeled or shipped as radioactive material. Samples will be considered radioactive until testing shows the levels are less than the limits for concentrations in air and water above natural background listed in RHA 3.21.

4.1.3) Material that exhibits greater than 0.5 mR/hr on contact.

4.2) Radiation Protection Officer: Primary individual responsible for the safe use and handling of radioactive material at the facility.

5.0 Procedures

5.1) During analysis, signs are posted at that work area indicating that analysis of radioactive samples is in progress. Gloves, eye protection, and lab aprons are required for the analysis of radioactive samples.

5.2) The analyst should identify the location of plastic 5 gallon containers to hold liquid waste generated from the analysis of the radioactive samples. Consult the group leader if the waste container cannot be located.

5.3) The analyst should take the following precautions to avoid cross contamination of the area.

5.3.1) Place absorbent material over the exposed work area.
5.3.2) When possible, keep the bulk sample in secondary containment. This may be a tub or a sample cart.
5.3.3) Do not take items out of the controlled area without first monitoring the item for contamination.
5.3.4) Keep dry samples closed and use a fume hood if particulates tend to go airborne.
5.3.5) Rinse all reusable glassware and equipment with DI water and place the rinses in the 5 gallon waste container.

5.4) The contact waste (i.e. paper towels, gloves, etc.) will be placed in a trash bag and taped shut. The analyst will transfer the contact material to a drum that is maintained by the waste coordinator or designee. Refer to EPI SOP S-005 "Radioactive Waste Handling Procedures" for instructions concerning the collection and segregation of radioactive waste.

5.5) Following analysis of radioactive samples, the subjected work areas must undergo wipe tests, as stated in EPI SOP S-001 "Radiation Survey Procedures".

6.0 Safety Health and Environmental Hazards

6.1) Adherence to this SOP should prevent a threat to safety, health or the environment.

7.0 Records Management

7.1) The Radiation Protection Officer or Designee will maintain a record of this SOP for review.

8.0 References

8.1) CRC Handbook of Laboratory Safety 2nd Ed. CRC Press.
8.2) South Carolina DHEC Radioactive Materials Regulation 61-63 Title A.

STANDARD OPERATING PROCEDURE

FOR

RADIATION WORKER TRAINING

Process Owner:	Signature:	Brian T. Morris, Technical Advisor
Technical Review by:	Signature:	James B. Westmoreland, Lab Manager
Quality Review by:	Signature:	Robert L. Pullano, Quality Assurance Mgr.
Approved & Authorized by:	Signature:	Heyward H. Coleman, President

SET |

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1.0 METHOD OBJECTIVE AND PURPOSE

This standard operating procedure (SOP) provides a basic radiation worker training document which is used to train General Engineering Laboratories, Inc. employees in the proper handling and disposal of potentially radioactive samples.

2.0 DISCUSSION

During routine handling of radioactive samples, contamination control and proper waste disposal procedures will minimize the inadvertent spread of possible contamination to the environment. This document provides the basic training necessary to ensure that all employees who handle potentially radioactive samples in the laboratory understand the handling procedures and safety precautions which are well suited to these objectives.

3.0 DEFINITIONS

3.1 **Radioactivity** - Ionizing radiation, particulate, or electromagnetic produced by the decay of unstable nuclei, which can interact with matter and which is not detectable by the human senses.

3.2 **REM** - Unit of radiation dosage that is used in radiation protection. It is a measure of biological damage caused by ionizing radiation. All samples analyzed in the laboratory are less than 2 millirem (mrem) per hour. Average background at sea level is 120 mrem per year.

3.3 **Contamination** - Surface radioactivity in unwanted areas (i.e.: sample residue on the floor).

3.4 **Radiation Protection Officer** - Individual responsible for the safe use and handling of radioactive material.

4.0 MINIMIZING EXPOSURE

4.1 Though the biological effects of radiation are not fully understood, it is generally believed that the harmful effects of radiation are proportional to the dose received. We, therefore, endeavor to keep radiation exposure to a minimum. This is accomplished in three primary ways:

4.1.1 Time - Minimize the time exposed to sources of radiation

4.1.2 Distance - Dose received from a radioactive source is inversely proportional to the square of the distance from the source. Doubling your distance quarters your exposure rate.

4.1.3 Shielding - Most objects will absorb incident radiation if placed in a field, lowering the intensity of radiation exiting the object.

5.0 HANDLING PROCEDURES FOR RADIOACTIVE SAMPLES

5.1 All radioactive sample containers received at login are surveyed for dose rate and swiped to check for surface contamination. If dose rate readings are in excess of 25 mrem/hour, the samples are returned to the client. If readings are less than 25 mrem/hour, the samples are assigned a login number.

5.2 When working with radioactive samples in the prep area, ensure the following is adhered to:

5.2.1 Radiation sign is clearly posted.

5.2.2 Lab coat, safety glasses, and disposable gloves are worn.

5.2.3 Several smears are taken of the prep area, once work has been completed. A smear consists of one "S" shaped wipe of approximately 18 inches. This will be approximately 100 square centimeters.

5.2.4 Liquid waste is disposed of in marked containers, and returned to the sample custodian.

5.2.5 Ensure work is performed on surfaces covered with absorbent material.

5.3 Procedures for a spill

5.3.1 Should there be a spill, immediately secure the source of the spill. Clean up the spill with absorbent material and dispose of it as solid contact waste.

5.3.2 Take and count several swipes to assess the extent of contamination. It is a good practice to take at least one floor smear in the area.

5.3.3 Inform the Radiation Protection Officer or designee of all spills.

6.0 SAFETY HAZARDS

6.1 In order to minimize the amount of contact waste generated, good judgment is required in determining whether lab disposables are potentially contaminated.

6.2 Segregate potentially contaminated glassware from other contact waste so as to minimize the likelihood of injury.

Process Owner(s):
Delores Gibbs, Sample Management Team Leader

Peter Wilber, EPI Support Team Leader

Technical Review by:
Wendy K. Morris, Assistant Radiation Safety Officer

Quality Review by:
Genie G. Bost, EPI Group Leader, Quality

Approved and Authorized by:
Robert L. Pullano, Quality Systems Manager

James B. Westmoreland, EPI Laboratory Manager

1.0 STANDARD OPERATING PROCEDURE FOR THE RECEIVING OF RADIOACTIVE SAMPLES

2.0 PURPOSE

This procedure provides guidelines on how to properly receive radioactive samples from Department of Energy facilities and other specified clients. This SOP is intended to address the radiation safety elements of the receiving process and should be used as a supplement to GEL SOP "Sample Receipt, Login, and Storage" (GL-SR-E-001) for sample receipt. This procedure also establishes controls on the handling of samples containing tritium and other radioisotopes.

3.0 DISCUSSION

The sample receiving area is the initial point of contact for all samples received by the laboratories. It is important; therefore, that special precautions be observed to preclude the

inadvertent spread of radioactive contamination throughout the laboratory which may be present in sample coolers due to spillage during transport. Additionally, these measures will ensure that unmarked radioactive samples are detected prior to being analyzed.

4.0 DEFINITIONS

4.1 Radioactive Material. According to SCDHEC, "There are no established levels at which a sample can be classified as radioactive or non-radioactive." See Appendix 5 of EPI SOP "Inventory and Tracking of Radioactive Material" (GL-EPI-E-S003) for the specific information notice dated June 6, 1994. Therefore any material received under the authority of our radioactive materials license will be considered "radioactive."

4.2 Radiation Safety Officer (RSO). Primary individual responsible for the safe use and handling of radioactive material in the laboratory.

4.3 DOE Facilities (clients and potential clients):

4.3.1 Allied Signal Aerospace Co., MO

4.3.2 Battelle Pacific Northwest Laboratories, WA

4.3.3 Bechtel Environmental

4.3.4 Brookhaven National Laboratory, NY

4.3.5 EG&G Energy Measurements, NV

4.3.6 EG&G Rocky Flats, CO

4.3.7 EG&G Idaho, ID

4.3.8 Lawrence Livermore National Laboratory, CA

4.3.9 Los Alamos National Laboratory, NM

4.3.10 DOE Oak Ridge SMO, TN

4.3.11 Lockheed Martin Energy Systems, TN

4.3.12 Mason & Hangar-Silas Mason Co. (Pantex Plant), TX

4.3.13 Reynolds Electrical and Engineering Co., NV

4.3.14 Sandia National Laboratories, NM

4.3.15 Science Applications International Corporation, NV

4.3.16 Supercollider Laboratory, TX

4.3.17 Westinghouse Savannah River Site, SC

4.3.18 FERMCO, OH

4.3.19 Westinghouse Hanford Site, WA

4.3.20 Jacobs Engineering Group, Inc., TN

4.3.21 Morrison Knudsen Corporation - Weldon Springs Site, MO

4.3.22 West Valley Nuclear Services, NY

4.3.23 Lockheed Idaho Technologies Company (LITCO), ID

4.3.24 Science Application International Corporation, TN

4.4 DOE Subcontractors/Miscellaneous

4.4.1 Other mixed waste laboratories. Quanterra, Mountain States Analytical, UT, TMA/Eberline, TMA/Norcal, Datachem, Clemson Technical Center, Babcock and Wilcox, etc.

4.4.2 Nuclear Power Plants. Duke Power, SCEG, Florida Power, Georgia Power, etc.

4.4.3 Mixed waste related companies. RUST Remediation Services, SAIC, SEG, DSSI, Permafix, Chem Nuclear Systems, Inc., GTS Duratek, Molten Metal Technologies, etc.

5.0 PROCEDURE

5.1 Receiving Shipments Marked "Radioactive II"

5.1.1 If a sample is labeled "Radioactive II" the RSO or designee should be informed. Login personnel are not to open the sample without the RSO or designee's assistance.

5.1.2 The sample should then be opened as outlined below for a radioactive sample.

5.2 Receiving Shipments Marked "Radioactive"

5.2.1 All samples marked "radioactive" shall be opened by two Login personnel. If two Login personnel are unavailable, then the RSO or designee shall oversee the procedure.

5.2.2 Using a survey meter that is calibrated for mR/hr, scan the exterior surface of the cooler as directed in EPI SOP "Radiation Survey Procedure" (GL-EPI-E-S001), keeping within one inch of the cooler. If readings are greater than 2 mR/hr, inform the RSO or designee. The RSO or his designee will assist in the receipt process to ensure exposure levels are acceptable. If upon opening the cooler, the samples register above 25 mR/hr, the client will be notified and arrangements will be made to return the samples or reduce the per sample exposure. Appendix 2 is the Login Safety Process Flow Diagram.

NOTE: The 25 mR/hr limit is an administrative control. If deemed appropriate by the RSO, some samples may be accepted with dose rates higher than 25 mR/hr. Samples above 25 mR/hr will be handled on a case-by-case basis.

5.2.3 Check to ensure that a chain of custody exists. If there is not a chain of custody, the samples are to be placed on hold in the original container with all of the packing material until a chain of custody is received.

5.2.4 Open the cooler and take one swipe of the inner surface. This swipe will be counted to check for leakage during transport. Remove the sample containers and scan each one with the survey meter documenting the highest reading obtained on the Radioactive Shipment Inventory Sheet (Appendix 1). If any sample registers above 2 mR/hr, contact the RSO or designee to verify the acceptability before commencing log-in procedures.

5.2.5 If the samples are not clearly marked radioactive, then place radioactive tape on each sample.

5.2.6 Take one swipe of the sample containers which represents the samples from each well or site sampled.

5.2.7 Do not dispose of any packing material until all of the samples are logged in and every sample on the chain of custody form is accounted for.

5.2.8 Forward the Radioactive Shipment Inventory Sheet (Appendix 1), client's shipping information sheet, any available activity data, and all swipes to EPI for counting and entry into the material balance.

5.3 Receiving Shipments from DOE facilities or others listed in Sections 4.3 and 4.4 that are not marked as "Radioactive"

5.3.1 Open the cooler and scan the inside of each cooler before removing sample containers. If there are no readings greater than 0.5 mR/hr. the samples may be treated as non-radioactive and normal log-in procedures followed.

5.3.2 If a reading greater than 0.5 mR/hr is obtained, scan each sample container individually to determine which samples are causing the high readings and inform the RSO. Handle the samples in accordance with EPI SOP "Radioactive Handling Procedures" (S-004).

5.3.3 Once it is determined that all samples are less than 0.5 mR/hr, log-in procedures may be commenced.

6.0 QUALITY CONTROL

Refer to EPI SOP "Radiation Survey Procedures" (S-001) for quality control requirements for the survey equipment.

7.0 SAFETY, HEALTH, AND ENVIRONMENTAL HAZARDS

Adherence to this procedure should prevent a threat to personnel or the environment.

8.0 RECORDS MANAGEMENT

The original Radioactive Shipment Inventory Sheet (Appendix 1) is kept in a bound file in the EPI laboratory.

9.0 REFERENCES

SCDHEC Title A Radioactive Materials Regulations, Parts 61-63.

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APPENDIX 1

Radioactive Shipment Inventory Sheet

Received By: _____ Date Received:_____

No. of Samples in Shipment: _____

Total Shipment Activity: Tritium (μCi):_____

Other (μCi):_____

If shipment activity is from non-tritium isotopes, list the isotopes below:

_____

_____

Sample Information:

Client:	
Lab ID's in Sample Receipt:	
Maximum Surface Reading (mR/hr):	
Maximum Removable Alpha Contamination ($dpm/100cm^2$):	
Maximum Removable Beta Contamination ($dpm/100cm^2$):	

Please attach applicable client supplied radiological information:

Shipment Reference Number:_____

Reviewed and Approved:_____Date:_____

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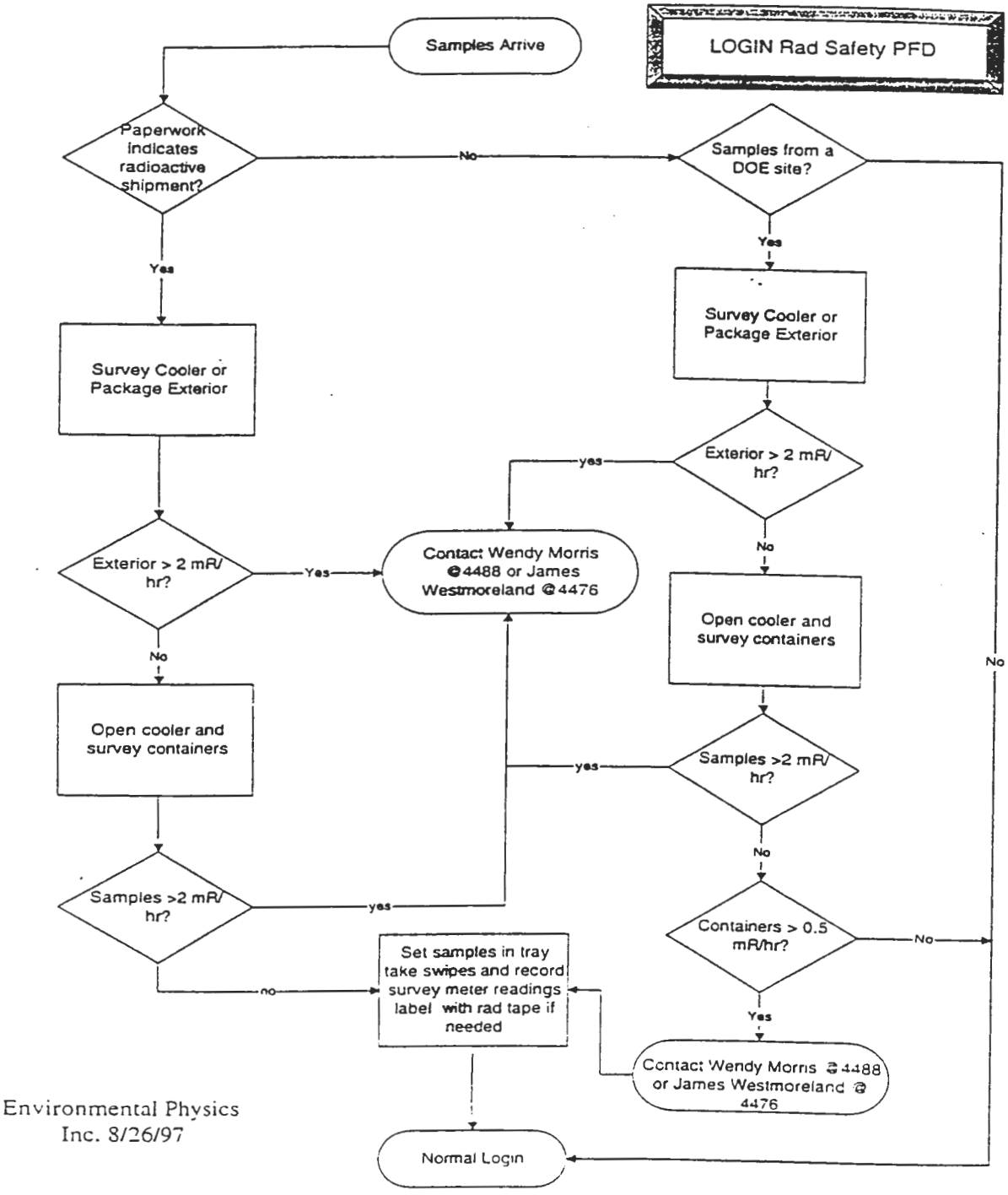
APPENDIX 2



Environmental Physics Inc. 8/26/97

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1.0 Shipping of Radioactive Samples and Sample Residues

UNCONTROLLED DOCUMENT

Process Owner:	Signature:	W. Byron Gainey
Technical Review by:	Signature:	James B. Westmoreland
Approved & Authorized by:	Signature:	Heyward H. Coleman

SET

2.0 Purpose

2.1) This procedure provides guidelines on how to properly ship radioactive samples to meet the Department of Transportation regulations for excepted Radioactive Material UN 2910..

3.0 Discussion

3.1) Environmental Physics Inc. will periodically return radioactive samples and sample residues to clients. Since activity levels are generally low, no special packaging or certification is required. These shipments are classified "excepted radioactive material" as defined in 49 CFR 173.421. This procedure deals exclusively with the packaging and documentation requirements for this case.

4.0 Definitions

4.1) Fissile Nuclides. Pu-239, U-235 or other nuclides for which safeguards must be taken to ensure against criticality during shipment.

4.2) Radioactive Material (RAM). For transportation purposes only, a level of activity above 2 nanocuries per gram is considered as RAM.

4.3) Radiation Protection Officer (RPO). Primary individual responsible for the safe use and handling of radioactive material in the laboratory.

5.0 Procedure

5.1) Determine the isotopes to be shipped and their activity. Confirm that the amounts of activity to be shipped meet the criteria as stated in 49 CFR 173.423 table 7 for excepted radioactive material.

5.2) Verify that the receiving party is licensed to receive the type and quantity of material being shipped and that they are expecting the shipment.

5.3) Package the material in a strong, tight container that will not leak any of the material during normal transport. For liquid and/or soil samples, tightly secure the containers by taping the lids. Seal the containers in a plastic bag to collect possible leakage during transport.

5.4) Secure the bagged material in an appropriately sized "inner" cardboard box, filling the void space with padding material (cardboard, foam, etc.).

5.5) Clearly mark the exterior of this inner box "Radioactive" on at least two opposite sides.

5.6) Secure the inner box in the center of an appropriately sized "outer" box, filling the void with padding material. A new box should be used for shipping radioactive material.

5.7) Before securing the outer box, scan the surface with an E-120 detector HP-270 probe, record the mR/hr. reading on the Limited Quantity Shipment Report (attachment 1), placing the original signed copy inside the box. If the reading is greater than 0.5 mR/hr. on contact, the material cannot be shipped as excepted radioactive material.

5.8) Clearly mark "this end up" on the outer box and include the following statement: "This package conforms to the conditions and limitations specified in CFR 49.173.422 for excepted Radioactive Material Limited Quantity NOS".

5.9) Perform a removable contamination survey as outlined in EPI SOP S-001 to confirm the exterior contamination is less than 20 dpm/ 100 cm^2 gross alpha activity and less than 100 dpm/100cm^2 gross beta activity.

5.10) Complete the Limited Quantity Shipping Report (attachment 1) and submit to the RPO for review. Include the shipping report in the shipment and tape the UPS Destination Label (attachment 2) to the box and secure the box.

5.11) Complete the UPS shipping books for each container shipped. An example of the completed forms is found in attachment 2. Note: **Do not** ship air freight.

6.0 Quality Control

6.1) The Radiation Control Officer or his designee shall be notified of all outgoing radioactive shipments. Additionally, the Laboratory Manager approves all shipments by signing the Limited Quantity Shipment Report.

7.0 Safety, Health, and Environmental Hazards

7.1) Adherence to this procedure prevents a threat to personnel or the environment.

8. References

8.1) CFR 49 Parts 100-177

Attachment 1

General Engineering Laboratory
Limited Quantity Shipment Record

From: General Engineering Laboratory
2040 Savage Road
Charleston, SC 29745

To: _____
_____
_____

Attn. James Westmoreland
803-556-8171

Attn. _____
Phone #_____

License # SC 362

License # _____

This Package Conforms to the conditions and limitations specified in 49CFR 173.421 for excepted radioactive material, n.o.s UN2910.

Radioactive receipt #: _____

Isotopic Activity: Tritium _____ uCi

Other (list isotopes)
_____ _____ uCi

Removable Contamination from package surface.

Gross Alpha _____ dpm/100 cm^2

Gross Beta _____ dpm/100 cm^2

Package Inventory:
_____
_____
_____

Radiation level on outside of package is less than 0.5 mR/hr. _____ (initial)

Inner Package is marked "RADIOACTIVE" _____ (initial)

Shipment prepared by:_____ Date_____
Shipment approved by:_____ Date_____

Attachment 2

United Parcel Service — UPS COPY

PICKUP RECORD

DATE 12/23/91

RECD FROM: General Engineering Labs, 2040 Savage Road, Charleston SC 29414

PICKUP RECORD NO. 168966900

ON-CALL CODE: 4

STAMP YOUR UPS SHIPPER NUMBER BELOW

ENTER EACH PACKAGE ON A SEPARATE LINE. IF RECORD IS VOIDED, PLEASE GIVE TO DRIVER.
INCREASE FRACTIONS OF A POUND TO NEXT FULL POUND.

REFERENCE #	NAME - STREET - CITY	STATE	ZIP CODE	TYPE SERVICE: GROUND ZONE	TYPE SERVICE: AIR ZONE	LBS	DECLARED VALUE	COD AMOUNT	FOR UPS OFFICE USE ONLY
Box 1	Westinghouse Savannah River Bldg 773-A Aiken	SC	29808	2		15			
2									
3									
4									
5									
6									
7									
8									
9									
10									
11									
12									
13									
14									
15									

RECEIVED BY | PICKUP TIME | NO. PKGS. | NO. CALLS

* OR (Oversized packages if less than 25 lbs. and more than 84 inches in length and girth combined.
** Unless a greater value is declared in writing on this receipt, the shipper hereby represents and agrees that the released value of each package or article not exceeding in a package delivered by this receipt is $100, which is a released value unless otherwise stated. The consignee hereby agrees that the shipment is delivered hereunder. This policy of a C.O.D. amount is not a declaration of value. In addition, the shipper hereby agrees that the maximum liability is $25,000 and the maximum carrier liability is $25,000. Claims not made in writing within 9 months of the scheduled delivery date are waived.
*** All checks in payment of C.O.D.'s accepted at shipper's risk.

.8 REV. 6-92 (BOOM 8KS. 6-921A

GENERAL ENGINEERING LABORATORIES
2040 Savage Road, Charleston, SC 29414
P. O. Box 30712, Charleston, SC 29417

To: Westinghouse Savannah River
Site
Bldg 773-A
Aiken SC 29808
Attention John Doe

15 x 227-848

Process Owner and Technical Review: Wendy K. Morris, Assistant Radiation Safety Officer

Quality Review by: Genie G. Bost, EPI Group Leader, Quality

Approved and Authorized by: Robert L. Pullano, Quality Systems Manager

James B. Westmoreland, EPI Laboratory Manager

1.0 STANDARD OPERATING PROCEDURE FOR DOSIMETRY PROCEDURES

2.0 PURPOSE

This operating procedure is designed to outline personnel dosimetry procedures utilized in the laboratory.

3.0 DISCUSSION

This SOP describes the dosimetry procedures utilized at Environmental Physics Inc. and General Engineering Laboratories, Inc. The method holds to the principles outlined in U.S. NRC Regulatory Guide 8.10 "Operating Philosophy for Maintaining Occupational Radiation Exposures As Low As Is Reasonably Achievable (ALARA)."

4.0 DEFINITIONS

4.1 Radioactive Material. According to SCDHEC, "There are no established levels at which a sample can be classified as radioactive or non-radioactive." See Appendix 5 to EPI SOP "Inventory and Tracking of Radioactive Material" (GL-EPI-E-S003), for the specific information notice dated June 6, 1994. Therefore any material received under the authority of our radioactive materials license will be considered "radioactive."

SE

4.2 Geiger-Mueller (GM) survey meter. A portable instrument with a radiation detection probe. An Eberline E-120 instrument with a HP-260 probe (or equivalent) is used for contamination surveys. An Eberline E-120 instrument with a HP-270 (or equivalent) probe is used for dose rate surveys.

4.3 Radiation Safety Officer (RSO). Primary individual responsible for the safe use and handling of radioactive material at the facility.

4.4 TLD. Acronym for "Thermoluminescent Dosimeter". This device absorbs radiation into a chip usually composed of lithium fluoride. The cumulative radiation exposure on the chip can be quantified at a later date.

4.5 ALARA. Acronym for "As Low As Is Reasonably Achievable". This acronym pertains to the handling of radioactive material and limiting exposure to personnel.

4.6 SCDHEC. Acronym for "South Carolina Department of Health and Environmental Control," specifically, the Bureau of Radiological Health.

5.0 PROCEDURES

5.1 External Dosimetry

5.1.1 Designated personnel handling radioactive material in the laboratory will be given a TLD badge. The RSO will designate the appropriate personnel for wearing a TLD badge. In general, preparatory personnel and log in personnel will be given TLD badges as they will be handling the bulk of activity in the laboratory. The badge must be worn at all times while in the laboratory.

5.1.2 The TLD must be worn on the front torso above the belt line and below the neck line.

5.1.3 The laboratory personnel should return the TLD to the badge rack with the control badge when not in use.

5.1.4 Personnel TLD badges will be exchanged on a quarterly basis to a facility that is accredited by NVLAP of the U.S. Department of Commerce.

5.1.5 Quarterly exposure reports are posted in the laboratory.

5.1.5 Any lost or destroyed badge must be reported to the RSO.

5.2 Internal Dosimetry

5.2.1 In the event that greater than 100 mCi of Hydrogen-3 is processed by an analyst in one month, bioassay samples will be taken for that analyst. This sample will be submitted for analysis to a laboratory licensed by the Nuclear Regulatory Commission or an Agreement State to perform the analysis.

5.2.2 The analyst will be notified by the RSO that a sample is required. The RSO will submit a Bioassay Sample Form (attachment 1) and a labeled bioassay bottle. The analyst will void into the bottle for a 24 hour period. At the end of the 24 hour interval the bottle will be returned to the RSO for shipment to the approved laboratory.

5.3 Limits for Exposures

5.3.1 External exposure to an individual will not exceed 1250 mR/quarter.

5.3.2 Internal limits will not exceed the limits specified in Part III, Appendix B, RHA 3.53 "Annual Limits on Intake."

5.3.3 In the event that any of the above limits are exceeded, the following will occur:

5.3.3.1 The RSO will notify the SCDHEC by written report as specified in RHA 3.46.

5.3.3.2 Any individual who has received occupational exposure above the specified limits, will be reassigned to work that will not involve radioactive exposures until deemed appropriate by the RSO.

5.3.3.3 A corrective action will be initiated to ensure the elevated exposure condition does not reoccur.

6.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

Adherence to this SOP should prevent a threat to safety, health or the environment.

7.0 RECORDS MANAGEMENT

The Radiation Safety Officer will maintain all dosimetry records indefinitely.

8.0 REFERENCES

8.1 South Carolina DHEC Radioactive Materials Regulation 61-63 Title A.

APPENDIX 1

Bioassay Sample Form

Analyst Name: _____

Social Security #: _____

Date of Birth: _____

Initial Void
Date & Time _____

Final Void
Date & Time _____

Approximate
Volume (mLs) _____

Comments _____

_____

_____

Analyst Signature: _____

Date _____

RSO Signature: _____

Date _____

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Process Owner(s):	Jeff T. Overcash, Radioanalytical Technician
Technical Review by:	James B. Westmoreland, EPI Laboratory Manager
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	Heyward H. Coleman, EPI President

1.0 STANDARD OPERATING PROCEDURE FOR THE HANDLING OF BIOLOGICAL MATERIALS

2.0 PURPOSE

2.1 This procedure provides guidance on the proper handling of samples that may be contaminated from normal biological pathways. This includes the handling of samples such as urine, feces, meats, and other animal tissues.

3.0 DISCUSSION

3.1 Care must be taken when handling biological materials to prevent personnel from becoming infected with a pathogen. Hepatitis is of particular concern when handling bioassay samples. Brucellosis, an infectious bacterial disease, is a concern when handling animal tissue. It is important therefore, that special precautions be observed to preclude the inadvertent spread of biological contamination throughout the laboratory.

3.2 Individuals handling human feces shall have an up to date (within 10 years) tetanus shot as well as having completed a series of polio vaccinations. Hepatitis vaccination shots (though not required) will be made available to all personnel handling biological samples.

3.3 A bioassay fume hood shall be established for sample preparation, fuming, and digestion. All bioassay samples will be completely segregated from other analyses in the laboratory.

4.0 DEFINITIONS

4.1 Biological Materials. Any material being analyzed in the laboratory that has been derived from a human or animal. This includes tissue, urine and feces.

4.2 Bioassay samples. Feces or urine collected from a human for the purpose of calculating uptake of a radionuclide.

5.0 PROCEDURE

5.1 During receipt, storage, and sample preparation bioassay samples shall be segregated from all other analyses in the laboratory. Bioassay samples will be unpacked in a fume hood and inspected for leakage. If there is any visible leakage, the leakage must be treated as a biohazard spill. The spill area will be decontaminated accordingly prior to continuing with the receipt. The exterior of the containers will be wiped with a disinfectant solution.

5.2 Once the samples are satisfactorily decontaminated the samples are to be labeled with their laboratory samples number. Urine samples shall be stored refrigerated; feces and tissue samples are to be stored frozen. All biological samples will be archived in a segregated area while awaiting analyses.

5.3 When handling biological samples protect the hands and forearms by wearing gloves and a laboratory coat to avoid contact of the biological material with the skin. Protect the eyes by wearing safety glasses with side shields and/or face splash shield.

5.4 If cutting of meats or other tissue with cutlery is required, the analyst will wear a cutting glove of mesh steel over disposable gloves to avoid cuts which could infect the analyst with pathogens.

5.5 Any procedure which volatilizes biological substances such as drying or ashing, must be conducted in a hood or other suitable containment device.

5.6 Decontamination of work surfaces exposed to biological samples is performed by wiping the work area with a diluted (1:10) bleach solution and water as soon as possible following analytical operations.

5.7 Exterior protective clothing shall be removed prior to exiting the bioassay sample preparation area in order to prevent the inadvertent spread of biohazards to the rest of the laboratory.

5.8 Wash hands with an antibacterial soap immediately after handling biological samples.

6.0 QUALITY CONTROL

6.1 Refer to specific analytical SOP's for quality control requirements such as duplicates, matrix spikes and laboratory control samples.

7.0 SAFETY, HEALTH, AND ENVIRONMENTAL HAZARDS

7.1 Urine and feces waste products may be disposed of by sanitary sewer followed by a dilute bleach rinse.

7.2 Non radiological biological material may be disposed of in the normal trash. It is recommended that tissue samples be ashed prior to disposal.

7.2 Liquid spills will be cleaned with a compound that absorbs the biological material.

8.0 REFERENCES

Not applicable.

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STANDARD OPERATING PROCEDURE

FOR

LABORATORY WASTE DISPOSAL AND EMERGENCY INSTRUCTIONS

UNCONTROLLED DOCUMENT

(GL-EPI-S-011 Revision 2)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

Process Owner	(sign & date)	Christopher S. Gobel (print name)
Technical Review	James B. Westmoreland 8/4/97 (sign & date)	James B. Westmoreland (print name)
Quality Review	8/4/97 (sign & date)	Gene G. Bost (print name)
Approval and Authorization	8/6/97 (sign & date)	(print name)

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR LABORATORY WASTE DISPOSAL AND EMERGENCY INSTRUCTIONS

2.0 PURPOSE

2.1 This document details the procedures for the collection and disposal of laboratory generated sample residue. It is intended for use by laboratory personnel after sample analysis. In addition, it addresses the final disposition of samples and wastes which is performed by a waste technician in accordance with the Laboratory Waste Management Plan. The purpose of this procedure is to ensure that discharges or shipments from this facility are in compliance with applicable environmental regulations. In addition, this SOP incorporates the RCRA Emergency/Contingency Plan for laboratory and environmental emergencies. (See Appendix 1.)

3.0 DISCUSSION

3.1 A variety of sample residues and wastes are generated and managed in the process of analyzing samples in the laboratory. It is important to distinguish between samples and wastes in the laboratory for regulatory purposes. According to 40 CFR 261.4(d), "A sample of solid waste or a sample of water, soil or air which is collected for the sole purpose of testing to determine its characteristics or composition, is not subject to any requirements of this part...". The exclusion is dependant on various conditions that are too numerous to consider in this SOP. For the purpose of this procedure the sample exclusion ends when the sample or sample residue is determined to be hazardous as defined in 40 CFR part 261.3.

3.2 Some of the waste streams produced in the laboratory have known hazards and are placed in **satellite accumulation areas** for proper disposal without further testing. An example of one of these known hazardous waste streams is a scintillation fluid with listed hazardous compounds.

3.3 Other waste streams are not known to be hazardous until further testing is performed. These samples are collected in **sample collection areas**.

3.4 When sample collection area containers or satellite accumulation area containers are filled, the assigned technician transfers the containers to a staging area. When satellite accumulation area containers are moved to the staging area, they must be disposed of within 180 days according to the regulations pertaining to small quantity generators of hazardous waste.

3.5 The waste technician maintains a log of satellite accumulation area movement and disposal manifests. When sample accumulation area containers are moved to the staging area they do not have to be moved until sample analysis has been performed to determine the material is hazardous. If the material is determined to be hazardous then the 180 day time limit is invoked.

3.6 Samples and sample residues which are returned to the client maintain their classification as samples and sample residues according to the regulations. Samples and sample residues which cannot be returned to the client are composited and then characterized. Based on the analytical results, sample residues are classified as wastes and disposed of appropriately. All samples and sample residues which become classified as wastes must be disposed of in accordance with the Laboratory Waste Management Plan.

3.7 **Important radioactive materials license compliance note**: No radioactive sample or residue produced from analysis, that is received for testing with a known quantity of radioactivity may be released into the sanitary sewer for disposal and then removed from the radioactive materials license. The radioactivity from these materials must be ultimately transferred to the original client or be disposed of at a licensed disposal facility. Failure to comply with this practice may result in fines and/or loss of radioactive materials license.

4.0 DEFINITIONS

4.1 Minimum Detectable Activity (MDA). The minimum activity level that is detectable at a 95% confidence level.

4.2 Radioactive contact waste - This includes gloves, paper towels, etc., which are used during analysis of radioactive samples. Note: contact waste is segregated by burnable items (paper and plastic) and non-burnable items (metals , glass, etc.) for disposal.

4.3 Radioactive material - Any material received under the authority of our radioactive materials license will be considered "radioactive". According to SCDHEC, "there are no established levels at which a sample can be classified as radioactive or nonradioactive". See appendix 7 for the specific information notice dated June 6, 1994.

4.4 Radiation Safety Officer (RSO) - Individual responsible for the safe use and handling of radioactive material at the facility.

4.5 Staging area: An area used to manage samples and waste prior to final disposition.

4.6 Sample - Any material received by the laboratory for analysis.

4.7 Sample residue - Any material generated during the analytical procedure which has come in contact with a sample.

4.8 Satellite accumulation area - A designated container for accumulating a hazardous waste in the laboratory.

4.9 Sample collection area - A designated container for accumulating a sample residual material in the laboratory. This material will be resubmitted for analysis

as a sample and is excluded from the regulations as specified in 40 CFR part 261.4.

5.0 PROCEDURES

5.1 Satellite accumulation area and sample collection area procedures. These procedures are intended to give instructions to laboratory personnel regarding laboratory waste. Refer to section 5.2 for instructions on movement and disposal of waste. To avoid spillage, please notify the waste technician or your supervisor when a container is nearly full. See Appendix 2 for the locations of satellite accumulation area and sample collection area containers.

5.1.1 Aqueous radioactive sample residue: This material is generated during the analytical process and contains reagents mingled with radioactive samples as received. This material is placed in sample collection area containers which are located in designated areas of the laboratory.

5.1.1.1 It is important to keep the sample collection area containers closed when not in use to avoid spillage.

5.1.1.2 Do not add oily, high % solids or known hazardous materials to these containers. Material of this nature will be placed in a separate container labeled with the date, client and sample numbers.

5.1.1.3 Two yellow aqueous radioactive sample residue 30 gallon drums are available for aqueous radioactive sample residues: One is for WSRC ground water only, the second is for other clients.

5.1.2 Aqueous non-radioactive sample residue: This material is generated during the analytical process and contains reagents mingled with non-radioactive samples. This material is placed in sample collection area containers which are located in designated areas of the laboratory.

5.1.2.1 It is important to keep the sample collection area containers closed when not in use to avoid spillage.

5.1.2.2 Do not add oily, high % solids or known hazardous materials to these containers. Material of this nature will be placed in a separate container labeled with the date, client and sample numbers.

5.1.3 Solid radioactive sample residue: This material is generally a sludge that is created when a radioactive sample is analyzed with a leaching

technique to remove the radionuclides of interest from the sample matrix. The residual solid sample and the leaching agent are placed in a plastic labeled container in the laboratory.

5.1.4 **Solid non-radioactive sample residue:** This material is generally a sludge that is created when a non-radioactive sample is analyzed with a leaching technique to remove the radionuclides of interest from the sample matrix. The residual solid sample and the leaching agent are placed in a plastic labeled container in the laboratory.

5.1.5 **Solid radioactive contact waste**: Material that is known to be incidentally contaminated with a radioactive sample during analysis is placed in one of the following locations.

5.1.5.1 If the contact waste is metal, glass or any material that is non-burnable, the material is placed in a lined 55 gallon metal drum labeled for non-burnable contact waste.

5.1.5.2 If the contact waste is paper, plastic or any material that is burnable, the material is placed in cardboard boxes labeled for radioactive waste.

5.1.6 **Liquid scintillation vials:** This material is kept in the vial and placed in one of the following locations. When adding vials to these drums please complete the LSC drum log (appendix 3). (Do not pour any free flowing fluid into these drums.)

5.1.6.1 The scintillation vial drums will be labeled for the isotopes that are acceptable for addition. This is done because certain isotopes used in the laboratory can cause disposal costs to escalate. Plutonium-241, Nickel-63, Selenium-79, and Tellurium-125m for example tend to be expensive to dispose of. Tritium, Carbon-14 and Technetium-99 tend to be less costly for disposal.

5.1.7 **Used resins:** Resins which are no longer useful in the laboratory are placed in labeled plastic or glass containers.

5.1.8 **Excess radioactive and non radioactive samples:** Excess sample material is kept in the original labeled bottles. The unused sample is returned to the sample archival area for final disposition.

5.2 Staging area procedures.

5.2.1 The waste technician or designee transfers the sample collection area containers to the staging area when filled. The waste technician maintains a log which is used to record drums created or moved into the staging area. On a monthly frequency, the satellite accumulation area and sample collection areas are inspected. See Appendix 5 for the satellite accumulation area monthly checklist.

5.2.2 **Aqueous radioactive sample residue.** If contractual arrangements have been made to return this material to the client, then package in accordance with DOT regulations and ship to the original client. If the material cannot be returned to the original client, then the material is screened for the presence of radioactivity. Depending on the isotopes suspected in the sample residue the following procedure(s) may be used to separate the radioactivity from the bulk solution for disposal: Alternately the material may be shipped without adjustment to a licensed disposal site after characterization for radioactive and hazardous constituents.

5.2.2.1 **Nuetralization**: Metals and many radionuclides such as the actinides and strontium may be carried effectively into a calcium phosphate/iron hydroxide precipitate. Note: cesium will not be significantly removed by this technique.

5.2.2.2 To the acidic solution add 1 mL calcium nitrate carrier (10 mg/mL) and 1 mL ferric nitrate carrier (10 mg/mL) per liter of aqueous material.

5.2.2.3 Note: to obtain a 10mg/mL carrier solution, dissolve 30 g of $Ca(NO_3)_2 * 4H_20$ in 500 mL of DI water. To obtain a 10 mg/mL iron carrier solution, dissolve 20 g of $Fe(NO_3)_3*9H_2O$ in 500 mL of DI water.

5.2.2.4 Add 1 ml of phosphoric acid per liter of solution and adjust the pH of the mixture to >7 with a basic reagent such as sodium hydroxide. Mix and allow the precipitate to settle. The clear supernate is then separated from the precipitate.

5.2.2.5 **Ion exchange**. Resin may be added directly to a solution and mixed or the solution may be passed through a column which has been packed with a resin. See the Laboratory Manager or Senior Chemist for the best resin(s) for a given waste stream.

5.2.2.6 **Distillation**: Place the solution in a suitable container for distillation. Connect a condenser to the distillation flask, begin the flow of cooling water and heat the solution to boiling. Boiling

chips or beads may be added to achieve a steady boil. Do not allow the distillation flask to go to dryness and do not continue distillation overnight. Any solid material resulting from distillation may be added to the radioactive solid contact waste stream mentioned in 5.2.6. Note: it is recommended that material be screened for tritium prior to distillation to avoid contamination of the distillate.

5.2.2.7 Aqueous non radioactive material collected after completion of any of the above techniques is submitted for sample screening as described in section 5.2.11. If no radioactivity is detected above the MDA, the eluant may be handled as an aqueous non-radioactive sample residue as listed in 5.2.3. If radioactivity is detected in the sample screen, the liquid may be re-evaluated for an alternative separation technique, or the material may be transported by an approved transporter to an approved TSD facility as listed in Appendix 4.

5.2.2.8 Solid radioactive material from the precipitation and/or ion exchange is composited and added to a sample collection area container for disposal as outlined in section 5.2.8.

5.2.3 Aqueous non-radioactive sample residue. This material is sample screened as outlined in 5.2.11. If no activity is detected in the sample screen above the MDA, the material is handled as outlined in the waste management plan. If activity is detected above the MDA, the material is handled as outlined in 5.2.2.

5.2.4 Solid radioactive sample residue.

5.2.4.1 Sludge material is filtered or dried to remove residual water. A sample of the dried material is then characterized for any hazardous constituents by TCLP.

5.2.4.1.1 If the material is determined to be non-hazardous, the radioactive constituents are determined as required by the disposal site listed in Appendix 4.

5.2.4.1.2 If the material is determined to be a mixed waste, the material may be solidified to determine if the hazardous characteristic can be contained when subjected to a TCLP. The material can then be shipped with prior permission and review of the low level disposal site. If solidification is impractical or deemed not cost effective.

the material may be transferred to a licensed broker or disposal site that can dispose or treat the mixed waste.

5.2.4.2 Oily material is characterized without treatment using the testing parameters required by the specific disposal site listed in Appendix 4. Extreme caution must be used in compositing oily material as explosive conditions may result. Please follow the compatibility testing outlined in 5.2.12 prior to compositing.

5.2.5 Solid non radioactive sample residue. This material is sample screened as outlined in 5.2.11. If no activity is detected in the sample screen above the MDA, the material is handled as outlined in the waste management plan. If activity is detected above the MDA, the material is handled as outlined in 5.2.4.

5.2.6 Solid radioactive contact waste.

5.2.6.1 Non-Burnable material is compacted into a plastic lined 55 gallon steel open top drum for disposal. A representative sample is taken for radiochemical characterization. This material is transported by an approved transporter to an approved TSD facility as listed in Appendix 4.

5.2.6.2 Burnable material is placed in plastic lined cardboard boxes for disposal. A representative sample is taken for radiochemical characterization as specified in Appendix 3. This material is transported by an approved transporter to an approved TSD facility as listed in Appendix 4.

5.2.7 Liquid scintillation vials. Liquid scintillation vials are placed in a plastic lined 55 gallon open top drum. The drums are labeled by the isotopes present. One drum is used for Carbon-14 and Tritium only, a second drum is used for other isotopes. This material is manifested for disposal and is transported by an approved transporter to an approved TSD facility as listed in Appendix 4.

5.2.8 Spent resins. Resin which is no longer usable to remove radioisotopes from aqueous media is placed in a lined drum for disposal. The resin is filtered and air dried in a fume hood prior to placing in the drum. A representative sample is taken for radiochemical characterization. This material is transported by an approved transporter to an approved TSD facility as listed in Appendix 4.

5.2.9 Excess radioactive sample.

5.2.9.1 Material that is not consumed and distributed into the collection streams mentioned above, is returned (if permitted by contract) to the original client for final disposition.

5.2.9.2 If the unused sample cannot be returned, it is composited by similar matrix for disposal. It is very important to check the parameters tested on the individual samples to ensure that a known hazardous material is not contaminating bulk non-hazardous material.

5.2.9.3 To facilitate the process of checking for known hazards, scan samples of like visible matrix (water, soil, oil, debris, cement etc.) to a new waste drum in LIMS.

5.2.9.4 Using the database program Microsoft Access or equivalent, the drum contents can be displayed by sample number and parameters tested in LIMS. The waste coordinator, or lab manager must review this data and approve before actual compositing may begin.

5.2.9.5 Remove samples that are suspect as revealed by the query of LIMS and composite the remaining samples into a waste drum that is compatible with the material.

5.2.9.6 A sample of the drum is then characterized for any hazardous constituents by TCLP.

5.2.9.7 If the material is determined to be non-hazardous, the radioactive constituents are determined as required by the disposal site listed in Appendix 4.

5.2.9.8 If the material is determined to be a mixed waste, the material may be solidified to determine if the hazardous characteristic can be contained when subjected to a TCLP. The material can then be shipped with prior permission and review of the low level disposal site. If solidification is impractical or deemed not cost effective, the material may be transferred to a licensed broker or disposal site that can dispose or treat the mixed waste. The individual radioactive samples that have been scanned into the waste drum in LIMS are then removed from the radioactive materials license database.

5.2.10 Empty sample containers.

5.2.10.1 The analyst should check to make sure the sample container has been correctly tracked to a status of EMTY in LIMS.

5.2.10.2 Containers that are labeled as radioactive are either compacted directly into the contact waste drum, or shredded in the bottle shredder. If the containers have visible residue it is best to avoid potential contamination of the shredder.

5.2.10.3 This material is sample screened as outlined in 5.2.11. If no activity is detected in the sample screen above the MDA, the material is handled as outlined in the waste management plan. If activity is detected above the MDA, the material is handled as outlined in 5.2.2.

5.2.11 Sample screening procedure: Add 1.5 mL of the liquid material or 0.1g of solid material to a 7 mL scintillation vial. Add 4.5 mL of "Ready Gel" liquid scintillation fluid, cap and mix thoroughly.

5.2.11.1 Label the vial cap with the drum number and count on a liquid scintillation counter under one of the sample screening programs.

5.2.11.2 Transfer the counted sample information to an excel spreadsheet and print the spectrum for review. Any activity greater than the detection limit is a positive sample screen.

5.2.12 Compatibility testing procedure: When compositing samples or sample residuals of unknown composition, this quick compatibility test should be followed.

5.2.12.1 Working within a well ventilated area place approximately 1 mL of each material to be composited in a dispo culture tube. Any evidence of a chemical reaction such as fuming, igniting, etc. indicates the materials are incompatible and should not be mixed.

6.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

6.1 When handling any sample or waste material the technician must wear personal protective equipment which includes, gloves, labcoat, and safety glasses. Depending on the operation, a face shield, apron, sleeve protectors, or dust mask may be used.

6.2 Care should be taken when moving sample collection area containers. When full, these containers are very heavy and should be moved with a drum or hand truck.

6.3 If a reaction occurs when adding material to a container, stop pouring and contact the waste technician or your supervisor. A reaction may produce heat, fumes, or other potentially dangerous conditions.

6.4 Collection containers must be closed when being transported in the laboratory.

7.0 RECORDS MANAGEMENT

7.1 Logbooks, manifests, and disposal records are maintained in the staging area for review.

8.0 REFERENCES

8.1 South Carolina Department of Health and Environmental Control (SCDHEC) Radioactive Material Regulations (Title A) Parts 31-33

APPENDIX 1

RCRA Emergency/ Contingency Plan

COMPANY: General Engineering Laboratories, Inc.

ADDRESS: 2040 Savage Road

LOCATION OF FACILITY: 2040 Savage Road

CITY: Charleston **STATE**: SC **ZIP**: 29414

TELEPHONE: 843-556-8171 **EPA #**: SCR000002915

PRIMARY EMERGENCY COORDINATOR: George McAbee **ADDRESS**: 2040 Savage Road
TELEPHONE (OFFICE): 843-556-8171 ext. 4452 **(HOME)**: 843-556-6955

SECONDARY EMERGENCY COORDINATOR: James Westmoreland **ADDRESS**: 2040 Savage Road
TELEPHONE (OFFICE): 843-556-8171 ext. 4476 **(PAGER)**: 843-805-9100

DESCRIPTION OF HAZARDOUS WASTES HANDLED

DOT Classification	Volume On Site	Monthly Volume	DOT ID #	DOT Emergency Guide #	EPA Waste Code
Waste Flammable Liquid, N.O.S	140 gal.	30 gal.	UN1993		F003,F005
Hazardous Waste Liquid, N.O.S.	30 gal.	5 gal.	NA3082		D002, D009

FEDERAL, STATE & LOCAL EMERGENCY RESPONSE CONTACTS

ORGANIZATION	CONTACT & LOCATION	PHONE
LOCAL		
Primary Fire Dept.	St. Andrews Fire Dept.	911
Secondary Fire Dept.	City of Charleston Fire Dept.	911
Emergency Response Team	Under direction of Fire Department hazardous material team	911
State Environmental	SC DHEC- Trident Office, 1000 Air Park Road	843-740-1590
Sheriff's Dept.	County of Charleston Dept., 3505 Pinehaven Ave.	911
Local State Police	SC State Highway Patrol, 6355 Fain St.	843-740-1660
Primary Hospital	Roper Hospital, 316 Calhoun St. , Charleston SC	843-724-2001
Secondary Hospital	St. Francis Xavier, 135 Rutledge Avenue	843-577-1000
STATE		
State Police HQ	SC State Highway Patrol, Columbia	803-737-1083
State Environmental HQ	SC DHEC 2600 Bull Street, Columbia	803-734-5200
State Emergency Response	SC DHEC 2600 Bull Street, Columbia	803-253-6448
FEDERAL		
Nearest EPA Office	EPA Region IV - Atlanta	404-874-0607
Nearest Coast Guard Office	US Coast Guard, 196 Tradd St.	843-724-7616

U.S. Coast Guard National Response Center — Chemtrec Emergency Response — Poison Center

800-424-8802 800-424-9300 800-922-1117

Arrangements with local police:
A copy of the contingency plan has been provided to the Charleston County Sheriff's Department. A letter of acceptance was provided for them to respond that they had received the plan.

Arrangements with local fire departments:
A copy of the contingency plan has been provided to the St. Andrews and City of Charleston Fire Departments. A letter of acceptance was provided for them to respond that they had received the plan.

Arrangements with local hospitals:
A copy of the contingency plan has been provided to Roper and St. Francis Xavier Hospitals. A letter of acceptance was provided for them to respond that they had received the plan.

Arrangements with local or state emergency response services:
A copy of the contingency plan has been provided to the St. Andrews Fire Department and the local office of SC Department of Health & Environmental Control. A letter of acceptance was provided for them to respond that they had received the plan.

PRIMARY EMERGENCY COORDINATOR

NAME: George McAbee, Facilities Manager **Office:** 843-556-8171 ext. 4452
ADDRESS: 2040 Savage Road, Charleston SC Home: 843-556-6955

FUNCTIONS/ACTIVITIES DURING EMERGENCY
Primary emergency coordinator will be responsible for assessment and control in the event of a fire, explosion or unplanned release of hazardous waste or hazardous waste constituents to the air, soil, or surface waters at this facility. The coordinator would provide the resources necessary to contain released materials, ensure the safety of employees and the public, and contact the proper emergency response officials.

SECONDARY EMERGENCY COORDINATOR

NAME: James Westmoreland, Division Director, Radiochemistry **Office:** 843-556-8171 ext. 4476
ADDRESS: 2040 Savage Road, Charleston SC **(PAGER):** 843-805-9100

FUNCTIONS/ACTIVITIES DURING EMERGENCY
Assist primary coordinator. In the event the primary coordinator is unavailable, the secondary coordinator would assume primary responsibilities for the event.

Facility Location	Facility #
2040 Savage Road, Charleston, SC 29414	SCR000002915

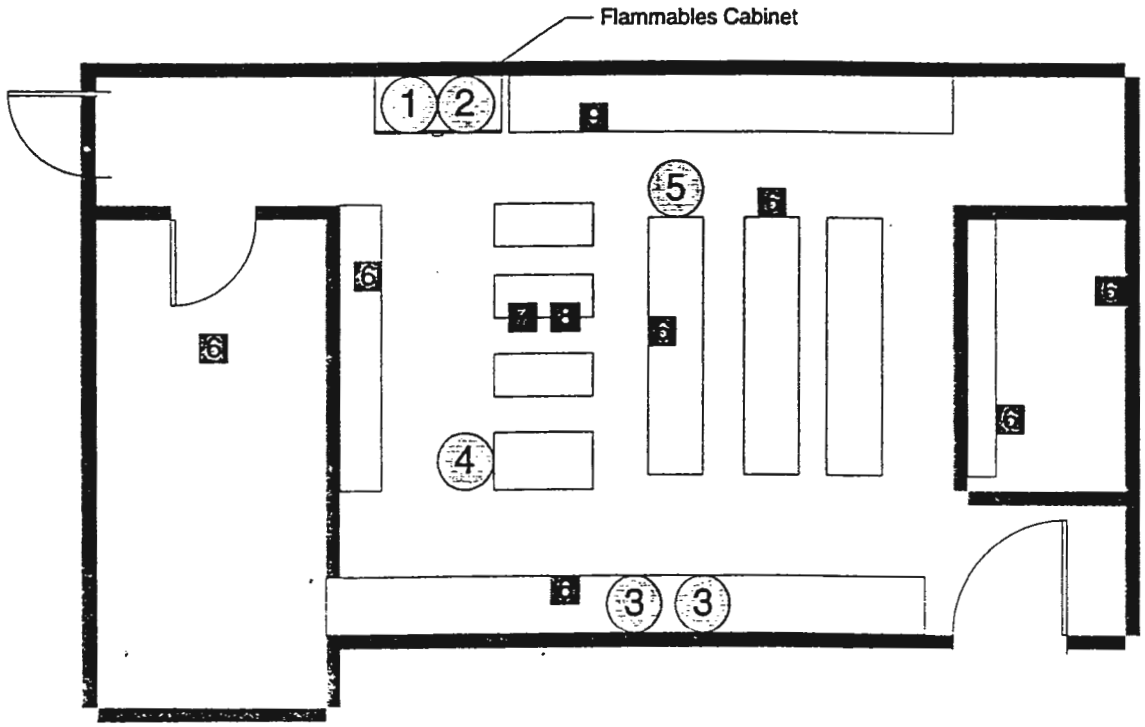
Facility Description
The facility is a full service environmental radiochemistry laboratory. The laboratory building has approximately 40,000 square foot of floor space for the laboratory equipment and office space. The building has card reader controlled access. Fire and security protection are provided on a 24 hour basis. Fire detectors and pull alarms are located throughout the facility. Additionally, some areas have temperature alarms to notify the 24 hour security service in the event of high temperature conditions not related to a fire. A facility diagram is attached to identify locations of emergency equipment.

Entrances Main entrances are identified on the facility diagram. The entrance to the waste management area is accessible to authorized personnel.	**First Aid Stations** Basic first aid stations for cuts & minor burns are located in various areas of the lab.	**Monitoring Equipment** Explosive meter, field gas chromatography equipment located in field services area.
Emergency Exits Exits are identified on the facility diagram.	**Guard Gates/Stations** Limited access facility	**Waste Storage Area** Identified on the facility diagram.
Fire Extinguishing System Individual fire extinguishers and automatic halon systems are identified on the facility diagram.	**Emergency Equipment Locations** Emergency equipment is identified on the facility diagram. The fire extinguisher is located adjacent the entrance/exit of the waste management area.	**Hazardous Materials Storage Area** Identified on the facility diagram.
Spill Control Equipment Absorbent, lime, granular carbon, spill booms, shovels and brooms are located in the waste management area. Closed trenches in the waste management area floor would provide containment in the event of a spill.	**Eye Wash Stations** Eye and full body wash station is located in the waste management area. Additional eye and full body wash stations are identified on the facility diagram.	**Communication System/Speakers** There is a public address system for announcements to all areas of the facility. Phones are located throughout the facility. A phone is located in the waste management area.
Evacuation Routes Evacuation routes and exits are shown on the attached diagram.	**Fire Systems/Alarms** Fire detectors are located throughout the facility. In the event an alarm is activated, a fire alarm sounds and a signal is sent to the security company.	**Fire Extinguishers** A hand held fire extinguisher is located in the waste management area. Other fire extinguisher locations are identified on the facility diagram.
Decontamination Equipment Decontamination would involve use of the same materials used for spill control listed above.	**Emergency Respirators** N/A	**Other**

APPENDIX 2

APPROXIMATE LOCATIONS OF LABORATORY SATELLITE ACCUMULATION AREA AND SAMPLE COLLECTION AREA CONTAINERS

#	Description of Collection area	Type of Container
1	Liquid Scintillation Vials (H-3, C-14)	open top steel drum with plastic liner
2	Liquid Scintillation Vials (Other Isotopes)	open top steel drum with plastic liner
3	Aqueous Radioactive Sample Residue	tight top drum (yellow)
4	Aqueous Non-Radioactive Sample Residue	tight top drum (non-yellow)
5	Radioactive Contact Waste (Non-Burnable)	open top steel drum with plastic liner
6	Radioactive Contact Waste (Burnable)	labeled cardboard boxes with plastic liner
7	Radioactive sludge sample residue	plastic container with secure lid
8	Non-radioactive sludge sample residue	plastic container with secure lid
9	Used resins	plastic or glass container with secure lid



APPENDIX 3

LSC Scintillation Cocktail Type_______________________ Drum

#_____________

APPENDIX 4

APPROVED DISPOSAL FACILITIES AND TRANSPORTERS

Low Level Radioactive Wastes

Company	Service	EPA Gen. ID #	DOT Reg. #
Bionomics, Inc.	Waste Broker	N/A	061995004014D
Chem Nuclear Services Inc.	Waste Broker/Disposal Site	SC004-8372-429	USDOT 059-357
Diversified Scientific Services Inc.	Mixed Waste Disposal Facility	TND98-210-9142	N/A
Perma-Fix of Florida, Inc.	Mixed Waste broker	FLD98-071-1071	N/A
Scientific Ecology Group	Rad Waste Consolidation Facility	TND98-215-7570	N/A
US Ecology, Inc.	Broker and Radioactive Waste Consolidation Facility	TND98-208-6969	061496005006E

Non Radiological Hazardous and Non-Hazardous Wastes

Company	Service	EPA Gen. ID #	DOT Reg. #
North American Environmental, Inc. (using JB HUNT and/or Envirotrans shipping services)	Waste Broker	N/A	JBHunt: ARD981908551 Envirotrans: GA0001040120 MC# 686560
Chemical Conservation Corp.	Disposal Facility	FLD98-055-9728	N/A
Fisher Industrial Service, Inc.	Disposal Facility	ALD98-102-0894	N/A

APPENDIX 5

SATELLITE ACCUMULATION AREA INSPECTION FORM

Area	*LSV*	*RCW*	*RA*	*RS*	*SPA*	*NR*
Is a sign present and legible?						
Is the area accessible?						
Is the area clean?						
Is any spillage evident?						
Containers						
Is there any leakage evident?						
Is there any rust which may cause container failure?						
Is there any physical damage to the container?						
Is the container bulging?						
Are the lids closed?						
Is there adequate segregation of incompatible materials?						
Labeling						
For hazardous materials does the comment read "hazardous waste- federal and sc law prohibits improper storage or disposal?						
Is an origin specified for the container?						
Is a waste category present?						
Epa waste code present?						
Equipment						
Are spill control and clean up materials available?						
Is a fire extinguisher charged and present?						
Is a phone available for emergency?						
Other comments:						

LSV: Liquid Scintillation Vials satellite accumulation area
RCW: Radioactive contact residue sample collection area
RA: Radioactive aqueous sample collection area
RS: Radioactive solid sample collection area
NR: Non Radioactive sample collection area

APPENDIX 6

SAMPLE AND WASTE DRUM DISPOSAL FORM

Drum ID#_____ Origin:_____ Matrix:_____

Accumulation Start Date:_____ Sample Date:_____ Lab ID_____

Final Classification:_____

Final Disposition:_____Disposition Date:_____

CONTENT LOG

Use if material is added to this drum that is not scanned into LIMS

Date	*Amount*	*Origin*	*Initials*

APPENDIX 7: SCDHEC Information Notice

South Carolina
DHEC
Department of Health and Environmental Control
2600 Bull Street, Columbia, SC 29201

Commissioner: Douglas E. Bryant

Board: Richard E. Jabbour, DDS, Chairman
Robert J. Stripling, Jr., Vice Chairman
Sandra J. McAlister, Secretary

Promoting Health, Protecting the Environment

William E. Applegate, III
John H. Burriss
Toney Graham Jr., MD
John B. Pate, MD

June 6, 1994

Bureau of Radiological Health

INFORMATION NOTICE

TO: S.C. Radioactive Material Licensees Authorized to Receive Samples for Analysis and Testing

FROM: James K. Peterson, Director
Division of Radioactive Material
Licensing and Compliance
Bureau of Radiological Health

SUBJECT: Disposal of Radioactive Wastes

This notice is intended to serve as a reminder to all licensees who receive samples (possibly containing radioactive material) for analysis, testing, research, etc. All samples received under the authority of your license should be disposed of by either (1) return to the sample generator, or (2) transfer to a facility specifically licensed to receive the waste. There are no other disposal options. Samples received under the authority of your license should not be stored or stockpiled at your facility.

You may not dispose of these samples via the sanitary sewer system, decay-in-storage, release to unrestricted areas, or by any other method except those mentioned in the above paragraph. Disposing of these samples at your facility in any manner would require you to obtain a waste disposal license from the Department. This type of license requires a substantial annual licensing fee.

Also, there are no established levels at which a sample can be classified as radioactive or non-radioactive. Therefore ALL samples received under the authority of your radioactive material license should be returned to the generator or transferred to a licensed disposal facility. Contact wastes may be disposed of under the authority of your specific license.

/sm

recycled paper

STANDARD OPERATING PROCEDURE

FOR UNCONTROLLED DOCUMENT

GAMMA SPECTROSCOPY SYSTEM OPERATION

(GL-EPI-I-001-Revision5)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Scott C. Moreland	10/15/99	SCOTT C. MORELAND
Process Owner	(sign and date)	(print name)
Richard M. Kinney	10/15/99	Richard M. Kinney
Technical Review	(sign and date)	(print name)
Robert L. Pilland	11/2/99	Robert L. Pilland
Quality Review	(sign and date)	(print name)
James B Westmoreland	11/2/99	James B Westmoreland
Approval and Authorization	(sign and date)	(print name)

SET II

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR GAMMA SPECTROSCOPY SYSTEM OPERATION

2.0 METHOD OBJECTIVE AND PURPOSE

This procedure establishes the method for the operation, calibration, and maintenance of the ND9900 Multichannel Analyzer System to obtain gamma spectra for samples containing gamma emitting radionuclides. This procedure applies to the operation of high energy (50 – 2000 KeV) gamma spectrometers.

3.0 DISCUSSION

3.1 A typical gamma-ray spectrometry system consists of a germanium detector (with its cryostat, liquid nitrogen dewar, and preamplifier) in conjunction with a detector bias supply, linear amplifier, liquid nitrogen level monitor, ADC (Analog to Digital Converter), AIM (Acquisition Interface Module), and a multichannel analyzer.

3.2 Germanium detectors used for the detection of gamma-rays are actually semiconductor diodes having a P-I-N (Positive-Intrinsic-Negative) structure in which the intrinsic (I) region is sensitive to ionizing radiation, particularly x-rays and gamma rays. Under the reverse bias applied, an electronic field extends across the intrinsic or depleted region. When gamma rays interact with the material within the depleted region of the detector, charge carriers (holes and electrons) are produced and are swept by the electric field to the P (positive) and N (negative) electrodes. This charge, which is proportional to the energy deposited in the detector by the incident gamma ray, is converted into a voltage pulse by an integral charge sensitive preamplifier.

The signal is then amplified and sent to an ADC where the signal is converted from an analog signal to a digital signal that can be interpreted by the central VAX computer. The digital signal is then sent to the AIM where the pulse is entered into a spectral channel corresponding to a particular gamma ray energy. The data is also sent to a central VAX computer, where the spectral data is stored and visually displayed. When acquisition is completed, the VAX computer quantifies the events recorded in each channel of the sample spectrum. The peaks of the spectrum, characteristic of both a particular radionuclide(s) and quantity of the radionuclide present in the sample, are identified by comparison to a nuclide library stored on a disk.

3.3 Prior to counting samples, the detector and associated electronics must be energy and efficiency calibrated. Energy calibration is performed by counting a radioactive source containing known gamma ray emitting radionuclides at a fixed amplifier gain. An energy calibration factor is then generated by determining the channel numbers corresponding to full energy peak centroids from gamma rays

emitted over the full energy range of interest from multipeaked and/or multinuclide radioactivity sources. Efficiency calibration is accomplished by counting a calibrated source of a particular geometry at a reproducible source to detector orientation. The measured emission rate of the calibration standard is then compared to the actual disintegration rate to determine the detector counting efficiency. The values for energy and efficiency calibration are maintained in configuration files that are referenced when analyzing samples.

4.0 DEFINITIONS

4.1 Efficiency: the percent of decay events from a standard radioactive source in a specific reproducible geometry that are seen and measured by a detector.

4.2 Traceable Calibration Standard: a calibrated radioactive source prepared from a standard reference material traceable to NIST (National Institute of Standards and Technology) or its equivalent.

4.3 Check Source: a radioactive source, not necessarily calibrated, which is used to confirm the satisfactory operation of the instrument.

4.4 ADC: analog to digital converter.

4.5 FWHM (Full Width Half Maximum). The full width of a gamma ray peak distribution measured at half the maximum peak height, measured above the continuum (background).

4.6 Dead Time: the time while the ADC is processing a pulse and is unable to process another pulse.

4.7 Geometry: a standard sample or source counting configuration (i.e., 20 mL vial, marinelli beaker, pertri dish, etc.) and its relationship to the detector.

5.0 PROCEDURES

Samples Counting:

5.1 Ensure the Daily QC Check has been performed and all parameters are in bounds, per section 9.3.

5.2 Ensure the sample is in a standard calibrated geometry and is clean.

5.3 From the Sample Counting System Main Menu, select 1) Sample Counting, to access the Sample Counting Menu.

5.4 Select 1) Count a Sample, to access the Alpha/Gamma Counting Menu.

5.5 Select 1) Samples.

5.6 Enter the Detector ID (i.e., Gamma 1).

5.7 Enter the Sample ID.

5.8 Enter the geometry used. If you are not sure of the available geometries, enter a ? to display the geometries currently available.

5.9 Enter the preset live time (in seconds).

5.10 Press "Return" to access the Parameter Editor and edit the Sample Counting Parameters:

Sample Identification:
Analyst's Initials:
Sample Quantity:
Sample Units:
Sample Date:
Detector ID:
Preset Live Time:
Sensitivity:

5.11 Press PF1 to exit the parameter editor.
5.12 Enter the appropriate nuclide library.
5.13 Enter "No" to examine/edit parameters.
5.14 Press "Return" to exit the Sample Counting Menu.

6.0 SAFETY, HEALTH, AND ENVIRONMENTAL HAZARDS

6.1 Individuals performing this procedure shall be aware of the precautions necessary for the proper handling of radioactive materials.

6.2 Follow the manufacturer's instructions for set-up, intercomponent connections, and preliminary testing of the equipment. Observe all of the manufacturer's limitations and cautions.

6.3 For HPGe (High Purity Germanium) detectors, the crystal may be allowed to warm to room temperature and subsequently recooled. These cycles should be minimized to extend the life of the crystal.

6.4 Ensure the detector coldfinger is submersed in liquid nitrogen at least 6 hours before applying a bias to the crystal.

6.5 Never exceed the manufacturer's recommended operating voltage for the detector. This may lead to detector damage.

6.6 Ensure the high voltage power supply is set to zero before energizing the unit. Turn on the high voltage power supply, press the reset button, and begin to slowly raise the voltage until the correct operating voltage is reached.

7.0 RECORDS MANAGEMENT

7.1 Quality control charts for instrument performance are kept on the VAX computer.

7.2 Instrument calibrations are kept in a file and are available for review.

8.0 REFERENCES

8.1 ASTM Annual Book of Standards, 1990, Volume 12.02, E-181.

8.2 Canberra NDSP Sample Counting Software.

9.0 STANDARDIZATION AND CALIBRATION

9.1 Energy and FWHM Calibration. This section shall be performed at initial instrument set up, after system maintenance that may effect the calibration, prior to efficiency calibrating, and whenever a problem is suspected. This section should be performed at least annually.

9.1.1 Position a calibration standard (containing ^{57}Co, 122 KeV and ^{88}Y, 1836 KeV as a minimum) on the detector and close the shield.

9.1.2 Following section 5.1, count the standard until a minimum of 10,000 counts have acquired in both the 122 KeV (^{57}Co) and 1836 KeV (^{88}Y) peaks.

9.1.3 From the Sample Counting System Menu, select 4) Create/ Delete/ Calibrate a Detector to access the Create /Delete /Calibrate Menu.

9.1.4 Select 1) FWHM and Energy Calibrate a Detector.

9.1.5 Answer the prompts:

9.1.5.1 Enter the name of the detector to be calibrated.

9.1.5.2 Enter "Yes" to use the spectrum acquired in 9/12 above.

9.1.5.3 Enter the ID of the standard used for calibration

9.1.5.4 Select "No" for hard-copy calibration plots.

9.1.5.5 Enter "Yes" to do a peak search after the calibration is completed.

9.1.5.6 If the Energy/FWHM calibration being performed is an initial calibration, enter "I", otherwise, enter "U" to update the present calibration parameters. If an initial calibration is performed, position the cursor over the 122 KeV and 1836 KeV peaks when prompted, and press "Return".

NOTE: The energy and FWHM calibration parameters will now be calculated and a graph will display for each. To clear the graphs, place the cursor arrow over the DISMISS key on the graph, press the left mouse button, then press "Return" to continue.

9.1.6 Review the calibration results displayed for energy and FWHM.

9.1.7 Submit the calibration report to the Supervisor (or designee) for review.

9.2 Efficiency Calibration. This section shall be performed during initial instrument set up, after system maintenance that may have effected the calibration, and/or annually.

9.2.1 Ensure the certificate files have been created for the calibration standard(s) to be used for efficiency calibration.

9.2.2 Position the traceable calibration source in the position to be calibrated (i.e., 20 ml vial, shelf 1) and close the shield.

9.2.3 Count the standard as a sample in accordance with section 5.1, ensuring at least 10,000 counts are attained in peak of interest.

9.2.4 Edit the sample parameters:

Sample ID:
Analyst's Initials:
Sample Quantity:
Sample Units:
Standard ID:
Sample Date:
Standard Date:
Detector ID:
Preset Live Time:
Sensitivity: Enter "5.0"

Press the PF1 key to exit the parameters editor.

9.2.5 After sample acquisition has been completed, from the Sample Counting System Main Menu, select 4) Create/ Delete/ Calibrate a Detector to access the Create/ Delete/ Calibrate Menu.

9.2.6 Select 3) Update Energy, FWHM, and Efficiency Calibration and answer the prompts.

9.2.6.1 Enter the detector to be calibrated.

9.2.6.2 Enter "Yes" to use the spectrum presently displayed in that detector configuration.

9.2.6.3 Enter the calibration standard ID.

9.2.6.4 Enter "Yes" to print out hard-copy plots of the calibration data.

9.2.6.5 Enter "Yes" to do a peak search after the calibration is complete.

9.2.6.6 Enter "Yes" to print a plot of the efficiency calibration curve.

9.2.6.7 Enter the name of the geometry calibrated.

9.2.6.8 Enter "E" to do an empirical fit of the efficiency data.

9.2.6.9 The graphs for Energy, FWHM, and Efficiency Calibration will now be displayed consecutively. After reviewing the graphs, clear them by placing the cursor arrow on the DISMISS key of the graph, pressing the left mouse key, and pressing "Return".

9.2.6.10 Compare new efficiencies to old efficiencies and ensure that they are within ± 10%.

9.2.7 Reposition the traceable calibration standard and repeat steps 9.2.2 and 9.2.3 using the correct and newly calibrated geometry. Compare the calculated activity of the recounted standard to the certified activity stated on the calibration certificate. These values should agree within ± 10% of each other. If the values do not agree within ± 10%, contact the Supervisor or designated alternate.

9.2.8 If the results do agree within ± 10%, place the activity comparison in the folder, complete the calibration checklist, and submit the data to the Technical Advisor for Instrumentation (or designee) for review.

9.3 Daily QC Checks. This performance check shall be performed daily prior to counting samples, following instrument maintenance which may effect the system calibration, or whenever a problem is suspected.

9.3.1 Position the appropriate calibration/check source on the detector in the proper shelf position.

9.3.2 Start data acquisition in accordance with section 5.1. Use a sample ID of QC_"Detector ID" (i.e., GC_GAMMA1). The QC prefix will automatically designate the sample as a QC sample and the results will be stored in the QC file for that detector.

9.3.3 After the completion of the data acquisition, a daily QC report is automatically generated. All of the predetermined parameters (i.e., ^{57}Co activity, ^{57}Co FWHM, etc.) are checked to verify that they are in bounds. If a parameter is out of bounds, a flag will be printed on the Daily QC Report indicating which parameter is out of bounds and whether the data point is out of bounds high or low. If any of the parameters are out of bounds, contact the Supervisor (or designee), investigate the cause and recount the standard if required by the Supervisor. If a recount is performed and a parameter is out of bounds a second time, contact the Supervisor and place the detector out of service. If no parameters are out of bounds, data and initial (or sign) the report and place it in the detector QC file.

9.4 Weekly Background Checks. This procedure should be performed at least once each calendar week.

9.4.1 Ensure the shield is empty and close the shield.

9.4.2 Start acquisition in accordance with section 5.1, entering:

The detector ID
Sample ID = BKG_"detector ID" (i.e., BKB_GAMMA1)
Sample geometry = 2L_MB
Preset Live Time = 60,000 seconds (or as otherwise determined_

9.4.3 Edit the Sample parameters:

Sample ID:
BKG_"detector ID"
Analyst's Initials:
Sample Quantity:
Sample Units:
BKG
Sample Date:
Today's Date:

Press PF1 to exit the sample parameters editor.

9.4.4 After acquisition is complete, review the data printout, checking for any shield contamination. If any contamination is noted, contact the Supervisor (or designee). Place the background in the detector background file.

10.0 CALCULAITONS

10.1 $$\text{Efficiency} = \frac{CPM_0}{DPMc}$$

Where: CPM_0 = observed cpm of the standard
DPM_c = certified disintegration rate of the standard

10.2 Decay Corrections

$$A_f = A_i * \exp^{-\frac{\ln 2}{t_{1/2}} * T}$$

Where: A_f = Final decay corrected activity
A_i = Original Activity
Exp = exponential (inverse natural log)
ln2 = natural log of 2
$t_{1/2}$ = half life of the isotope
T = decay time

10.3 Standard Deviation (sigma)

$$\text{Standard Deviation (s)} = \sqrt{\frac{N}{t}}$$

Where: N = Sample count rate (cpm)
t = Sample count time (minutes)

10.4 Minimum Detectable Activity (MDA) Calculation

$$MDA = \frac{2.71 + 4.66\sqrt{Sb}}{2.22 * V * E * A * T_s} * \exp^{-\frac{\ln 2}{t_{1/2}} * T}$$

Where: 2.71 = K factor (95% confidence level)

4.66 = statistical factor (95% confidence level)
S_b = standard deviation of the background
2.22 = conversion factor (dpm/pCi)
T = difference in time between sample date/time and count date/time
V = sample volume or weight
E = counting efficiency
A = isotopic abundance
T_s = sample count time

10.5 Activity Calculation

$$\text{ACTIVITY (pCi/unit)} = \frac{C_N}{2.22 * V * E * A * T_s} * \exp^{\frac{-\ln 2}{t_{1/2}} * T}$$

Where: A = isotopic abundance
C_N = net sample count (area)
T_s = sample count time
T = time between sample date/time and count date/time

11.0 QUALITY ASSURANCE REQUIREMENTS

11.1 Efficiency calibration shall be performed with a traceable calibration standard that closely approximates the sample counting geometry.

11.2 All data generated by this procedure shall be reviewed by the Section Leader (or designee)

11.3 The calibration portions of this procedure shall be performed at initial installation, following system maintenance which may effect the electronics, when a problem is suspected, and at the following frequencies:

11.3.1 Efficiency Calibration – verified or reperformed annually

11.3.2 Energy and FWHM Calibration – annually

11.3.3 Detector backgrounds – once each calendar week

11.4 The operator performing this procedure shall ensure that all daily instrument checks have been performed and meet performance criteria before counting any samples.

APPENDIX 1

APPENDIX 2

STANDARD OPERATING PROCEDURE

FOR

UNCONTROLLED DOCUMENT

BECKMAN LS 6000/6500

(GL-EPI-E-I-004-Revision 2)

PROPRIETARY INFORMATION

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	8/10/99	Tim C. Winters
Process Owner	(Sign and Date)	(Print Name)
	8/10/99	Richard M. Kinney
Technical Review	(Sign and Date)	(Print Name)
	8/16/99	Genie Bost
Quality Review	(Sign and Date)	(Print Name)
	8/16/99	James B Westmoreland
Approval and Authorization	(Sign and Date)	(Print Name)

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TABLE OF CONTENTS

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4.4 Quench. Something that interferes with either the production or the detection of a light pulse in or from a scintillation sample. For more information refer to the instrument reference manual, page R 3-64.

4.5 H Number. A parameter which indicates the level of quench in a sample using the energy distribution pattern of Compton electrons induced in the sample by an external Cs-137 source. This value is then used to automatically adjust the counting region to exclude unwanted background events.

4.6 Cocktail. The solution in which samples are placed for measurement in a liquid scintillation counter. Solvents and scintillators are the major components of scintillation cocktails.

4.7 Dual Label. Two different radionuclides in a sample.

4.8 Single Label. One radionuclide in a sample.

4.9 Scintillator. The substance in the scintillation cocktail which absorbs decay energy transferred from the solvent and emits light energy (photons) approximately proportional in intensity to the decay energy.

4.10 E^2/B. Figure of Merit, FOM, is the measure of the instrument sensitivity based on the measured counting efficiency and background.

5.0 SAFETY, HEALTH AND ENVIROMENTAL HAZARDS

5.1 Personnel performing this procedure shall be aware of the risks associated with the handling of radioactive materials.

5.2 Wipe all sample vials clean with a dry, lint-free cloth prior to placing them in the sample cassettes. This will minimize the spread of contamination to the instrument and other samples, as well as minimize any light interferences caused by dirt on the vial.

6.0 EQUIPMENT LIST

6.1 Beckman Liquid Scintillation Counter.
6.2 Parallel Printer.
6.3 Sample cassettes.
6.4 Protocol flags.

7.0 MATERIALS LIST

7.1 NIST traceable calibration standard(s), such as ^{3}H, ^{14}C, etc..
7.2 Quench standard sets. These should be made for each isotope(s) to be counted and in each cocktail to be used.
7.3 20 ml unquenched carbon-14 calibration standard.
7.4 20 ml unquenched tritium (^{3}H) calibration standard.
7.5 20 ml unquenched background standard.

8.0 STANDARDIZATION AND CALIBRATION

8.1 Daily Instrument Checks. This procedure will be performed after initial startup, daily when counting samples, weekly when idle, or whenever a problem is suspected.

8.1.1 Ensure the sample cassette with the protocol flag "AUTO" contains the unquenched H-3 standard in the first position, followed by sample cassette "1" containing the Background, H-3, and C-14 standards.

8.1.2 Place the cassettes in the instrument with the protocol flag positioned towards the rear of the instrument.

8.1.3 Press the Green "AUTO START" button on the keyboard to begin the counting procedure.

8.1.4 Ensure the printer is selected for the liquid scintillation counter and observe the messages printed out at the end of the daily checks. The message printed should say "Calibration Successful". If any other message is received, contact the Group Leader (or designee) and place the instrument out of service.

8.1.5 Enter the data received from the analysis of the background and C-14 standard into the appropriate instrument file on the QC report. The QC report will automatically compare the last results entered to the existing QC limits for the database and flag any out of specification results. If any results are out of specification re-perform section 8.1 of this procedure.

8.2 Establishing Quench Curves. A quench curve correlates the known DPM of a standard to some measured amount of chemical quench. The quench curve is generated by counting a series of standard samples, each containing the same amount of nuclide and varying amounts of the quenching agent. Generally, the

STANDARD OPERATING PROCEDURE

FOR

UNCONTROLLED DOCUMENT

LB-5110W GROSS ALPHA/BETA COUNTER

OPERATING INSTRUCTIONS

(GL-EPI-I-005 Revision 2)

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Quality Review	(sign and date)	(print name)
Approval and Authorization	(sign and date)	(print name)

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TABLE OF CONTENTS

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1.0 Method Purpose and Objective

This standard operating procedure provides the necessary instructions to operate the low background, high efficiency, gas flow proportional counter, LB5100/W.

2.0 Discussion

The Tennelec Model LB5100/W Automatic Alpha/Beta Counting System is designed to measure very low levels of alpha and beta radiation in environmental samples. The detector assembly consists of a dual proportional detector assembly. This consists of a large area guard detector and a thin window sample detector. The guard detector measures the extraneous radiation (background) which typically comes from cosmic anticoincidence mode with the sample detector to eliminate the unwanted background events. The result is an extremely low background counting system. The sample detector is a gas flow type proportional counter. When samples are placed under the thin detector window the sample radiates a fraction of its total alpha and beta particles towards the detector. Once the beta of alpha particle enters the detector, the particle ionizes the gas molecules contained within the volume of the detector. The electrical field present in the detector chamber accelerates the charged particles, causing secondary ionization's introducing more charged particles into the detection chamber. The total number of ions present in the chamber is thus a multiple of the ions formed by the incident radiation. The resulting charge built up on the anode is then processed to determine if the incident particle was an alpha of a beta. This is accomplished by measuring the relative intensity of the voltage pulse. The separation between alpha and beta particles is easily accomplished since the alpha particle produces a pulse that is approximately 40 times larger than that produced from a beta particle.

3.0 Definitions

3.1 **Efficiency-** the percent of decay events from a standard source that are seen and measured by a detector.

3.2 **Crosstalk-** the detection of alpha events in the beta channel or the detection of beta events in the alpha channel during simultaneous counting.

3.3 **Simultaneous Counting-** the measurement of both gross alpha and gross beta activity at the same time.

3.4 **Proportional Counter-** a gas filled radiation counter tube operated in the range of high voltage in which the total charge collected for each ionizing event is proportional to the number of ion pairs formed in the tube by the initial event.

3.5 **Self Absorption-** absorption of radioactive emissions by the solids contained on the counting planchet, thereby preventing the emission from reaching the detector.

3.6 **Check Source-** a radioactive source, not necessarily calibrated, which is used to confirm the satisfactory operation of the instrument.

4.0 Health Safety and Environmental Hazards

4.1 P-10 gas (or equivalent) must be supplied to the machine at .1-.3 (scfh), for a minimum of 20 min. before starting a counting cycle.

4.2 Gas flow exceeding .3 scfh or gas pressure above 10 psi to the flow meters may result in detector window rupture or deformation.

4.2.1 If the parameters are exceeded, quickly correct the cause, then perform an instrument background and QA check to determine detector performance. Note the date, time, cause, action taken, and any other pertinent information in the maintenance log. Inform the group leader of the occurrence.

4.3 Bumping or moving the LB5100/W while counting can result in several problems.

4.3.1 Noise generated can greatly interfere with sample counting already in progress.

4.3.2 Planchettes can become misaligned in the holders, and subsequently damage the detector windows.

4.4 The thin detector windows are easily damaged by hot or fuming samples.

4.5 Samples must not protrude above the surface of the carrier. Smears have been known to scratch, even cut detector windows.

5.0 Instrumentation

5.1 Oxford Model LB5100/w Gross Alpha/Beta Counting System with PC capable of supporting Windows^{tm} 3.1 and Microsoft Excel^{tm}.

5.2 Printer. Recommend 24 pin dot matrix or better.

5.3 Gas Regulator. A dual stage regulator is recommended.

6.0 Apparatus and Materials

6.1 **Radioactive Check Sources.** Containing an alpha and/or beta emitting isotope(s).

6.2 **Traceable Calibration Standard.** NIST traceable standard based upon the isotope to be measured.

6.3 **P-10 Gas** (10% Argon/90% Methane).

7.0 Calibrations

7.1 Instrument Window Settings:

This procedure is used to adjust the discriminator window to the optimum setting for the simultaneous measurement of alpha and beta particles and may be omitted if calibrating for only alpha or beta counting. The Procedure for determining discriminator window settings is explained in detail in Section 4.3 (pg. 56-61) of the LB5100/W Low-Background System Operating Manual provided by Oxford Instruments.

7.2 Self Absorption Correction

The Procedure for correcting for self-attenuation/absorption is explained in detail in Section 4.8 & 4.9 (pg. 67-70) of the LB5100/W Low-Background System Operating Manual provided by Oxford Instruments.

7.3 Determining the Operating Voltage Plateau

7.3.1 Select "LB5100", "Edit Parameters", "(The type of Plateau to be performed)", "OK". The Plateau Presets will be displayed. Select the parameters desired for the Plateau, then chose "OK". Follow the macro provided user prompts during this process.

7.3.2 After the Plateau is completed the data is placed on an Excel Spreadsheet and graphed. The optimum operation voltage for beta counting, is approximately 50 volts above the knee of the curve. The knee is determined by drawing straight lines along the rising slope and the plateau portions of the curve. The knee is the point where these two lines intersect. The operating voltage is typically the first point on the plateau (flat portion of the curve). This will yield the good efficiency with the lowest background. Selecting a voltage above that may result in increased efficiencies but will increase the background values for the beta channel.

Since the alpha voltage may be increased without increasing the background significantly, the operating voltage for alpha counting will be the mid point of the alpha plateau.

8.0 Procedures

8.1 Startup

8.1.1 The LB5100/W is normally left in a running condition, although the monitor is turned off over night to prevent "burn-in", or the screen saver option may be selected to provide protection. In the case, that the entire unit is off, simply lift the rocker switch on the back of the counting unit to the "on" position. This will supply power to all of the instrument components, and display the LB5100/W main operating menu bar above the picture provided by Oxford Instruments.

8.2 Starting a Count

8.2.1 Edit the counting parameters for the counting program to be used by selecting "LB5100", "Edit Parameters", and then the type of count to be performed.

8.2.2 Place a Group Carrier (A-J) on the right side of the sample changer. Place the sample carriers to be counted above the group carrier. Repeat this process for additional counting groups or place the "END" carrier on top of the carriers to be counted.

8.2.3 Select "LB5100", "Start Count," "(Type of count to be performed, normally Generic)," "Letter of group to be counted," then highlight the file name. Name the file to be counted in the following format (all by convention, for consistency) Month, Underscore, Date, Underscore, Sequential count for that day, Period, xld (i.e. 6_14_1.xld). Detector QC checks may be named 6_14bac.xld or 6_14eff.xld. Choose "Run".

8.2.4 Enter the file name into the Runlog Dialog Box that appears, then enter the sample IDs and the users initials. Complete the prompted inputs then repeat this process or choose "Cancel".

8.2.5 Enter the file name and description on the notebook provided next to the computer. This is done to keep track of the next sequential file name.

8.2.6 The count in progress may be monitored by selecting "LB5100", "Unit Status".

8.3 Daily Checks

8.3.1 Ensure the daily QA and Background checks are completed (explained below) during the first part of the workday. The results of this daily check are maintained in the Gas Flow Proportional Counters Binder. The control charts provide a means to evaluate detector performance, conformance, and trends.

8.3.1.1 *Background Check*: Select the background carrier to be used. Background is normally counted for 60 minutes for a daily check to allow some alpha counts to be accumulated, due to the very low count rate. Start the count per section 5.2 above.

8.3.1.2 *QA check*: Select the Alpha and Beta Source Carriers used. The count of Alpha and Beta for the applicable carrier should exceed 10,000 counts. Start the count per section 5.2 above.

8.3.1.3 The daily checks can be printed out by using the macro provided. Select the icon that looks like a date book. When prompted, select the name of the Background file. When prompted select the name of the QA check file. The daily check will then be printed.

8.3.1.4 Any Background or QA check that is flagged by the control chart program should be evaluated to determine the cause. The problem should be corrected prior to counting any samples for that day. In either case, the group leader should be consulted to give the final decision on actions to be taken.

8.4 Weekly Checks

8.4.1 Prior to processing samples, ensure the instrument background for that week has been determined. The instrument background is determined weekly using the same procedure as for the Daily Background Check except it is counted for 480 minutes. This value is written on the LB5100/W status sheet laminated next to the LB5100/W computer. The weekly value for background can then be used by the analyst on method spreadsheets to correct for background.

8.4.2 Place a Group Carrier and the source on the sample changer. Proceed to the Starting a Count Section (5.2).

8.5 Viewing Data

8.5.1 Counted data may be obtained by selecting "LB5100", "Data Output", and then the file name. Follow macro provided prompts to obtain the data.

9.0 Quality Assurance Requirements

9.1 Efficiency calibration shall be performed with a traceable calibration standard that closely approximates the sample counting geometry.

9.2 All data generated by this procedure shall be reviewed by the Section Head or Designee.

9.3 The calibration portions of this procedure shall be performed at initial installation, on a 12-month frequency, after changing detectors, following system maintenance which may effect the electronics, or when a problem is suspected.

9.4 The operator performing this procedure shall ensure that all daily instrument checks have been performed and meet performance criteria before counting any samples.

10.0 References

10.1 "Model LB-5100/W, Low-Background System User's Manual," Oxford Instruments Inc.

STANDARD OPERATING PROCEDURE

UNCONTROLLED DOCUMENT

FOR

LB-4110 GROSS ALPHA/BETA COUNTER

OPERATING INSTRUCTIONS

(GL-EPI-I-006 Revision 1)

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Process Owner (sign and date)	(print name)
6/8/99	Joe Davis
Technical Review (sign and date)	(print name)
6/8/99	Genie Bost
Quality Review (sign and date)	(print name)
6/8/99	James B Westmoreland
Approval and Authorization (sign and date)	(print name)

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TABLE OF CONTENTS

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2.4 To insure the proper operation of the instrument, routine quality assurance (QA) checks are performed on the instrument. These checks consist of a daily background and efficiency check, the data is then archived and then analyzed to ensure that all detectors are operating within specifications.

3.0 Definitions

3.1 **Efficiency-** the percent of decay events from a standard source that are seen and measured by a detector.

3.2 **Crosstalk-** the detection of alpha events in the beta channel or the detection of beta events in the alpha channel during simultaneous counting.

3.3 **Simultaneous Counting-** the measurement of both gross alpha and gross beta activity at the same time.

3.4 **Proportional Counter-** a gas filled radiation counter tube operated in the range of high voltage in which the total charge collected for each ionizing event is proportional to the number of ion pairs formed in the tube by the initial event.

3.5 **Self Absorption-** absorption of radioactive emissions by the solids contained on the counting planchet, thereby preventing the emission from reaching the detector.

3.6 **Check Source-** a radioactive source, not necessarily calibrated, which is used to confirm the satisfactory operation of the instrument.

4.0 Health, Safety and Environmental Hazards

4.1 P-10 gas (or equivalent) must be supplied to the machine at .1-.3 (scfh), for a minimum of 20 min. before starting a counting cycle.

4.2 Gas flow exceeding .3 scfh or gas pressure above 10 psi to the flow meters may result in detector window rupture or deformation.

4.2.1 If the parameters are exceeded, quickly correct the cause, then perform an instrument background and QA check to determine detector performance. Note the date, time, cause, action taken, and any other pertinent information in the maintenance log. Inform the group leader of the occurrence.

4.3 Bumping the LB4110 while counting, can result in several problems.

1.0 Method Objective and Purpose

This method describes the operation of the Tennelec LB4110 Alpha/Beta Counting System for routine sample analysis. The operation includes access to the computer operating system, calibration, performance checks, starting of data collection, and the printing of data plots and reports.

2.0 Discussion

2.1 The Tennelec LB4110 Alpha/Beta Counting System is a multiple detector unit, designed to measure very low levels of alpha and beta radiation in environmental samples. The sample detectors are gas flow type proportional counters, each consisting of a dual proportional assembly with a large area guard detector and a thin window sample detector. The guard detector measures the extraneous (background) radiation which typically comes from cosmic radiation, lead x-rays, or gamma radiation, and is operated in an anticoincidence mode with the sample detector to eliminate unwanted background events. The result is an extremely low background counting system. When samples are placed under each thin detector window, the sample radiates a fraction of its total alpha and beta particles towards the detector. Once the alpha or beta particle enters the detector, the particle ionizes the gas molecules contained within the volume of the detector. The electrical field present in the detector chamber accelerates the charged particle, causing secondary ionization that introduces more charged particles into the detection chamber. The total number of ions present in the chamber is thus a multiple of the ions formed by the incident radiation. The resulting charge built up on the anode is then processed to determine if the incident particle was an alpha or a beta. This is accomplished by measuring the relative intensity of the voltage pulse. The separation between alpha and beta particles is easily accomplished since the alpha particle produces a pulse that is approximately 40 times larger than that produced by a beta particle.

2.2 Calibration of the LB4110 is accomplished in three basic steps, which are: determining the operations voltage, system calibration (which calculates and stores alpha and beta efficiencies, crosstalk values and discriminator window values), and measuring backgrounds for alpha, beta and simultaneous alpha/beta channel counting configurations.

2.3 The efficiency of the gross alpha/beta counters is adversely affected by the absorption of alpha and beta particles from samples containing increased amounts of solids (greater than 5 mg/cm^2). A series of standards containing known amounts of a radioactive standard are prepared containing various amounts of solids. The relative efficiency of each standard is measured, a correction factor calculated, and a curve is drawn of absorption correction factor versus sample solids.

4.3.1 Noise generated can greatly interfere with sample counting already in progress.

4.3.2 Planchettes can become misaligned in the holders, and subsequently damage the detector windows.

4.4 The thin detector windows are easily damaged by hot, or fuming samples.

4.5 Samples must not protrude above the surface of the carrier. Smears have been known to scratch, even cut detector windows.

5.0 Instrumentation

5.1 Oxford Model LB-4110 Gross Alpha/Beta Counting System.

5.2 Printer. Recommend a 24 pin dot matrix or better.

5.3 Gas Regulator. Recommend a dual stage regulator.

6.0 Apparatus and Materials

6.1 **Radioactive Check Source.** Containing an alpha and/or beta emitting isotope(s). The actual isotope is dependent on the radionuclide to be measured. Normally a SR-90/Y-90 or Th-230 source is used.

6.2 **Traceable Calibration Standard.** NIST traceable standard based upon the isotope to be measured.

6.3 P-10 Gas (10% Argon/90% Methane)

7.0 Calibrations

7.1 Setting Threshold Levels and Determining Crosstalk

7.1.1 Adjusting Window settings is performed during method calibration and should not be performed unless the operator has a through understanding of the consequences of changing window settings. To gain an understanding of the term "Crosstalk", the operator should read Section 5.2 (pg19) of the LB4100 Low Background Counting System Instruction Manual.

7.1.2 To adjust the Counting windows refer to Section 2.3.4 (pg5) and Section 4.3.3(pg12) of the LB4100 Low Background Counting System Instruction Manual. Record the setup parameters on the LB4100 Instrument Settings form.

7.2 Performing a Plateau

7.2.1 Plateau generation is normally performed during method calibration. To perform a plateau, refer to Section 2.3.7 (pg8) of the LB4100 Low Background Counting System Instruction Manual.

7.2.2 After the Plateau is completed the data may be printed or graphed from the plateau counting page. The optimum operation voltage. for beta counting, is approximately 50 volts above the knee of the curve. The knee is determined by drawing straight lines along the rising slope and the plateau portions of the curve. The knee is the point where these two lines intersect. The operating voltage of typically the first point on the plateau (flat portion of the curve). This will yield the good efficiency with the lowest background. Selecting a voltage above that may result in increased efficiencies but will increase the background values for the beta channel Since the alpha voltage may be increased without increasing the background significantly, the operating voltage for alpha counting will be the mid point of the alpha plateau. Include a printout of the plateau with the operating voltages in the calibration package.

8.0 Procedures

8.1 Startup

8.1.1 The LB4110 is normally left in a running condition, although the monitor is turned off over night to prevent "burn-in". In the case, that the entire unit is off, simply lift the rocker switch on the front of the counting unit to the "up" position. This will supply power to all of the instrument components. The LB4110 software is started by double clicking on the LB4000 icon in the Applications window in program manager.

8.2 Weekly Checks

8.2.1 Prior to processing samples, ensure the instrument background for that week has been determined and entered into the control chart system prior to sample counting. The instrument background is determined weekly

using the same procedure as for the Daily Background Check (below) except it is counted for 500 minutes.

8.3 Daily Checks

8.3.1 Ensure the daily QA and Background checks are completed (explained below) during the first part of the workday and entered into the control chart program. The results of this daily check are maintained in the Gas Flow Proportional Counters Binder. The control charts provide a means to evaluate detector performance, conformance, and trends.

8.3.1.1 *Background Check*: Place a contamination free planchet into each detector holder. Background is normally counted for 60 minutes for a daily check to allow some alpha counts to be accumulated, due to the very low count rate. Start the count per section 5.6 below.

8.3.1.2 *QA check*: Place the prepared check sources in the applicable detector holders. The count of Alpha and Beta for the applicable carrier should exceed 10,000 counts. Start the count per section 5.6 below.

8.4 Starting a Count

8.4.1 To insert samples into the counting unit, refer to Section 4.1 (pg10) of the LB4100 Low Background Counting System Instruction Manual.

8.4.2 From the LB4110 software screen, toggle to page 3 by pushing the page up or page down keys.

8.4.3 Determine the sample configuration to count the samples (eff.cnf). Push F7 (Load configuration) and type in the applicable configuration.

8.4.4 The default settings for printing and saving information can be checked by pressing ALT+X. More information about these settings are contained in Section 4.3 paragraph 7 (pg11) of the LB4100 Low Background Counting System Instruction Manual.

8.4.5 The following lab guidelines should be followed to ensure consistency.

8.4.5.1 All counting data is to be saved into the directory "D:\LB4000\DATA" (A-D drawers), "D:\LB4000\DATA\EF"

(E,F,&G drawers), "D:\LB4000\DATA\IJKL" (I-L drawers).

8.4.5.2 Background counts are to be saved under the file name BKG. Efficiency counts are to be saved under the file name EFF.

8.4.6 The Sample ID's and parameters may be entered prior to the start of the count or during a count.

8.4.7 To start the count refer to Section 4.3.1 (pg11) of the LB4100 Low Background Counting System Instruction Manual.

8.5 Self Absorption Correction

8.5.1 For certain methods the operator is required to correct for the self-absorption effects due to varying sample weight. The Procedure for correcting for self attenuation/absorption is explained in the applicable method.

9.0 Quality Assurance Requirements

9.1 Efficiency calibration shall be performed with a traceable calibration standard that closely approximates the sample counting geometry.

9.2 All data generated by this procedure shall be reviewed by the Section Head or Designee.

9.3 The calibration portions of this procedure shall be performed at initial installation, on a 12-month frequency, after changing detectors, following system maintenance which may effect the electronics, or when a problem is suspected.

9.4 The operator performing this procedure shall ensure that all daily instrument checks have been performed and meet performance criteria before counting any samples.

10.0 Records Management

10.1 Calibration packages are reviewed by the Technical Advisor for Instrumentation.

10.2 Daily checks are reviewed by the Technical Advisor for Instrumentation.

11.0 References:

11.1 "LB4100 Low Background Counting System Instruction Manual", Oxford Instruments Inc.

STANDARD OPERATING PROCEDURE

FOR

LUDLUM LUCAS CELL COUNTER

(GL-EPI-E-I-007 REVISION 2)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

Process Owner	10/11/99 (Sign and Date)	Tim C. WINTERS (Print Name)
Technical Review	10/14/99 (Sign and Date)	Richard M. Kinney (Print Name)
Quality Review	10/15/99 (Sign and Date)	Robert L. Rillard (Print Name)
Approval and Authorization	10/15/99 (Sign and Date)	James B Westmoreland (Print Name)

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR LUDLUM LUCAS CELL COUNTER

2.0 PURPOSE

The purpose of this document is to give the analyst a summary of the operation of the Lucas Cell Counter. The analyst should refer to the users manual for more detailed information.

3.0 DISCUSSION

General Engineering Laboratories, Inc. (GEL) has several Ludlum scalers that are connected to Radon Flask Counters (Model 182). The systems are employed to count lucas cells that have been prepared according to specified analytical procedures. The components utilized in this procedure are available from Ludlum Measurements, Inc. in Sweetwater Texas.

4.0 DEFINITIONS AND DESCRIPTION OF CONTROLS

4.1 See Appendix 1 for a diagram of the Ludlum front panel.

4.2 The "Power Switch" is a 4-position switch. "Off" indicates that the power is off. "Line" indicates that the line power is 115 volts Hz and the power is on.

4.3 The "Count" button resets and starts the scaler counting. The scaler turns off automatically when the preset count is reached.

4.4 The "Count Lamp" is a red light that indicates the scaler is counting.

4.5 The "Hold" button pauses the scaler without resetting the count. Pressing the "hold" button again will resume the count.

4.6 The "Meter Readout" displays the high voltage while the power switch is in the "line" position.

4.7 The "Discriminator" potentiometer is a 1-turn pot used to set the detector discriminator level. This potentiometer is set at the factory and should be turned without first checking with the Senior Analyst or Group Leader.

4.8 The "High Voltage" potentiometer is used to set the detector high voltage as determined by the plateau calibration. Refer to Section 4.2 for specific calibration instructions. The instrument will support up to 1500 volts.

4.9 The "Minutes" thumbwheel settings are 2-decade switches for setting the preset counting time. The time base is minutes from 0 to 99 with multiples of 0.1, 1 or 10.

4.10 The "Detector Input" is a series "C" coaxial connector used to connect the lucas cell detector with the scaler.

4.11 The "Count" readout is a 6-decade LED, which indicates the total counts.

5.0 PROCEDURE

5.1 Place the Lucas Cell in the Radon Flask Counter and center the cell on the photomultiplier tube. Gently press the top portion of the flask counter down onto the base and turn the thumbscrews to tighten the unit in place. Allow the unit to light adjust for a minimum of 2 minutes.

5.2 Select "line" operation with the power switch.

5.3 Select an appropriate count scale by turning the "minutes scale" as follows:

Time Interval	Multiplier Setting
1-99 minutes	X 1
100-990 minutes	X 10

5.4 Select an appropriate count interval by dialing in the corresponding time in minutes. (Example, for a 30 minute count dial in "3" for the left dial and "0" for the right dial with the minutes scale at X 1.)

5.5 Press the "hold" button and release. This will reset the counter.

5.6 Press the "count" button and release. This will begin the count. A red line will activate indicating that the count is in process.

5.7 When the count is completed, record the number present on the readout on the run log. (Appendix 2.)

5.8 Remove the counting cell from the counter by loosening the thumbscrews on the sides of the flask counter.

6.0 QUALITY CONTROL

6.1 **Daily Checks:** Each day the instrument is used the following checks are performed to ensure the consistent performance of the instrument.

6.1.1 **Instrument Performance Check:** Place the source counting cell in the flask counter and count for 1 minute. Record the gross counts in the instrument run log. Enter the gross counts into the QC Menu of the MicroVax 3100. (See "QC Data Entry on the MicroVax 3100," GL-EPI-

E-I-008, for instructions on using the software.) The QC program will compare the entered results with the historical 2 and 3 sigma limits. The report will flag an out of control condition.

6.1.2 **Daily Background Check:** Place the appropriate Radon counting cell into the counter and count for a period of time equal to the duration of the sample count. Record the gross counts per minute in the instrument run log. Enter the gross counts into the QC Menu of the MicroVax 3100. (See GL-EPI-E-I-008.) The QC program will compare the entered results with the historical 2 and 3 sigma limits. The report will flag an out of control condition.

6.2 **Instrument Calibration:** The instrument will be calibrated in accordance with GL-EPI-E-A-008 on an annual basis, or whenever the daily checks indicate a condition that warrants recalibration.

7.0 Safety, Health and Environmental Hazards

Following this procedure poses no safety, health or environmental hazards.

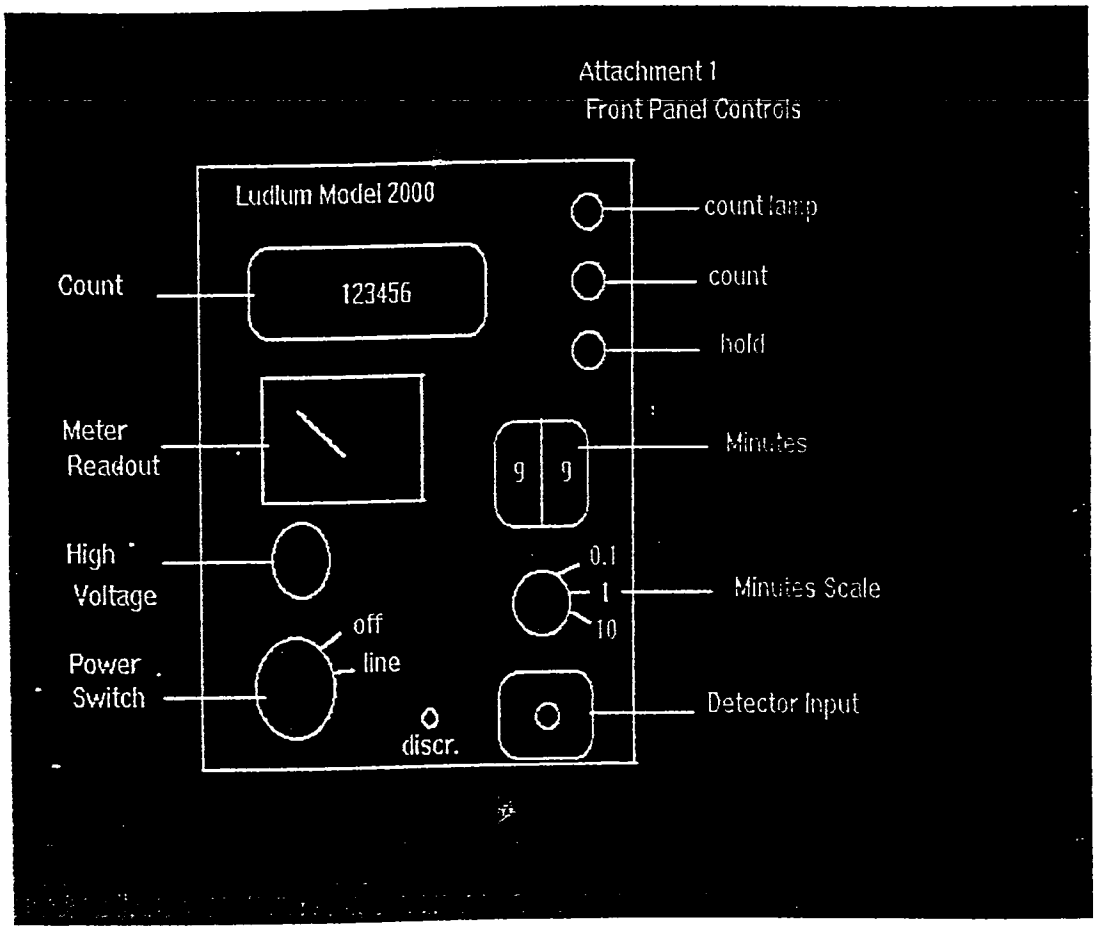
8.0 Records Management

8.1 Quality control charts for instrument performance are kept on the MicroVax 3100.

8.2 Instrument calibration records are kept in a file and are available for review.

APPENDIX 1

LUDLUM MODEL FRONT PANEL



APPENDIX 2

LUCAS CELL INSTRUMENT RUN LOG

Analysis ID	Start Count Date/time	Analyst Initials	Cell #	Det #	Count Time(min)	Total Counts

Reviewed By:_____ **Date:**_____

STANDARD OPERATING PROCEDURE

FOR UNCONTROLLED DOCUMENT

VAX/VMS QUALITY CONTROL SOFTWARE PROGRAM

(GL-EPI-I-008 Revision 2)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of the General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Process Owner (sign and date) 070699	Robert D Tinnm (print name)
Technical Review (sign and date) 07/06/99	Scott C. Moreland (print name)
Quality Review (sign and date) 7/6/99	Janie G. Bost (print name)
Approval and Authorization (sign and date) 7/8/99	James B. Westmoreland (print name)

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TABLE OF CONTENTS

SECTION PAGE

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1. Method Objective and Purpose

This procedure describes the use of the VAX/VMS Quality Control Package employed for the monitoring of analytical quality control samples (i.e. blanks, LCS, MS, etc.) and tracking the routine instrument checks performed on counting room instrumentation.

2. Discussion

2.1 Quality Control for analytical analyses performed in the Radiochemistry Section requires the complete monitoring and documentation of all quality control samples performed during routine operations. The quality control samples monitored include not only the individual QC samples performed with each batch, but also the instrument checks which are performed on a routine basis to ensure proper instrument operation prior to the analysis of any samples. The QC Menu software developed for the VAX Workstation utilizes the Canberra/Nuclear Data VAX/VMS Quality Assurance Package (07-0323) to perform these monitoring and analytical functions. The software is run using command procedures written in DCL (Digital Command Language). The command procedure as written provides the user with a menu driven interface facilitating an ease of operation.

2.2 The features present in the package include the ability to add, plot, reject, and/or delete results to or from the database. The quality assurance files are created manually to contain any parameter required for monitoring.

3. Definitions

3.1 **QC Menu Software.** The command procedure developed by and for EPI for the purpose of monitoring QC parameters in all phases of sample analysis.

3.2 **DCL.** The programming language developed by Digital Equipment Company for use on VAX/VMS operation platforms.

3.3 **QC Report.** An automatically generated report which compares the last entry into the QC data record with the user defined limits (either n-sigma or boundary mode).

4. Procedure

This procedure assumes the operator possess a minimal proficiency with the use of the VAX Workstations.

4.1 If in the counting procedures, exit to the command prompt.

4.2 Type "@qc_menu",<CR>, to enter the QC MENU program.

4.3 If entering sample QC select "1", otherwise select the number corresponding to the instrument for which the data is to be entered/reviewed.

4.3.1 Sample QC

4.3.1.1 Select the instrument the samples were analyzed on. Select the correct analysis and sample matrix.

4.3.1.2 Once in the QA Software select "6" (PROCESS RESULTS) and press PF1.

4.3.1.3 Select the desired operation from the remaining menu(s). When the operations are complete, select "EXIT" to save the changes or select "QUIT" to exit without updating the file.

4.3.1.4 Exit the QC Menu program until the command prompt is displayed.

4.3.2 Instrument QC

4.3.2.1 After choosing the appropriate instrument to update, press <CR>.

4.3.2.2 Select the desired operation (i.e., update or plotting results) and press <CR>.

4.3.2.3 Once in the QA Software, select "6" (PROCESS RESULTS) and press PF1.

4.3.2.4 Select the desired operation from the remaining menu(s). When the operations are complete, select "EXIT" to save the changes or select "QUIT" to exit without updating the file.

4.3.2.5 If after entering instrument data a QC REPORT is desired select "Generate a QC Report".

4.3.2.6 Exit the QC Menu program until the command prompt is displayed.

5. Safety, Health, and Environmental Hazards

Not applicable.

6. Records Management

6.1 All computer-generated files are maintained on the VAX. Workstation and backed up with the routine system backups.

6.2 Hard copy printouts of instrument QC REPORTs are maintained in logbooks.

7. References

7.1 VAX/VMS Quality Assurance Package User's Manual (07-0323), Nuclear Data Systems, June, 1991.

7.2 Digital Equipment Company, VMS Users Manual.

STANDARD OPERATING PROCEDURE

FOR UNCONTROLLED DOCUMENT

ALPHA SPECTROSCOPY SYSTEM

(GL-EPI-I-009 Revision 3)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Robert D Timm	09/24/99	ROBERT D TIMM
Process Owner	(Sign and Date)	(Print Name)
Scott C Moreland	9/24/99	SCOTT C. MORELAND
Technical Review	(Sign and Date)	(Print Name)
Robert L Pellam	9/24/99	Robert L. Pellam
Quality Review	(Sign and Date)	(Print Name)
James B Westmoreland	9/27/99	James B Westmoreland
Approval and Authorization	(Sign and Date)	(Print Name)

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TABLE OF CONTENTS

SECTION PAGE

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1.0 STANDARD OPERATING PROCEDURE FOR THE ALPHA SPECTROSCOPY SYSTEM

2.0 METHOD OBJECTIVE AND APPLICABILITY

This method establishes the procedures for general use and calibration of the Nuclear Data Multichannel Analyzer System used to obtain and analyze alpha spectra for samples containing single or multiple alpha-emitting radionuclides. The operation of the Canberra alpha spectrometers is discussed. The method also describes how specific radionuclide analyses are determined from the alpha spectral data.

3.0 DISCUSSION

3.1 Alpha particles with discrete energies are emitted during the decay of some radionuclides. The number of alpha particles emitted per unit time is directly related to the disintegration rates of the radionuclides, and the energies of the alpha particles are characteristic of the radionuclides in a sample. By obtaining an alpha spectrum, one can identify the radionuclides in a mixture and establish the relative amounts of each.

3.2 Alpha spectrometry is normally performed with silicon surface barrier detectors. This type detector produces a current pulse, whenever an alpha particle dissipates its energy in the sensitive volume of the detector. The amplitude of the current pulse is directly proportional to the energy of the alpha particle, and the number of alpha particles entering the sensitive volume, of the detector per unit time, is directly related to the disintegration rate of the sample. Current pulses are amplified to give a voltage output that is proportional to the incident alpha particle energy. These voltage pulses are processed and stored by a multichannel analyzer, for subsequent display and analysis.

3.3 Energy and efficiency calibrations are performed after initial installation, monthly, following system maintenance, and/or whenever any problem is suspected. A NIST traceable standard, or equivalent source, approximating the sample geometry is counted in a reproducible shelf position, a spectrum collected, and subsequent analysis performed. The energy calibration is generated from the newly acquired data by plotting peak centroids versus actual energies of the standard. The efficiency calibration is attained by comparing the observed count rate of the calibration standard to the known decay corrected emission rate of the standard. This efficiency value is then used for the energy range of 3 - 7 MeV.

4.0 DEFINITIONS

4.1 Background - Those counts that can be observed and thereby, allowed for by measuring an empty chamber background. These counts are attributable to environmental radioactivity, recoil contamination of the detector, electronic noise pulses, etc.

4.2 Traceable Calibration - Standard: A calibrated radioactive source, with stated accuracy, whose calibration is certified by or to NIST (National Institute of Standards and Technology) or an equivalent organization.

4.3 Efficiency - A percent of decay events from a standard radioactive source that are seen and measured by a detector.

4.4 FWHM (Full Width Half-Maximum) - The full width of an alpha peak distribution measured at half the maximum peak height.

5.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

5.1 Individuals performing this procedure shall be aware of the precautions necessary for the proper handling of radioactive materials.

5.2 The detector bias supply must remain off, until the detector chamber reaches a minimum of 500 microns of vacuum, to prevent damage to the surface barrier detectors.

5.3 Turning off, or loss of power to, the vacuum pumps could lead to oil contamination of the alpha detectors. Therefore, all detectors must be brought to atmospheric pressure prior to turning the vacuum system off or immediately after a loss of power.

5.4 Follow the manufacturer's instructions for set up, intercomponent connections, and preliminary testing of the equipment. Observe all of the manufacturer's limitations and precautions.

5.6 Never exceed the manufacturer's recommended operating voltage for the detector; this may lead to detector damage.

6.0 EQUIPMENT

6.1 Alpha Spectrometer

6.2 Surface Barrier Detector (or equivalent)

6.3 Multichannel Analyzer (VAX workstation or equivalent)

6.4 Vacuum Pump

6.5 Remote Parallel Interface (RPI)

6.6 AMX analog multiplexor module

6.7 Acquisition Interface Module

6.8 ADC (analog to digital converter)

7.0 MATERIALS LIST

Traceable calibration standard - The actual standard is dependent upon the sample geometry being calibrated.

8.0 STANDARDIZATION AND CALIBRATION

8.1 Energy and Efficiency Calibration (Monthly Checks). Alpha calibration standards are counted once each calendar month to update the detector energy and efficiency calibrations.

8.1.1 Select **2) List Status of Detectors**; ensure detectors are free for use. Press **Return** to exit.

8.1.2 Load a standard into each counter. Close the chamber doors and start evacuation of the chambers.

8.1.3 After the vacuum has fallen below 500 microns, turn on detector bias.

8.1.4 From the VMS **$** prompt type **Count** to access the **Sample Counting Main Menu**. If the **Sample Counting Main Menu** is displayed, proceed with 8.1.4.

8.1.5 Select **1) Sample Counting** to access the **Sample Counting Menu**.

8.1.6 Select **1) Count a New Sample** to access the **Alpha/Gamma Counting Menu**.

8.1.7 Select **3) Monthly Calibration Check.**

8.1.8 Verify that all detector bias supplies are on and press **Return**. The system then starts data acquisition on all alpha counters and counts the standards for four (4) hours.

8.2 Processing the Monthly Calibrations

8.2.1 Exit to the VMS $ prompt by pressing **R** and **Return.**

8.2.2 Check the contents of the file **Names.dat** by typing **Edit Names.dat**. Edit the contents of the file so it contains the entries **W0## - W##**, depending on the number of detectors in the system. When the contents of the file are

correct, press **Control Z** followed by **Quit** if the changes are not to be saved, or **Exit** if the changes are to be saved. Assign the system to the banks being processed, **assign #system_id_start - assign #system_id_end**, where the first number is the first bank number and the second number is the last bank number.

8.2.3 At the **$** prompt, type **Process** to start the processing of the calibrations.

8.2.4 At the **Initial or Update calibration? (I/U)** prompt, enter **U** to update the calibration or an **I** to perform an initial calibration. Note that an initial calibration is done only under specific circumstances, such as initial setup of a counter. Consult the Section Leader (or designee) prior to performing an initial calibration. If a calibration update was chosen, the operator will be asked to verify the updated energy and efficiency parameters for each detector.

8.2.5 Initial Calibration Only

8.2.5.1 At the prompt, use the mouse to position the cursor over the center of the specified nuclide on the spectrum display. Press **Return.**

8.2.5.2 Repeat for each nuclide.

8.2.6 After data is processed, a supervisor's action report will be printed. Review the report for any out of control condition. Contact section leader or designee for out of control condition. Detector should be removed from service if any of the following conditions exist: HIGH/LOW PSAREA, NLACTIVITY, ECOFFSET, ECSLOPE, AVRGEFF.

8.2.7 Update detector status board.

8.2.8 Place sample shelf for out of service detectors in the bottom of the chamber.

8.3 Daily Pulser Checks. This pulser check is performed daily, prior to counting samples, to verify the proper operation of the detectors. Peak centroid, Pulser count rate, and peak FWHM are monitored and stored in quality assurance files.

8.3.1 Select 2) **List Status of Detectors**. Press **Return** to exit.

8.3.2 Ensure all detectors are empty (for Alpha 9, position appropriate sources on sample shelves).

8.3.3 After the vacuum has fallen below 500 microns, turn on the detector bias supplies.

8.3.4 From the VMS $ prompt, type **Count** to access the **Sample Counting Main Menu**. If the **Sample Counting Main Menu** is displayed, proceed with 8.3.4.

8.3.5 Select **1) Sample Counting** to access the **Sample Counting Menu**.

8.3.6 Select **1) Count a New Sample** to access the **Alpha/Gamma Counting Menu**.

8.3.7 Select **2) Daily Pulser Check**.

8.3.8 Verify that all pulsers are on and the detector bias supplies are on. The system then starts data acquisition on all alpha counters and counts the pulsers for 10 minutes.

8.4 Processing the Daily Pulser Checks

8.4.1 Exit to the VMS **$** prompt by pressing **R** and **Return**.

8.4.2 Check the contents of the file **Names.dat** by typing **Edit Names.dat**. Edit the contents of the file so it contains the entries **D## - D##**, depending on the number of detectors in the system. When the contents of the file are correct, press **Control Z** followed by **Quit** if the changes are not to be saved, or **Exit** if the changes are to be saved. Assign the system to the banks being processed.

8.4.3 At the **$** prompt type **Process** to start the processing of the calibrations. The program proceeds to automatically process the pulser data one counter at a time. After the data is processed, a Supervisor's Action Report will be printed. Review the printout for any out of control conditions. If any of the data is out of control, contact the Section Leader (or designee) immediately. Out of control conditions are: HIGH/LOW PSFWHM, PSENERGY, PSCENTRD.

8.5 Weekly Background Checks

8.5.1 Select **2) List Status of Detectors**. Press **Return** to exit.

8.5.2 Place background planchets into each detector. Close the chamber doors and start evacuation of the chambers.

8.5.3 After the vacuum has fallen below 500 microns, turn on the detector bias.

8.5.4 Assign the system. From the VMS $ prompt, type **Count** to access the **Sample Counting Main Menu**. Assign the system. If the Sample

Counting Main Menu is displayed, proceed with 8.5.5.

8.5.5 Select 1) **Sample Counting** to access the **Sample Counting Menu.**

8.5.6 Select 1) **Count a New Sample** to access the **Alpha/Gamma Counting Menu.**

8.5.7 Select 4) **Backgrounds.**

8.5.8 Verify that all detector bias supplies are on, all pulsers are off, and press **Return.** The system then starts data acquisition on all alpha counters and counts the backgrounds for predetermined count time.

8.6 Processing Weekly Backgrounds

8.6.1 Exit to the VMS **$** prompt by pressing **R** and **Return.**

8.6.2 Check the contents of the file **Names.dat** by typing **Edit Names.dat.** Edit the contents of the file so it contains the entries **B## - B##,** depending on the number of detectors in the system. When the contents of the file are correct, press **Control Z** followed by **Quit** if the changes are <u>not</u> to be saved, or **Exit** if the changes are to be saved. Assign the system.

8.6.3 At the **$** prompt, type **Process** to start the processing of the background counts. The program proceeds to automatically process the background data one detector at a time. After the data is processed, a Supervisor's Action Report will be printed. Review the printout for any out of control conditions. If any of the data is out of control, contact the Section Leader (or designee) immediately. Detector should be logged out for isotopes that have high background.

9.0 OPERATING PROCEDURE

9.1 Sample Counting

9.1.1 Select 2) **List Status of Detectors.** Press **Return** to exit.

9.1.2 Ensure that there are no jobs active which use the alpha spectrometer(s) that is to be loaded. Ensure the bias supply to the detector is off, then vent the alpha spectrometer sample chamber to atmosphere by placing the "pump/vent" switch, on the spectrometer, to the "vent position.

9.1.3 Open the door of the sample chamber. Carefully remove any sample that is in the chamber with a pair of forceps and place it in a proper storage container.

9.1.4 Using a pair of forceps, carefully position the next sample that is to be counted on the sample shelf, taking care to center the sample beneath the detector face and ensure the sample shelf is in the proper location.

9.1.5 Close the door on the sample chamber and begin to evacuate the chamber.

9.1.6 Before starting acquisition verify the proper operating bias by selecting the "bias" position, with function selector switch on the front of the alpha spectrometer.

9.1.7 From the VMS **$** prompt, type **Count** to access the **Sample Counting Main Menu**. If the **Sample Counting Main Menu** is displayed, proceed with 9.1.7.

9.1.8 Select **1) Sample Counting** to access the **Sample Counting Menu**.

9.1.9 Select **1) Count a New Sample** to access the **Alpha/Gamma Counting Menu**.

9.1.10 Select **1) Samples**.

9.1.11 Enter appropriate parameters.

9.1.12 Enter the number of samples to be counted.

9.1.13 Enter the detector number containing the first sample.

9.1.14 The system then enters the Parameter Editor. Enter the applicable information for each sample.

- ü Sample Identification: (if empty enter an **E** followed by the detector number.)
- ü Sample Collection Time: enter the Decay date for the tracer.
- ü Sample Quantity:
- ü Sample Units: (grams, ml, liters, etc.)
- ü Analyst' Initials
- ü Activity Units: pCi
- ü Activity Multiplication Factor: 1E+6
- ü Configuration Title: GARXXX
- ü Tracer DPM:
- ü Neptunium Yield:

Press PF1 to exit the Parameter Editor. Continue with the next sample if applicable. When all of the sample data is entered, the system exits to the **Sample Counting Menu**.

9.2 Processing Sample Data

9.2.1 Visually review the data collected. Each spectrum should contain an individual sample spectrum corresponding to the sample loaded in that particular chamber. If any of the spectrums are missing or appear to be wrong, contact the Section Leader (or designee).

9.2.2 Return to VMS and **Edit Names.dat**. It should contain all of the sample ID numbers in the order they were loaded and an **EXX** number, where **XX** corresponds to the number of an empty chamber. Exit the editor by typing **Control Z** and **Exit** (use **Quit** to exit without changing the contents of the original file).

9.2.3 Type **Process** or **@Auto-Process** to begin processing the sample data. After each spectrum is displayed, answer the questions **Does peak resolution look OK?** and **Are you ready for the next spectrum?** by entering a **Y** (for yes). An **N** (for NO) answer to either of these questions will abort the processing. Processing may then be resumed by deleting any suspect data from the **Names.dat** file and repeating step 9.2.3.

10.0 CALCULATIONS

10.1 Efficiency

$$\text{Efficiency} = \frac{CPM_o}{DPM_c}$$

Where:

CPM_O = Observed cpm of the standard
DPM_C = Certified disintegration rate of the standard

10.2 Decay Corrections

$$A_f = A_i * \exp^{-\frac{\ln 2}{t_{1/2}} T}$$

Where:

A_f = Final decay corrected activity
A_O = Original activity
exp = Exponential (inverse natural log)

ln2 = Natural log of 2
$t_{1/2}$ = Half-life of the isotope
T = Decay time

10.3 Standard Deviation (sigma)

$$\text{Standard Deviation (s)} = \sqrt{\frac{N}{t}}$$

Where:

N = Sample count rate (cpm)
t = Sample count time (minutes)

10.4 Minimum Detectable Activity (MDA) Calculation

$$MDA = \frac{2.71 + 4.66\sqrt{s_b}}{2.22 * V * E * A * R}$$

Where:

2.71 = k factor (95% confidence level)
4.66 = Statistical factor (95% confidence level)
s_b = Standard deviation of the background
2.22 = Conversion factor (dpm/pCi)
V = Sample volume or weight
E = Counting efficiency
A = Isotopic abundance
R = Chemical recovery

10.5 Chemical Recovery

$$R = \frac{C_T}{t * E * D_T}$$

Where:

C_T = Tracer regions net counts
D_T = Tracer DPM added

10.6 Sample Activity

$$\text{Activity} = \frac{C_s - C_b}{E * R * T * A * V}$$

Where:

C_s = Gross sample counts
C_b = Gross background counts

11.0 QUALITY ASSURANCE (QA) REQUIREMENTS

11.1 Pulser checks shall be performed daily prior to counting samples.

11.2 Monthly checks shall be performed once each calendar month to verify/update energy and efficiency calibration.

11.3 Detector backgrounds shall be performed once each calendar week.

12.0 REFERENCES

12.1 1990 Annual Book of ASTM Standards, Volume 12.02, E181.

12.2 Canberra Model 7401 Alpha Spectrometer Operations Manual.

STANDARD OPERATING PROCEDURE

FOR

COUNTING ROOM INSTRUMENTATION

MAINTENANCE AND PERFORMANCE CHECKS

(GL-EPI-I-010 Revision 3)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

Process Owner (Sign and Date)	SCOTT C. MORELAND (Print Name)
Technical Review (Sign and Date)	TIM C. WINTERS (Print Name)
Quality Review (Sign and Date)	Genie G. Bost (Print Name)
Approval and Authorization (Sign and Date)	James B Westmoreland (Print Name)

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TABLE OF CONTENTS

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1.0 COUNTING ROOM INSTRUMENTATION MAINTENANCE AND PERFORMANCE CHECKS

2.0 PURPOSE

This procedure outlines the required scheduled maintenance and performance checks for each of the counting room instruments.

3.0 DISCUSSION

In order to assure the optimum performance of counting room instrumentation it is necessary to perform regularly scheduled maintenance and instrument checks. The instrument checks include periodic verification of instrument background, stability, and efficiency calibration. The scheduled maintenance, which is often included as part of an instrument check procedure, provides a means of maintaining instrument performance, while minimizing the "down time" due to instrument failure and subsequent repair.

4.0 DEFINITIONS

4.1 Performance Check - any operation performed on an instrument to verify it's ability to conform to required specifications.

4.2 Scheduled Maintenance - any operation performed on an instrument to prevent premature equipment failure.

4.3 SOP - Standard Operating Procedure.

5.0 PROCEDURES

5.1 Gamma Spectrometers

5.1.1 Daily Instrument Check - performed daily in accordance with GEL SOP "Gamma Spectroscopy System Operation" (GL-EPI-I-001), to verify proper performance of the detector prior to the analysis of any samples. Out of specification parameters are to be noted in the instrument maintenance log and if necessary the detector removed from service until satisfactory completion of the daily instrument checks.

5.1.2 Weekly Instrument Background - performed weekly in accordance with GEL SOP "Gamma Spectroscopy System Operation" (GL-EPI-I-001), to verify the absence of any detector or shield contamination. If any contamination is noted the affected detector shall be removed from service until the source of the contamination can be identified and removed.

5.1.3 Weekly Liquid Nitrogen Fill - performed weekly, each dewar must be filled with liquid nitrogen to ensure proper operation of the gamma detectors. Intrinsic germanium detectors are cooled with liquid nitrogen to eliminate the thermal excitation (noise) in the germanium crystal. The completion of the filling shall be noted on the detector fill log.

5.1.4 Monthly Software Backups - approximately once a month the sample analysis records accumulated on the workstation data disk are backed up onto 4mm DATA tapes. This function can be accomplished on a more frequent basis if the availability of disk space for sample archival becomes a problem.

5.1.5 Annual Efficiency Calibration - performed annually in accordance with GEL SOP "Gamma Spectroscopy System Operation" (GL-EPI-I-001), the detection efficiency is verified and/or updated to ensure the accurate quantitation of samples.

5.1.6 Filter Cleaning - the filter on the air intake of the instrument cabinet shall be cleaned as a minimum quarterly.

5.2 Alpha Spectrometry System

5.2.1 Daily Pulser Checks - performed in accordance with GEL SOP "Alpha Spectroscopy System" (GL-EPI-I-009), a daily pulser check is performed on each alpha spectrometer to verify the detector operation and energy calibration. Out of specification parameters are to be noted in the instrument maintenance log and if necessary the detector removed from service until satisfactory completion of the daily instrument checks.

5.2.2 Monthly Efficiency Calibration - performed in accordance with GEL SOP "Alpha Spectroscopy System" (GL-EPI-I-009), each alpha spectrometer is recalibrated for energy, FWHM, and efficiency. Out of specification parameters are to be noted in the instrument maintenance log and if necessary the detector removed

from service until satisfactory completion of the daily instrument checks.

5.2.3 Weekly Background Checks - performed weekly in accordance with GEL SOP "Alpha Spectroscopy System" (GL-EPI-I-009), the alpha spectrometer chambers are cleaned and the background for each detector is measured, archived, and compared to established limits to identify any out of specification conditions. Out of specification parameters are to be noted in the instrument maintenance log and if necessary the detector removed from service until satisfactory completion of the weekly instrument checks.

5.2.4 Monthly Software Backups - approximately once a month the sample analysis records accumulated on the workstation data disk are backed up onto 4mm DATA tapes. This function can be accomplished on a more frequent basis if the availability of disk space for sample archival becomes a problem.

5.2.5 Vacuum Pump Oil - the oil in the alpha spectrometry system vacuum pumps shall be changed as a minimum of semi-annually.

5.2.6 Filter Cleaning - the filter on the air intake of the instrument cabinet shall be cleaned as a minimum quarterly.

5.3 Gas Flow Proportional Counters

This section is generic in nature and is intended to address the required instrument checks and maintenance on any of the gas flow proportional counters including the Oxford's LB-4100 and LB5100W.

5.3.1 Daily Checks - performed in accordance with the applicable instrument SOP, a daily background and efficiency check are performed on each gas flow proportional counter to verify the detector operation and energy calibration. Out of specification parameters are to be noted in the instrument maintenance log and if necessary the detector removed from service until satisfactory completion of the daily instrument checks.

5.3.2 Weekly Background Checks - performed weekly in accordance with the applicable instrument SOP, the instrument is cleaned and the background for each detector is measured, archived, and compared to established limits to identify any out of specification

conditions. Out of specification parameters are to be noted in the instrument maintenance log and if necessary the detector removed from service until satisfactory completion of the weekly instrument checks.

5.3.3 Annual Efficiency Calibration - performed annually in accordance with the applicable instrument and method SOP's, the detector efficiency is verified and/or updated to ensure the accurate quantitation of samples.

5.3.4 Monthly Software Backups - approximately once a month the sample analysis records accumulated on the instrument data disk are backed up onto 4mm DAT Disks. This function can be accomplished on a more frequent basis if the availability of disk space for sample archival becomes a problem.

5.3.5 Sample Changer Cleaning - the sample changer assembly should be vacuum periodically to minimize the accumulation of dust in the instrument.

5.3.6 Gas Supply Changes - at or near depletion of the gas supply to the instrument the gas bottle shall be changed and the appropriate entry shall be made in the P-10 log book.

5.4 Liquid Scintillation Counter

5.4.1 Daily Checks - performed in accordance with GEL SOP "Beckman LS-6000/6500 Operating Procedures" (GL-EPI-I-004), a daily instrument check is performed to verify the detector operation and energy calibration. Out of specification parameters are to be noted in the instrument maintenance log and if necessary the detector removed from service until satisfactory completion of the daily instrument checks.

5.4.2 Annual Efficiency Calibration - performed annually in accordance with the applicable instrument and method SOP's, the detection efficiency is verified and/or updated to ensure the accurate quantitation of samples.

5.4.3 Sample Changer Cleaning - the sample changer assembly should be vacuum periodically to minimize the accumulation of dust in the instrument.

5.5 Health Physics Radiation Detection Equipment

5.5.1 Daily Checks - performed daily when in use, the background and efficiency of each instrument is checked to ensure the proper operation of radiation detection equipment. The results of the daily checks are recorded in the applicable instrument log book.

5.5.2 Annual Efficiency Calibration - performed annually in accordance with the manufacturers specification, the detection efficiency is verified and/or updated to ensure the accuracy.

5.6 Lucas Cell Counters

5.6.1 Daily Checks - performed in accordance with GEL SOP "Ludlum Model 2000 Lucas Cell Counter Operating Instructions" (GL-EPI-I-007), a daily instrument check is performed to verify the detector operation. Out of specification parameters are to be noted in the instrument maintenance log and if necessary the detector removed from service until satisfactory completion of the daily instrument checks.

5.6.2 PMT Cleaning -weekly the window of the PMT shall be cleaned to reduce the interference from dust accumulation.

6.0 SAFETY, HEALTH, AND ENVIRONMENTAL HAZARDS

6.1 All individuals performing the required instrumentation checks shall have demonstrated a proficiency in instrument operation.

6.2 All individual s performing the instrumentation checks shall be familiar with the hazards associated with the handling of radioactive materials.

7.0 RECORDS MANAGEMENT

7.1 Instrument logs shall be maintained for each instrument to allow for the documentation of instrument problems, maintenance functions, etc.

8.0 REFERENCES

None.

STANDARD OPERATING PROCEDURE

FOR

PREPARATION OF RADIOACTIVE STANDARDS

UNCONTROLLED DOCUMENT

Process Owner(s):	Richard M. Kinney, EPI Analyst III
Technical Review by:	Barry D. Stewart, Senior Technical Specialist
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	James B. Westmoreland, EPI Laboratory Director

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TABLE OF CONTENTS

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1.0 TITLE: STANDARD OPERATING PROCEDURE FOR PREPARATION OF RADIOACTIVE STANDARDS

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

The purpose of this document is to provide necessary instructions for the preparation of radioactive standards.

3.0 METHOD APPLICABILITY

Not applicable.

4.0 DEFINITIONS

4.1 Certification. Written documentation that a particular standard complies with the stated criteria.

4.2 Standard Reference Material (SRM). A source of certified reference material issued by the National Institute of Standards and Technology (NIST), the U. S. Environmental Protection Agency (USEPA), or other national reference organizations.

4.3 Primary Stock Solution. A solution created by the dilution of a SRM to a known volume.

4.4 Secondary Stock Solution. A solution created by diluting a known aliquot from the primary stock solution to a known volume.

4.5 Tertiary Stock Solution. A solution created by diluting a known aliquot from the secondary stock solution to a known volume.

5.0 METHOD VARIATIONS

Not applicable.

6.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

6.1 Treat each chemical, sample and radioactive isotope solution as a potential health hazard, and exposure to these chemicals must be reduced to the lowest level possible. Environmental Physics, Inc. (EPI) maintains a current awareness file of OSHA regulations regarding the safe handling of the chemicals in the radiological laboratory as well as a reference file of Material Safety Data Sheets (MSDS.) These documents as well as individual sample MSDS forms provided by the clients are kept in EPI.

6.2 Personal protective equipment:

6.2.1 Always wear safety glasses with side shields while in the laboratory.

6.2.2 Strong rubber or latex gloves are required when handling concentrated acids (such as sulfuric acid and acetic acid), or ethanol.

6.2.3 Work under a hood when using concentrated sulfuric acid, acetic acid, and ethanol.

6.3 Prior to handling radioactive samples analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

6.3.1 Wear a plastic apron over lab coat when working with radioactive samples.

6.3.2 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.3.3 Prohibit admittance to immediate work area.

6.3.4 Take swipes of counter top(s) upon completion of work.

6.3.5 Segregate radioactive waste from non-radioactive waste. Radioactive waste containers are obtained from Waste Management. Liquid and solid sample waste are segregated. Other disposable solids such as pipet tips, instrument vials, etc., are disposed of in the radioactive waste trash boxes.

6.4 Refer to EPI SOP "EPI Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011).

7.0 INTERFERENCES

Not applicable.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 Calibrated analytical balance capable of weighing to 0.0001 g

8.1.2 Calibrated Oxford or Eppendorf pipets

8.1.3 Disposable cups (30 mL)

8.1.4 Volumetric flasks

8.1.5 Disposable droppers

8.1.6 Pasteur pipet (5 ¾ inches)

8.1.7 Latex bulb (1 inch)

9.0 SAMPLE HANDLING AND PRESERVATION

Radioactive material is handled as outlined in EPI SOP "Radioactive Material Handling Procedures" (GL-EPI-E-S004).

10.0 SAMPLE PREPARATION

Not applicable.

11.0 PREPARATION OF STANDARD SOLUTIONS, REAGENT SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Primary Stock Solution Preparation:

11.1.1 Wipe the outside of the SRM vial with DI water and lab wipes to ensure the vial is free from dirt and other potential contaminants. Invert the vial several times and allow the solution to drain within the vial for several minutes.

11.1.2 Carefully break the vial by applying pressure to the neck area.

NOTE: If the vial is not scored by the vendor, use a diamond tip pen or vial scoring file to establish the break area.

11.1.3 Using a glass Pasteur pipet and latex bulb, aliquot the solution into a tared volumetric flask which has been placed on the analytical balance. Measure the aliquot and record the net weight on the Primary/Secondary Stock Solution form (Appendix 1).

11.1.4 Dilute the solution in the volumetric flask to volume with the appropriate solvent. Stopper the flask and ensure good mixing of the solution. Record the final diluted volume and net weight on the Primary/Secondary Stock Solution form (Appendix 1).

11.1.5 Pour the primary stock solution into a labeled nalgene HDPE bottle. The bottle should be labeled with the following information:

11.1.5.1 Analyst's initials and date

11.1.5.2 The isotope(s) present

11.1.5.3 The concentration of the isotope(s) in dpm/mL (decay corrected to the reference date)

11.1.5.4 The primary stock solution code (see Step 11.1.6)

11.1.5.5 The expiration date (see Step 11.4.1)

11.1.5.6 The reference date and solution of standard

11.1.6 The following nomenclature will be used to establish the primary stock solution code: [Assigned consecutive logbook number]-A.

NOTE: All sources are verified when making a new primary stock solution. An appropriate aliquot is taken and counted on the appropriate instrument. A senior analyst (see approved list of analysts inside Primary/Secondary Standard Logbooks) will determine if standard is to be made available for use. Verification data will be maintained in the Standard Verification notebook.

11.1.7 After all standards are made, the balance and bench top area are scanned with a portable survey meter and swiped and counted to test for possible contamination.

11.2 Secondary Stock Solution Preparation:

11.2.1 Invert the primary stock solution several times and allow the solution to drain within the bottle.

11.2.2 Open the primary stock solution carefully. Remove an appropriate aliquot from the primary stock solution into a tared volumetric flask which has been placed on the analytical balance. Measure the aliquot and record the net weight on the Primary/Secondary Stock Solution form (Appendix 1).

11.2.3 Dilute the solution in the volumetric flask to volume with the appropriate solvent. Stopper the flask and ensure good mixing of the solution. Record the final diluted volume and net weight on the Primary/Secondary Stock Solution form (Appendix 1)

11.2.4 Pour the secondary stock solution into a labeled nalgene HDPE bottle. The bottle should be labeled with the following information:

11.2.4.1 Analyst's initials and date

11.2.4.2 The isotope(s) present

11.2.4.3 The concentration of the isotope(s) in dpm/mL (decay corrected to the reference date)

11.2.4.4 The solution of standard

11.2.4.5 The secondary standard code (see Step 11.2.5)

11.2.4.6 The expiration date (see Step 11.4.1)

11.2.4.7 The reference date and solution of standard

11.2.5 The following nomenclature will be used to establish the secondary stock solutions code as subsequent standards are created: [Assigned consecutive logbook number]-B through Z. If more than 25 secondary solutions are made from one primary, begin labeling as AA-ZZ.

11.2.6 After all standards are made, the balance and bench top area are scanned with a portable survey meter and swiped and counted to test for possible contamination.

11.3 Tertiary Stock Solution Preparation

11.3.1 Invert the secondary stock solution several times and allow the solution to drain within the bottle.

11.3.2 Open the secondary stock solution carefully. Remove an appropriate aliquot from the secondary stock solution into a tared volumetric flask which has been placed on the analytical balance. Measure the aliquot and record the net weight on the Primary/Secondary Stock Solution form (Appendix 1).

11.3.3 Dilute the solution in the volumetric flask to volume with the appropriate solvent. Stopper the flask and ensure good mixing of the solution. Record the final diluted volume and net weight on the Primary/Secondary Stock Solution form (Appendix 1).

11.3.4 Pour the secondary stock solution into a labeled nalgene HDPE bottle. The bottle should be labeled with the following information:

11.3.4.1 Analyst's initials and date

11.3.4.2 The isotope(s) present

11.3.4.3 The concentration of the isotope(s) in dpm/mL (decay corrected to the reference date)

11.3.4.4 The tertiary standard code (see Step 11.3.5)

11.3.4.5 The expiration date (see Step 11.4.1)

11.3.4.6 The reference date and solution of standard

11.3.5 The following nomenclature will be used to establish the tertiary stock solutions code as subsequent standards are created: [Assigned consecutive logbook number]-[secondary standard letter]-[1-100].

11.3.6 After all standards are made, the balance and bench top area are scanned with a portable survey meter and swiped and counted to test for possible contamination.

11.4 Expiration Dates for Standards

11.4.1 The expiration dates for aqueous sources are determined by the standards vendor. If no expiration date is given by the vendor, use two years or five half lives, whichever is smaller.

11.4.2 The expiration dates for non-aqueous sources and non-aqueous primary/secondary sources are given by the standard vendor. If no expiration date is given then the expiration date is equal to five half lives of the isotope of interest.

NOTE: Standards that are used for obtaining tracer recovery by comparison of counts between a sample and reference standard have an expiration date of eight half lives or two years, whichever is smaller, if none is provided by vendor. (Examples: Fe-59, Ba-133)

11.4.3 Solid standards (electroplated, precipitated or spiked matrix standards) which are used for calibration are not assigned specific expiration dates. These sources can be used until control charts indicate a change in instrument response or they have decayed beyond 5 half lives. At such time, the source must be verified or discarded.

11.4.4 Expired standards can be used if verified. Each bottle to be verified will have an appropriate aliquot taken and counted on the appropriate instrument. A senior analyst (see approved list of analysts inside Primary/Secondary Logbooks) will determine if standard is to be made available for use. The expired bottle that has been verified will be labeled with verification date, verification expiration date, and how standard can be used. Verification data will be maintained in the Standard Verification notebook. Standard logbooks will be updated with the same information. Verifications will be performed annually.

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 All balances at EPI are on a service contract for a yearly calibration check. maintenance, and cleaning. Each balance is tagged with the balance serial number, service date, date of the next scheduled service, and the signature of the service technician. EPI maintains test weights to verify balances on a usage basis. These weights are validated yearly by a National Institute of Standards and Technology (NIST) traceable service center.

12.2 The calibration may be verified or checked by the use of weights other than class S. This verification is not to be confused with calibrating the balance. The records of calibration of weights are kept in Quality Records and other appropriate locations.

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 The use of the analytical balance is outlined in GEL SOP "Standard Operating Procedures for Balances" (GL-LB-E-002).

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 Instructions for equipment and instrument maintenance are contained in Section 12.0 of this operating procedure.

15.0 DATA COLLECTION, CALCULATION AND REDUCTION METHODS

15.1 The following calculation is used to determine the standard concentrations in dpm/mL. The following example shows the calculation of a primary stock solution prepared from a SRM:

$$\frac{uCi(srm)}{g} * \frac{g(measured)}{mL(diluted)} * \frac{(2.22e+6)dpm}{uCi} = \frac{dpm(primary)}{mL}$$

This can also be calculated for dpm/g using the following calculation:

$$\frac{uCi(srm)}{g} * \frac{g(measured)}{g(diluted\,net\,weight)} * \frac{(2.22e+6)dpm}{uCi} = \frac{dpm(primary)}{g}$$

15.2 Primary stock standard solutions should be checked for decay corrections every week prior to using them.

16.0 QUALITY CONTROL REQUIREMENTS

Not applicable.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

17.1 Log books are regularly reviewed by the respective Group Leader.

18.0 RECORDS MANAGEMENT

18.1 Each time a standard is prepared the analyst records the appropriate information on the Primary/Secondary Stock Solution form (refer to Steps 11.1.3, 11.2.3, and 11.3.3).

18.2 All Primary/Secondary Stock Solution forms are kept in log books and maintained by the Group Leader, Quality. A copy of this log book and the source certificates are made and used in the laboratory. The original standard source certificates are kept in a fireproof cabinet in the Quality department.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Radioactive material is handled and disposed of as outlined in EPI SOP "EPI Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011), and the GEL/EPI Laboratory Waste Management Plan (GL-LB-G-001).

20.0 REFERENCES

20.1 Compilation of ASTM standard definitions sponsored by ASTM Committee on Terminology. Seventh edition, Philadelphia, American Society for Testing and Material, 1990.

20.2 Taylor, John K., Quality Assurance of Chemical Measurements. Chelsea. Michigan, Lewis Publishers, 1987.

20.3 Table of Isotopes. Seventh Edition, John Wiley & Sons, Inc., New York. 1978.

APPENDIX 1

Primary Standard Log

Parent Code: _____ Isotope: _____

Prepared By: _____ Prep Date: _____

Carrier Conc:_____ Primary Code _____

Reference date:_____

Mass of Parent (g) _____ Dilution (mL) _____

Show calculation converting parent activity to dpm/mL:

_____

Reviewed By:_____ Date:_____

==========

Secondary Standards:

Note: Decay correction is not performed on these secondary standards.

Date prepared	Init.	Mass(g) primary	Dilution(mL)	Secondary code	Secondary conc (dpm/mL)

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1.0 Verification of Customer Work Order and Billing Procedures

Process Owner:	Signature:	Mack D. Swafford Mack D. Swafford
Technical Review by:	Signature:	James B. Westmoreland James B. Westmoreland
Approved & Authorized by:	Signature:	Heyward H. Coleman Heyward H. Coleman

SET I.I

SOP for Verification of Customer Work Order and Billing

1. When a customer requests a price quotation, the Project Manager fills out a Project Code Request (attachment 1) with the appropriate discount and submits it to the accounting department to get a project number.

2. The Project Manager creates a Client Confirmation Letter (attachment 2) and faxes or mails a copy to the customer. The Project Manager retains a copy and places it in his project notebook.

3. When the samples arrive, the Project Manager checks the Client Confirmation Letter for accuracy and reprints it, leaving off the client acceptance signature line. The Project Manager then gives a copy to Log-In and sends a copy to the client confirming receipt of samples and any other necessary arrangements

4. Log-In takes the confirmation letter and logs in the samples using the Chain of Custody sheets provided by the client.

5. The Project Manager uses the custody sheets, the PM Report (attachment 3), and the confirmation letter to insure that the samples have been logged in properly. Note: This is a critical step - mistakes in Log-In can result in incorrect billing to clients. Particular care needs to be exercised in determining that combination analyses are logged in as combinations rather than as individual tests (as a rule, if multiple analyses are requested on a chain of custody and if there is a combined rate for these analyses, analyses are to be logged in using the combined rate, even if this was not specified on the chain of custody).

6. If the Project Manager detects an error in Log-In, he fills in and submits a Lab Number Change form to the data processing department (attachment 4).

7. When a sample's status changes to PMRV, QC generates the Certificate of Analysis. The Certificate of Analysis is delivered to the Project Manager who in turn reviews and returns it to the client.

8. After the Project Manager reviews the data package/Certificate of Analysis per the PM Data Package Review, it is reviewed by the Radiochemistry Laboratory Manager or his designee. After review, the data package/Certificate of Analysis is sent to the client.

9. When the Certificate of Analysis has been sent to client, the Project Manager changes status to REPT and Accounting prepares an invoice. The prices

used in the Accounting invoice is taken directly from the information that was entered into LIMS by Log-in in step 5 above.

10. A copy of the invoice goes to the Project Manager who reviews the invoice for accuracy. This review is to be done promptly (within 24 hours) and if errors are found, corrections are promptly submitted to Accounting. If an invoice disk has been prepared for the client, this should also be reviewed by the Project Manager.

11. Once a confirmation of correctness is received from the Project Manager, the accounting department sends an invoice to the customer.

Attachment 1

Project Information Sheet

I. PROJECT CODE REQUEST

Project Information

Project Number: _____ PM Initials: _____ Service Area: _____
Project Title: _____
Project Location: City: _____ State: _____
Project Duration: _____
Proposal Submtl Date: _____
Projected Cost: _____

Client Information

Company Name: _____ SIC Code _____
Contact: _____
Address: _____
City/State/Zip: _____
Phone Number: _____ Fax Number: _____

Client Contact Information

_____ Existing Analytical Client
_____ Existing Consulting Client
_____ Request for Proposal
_____ New Client
- _____ Reference. Referred by: _____
- _____ Conference/Convention: _____
- _____ Business Development: _____
- _____ Advertisement: _____
- _____ Industrial Guide: _____
- _____ Other: _____
- _____ Former Client: Why return: _____

Additional Job Opportunities

_____
_____
Referred To (Internal): _____

II. PROPOSAL SUBMITTAL INFORMATION

Proposal Submittal Date: _____
Proposed Cost: _____
Laboratory: _____ Consulting: _____

Proposal Follow-up (include date)

_____
_____
Referred To (Internal): _____

Proposal Status

Accepted: _____ Rejected: _____
Date Accepted: _____ Date Rejected: _____
Why Accepted/Rejected? _____
_____
_____

Attachment 2

Mr. Rolf Larsen
Mountain States Analytical
1645 West 2200 South
Salt Lake City, Utah 84119

Quote Number: 9122
Date: 10/25/93
Project Number: MTSA00292
Phone Number: 801-973-0050

The following is a quotation for the analytical services you requested:

SAMPLE GROUP: 1
MATRIX: GroundH2O
SAMPLES: 1
FREQUENCY: One-time
GEN COMMENTS:
LAB COMMENTS:

EXPECTED ARRIVAL: 10/25/93
PRIORITY: 3 - STANDARD
DUE DATE: 11/08/93
DAYS TO PROCESS: 14

ANALYSIS		METHOD		FEE
Other Isotopes			1 @ 105.00	105.00
RA-226	Radium-226	EPA 903		
			Sample Group Total:	105.00
			Total:	$105.00

Analytical results are reported to client contact by close of business on the due date. Field and Consulting services are estimated amounts. You will be invoiced for net expenditures.

Thank you for the opportunity to assist with your environmental testing needs. I look forward to working with you and serving as your representative at General Engineering Laboratories. Please call me if you have any questions.

Mack Swafford
Project Manager
Customer Services
(803) 556-8171
(803) 766-1178 Fax

Attachment 3

Daily PM Log for: 22-OCT-93 Project Manager: Heyward Coleman Page: 1

Sample #	Project / Description / COC Number / Test	Mgr	Collector / CCL No. / Matrix	Matrix / Comments / PO Number / Method	Time	Date Coll	P	Days	Due Date	Rec'd by
9310410-01	WSRC00192 HHC WSRC HSB109C			GroundH2O	1006	20-OCT-93	4	21	12-NOV-93	DFW
	SECE		8122.1	AA86567N						
	ALPHA SPEC AM			GroundH2O	EPI A-011					
	ALPHA SPEC CM			GroundH2O	EPI A-011					
	ALPHA SPEC PU			GroundH2O	EPI A-012					
	ALPHA SPEC TH			GroundH2O	EPI A-012					
	ALPHA SPEC U			GroundH2O	EPI A-011					
	GAMMA PHA			GroundH2O	EPI A-013					
	RA-226			GroundH2O	EPI A-008					
	RA-228			GroundH2O	EPI A-009					
	SR-89 & 90			GroundH2O						
	SR-89			GroundH2O	EPI A-004					
	SR-90			GroundH2O	EPI A-004					
9310410-02	WSRC00192 HHC WSRC QA 71D			GroundH2O	1006	20-OCT-93	4	21	12-NOV-93	DFW
	SECF		8122.1	AA86567N						
	ALPHA SPEC AM			GroundH2O	EPI A-011					
	ALPHA SPEC CM			GroundH2O	EPI A-011					
	ALPHA SPEC PU			GroundH2O	EPI A-012					
	ALPHA SPEC TH			GroundH2O	EPI A-012					
	ALPHA SPEC U			GroundH2O	EPI A-011					
	GAMMA PHA			GroundH2O	EPI A-013					
	RA-226			GroundH2O	EPI A-008					
	RA-228			GroundH2O	EPI A-009					
	SR-89 & 90			GroundH2O						
	SR-89			GroundH2O	EPI A-004					
	SR-90			GroundH2O	EPI A-004					
9310410-03	WSRC00192 HHC WSRC QA 72D			GroundH2O	1006	20-OCT-93	4	21	12-NOV-93	DFW
	SECG		8122.1	AA86567N						
	ALPHA SPEC AM			GroundH2O	EPI A-011					
	ALPHA SPEC CM			GroundH2O	EPI A-011					
	ALPHA SPEC PU			GroundH2O	EPI A-012					
	ALPHA SPEC TH			GroundH2O	EPI A-012					
	ALPHA SPEC U			GroundH2O	EPI A-011					
	GAMMA PHA			GroundH2O	EPI A-013					
	RA-226			GroundH2O	EPI A-008					
	RA-228			GroundH2O	EPI A-009					
	SR-89 & 90			GroundH2O						
	SR-89			GroundH2O	EPI A-004					
	SR-90			GroundH2O	EPI A-004					

Attachment 4

Lab Number Change

Date: _____

Change requested by: _____

Change made by: _____

Client Code: _____

Lab Number(s): _____

Description: _____

Type of change:

☐ Add Tests (list test codes)

_____ _____ _____ _____
_____ _____ _____ _____
_____ _____ _____ _____
_____ _____ _____ _____

☐ Delete Tests (list test codes)

_____ _____ _____ _____
_____ _____ _____ _____
_____ _____ _____ _____
_____ _____ _____ _____

☐ Priority Change Old Priority: _____ New Priority: _____

☐ Change Sample Description to:

_____

☐ Other specify _____

_____

Comments

_____

_____

_____

_____

Forward to accounting after changes have been made to ensure proper invoicing.

fc limslab number change

Magnetic Backup of Hard Drives

Process Owners:	Signature:	Russell L. Moser
	Signature	Wilbur B. Sigmon, III
Technical Review by:	Signature:	James B. Westmoreland
Quality Review by:	Signature:	Robert L. Pullano
Approved & Authorized by:	Signature:	Heyward H. Coleman

SET

2.0 Method Objective and Applicability

This operating procedure is specific to backup and removal of files from hard drives to diskettes or tapes

3.0 Discussion

3.1 The ability to retrieve data and to recreate the resulting spreadsheets is a necessity in the laboratory. Therefore, regular and complete storage of files must take place.

3.2 Overview

3.2.1 The VAX/VMS (Virtual Address Extension/Virtual Memory System) operating system consists of a pair of hard disk drives (dkb200: and dkb300:). The VAX/VMS operating system is located on dkb200: and is only backed up after a system upgrade. The command procedures and archived data are located on dkb300:[ALPHA] and dkb300:[QA],and are backed up on a regular interval (normally every 2-4 weeks).

3.2.2 The remaining personnel computers in the laboratory save their data to the network drive (E:\), if possible. If the computer software doesn't support saving data to alternate drives, macros are provided to duplicate data to the network drive. The applicable directories on E:\ drive are backed up are GFPC (gas flow proportional counting) and RADGROUP. These two directories and backed up using ms backup every day if practical. Once data is no longer needed, (greater than about 1 month) it is archived on floppy disks and later stored in fire proof cabinets.

4.0 Definitions

4.1 Archive: Saving of files from the hard drive of the computer diskette for long term storage.

4.2 Backup: Duplicating drive or directories onto tape or compressed onto another drive. This method allows retrieval of lost data.

5.0 Procedures

5.1.3.3 After completion of the backup, which is signified by the return of the $ prompt type "Backup/rewind/list=lp mka500:qa.bck". This will create a listing of the backup and print it to line printer.

5.1.3.4 After completion of the listing, type "dismount mka500:". Once the green light is illuminated on the tape drive, slide the lever to the left and remove the tape cartridge. Label the cartridge with the directory ([QA]), the date and the volume if more than one tape was used to create the backup.

5.1.4 The listings for each of the backups are stored in the binder labeled "[Alpha] Backups or [QA] Backups."

5.2 Those computers with a largely organizational or management function should also be backed up in a regular manner with sensitive information stored and locked.

5.3 Personal computers in the lab.

5.3.1 Computers that have critical files on the internal hard drives have a macro available to copy these files onto the network drive. This is actuated by depressing the "UPDATE E:\" button on the Excel "Blank.xls" screen.

5.3.2 Selected directories on the network drive (i.e....E:\GFPC, E:\RADGROUP) are backed-up using Microsoft Backup Version 6.0. The directories are backed-up onto a hard drive. Instructions for use of Microsoft Backup are contained in the users manual. This is performed daily if practical.

5.3.3 When files are no longer needed they are archived. The archiving of data is necessary to minimize the amount of data backed up daily. The following procedure is used.

5.3.2.1 Files are moved to a 3.5 inch floppy drive. These files should be grouped together to the machine that users used.

5.3.2.2 Install the disk into the LBW5100 disk drive.

5.1 Creating backups of the micro-Vax 3100 is accomplished for three separate catagories. Individual backups of the [SYSTEM], [ALPHA], and [QA] directories are performed as separate functions

5.1.1 To perform a backup of the [SYSTEM] account refer to the VAX/VMS Operating Manual.

5.1.2 Creating a backup of the [ALPHA] directory.

5.1.2.1 To perform a backup of the [ALPHA] account first place a new TK50 magnetic tape in the tape drive and push the lever to the right.

5.1.2.2 At the $ prompt type @BACKUP and select "Alpha Only" on the menu screen. This will start an automatic backup of all files in the [ALPHA...] account. If the size of the backup exceeds the capacity of a single tape cartridge it will be necessary to insert a second tape cartridge into the tape drive and press "RETURN".

5.1.2.3 After completion of the backup, which is signified by the return of the $ prompt type "Backup/rewind/list=lp mka500:alpha.bck". This will create a listing of the backup and print it to line printer.

5.1.2.4 After completion of the listing, type "dismount mka500:". Once the green light is illuminated on the tape drive, slide the lever to the left and remove the tape cartridge. Label the cartridge with the directory ([ALPHA]), the date and the volume if more than one tape was used to create the backup.

5.1.3 Backing up the [QA] Directory.

5.1.3.1 To perform a backup of the [QA] account, first place a new TK50 magnetic tape in the tape drive and push the lever to the right.

5.1.3.2 At the $ prompt type @BACKUP and select "QA only" on the menu screen. This will start an automatic backup of all files in the [QA...] account.

5.3.2.3 Activate the "Blank.xls" screen on the Microsoft Excel program. Depress the "What's in B:\" button. This macro determines which files are on the disc.

5.3.2.4 The macro requires the input of the disk name. This is obtained by determining the next name in the "Disk Archive Guide" book. Two pages are then printed. The first in installed into the "Disk Archive Guide", the second is kept with the disk.

5.3.2.5 The name of the disk is written on the disk and stored in the LBW5100 desk disk rack. After about one month the disk is moved into fireproof storage.

6.0 Safety, Health and Environmental Hazards

This section is reserved for future development.

7.0 Records Management

Backup information should be kept on hand for a time not shorter than 1 year, and should be kept intact as long as possible not shorter than 5 years..

8.0 References

None

STANDARD OPERATING PROCEDURE

FOR

SEGREGATION OF RADIOACTIVE SAMPLES

UNCONTROLLED DOCUMENT

Process Owner(s):	FOR MARVIN S. MURDAUGH Marvin S. Murdaugh, Radioanalytical Technician
	Russ L. Moser, Group Leader
Technical Review by:	Barry D. Stewart Barry D. Stewart, Sr., Technical Specialist
Management Review by:	James B. Westmoreland James B. Westmoreland, EPI Laboratory Manager
Quality Review by:	Genie G. Bost Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	Heyward H. Coleman, EPI President

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TABLE OF CONTENTS

1.0 STANDARD OPERATING PROCEDURE FOR SEGREGATION OF RADIOLOGICAL SAMPLES

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 Sample segregation is extremely important in the radioanalytical laboratory. Sample segregation reduces the potential for inadvertent spread of contamination from higher level samples to low level samples. A difficulty arises in determining the activity levels present in a sample. Liquid Scintillation Counting is a quick, effective scanning technique for total activity in a sample. The Liquid Scintillation Counter will detect alpha and beta particulate activity. Although gamma activity is not effectively detected, most gamma emitting isotopes also yield alpha and beta emissions. Liquid Scintillation Counting is used to scan liquid and solid samples for total activity. Once total activity is determined on a sample, the sample can then be classified according to that total activity (see Appendix 1). All subsequent activity on that sample can then be segregated so that no intermingling of high and low level samples will occur.

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit (MDL) and Practical Quantitation Limit (PQL): This method is capable of achieving 250 pCi/mL on routine samples.

3.2 Method Precision: Typical Relative Percent Difference (RPD) between sample duplicates are less than 20%.

3.3 Method Bias (Accuracy): Typical recoveries are between 75 and 125%.

4.0 DEFINITIONS

4.1 Dispersibility - The radioactive material, its physical form, and its intended use affect the chance, and the outcome, of an accidental re-lease. The following list of conditions of relative dispersibility aids in understanding the degree of physical containment/confinement, engineered safe-guards, and administrative controls needed when working with radioactive materials.

4.1.1 Nondispersible conditions include:

4.1.1.1 Simple wet operations.

4.1.1.2 Using encapsulated or sealed sources nondestructively.

4.1.2 Limited dispersible conditions include:

4.1.2.1 Normal chemistry

4.1.2.2 Using radioactive or hazardous materials strongly bound in a solid matrix or biological system

4.1.2.3 Performing operations that can result only in fractional releases of material.

4.1.3 Dispersible conditions include:

4.1.3.1 Risk of wet spills

4.1.3.2 Simple dry operations.

4.1.4 Highly or readily dispersible conditions include:

4.1.4.1 Using radioactive powders, gases, vapors, or other aerosols.

4.1.4.2 Using radioactive in combustible or explosive conditions.

4.1.4.3 Using radionuclides in dry, dusty operations.

4.1.4.4 Using pyrophoric radioactive materials.

4.1.4.5 Performing high-temperature or high-pressure operations that may increase the chance of producing radioactive aerosols.

5.0 METHOD VARIATIONS

5.1 Variations may be necessary to handle unusual sample types.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. Environmental Physics, Inc. (EPI) maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the library and in the laboratory, respectively. Individual sample MSDS forms provided by the clients are kept in Login.

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are:

6.3.1 Nitrile gloves for concentrated acids and bases.

6.4 Never leave gas cylinders unchained or untied, including when they are on a moving cart.

6.5 Instructions on the handling of radioactive samples is outlined in EPI SOP M-001, "Handling of Radioactive Samples." The following general guidelines are applicable:

6.5.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling.

6.5.2 Wear a plastic apron over lab coat when working with radioactive samples.

6.5.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.5.4 Prohibit admittance to immediate work area.

6.5.5 Post signs indicating radioactive samples are in the area.

6.5.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in EPI.

6.5.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management."

6.6 Refer to EPI SOP S-005 "Radioactive Waste Handling Procedures" for instructions on how materials are disposed.

7.0 INTERFERENCES/LIMITATIONS

7.1 Organic material in the sample can cause quenching in the liquid scintillation counting.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Apparatus and equipment:

8.1.1 Electric hot plate

8.1.2 20 mL glass scintillation vial

8.2 Reagents, chemicals, and standards: All chemicals should be of reagent grade or equivalent whenever they are commercially available.

8.2.1 Distilled or deionized water

8.2.2 Scintillation cocktail (Ready Gel® or equivalent)

8.2.3 Nitric acid (concentrated)

8.2.4 Hydrochloric acid (concentrated)

8.3 Instrumentation:

8.3.1 Liquid scintillation counter and associated equipment.

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 Samples are collected as specified in the specific isotopic method.

10.0 SAMPLE PREPARATION

10.1 Segregation Procedure. This procedure will be followed for samples that are to be analyzed for radiological analytes. Some clients may request use of this procedure on non-radiological samples. Certain projects may be exempted from this procedure if the client performs a similar scan for activity prior to shipping samples to the laboratory.

Determination of Radiological Classification

10.2 This procedure is supplemental to the standard operating procedure GL-EPI-S-007 "Receiving of Radioactive Samples" and all the normal login procedures are to be followed which includes gamma dose rate surveys and smear testing.

10.3 Samples that cannot be easily aliquotted, such as a piece of concrete, will be classified according to the dose rate level on the surface of the sample.

NOTE: If the client supplies activity levels, these may be used to establish the correct classification. Samples reading at background level (Approximately 0.1 mR/Hr) will be classified as "Environmental". Samples reading above background (up to 2 mR/Hr) will be classified as "Radioactive I." Samples reading above 2 mR/Hr will be classified as "Radioactive II."

10.4 Samples that can be easily aliquotted, will be classified according to the total activity of the sample. Note: Bioassay samples are labeled without further investigation. The analyst will take a small aliquot of sample directly from the sample container which is then placed in a liquid scintillation vial. Liquid samples will be dispensed using a disposable transfer pipet with a known volume. Solid samples will be aliquotted using a disposable spoon with a known volume. If the analyst observes significant dose, i.e. greater than 2 mR/Hr, the sample may be labeled as "Radioactive II" with no aliquoting required.

10.5 An appropriate volume of scintillation cocktail is added and the vial is capped and shaken vigorously to suspend the sample in the scintillation fluid.

10.6 The samples are then introduced into the scintillation counter for a 1 to 5 minute count. The window settings on the liquid scintillation counter will be such that the presence of any significant alpha or beta activity will be detected.

10.7 If the sample exceeds the quench limits, a smaller aliquot of the sample will be run. If high quench persists, a similar aliquot will be evaporated onto a 2 inch planchet for gas flow proportional counting.

10.8 The raw counts observed will be evaluated and converted to units of pCi/g or pCi/mL. Based on this approximate cumulative activity, the samples will be labeled according to classifications as listed in Appendix 1.

Sample Storage, Batching and Analysis Considerations

10.9 Samples are stored separately according to their classification. The only exception is that "Radioactive I" and "Radioactive II" samples are stored in a common storage area.

10.10 Samples will only be batched with other samples in the same group classification.

10.11 Initial handling of "Radioactive II" samples will be done in the glass enclosed area of the analytical lab. This includes weighing, drying ashing and initial sample dissolution.

10.12 Initial handling of "Radioactive I" samples may be done in the glass enclosed area or other areas within the lab as long as no "Radioactive II" or "Environmental" samples are in the immediate proximity of the "Radioactive I" batch.

10.13 Initial handling of "Environmental" samples will not be done in the glass enclosed area or in the immediate proximity of a "Radioactive I" or "Radioactive II" batch.

Segregation of Materials Used in Analysis

10.14 Whenever practical, disposable materials should be used to process "Radioactive II" materials. If not disposed of, materials used to process "Radioactive II" batches will be rinsed into a waste container upon completion. Some items may be disposed as radioactive material with the permission of the group leader. The materials that need to be reused, will be placed in a bin labeled "Radioactive II" which will be filled with a soap solution.

10.15 The material in the "Radioactive II" bin will then be removed for cleaning. The analyst will clean the material with a brush or other physical cleaning tool. The analyst will then rinse the material with tap water and do a final rinse with deionized water. Materials used for "Radioactive II" samples may be used for other "Radioactive II" samples and must not be used for other sample groups until residual contamination is proven to be removed for the isotope of interest.

10.16 "Radioactive I" samples will be handled just as the "Radioactive II" materials only with a separate labeled holding bin. "Environmental" materials may be returned to use with normal cleaning procedures without any follow up screening being performed.

Segregation of Alpha Spectrometers, Gas Flow Proportional Detectors and Lucas Cell Detectors

10.17 Certain detectors will be labeled for "Radioactive II" samples only, and these detectors will be used only for "Radioactive II" samples. However, if a blank is run on the detector and no activity is detected above normal background levels, then the detectors may be used for "Radioactive I" or "Environmental" samples.

10.18 Certain detectors will be labeled for "Radioactive I" samples only, and these detectors will be used only for "Radioactive I" samples. However, if a blank is run on the detector and no activity is detected above normal

background levels, then the detectors may be used for "Environmental" samples.

Segregation of Gamma Spectrometers and Liquid Scintillation Counters.

10.19 To prevent cross-contamination of gamma samples, samples are placed in disposable plastic bags. No further segregation is required for Germanium detectors.

10.20 Liquid Scintillation detectors do not come in direct contact with samples, therefore no segregation is required for Liquid Scintillation Counters

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to EPI SOP M-001 "Preparation of Radioactive Standards."

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 Instrument Calibration.

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 Please refer to EPI SOP I-007 "Beckman 6500 Liquid Scintillation Counter Operating Instructions" for guidance concerning analysis procedures and instrument operation.

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 Refer to Environmental Physics, Inc. SOP I-010 "Counting Room Instrumentation Maintenance and Performance Checks."

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 Calculate the sample pCi/L according to the following equation:

$$\text{Result (pCi / g)} = \frac{(S_{cpm} - B_{cpm}}{2.22 * E * V}$$

15.2 The counting uncertainty is calculated according to the following equation:

$$\text{Error (pCi / g)} = \frac{1.96\sqrt{\frac{S_{cpm}}{T_c} + \frac{B_{cpm}}{T_{cb}}}}{(2.22 * E * V)}$$

15.3 The method detection limit (MDA) is calculated according to the following equation:

$$\text{MDA (pCi / g)} = \frac{(2.71 + 4.66\sqrt{B_{CPM} * T_C})}{(2.22 * E * V * T_C)}$$

where:

C = net count rate, cpm
E = detector efficiency
V = milliliters or grams of sample used
2.22 = conversion factor from dpm/pCi.
Bcpm=Background cpm
Scpm= Sample cpm
Tc= Sample count time
Tcb= Background count time

15.4 Record the information required on the que sheet.

15.5 Transfer applicable data from the que sheet to a verified spreadsheet.

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification Requirements

16.1.1 Refer to EPI SOP D-003 "Analyst and Analytical Methods Validation Procedures" for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 No added quality control samples such as duplicates or method blanks are required due to the screening nature of this procedure.

16.2.2 Actions Required if the Quality Control Requirements Are Not Met. If any of the QC criteria cannot be satisfied, the analyst should inform their group leader and initiate a Non-conformance Report as outlined in GEL SOP GL-QS-E-004 "Non-conformance

Identification, Control, Documentation, Reporting, and Dispositioning."

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

17.1 The analyst turns in the raw data, review checklist, que sheet, spreadsheet, data report, NCR's (if applicable), and other appropriate information in a batch to the data review specialist.

17.2 After the review process is complete, the data is transmitted from the laboratory personnel to the reporting personnel as outlined in EPI SOP D-003, "Data Review and Validation Procedures."

17.3 The Data Review/Quality Group Leader is responsible for reporting the data.

18.0 RECORDS MANAGEMENT

18.1 The following records are retained to document the analytical process

18.1.1 A runlog that lists the sequence the analyses that were performed.

18.1.2 A maintenance log which describes the routine and non-routine maintenance.

18.1.4 A batch que sheet and spreadsheet which contains applicable dates, times, detectors used etc.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

19.1 Radioactive samples and material shall be handled and disposed of as outlined in EPI SOP S-005 "Radioactive Waste Handling Procedures."

20.0 REFERENCES

20.1 10 CFR 835, 1993. "Occupational Radiation Protection." U.S. Code of Federal Regulations.

20.2 29 CFR 1910. 1992. "Occupational Safety and Health Standards." U.S. Code of Federal Regulations.

20.3 DOE Methods for Evaluating Environmental and Waste Management Samples, DOE/EM-0089T, March 1993.

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Appendix #1: Group Classifications

Group classification	Activity Level	Units
"Bioassay"	N/A	N/A
"Environmental"	<500	pCi/L or pCi/g
"Radioactive I"	>500 and < 10,000	pCi/L or pCi/g
"Radioactive II"	>10,000	pCi/L or pCi/g

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Process Owner:	Nancy A. Slater, EPI Project Manager
Technical Review by:	Russel L. Moser, EPI Group Leader
Management Review by:	James B. Westmoreland, EPI Laboratory Manager
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	Heyward H. Coleman, EPI President

1.0 STANDARD OPERATING PROCEDURE FOR GENERATION AND SHIPMENT OF ELECTRONIC DISK DELIVERABLES

2.0 PURPOSE

To describe the procedure for generating data files and for shipping data files for West Valley Nuclear Services Company, Inc. (WVNS), Fernald Environmental Restoration Management Corporation (FERMCO), and Science Applications International Corporation (SAIC).

3.0 DISCUSSION

West Valley Nuclear Services (WVNS), Science Applications International Corporation (SAIC), and Fernald Environmental Restoration Management Corporation (FERMCO) require that specific data be reported via an electronic disk deliverable. This standard operating procedure describes the process by which the data files are generated, reviewed, and shipped. A certain level of training, computer experience, and computer privileges is necessary to generate the disk deliverable.

4.0 DEFINITIONS

4.1 PMRV (project manager review) - Sample status necessary for the capture of data.

S

4.2 LIMS - Laboratory Information Management System

4.3 EDD - Electronic Data Deliverable

4.4 Release number - A client designated number for a group of samples.

4.5 ASCII - American Standard Code for Information Interchange, a computer code for representing alpha-numeric information.

5.0 PROCEDURES:

5.1 Initiation: When to Generate a Data File

Samples which have been updated to "PMRV" (project manager review) are ready to be captured in the data tables so that the EDD can be generated.

5.2 Generating the Data File for WVNS

5.2.1 At the LIMS prompt, the WVNS specific program is run to generate the WVNS EDD (ASCII Format) using the client's samples numbers. The UNIX work file is then converted to a DOS file using the client's release number. Then the file is copied onto a diskette, printed and compared to the Certificates of Analysis. The work file is then deleted. An example follows:

```
nas>nohup idxl /oracle/edd/wvns/wvns_disk.idxl - lab/lab
9601234-01 9601234-09>9601234.res &
nas>unix2dos wvns.csv wvns080.dat
nas>cp wvns080.dat /export/home/radio
nas>rm wvns.csv
```

5.3 Generating the Data File for FERMCO and SAIC

5.3.1 At the LIMS prompt, the client specific UNIX program is run to generate the client specific EDD using the client's release number (FERMCO) or sample numbers (SAIC). Then the file is copied onto a diskette. An example follows:

```
nas>cd /oracle/edd/ferm
nas>run_ferm_disk 1000003358 lwepi
nas>cp 3358.edd /export/home/radio
```

5.3.2 The program is designed to look for errors and if any are found it aborts the program and gives an error message. If necessary, correct any errors and then rerun the program using the following:

nas>!!

5.4 Shipment

Floppy disks and hard copies of the files are sent by certified, registered mail in care of the client, along with the cover letter and chains of custody by the project manager.

6.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

No potential safety, health or environmental hazards exist for the activity described in the SOP.

7.0 RECORDS MANAGEMENT

7.1 Refer to GEL SOP "Standard Operating Procedure For Quality Records Management and Disposition" (GL-QS-E-008) for the procedure for inactive data storage.

7.2 Electronic copies of the data files are kept in LIMS in the Project Manager's file and are backed up and archived.

8.0 REFERENCES

8.1 SunOS User's Guide: Getting Started, Revised March 1990.

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STANDARD OPERATING PROCEDURE

FOR

THE OPERATION OF THE

CHEMCHEK KINETIC LASER PHOSPHORIMETER

(GL-EPI-I-011 Revision 5)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

Julie Hutto 9/22/99		Julie Hutto
Process Owner	(Sign and Date)	(Print Name)
Michael Schultz 9/22/99		Michael Schultz
Technical Review	(Sign and Date)	(Print Name)
Robert L. Pullano 9/24/99		Robert L. Pullano
Quality Review	(Sign and Date)	(Print Name)
James B Westmoreland 9/24/99		James B Westmoreland
Approval and Authorization	(Sign and Date)	(Print Name)

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TABLE OF CONTENTS

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4.4 Reference Cell - The cell that is used to monitor laser performance. The cell is to the left hand side of the sample cell. R^2 is monitored as well as, lifetime as two critical indicators of performance.

4.5 Sample Cell - The cell to the right hand side of the door with the door closed and facing the instrument. The sample cell is the cell position which is used to calibrate the instrument and subsequently analyze samples.

4.6 Low Range - The range used to analyze samples with little or no uranium content.

4.7 High Range - The range used to analyze samples with a higher uranium concentration.

4.8 Background - A cell with only Uraplex and deionized water used to measure the phosphorescence of the "Uraplex" with no uranium presence for subtraction from analysis.

5.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS

5.1 Under no circumstances should the safety locks be defeated, and the door of the instrument opened while the laser is running. This could give the analyst exposure to radiation from the laser.

5.2 Caution should be used when dealing with acids, and other laboratory reagents. Safety procedures for these are set forth in the Handbook for Good Laboratory Practices, found in the library of the laboratory.

5.3 If there is any question regarding the safety of any laboratory practice, **stop immediately** and consult the group leader prior to carrying out the rest of the procedure.

6.0 EQUIPMENT

6.1 Plastic or quartz cells

6.2 Chemchek Laser Phosphorimeter system

6.3 Calibrated pipettes

6.4 Vacuum pump and slurp line (if using non-disposable cells)

7.0 MATERIALS LIST

7.1 NIST traceable standard solutions

1.0 STANDARD OPERATING PROCEDURE FOR THE OPERATION OF THE CHEMCHEK KINETIC LASER PHOSPHORIMETER

2.0 METHOD OBJECTIVE AND APPLICABILITY

This standard operating procedure provides the necessary instructions to operate the Chemchek laser phosphorimeter.

3.0 DISCUSSION

3.1 The chemcheck phosphorimeter uses a proprietary complexing agent "Uraplex" to promote phosphorescence in the presence of uranium. A laser is used to pass a beam through two cells, the reference cell and the sample cell. The laser causes the excitation of the Uraplex. The complex then loses the energy through phosphorescence, which is measured perpendicular to the laser beam path.

3.2 A calibration curve of known standards is used to compare the sample for quantification. The instrument uses a least squares fit of the standard curve. Two ranges are available on the chemchek system, an upper range and a lower range. These two ranges are calibrated separately for high uranium content samples and low concentration samples. Care should be taken to screen the sample on the higher range before using the lower range, if the sample is thought to contain a high uranium concentration.

3.3 Interferences are those which hamper the ability of the complexing agent to decay through phosphorescence by undergoing chemical or physical quench. Halides, chloride in particular, are a source of interference in that they allow the complexed solution to decay by physical vibration and not through phosphorescence. Undissolved particles in the cell, such as organics, can also give this interference. Short phosphorescence lifetime of the complexed solution is an indication of interference. Another source of possible interference is fluorescence. The Chemchek system uses a pulsed laser system and delays the measurement of the phosphorescence until after the fluorescence is completed.

4.0 DEFINITIONS

4.1 Uraplex - A proprietary complexing agent used to promote phosphorescence.

4.2 Lifetime - The measurement of the length of phosphorescence in a sample or standard.

4.3 R^2 or Linear Regression Coefficient - The measurement of the linearity of decay. A low R^2 is indicative of poor phosphorescence, or quench.

7.2 Deionized water

7.3 Stilbene 420 (Lasing compound)

7.4 Uraplex (proprietary complexing solution) prepared fresh. Uraplex can be kept fresh for several days in a darkened refrigerator.

8.0 STANDARDIZATION AND CALIBRATION

8.1 Invoke the Chemchek operational menus.

8.2 All cells are handled from the top and bottom of the cell and not from side to side. Gloves are to be worn.

8.3 The analyst follows the directional menus by selecting the calibration mode.

8.4 Select which range to undergo calibration, high or low range.

8.5 A reference cell is prepared using a 1 µg/L reference solution. A background is the first part of the calibration to be run. A cell with 1 mL of deionized water and 1.5 mL of Uraplex is inserted into the sample holding position. The background is plotted each day it is performed and subsequent results should fall within 2 Sigma. If outside 2 Sigma but within 3 Sigma, investigate results. If outside 3 Sigma, corrective action is taken. If disposable cells are being used, then a new cell should be used. If the background remains out the reagents should be replaced and the background reanalyzed. Uraplex upon degrading will demonstrate a higher background. If the solution is more than several days old, it should be prepared again which would then bring the background down.

8.6 A series of standards is selected to encompass the concentration range of the samples being analyzed. It is best to calibrate both ranges. The low range can be from approximately 1 to 20 µg/L, and the high range from 15 to 10,000 µg/L. Several standards are selected throughout the range. At least three standards for each range are chosen. The calibration standards are NIST traceable.

8.7 The standard concentrations are entered into the curve information window under calibration mode of the Chemchek system. Each standard is run in consecutive order. The R^2 is monitored as well as the lifetime of each standard. The reference cell is also monitored for both criteria. For both the reference cell and the sample cell, which contains the standard, the criteria for acceptance is linear regression of >0.95 and the lifetime greater than 120 us.

8.8 If the criteria are not met, then the analysis is rerun. If the standard continues to fail, then consult the group leader. Possible causes could be degrading of lasing compound or needed maintenance on instrument windows.

8.9 At the completion of the standard curve the summary report is generated for each range calibrated. The final review includes all lifetimes, R^2 of standards and reference cells, a least squares fit of the standard curve of >0.95, and ana percent descrepancy of <10%.

NOTE: Check Special Batch Requirements for client specific criteria for calibration and blank population management.

9.0 OPERATING PROCEDURE

9.1 Care should be taken when using the Chemchek Laser Phosphorimeter not to touch the inside windows and when handling the cells. Plastic gloves are used when handling cells and held from top to bottom not side to side of the cell.

NOTES: For SNLS and MKFG samples run a CCV (continuing calibration verification) and CCB (continuing calibration blank) every 10 samples for the range of the samples. Acceptable recovery of the CCV is ±25 %. The acceptable criteria for the CCB is to be lower than the CRDL (i.e. for the low range run a 10 µg/L CCV and the CCB, and for the high range run a 500 µg/L CCV and the CCB).

KPA measurements of total uranium (for KHCO) on cellulose ester filters are made by the method of standard additions. Two measurements are conducted for each filter sample: one un-spiked aliquot and one spiked aliquot. The two results must agree to within 10%.

9.2 Ensure that the calibration of the instrument has taken place according to Section 8.

9.2.1 During sample analysis, the calibration is monitored by analyzing two reference solutions, at the beginning and end of each batch. One of these (within the calibration range) reference solutions must be a non-calibration curve solution.

9.3 Following the directional menus, select the menu for the sample analysis mode.

9.3.1 For specific operating instructions for sample analysis, refer to "Operation and Service Manual, Automatic FPA," (Chemcheck Instruments).

9.4 The sample analysis can be brought up on the screen and printed out, if selected, and reviewed for the lifetime and R^2. Acceptable criteria would be greater than 120 us and less than 350 us lifetime, a linear regression coefficient of decay, R^2, >0.95, and a reference intensity ratio between 0.8 and 1.2. Check special batch requirements for specific client needs.

9.5 The instrument maintains a run log of sample identification, run date and time.

9.6 If the sample does not meet the criteria for R^2 and/or lifetime, a post-spiked sample must be run. If this post-spiked sample does not meet the acceptance criteria, see the Group Leader or Senior Analyst.

9.7 The reference cell is monitored during analysis to check the laser performance. If the reference cell response falls below acceptable lifetime or R^2, a recalibration is required and a reanalysis of all samples in that sample set is required. If the criteria falls out again during the next set of analysis, consult the group leader. Replacement of the lasing compound is probably necessary.

9.8 After the completion of analysis, the sample results shall be entered on the Total Uranium spreadsheet and the corrected results along with the run date, time, and analyst's initials is entered into LIMS.

9.9 The analyst reviews the data for completeness and accuracy according to "Data Review and Validation Procedures" (GL-EPI-D-003), and turns it in to the Group Leader for subsequent reviews.

9.10 For instrument care, please see the Chemcheck Operation and Service Manual.

10.0 CALCULATIONS

10.1 The linear regression equation for R^2 is of the form of linear equation:

$$y = b_0 + b_1 x$$

10.2 Method of Least Squares Equation to solve for b and b_1:

$$nb_0 + b_1 \Sigma x_i = \Sigma y_i$$

and:

$$b_0 \Sigma x_i + b_1 \Sigma x_i^2 = \Sigma x_i y_i$$

$$r = \frac{\Sigma (x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\left[\Sigma (x_i - \bar{x})^2 I \Sigma (y_i - \bar{y})^2\right]}}$$

where:

r = correlation coefficient

10.3 For MDA determination, see page 45, Section 6.3.4 of the "Operation and Service Manual, Automatic FPA." See special batch requirements for specific client needs.

11.0 QUALITY ASSURANCE REQUIREMENTS

11.1 A method blank accompanies each batch of 20 or less samples. The reported value should be less than the CRDL for all target isotopes

11.2 A matrix spike and laboratory control sample is run with each batch of 20 samples or less. The requirements for recovery is between 75 and 125%. Recovery is calculated as follows:

$$MS(\%) = \frac{\text{Spiked result} - \text{Sample result}}{\text{Spike added}} * 100\%$$

$$LCS(\%) = \frac{\text{observed concentration}}{\text{known concentration}} * 100\%$$

11.3 A sample duplicate is analyzed with each sample batch of 20 or less samples. The limits on %RPD is 20 percent, if both samples are above 5 times the CRDL.

If either result is below 5 times CRDL but above CRDL, then the %RPD can be equal to or less than 100%. If either result is below CRDL, then the limits on %RPD are not applicable. Relative percent difference (%RPD) is calculated as follows:

$$RPD(\%) = \frac{\text{High Dup}(\mu g/L) - \text{Low Dup}(\mu g/L)}{\text{Average } (\mu g/L)} * 100\%$$

11.4 If any of the above criteria cannot be satisfied, inform the group leader and initiate a nonconformance report as outlined in GEL SOP "Nonconformance Identification Control, Documentation, Reporting and Dispositioning" (GL-QS-E-004).

NOTE: Some clients contractually override these limits and may be more or less restrictive.

11.5 For SNLS and MKFG, a CCB and CCV shall be run after every tenth sample during analysis of the samples. Acceptable criteria of the CCV (Continuing Calibration Verification) is ±25% and the criteria of the CCB (Continuing Calibration Blank) is for the result to be less than CRDL (Contract Required Detection Limit for the samples). If the criteria is not met then the analyst should recalibrate the instrument and rerun the analysis. If the criteria continue to fail then the analyst should consult the group leader or designee, possible problems are degredation of the lasing dye or needed maintenance on the instrument.

11.6 Run Sequence

11.6.1 Calibration background.

11.6.2 Calibration Standards: Calibrate each range to be used for the analysis.

11.6.3 Standard Checks.

11.6.4 Samples.

11.6.5 Standard Checks.

11.7 Records, Management, and Document Control

11.7.1 Raw data and associated documentation are stored in binder books or in files and kept on hand for any subsequent reviews necessary.

11.7.2 Data generated on the chemchek system is backed up during routine software backup. See EPI SOP "Magnetic Backup of Hard Drives" (M-003) for magnetic backup and storage instructions, concerning how often the information is backed up and how long it is kept.

11.7.3 Quality control charts for spike, blank, and duplicates are kept on the Micro-Vax 100.

12.0 REFERENCES

12.1 CRC Standard Math Tables, 25th edition, Editor, William H. Beyer, Ph.D., CRC Press, 1981. pg. 509

12.2 Operation and Service Manual, Kinetic Phosphorescence Analyzer, Chemchek Instruments, Inc., Richland, WA.

12.3 *Determination of Trace Uranium by Kinetic Phosphorescence Analysis*, Westinghouse Materials Co. of Ohio FMPC, Analytical Laboratory Procedure.

12.4 *Standard Test Method for Trace Uranium in Water by Pulsed-Laser Phosphorimetry* 1992 Book of ASTM Standards. Section 11, Vol. 11.02 Water (II)

STANDARD OPERATING PROCEDURE

FOR UNCONTROLLED DOCUMENT

MANAGING STATISTICAL DATA IN THE RADIOCHEMISTRY LABORATORY

(GL-EPI-I-012 REV3)

[signature] 6/7/99	Tim C Winters
Process Owner (Sign and Date)	(Print Name)
[signature] 6/7/99	Scott C. Moreland
Technical Review (Sign and Date)	(Print Name)
Erica L. Elias 6/7/99	Erica L. Elias
Quality Review (Sign and Date)	(Print Name)
[signature]	James B Westmoreland
Approval and Authorization (Sign and Date)	(Print Name)

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Table of Contents

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1.0 MANAGING STATISTICAL DATA IN THE RADIOCHEMISTRY LABORATORY

2.0 METHOD OBJECTIVE AND PURPOSE:

This procedure provides guidelines on how statistical limits are derived and used at General Engineering Laboratories, Inc.

3.0 DISCUSSION:

Decisions on the performance of instrumentation and methods are critical in the production of quality radioanalytical data. Statistical data can be used to determine acceptable limits, identify trends and provide other useful information.

4.0 DEFINITIONS:

4.1 Detector Lockout. A condition which causes a detector to be removed from service. To change this status, the analyst must evaluate the cause of the lockout.

4.2 Detector Watch. A condition which alerts the analyst to watch for a reoccurrence of a problem condition.

4.3 Detector Outlier. A condition that is outside the detectors acceptance criteria. This condition can be removed if two additional confirmation checks show acceptable results. If the detector continues to fail, a Detector Lockout occurs.

4.4 Detector Trend. When greater than 5 points are above or below 1 standard deviation of the statistical mean.

4.5 FWHM. (Full width at half maximum) A measure of the resolution of a peak width.

4.6 CPM. Counts per minute.

5.0 SAFETY, HEALTH AND ENVIRONMENTAL HAZARDS:

Adherence to this procedure does not present a threat to personnel or the environment.

6.0 PROCEDURE:

6.1 Appendix 1 gives the instrument parameters that are monitored in the laboratory. Selected charts are printed quarterly for review as outlined in Appendix 1. The limits for these parameters are established using one of two principal mechanisms:

6.1.1 By running at least 10 replicates of the given parameter and then calculating the mean and standard deviation. The limits are set as plus or minus three times the standard deviation of the mean. These established limits are static until the next statistical evaluation that is determined by the group leader. New limits are normally established when a change occurs in the process such as a window replacement or detector repair.

6.1.2 By setting a fixed upper and lower boundary level. These limits are based on well-established performance specifications. An example is a surface barrier detector must have an average efficiency above 19.5%.

6.2 The following guidelines are used to evaluate the detectors in the laboratory for all instruments that use the database to evaluate their statistical data:

6.2.1 A "detector watch" occurs when a check has a standard deviation outside of plus or minus 2 sigma. During a "detector watch," the detector may be used but will be locked out if the condition occurs again.

6.2.2 A "detector outlier" occurs if the standard deviation is between 3 and 5 sigma. The detector cannot be used without recounting the check twice. If the condition occurs a second time a "detector lockout" occurs.

6.2.3 The following will cause a "detector lockout" to occur.

6.2.3.1 A check with a standard deviation outside of 5 sigma of the statistical mean.

6.2.3.2 Two consecutive checks with a standard deviation outside of 2 sigma of the statistical mean.

6.2.3.3 Five consecutive checks where the standard deviation is changing in the same direction (up or down).

6.3.3.4 Five consecutive checks with standard deviation above 1 sigma or below -1 sigma.

Gas Flow Proportional Counters

6.3 Daily instrument checks. A radioactive source and background check is counted each day the detector is used. The radioactive source is counted for a minimum of 30 minutes and the background is counted for a minimum of 60 minutes.

6.4 Weekly background checks. A background planchet is counted for a minimum of 480 minutes. The weekly background checks are included with the daily background checks in the determination of statistical limits and control chart. An administrative cap on the allowable background is 0.3 cpm alpha and 2 cpm beta.

Liquid Scintillation Counters

6.5 Daily instrument checks. A radioactive source and background check is counted each day the detector is used. The radioactive source is counted for a minimum of 10,000 counts, and the background is counted for a minimum of 30 minutes.

Alpha Spectrometers

6.6 Daily Pulser Checks. A daily pulser check is run on each detector that is to be used for analyzing samples that day. The pulser is an electronic signal at a set KeV, usually 5000, and is monitored for FWHM, centroid channel and centroid energy. The monitoring technique used is a boundary chart using factory suggested values. The values used are, for FWHM an upper limit of 25 KeV, for centroid channel it is -35 channel lower limit and +35 channels for upper limit, and ± 100 KeV for centroid energy.

6.7 Weekly Backgrounds. Once per week a background of 1000 minutes is counted on each detector. The background is then processed and results charted. Recorded results include, total background counts by isotopic region, total background counts of spectrum and total background count rate for entire spectrum. An administrative cap of 50 counts is set as an upper boundary for each region of interest.

6.8 Monthly Calibrations. There are twenty charts kept on data accumulated from the monthly calibration. These twenty charts are centroid channel, centroid energy, peak area, activity for four isotopes for the standards, which are Pu-239, Cm-244 and Np-237, as well as average efficiency, energy calibration offset, FWHM, energy calibration slope. All charts are using the boundary mode; administrative limits have been set for each chart based upon results of initial calibrations.

Gamma Spectrometers

6.9 Daily Checks. Each day a daily standard is counted on the detector if it is to be used for analysis. The daily standard is a mixed gamma standard that has traceability to NIST. Three lines are chosen and monitored. These three lines are usually Cd-109 at 88 KeV, Cs-137 at 661 KeV, and Y-88 at 1836 KeV. These isotopes are changed for low energy detectors such as x-ray detectors, but the idea is to cover the largest energy range possible with the isotopes available. Charts are maintained on peak centroid channel, peak centroid energy, peak FWHM, and isotopic activity (indirectly a measure of detector efficiency). The charts use a boundary mode based upon 2 and 3 sigma of 20 runs of the daily standard in one day, after the twelve month calibration has been completed. If after the twelve month calibration a daily standard is run and all items are found to be in conformance the previous settings may be left in place.

6.10 Weekly Backgrounds. A 1000 minute background is run on each detector once a week. The background is analyzed for two items, total spectrum counts and total spectrum counts per minute. These charts also use a boundary mode, which is a 2 and 3 sigma of 20 backgrounds on the detector. These backgrounds may be collected over a 20 week period and the detector used for this period as long as a close check of the previous weeks results are completed prior to putting the detector in use for the next week. At the end of twenty weeks the results of gross counts and cpm are analyzed for 2 and 3 sigma and the boundaries for investigate and action are set.

Lucas Cells

6.11 Daily Check. Each day a daily check is counted for 1 minute on each detector that is to be used for analysis. The standard is a nominal source standard of Radium-226 sealed in a Lucas cell. The standard is then analyzed for total counts. The boundaries are set by 2 and 3 sigma boundaries from 20 runs in one day of the daily standard after the six month calibration. These boundaries are also known as investigate for 2 sigma and action for 3 sigma.

6.12 Backgrounds. Each cell prior to being used for analysis is evacuated and a 30 minute background is counted on it. An administrative limit of 0.267 cpm has been set as the upper limit for the background. If the background fails the cell may not be used for analysis until the cell has been purged and cleaned and demonstrated a background level of less than 0.267 cpm.

Kinetic Phosphorimeter

6.13 Daily checks. Each day that the kinetic phosphorimeter is used to analyze samples the detector is calibrated and daily checks run. The daily checks consist of a bkg check and a check standard, preferably a cross check sample of known concentration, for each range to be used that day. The control limits for the low range is <10,000 intensity for the background and between 75% and 125% for the check standard. The high range limits are <10 intensity for the background and between 75% and 125% for the check standard.

Analytical Methods

6.14 Analytical method performance is tracked by printing four primary charts in the first month of a calendar quarter. These charts include method blanks, duplicate relative percent difference, matrix spike recovery and laboratory control sample recovery. No limits are established for these parameters based on their statistical performance. These parameters are given specific acceptable ranges in the analytical standard operating procedure.

7.0 RECORDS MANAGEMENT:

7.1 Charts are maintained by the Group Leader. The control charts are printed quarterly for both instrumentation and methods.

7.2 Each analysis that is performed on the instrument is documented in the run log according to GEL SOP "Run Logs" (GL-LB-E-009).

7.3 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

8.0 REFERENCES:

8.1 Chieco, N.A, Bogen, D.C., Knutson, E.O. Environmental Measurements Laboratory (EML) Procedures Manual. 27th Edition, Volume 1. US Department of Energy February 1992.

8.2 Krieger, H.L., Whittaker, E.L. Prescribed Procedures for Measurement of Radioactivity in Drinking Water. EPA 600 4-80-032. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. August 1980.

8.3 ANSI N42.2 "Measurement Quality Assurance for Radioassay Laboratories" Feb. 9, 1994 Final Revision.

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APPENDIX 1

Instrument	Parameter	Limit	Print
Proportional oucnter	Daily source check	3 Sigma	Yes
Proportional count	Background checks	3 Sigma (0.3 cpm alpha max., 2.0 cpm beta max.)	Yes
Scintillation count	Daily source check	3 Sigma	Yes
Scintillation count	Daily background check	3 Sigma	Yes
Alpha spectromete	Daily pulser check FWHM	Boundary (0-25 KeV)	No
Alpha spectromete	Daily pulser check Channel	Boundary (+-35 channel)	No
Alpha spectromete	Weekly background check for total spectrum	Boundary (0-0.05 cpm) per isotopic region	Yes
Alpha spectromete	Weekly background check by isotopic region	Boundary (0-.025 cpm) per isotopic region	No
Alpha spectromete	Monthly Cal. peak centroid of 4 isotopes	3 Sigma	No
Alpha spectromete	Monthly Cal. peak energy of 4 isotopes.	3 Sigma	No (Am-241)
Alpha spectromete	Monthly Cal. peak area of 4 isotopes.	3 Sigma	No
Alpha spectromete	Monthly Cal. peak activity of 4 isotopes.	3 Sigma	No
Alpha spectromete	Monthly Cal. average efficiency.	Boundary • 19.5%	No
Gamma spectrome	Daily peak centroid channel check using 3 energy lines.	3 Sigma	No
Gamma spectrome	Daily peak centroid energy check using 3 energy lines.	3 Sigma	No (Y-88)
Gamma spectrome	Daily peak FWHM check using 3 energy lines.	3 Sigma	No (Cs-137)
Gamma spectrome	Daily activity check using 3 energy lines.	3 Sigma	No (Cs-137)
Gamma spectrome	Weekly background check of total spectrum counts	3 Sigma	No
Gamma spectrome	Weekly bkg total spectrum cpm	3 Sigma	No
Lucas cells	Daily Source Check	3 Sigma	No
Lucas cells	Daily Background Check	3 Sigma	No

Instrument	Parameter	Limit	Print
Kinetic Phosphorimeter	Daily Bkg Check High Range	Boundary (<10)	No
Kinetic Phosphorimeter	Daily Source Check High Range	Boundary (75-125%)	No
Kinetic Phosphorimeter	Daily Bkg Check Low Range	Boundary (<10,000)	No
Kinetic Phosphorimeter	Daily Source Check Low Range	Boundary (75-125%)	No

STANDARD OPERATING PROCEDURE

FOR UNCONTROLLED DOCUMENT

COLUMN PREPARATION

(GL-EPI-E-I-013

PROPRIETARY INFORMATION

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

Process Owner	3/3/99 (Sign and Date)	Tim C. Winters (Print Name)
Technical Review	9/3/99 (Sign and Date)	Richard M. Kinney (Print Name)
Quality Review	9/7/99 (Sign and Date)	Gene C. Bost (Print Name)
Approval and Authorization	(Sign and Date)	James B. Whitmire, Jr. (Print Name)

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TABLE OF CONTENTS

1.0 STANDARD OPERATING PROCEDURE FOR COLUMN PREPARATION

2.0 METHOD OBJECTIVE

2.1 This standard operating procedure provides the necessary instructions for the preparation of Eichrom resin columns.

3.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

3.1 Care should be taken when handling nitric acid as contact with skin may cause severe burns.

4.0 APPARATUS AND MATERIALS

4.1 Ancillary Equipment

4.1.1 Columns (EG&G WALLAC)

4.1.2 Column Caps (EG&G WALLAC)

4.1.3 Column Discs (EG&G WALLAC)

4.1.4 1000 mL poly bottles

4.1.5 Magnetic stirring bars

4.1.6 Magnetic stirrer

4.1.7 5 mL pipette

4.1.8 5 mL pipette tips

4.1.9 Column boxes

4.1.10 Permanent colored markers (Red, Green, Blue, Black)

4.1.11 Column racks

4.2 Reagents, Chemicals, Standards

4.2.1 Type II deionized water

4.2.2 Nitric acid

5.0 OPERATING PROCEDURE

5.1 Place 100 gms of dry resin, 700 mLs of DI water, and 2 mls of concentrated nitric acid in a 1000 mL poly bottle. Agitate vigorously and allow the resin to sit until it settles out in the bottom of the bottle.

NOTE: This will take about 24 hours.

5.2 Place columns in the column racks. Pre-soak bottom discs with 1 mL of DI water for about one hour. This allows the air in the discs to be displaced by the DI water and prevents flow problems from occuring.

5.3 When preparing columns, place a magnetic stirring bar in the prepared resin bottle. Stir resin on a magnetic stirrer and aliquot 5 mLs into each column.

NOTE: Minimize the time resin is stirring as prolonged agitation will break down the resin and seriously affect column flow rates.

5.4 Let the resin settle in the columns. Do a quick visual check to see that the resin level in the columns are all equal.

5.5 Remove excess water. If top disc is required, place pre-soaked disc in column. Rinse to ensure that there is no resin left on top of the disc. Place caps on columns and then place columns in boxes.

5.6 Color code the column caps with a permanent marker as follows:

Black	=	Tru resin
Red	=	Teva resin
Blue	=	Sr resin
Green	=	Ln resin

5.7 Label boxes and record information in the column prep log book (Appendix 1).

APPENDIX 1

DATE	LOT #	RESIN TYPE	BOX #	INITIALS	COMMENTS

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VOLUME RAD 2
TABLE OF CONTENTS

VOLUME RAD 2
TABLE OF CONTENTS

VOLUME RAD 2
TABLE OF CONTENTS

STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF GROSS ALPHA

AND GROSS NON-VOLATILE BETA

IN WATER

CONTROLLED DOCUMENT

(GL-EPI-A-001 Revision 3)

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Tim C. Winters 7/27/99		
Process Owner (sign & date)		(print name)
Richard M. Kinney 7/27/99		Richard M. Kinney
Technical Review (sign & date)		(print name)
7/27/99		Gene G. Bast
Quality Review (sign & date)		(print name)
James B. Westmoreland 7/27/99		James B. Westmoreland
Approval and Authorization (sign & date)		(print name)

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TABLE OF CONTENTS

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1.0 THE DETERMINATION OF ALPHA AND NON-VOLATILE BETA IN WATER

2.0 METHOD OBJECTIVE AND PURPOSE

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for gross alpha and non-volatile beta emitting isotopes in water.

2.2 GEL utilizes methods that are derived from established sources. This method is based on the source method EPA 600 4-80-032 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water" August 1980, Method 900.0, and uses similar principles of radiochemical concentration and counting.

3.0 METHOD SUMMARY

A sample is evaporated from a measured volume, then quantitatively transferred to a planchet. The planchette is flamed, allowed to cool, and weighed. The sample is then counted in a gas flow proportional counter at the appropriate voltage to simultaneously count alpha and beta activity.

4.0. SAMPLE COLLECTION AND PRESERVATION

A representative sample must be collected from a source of water and should be large enough so that adequate aliquots can be taken to obtain the required sensitivity. Samples should be preserved at the time of collection with HNO_3 to pH=2. If samples are collected without preservation, they should be brought to the laboratory within 5 days and preserved in the original container for a minimum of 16 hours before analysis. The container of choice should be plastic over glass to prevent loss due to breakage during handling.

5.0 INTERFERENCES

Some types of dissolved solids, when converted to nitrate salts, are quite hygroscopic even after dried. When such hygroscopic salts are present with samples that are put into an automatic counting system, those samples gain weight while they are waiting to be counted and inaccurate counting data can result. This problem is minimized by flaming the samples and storing in a desiccator.

6.0 APPARATUS

6.1 Gas-flow proportional counting system

6.2 Stainless steel planchets (2" x 1/8"), concentric ring

6.3 Electric hot plates

6.4 Various sized beakers

6.5 Desiccator

6.6 Analytical balance

7.0. REAGENTS

7.1 Type II deionized water

7.2 Nitric acid, concentrated

8.0. PROCEDURE

8.1 Transfer an aliquot to an appropriate sized beaker. Record the information on the que sheet (Appendix 1).

NOTE: A volume size should be chosen so that no more than 100 mg of total water solids concentrate onto the planchet.

Evaporate the aliquot to near dryness on a hot plate and add 5 mL concentrated Nitric acid. Repeat the evaporation and addition of 5 mL concentrated nitric acid. Quantitatively transfer the concentrate in small portions (not more than 5 mL. at a time) to a preweighed planchet, evaporating each portion to near dryness. Rinse the beaker with 10% HNO_3 and transfer to the planchet. Dry planchets on a hot plate. Avoid splattering by reducing heat as necessary.

8.2 Flame planchetted samples to a dull red with a burner to convert the nitrate salts to oxides before weighing and counting. Rotate the samples over the flame to ensure a complete conversion to oxide.

NOTE: Drinking water samples are not flamed.

8.3 FOR DRINKING WATER SAMPLES ONLY:
Dry the sample residue in a drying oven at 105°C for at least two hours. Cool in a desiccator; weigh, and count. Store the sample residue in a desiccator until ready for counting.

NOTE: Some types of water dissolved solids, when converted to nitrate salts, are quite hygroscopic even after being dried at 105°C for two hours. When such hygroscopic salts are present with samples that are put into an automatic counting system, those samples gain weight while they are waiting to be counted. When there is evidence of hygroscopic salts in sample counting planchets, it is recommended that they be flamed for a few minutes to convert the nitrate salts to oxides before weighing and counting.

8.4 Allow the sample to cool and weigh the sample residue. Store the sample residue for a minimum of 4 hours until ready for counting.

8.5 Count in a gross alpha beta counter for a time period that will meet the contract required detection limit (CRDL).

9.0. QUALITY CONTROL

9.1 A matrix spike (MS) should be run with every batch of 20 samples. The MS is calculated as follows:

9.1.1 $$MS(\%) = \frac{\text{SPIKE(pCi/l)} - \text{SAMPLE (pCi/l)}}{\text{SPIKE CONCENTRATION (pCi/l)}} * 100\%$$

A Duplicate of a sample should be run with every batch of 20 or less samples.

9.2 Relative percent difference (RPD) between the duplicate (DUP) and the sample should be less than or equal to 20%. The RPD is calculated as follows:

9.2.1 $$RPD(\%) = \frac{\text{HIGH DUP (pCi/l)} - \text{LOW DUP (pCi/l)}}{\text{AVERAGE (pCi/l)}} * 100\%$$

9.3 A method blank should accompany each batch of 20 samples. The reported value should be less than or equal to the CRDL.

10.0 CALCULATIONS

10.1 The instrument or spreadsheet will calculate the alpha radioactivity, alpha uncertainty, and alpha MDA (minimum detectable activity) in pCi/L by the following equation:

10.1.1 $$\text{ALPHA} = \frac{(A_{NET})}{(2.22)(C)(V)}$$

10.1.2 $$\text{ALPHA}_{\text{UNCERTAINTY}} = \frac{(1.96)\sqrt{\frac{B+A}{ct}}}{(2.22)(C)(V)}$$

10.1.3 $$\text{MDA}_{\text{ALPHA}} = \frac{2.71+4.66\sqrt{\frac{B}{ct}}}{(2.22)(C)(V)(ct)}$$

Where:

A_{NET} = (A-B)-D*beta crosstalk
A = alpha count rate
B = alpha background count rate
C = alpha efficiency factor, from graph of efficiency versus mg of solids per cm^2 of planchet area
D= beta count rate
V = volume of sample aliquot (L)
2.22 = conversion factor from dpm to pCi
ct = count time (min)

10.2 The instrument or spreadsheet will calculate the beta radioactivity, beta uncertainty, and beta MDA in pCi/L by the following equations:

10.2.1 $$\text{BETA} = \frac{(A_{NET})}{(2.22)(C)(V)}$$

10.2.2 $$\text{BETA}_{\text{UNCERTAINTY}} = \frac{(1.96)\sqrt{\frac{A+B}{ct}}}{(2.22)(C)(V)}$$

10.2.3 $$MDA_{BETA} = \frac{2.71 + 4.66\sqrt{\frac{B}{ct}}}{(2.22)(D)(V)(ct)}$$

Where:

A_{NET} = (A-B) – C*Alpha Crosstalk

A = beta count rate at the beta voltage plateau.

B = beta background count rate

C = alpha count rate

D = beta efficiency factor, from the graph of efficiency versus mg of solids per cm^2 of planchet area.

V = volume of sample aliquot (L)

2.22 = conversion factor from dpm to pCi

ct = count time(min)

11.0 CALIBRATION

11.1 Plateau Generation

11.1.1 In the alpha/beta mode, only the beta plateau needs to be determined. The plateau curve should be established with sources that simulate the samples to be analyzed. The standard used to perform the beta plateau is Sr-90.

11.1.2 A plateau is generated by a series of source counts while increasing (stepping) the high voltage applied to the detector after each count. This procedure should be performed with small voltage increments (e.g. 10 V but no more than 50V per step) from about 800V to about 1500V.

11.1.3 After a source is counted, a graph should be prepared which plots high voltage vs. the total number of counts accumulated at each high-voltage setting.

11.1.4 The optimum operational voltage for simultaneous alpha/beta operation is approximately 50V above the knee of the curve. The knee is determined by drawing straight lines along the rising slope and the plateau portions of the curve. The knee is the point where these two lines intersect.

11.2 Efficiency Determination

11.2.1 The instrument is calibrated using NIST traceable Th-230 and Sr-90 sources. The efficiency source activity should be large enough that calibration data on that source can be acquired in a reasonable amount of time, yet without creating too much dead time in the instrument (usually 10,000 DPM)

11.2.2 Calibration quench curves are generated by adding a known amount of activity to 8 to 12 preweighed planchettes. Separate standards are prepared for alpha and beta. The activity is added to standards of increasing solids. Record the initial weights of the planchettes on the standard preparation sheet. (Appendix 2)

11.2.3 Increasing amounts of barium chloride is used in the preparation of the calibration standards to vary the weights. The first standard should have no barium chloride added to it. The weights should vary from about 0 mg to just over 100 mg.

11.2.4 The planchettes are evaporated to dryness on a low heat hotplate. They are then allowed to cool and weighed again. Record the final weights on the standard preparation sheet.

11.2.5 Each standard is then counted in each detector to be calibrated for a time sufficient enough to accumulate at least 10,000 counts.

11.2.6 Efficiency of each detector is then plotted as a function of weight.

11.3 Crosstalks

11.3.1 Alpha crosstalk is the fraction of all alpha counts recorded in the beta channels. The alpha crosstalk values are determined by counting a pure alpha emitting source (e.g. Po-210) in each detector.

11.3.2 Beta crosstalk is the fraction of all beta counts recorded in the alpha channels. The beta crosstalk values are determined by counting a pure beta emitting source (e.g. Sr-90) in each detector.

11.4 Verifications

11.4.1 The gross alpha and beta calibration curves will be verified by running calibration standards (at different weights) against the curves. The

acceptance criteria being 100% ± 10% accuracy measured against a known value.

12.0 REFERENCES

12.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, USEPA, Method 900.00, pp. 1-9, August 1980

12.2 Standard Methods for the Examination of Water and Wastewater, 14th edition, American Public Health Association, Washington, DC, (1976)

12.3 1979 Annual Book of ASTM Standards, Part 31, American Society for Testing and Materials, Philadelphia, PA, (1979)

12.4 Friedlander, G., J. W. Kennedy, and J. Miller, Nuclear and Radiochemistry, John Wiley and Sons, Inc., New York, NY, (1964)

12.5 Youden, W. J. and F. J. Massey, Jr., Introduction to Statistical Analysis, 3rd edition, McGraw-Hill.

12.6 Harley, J. H., N. A. Hallden, and I. M. Fisenne, "Beta Scintillation Counting with Thin Plastic Phosphors", Nucleonics, pp. 20-59, (1961)

12.7 Hallden, N. A. and J. H. Harley, "An Improved Alpha-Counting Technique," Analytical Chemistry, Vol. 32:1861, (1960).

12.8 American National Standard, Calibration and Usuage of Alpha/Beta Proportional Counters, ANSI N42.25 1997.

APPENDIX 1

Gross Alpha Beta Que Sheet Batch #: 92624 Analyst: Jeff Overnight Comments:

Alpha Spike Isotope: Th-230 Spike Code:_____ Vol:_____ Prep Date:_____
Beta Spike Isotope: Sr-90 Spike Code:_____ Vol:_____ Initials :_____
LCS Isotope: Th-230/Sr-90 LCS Code: _____/_____ Vol:_____ Minimum Duedate: 11/15/96

Sample ID	Client Description		RDL		Client	Tests Requested		Matrix	BKR #	Vol (mL or g)	Carrier or Det #	Initial Weight (g)	Final Weight (g)	Net Weight (mg)
9610415-01	ASB 9		1.5	PCL		A	B	GroundH2O						
9610415-02	ASB 4		1.5	PCL		B	A	GroundH2O						
9610415-03	ASB 3AR		1.5	PCL		A	B	GroundH2O						
9610415-04	ASB 6A		1.5	PCL		A	B	GroundH2O						
9610415-05	ASB 2AR		1.5	PCL		A	B	GroundH2O						
9610415-06	ASB 1A		1.5	PCL		B	A	GroundH2O						
9610415-07	HSB109C		1.5	PCL		A	B	GroundH2O						
9610415-08	HSB110D		1.5	PCL		A	B	GroundH2O						
9610415-09	HSB117D		1.5	PCL		A	B	GroundH2O						
9610415-10	HSB110C		1.5	PCL		A	B	GroundH2O						
9610415-11	BGO 29D		1.5	PCL		B	A	GroundH2O						
9610415-12	BGO 29C		1.5	PCL		A	B	GroundH2O						
9610415-13	BGO 45D		1.5	PCL		A	B	GroundH2O						
9610415-14	BGO 29A		1.5	PCL		A	B	GroundH2O						
9610415-15	BGO 27D		1.5	PCL		A	B	GroundH2O						
9610415-16	BGO 45C		1.5	PCL		B	A	GroundH2O						
9610415-17	BGO 47A		1.5	PCL		A	B	GroundH2O						
9610415-18	BGO 45A		1.5	PCL		A	B	GroundH2O						
9610415-19	BGO 45B		1.5	PCL		A	B	GroundH2O						
9610415-20	BGO 47C		1.5	PCL		A	B	GroundH2O						
QC395272	blank	BLANK	1.5	pCi/L	QC	A	B	GroundH2O						
QC395273	9610415-20	DUP	1.5	pCi/L	QC	A	B	GroundH2O						
QC395274	9610415-20	MS	1.5	pCi/L	QC	A	B	GroundH2O						
QC395275	lcs	LCS	1.5	pCi/L	QC	A	B	GroundH2O						

Instrument Used: (Circle One) LB4110 S/N: 3219 HT1000 S/N: 10912771 Data Reviewed By:_____
LB5100 S/N: 203 LB5100W S/N: 14740

APPENDIX 2

Applicable SOP Number _____ Isotope _____

Date Standards Prepared _____ Cocktail Type Used _____

Standard ID _____ Matrix of Vial/Planchett _____

Amount Used (g or ml) _____

Standard Activity (DPM/g or mL) _____ Type of Scintillation Vial _____

Reference Date _____ Pipette ID Used _____

Expiration Date _____ Balance ID Used _____

Residue/Carrier Agent _____ Quenching Agent _____

EPI Samplenumber	Standard Number	Quenching Vol (uL)/ Residue Volume(mL)	Initial Wt. (g)	Final Wt. (g)	Net Wt. (mg)

Prepared By: _____ Date _____

Reviewed By: _____ Date _____

STANDARD OPERATING PROCEDURE

FOR

UNCONTROLLED DOCUMENT

THE DETERMINATION OF GROSS ALPHA AND GROSS NON-VOLATILE BETA IN SOIL

(GL-EPI-A-001b REV3)

6/3/99 — Tim Winters

Process Owner (Sign and Date) (Print Name)

6/4/99 — Richard M. Kinney

Technical Review (Sign and Date) (Print Name)

6/4/99 — Genie G. Bost

Quality Review (Sign and Date) (Print Name)

James B. Westmoreland

Approval and Authorization (Sign and Date) 6/4/99 (Print Name)

SET I.D.

TABLE OF CONTENTS

1.0 DETERMINATION OF GROSS ALPHA AND GROSS NON-VOLATILE BETA IN SOIL

2.0 METHOD OBJECTIVE AND APPLICABILITY

This standard operating procedure provides the necessary instructions to conduct the analysis for gross alpha and non-volatile beta emitting isotopes in soil.

3.0 INTERFERENCES

Some types of dissolved solids, when converted to nitrate salts, are quite hygroscopic even after dried. When such hygroscopic salts are present with samples that are put into an automatic counting system, those samples gain weight while they are waiting to be counted and inaccurate counting data can result. Flaming the samples minimizes this problem.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 Care should be taken when handling HF, HNO3, and HCl as contact with skin may cause severe burns.

4.2 Keep HF out of glass containers. HF reacts violently with glass.

4.3 Vent the microwave bombs under a hood and point away from the body. When contents of the bombs are under pressure, caustic fumes may be emitted.

5.0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment

5.1.1 Stainless steel planchets (2" x 1/8"), concentric ring

5.1.2 Electric hot plates

5.1.3 Desiccator

5.1.4 Analytical balance

5.1.5 Microwave oven

5.1.6 50 mL centrifuge tubes

5.1.7 Microwave bombs (100 mL)

5.1.8 100 mL Teflon® beakers

5.2 Reagents, Chemicals and Standards

5.2.1 Type II deionized water

5.2.2 Nitric acid, concentrated

5.3 Instrumentation

5.3.1 Gas-flow proportional counting system

6.0 SAMPLE COLLECTION AND PRESERVATION

A representative sample must be collected from a source of soil and should be large enough (50-100 grams) so that adequate aliquots can be taken to obtain the required sensitivity. The container of choice should be plastic over glass to prevent loss due to breakage during handling.

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to EPI SOP "HT-1000 Gross Alpha/Beta Counter Operating Instructions" (I-002) or EPI SOP "LB4110 Gross Alpha/Beta Counter Operating Instructions" (I-006) for instructions concerning gas flow proportional counters.

7.2 Refer to EPI SOP "Counting Room Instrumentation Maintenance and Performance Checks" (I-010) for instructions concerning instrument maintenance.

8.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

Refer to EPI SOP "Preparation of Radioactive Standards" (M-001) for instructions concerning the preparation of standard solutions.

9.0 OPERATING PROCEDURE

9.1 Sample Preparation Techniques

9.1.1 Homogenize a representative aliquot of soil sample as outlined in EPI SOP "Soil Preparation for the Determination of Radionuclides" (A-021). Digest a representative aliquot of the dried and homogenized soil according to EPI SOP "Microwave Digestion of Soil and Sand" (A-015). Record applicable information on the que sheet (Attachment 1).

9.1.2 Evaporate the digested sample to less than 5 mL.

9.1.3 Transfer the sample to a labeled and pre-weighed planchet. Record the aliquot volume on the que sheet (Attachment 1).

9.1.4 Evaporate on a low heat hot plate.

9.1.5 Flame plancheted samples to a dull red with a burner to convert the nitrate salts to oxides before weighing and counting. Rotate the samples over the flame to ensure a complete conversion to oxide.

9.1.6 Allow the sample to cool and weigh the sample residue. The net solids should be less than 100 mg. If the weight is >100 mg, then reprep sample with a smaller aliquot.

9.1.7 Store the sample residue for a minimum of 4 hours before counting.

9.1.8 Count in a gross alpha beta counter for a time period that will meet the contract required detection limit.

9.2 Instrument Calibration

9.2.1 Plateau Generation

9.2.1.1 In the alpha/beta mode, only the beta plateau needs to be determined. The plateau curve should be established with sources that simulate the samples to be analyzed. The standard used to perform the beta plateau is Sr-90.

9.2.1.2 A plateau is generated by a series of source counts while increasing (stepping) the high voltage applied to the detector after each count. This procedure should be performed with small voltage increments (e.g., 10V but no more that 50V per step) from about 300V to about 1500V.

9.2.1.3 After a source is counted, a graph should be prepared which plots high voltage vs. the total number of counts accumulated at each high-voltage setting.

9.2.1.4 The optimum operational voltage for simultaneous alpha/beta operation is approximately 50V above the knee of the curve. The knee is determined by drawing straight lines along the rising slope and the plateau portions of the curve. The knee is the point where these two lines intersect.

9.2.2 Efficiency Determination

9.2.2.1 The instrument is calibrated using NIST traceable Th-230 and Sr-90 Sources. The efficiency source activity should be large enough that calibration data on that source can be acquired in a reasonable amount of time, yet without creating too much dead time in the instrument (usually 10,000 DPM).

9.2.2.2 Calibration quench curves are generated by adding a known amount of activity to 8 to 12 preweighed planchettes. Separate standards are prepared for alpha and beta. The activity is added to standards of increasing solids. Record the initial weights of the planchettes on the standard preparation sheet (Attachment 2).

9.2.2.3 Increasing amounts of barium chloride is used in the preparation of the calibration standards to vary the weights. The first standard should have no barium chloride added to it. The weights should vary from about 0 mg to just over 100 mg.

9.2.2.4 The planchettes are evaporated to dryness on a low heat hotplate. The are than allowed to cool and weighed again. Record the final weights on the standard preparation sheet.

9.2.2.5 Each standard is then counted in each detector to be calibrated for a time sufficient enough to accumulate at least 10,000 counts.

9.2.2.6 Efficiency of each detector is then plotted as a function of the weight.

9.2.3 Crosstalks

9.2.3.1 Alpha crosstalk is the fraction of all alpha counts recorded in the beta channels. The alpha crosstalk values are determined by counting a pure alpha emitting source (e.g. Po-210) in each detector.

9.2.3.2 Beta crosstalk is the fraction of all the beta counts recorded in the alpha channels. The beta crosstalk values are determined by counting a pure beta emitting source (e.g. Sr-90) in each detector.

9.2.4 Verifications

9.2.4.1 The gross alpha and beta calibration curves will be verified by running calibration standards (at different weights) against the curves. The acceptance criteria being 100% +-10% accuracy measured against a known value.

9.3 Instrument Performance Requirements

9.3.1 Refer to GEL SOP "HT1000 Gross Alpha/Beta Counter Operating Instructions" (EPI-I-002) or GEL SOP "LB4110 Gross Alpha/Beta Counter Operating Instructions" (EPI-I-006) for instructions concerning gas flow proportional counters.

9.3.2 Refer to GEL SOP "Counting Room Instrumentation Maintenance and Performance Checks" (EPI-I-010) for instructions concerning instrument maintenance.

10.0 CALCULATIONS AND DATA REDUCTION METHODS

10.1 The instrument or spreadsheet will calculate the alpha radioactivity in pCi/g by the following equations:

10.1.1 $$\text{Alpha} = \frac{A_{net}}{(2.22)(C)(g)}$$

10.1.2 $$\text{Alpha}_{\text{UNCERTAINTY}} = \frac{(1.96)\sqrt{\frac{B}{ctb} + \frac{A}{cts}}}{(2.22)(C)(g)}$$

10.1.3 $$\text{MDA}_{\text{ALPHA}} = \frac{\left(2.71 + 4.66\sqrt{(B)(ct)}\right)}{2.22 * C * g * ct}$$

Where: Anet = (A-B)-D*beta crosstalk
A = alpha count rate
B = alpha background count rate.
C = alpha efficiency factor, from graph of efficiency versus mg of solids per cm^2 of planchet area
D = count rate
g = grams used in the sample digestion.
2.22 = conversion factor from dpm to pCi.
ctb = background count time (min.)
A-B = net count rate
cts = sample count time

10.2 The instrument or spreadsheet will calculate the non volatile beta radioactivity in pCi/g by the following equations:

10.2.1 $$\text{BETA} = \frac{A_{net}}{(2.22)(C)(g)}$$

10.2.2 $$\text{BETA}_{\text{UNCERTAINTY}} = \frac{(1.96)\sqrt{\frac{A}{cts} + \frac{B}{ctb}}}{(2.22)(D)(g)}$$

10.2.3 $$\text{MDA} = \frac{2.71 + 4.66\sqrt{(B)(ct)}}{2.22 * D * ct * g}$$

Where: Anet = (A-B)-C*alpha crosstalk
A = beta count rate
B = background count rate
C = alpha count rate
D = beta efficiency factor, from the graph of efficiency versus mg of solids per cm^2 of planchet area.
g = grams used in sample digestion
2.22 = conversion factor from dpm to pCi
cts = sample count time (min.)
ctb = background count time
A-B = net count rate

11.0 DATA RECORDING, REVIEW AND REPORTING

11.1 Data Recording.

Record the information required the gross alpha beta in water on the que sheet (Appendix 1).

11.2 Data Review

Refer to EPI SOP "Data Review and Validation Procedures" (D-003) for instructions concerning the data review process.

11.3 Data Reporting.

The analyst will take the applicable gross alpha beta spreadsheet and enter the data into LIMS. The following information should be included: analyst's initials, date and time the sample was counted, sample results and accuracy in pCi/g.

12.0 QUALITY CONTROL REQUIREMENTS

12.1 Analyst and Method Verification Requirements

Refer to EPI SOP "Analyst and Analytical Methods Validation Procedures" (D-002) for instructions concerning the validation of analysts and analytical methods.

12.2 Method Specific Quality Control Requirements

12.2.1 A matrix spike(MS) should be run with every batch of 20 or less samples. The MS is calculated as follows:

$$MS(\%) = \frac{SPIKE(pCi/g) - SAMPLE(pCi/g)}{SPIKE\ CONCENTRATION(pCi/g)} * 100\%$$

12.2.2 A duplicate of a sample should be run with every batch of 20 or less samples. The relative percent difference (RPD) between the duplicate (dup) and the sample should be less than or equal to 20%. The RPD is calculated as follows:

$$RPD(\%) = \frac{HIGH\ DUP(pCi/g) - LOW\ DUP(pCi/g)}{AVERAGE(pCi/g)} * 100\%$$

12.2.3 A method blank should accompany each batch of 20 or less samples. The reported value should be less than or equal to the contract required detection limit (CRDL).

12.3 Actions Required if the Quality Control Requirements Are Not Met.

12.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non-conformance report as outlined in GEL SOP "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

12.3.2 If the contract MDA cannot be satisfied due to high sample weight and low sample volume, with a count time of 480 minutes, the client should be notified of the matrix interference.

13.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

13.1 Each analysis performed on the LB-4110 is automatically documented in the run log of that particular instrument.

13.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

14.0 LABORATORY WASTE HANDLING AND DISPOSAL

Radioactive material is handled and disposed of as outlined in EPI SOP "Radioactive Waste Handling Procedures" (S-005).

15.0 REFERENCES

15.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, USEPA, Method 900.00, pp. 1-9, (Aug. 1980).

15.2 Standard Methods for the Examination of Water and Wastewater, 14th edition, American Public Health Association, Washington, DC (1976).

15.3 1979 Annual Book of ASTM Standards, Part 31, American Society for Testing and Materials, Philadelphia, PA (1979).

15.4 Friedlander, G., J. W. Kennedy, and J. Miller, Nuclear and Radiochemistry, John Wiley and Sons, Inc., New York, New York, (1964).

15.5 Youden, W. J. and F. J. Massey, Jr., Introduction to Statistical Analysis, 3rd edition, McGraw-Hill,

15.6 Harley, J. H.., N. A. Hallden, and I. M. Fisenne, "Beta Scintillation Counting with Thin Plastic Phosphors", Nucleonics, p. 20-59, (1961).

15.7 Hallden, N. A. and J. H. Harley, "An Improved Alpha-Counting Technique", Analytical Chemistry, p. 32:1861, (1960).

15.8 American National Standard, Calibration and Usage of Alpha/Beta Proportional Counters, ANSI N42.25-1997

ATTACHMENT 1

Gross Alpha Beta Que Sheet

Batch #: 150170 Analyst: Tom Cushman Comments:

Alpha Spike isotope: Th-230 Spike Code:_____ Vol:_____ Balance #:_____ Prep Date:_____
Beta Spike isotope: Sr-90 Spike Code:_____ Vol:_____ Pipet #:_____ Initials :_____
LCS isotope: Th-230/Sr-90 LCS Code: _____/_____ Vol:____/____ Minimum Duedate: 6/4/1999

Sample ID	Client Description		RDL	Client	Tests Requested	Matrix	BKR #	Vol (mL or g)	Carrier or Det #	Initial Weight (g)	Final Weight (g)	Net Weight (mg)
9905709-10	BGO 19DR		2 PCL	WSRC	A B	GroundH2O						
9905709-11	BGO 11DR		2 PCL	WSRC	A B	GroundH2O						
9905709-12	BGO 6B		2 PCL	WSRC	A B	GroundH2O						
9905709-13	BGO 3C		2 PCL	WSRC	A B	GroundH2O						
9905730-01	BGO 13DR		2 PCL	WSRC	A B	GroundH2O						
9905730-02	BGO 17DR		2 PCL	WSRC	A B	GroundH2O						
9905730-03	BGO 18D		2 PCL	WSRC	A B	GroundH2O						
9905882-01	BGO 7D		2 PCL	WSRC	A B	GroundH2O						
9905926-01	GSC-38D		1.5 pCi/	GSTL	B A	GroundH2O						
9905926-02	GSC-38S		1.5 pCi/	GSTL	A B	GroundH2O						
9905926-03	GSC-1S		1.5 pCi/	GSTL	A B	GroundH2O						
9905926-04	GSC-1D		1.5 pCi/	GSTL	A B	GroundH2O						
9905926-05	GSC-2S		1.5 pCi/	GSTL	A B	GroundH2O						
9905926-06	GSC-2D		1.5 pCi/	GSTL	A B	GroundH2O						
9905926-07	DH-8		1.5 pCi/	GSTL	A B	GroundH2O						
9905926-08	B-15A		1.5 pCi/	GSTL	A B	GroundH2O						
9905926-09	GSC-4D		1.5 pCi/	GSTL	A B	GroundH2O						
9905926-10	GSC-4S		1.5 pCi/	GSTL	A B	GroundH2O						
9905926-11	GSC-5D		1.5 pCi/	GSTL	A B	GroundH2O						
9905926-12	GSC-5S		1.5 pCi/	GSTL	A B	GroundH2O						
QC615839	blank	LANK	1.5 pCi/	QC	A B	GroundH2O						
QC615840	9905882-01	DUP	1.5 pCi/	QC	A B	GroundH2O						
QC615841	9905882-01	MS	1.5 pCi/	QC	A B	GroundH2O						
QC615842	lcs	LCS	1.5 pCi/	QC	A B	GroundH2O						

Instrument Used: (Circle One) LB4110 S/N: 3219 HT1000 S/N: 10912771 Data Reviewed By:_____
LB5100 S/N: 203 LB5100W S/N: 14740

ATTACHMENT 2

General Engineering Laboratories
Calibration Source Preparation Sheet

Applicable SOP Number _____ Isotope _____
Date Standards Prepared _____ Cocktail Type Used _____
Standard ID _____ Matrix of Vial/Planchet _____
Amount Used (g or ml) _____ _____
rd Activity (DPM/g or mL) _____ Type of Scintillation Vial _____
Reference Date _____ Pipette ID Used _____
Expiration Date _____ Balance ID Used _____
Residue/Carrier Agent _____ Quenching Agent _____

Sample numbe	Standard Number	uenching Vol (uL Residue Volume(n	Initial Wt. (g)	Final Wt. (g)	Net Wt. (mg)

Prepared By: _____ Date _____

Reviewed By: _____ Date _____

Rev 1 RLM 9/10/97

STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF GROSS ALPHA IN WATER BY COPRECIPITATION

UNCONTROLLED DOCUMENT

Process Owner(s):	Tim C. Winters, EPI Team Task Leader
Technical Review by:	Russell L. Moser, EPI Group Leader
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	James B. Westmoreland, EPI Laboratory Manager

SET I.D.

1.0 STANDARD OPERATING PROCEDURES FOR THE DETERMINATION OF GROSS ALPHA IN WATER BY COPRECIPITATION

2.0 METHOD OBJECTIVE AND APPLICABILITY

An aliquot of drinking water is acidified with H_2SO_4. Barium carrier is added to precipitate barium sulfate and the sample is digested while warming up to 50°C for 30 minutes. Iron carrier is then added. After 30 minutes the sample is neutralized with dilute NH_4OH to precipitate ferric hydroxide. The sample is filtered and the precipitate is radioassayed after drying. This procedure precipitates radium and all alpha and beta emitting actinide elements.

3.0 INTERFERENCES

3.1 Excess dissolved solids in the sample can yield a heavy precipitation on the filter. If greater than 100 mg is observed on the filter, the alpha particles will be highly quenched. The sample will be rerun with less volume to achieve a net of <100 mg to compensate for this interference.

3.2 Radon and short lived daughter isotopes dissolved in the sample can yield erratic results. It is important to bring the sample to a vigorous boil to remove radon-222 and to wait the prescribed 3 hours to allow the short lived progeny to decay.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

Care should be taken when handling NH_4OH, HNO_3, and H_2SO_4 as contact with skin may cause severe burns.

5.0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment

5.1.1 Drying lamps

5.1.2 Filter membranes, 47 mm diameter, 0.45 micrometer pore size.

5.1.3 Glassware

5.1.4 Planchets, stainless steel, 2 inch diameter

5.2 Reagents, Chemicals and Standards

5.2.1 Ammonium hydroxide, 6 M

5.2.2 Nitric acid, 16 M, 70% HNO_3 reagent.

5.2.3 Barium carrier, 5 mg Ba^{+2}/mL. Dissolve 4.4 g $BaCl_2*2H_20$ in 500 mL DI water.

5.2.4 Bromocresol purple, 0.1 percent. Dissolve 100 mg of the water soluable reagent in 100 mL DI water.

5.2.5 Iron carrier, 5 mg Fe^{+3}/mL. Dissolve 17.5 g $Fe(NO_3)_3*9H_2O$ in 200 mL DI water containing 2 mL 16 M HNO_3. Dilute to 500 mL.

5.2.6 Sulfuric acid, 1 M. Dilute 55 mL of the 96% reageant grade H_2SO_4 to 1 liter with DI water.

5.3 Instrumentation

5.3.1 Gas-flow proportional counting system

6.0 SAMPLE COLLECTION & PRESERVATION

A representative sample must be collected from a source of water and should be large enough so that adequate aliquots can be taken to obtain the required sensitivity. Samples should be preserved at the time of collection with HNO_3 to pH=2. If samples are collected without preservation, they should be brought to the laboratory within 5 days and preserved in the original container for a minimum of 16 hours before analysis. The container of choice should be plastic over glass to prevent loss due to breakage during handling.

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to EPI SOPs "HT-1000 Gross Alpha/Beta Counter Operating Instructions" (I-002), or "LB-4110 Gross Alpha/Beta Counter Operating Instructions" (I-006) for instructions concerning gas flow proportional counters.

7.2 Refer to EPI SOP "Counting Room Instrumentation Maintenance and Performance Checks" (I-010) for instructions concerning instrument maintenance.

8.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

8.1 All standards used in this method must be NIST traceable.

8.2 Dilution of standards should be noted in standard logbook in keeping with the EPI SOP "Preparation of Radioactive Standards" (M-001) for labeling and tracking of the standards. The weight of the standard to be diluted and diluting solutions should be noted in the standards logbook. The weight should be taken on a

balance of at least four places and calculation of the new standard activity carried out and noted in appropriate significant figures.

8.3 A reagent blank should be used as the batch blank.

8.4 A duplicate and spiked sample should be made on the same sample in a sample batch.

8.5 A laboratory control sample (LCS) should be made by spiking deionized water with a known quantity of analyte isotope.

9.0 OPERATING PROCEDURE

9.1 Sample Preparation Techniques

9.1.1 Use a measured aliquot of water sample. if the sample is less than 500 mL, dilute to 500 mL with DI water. Record the aliquot on the que sheet (Appendix 1).

9.1.2 If the sample used is preserved with acid, neutralize to color change with 6 M NH_4OH using 1 mL bromocresol purple.

9.1.3 Add 20 mL 1 M H_2SO_4 and flush the radon from the sample by boiling for a minimum of two minutes.

9.1.4 Allow the sample to stand for a minimum of three hours for radon progeny to decay.

9.1.5 Add 1 mL barium carrier.

9.1.6 Heat the sample to approximately 50°C with stirring for 30 minutes.

9.1.7 Add 1 mL iron carrier and 1 mL bromocresol purple reagent.

9.1.8 Continue stirring and add 6 M NH_4OH dropwise to the sample until the indicator changes color.

9.1.9 Continue warming and stirring for another 30 minutes.

9.1.10 Filter sample through a pre weighed membrane filter and wash precipitate with 25 mL DI water.

9.1.11 Dry the filter for a minimum of one hour at 100°C. Allow the filter to cool and weigh. Record the filter gross weight on the que sheet (Appendix 1). Mount the filter membrane on a planchet using double stick tape or glue stick.

9.1.12 Store samples in a desiccator or dry carefully under a heat lamp if recounted at a later date.

9.2 Instrument Calibration

9.2.1 The instrument is calibrated with an NIST traceable Th-230 source. The sources are counted at the operating voltage and gain of the samples.

9.2.2 A calibration quench curve is generated by spiking with a known dpm of alpha activity from Th-230 . The activity is added to samples of increasing solids. A quench curve is derived by plotting percent efficiency versus mg of solids. The quench curve slope and intercept are used in the spreadsheet to calculate the sample activity.

9.2.3 The gross alpha and beta calibration curves will be verified by running two calibration standards (at different weights) against the new curve, with acceptance criteria being 100% ±10% accuracy measured against a known value.

9.3 Instrument Performance Requirements

9.3.1 Refer to EPI SOPs "HT-1000 Gross Alpha/Beta Counter Operating Instructions" (I-002), or "LB-4110 Gross Alpha/Beta Counter Operating Instructions" (I-006) for instructions concerning gas flow proportional counters.

9.3.2 Refer to EPI SOP "Counting Room Instrumentation Maintenance and Performance Checks" (I-010) for instructions concerning instrument maintenance.

9.4 Analysis Procedures and Instrumental Operation

9.4.1 After a minimum of 4 hours, count the sample on the gas flow proportional counter under the prescribed program for alpha activity.

9.4.2 If sample results are >100 pCi/L, verify results by reprepping another aliquot. Calculate RPD between the two counts. If RPD >40%, return to step 9.1.1.

10.0 CALCULATIONS AND DATA REDUCTION METHODS

10.1 Calculate the concentration, Z, of gross alpha in picocuries per liter (pCi/L) as follows:

$$\text{Activity (pCi/L)} = \frac{C_l - C_b}{2.22 * E * V}$$

$$\text{uncertainty (pCi/l)} = \frac{1.96\sqrt{\frac{C_l}{T_s} + \frac{C_b}{T_b}}}{2.22 * E * V}$$

where:

E	=	counter efficiency
V	=	volume analyzed (liters)
2.22	=	dpm/pCi
C_l	=	sample counts per minute
C_b	=	reagent blank counts per minute
Ts	=	Count time of sample
Tb	=	Count time of Blank

10.2 Calculate the minimum detectable activity (MDA) as follows:

$$MDA = \frac{2.71 + 4.66\sqrt{C_b T}}{2.22 * E * V * T}$$

where:

C_b	=	reagent background counts per minute
T	=	Sample count time
E	=	counter efficiency
V	=	sample volume in liters
2.22	=	dpm/pCi

11.0 DATA RECORDING, REVIEW AND REPORTING

11.1 Data Recording.

Record the information required for gross alpha in water on the que sheet (Appendix 1).

11.2 Data Review

Refer to EPI SOP "Data Review and Validation Procedures" (D-003) for instructions concerning the data review process.

11.3 Data Reporting

The analyst will take the applicable gross alpha beta spreadsheet and enter the data into LIMS. The following information should be included: analyst's initials, date and time the sample was counted, sample results, and accuracy in pCi/L.

12.0 QUALITY CONTROL REQUIREMENTS

12.1 Analyst and Method Verification Requirements

Refer to EPI SOP "Analyst and Analytical Methods Validation" (D-002) for information concerning analyst and method verification.

12.2 Method Specific Quality Control Requirements

12.2.1 A matrix spike (ms) should be run with every batch of samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows.

$$Rec = \frac{Spike(pCi/unit) - Sample(pCi/unit)}{No\ minal\ Concentration(pCi/unit)} * 100$$

12.2.2 A sample duplicate should be run with every batch of samples. The relative percent difference (RPD) between the sample and the sample duplicate should be less than or equal to 20%. If both results are greater than 5X MDA. If either result is between 5X MDA and MDA limit is 7 100. If either result is less than MDA then limits are not applicable. The RPD is calculated as follows.

$$\%RPD = \frac{High\ Activity(pCi/unit) - Low\ Activity(pCi/unit)}{Average(pCi/unit)} * 100$$

12.2.3 A method blank should accompany each batch of samples. The reported value should be less than or equal to requested MDA. If the value of the blank activity is not below the requested MDA then an adequate explanation should be given.

12.2.4 A Laboratory Control Sample (LCS) should be run with each batch of samples. The recovery of the LCS should fall between 75-125%. The recovery is calculated as follows:

$$\%\,Rec = \frac{Observed\ pCi/l}{Known\ pCi/l} * 100$$

12.3 Actions Required if the Quality Control Requirements Are Not Met

If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non conformance report as outlined in GEL SOP "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

13.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

13.1 Each analysis that is performed on the instrument is documented in the run log according to GEL SOP "Run Logs" (GL-LB-E-009).

13.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

14.0 LABORATORY WASTE HANDLING AND DISPOSAL

Radioactive material is handled and disposed of as outlined in EPI SOP "Radioactive Waste Handling Procedures" (S-005).

15.0 REFERENCES

15.1 Eastern Environmental Radiation Facility Radiochemistry Manual, EPA 520/5-84-006, Method 00-02-1. US EPA, P.O. Box 3009, Montgomery, AL 36193.

15.2 Krieger, H.L., and Whittaker, E.L., Prescribed Procedures for Meansurement of Radioactivity in Drinking Water, EPA-600/4-80-032. Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH. August 1980.

APPENDIX 1

Gross Alpha in Water by Coprecipitation Que Sheet

Comment: *none*

Batch # 38291 Prep Date:_____ Initials:_____

Spike Isotope:_____ Spike Code:_____ Vol:_____

LCS Isotope:_____ LCS Code:_____ Vol:_____

Instrument Used: (circle one)		LB5100W	HT1000	LB5100	Lb4110	Batched to:	
s/n:		14740	10912771	203	8219	Shane	Mull

Sample ID	*Sample Descrip.*	*Bkr #*	*Vol (mls)*	*Carrier or Det #*	*Initial Weight (g*	*Final Weight (g)*	*Net Weight (mg)*
9310365-09	QA 26D						
9310392-01	HSB109C						
9310392-02	HSB129C						
9310392-03	HSB134D						
9310392-04	HSB151D						
9310392-05	QA 71D						
9310392-06	QA 72D						
9310392-12	HTF 9						
9310392-13	HTF 10						
9310392-14	HTF 11						
9310392-15	HTF 12						
9310392-16	HTF 13						
9310392-17	HTF 14						
9310392-18	HTF 15						
9310392-19	HTF 18						
9310392-20	HTF 19						
9310392-21	HTF 20						
9310392-22	HTF 21						
9310392-23	HTF 34						
9310414-07	HSB130C						
QC151394	blank						
QC151395	9310414-07						
QC151396	9310414-07						
QC151397	lcs						

Data Reviewed By: _____

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STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF TRITIUM

UNCONTROLLED DOCUMENT

(GL-EPI-E-A-002 Revision 5)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Process Owner	8/10/99 (Sign and Date)	Tim C. Winters (Print Name)
Technical Review	8/26/99 (Sign and Date)	Richard M. Kinney (Print Name)
Quality Review	8/27/99 (Sign and Date)	Carie Bost (Print Name)
Approval and Authorization (Sign and Date)	8/30/99	James B. Wommack (Print Name)

SET I.D.

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310

TABLE OF CONTENTS

1.0 STANDARD OPERATING PROCEDURES FOR THE DETERMINATION OF TRITIUM

2.0 METHOD OBJECTIVE AND APPLICABILITY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for tritium in various matrices.

2.2 GEL utilizes methods that are derived from established sources. This method is based on the source method EPA 600 4-80-032, "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," August 1980, Method 906.0, and uses similar principles of radiochemical concentration and counting.

3.0 INTERFERENCES

3.1 Samples with color or chemical quenching agents may reduce the tritium counting efficiency. Generally, these problems are overcome by diluting the sample aliquot. However, in some cases distillation may be necessary.

3.2 Other beta emitters in the sample may bias the tritium results high.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 Carbon Tetrachloride is a toxic substance that should be handled with care. Follow all safety procedures when handling any chemical substance.

4.2 All radioactive materials are handled according to "Radioactive Material Handling Procedure,".(GL-EPI-S-004).

4.3 Follow manufacturer's safety precautions when handling Ready Safe® (Beckman) and Ultima Gold® (Packard), and TriSsafe (Wallac).

4.4 Care must be taken when handling liquid nitrogen. Insulated gloves and insulated containers are provided in the lab for handling nitrogen.

4.5 Inspect all glassware and equipment prior to use. Any cracks or chipped glassware should be discarded. If there is any question on the condition of the glassware or equipment, it should be repaired or replaced.

5.0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment

5.1.1 20 ml plastic scintillation vials and caps

5.1.2 5 ml Oxford® pipet or equivalent

5.1.3 10 ml Oxford® pipet or equivalent

5.1.4 Eppendorf® Repeater™ pipet or equivalent

5.1.5 Distillation apparatus

NOTE: All glassware used in this method must be pre-soaked in a solution of 10% nitric acid for a minimum of one hour, then rinsed with DI water prior to use.

5.1.6 Vacuum pump

5.2 Reagents, Chemicals and Standards

5.2.1 Type II deionized water

5.2.2 Background well water (fossil water)

5.2.3 Liquid scintillation cocktail (Beckman Ready Safe® or Packard Ultima Gold®), TriSafe (Wallac)

5.2.4 Potassium permanganate

5.2.5 Sodium Hydroxide pellets, reagent grade

5.2.6 Liquid Nitrogen

5.2.7 Carbon Tetrachloride

5.3 Instrumentation

5.3.1 Liquid Scintillation Counter

6.0 SAMPLE COLLECTION & PRESERVATION

6.1 The preferred method for sample collection is in a glass container. This is preferred due to the possibility of tritium exchange between the sample and container if plastic is used. Plastic containers may be used if sample breakage is more probable in large solid samples or for specific clients.

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to "Beckman LS-6000/6500 Operating Procedures," (GL-EPI-E-I-004) for instructions concerning instrument maintenance.

8.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

8.1 All standards used in this method must be NIST traceable.

8.2 Dilution of standards should be noted in standard logbook in keeping with accepted procedure, EPI SOP M-001 "Preparation of Radioactive Standards," (GL-EPI-E-M-001). The weight of the standard to be diluted and diluting solutions should be noted in the standards logbook. The weight should be taken on a balance of at least four places and calculation of the new standard activity carried out and noted in appropriate significant figures.

8.3 A "blank" water should be used for the batch blank and the laboratory control sample (LCS). The LCS is prepared by adding a known amount of a NIST standard and recording the applicable information on the que sheet (Appendix 5).

8.4 A duplicate and spiked sample should be made on the same sample in a sample batch. For the spiked sample, add a known amount of a NIST tritium standard and record the applicable information on the que sheet.

8.5 When measuring tritium for low level analysis, fossil water from deep wells is used in the preparation of samples and blanks. This water has been isolated from natural generation of tritium from the cosmic rays. The tritium concentration of this water is approximately 1,000 times lower than surface water

9.0 OPERATING PROCEDURE

From the sample matrix and client requirements, determine the procedure to be used. The project manager or group leader may need to be consulted.

9.1 Determination of Tritium via the Direct Counting of the Sample in a Sample Vial

9.1.1 Transfer a known amount of sample and a known amount of scintillation cocktail into a scintillation vial. The proper ratio between sample volume and scintillation cocktail is obtained from the calibration file maintained by the group leader. Record the sample volume on the Que sheet.

9.1.2 Add Carbon Tetrachloride (normally 10 uL) to bring the quench value

within the quench limits of the calibration. Cap the vial and shake vigorously to cause complete dissolution of the sample in the cocktail.

NOTE: A milky appearance is an indicator of cocktail loading. A volume should be chosen so that the vial appears clear. Consult the group leader or method development person if questions arise.

9.1.3 Wipe each sample vial with an anti-static cloth and place in a counting rack. Allow the samples to dark adapt, normally two hours, and count the samples under the proper program for sufficient time to meet client detection limits and error requirements.

9.2 Determination of Tritium Using the Distillation Apparatus (Appendix 1)

NOTE: These steps can be used for all solid or water matrices. When determining low level tritium, use steps 9.3 or 9.4.

9.2.1 Weigh a known amount of sample into a round bottom distillation flask. Record the weight on the Que sheet.

9.2.2 For samples with insufficient water content (at least 100-150 ml), add a known amount of water to the sample. Record this volume on the Que sheet.

9.2.3 For water samples and samples that do not have a noticeable color, add 50-100 mg of dry $KMnO_4$.

NOTE: The exact amount of $KMnO_4$ is not critical. The intent is to cause the sample to take on a purple appearance.

9.2.4 Add 5-10 pellets of NaOH.

9.2.5 Supply cooling water to the condenser and heat the distillation flask to boiling. Record distillation rig number on the Que sheet.

9.2.6 Collect the first 10 ml of the distillate and discard. Collect the next 10 mls of distillate and save for analysis.

9.2.7 Prepare the tritium sample for counting using steps in 9.1.

9.3 Determination of Tritium Using the Microwave (Appendix 2)

NOTE: This procedure is to be used for low level analysis of samples with low sample volumes when the direct method is not an option (i.e., 10-30

grams or ml of sample).

NOTE: All glassware used in this method must be pre-soaked in a solution of 10% nitric acid for a minimum of one hour, then rinsed with DI water prior to use.

9.3.1 All glassware must be completely dry. Glassware may be placed in an oven to aid in drying.

9.3.2 Weigh a known amount of sample into a digestion tube. Record the weight on the Que sheet.

9.3.3 For samples with insufficient water content (at least 10 ml), add a known amount of fossil water to the sample. Record this volume on the Que sheet.

9.3.4 For water samples and samples that do not have a noticeable color to them, add a small amount of dry $KMnO_4$.

NOTE: The exact amount of $KMnO_4$ is not critical. The intent is to cause the sample to take on a purple appearance.

9.3.5 Add 1-2 pellets of NaOH to water samples. Assemble the distillation flask to the condenser. Place a labeled centrifuge tube under the condenser

9.3.6 Supply cooling water to the condenser and microwave the sample for 10 minutes.

9.3.7 Prepare the tritium sample for counting using steps in 9.1.

9.4 Determination of Tritium using distillation Apparatus II (Appendix 3)

NOTE: All glassware used in this method must be pre-soaked in a solution of 10% nitric acid for a minimum of one hour, then rinsed with DI water prior to use.

9.4.1 Weigh a known amount of sample into an Erlenmeyer flask.

9.4.2 This step is used only if approved by the client. For solid samples add a known amount of water to the sample. Record this volume on the Que sheet.

9.4.3 Add 2 mL 1% $KMNO_4$, 4-6 NaOH pellets, and 3-6 boiling beads (water samples).

9.4.4 Assemble the Erlenmeyer flask to the air condenser. Record the distillation rig number on the Que sheet.

9.4.5 Supply cooling air to the condenser and place the distillation flask on a hotplate.

9.4.6 Allow the inner container to fill then discard the distillate. Allow the inner container to refill and save for analysis.

9.4.7 Prepare the tritium sample for counting using steps in 9.1.

9.5 Determination of Tritium Using the Vacuum Rig (Appendix 4)

NOTE 1 These steps are used for large samples being analyzed for low level tritium.

NOTE 2 This procedure is used to remove approximately 10 mL or more of water from the sample.

9.5.1 All residual moisture in the distillation system must be removed. If droplets are present, dry areas with tissue or place in oven to dry. This must be accomplished before proceeding.

9.5.2 Weigh the sample container to be used. Record this weight on the Que Sheet. (Appendix 5)

9.5.3 Tare Balance

9.5.4 Add sample to the sample container. Weigh the container and sample. Record weight on the Que Sheet.

9.5.5 Assemble the vacuum distillation system. Check vacuum pump oil.

NOTE: The vacuum pump must be started and stopped with the vent valve open. The pump cannot be started or stopped under load or pump damage may result.

9.5.6 Open or check open valves A, B, C and D. Start the vacuum pump.

9.5.7 If a rig is not connected to valve C or D, slowly close that associated valve. Slowly close valve A. When the pump has quieted, immerse the sample trap in liquid nitrogen.

9.5.8 Heat the sample by placing it on hotplate at low to medium heat.

9.5.9 Allow the sample to distill for about 20 minutes or until approximately 20 ml of water is collected.

9.5.10 Remove the heat source and close valve B. Open valve A and turn off the vacuum pump.

9.5.11 Remove sample trap from the liquid nitrogen and open associated isolation valve C or D.

9.5.12 Label the sample trap or melt sample inside trap and transfer sample to a centrifuge tube for sample preparation.

9.5.13 Place the sample container in the oven at 100° C for at least 6 hours. If after 6 hours the sample does not appear dry, place in oven until dryness is obtained.

9.5.14 Allow sample to cool. Weigh sample container and dried sample. Record weight on Que Sheet.

9.5.15 Wash all glassware and dry in oven at 100° C. If high activity sample is run or sample splatter is evident, clean the section between the sample and the sample trap and dry in oven at 100• C.

9.5.16 Prepare the tritium sample for counting using steps in 9.1.

9.6 Determination of Tritium Using the Oven

NOTE: All glassware used in this method must be pre-soaked in a solution of 10% nitric acid for a minimum of one hour, then rinsed with DI water prior to use.

9.6.1 Weigh a known amount of sample into a 1000 mL Erlenmeyer flask. Record this volume on the que sheet.

9.6.2 Prepare the tritium sample for counting using the steps in 9.1.

9.7 Instrument Calibration

9.7.1 The liquid scintillation counter is calibrated by counting 8 to 12 standards with equal activity and different amounts of a quenching agent (Carbon Tetrachloride), and plotting efficiency verses quench number (H#). Refer to "Liquid Scintillation Counter Operating Instructions", (GL-EPI-I-004), for instructions on establishing a quench curve.

9.7.2 Each standard used as part of quench calibration for the Rocky Flats project must be counted long enough to acquire 100,000 counts.

9.8 Instrument Performance Requirements

9.8.1 Refer to "Liquid Scintillation Counter Operating Instructions," (GL-EPI-E-I-004), for performance requirements.

9.9 Analysis Procedures and Instrumental Operation

9.9.1 Refer to "Liquid Scintillation Counter Operating Instructions," (GL-EPI-E-I-004), for performance requirements.

10.0 CALCULATIONS AND DATA REDUCTION METHODS

10.1 The instrument will calculate the tritium radioactivity, uncertainty, and MDA in pCi/unit by the following equations:

NOTE: Certain clients may request other equations to be used.

10.1.1 Calculations for aqueous samples:

10.1.1.1 $$\text{Tritium} = \frac{A}{2.22 * C * V}$$

10.1.1.2 $$\text{Tritium Uncertainty} = \frac{1.96\sqrt{(cpm_{bkg}/T_{CB}) + (cpm_s/T_C)}}{2.22 * C * V}$$

10.1.1.3 $$MDA = \frac{2.71 + 4.65\sqrt{cpm_{bkg} * T_c}}{2.22 * C * V * T_c}$$

10.1.2 Calculations for solid samples:

10.1.2.1 $$\text{Tritium} = \frac{A}{2.22 * C * V}$$

10.1.2.2 $$\text{Tritium Uncertainty} = \frac{(1.96\sqrt{(cpm_{bkg}/T_{CB}) + (cpm_s/T_C)}}{2.22 * C * V}$$

10.1.2.3 $$MDA=\frac{2.71+4.65\sqrt{cpmbkg * Tc)}}{2.22 * C * V * Tc}$$

10.1.2.4 $$\%\ Moisture=\frac{Wi - Wf}{Wi} * 100$$

Where:

A=Net beta count rate (gross beta count rate minus the background count rate) at the tritium window

C=Tritium efficiency factor, read from efficiency H#(quench factor).

V=Volume of sample aliquot (mL)

2.22=Conversion factor from dpm/pCi

T_{CB}=Background count time

cpm_{bkg}=Background counts per minute

cpm_S=Observed sample counts per minute in the tritium window

Tc=Sample count time (minutes)

Wi=Weight initial(g)

Wf=Weight final(g)

11.0 DATA RECORDING, REVIEW AND REPORTING

11.1 Data Recording

11.1.1 Record the information required on the Que sheet (Appendix 5).

11.2 Data Review

11.2.1 Refer to "Data Review and Validation Procedures," (GL-EPI-E-D-003), for instructions concerning the data review process.

11.3 Data Reporting

11.3.1 The analyst will take the applicable tritium spreadsheet and enter the data into LIMS. The following information should be included: analyst's initials, date and time the sample was counted, sample results and accuracy in pCi/ml. Refer to "Validation and Verification of Software," (GL-EPI-M-006), for spreadsheet verification.

12.0 QUALITY CONTROL REQUIREMENTS

12.1 Analyst and Method Verification Requirements

12.1.1 Refer to "Analyst and Analytical Methods Validation," (GL-EPI-D-002),

for information concerning analyst and method verification.

12.2 Method Specific Quality Control Requirements

12.2.1 A matrix spike(ms) should be run with every batch of samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\text{Rec} = \frac{\text{Spike (pCi/unit)} - \text{Sample (pCi/unit)}}{\text{Nominal Concentration (pCi/unit}}$$

12.2.2 A duplicate should be run with every batch of samples. If both the sample and duplicate values (pCi/ml) are greater than 5 times the CRDL, the allowable RPD is less than or equal to 20%. If the sample and the duplicate values (pCi/ml) are greater than or equal to the CRDL and less than 5 times the CRDL, the allowable RPD is less than or equal to 100%. The RPD is not applicable if either sample or duplicate values are less than the CRDL. The RPD is calculated as follows:

$$\%\text{RPD} = \frac{\text{High Result} - \text{Low Result}}{\text{Average}} * 100$$

12.2.3 The blank reported value should be less than or equal to the CRDL.

12.2.4 The LCS recovery should fall between 75 and 125%. The recovery is calculated as follows:

$$\text{Rec} = \frac{\text{LCS (pCi/unit)}}{\text{Nominal Concentration (pCi/unit)}}$$

12.3 Actions Required if the Quality Control Requirements Are Not Met

12.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non-conformance report as outlined in "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items," (GL-QS-E-004).

13.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

13.1 Raw data and associated documentation will be stored in binder books or in files and kept on hand for any subsequent reviews necessary.

13.2 Data generated by the instrument will be backed up during routine software

backup.

13.3 Quality control charts are kept on a local database as outlined in "VAX/VMS Quality Control Software Program," (GL-EPI-I-008).

14.0 LABORATORY WASTE HANDLING AND DISPOSAL

14.1 Radioactive material is handled and disposed of as outlined in "Laboratory Waste Disposal and Emergency Instructions," (GL-EPI-S-005).

15.0 REFERENCES

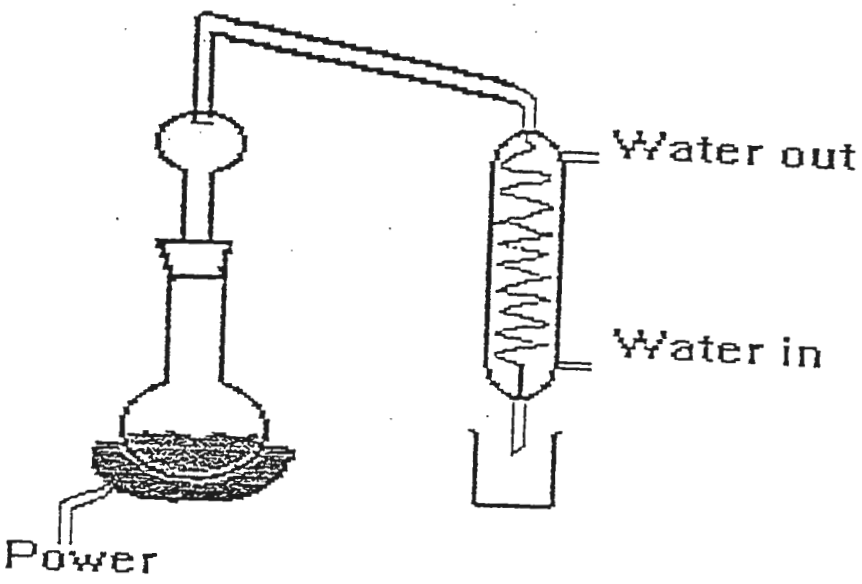
15.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, USEPA, Method 906, Aug. 1980.

15.2 Standard Methods for the Examination of Water and Wastewater, 14th edition, American Public Health Association, Washington, DC, 1976.

APPENDIX 1

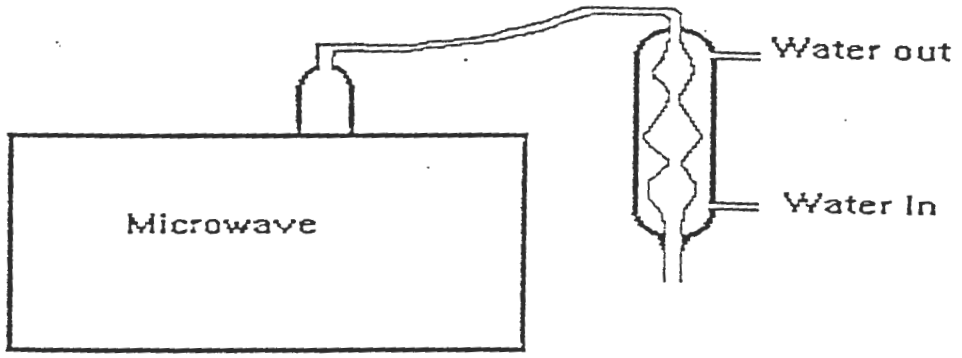
APPENDIX 1

DISTILLATION APPARATUS DIAGRAM



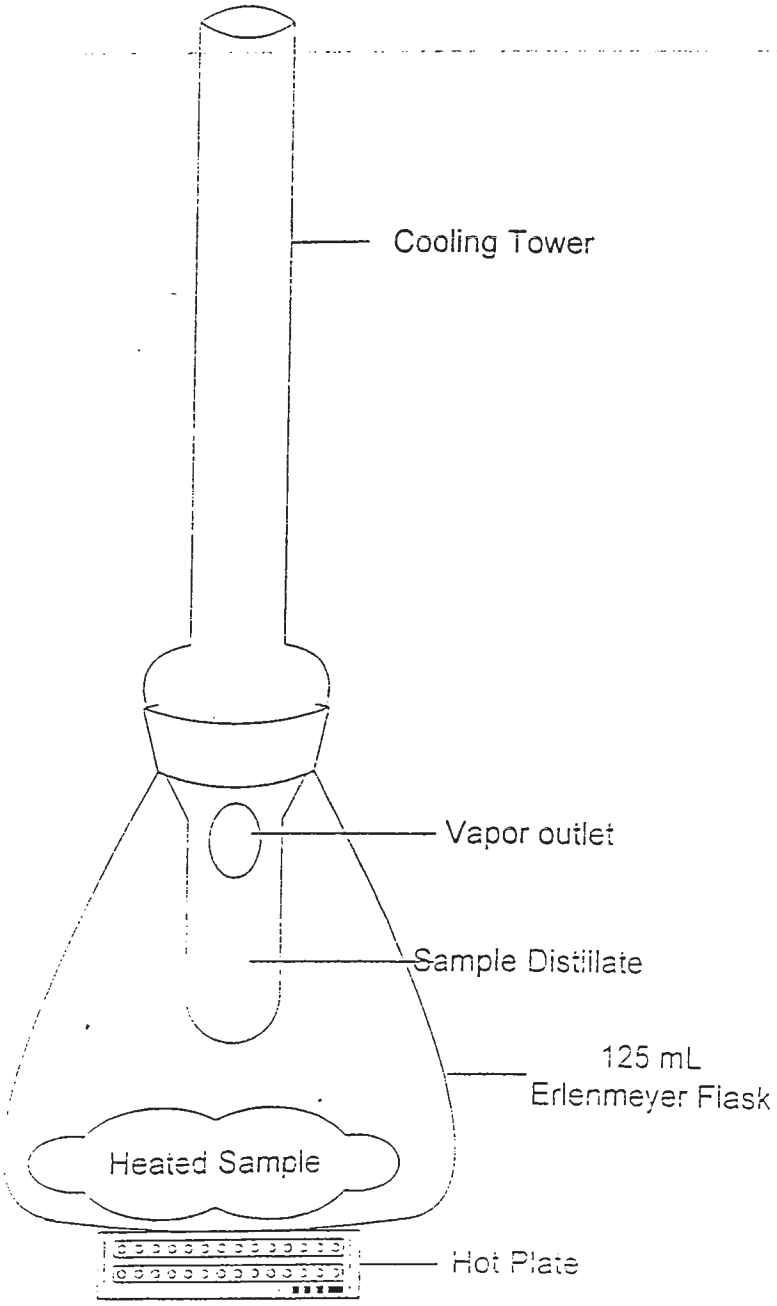
APPENDIX 2

MICROWAVE APPARATUS DIAGRAM



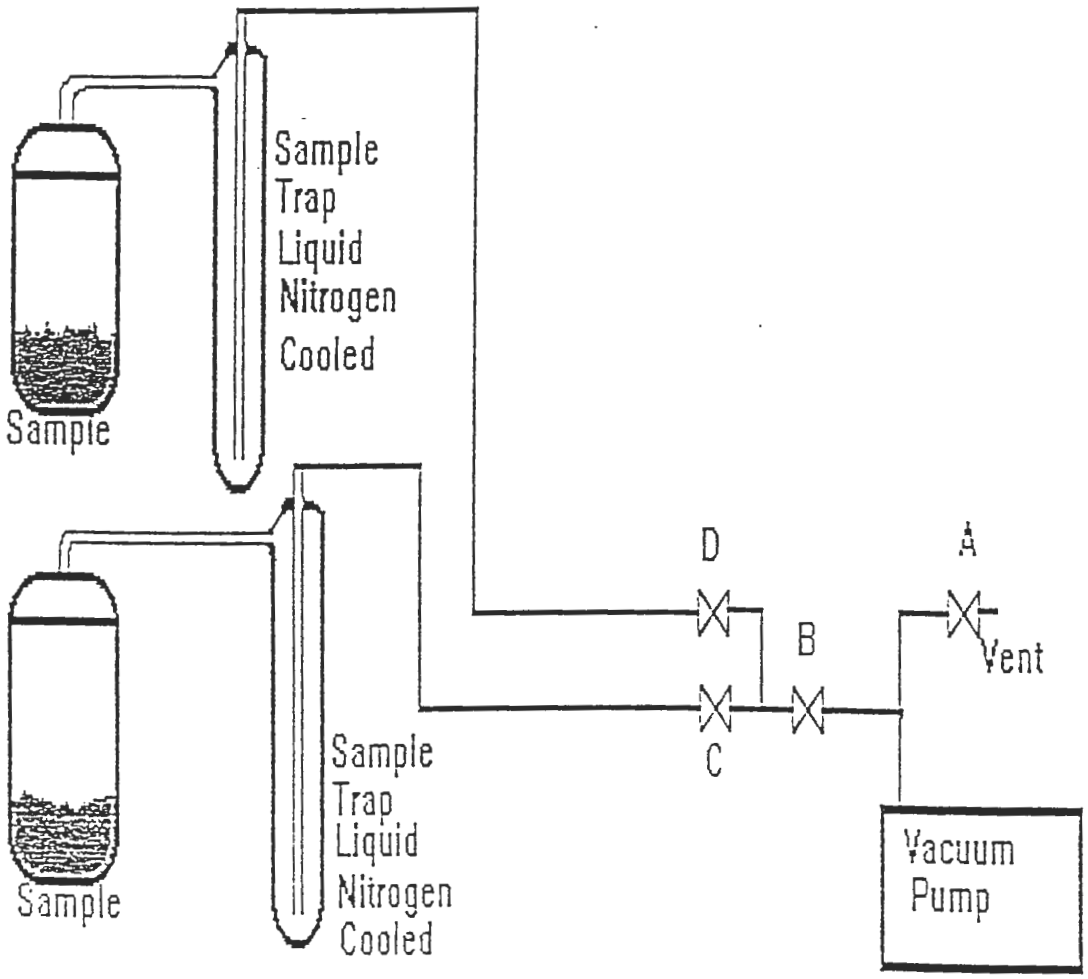
APPENDIX 3

VACUUM RIG DIAGRAM



APPENDIX 4

DISTILLATION APPARATUS II DIAGRAM



APPENDIX 5

TRITIUM QUE SHET

Tritium Que Sheet Batch #: 105053 Analyst: IDXL Automatic Comments:

H-3 Spike Code:_____ Vol:_____ Balance # :_____ Prep Date:_____

H-3 LCS Code:_____ Vol:_____ Pipet # :_____ Initials :_____

Minimum Duedate: 8/12/97

Sample ID	Client Description		Min RDL	Client	Aliquot (mL or g)	LCS Rack #	Dist Rig #	Vol. Added For Dist.	Initial Weight (g)	Final Weight (g)	Dist. Vol. Collected
9707287-12	Description		700 PCL	Client					N/A	N/A	
9707287-13	Description		700 PCL	Client					N/A	N/A	
9707287-14	Description		700 PCL	Client					N/A	N/A	
9707287-15	Description		700 PCL	Client					N/A	N/A	
9707287-16	Description		700 PCL	Client					N/A	N/A	
9707287-18	Description		700 PCL	Client					N/A	N/A	
9707287-19	Description		700 PCL	Client					N/A	N/A	
9707287-20	Description		700 PCL	Client					N/A	N/A	
9707287-21	Description		700 PCL	Client					N/A	N/A	
9707287-22	Description		700 PCL	Client					N/A	N/A	
9707287-23	Description		700 PCL	Client					N/A	N/A	
9707287-24	Description		700 PCL	Client					N/A	N/A	
9707322-01	Description		700 PCL	Client					N/A	N/A	
9707322-02	Description		700 PCL	Client					N/A	N/A	
9707322-03	Description		700 PCL	Client					N/A	N/A	
9707322-04	Description		700 PCL	Client					N/A	N/A	
9707322-05	Description		700 PCL	Client					N/A	N/A	
9707322-06	Description		700 PCL	Client					N/A	N/A	
9707322-07	Description		700 PCL	Client					N/A	N/A	
9707322-08	Description		700 PCL	Client					N/A	N/A	
QC443729	BLANK	BLANK	0.7 pCi/mL	QC					N/A	N/A	
QC443730	9707322-08	DUP	0.7 pCi/mL	QC					N/A	N/A	
QC443731	9707322-08	MS	0.7 pCi/mL	QC					N/A	N/A	
QC443732	LCS	LCS	0.7 pCi/mL	QC					N/A	N/A	

Instruments Used (circle as appropriate):

LS6000 (Red) 7065155 WALLAC (Yellow) 4040127

LS6500 (Blue) 7067083

LS6500 (Green) 7067404

Data Reviewed By:_____

Page 1 of 1

STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF CARBON-14 IN WATER

(GL-EPI-E-A-003 Revision 2)

UNCONTROLLED DOCUMENT

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Process Owner	5/30/99 (Sign and Date)	Tim C. Winters (Print Name)
Technical Review	9/3/99 (Sign and Date)	Richard M. Kinney (Print Name)
Quality Review	9/7/99 (Sign and Date)	Gene G. Sat (Print Name)
Approval and Authorization	9/7/99 (Sign and Date)	James B Winstead (Print Name)

SET ID

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF CARBON-14 IN WATER

2.0 METHOD OBJECTIVE AND APPLICABILITY

A sample is digested in a flask. Inorganic carbon-14 is released as carbon dioxide by the acid reaction. Potassium persulfate with acid in the presence of a silver nitrate catalyst oxidizes organic carbon to carbonate that is distilled and trapped into an ethanolamine/ methanol solution. The extracted carbon-14 is transferred to a scintillation vial and counted in a liquid scintillation counter.

3.0 INTERFERENCES

Tritium can interfere with the determination of carbon-14. To eliminate the interference, a 0.4 M HCl trap is used to remove tritium prior to the final carbon-14 trapping in ethanolamine solution.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 Caution should be used when dealing with acids, ethanolamine, and any other laboratory reagents. Safety procedures for these are set forth in the "Handbook for Good Laboratory Practices," found in the library of the laboratory.

4.2 If there is any question regarding the safety of any laboratory practice, stop immediately and consult the group leader prior to carrying out the rest of the procedure.

5.0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment

5.1.1 Carbon-14 apparatus

5.1.2 Calibrated pipettes

5.1.3 Heating mantels

5.1.4 Glass wool

5.1.5 Calibrated balance

5.2 Reagents. Chemicals and Standards

5.2.1 Ascarite

5.2.2 Absolute methanol

5.2.3 Scintillation cocktail (suggested Ready Gel™ cocktail)

5.2.4 Nitric acid, concentrated

5.2.5 Potassium persulfate

5.2.6 1 M silver nitrate. 169.89 grams to 1,000 mL of DI water.

5.2.7 0.4 M hydrochloric acid. Add 33 mL of concentrated acid to 500 mL of DI water and bring up the volume to 1,000 mL.

5.2.8 Carbon-14 standard solution traceable to NIST

5.2.9 Ethanolamine reagent

5.2.10 1 M sodium carbonate. 105.99 g to 100 mL DI water.

5.2.11 Carbon tetrachloride

5.3 Instrumentation

5.3.1 Liquid scintillation counting system

6.0 SAMPLE COLLECTION AND PRESERVATION

Samples are collected unpreserved in a suitable container.

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to GL-EPI-E-I004 for instructions concerning the scintillation counter.

7.2 Refer to GL-EPI-E-I010 "Counting Room Instrument Maintenance and Performance Checks" for instructions concerning instrument maintenance.

8.0 PREPARATION OF STANDARD SOLUTION AND QUALITY CONTROL SAMPLES

8.1 Refer to GL-EPI-M001 "Preparation of Radioactive Standards" for instructions concerning the preparation of standard solutions.

8.2 A blank should be performed with the batch. A DI water blank should be used.

8.3 A duplicate sample should be run for each sample batch.

8.4 A matrix spike sample is prepared by adding a volume of standard directly to the sample. The volume and standard ID are recorded on the que sheet (Appendix 1).

8.5 A laboratory control sample is prepared by adding a volume of standard directly to DI water. The volume and standard ID are recorded on the que sheet (Appendix 1).

9.0 OPERATING PROCEDURE

9.1 Sample Preparation Techniques

9.1.1 Measure out an appropriate aliquot of sample and transfer it to a two-neck flask. Generally, 250 mls is a sufficient sample volume. Record the volume of sample on the que sheet (Appendix 1).

9.1.2 Slide a plug of glass wool into the bottom portion of the addition tube. Fill up to the middle with Ascarite. Slide another plug of glass wool into the tube. Fill up to the top portion with drierite and pack with another piece of glass wool.

9.1.3 Add 4 mL of ethanolamine and 1 mL of methanol to the C-14 trap. Add 50 mL of 0.4 M of HCl acid to the H-3 trap. Connect the traps to the respective absorber jackets (see Figure 1).

9.1.4 Add the carbon carrier 1 mL of the two-neck flask.

9.1.5 Start vacuum adjusting to 100 cc/min.

9.1.6 Add 5.0 g of potassium persulfate, 5 mL of silver nitrate and 20 mL of concentrated nitric acid.

9.1.7 Attach the addition tube to the flask immediately and turn on the heat and the water line for cooling the condenser.

9.1.8 Heat the flask for approximately 90 minutes. Turn the heat off after the required time and let the flask cool for about 30 minutes. The vacuum and the water should remain on.

9.1.9 After shutting off the vacuum and the water, disconnect the absorber jacket (see Figure 1) from the H-3 trap and disconnect the absorber jacket of the C-14 trap (next to the H-3 trap). Let the solution from the absorber jacket tube drain out completely into the trap. Disconnect the absorber jacket of the C-14 jacket.

9.1.10 Transfer the ethanolamine-methanol solution from the C-14 trap to a glass scintillation vial. Rinse the trap with 5 mL of methanol and transfer the rinse to the vial.

9.1.11 Add 10 mL of scintillation cocktail to each vial. Cap the vial and shake to mix well. The solution should now be clear.

9.1.12 Wipe the exterior of the vial with a Kim wipe dampened with methanol, then wipe dry with a lint-free paper.

9.1.13 Place the vial in the scintillation counter to adapt to darkness for at least 1-2 hours before counting. Record the rack and position number on the sample que sheet (Appendix 1).

9.1.14 Count the sample in the scintillation system. The activity is determined by the comparison of the sample's quench number to a previously counted standard quench curve. If the sample quench number is below the standard quench curve limits, add carbon tetrachloride. If the sample is above the quench limits, then the sample must be diluted or extracted again and recounted.

9.2 Instrument Calibration

9.2.1 Establishing a quench curve

9.2.1.1 Prepare a set of standards consisting of 8-12 standards using the same matrix and cocktail as the samples to be measured. Add approximately 10,000 DPM to each standard, cap and shake.

NOTE: Do not add any quenching agent at this point.

9.2.1.2 Allow the standards to dark adapt for a minimum of 1 hour.

9.2.1.3 Using an available counting program, measure the observed CPM of each of the standards to verify accurate pipetting. All standards should agree within +/- 5% of the mean. Discard any standards that do not meet this criteria.

9.2.1.4 Add 0-220 ul of carbon tetrachloride in 20 ul increments to each of the standards.

9.2.1.5 Recount the standards for a period of 1 minute to determine if the range of H#'s covers the desired range of sample quench. If the range of the standards is not large enough, create more standards, adjusting the amount of quenching agent appropriately. Record

the amount of quenching agent used for each standard on the standard preparation sheet (Appendix 2).

9.2.1.6 Count the standards for a sufficient length of time to accumulate at least 10,000 counts in the highest quenched standard. Plot efficiency vs. H#.

9.2.2 Calibration verification

9.2.2.1 Count the standards against the newly established curves. The acceptance criteria being 100% +/- 10% accuracy measured against a known value.

9.3 Instrument Performance Requirements

9.3.1 Refer to GL-EPI-E-I004 for instructions concerning the scintillation counter.

9.3.2 Refer to GL-EPI-E-I010 "Counting Room Instrument Maintenance and Performance Checks" for instructions concerning instrument maintenance.

9.4 Analysis Procedures and Instrumental Operation

9.4.1 Refer to the instrument procedure GL-EPI-E-I004 for operation of the liquid scintillation counting system.

10.0 CALCULATIONS

10.1 Calculate the activity in pCi/L by the following equation:

$$C-14=\frac{(S-B)}{(2.22)(E)(v)}$$

10.2 Calculate the uncertainty in pCi/L by the following equation:

$$C-14_{UNCERTAINTY}=\frac{(1.96)\sqrt{\frac{B}{CT_b}+\frac{S}{CT_s}}}{(2.22)(E)(v)}$$

Where:
S = gross beta count rate per minute (cpm)
B = background cpm
E = counting efficiency
v = volume of sample taken for analysis

2.22 = conversion factor from dpm/pCi
CT_b = count time background
CT_s = count time sample

10.3 The method MDA is calculated according to the following equation:

$$MDA = \frac{2.71 + 4.66 * \sqrt{B_{cpm} * C}}{2.22 * E * V * C}$$

Where:
S_{cpm} = sample counts per minute
B_{cpm} = background counts per minute
E = counting efficiency
C = sample count time
V = volume of sample used for analysis

11.0 DATA RECORDING, REVIEW AND REPORTING

11.1 Data Recording

11.1.1 The data printed out from the liquid scintillation counter should be entered onto the master spreadsheet for carbon-14.

11.1.2 Once the batch is complete, the values from the spreadsheet are entered into the LIMS.

11.2 Data Review and Validation

11.2.1 Refer to GL-EPI-E-D003 "Data Review and Validation Procedures" for instructions concerning the data review process.

11.3 Data Reporting

11.3.1 The analyst will take the applicable spreadsheet and enter the data into the LIMS. The following information should be included: Analyst's initials, run date and time of the sample, results in pCi/L, the accuracy in pCi/L and the nominal concentration of spike and LCS.

12.0 QUALITY CONTROL REQUIREMENTS

12.1 Analyst and Method Verification Requirements

12.1.1 Refer to GL-EPI-E-D003 "Analyst and Analytical Methods Validation Procedures" for instructions concerning the validation of analysts and analytical methods.

12.2 Method-Specific Quality Control Requirements

12.2.1 A method blank should accompany each batch of samples. The reported value should be less than or equal to the contract-required detection limit (CRDL).

12.2.2 A matrix spike (ms) should be run with every batch of 20 or fewer samples. The recovery of the spikes should fall between 75% and 125%. The recovery is calculated as follows:

$$MS(\%) = \frac{Spike\,(pCi/L) - Sample\,(pCi/L)}{Spike\ Concentration\,(pCi/L)} * 100$$

12.2.3 A duplicate sample should be run with every batch of 20 or fewer samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20% if both results are greater than 5 * CRDL. If either result is between 5 * CRDL and CRDL, then the limit on RPD is equal to or less than 100%. If either result is less than CRDL, then the limits on RPD are not applicable. The RPD is calculated as follows:

$$RPD(\%) = \frac{High\ Dup\,(pCi/L) - Low\ Dup\,(pCi/L)}{Average\,(pCi/L)} * 100$$

12.2.4 A Laboratory Control Sample (LCS) should be run with every batch of 20 or fewer samples. The recovery of the LCS should fall between 75% and 125%. The recovery is calculated as follows:

$$LCS(\%) = \frac{Observed\ LCS\,(pCi/L)}{LCS\ \ Known\ Concentration\,(pCi/L)} * 100$$

12.3 Actions Required if the Quality Control Requirements Are Not Met

12.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a nonconformance report as outlined in GEL

SOP GL-QS-E-004 "Nonconformance Identification Control. Documentation. Reporting and Dispositioning."

13.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

13.1 Raw data and associated documentation will be stored in binder books or in files and kept on hand for any subsequent reviews necessary.

13.2 Data generated by the liquid scintillation counting system will be backed up during routine software backup. See procedure for magnetic backup and storage instructions GL-EPI-E-M003.

13.3 Quality control charts for spike, blank and relative percent difference results are kept on a local database.

14.0 LABORATORY WASTE HANDLING AND DISPOSAL

14.1 The ethanolamine waste is segregated for disposal as an organic waste.

14.2 Radioactive material is handled and disposed as outlined in GL-EPI-E-S005 "Radioactive Waste Handling Procedures."

15.0 REFERENCES

15.1 Carbon-14 Radionuclide Separation and Analysis, Peter Lindahl, Chemical Technology Div., Argonne National Lab.

Appendix 1

C-14 Que Sheet

Batch #: 144030 **Analyst:** Kim Allen **Comments:** blowndown tank #

C-14 Spike Code: _____ **Vol:** _____ **Balance #:** _____ **Prep Date:** _____

C-14 LCS Code: _____ **Vol:** _____ **Pipet #:** _____ **Initials:** _____

Minimum Due Date: 3/23/99

Sample ID	Client Description		Min RDL	Client	Sample #	Aliquot (mL or g)	LCS Rack #	C-14 Rig #
9903104-02	99-CIF-0403		200 PCL	WSRC				
QC591702	blank	BLANK	200 pCi/L	QC				
QC591703	9903104-02	DUP	200 pCi/L	QC				
QC591704	9903104-02	MS	200 pCi/L	QC				
QC591705	lcs	LCS	200 pCi/L	QC				

Instruments Used (circle as appropriate):

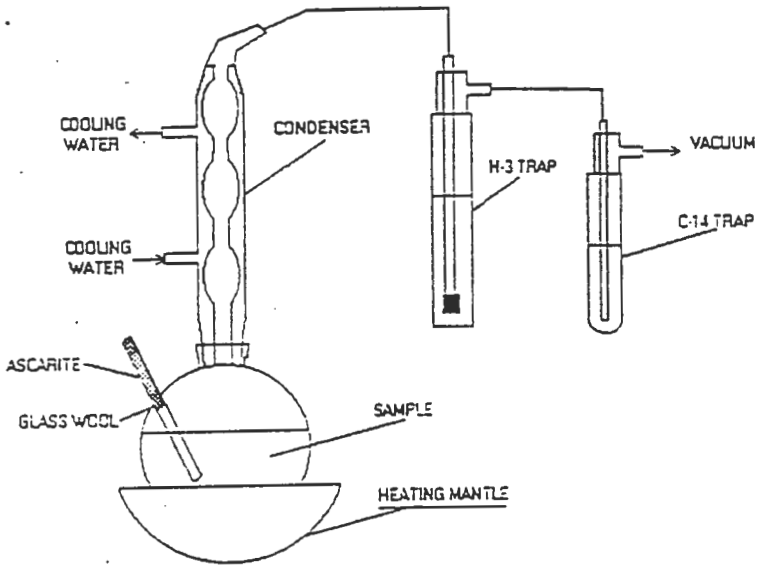
LS6000 (Red) 7065155 **LS6500 (Blue) 7067083** **LS5801 (Orange) 7012880** **Data Reviewed by:** _____

LS6500 (Green) 7067404 **LS6500 (Black) 7069123** **WALLAC (Yellow) 4040127**

Page 1 of 1

FIGURE 1

CARBON-14 DISTILLATION APPARATUS



Appendix 2

General Engineering Laboratories
Calibration Source Preparation Sheet

Applicable SOP Number _____ Isotope _____

Date Standards Prepared _____ Cocktail Type Used _____

Standard ID _____ Matrix of Vial/Planchett _____

Amount Used (g or ml) _____

Standard Activity (DPM/g or mL) _____ Type of Scintillation Vial _____

Reference Date _____ Pipette ID Used _____

Expiration Date _____ Balance ID Used _____

Residue/Carrier Agent _____ Quenching Agent _____

EPI Samplenumber	Standard Number	Quenching Vol (uL)/ Residue Volume(mL)	Initial Wt. (g)	Final Wt. (g)	Net Wt. (mg)

Prepared By: _____ Date _____

Reviewed By: _____ Date _____

STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF CARBON-14 IN SOIL, VEGETATION AND OTHER SOLID MATRICES

(GL-EPI-A-003B Revision 3) UNCONTROLLED DOCUMENT

PROPRIETARY INFORMATION

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

Process Owner	3/30/99 (Sign and Date)	Tim C. Winters (Print Name)
Technical Review	9/3/99 (Sign and Date)	Richard M. Kinney (Print Name)
Quality Review	9/7/99 (Sign and Date)	Gene C. Post (Print Name)
Approval and Authorization (Sign and Date)	9/7/99	James B. Westmoreland (Print Name)

SET I.D.

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310

TABLE OF CONTENTS

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1.0 THE DETERMINATION OF CARBON-14 IN SOIL, VEGETATION AND OTHER SOLID MATRICES

2.0 METHOD OBJECTIVE AND APPLICABILITY

2.1 A soil sample is digested in a flask. Inorganic carbon-14 is released as carbon dioxide by the acid reaction. Potassium persulate with acid in the presence of a silver nitrate catalyst oxidizes organic carbon to carbonate that is distilled and trapped into an ethanolamine/methanol solution. The extracted carbon-14 is transferred to a scintillation vial and counted in a liquid scintillation counter.

3.0 INTERFERENCES

3.1 Tritium can interfere with the determination of carbon 14. To eliminate the interference, a 0.4 M HCl trap is used to remove tritium prior to the final carbon-14 trapping in ethanolamine solution.

3.2 There are some limitations of importance to the analyst. Quantitative recovery of the CO_2 produced in the sample decomposition is limited by the capacity of the ethanolamine trap. The trap capacity is 2.7 grams of CO_2 when using 4 mL of ethanolamine trap. However, as CO_2 reacts with ethanolamine, the methanol-ethanolamine mixture can become immiscible with the LS cocktail. When this happens, the sample and cocktail form two layers, rendering the mixture unsuitable for LS counting. Immiscibility starts to occur when more than one gram of CO_2 has been collected in the trap. Therefore, although the trap can accept more CO_2, the LS cocktail cannot, and the sample size must be limited to that which will produce less than one gram of CO_2. The addition of more methanol to the LSC vial to increase miscibility is not acceptable if the vial is filled above the shoulder. When this occurs, part of the scintillation process occurs in the upper part of the vial, which is unobserved by the detector. Thus, the sample result will be biased low.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 Caution should be used when dealing with acids, ethanolamine, and any other laboratory reagents. Safety procedures for these are set forth in the Handbook for Good Laboratory Practices, found in the library of the laboratory.

4.2 If there is any question regarding the safety of any laboratory practice, **stop immediately** and consult the Group Leader prior to carrying out the rest of the procedure.

0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment

5.1.1 Carbon-14 apparatus

5.1.2 Calibrated pipettes

5.1.3 Heating mantels

5.1.4 Glass wool

5.1.5 Calibrated balance

5.1.6 Disposable volumetric pipettes

5.2 Reagents, Chemicals and Standards

5.2.1 Ascarite

5.2.2 Absolute methanol

5.2.3 Scintillation cocktail (suggested Ready GEL^{tm} cocktail)

5.2.4 Nitric acid concentrated

5.2.5 Potassium persulfate

5.2.6 1 M silver nitrate, 169.89 grams to 1000 ml of DI water

5.2.7 0.4 M hydrochloric acid. Add 33 ml of concentrated acid to 500 ml of DI water and bring up the volume to 1000 ml.

5.2.8 Carbon-14 standard solution

5.2.9 Ethanolamine

5.3 Instrumentation

5.3.1 Liquid Scintillation counting system

6.0 Sample Collection & Preservation

6.1 Samples are collected unpreserved in a suitable container.

7.0 Equipment and Instrument Maintenance

7.1 Refer to GL-EPI-I-004, "Operation of the Beckman LS 6000 Series Scintillation Counter," for instructions concerning the scintillation counter.

7.2 Refer to GL-EPI-I-010, "Counting Room Instrument Maintenance and Performance Checks," for instructions concerning instrument maintenance.

8.0 Preparation of Standard Solution and Quality Control Samples

8.1 Refer to GL-EPI-M-001, "Preparation of Radioactive Standards," for instructions concerning the preparation of standard solutions.

8.2 A blank should be performed with the batch. DI water is used as the blankl.

8.3 A duplicate sample should be run for each sample batch.

8.4 A matrix spike sample is prepared by adding a volume of standard directly to the sample soil distillation slurry. The volume and standard ID are recorded on the que sheet (Appendix 1).

8.5 A laboratory control sample is prepared by adding a volume of standard directly to DI water. The volume and standard ID are recorded on the que sheet (Appendix 1).

9.0 OPERATING PROCEDURE

9.1 Sample Preparation Techniques

9.1.1 Weigh out an appropriate amount of sample and transfer it to a two neck flask. Record the weight of sample on the que sheet (Appendix 1). Add about 100 ml of DI water and connect to the condenser.

9.1.2 Slide a plug of glass wool into the bottom portion of the addition tube. Fill up to the middle Ascarite. Fill up to the top portion with drierite and pack with another piece of glass wool.

9.1.3 Add 4 ml of ethanolamine and 1 ml of methanol to the C-14 trap. Add 50 ml of 0.4 M of HCl acid to the H-3 trap. Connect the traps to the respective absorber jackets (see Figure 1).

9.1.4 Start vacuum adjusting to 100 cc/min.

9.1.5 Add a slurry of 5.0 grams of potassium persulfate, 2 ml of silver nitrate and 25 ml of concentrated nitric acid by temporarily removing the addition tube.

9.1.6 Attach the addition tube to the flask immediately and turn on the heat and the water line for cooling the condenser.

9.1.7 Heat the flask for approximately 90 minutes. Turn the heat off after the required time and let the flask cool for about 30 minutes. The vacuum and the water should remain on.

9.1.8 Disconnect the absorber jacket (see Figure 1) from the H-3 trap and disconnect the absorber jacket of the C-14 trap (next to the H-3 trap). Let the solution from the absorber jacket tube drain out completely into the trap. Disconnect the absorber jacket of the C-14 jacket.

9.1.9 Transfer the ethanolamine-methanol solution from the C-14 trap to a glass scintillation vial. Rinse the trap with 3 ml of methanol and transfer the rinse to the vial.

9.1.10 Add 10 ml of scintillation cocktail gel to each vial. Cap the vial and shake to mix well (a milky white solution may be formed). Uncap the vial and add another 1 ml of methanol. Cap the vial and shake well. The solution should now be clear. (See section 3.2.)

9.1.11 Wipe the exterior of the vial with a Kimwipe.

9.1.12 Place the vial in the scintillation counter to adapt to darkness for at least 2 hours before counting. Record the rack and position number on the sample que sheet (Appendix 1).

9.1.13 Count the sample in the scintillation system. The activity is determined by the comparison of the samples quench number to a previously counted standard quench curve that has been stored in system memory. If the sample quench number is either above or below the standard quench curve limits, the sample must be diluted or extracted again and recounted.

9.2 Sample Preparation Techniques: Oily matrices

9.2.1 The carbon dioxide traps currently used (4 mL of ethanolamine and 2 mL methanol) will handle a sample that contains up to 0.27 grams of carbon. If it is known, for example, that a sample is 10% carbon, a sample up to 2.7 grams could be used. Since the carbon content will usually never be known, the sample size should be limited to 0.5 grams unless that poses a problem for mda requirements. (See section 3.2.)

9.2.2 Place the sample into the reaction flask and connect the apparatus in the normal fashion, temporarily omitting the Ascarite tube.

9.2.3 Add 10 mL of sulfuric acid to the flask, immediately insert the Ascarite tube and establish air flow.

9.2.4 It can now begin heating. It is difficult to predict how the sample will react. The desired result is the formation of a black amorphous residue without copious evolution of white fumes of sulfuric acid. With such a small amount of liquid present, it is easy to overheat the flask. Once the acid is hot, the charring should only require a few minutes.

9.2.5 When the charring is complete, shut off heat and remove the heating mantle to speed up cooling. DO NOT DISCONNECT THE ASCARITE

TUBE OR INTERRUPT FLOW THROUGH THE CARBON DIOXIDE TRAP UNTIL THE FLASK IS COOL.

9.2.6 When the flask is cool to the touch, stop flow through the system and remove the Ascarite tube. Now add all the reagents normally used for the C-14 analysis, except $K_2S_2O_8$ to 25 g. Replace the Ascarite tube and continue with the digestion process as usual.

9.3 Sample Preparation Techniques: Vegetation matrices

9.3.1 For vegetation samples use 0.5 g (or less) based on dry weight. Add 100 mL of DI water to the digestion flask.

9.3.2 Add 3 mL ethanolamine and 3 mL of methanol to the C-14 trap.

9.3.3 Add 10 g potassium persulfate, 5 mL silver nitrate and 100 mL concentrated nitric acid to the digestion flask.

9.4 Instrument Calibration

9.4.1 Establishing a quench curve

9.4.1.1 Prepare a set of standards consisting of 8 to 12 standards using the same matrix and cocktail as the samples to be measured. Add approximately 10,000 dpm to each standard, cap and shake.

NOTE: Do not add any quenching agent at this point.

9.4.1.2 Allow the standards to dark adapt for a minimum of 1 hour.

9.4.1.3 Using an available counting program measure the observed CPM of each of the standards to verify accurate pipetting. All standards should agree within ± 5% of the mean. Discard any standards that do not meet this criteria.

9.4.1.4 Add 0 to 220 ul of carbon tetrachloride in 20 ul increments to each of the standards.

9.4.1.5 Recount the standards for a period of 1 minute to determine if the range of H#s covers the desired range of sample quench. If the range of the standards is not large enough, create more standards, adjusting the amount of quenching agent appropriately. Record the amount of quenching agent used for each standard on the standard preparation (Appendix 2).

9.4.1.6 Count the standards for a sufficient length of time to accumulate at least 10.000 counts in the highest quenched standard. Plot efficiency vs. H#.

9.4.2 Calibration verification

9.4.2.1 Count the standards against the newly established curves. The acceptance criteria being 100% ± 10% accuracy measured against a known value.

9.5 Instrument Performance Requirements

9.5.1 Refer to GL-EPI-I-004, "Operation of the Beckman LS 6000 Series Scintillation Counter," for instructions concerning the scintillation counter.

9.5.2 Refer to GL-EPI-I-010, "Counting Room Instrument Maintenance and Performance Checks." for instructions concerning instrument maintenance.

9.6 Analysis Procedures and Instrumental Operation

9.6.1 Refer to the instrument procedure GL-EPI-SOP I-004 for operation of the liquid scintillation counting system .

10.0 CALCULATIONS

10.1 Calculate the activity in pCi/g by the following equation:

$$C\text{-}14 = \frac{(S - B)}{(2.22)(E)(g)}$$

10.2 Calculate the uncertainty in pCi/g by the following equation:

$$C\text{-}14_{UNCERTAINTY} = \frac{(1.96)\sqrt{\frac{B}{CTb} + \frac{S}{CTs}}}{(2.22)(E)(g)}$$

Where:
S = gross beta count rate per minute
B = background cpm
E = counting efficiency
g = grams of sample taken for analysis
2.22 = conversion factor from dpm/pCi

CT_b= Count time background
CT_S= Count time sample

10.3 The method MDA is calculated according to the following equation:

$$MDA = \frac{2.71 + 4.66 * \sqrt{Bcpm * C}}{2.22 * E * g * C}$$

where:
S_{cpm} = Sample counts per minute
B_{cpm} = Background counts per minute
E = Counting Efficiency
C = Sample count time
g = grams of sample used for analysis

11.0 DATA RECORDING, REVIEW AND REPORTING

11.1 Data Recording

11.1.1 The data printed out from the liquid scintillation counter should be entered onto the master spreadsheet for carbon 14.

11.1.2 Once the batch is complete, the values from the spreadsheet are entered into LIMS.

11.2 Data Review and Validation

11.2.1 Refer to GL-EPI-D-003, "Data Review and Validation Procedures," for instructions concerning the data review process.

11.3 Data Reporting

11.3.1 The analyst will take the applicable spreadsheet and enter the data into LIMS. The following information should be included: Analyst initials, run date and time of the sample, results in pCi/g, the accuracy in pCi/g and the nominal concentration of spike and LCS.

12.0 QUALITY CONTROL REQUIREMENTS

12.1 Analyst and Method Verification Requirements

12.1 Refer to GL-EPI-D-003, "Analyst and Analytical Methods Validation Procedures," for instructions concerning the validation of analysts and analytical methods.

12.2 Method Specific Quality Control Requirements

12.2.1 A method blank should accompany each batch of samples. The reported value should be less than or equal to the contract required detection limit (CRDL).

12.2.2 A matrix spike(ms) should be run with every batch of 20 or less samples. The recovery of the spikes should fall between 75 and 125%. The recovery is calculated as follows.

$$MS(\%) = \frac{SPIKE(pCi/g) - SAMPLE(pCi/g)}{SPIKE\ CONCENTRATION(pCi/g)} * 100\%$$

12.2.3 A duplicate sample should be run with every batch of 20 or less samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20%. The RPD is calculated as follows:

$$RPD(\%) = \frac{HIGH\ DUP(pCi/g) - LOW\ DUP(pCi/g)}{AVERAGE(pCi/g)} * 100\%$$

12.2.4 A Laboratory Control Sample (LCS) should be run with every batch of 20 or less samples. The recovery of the LCS should fall between 75 and 125%. The recovery is calculated as follows.

$$LCS(\%) = \frac{Observed\ LCS(pCi/g)}{LCS\ Known\ CONCENTRATION(pCi/g)} * 100\%$$

12.3 Actions required if the Quality Control Requirements are not met

12.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the Group Leader and initiate a non-conformance report as outlined in GL-QS-E-004, "Nonconformance Identification Control, Documentation, Reporting and Dispositioning."

13.0 Records Management and Document Control

13.1 Raw data and associated documentation will be stored in binder books or in files and kept on hand for any subsequent reviews necessary.

13.2 Data generated by the liquid scintillation counting system will be backed up during routine software backup. (See GL-EPI-M-004 for the procedure for magnetic backup and storage)

13.3 Quality control charts for spike, blank and relative percent difference results are kept on a local database.

14.0 LABORATORY WASTE HANDLING AND DISPOSAL

14.1 The ethanolamine waste is segregated for disposal as an organic waste.

14.2) Radioactive material is handled and disposed as outlined in GL-EPI-S-011, "Laboratory Waste Disposal and Emergency Instructions."

15.0 REFERENCES

15.1 Carbon - 14 Radionuclide Separation and Analysis. Peter Lindahl, Chemical Technology Division, Argonne National Lab.

APPENDIX 1

C-14 Que Sheet

Batch#: 144030 **Analyst: Kim Allen** **Comments** Blowdown tank #

C-14 Spike Code:_____ Vol:_____ Balance #:_____ Prep Date:_____
C-14 LCS Code:_____ Vol:_____ Pipet #:_____ Initials:_____
Minimum Due Date: 8/05/99

Sample ID	Client Desc.		Min RDL	Client	Sample#	Aliquot (mL or g)	LCS Rack#	C-14 Rig#
9903104-02	99-CIF-0403		200 PCL	WSRC				
QC591702	Blank	BLANK	200 pCi/L	QC				
QC591703	9903104-02	DUP	200 pCi/L	QC				
QC591704	9903104-02	MS	200 pCi/L	QC				
QC591705	lcs	LCS	200 pCi/L	QC				

Instrument Used (circle as appropriate) Data Reviewed By:_____
LS6000 (Red) 7065155 LS6500 (Blue) 7067083 LS5801 (Orange) 7022880
LS6500 (Green) 7067404 LS6500 (Black)7069123 WALLAC (Yellow) 4040127

APPENDIX 2

General Engineering Laboratories
Calibration Source Preparation Sheet

Applicable SOP Number _____ Isotope _____

Date Standards Prepared _____ Cocktail Type Used _____

Standard ID _____ Matrix of Vial/Planchett _____

Amount Used (g or ml) _____

Standard Activity (DPM/g or mL) _____ Type of Scintillation Vial _____

Reference Date _____ Pipette ID Used _____

Expiration Date _____ Balance ID Used _____

Residue/Carrier Agent _____ Quenching Agent _____

EPI Samplenumber	Standard Number	Quenching Vol (uL)/ Residue Volume(mL)	Initial Wt. (g)	Final Wt. (g)	Net Wt. (mg)

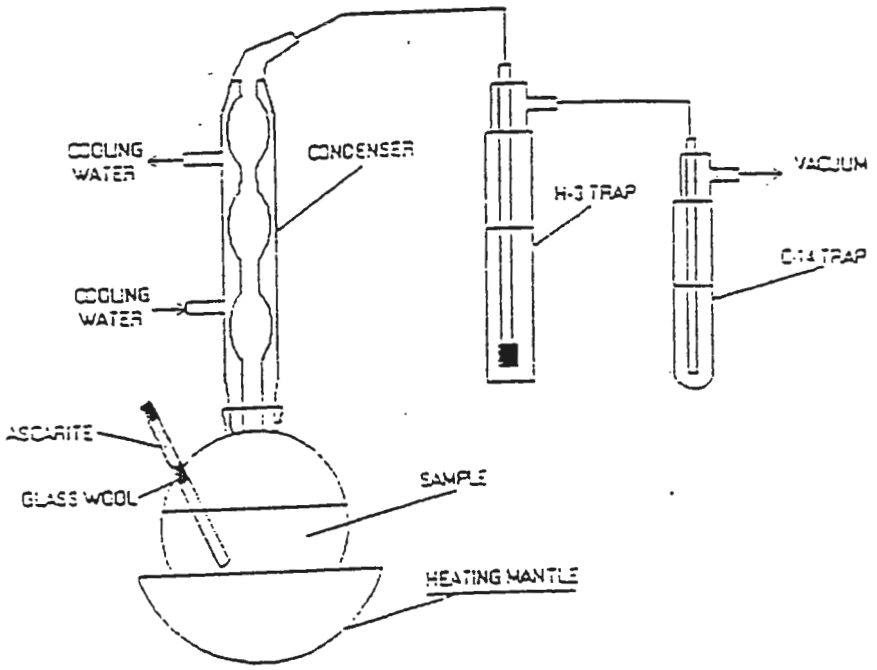
Prepared By: _____ Date _____

Reviewed By: _____ Date _____

Rev 1 RLM 9/10/97

Figure 1

CARBON-14 DISTILLATION APPARATUS



UNCONTROLLED DOCUMENT

STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF STRONTIUM 89/90

IN WATER, SOIL, MILK, MEATS, FISH,

VEGETATION, AND OTHER TISSUES

Process Owner:	Aadli Abdul-Kareem. EPI Laboratory Technician
Technical Review by:	John Dornisch. EPI Laboratory Technician
Quality Review by:	Genie G. Bost. EPI Group Leader. Quality
Approved & Authorized by:	James B. Westmoreland. EPI Laboratory Director

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF STRONTIUM 89/90 IN WATER, SOIL, MILK, MEATS, FISH, VEGETATION, AND OTHER TISSUES

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic Sr-89 and Sr-90. Environmental Physics, Inc. (EPI) utilizes methods that are derived from established sources. This method is based on the source procedures manual EPA 600 4-80-032 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water" August 1980 Method 905.0 and uses similar principles of radiochemical concentration and counting. This method is also very similar in concept to the source method from the DOE Methods Manual for Evaluating Environmental and Waste Management samples 1997 Edition, RP501 Revision 1: "Determination of Total Radioactive Strontium in High Level Samples using Extraction Chromotography".

2.2 This procedure is applicable for the following matrices: water, soil, meats, fish, vegetation, and other tissues.

2.3 Milk fats tend to coat resin beads and clog ion exchange columns, so a batch process is used. Stable strontium is added to equilibrate the radiostrontium with the stable Sr carrier. Sr is stripped from the resin with 8M HNO3, converted to carbonate to concentrate the Sr, and separated from calcium and other elements and isotopes by Sr Resin.

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit (MDL): Typical minimum detectable activity (MDA) for samples analyzed for Sr-89 and Sr-90 is 1pCi/L or 1pCi/g.

3.2 Method Precision: Typical Relative Percent Difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ± 25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample as well as completed an unknown sample within ± 25% of true value. Analyst training records are kept on file in the Human Resources department.

4.0 DEFINITIONS

Not applicable

5.0 METHOD VARIATIONS

Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a group leader or Senior Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard andexposure to these chemicals must be reduced to the lowest level possible. Environmental Physics, Inc. (EPI) maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the laboratory, respectively. Individual sample MSDS forms provided by the clients are kept in Login.

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are:

6.3.1 Nitrile gloves for concentrated acids and bases.

6.4 Never leave gas cylinders unchained or untied, including when they are on a moving cart.

6.5 Instructions on the handling of radioactive samples is outlined in EPI SOP "Handling of Radioactive Samples" (GL-EPI-E- M-001). The following general guidelines are applicable:

6.5.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling.

6.5.2 Wear a plastic apron over lab coat when working with radioactive samples.

6.5.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.5.4 Prohibit admittance to immediate work area.

6.5.5 Post signs indicating radioactive samples are in the area.

6.5.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in EPI.

6.5.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

6.6 Refer to EPI SOP "EPI Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011), and GEL/EPI Laboratory Waste Management Plan (GL-LB-G-001) for instructions on how materials are disposed.

7.0 INTERFERENCES

7.1 Stable strontium in the samples will compete for sites on the Sr Resinâð column and will affect the chemical yields.

7.2 Most milk contains 1400-1700 mg of potassium (K) per liter. Because the ionic radii of K and Sr are very close, K will compete with Sr for resin sites on the Sr Resin. Therefore, removal of K is necessary to prevent extremely low Sr yields.

7.3 Ammonium ion interferes with milk. Sodium hydroxide is used instead of ammonium hydroxide to eliminate this interference.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 Stainless steel planchettes (2"x 1/8")

8.1.2 Hot plates

8.1.3 Centrifuge tubes (plastic)

8.1.4 Beakers; 600 mL, 1000 mL, 2000 mL

8.1.5 Drying lamps/drying oven

8.1.6 Desiccator

8.1.7 0.45 µm 47 mm diameter Gelman DM-450® Tuffryn filters or equivalent

8.1.8 47 mm filter funnels

8.1.9 Sr Resin® columns EIChroM Industries Inc.

8.1.10 Culture tubes (150 mm x 20 mm)

8.1.11 .45 µm 47 mm diameter cellulose nitrate membrane filters

8.1.13 Teflon-coated stirring magnets

8.2 Reagents, Chemicals and Standards

8.2.1 Deionized water (DI water).

8.2.2 Strontium carrier, 5 mg/mL: Dissolve 12.08 g $Sr(NO_3)_2$ in DI water and dilute to 1000 mL.

8.2.3 Nitric acid, 8 M: Add 500 mL concentrated nitric acid (16 M HNO_3) to 400 mL DI water, allow to cool, then dilute to 1000 mL with DI water.

8.2.4 NaOH, 6 M: Cautiously add 120 g NaOH pellets to approximately 300 mL water when cool, dilute to 500 mL.

8.2.5 Sodium carbonate, .75 M: Add 79.5 g Na_2CO_3 to 1000 mL with DI water.

8.2.6 Nitric acid, 3 M: Carefully mix 189 mL 16 M HNO_3 (concentrated) with 500 mL DI water , allow to cool and dilute to 1000 mL.

8.2.7 Nitric acid, 0.05 M: Carefully mix 3.1 mL 16 M HNO_3 (concentrated) with 500 mL DI water and dilute to 1000 mL with DI water.

8.2.8 Ammonium hydroxide, concentrated (14.8 M)

8.2.9 Yttrium carrier, dissolve 22.90 g Y_2O_3 in 500 mL DI water. Add 150 mL concentrated HNO_3 and dilute to 1000 mL.

8.2.10 Phenolphthalein solution. Dissolve 1.0g phenolphthalein in 50 mL ethyl alcohol. Dilute to 100 mL with DI water.

8.2.11 Thymol Blue pH indicator.

8.2.12 1 M Oxalic acid. Dissolve 126.07 g and dilute to 1000 mL.

8.3 Reagents, chemical and standards for milk sample preparation.

8.3.1 Strontium carrier, 10 mg Sr/mL. Dissolve 2.42 g of Sr (NO3)2 in 100 mL of water. The reagent must be standardized by precipitating as SrCO3 and drying at the same temperature used for samples. Standardize by precipitating three 5.00 mL aliquots as SrCO3. Filter through tared Gooch crucibles, dry under a heat lamp, and weigh. Acceptable RSD for a triplicate determination is 0.5%.

NOTE: Standardization must be performed even if the carrier is a certified solution, e.g., from HPS. This is necessary because drying conditions may result in the retention of water of hydration that makes the empirical formula of the precipitate different from the theoretical. This practice will ensure equivalence between standards and samples.

8.3.2 Nitric acid, 8 M. Add 500 mL of concentrated nitric acid to 400 mL of DI water and mix. Dilute to 1 liter.

8.3.3 Cation resin, Dowex 50-X8, sodium form, or equivalent. Use 15-50 mesh material for easier recovery from the milk.

8.3.4 Sodium hydroxide, 10M. Add 400g of NaOH pellets to 800 mL of DI water and mix. Dilute to 1 liter when cool.

8.3.5 Sodium carbonate, 0.75 M. Add 79 g of Na2CO3 to 900 mL of DI water. Stir until dissolved and dilute to 1 liter.

8.4 Instrumentation

8.4.1 Gas-flow proportional counting system

8.4.2 Analytical balance.

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 Water samples should be collected in plastic bottles and preserved with concentrated nitric acid to pH 2.

9.2 If the sample is received with pH greater that 2, the analyst should modify the sample to pH 2, mix and allow to sit overnight before proceeding.

9.3 Sample handling and preservation for milk samples.

9.3.1 Samples should be preserved with 50 mL of formaldehyde per liter so that storage in a refrigerator is not necessary. Samples which are not preserved at collection must be kept refrigerated until preserved.

10.0 SAMPLE PREPARATION

10.1 Sample preparation techniques for water matrix.

10.1.1 Transfer an aliquot of water sample to an appropriate beaker and record the volume on the que sheet (Appendix 1). Add several drops of phenolphthalein solution and add 6 M NaOH to a light purple color. Add 1.0 mL strontium carrier, then cover the beaker with a watch glass and heat to rapid boiling.

10.1.2 Remove the watch glass and allow the sample to partially cool. Slowly add 10 mL .75 M Na_2CO_3 while stirring. Allow the precipitate to settle and cool for at least one hour.

10.1.3 Filter the sample through a .45µm cellulose nitrate membrane filter. Transfer the remaining precipitate to the filter with a fine stream of DI water. Rinse the sides of the beaker with a minimum amount of 8 M nitric acid and save the rinse.

10.1.4 Place a culture tube in a side arm vacuum flask. Place the funnel apparatus with the filter on the flask so the tip of the funnel is in the culture tube.

10.1.5 Without vacuum applied, add the rinse from beaker in 10.1.3. Apply vacuum after 20-30 seconds. Add an additional 3 mL 8 M nitric acid to the filtering funnel to rinse the funnel sides of any precipitate.

10.1.6 Add a final 3 mL 8 M nitric acid to complete the dissolution of remaining precipitate on the filter into the culture tube.

10.1.7 Prepare the Sr Resin® columns by pre-rinsing with 5 mL 8 M nitric acid.

10.1.8 Load the sample onto the column. Rinse the tube onto the column with an additional 1-2 mL 8 M HNO_3.

10.1.9 When the sample has passed completely through the column, rinse the column with 10 mL 3 M HNO_3. Discard the rinse.

10.1.10 When the rinse has passed completely through the column, place a clean, labeled centrifuge tube under the column and elute the strontium with 15 mL 0.05 M nitric acid. Record the date and time as the beginning of Y-90 ingrowth.

NOTE: The remaining steps should be performed as quickly as possible to avoid excess Y-90 ingrowth. Save the labeled Sr Resin® column for later Y-90 separation.

10.1.11 Add 2 drops Thymol Blue indicator, the add 6 N NaOH until color blue, then add 3 mL 0.75M sodium carbonate and heat in microwave for approximately 30 seconds - 1 minute. Allow precipitate to settle for 30 minutes.

10.1.11 Add 5 mL 6 M NaOH to the centrifuge tube and 10 mL .75 M sodium carbonate. Cap the tube and shake it vigorously then allow the precipitate to collect for 30 minutes.

10.1.12 Label and weigh a 47 mm DM-450® Tuffryn filter for each sample. Filter the precipitate quantitatively through the pre-weighed Tuffryn filter, rinsing the final precipitate out of the tube with a fine jet of water.

10.1.13 Dry the filters in an oven at 100°C. Move the filters to a dessicator for 10 minutes and weigh. Record the weight on the que sheet.

10.1.14 Count the filters in the low background gas flow gross alpha/beta counter for a duration which will meet the CRDL and uncertainty.

10.1.15 Once counted, store the samples for a minimum of 5 to 7 days, to allow for Y-90 ingrowth.

10.1.16 Remove the filter from the planchette and place in the 47 mm filtering funnel. Place a labeled culture tube in a side arm flask and position the filter in a funnel above the culture tube.

10.1.17 Add 5 mL 8 M nitric acid and allow the solution to flow by gravity through the filter for 20-30 seconds. (This will ensure the strontium carbonate is being completely dissolved.). Pull the residual solution through the filter by applying a vacuum to the flask.

10.1.18 Add an additional 10 mL 8 M nitric acid to remove. and rinse the remaining precipitate into the culture tube.

10.1.19 Add 1.0 mL yttrium carrier to the culture tube and swirl.

10.1.20 Prepare the original Sr Resin® column for Y-90 separation by adding 5 mL 8 M nitric acid. Discard the washings.

10.1.21 Place a clean, labeled tube under the column and load the filtrate in the culture tube onto the column. Rinse the culture tube with 1-2 mL 8 M nitric acid into the column. Record the date and time as this marks the end of the Y-90 ingrowth and the beginning of the Y-90 decay.

10.1.22 Precipitate the yttrium by slowly adding 1-2 drops Thymol blue indicator, 1mL 1 M oxalic acid, then slowly add ammonium hydroxide until yellowish color throughout sample. Allow the precipitate tto stand with occasional swirling for 30 minutes.

10.1.23 Filter the yttrium oxalate through a pre-weighed 47 mm Tuffryn filter.

10.1.24 Dry the filters under a heat lamp or in a drying oven. Move the filters to a dessicator for 10 minutes and weigh. Calculate the net precipitate on the filter to determine the yttrium yield.

10.1.25 Mount the filter on a planchette and count for yttrium-90 beta activity in the low background gas flow gross alpha/beta counter.

10.1.26 Enter all necessary data into the calculation spreadsheet. Update method control charts, and enter final results into LIMS.

10.2 Sample preparation techniques for soil matrix.

10.2.1 Perform procedure EPI SOP A-021 "Soil Preparation for Radionuclide Analysis" including the ashing routine using 1 mL strontium carrier and up to 2.0 g sample aliquot. Record the aliquot information on the Sr que sheet.

10.2.2 Leach method (common):

10.2.2.1 After the sample has been ashed. Transfer the sample to a suitable beaker for acid leaching.

10.2.2.2 Slowly add 30 - 50 mL 8M HNO_3 to the beaker. Cover the sample with a watchglass and reflux.

10.2.2.3 Slowly add 2-3 drops 30% H_2O_2, stir and continue refluxing for 30 min.

10.2.2.4 Repeat step 10.2.2.3 two more times.

10.2.2.5 Slurry the sample and transfer the mixture to a centrifuge tube.

10.2.2.6 Centrifuge the mixture to separate the leachate from the remaining soil. Transfer the leachate into a Teflon beaker.

10.2.2.7 Add 10 mL 8 M HNO_3 to the centrifuge tube, cap and mix. Repeat step 10.2.2.6 and this step two additional times.

10.2.2.8 Centrifuge and discard any remaining solid matter. Heat the leached sample in the Teflon beaker to dryness. Add a dropper of 5% boric acid and 10 mL 8 M nitric acid. Cover and boil for approximately 10 minutes. When cool dilute to desired volume.

10.2.2.9 Add 1-2 drops of phenolphthalein . Add 6 M NaOH until the solution shows a basic (purple) color.

10.2.2.10 Add 10 mL .75 M Na_2CO_3. Stir and allow the precipitate to digest for 30 min.

10.2.2.11 Proceed as directed for water matrix at step 10.1.3.

10.2.3 Microwave digestion method (rapid leach for smaller samples):

10.2.3.1 After the sample has been ashed, perform procedure EPI SOP "Microwave Digestion of Soil and Sand" (GL-EPI-E-A015).

10.2.3.2 After HNO_3 conversion is complete, increase the sample volume to 50 mL with DI H_2O and then to 10 mL with DI H_2O. The sample is now ready for carbonate precipitation.

10.2.3.3 Proceed as directed for soil matrix at step 10.2.1.9.

10.2.4 The determination of Sr in large soil samples.

10.2.4.1 For each sample to be analyzed, pipet a 0.500 mL aliquot of Sr carrier (10 mg/mL) into a 50 mL flask.

10.2.4.2 Prepare a standard by diluting 0.500 mL of Sr carrier to 50 mL with water in a volumetric flask. Pipet 1.00 mL into a second 50 mL volumetric flask and dilute to the mark with 0.1 M HON. This 2 ppm standard will be used as the reference for all subsequent ICP determinations of Sr.

10.2.4.3 Transfer 10 grams of soil to a 150 mL beaker. Add 25 mL of 8M HNO3. Mix well, cover, and heat to just below boiling for 2 hours. Cool.

10.2.4.4 Centrifuge. Decant the supernate into the respective volumetric flask from Step 10.2.4.1.

10.2.4.5 Slurry the precipitate with 10 mL of 2M nitric acid and centrifuge. Decant the supernate into the volumetric flask from Step 10.2.4.3.

10.2.4.6 Slurry the precipitate with a second 10 mL portion of 2M nitric acid and centrifuge. Decant the supernate into the volumetric flask from Step 10.2.4.3. Fill the flask to the mark with water. Cap and thoroughly mix the contents.

10.2.4.7 Remove a 1.00 mL aliquot and transfer to a second 50 mL volumetric flask. Dilute to the mark with water. Cap, mix, and transfer to a centrifuge tube. Label with sample number and "initial Sr".

10.2.4.8 Transfer the contents of the first volumetric flask from Step 10.2.4.7 to a Teflon beaker. Rinse the flask with several small portions of water. Add 10 mL of 5% boric acid. Cover and boil for 10 minutes. Cool.

10.2.4.9 Dissolve the residue in 10 mL of 8M nitric acid. Load the sample onto the column and discard the effluent.

10.2.4.10 Prepare Sr columns by rinsing with 5 mL of 8M nitric acid. Load the sample onto the column and discard the effluent.

10.2.4.1 Rinse the column with 10 mL of 3M HNO3. Discard the effluent. Record the hour and date as the beginning of Y-90 ingrowth.

10.2.4.12 Elute Sr with 20 mL of 0.05M HNO3 collecting the effluent in a centrifuge tube. If Sr-90 only is needed, cap the tube from Step 10.2.4.12 and set aside for 5-7 days to allow Y-90 ingrowth, and then continue with Step 10.2.4.26. If Sr-89 and Sr-90 are both needed, continue with Step 10.2.4.13.

10.2.4.13 Add 2 drops of phenolphthalein indicator solution and 2 mL of 6M NaOH. If the indicator has not turned pink, add additional 6M NaOH dropwise. Now add 1 mL of 0.75M Na2CO3 and mix. Heat to just below boiling. (This might take 30 seconds or so in the microwave.) Allow the precipitate to digest for 15 minutes.

10.2.4.14 For each sample, rinse a 47-mm DM-450® or Tuffryn HT-450® filter with water and alcohol, dry, and weigh.

10.2.4.15 Place a tared filter from Step 10.2.4.14 in the filtration apparatus, rinse the funnel with 5 mL of 80% alcohol, and disconnect the vacuum.

10.2.4.16 Pour the sample into the filter chimney. Connect the vacuum and suck dry. Be careful not to disturb the precipitate with subsequent rinses.

10.2.4.17 Disconnect the vacuum and add 5 mL of water to rinse the funnel. Connect the vacuum and suck dry.

10.2.4.18 Disconnect vacuum and add 5 mL of water to rinse and funnel. Connect the vacuum and suck dry.

10.2.4.19 Disconnect vacuum and add 5 mL of water to rinse the funnel. Connect the vacuum and suck dry.

10.2.4.20 Disconnect vacuum and add 5 mL of 80% alcohol to rinse the funnel. Connect the vacuum and suck dry.

10.2.4.21 With the vacuum still connected, remove the filter chimney and rinse the outside edges of the filter with water and 8% alcohol. Suck dry.

10.2.4.22 Disconnect the vacuum, remove the filter, and dry in the oven at 105°C. Mount the filter for counting using double-sided tape to keep the filter flat.

10.2.4.23 Count immediately in the low background counter for a duration necessary to meet client specified detection limit.

10.2.4.24 After counting, rinse the $SrCO_3$ precipitate into a 50 mL volumetric flask using 2M HNO_3. Fill the flask to the mark with water. Remove a 1.00 mL aliquot and dilute to 50 mL with 0.1 M HNO_3 in a second volumetric flask. Transfer this solution to a centrifuge tube and label with sample number and "final Sr".

10.2.4.25 Send the Sr standard and the "initial Sr" and "final Sr" samples for ICP analysis. Since all dilutions have been made in the same way, the Sr yield is calculated by dividing the "final Sr" ICP ppm result by the "initial Sr." This provides correction for stable Sr which may have been present in the sample. An additional factor of 0.98 in the denominator of the activity calculation is necessary to correct for the aliquot removed in Step 10.2.4.7 for ICP analysis.

NOTE: If Sr-90 only is being determined, begin here after Step 10.2.4.12.

10.2.4.26 To the sample from Step 10.2.4.12 add 1.00 mL of yttrium carrier (10mg/mL). Add a few drops of phenolphthalein and then add NH_4OH until the indicator turns pink. Heat in the microwave coagulate the precipitate. (Depending on the number of samples, 10-30 seconds should be sufficient.)

10.2.4.27 Centrifuge. Decant the supernate into a 50 mL volumetric flask. record the hour and dates as time of separation of Y and Sr.

10.2.4.28 Dissolve the precipitate in 10 drops of HCl and dilute to 20 mL with water. Add NH_4OH dropwise until $Y(OH)_3$ precipitates and then heat in the microwave as before.

10.2.4.29 Centrifuge. Decant the supernate into the 50 mL volumetric flask from Step 10.2.4.27. Fill the flask to the mark with water. Transfer 1.00 mL to a second volumetric flask. Fill to the mark with 0.1 M nitric acid. Transfer to a centrifuge tube and label with sample number and "final Sr". Submit the "initial Sr," "final Sr," and standard for ICP analysis.

10.2.4.30 Dissolve the precipitate from Step 10.2.4.29 in 10 drops of HCl and dilute 25 mL with water. Add NH4OH dropwise until Y(OH)3 precipitates, and then heat in the microwave as before.

10.2.4.31 Centrifuge. Decant and discard the supernate. Dissolve the precipitate from Step 10.2.4.29 in 10 drops of HCl and dilute to 25 mL with water. Add 1 drop of thymol blue indicator and 1 mL of 1M oxalic acid.

10.2.4.32 Add NH4OH dropwise with mixing until the indicator turns orange. The pH will be 1-2, which is necessary to ensure that yttrium oxalate precipitates as the 9-hydrate. Heat in the microwave as before, and then let the precipitate digest for 15 minutes.

10.2.4.33 For each sample, rinse a 47-mm DM-450® or Tuffryn HT-450 filter with water and alcohol, dry, and weigh.

10.2.4.34 Place a tared filter from Step 10.2.4.33 in the filtration apparatus, rinse the funnel with a 5 mL 0f 80% alcohol, and disconnect the vacuum.

10.2.4.35 Pour the sample into the filter chimney. Connect vacuum and suck dry. Be careful not to disturb the precipitate with subsequent rinses.

10.2.4.36 Disconnect vacuum and add 5 mL of water to rinse the funnel. Connect the vacuum and suck dry.

10.2.4.37 Disconnect vacuum and add 5 mL of water to rinse the funnel. Connect the vacuum and suck dry.

10.2.4.38 Disconnect vacuum and add 5 mL of water to rinse the funnel. Connect the vacuum and suck dry.

10.2.4.39 Disconnect vacuum and add 5 mL of water to rinse the funnel. Connect the vacuum and suck dry.

10.2.4.40 With the vacuum still connected, remove the filter chimney and rinse the outside edges of the filter with water and 8% alcohol. Suck dry.

10.2.4.41 Disconnect the vacuum, remove the filter, and dry in the oven at 105°C. Mount the filter for counting using double-sided tape to keep the filter flat.

10.2.4.42 Count immediately in the low background counter for a duration necessary to meet client specified detection limit.

10.2.4.43 Yttrium yield is determined from the weight of the Y2(C2O4)3-9H2O precipitate. Sr yield is calculated by dividing the "final Sr" ICP ppm result by the "initial Sr". This provides correction for stable Sr which may have been present in the sample. An additional factor of 0.98 in the denominator of the activity calculation is necessary to correct for the aliquot removed in Step 10.2.4.7 for ICP analysis.

10.3 Sample preparation for milk samples.

10.3.1 Transfer a measured volume of sample (0.5-1L) to a beaker and add 1.0 mL (10mg) of Sr carrier. Add a Teflon-coated stirring magnet and stir for 15 minutes.

10.3.2 Add 60 mL of cation resin and continue stirring for 30 minutes. Stirring must be vigorous enough to distribute the resin throughout the sample.

10.3.3 Remove the beaker from the stirrer and allow the resin to settle.

10.3.4 Slowly decant and discard the milk, ensuring that no resin is lost. Rinse the beaker and resin with several 200 mL portions of hot water, continuing until the rinse is clear. It is necessary that the milk be completely removed from the resin.

10.3.5 Transfer the resin to a large chromatography column which has been fitted with a control valve at the bottom. The resin will completely fill the column and extend slightly into the column reservoir. This will not affect subsequent operations. Drain water from the column down to the top of the resin bed.

10.3.6 Elute the Sr from the resin by passing 400 mL of 8M nitric acid through the column at a flow rate of 1-2 drops per second. Collect the effluent in a 600 mL beaker.

NOTE: If the flow rate is greater than 1-2 drops per second. Sr stripping will be incomplete. Slower flow rates will not adversely affect the stripping except to increase the time necessary for elution.

10.3.7 Evaporate the effluent to a volume of about 50 mL. Some insoluble material may be present. Transfer the sample to a 1 L beaker and dilute to 500 mL with water.

10.3.8 Add 10M NaOH with stirring to the sample until the solution is alkaline to phenolphthalein (pink) or thymol blue (blue). Then add 50 mL of 0.75M Na2CO3 and stir. cover and heat to near boiling. Let the precipitate stand for several hours, or overnight, if possible, to allow all insoluble material to settle.

10.3.9 Carefully decant and discard the clear solution. Transfer the voluminous precipitate to a centrifuge tube and centrifuge. Discard the supernate. Wash the precipitate with 35 mL of water, ensuring that the precipitate is thoroughly resuspended in the wash.

10.3.10 Repeat step 10.3.9.

NOTE: Most milk contains 1400-1700 mg of K per liter. Because the ionic radii of K and Sr are very close, K will compete with Sr for resin sites on the Sr Resin. Therefore, removal of K is necessary to prevent extremely low Sr yields.

10.3.11 In small portions, very carefully add 8M HNO3 until the precipitate is dissolved. Copious amounts of CO2 will be evolved, and care must be exercised to avoid loss of sample due to spattering.

10.3.12 Complete the analysis using steps 10.1.7 through 10.1.26.

10.4 Sample preparation techniques for meats, fish, vegetation, and other tissues.

NOTE: The following twelve steps are in addition to the steps in Section 10.1 for sample preparation of water samples.

10.4.1 Ash the sample as specified in EPI SOP "Soil Sample Ashing for the Determination of Radionuclides" (GL-EPI-E-A023B).

10.4.2 Add 300 mL of 8M nitric acid, 30 mg of calcium carrier and 0.5 mL stable strontium carrier to the ashed tissue.

10.4.3 Cover the sample and heat to reflux for 1-2 hours. Allow to cool and filter through a glass fiber filter. Collect the filtrate.

10.4.4 Boil the filtrate and evaporate to approximately 25-50 mL. Dilute to 700 mL.

10.4.5 Add 1 mL of methyl orange and 10 g of oxalic acid. Stir the solution until the oxalic acid has dissolved.

10.4.6 Add ammonium hydroxide until the solution turns yellow, giving a pH of approximately 4. Allow the samples to sit overnight.

10.4.7 Decant the supernate and collect the precipitate in a centrifuge. Centrifuge, then discard the excess supernate. Rinse the precipitate with deionized water and recentrifuge. Discard the deionized water.

10.4.8 Dissolve the precipitate in 50 mL of 8M nitric acid. Transfer to a 250 mL beaker and very slowly take to dryness.

10.4.9 Wet ash the samples with 5-10 mL of 8M nitric acid, and 1 mL of hydrogen peroxide. (Repeat this process until the sample no longer produces a brownish gas).

10.4.10 Add 8M nitric acid and reflux until the sample dissolves. Take this to dryness.

10.4.11 Ash the samples in a furnace at 500° for 30 min. Allow the samples to cool.

10.4.12 Place the samples on a hotplate and add 10-15 mL of 8M nitric acid and heat until the solids dissolve. (This is the load solution that is run through the column).

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to EPI SOP "Preparation of Radioactive Standards" (GL-EPI-E-M001).

11.2 Preparation procedures

11.2.1 A "blank" water should be sued as the the batch blank.

11.2.2 A duplicate and spied sample should be made on the same sample in a sample batch. The spiked sample is made by adding a known quantity of Sr-89 and Sr-90 to the sample.

11.2.3 A laboratory control sample should be run with each batch by spiking a DI water sample with a known quantity of strontium standard.

11.3 Source materials are ordered from any of four main suppliers listed below. All four companies are called for a quote, and the most economical price for the laboratory needs is selected. All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in Section 19.0 of EPI SOP "Preparation of Radioactive Standards" (GL-EPI-E-M001).

11.3.1 National Institute of Standards and Technology (NIST), Gaithersburg, MD.

11.3.2 Isotope Products Laboratories, Burbank, CA.

11.3.3 Amersham Corporation, Arlington Heights, IL.

11.3.4 North American Scientific Products and Services, North Hollywood, CA.

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 Standardization of Sr carrier.

12.1.1 Pipet four 5.00 mL aliquots of Sr carrier (nom. conc. 10 mg Sr/mL) into tared 50 mL centrifuge tubes. Record the weight of each carrier aliquot.

12.1.2 Dilute to 30 mL of water. Add 2 drops thymol blue indicator (or phenolphthalein). Add 1 mL of 10 M NaOH. The indicator should turn blue (or pink if using phenolphthalein). Add additional NaOH dropwise if necessary until the indicator changes color. Add 2 mL of 0.75 M Na2CO3 to precipitate Sr CO3. Heat in the microwave for 20-30 seconds. Set aside for 15 minutes.

12.1.3 Filter through a tared Gooch crucible containing a double 2.1 cm glass fiber filter. Rinse with two 5 mL portions of water, and one 5 mL portion of 80% alcohol.

12.1.4 Dry in the oven for 30 minutes at 105°-110°C. Cool. Weigh. Calculate the standard weight of Sr carrier in mg/mL and mg/g of solution as follows to obtain the standard weight for 1.00 mL or 1.00 gram of Sr carrier solution:

$$Sr.\ Std.\ Wt.\ mg/mL\ of\ carrier\ solution = \frac{Net\ Wt.,\ mg\ of\ SrCO_3}{5.00}$$

$$Sr.\ Std.\ Wt.\ mg/gram\ of\ carrier\ solution = \frac{Net\ Wt.,\ mg\ of\ SrCO_3}{Net\ Wt.\ g\ of\ 5-mL\ aliquot}$$

12.1.5 Acceptable precision is a relative standard deviation of (0.5%. Record the carrier lot number if obtained from HPS) and the standardization results in the lab notebook. Label the carrier solution with the standardization results. The solution should be restandardized every 3 months.

12.2 Standardization of Y carrier

12.2.1 Pipet four 5.00 mL aliquots of Y carrier (nom. conc. 10 mg Y/mL) into tared 50 mL centrifuge tubes. Record the weight of each carrier aliquot.

12.2.2 Dilute to 30 mL with water. Add 2 drops thymol blue indicator. Add 1 mL of 1M oxalic acid. Add NH4OH dropwise until the indicator changes from red to orange.

NOTE: The desired pH is 1.9 which is necessary to ensure that Y2(C2O4)3 precipitates with the correct stoichiometric form. Heat in the microwave for 20-30 seconds. Set aside for 15 minutes.

12.2.3 Filter through a tared Gooch crucible containing a double 2.1 cm glass fiber filter. Rinse with two 5 mL portions of water, and one 5 mL portion of 80% alcohol.

12.2.4 Dry in the oven for 30 minutes at 105-110°C. Cool. Weigh. Calculate the standard weight of Y carrier in mg/mL and mg/gram of solution as follows to obtain the standard weight for 1.00 mL or 1.00 gram of Y carrier solution:

$$Y\ Std.\ Wt.\ mg/mL\ of\ carrier\ solution = \frac{Net\ Wt.,\ mg\ of\ Y_2(C_2O_4)^3}{5.00}$$

$$Y\ Std.\ Wt.\ mg/gram\ of\ carrier\ solution = \frac{Net\ Wt.,\ mg\ of\ Y_2(C_2O_4)^3}{Net\ Wt.,\ g\ of\ 5-mL\ aliquot}$$

12.2.5 Acceptable precision is a relative standard deviation of 0.5%. Record the carrier lot number (if obtained from HPS) and the standardization results in the lab notebook. Label the carrier solution with the standardization results. The solution should be restandardized every 3 months.

12.3 Preparation of Sr-89 Standards

NOTE Eppendorf pipets are used to quickly add estimated amount of carrier in the preparation of standards. To unsure highest accuracy, the amount added is calculated by weight. It is important to follow the filter preparation and precipitate washing and rinsing steps carefully to ensure highest accuracy in yield determination.

12.3.1 For each standard (usually 8), soak a HT-450® Tuffryn filter in 80% ethanol for 5 minutes. Remove the filters, place in filter holders, connect vacuum, and suck dry. Remove the filters and dry in an oven at 105°-110°C. Cool. Weigh.

12.3.2 Weigh and record an aliquot of Sr-89 tracer (10,000 dpm) into each of 8 disposable centrifuge tubes. Add Sr carrier to each tube according to the following table. This will provide a curve that extends to 40 mg of precipitate.

Table 12.3.2.1

Tube	mL of Sr carrier, g	Expected Precipitate Wt., mg*
1	.2	3.37
2	.4	6.74
3	.6	10.10
4	.8	13.47
5	1.1	18.52
6	1.4	23.58
7	1.7	28.63
8	2.0	33.68

*Assumes the Sr carrier is 10.0 mg Sr/mL

12.3.3 Dilute to 30 mL with water and add 2 drops of thymol blue. Add 1mL 10M NaOH to raise pH>8. If the indicator does not turn blue, add more 10M NaOH dropwise. Do not add a large excess. Then add 1 mL of 0.75M Na2CO3. Heat for 20-30 seconds in the microwave, or 5 minutes in a hot water bath. Allow the precipitate to stand for 15 minutes.

12.3.4 Place a tared filter from Step 12.3.1 into the filter holder, rinse the assembly with 5mL of 80% ethanol, and connect the vacuum. Suck dry and disconnect vacuum. Swirl the centrifuge tube containing the standard to be filtered, and pour the entire sample into the filter reservoir. Apply vacuum and suck dry.

12.3.5 Disconnect vacuum. Rinse the filter with 5 mL of water. Connect the vacuum and suck dry.

12.3.6 Disconnect vacuum. Rinse the filter with 5 mL of water. Connect the vacuum and suck dry.

12.3.7 Disconnect vacuum. Rinse the filter with 5 mL of water. Connect the vacuum and suck dry.

12.3.8 Disconnect vacuum. Rinse the filter with 5 mL of 80% ethanol. Connect vacuum and suck dry. Continue to rinse the funnel assembly with 80% ethanol as necessary to recover any precipitate clinging to the walls.

12.3.9 With vacuum connected, remove the reservoir and rinse the edges of the filter with 80% ethanol. Disconnect vacuum and remove filter. Place on a Kim Wipe to remove excess moisture, and then dry in the oven at 105°-110°C. Cool. Weigh.

12.4 Preparation of Sr-90 standards

12.4.1 For each standard (usually 8), soak a HT-450® Tuffryn filter in 80% ethanol for 5 minutes. Remove the filters, place in filter holders, connect vacuum, and suck dry. Remove the filters and dry in an oven at 105°-110°C. Cool. Weigh.

12.4.2 Weigh and record an aliquot of Sr-90 tracer (10,000 dpm) into each of 8 disposable centrifuge tubes. Use a pipet to make the transfer. Add 1.0 mL of Y carrier and Sr carrier to each tube according to table 12.3.2.1. This will provide a curve that extends to 40 mg of precipitate.

12.4.3 Dilute to 30 mL with water and add 2 drops of thymol blue. Add 1 mL NH4OH to raise pH > 8. If the indicator does not turn blue, add more ammonia dropwise. Do not add a large excess. Heat for 20-30 seconds in the microwave, or 5 minutes in a hot water bath. Allow the precipitate to stand for 15 minutes.

12.4.4 Filter the standard through a 25 mm glass fiber filter, collecting the supernate in a clean tube. Record the hour and date as Y-Sr separation time.

12.4.5 Add 1 mL of 0.75 M Na2CO3. Heat for 20-30 seconds in the microwave, or 5 minutes in a hot water bath. Allow the precipitateto stand for 15 minutes.

12.4.6 Place a tared filter from Step 12.4.1 into the filter holder, rinse the assembly with 5 mL of 80% ethanol, and connect the vacuum. Suck dry and disconnect vacuum. Swirl the centrifugetube containing the standard to be filtered, and pour the entire sample into the filter reservoir. Apply vacuum and suck dry.

12.4.7 Disconnect vacuum. Rinse the filter with 5 mL of water. Connect vacuum and suck dry.

12.4.8 Disconnect vacuum. Rinse the filter with 5 mL of water. Connect vacuum and suck dry.

12.4.9 Disconnect vacuum. Rinse the filter with 5 mL of water. Connect vacuum and suck dry.

12.4.10 Disconnect vacuum. Rinse the filter with 5 mL of 80% ethanol. Connect vacuum and suck dry. Continue to rinse the funnel assembly with 80% ethanol as necessary to recover any precipitate clinging to the walls.

12.4.11 With vacuum connected, remove the reservoir and rinse the edges of the filter with 80% ethanol. Disconnect vacuum and remove filter. Place on a Kim Wipe to remove excess moisture, and then dry in the oven at 105°-110°C. Cool. Weigh.

12.5 Preparation of Y-90 standards

NOTE: A self-absorption curve is not needed for Y-90 because it emits 2.27 MeV beta particles, which do not undergo measurable attenuation in the range of precipitate weights obtained during Sr analysis.

12.5.1 Soak 4 HT-450® Tuffryn filters in 80% ethanol for 5 minutes. Remove the filters, place in filter holders, connect vacuum, and suck dry. Remove the filters and dry in an oven at 105°-110°C. Cool. Weigh.

12.5.2 Pipet 1.00 mL of Y carrier into each of 4 disposable centrifuge tubes.

Add a certified known amount of Sr-90 standard (10,000 dpm) to each tube.

12.5.3 Dilute to 30 mL with water and add 2 drops thymol blue indicator. Add NH4OH until the indicator turns blue. Heat in the microwave for 20 - 30 seconds or 5 minutes in a hot water bath to coagulate the Y(OH)3 precipitate. Cool.

12.5.4 Centrifuge. Decant and discard the supernate in radwaste. Record the hour and date as time of separation of Y-Sr.

12.5.5 Dissolve the precipitate in 10 drops of HCl. Dilute to 30 mL with water. Add NH4OH dropwise until Y(OH)3 precipitates, and then 4 drops in excess. Heat in the microwave for 20 - 30 seconds or 5 minutes in a hot water bath to coagulate the Y (OH)3 precipitate. Cool.

12.5.6 Centrifuge. Decant and discard the supernate in radwaste. Dissolve the precipitate in 1 mL of HCl add 2 drops of thymol blue indicator, and dilute to 30 mL.

12.5.7 Add 1 mL of 1 M oxalic acid. Add NH4OH dropwise until the indicator barely turns orange. Heat in the microwave for 20 - 30 seconds or 5 minutes in a hot water bath. Let stand 15 minutes.

12.5.8 Place a tared filter from Step 12.5.1 into the filter holder, rinse the assembly with 5 mL of 80% ethanol, and connect vacuum. Suck dry and disconnect vacuum. Swirl the centrifuge tube containing the standard to be filtered, and pour the entire sample into the filer reservoir. Apply vacuum and suck dry.

12.5.9 Disconnect vacuum. Rinse the filter with 5 mL of water. Connect vacuum and suck dry.

12.5.10 Disconnect vacuum. Rinse the filter with 5 mL of water. Connect vacuum and suck dry.

12.5.11 Disconnect vacuum. Rinse the filter with 5 mL of water. Connect vacuum and suck dry.

12.5.12 Disconnect vacuum. Rinse the filter with 5 mL of 80% ethanol. Connect vacuum an suck dry. Continue to rinse the funnel assembly with 80% ethanol as necessary to recover any precipitate clinging to the walls.

12.5.13 With vacuum connected, remove the reservoir and rinse the edges of the filter with 80% ethanol. Disconnect vacuum and remove filter. Place on a Kim Wipe to remove excess moisture, and then dry in the oven at 105°-110°C. Cool. Weigh.

13.0 ANALYSIS AND INSTRUMENT OPERATION

Refer to EPI SOPs "HT1000 Gross Alpha/Beta Counter Operating Instructions" (GL-EPI-E-I002), or "LB4110 Gross Alpha/Beta Counter Operating Instructions" (GL-EPI-E-I006) for instrumentation operation procedures.

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 Routine Preventative Maintenance

14.1.1 Refer to EPI SOPs "HT-1000 Alpha/Beta Counter Operating Instructions" (GL-LB-E-I002), and "LB-4110 Alpha/Beta Counter Operating InstructiosOperation" (GL-LB-E-I006) for instructions concerning the gas flow proportional counters.

14.1.2 Refer to EPI SOP "Counting Room Instrument Maintenance and Performance Checks" (GL-LB-E-I010) for instructions concerning instrument maintenance.

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 The spreadsheet will calculate Sr-90 and Sr-89 in pCi/L according to the following equations:

15.1.1 $$Sr-90(pCi/L)=\frac{(CPM_s-CPM_b)}{(2.22)(V)(E_s)(Y_g)Y_d)(T_c)(R_s)}$$

15.1.2 $$Sr-89(pCi/L)=\frac{(CPM_{89}-CPM_{b1})}{(2.22)(V)(E_{89})(Sr_d)(R_s)(T_c)}$$

15.2 Counting uncertainty is calculated according to the following equations:

15.2.1 $$Sr-90(pCi/L)=\frac{1.96\sqrt{\frac{CPM_{90}}{T_c}+\frac{CPM_b}{C_b}}}{(2.22)(V)(E_s)(Y_g)Y_d)(R_s)(T_c)}$$

15.2.2 $$Sr-89(pCi/L)=\frac{1.96\sqrt{\frac{CPM_{89}}{T_c}+\frac{CPM_b}{C_b}}}{(2.22)(V)(E_{89})(Sr_d)(T_c)(R_s)}$$

15.3 The method MDA is calculated according to the following equation:

15.3.1 $$Sr-90\,MDA(pCi/L)=\frac{2.71+4.66\sqrt{CPM_b * T_c}}{2.22 * V * E_y * Y_d * Y_d * R_y * T_c}$$

15.3.2 $$Sr-89\,MDA(pCi/L)=\frac{2.71+4.66\sqrt{CPM_{b1} * T_c}}{2.22 * V * E_{89} * Sr_d * R_s * T_c}$$

Where:

CPM_Y=Yttrium cpm
CPM_b= Yttrium background cpm
CPM_{89}=Sr-89 cpm
CPM_{b1}=Sr-89 background cpm
2.22=Conversion from dpm to pCi
V=Sample Volume in liters
E_y=Yttrium counting efficiency
E_{89}=Sr-89 counting efficiency
Y_g=Yttrium ingrowth
Y_d=Yttrium decay
Sr_d=Strontium 89 decay
T_c=Count duration of the sample (min.)
C_b=Count duration of the background (min.)
R_S=Sr chemical recovery
R_y=Y chemical recovery

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification Requirements

Refer to EPI SOP "Analyst and Analytical Methods Validation Procedures" (GL-EPI-E-D002) for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank should accompany each batch of samples. The reported value of the blank should be less than or equal to the contract required detection limit (CRDL).

16.2.2 The carrier added to all samples is used to calculate the chemical yield. The chemical yield of all samples should be between 25-125%. The chemical yield is calculated as follows:

$$Chemical\ Yield = \frac{Sample\ ppt\ Weight}{Standardized\ ppt\ Weight}$$

16.2.3 A matrix spike (ms) should be run with every batch of samples. The recovery of the ms should be between 75-125%. The recovery is calculated as follows:

$$MS\ Recovery\ (\%) = \frac{Spike\ (pCi/L) - Sample\ (pCi/L)}{SpikeNominalConcentrion(pCi/L)}$$

Where:

Spike=results of matrix spike
Sample=results of sample without added spike
Spike Nominal Concentration (SNC) = concentration of Sr in spike, calculated as follows:

$$SNC = \frac{Standard\ dpm * mL\ of\ spike\ added}{2.22 * volume\ in\ liters}$$

16.2.4 A duplicate sample should be run with every batch. The relative percent difference (RPD) between the actual sample and the QC duplicate should be less than or equal to 20% if both the sample and the QC duplicate results are greater than 5 times MDA or 100% if they are both less than 5 times MDA. If either results less than MDA then limits on RPD are not applicable. The RPD should be calculated as follows:

$$RPD\ (\%) = \frac{ABS(DUP_1 - DUP_2)}{\frac{(DUP_1 + DUP_2)}{2}} * 100\%$$

Where:

ABS=Absolute value

16.2.5 A Laboratory Control Sample (LCS) should be run with every batch. The recovery of the LCS should fall between 75-125%. The LCS recovery is calculated as follows:

$$LCS_{RECOVERY} = \frac{LCS\ Result\ (pCi/L)}{Nominal\ Concentration\ of\ LCS\ (pCi/L)} * 100\%$$

16.3 Actions Required if the Quality Control Requirements Are Not Met

If any of the QC criteria from 12.2.1 through 12.2.5 cannot be satisfied, the analyst should inform their group leader and initiate a Non-conformance Report as outlined in GEL SOP "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

17.1 The analyst turns in the raw data, review checklist, que sheet, spreadsheet, data report, NCR's (if applicable), and other appropriate information in a batch to the Data Review Specialist.

17.2 Analysts data go through the following process of review:

17.2.1 The first level of review is the analyst review. The analyst will perform the following review procedure:

17.2.1.1 Visually check the que sheet, spreadsheet, raw data, and data report to make sure the information has been transcribed correctly.

17.2.1.2 Check to see that the required detection limit (RDL) is met if required.

17.2.1.3 Complete the batch checklist.

17.2.2 The second level review is performed by the Data Review Clerk who reviews the batch checklist, checks for special client requirements, and reviews the transcription.

17.2.3 The trird level review is complested by the Group Leader/Data Review Specialist who reviews the checklist and checks for special client requirements.

17.3 After the review process is complete, the data is transmitted from the laboratory personnel to the reporting personnel as outlined in EPI SOP D-003, "Data Review and Validation Procedures."

17.4 The Data ReviewSpecialist/Quality Group Leader is responsible for reporting the data.

18.0 RECORDS MANAGEMENT

18.1 Each analysis that is performed on the instrument will be documented in the run log according to GEL SOP "Run Logs" (GL-LB-E-009).

18.2 All raw data printouts, calculations, spreadsheets, and batch checklists are filed with the sample data for review and achival.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Radioactive samples and material shall be handled and disposed of as outlined in EPI SOP "EPI Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011), and GEL/EPI Laboratory Waste Management Plan (GL-LB-G-001).

20.0 REFERENCES

20.1 B.D. Stewart "Preparation of Milk Samples for Strontium Analysis," Sr-01, Radiochemistry Procedures Manual, Arizona State University Radiation Measurements Facility, 1992.

20.2 US EPA Method 900.1

20.3 EML Procedures Manual. HASL-300-Ed25. 1982.

20.4 Los Alamos National Laboratories Methods Manual.

20.5 EIChroM technical method data.

20.6 Special thanks to Dr. Frank Kinard with the College of Charleston for his help in reviewing the Strontium method.

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APPENDIX 1

Strontium Que Sheet Batch #: 119302 Analyst: Aadli Kareem Comments:

Sr-89 Spike Code:_____ Vol:_____ Sr Sep. Date/Time:_____ Prep Date:_____
Sr-90 Spike Code:_____ Vol:_____ Y Sep. Date/Time:_____ Initials :_____
Sr-89/90 LCS Code:_____/_____ Vol:_____ Minimum Duedate: 4/9/98

Sample ID	Client Description	Min RDL	Client	Tests	Sample Date/Time	BKR #	Aliquot (mL or g)	Carrier or Det #	Initial Sr Weight (g)	Final Sr Weight (g)	Net Sr Weight (mg)	Initial Y Weight (g)	Final Y Weight (g)	Net Y Weight (mg)
9803521-01	075-2118	2 pCi/L	CODE	-90 -90	3/18/98									
9803521-02	075-2120	2 pCi/L	CODE	-90 -90	3/18/98									
9803521-03	075-2119	2 pCi/L	CODE	-90 -90	3/18/98									
9803525-04	98-CIF-0400	5 PCL	WSRC	-90 -90	3/22/98 1312									
9803652-01	HAA 5A	2 PCL	WSRC	-90 -90	3/26/98 0930									
9803652-02	HAA 5C	2 PCL	WSRC	-90 -90	3/26/98 0840									
9803653-01	QA 99A	2 PCL	WSRC	-90 -90	3/26/98 1459									
9803653-02	QA 100A	2 PCL	WSRC	-90 -90	3/26/98 1459									
9803653-03	HXB 4D	2 PCL	WSRC	-90 -90	3/26/98 1236									
9803653-04	HXB 5D	2 PCL	WSRC	-90 -90	3/26/98 1459									
9803680-01	CSO 1	2 PCL	WSRC	-90 -90	3/27/98 1127									
9803722-03	980326147	2 pCi/L	MLAB	-90 -90	3/25/98									
9803722-04	980326148	2 pCi/L	MLAB	-90 -90	3/25/98									
QC497147	blank	2 pCi/L	QC	-90 -90	3/31/98									
QC497148	9803653-04	2 pCi/L	QC	-90 -90	3/31/98									
QC497149	9803653-04	2 pCi/L	QC	-90 -90	3/31/98									
QC497150	LCS	2 pCi/L	QC	-90 -90	3/31/98									

Instrument Used: (Circle On LB4110 S/N: 8219 HT1000 S/N: 10912771 Data Reviewed By:_____
LB5100 S/N: 203 LB5100W S/N: 14740

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STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF TECHNETIUM-99

(GL-EPI-A-005 Revision 5)

UNCONTROLLED DOCUMENT

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Process Owner	8/12/99 (Sign and Date)	Tim C. Winters (Print Name)
Technical Review	8/10/99 (Sign and Date)	Richard M. Kinney (Print Name)
Quality Review	8/16/99 (Sign and Date)	Genie Bost (Print Name)
Approval and Authorization	8/16/99 (Sign and Date)	James B. Westmoreland (Print Name)

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TABLE OF CONTENTS

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1.0 THE DETERMINATION OF TECHNETIUM-99

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for Technetium-99.

2.2 GEL utilizes methods that are derived from established sources. This method is based on two source methods from the DOE: the Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP550, "Technetium-99 Analysis using Extraction Chromatography," and the DOE's Environmental Measurement Laboratory (EML) HASL 300 Procedures Manual. This method uses similar principles of radiochemical separation and counting.

2.3 Samples are oxidized to pertechnetate using hydrogen peroxide. The remaining organic material, if present in the sample, is removed by a pre-filter column containing Amberlite® XAD-7 resin. Tc-99 is then separated using a Teva® resin column. Method recovery is calculated by addition of Tc-99M.

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit (MDL): Typical minimum detectable activity (MDA) for samples analyzed for Tc-99 is 50 pCi/L or 5 pCi/g.

3.2 Method Precision: Typical relative percent difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value. Analyst training records are kept on hand in the Human Resources department.

4.0 DEFINITIONS

4.1 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.2 Type II water: Deionized water.

4.3 LIMS: Laboratory Information Management System. The database system used to store and report data.

5.0 METHOD VARIATIONS

5.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Senior Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Caution should be used when dealing with acids, and any other laboratory reagents.

6.2 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the Group Leader prior to carrying out the rest of the procedure.

6.3 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

6.4 Refer to, "Radioactive Waste Handling Procedures," (GL-EPI-S-005), for instructions on how materials are safely and properly disposed.

7.0 INTERFERENCES

7.1 Organic material in the sample can cause quenching in the liquid scintillation counting. This problem can be resolved with the Amberlite® pre-filter column.

7.2 Tritium can cause an interference at elevated levels. This interference can be eliminated by initial boiling of the sample as well as multiple rinses of the Teva® column with 0.1N HCl.

7.3 Actinide elements can cause an interference. This interference can be eliminated by performing an iron scavenge before proceeding.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 Beakers, approximately 150 mL

8.1.2 20 mL plastic scintillation vial

8.1.3 Eppendorf repeating pipettor, 1 to 5 mL

8.1.4 Watchglasses

8.1.5 Empty resin columns 10 mL capacity

8.1.6 Hot plate

8.1.7 Polypropylene centrifuge tubes

8.2 Reagents, Chemicals and Standards

8.2.1 Scintillation cocktail (Ready Gel® or equivalent)

8.2.2 Nitric acid (concentrated)

8.2.3 Hydrogen peroxide, 30% solution (concentrated)

8.2.4 0.1 M nitric acid, 3 mL concentrated acid diluted to 4750 mL with de-ionized water

8.2.5 Teva® columns

8.2.6 Amberlite® XAD-7 resin columns

8.2.7 NIST traceable Tc-99 standard

8.2.8 Tc-99M

8.2.9 Carbon Tetrachloride

8.2.10 DI Water

8.2.11 0.1 N Hydrochloric acid. 8.3 mL concentrated HCl diluted to 1000 mL with deionized water

8.2.12 1.0 M Hydrochloric acid. 166.7 mL concentrated HC1 diluted to 2000 mL with deionized water.

8.2.13 Ferric Nitrate Carrier (10 mg Fe/mL). Dissolve 14.5 g Fe(NO3)3*9 H2O in 200 mL deionized water

8.2.14 Sodium Hydroxide (10M). Dissolve 200 g NaOH in 500 mL deionized water

8.2.15 Methyl Violet Indicator (0.02% w/v)

8.2.16 8M Nitric acid. 500 mL concentrated HNO3 diluted to 1000 mL with deionized water

8.3 Instrumentation

8.3.1 Liquid scintillation counter and associated equipment

8.3.2 Gamma spectrometer and associated electronics and data reduction package

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 Water samples are collected in a plastic container and preserved with nitric acid to a pH of 2. Soil samples are collected without preservation in a suitable container.

10.0 SAMPLE PREPARATION

10.1 Measure an aliquot of sample into a beaker. For soils and other matrices weigh a representative aliquot from the homogenized sample. Use an appropriate amount of 0.01 M HNO_3 for the blank and LCS. Typically, 100 mL and 5 g are sufficient to meet client MDA requirements. Record the data on the que sheet (Appendix 1).

10.2 Add 1 mL tracer to each sample. Spike appropriate samples.

10.3 Sample matrices:

10.3.1 Aqueous samples: Add 2 mL H_2O_2. Cover samples with a watchglass and bring to a boil. Once the samples start to boil, take the covers off, turn the heat down, and let digest for one hour. Proceed to Step 10.5

10.3.2 Soil matrices: Add 10 mL H_2O_2. Digest soil samples for one hour then heat to apparent dryness. Let samples cool.

10.4 Leach soil samples using 50 mL 1.0 M HCl with occasional agitation for a minimum of one hour. Cover with a watchglass and apply low heat for two hour, agitating occasionally. Allow samples to cool. Centrifuge and pour supernate into a clean beaker.

NOTE: If high concentrations of actinide elements are suspected, go to step 10.4.1, otherwise, go to step 10.5.

10.4.1 Add 10-20 mg of ferric carrier, then add a sufficient amount of sodium hydroxide for precipitation to occur. Heat until samples reach the boiling point. Reduce heat for 10 minutes and then allow sample to settle and cool.

10.4.2 Centrifuge and pour supernate into a clean beaker. Re-dissolve precipitate in dilute acid, re-precipitate with an appropriate amount of NaOH and centrifuge. Add supernate to previous supernate.

10.4.3 Add 2-3 drops of 0.02 % w/v Methyl Violet Indicator; supernate will turn to a light shade of violet. Add 8 M HNO_3 dropwise until a light shade of blue appears.

10.5 Fill an empty column to a height of 20 mm with the pre-filter material, Amberlite® XAD-7 resin.

NOTE: If the analyst suspects the absence of organics in the sample (e.g., the sample is clear and colorless), this step may be omitted. Continue to Step 10.7.

10.6 Place a clean beaker under the column. Load the sample onto the column and collect eluant.

10.7 Load sample onto the Teva column and discard eluant. Pass the entire sample through the column. (This process usually takes 4-5 hours unless vacuum is used to aid in the transfer).

10.8 Rinse the column with 10 mL 0.1 M HCl and discard eluant. Repeat this rinse at least three times. If contaminants are suspected, several more rinses may be necessary.

10.9 Extrude the resin from the Teva column and place in a plastic scintillation vial. Add 4 mL DI water and 10 mL Ready Gel®. A gelatinous mass is formed which tends to stick to the top of the vial. Tap the vial on the lab bench to allow the gel to settle.

10.10 Adjust the quench when necessary by adding an appropriate amount of carbon tetrachloride to each vial. Shake to mix.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to "Preparation of Radioactive Standards," (GL-EPI-M-001).

11.2 Add 200 microliters of the Tc-99M to a 100 mL volumetric flask and dilute to 100 mL with DI water. Record the tracer in the Standards Log Book.

11.3 Add 1.0 mL of tracer to an empty vial. Add 0.4g Teva® resin, 3 mL H_2O and 10 mL scintillation cocktail. This vial will be used as the 100% chemical yield level.

11.4 Source materials are ordered from any of the suppliers listed below. All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in Section 19.0 of "Preparation of Radioactive Standards," (GL-EPI-M-001).

11.4.1 National Institute of Standards and Technology (NIST), Gaithersburg, Maryland.

11.4.2 Isotope Products Laboratories, Burbank, California.

11.4.3 Amersham Corporation, Arlington Heights, Illinois.

11.4.4 North American Scientific Products and Services, North Hollywood, California.

11.4.5 Analytics, Inc., Atlanta, Georgia.

12.0 INSTRUMENT CALIBRATION

12.1 Establishing a quench curve

12.1.1 Prepare a set of standards consisting of 8 to 12 standards using the same matrix and cocktail as the samples to be measured. Add approximately 10,000 DPM to each standard, cap and shake.

NOTE: Do not add any quenching agent at this point.

12.1.2 Allow the standards to dark adapt for a minimum of one hour.

12.1.3 Using an available counting program, measure the observed CPM of each of the standards to verify accurate pipetting. All standards should agree

within ± 5% of the mean. Discard any standards that do not meet this criteria.

12.1.4 Add 0 to 220 ul of carbon tetrachloride in 20 ul increments to each of the standards.

12.1.5 Recount the standards for a period of 1 minute to determine if the range of H#s covers the desired range of sample quench. If the range of the standards is not large enough, create more standards, adjusting the amount of quenching agent appropriately. Record the amount of quenching agent used for each standard on the standard preparation sheet.

12.1.6 Count the standards for a sufficient length of time to accumulate at least 10,000 counts in the highest quenched standard. Plot efficiency vs. H#.

12.2 Calibration verification

12.2.1 Count the standards against the newly established curves. The acceptance criteria being 100% ± 10% accuracy measured against a known value.

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 Please refer to "Micro-Vax 3100 Gamma Spectroscopy System Operating Procedure" (GL-EPI-I-004) for instructions concerning the Gamma Spectrometer.

13.2 Prepare the sample as outlined in Section 10.0.

13.3 Count the reference and samples to determine Tc-99M chemical recovery using either 13.3.1 or 13.3.2.

13.3.1 Count the reference and the samples for 5 minutes on the same gamma spectrometer. Divide the sample result by the reference result to determine the chemical yield.

13.3.2 Place reference and samples in a scintillation rack with the proper user and number. Note the rack number and position on the que sheet. Place the rack in the scintillation counter and count for 10,000 counts after completing Step 10.10. Save data for calculating chemical yield.

13.4 Allow the TC-99M to decay (takes 5 – 7 days).

13.5 If not already performed in Step 13.3, place samples in a scintillation rack with the proper user number. Note the rack number and position on the que sheet.

13.6 Place the rack in the scintillation counter, and allow it to dark adapt for 1 – 2 hours before counting.

13.7 Count samples for a duration that is at least sufficient to meet the CRDL and uncertainty requirements.

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 Refer to "Operation of the Beckman LS 6000 Series Scintillation Counter" (GL-EPI-I-004) for instructions concerning the scintillation counter

14.2 Refer to "Counting Room Instrument Maintenance and Performance Checks" (GL-EPI-I-010) for instructions concerning instrument maintenance.

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 The analyst will use an Excel spreadsheet to calculate the sample pCi/unit according to the following equations:

$$\text{Result (pCi/unit)} = \frac{(\text{Scpm} - \text{Bcpm})}{2.22 * E * V}$$

15.2 The counting uncertainty is calculated according to the following equation:

$$\text{Error (pCi/unit)} = \frac{1.96\sqrt{\frac{\text{Scpm}}{Tc} + \frac{\text{Bcpm}}{Tcb}}}{(2.22 * E * V * R)}$$

15.3 The method detection limit (MDA) is calculated according to the following equation:.

$$\text{MDA (pCi/unit)} = \frac{\left(2.71 + 4.66\sqrt{\text{Bcpm} * Tc}\right)}{(2.22 * E * V * Tc * R)}$$

Where:

T_c=Sample count duration
T_{cB}=Background count duration
B_{CPM}=Background counts per minute
S_{CPM}=Sample counts per minute
E=Efficiency of counting

V=Sample volume
R=Radiometric recovery

15.4 Record the information required on the Tc-99 que sheet.

15.5 Transfer applicable data from Tc-99 que sheet and instrument raw data to Tc-99 batch spreadsheet in Excel.

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification Requirements

16.1.1 Refer to "Analyst and Analytical Methods Validation Procedures" (GL-EPI-D-003), for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank should accompany each batch of samples. The reported value of the blank should be less than or equal to the contract required detection limit (CRDL).

16.2.2 A matrix spike (MS) should be run with every batch of samples. The recovery of the MS should be between 75-125%. The MS recovery is calculated as follows:

$$\text{MS Recovery (\%)} = \frac{\text{Spike (pCi/unit)} - \text{Sample (pCi/unit)}}{\text{Spike Nominal Concentration (pCi/unit}} * 100\%$$

Where:
Spike=Results of matrix spike
Sample=Results of sample without added spike
Spike Nominal Concentration=SNC:

$$\text{SNC} = \frac{\text{standard dpm} * \text{ml of spike added}}{2.22 * \text{volume in gram or liters}}$$

16.2.3 A duplicate sample should be run with every batch. The relative percent difference (RPD) between the actual sample and the QC duplicate should be less than or equal to 20% if both the sample and the QC duplicate results are greater than 5 times MDA or 100% if they are both less than 5 times MDA. The RPD should be calculated as follows:

$$RPD\,(\%) = \frac{ABS\,(DUP_1 - DUP_2)}{(DUP_1 - DUP_2)/2} * 100$$

16.2.4 A Laboratory Control Sample (LCS) should be run with every batch. The recovery of the LCS should fall between 75-125%. The LCS recovery is calculated as follows:

$$LCS_{RECOVERY} = \frac{LCS\ Result\,(pCi/unit)}{Nominal\ Concentration\ of\ LCS\,(pCi/unit)} * 100$$

16.3 Actions Required if the Quality Control Requirements are not met

16.3.1 If any of the QC criteria from 12.2.1 through 12.2.5 cannot be satisfied, the analyst should inform their Group Leader and initiate a Non-conformance Report as outlined in "Non-conformance Identification, Control, Documentation, Reporting, and Dispositioning," (GL-QS-E-004).

17.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

17.1 For Data Review, Approval and Transmittal, see "Data Review and Validation Procedures," (GL-EPI-D-003).

18.0 RECORDS MANAGEMENT

18,1 All raw data printouts, calculations, spreadsheets, and batch checklists shall be filed with the sample data for archival and review.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

19.1 Radioactive samples and material shall be handled and disposed of as outlined in "Radioactive Waste Handling Procedures," (GL-EPI-S-005).

19.2 Refer to the waste management plan for specific information on waste handling and disposal.

20.0 REFERENCES

21.1 Theresa M. Davis, Donald M. Nelson, and Emmer G. Thompson "Monitoring for Tc-99 in Borehole Waters Using an Extraction Chromatographic Resin." Radioactivity & Radiochemistry, Volume 4, No.2, 1993.

21.2 EML Procedures Manual. HASL-300, Ed. 25, 1982.

APPENDIX 1

Technetium Que Sheet

Batch #: 14123. Analyst: IDXL Automatic

Tc-99 Spike Code:_____ Vol:_____ Balance # :_____
Tc-99 LCS Code:_____ Vol:_____ Pipet #:_____
Tc-99m Tracer Code:_____ Vol:_____

Comments: Radioactive
Prep Date:_____
Initials :_____
Minimum Duedate: *2/19/99*

Sample ID	Client Description		Min RDL	Client	Aliquot (mL or g)	LCS Rack #	Tc-99m Chemical Yield
9901717-01	HSB136D		300 PCL	WSRC			
9901717-03	HSB145D		300 PCL	WSRC			
9901821-02	99-CIF-0180		300 PCL	WSRC			
9901837-07	HSB111D		300 PCL	WSRC			
9901837-08	HSB114C		300 PCL	WSRC			
9901837-09	HSB115C		300 PCL	WSRC			
9901837-10	HSB117C		300 PCL	WSRC			
9901837-11	HSB137C		300 PCL	WSRC			
9901837-12	HSB137D		300 PCL	WSRC			
9901914-03	HSB101C		300 PCL	WSRC			
9901914-06	HSB129C		300 PCL	WSRC			
9901914-07	HSB136C		300 PCL	WSRC			
9901914-08	HSB139C		300 PCL	WSRC			
9901988-01	North Mound Sewer		300 pCi/L	NFSR			
QC581015	blank	BLANK	300 pCi/L	QC			
QC581016	9901914-08	DUP	300 pCi/L	QC			
QC581017	9901914-08	MS	300 pCi/L	QC			
QC581018	lcs	LCS	300 pCi/L	QC			

Instruments Used (circle as appropriate):

LS6000 (Red) 7065155 LS6500 (Black) 7069123
LS6500 (Blue) 7067083 WALLAC (Yellow) 4040127
LS6500 (Green) 7067404 WALLAC (Pink) 2200082

Data Reviewed By:_____

STANDARD OPERATING PROCEDURE

UNCONTROLLED DOCUMENT

FOR

THE DETERMINATION OF IODINE-129

Process Owner(s):	Robert D. Timm, EPI Team Task Leader
	Wilbur B. Sigmon, III, EPI Group Leader
Technical Review by:	Barry D. Stewart, Senior Technical Specialist
Management Review by:	James B. Westmoreland, EPI Laboratory Manager
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	Heyward H. Coleman, EPI President

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TABLE OF CONTENTS

SECTION Page

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1.0 TITLE: STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF IODINE-129

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for I-129 in a variety of matrices including water, soil, charcoal canisters, milk and swipes. Iodine is separated from the sample matrix (when required) by sorption on AG-1 anion resin. Since the affinity of the resin for iodide is greater than other singly charged anions, iodide is preferentially held.

2.2 The Department of Energy (DOE) EML Procedures Manual source method number for Iodine in soils and sediments by Gamma analysis is HASL-300. For water the method code is EPA 901.1.

2.3 This revision combines two related procedures. The following EPI standard operating procedure is canceled without replacement having served its purpose:

EPI SOP "The Determination of Iodine-129 in Soil" (A-006b).

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for I-129 is 2 pCi/L or 2 pCi/g.

3.2 Method Precision: Typical relative percent difference (RPD) is less than 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value. Analyst training records are kept on file in Human Resources.

4.0 DEFINITIONS

Not applicable.

5.0 METHOD VARIATIONS

5.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Senior Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Care should be taken when handling/heating the acids used in this method.

6.1.1 Work under a hood when using concentrated acids and bases.

6.1.2 Wear gloves, laboratory coat and eye protection with side shields while in the laboratory.

6.1.3 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. Environmental Physics, Inc. (EPI) maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the laboratory. Individual sample MSDS forms provided by the clients are kept in the administrative area.

6.2 Keep hands free from moving parts of canning device and Gamma shields.

6.3 For special conditions of handling radioactive materials, refer to EPI SOP "Radioactive Material Handling Procedure" (S-004).

7.0 INTERFERENCES

7.1 High levels of I-131 can give a positive bias from 29 kev x-rays. This problem is minimized by allowing the I-131 to decay prior to gamma counting.

7.2 Samples which are acidified to pH of 2 at the time of collection may lose some iodine as gas.

7.3 High concentrations of other anions such as sulfate or phosphate can reduce the iodine sorption on anion resin.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 1x8 ion exchange resin, 50-100 mesh, Cl^- form.

8.1.2 Ion exchange columns -- 2 cm ID by ~15 cm.

8.1.3 125 mL Nalgene plastic bottles with screw caps.

8.1.4 pH paper (general purpose 0-14 range).

8.1.5 Low energy gamma ray spectroscopy system capable of resolving gamma lines in the 20-50 KeV range.

8.1.6 Plastic petri dishes, 1.2" diameter.

8.1.7 2 L beakers.

8.1.8 Laboratory stirrer and teflon coated stirring magnets.

8.2 Reagents, chemicals, and standards

8.2.1 NIST traceable standard I-129.

8.2.2 1M sodium bisulfite ($NaHSO_3$) solution (prepare by dissolving 10.46g $NaHSO_3$ per 100 mL of DI water. Prepare weekly.

8.2.3 50% NaOH solution.

8.2.4 10% HNO_3 solution.

8.2.5 Iodine carrier, 20 mg I^-/mL. Dissolve 2.62 g of KI in 100 mL of DI water. Discard if color is present.

8.2.6 0.5% $NaHSO_3$. 0.5g $NaHSO_3$ per 100 mL of DI water. Prepare fresh for each use.

8.3 Instrumentation

8.3.1 X-Ray Spectrometer.

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 Samples should be collected in a suitable container which will maintain its integrity during transportation.

9.2 Milk samples should be preserved with 10 mL of formaldehyde or by refrigeration.

10.0 SAMPLE PREPARATION

10.1 Water samples

10.1.1 Transfer a known volume to a suitable container. Add 1 mL of iodine carrier and 2 mL of 1 M $NaHSO_3$. Stir with a stir rod and allow to set for 30 minutes.

10.1.2 Adjust pH to ≈ 6.5 using 50% NaOH to raise pH, or 10% HNO_3 to lower pH. Continue to stir for several minutes.

10.1.3 Proceed to Section 13.1 for ion exchange separations.

10.2 Soil samples

10.2.1 Aliquot the sample into a tared petri dish being careful to fill the dish to the lid without overfilling. Record the sample mass on the que sheet.

10.2.2 Proceed to Section 13.2 for sample counting.

10.3 Charcoal canisters

10.3.1 If the sample consists of one canister only, proceed to Section 13.2 for sample counting.

10.3.2 If the sample contains multiple canisters, open all canisters and homogenize by placing in a soil prep can and shaking in a soil shaker for 5 minutes. Determine the total sample charcoal weight and return appropriate fractional aliquot to one of the canisters. For example, if the sample is 2 canisters and total charcoal weight is 50 mg, put 25 mg of homogenized charcoal back in the canister. Proceed to Section 13.2 for sample counting.

10.3.3 If direct counting the canister as prepared in step 10.3.2 will not allow you to analyze enough sample to meet the Required Detection Limit (RDL), prepare the sample as follows:

10.3.3.1 Place appropriate fraction of the homogenized sample into a suitable container.

10.3.3.2 Add DI water to cover the charcoal and stir on a stir plate.

10.3.3.3 Suspend the charcoal in a freshly prepared weight per volume $NaHSO_3$ solution and stir for 20 minutes

10.3.3.4 Separate the charcoal from the liquid reserve the liquid for subsequent ion exchange.

10.3.3.5 Proceed to Section 13.1 for ion exchange separation.

10.4 Swipes

10.4.1 Transfer swipe to a suitable container, add 1 mL of iodine carrier, and 20 mL of DI water. Ensure that the swipe is completely immersed in water.

10.4.2 Add 2 mL of 1 M $NaHSO_3$, stir, and allow to sit for 1 hour to leach Iodine from the swipe.

10.4.3 Remove swipe from the sample and rinse with DI water, catching the rinse in the sample container.

10.4.4 Proceed to Section 13.1 for ion exchange separations.

10.5 Milk Samples.

10.5.1 Transfer the sample to a 2 L beaker and add 1 mL of I carrier. Add a teflon coated stirring magnet and stir for 15 minutes to equilibrate iodine species.

10.5.2 Add 10 mL of AG-1 resin and continue stirring for 30 minutes. Stirring must be vigorous enough to distribute the resin throughout the sample.

10.5.3 Remove the beaker from the stirrer and allow the resin to settle for 5-10 minutes.

10.5.4 Slowly decant the milk into a second beaker, ensuring that no resin is lost. Rinse the beaker and resin with several small portions of water.

NOTE: It is not necessary that the milk be completely removed from the resin. Since no chemical yield determination is made, analytical accuracy will depend on quantitative recovery of the resin.

10.5.5 Add a second 10.0 mL portion of AG-1 resin to the beaker containing the milk and stir for 30 minutes.

10.5.6 Remove the beaker from the stirrer and allow the resin to settle for 5-10 minutes. Very carefully decant and discard the milk.

10.5.7 Rinse the beaker and resin with several portions of water, and transfer the resin to the bottle containing the first portion of resin.

10.5.8 Cap the bottle and place inside a small plastic bag. Count on the x-ray spectrometer by placing the bottle directly on the detector end cap as specified in 13.2.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

Refer to EPI SOP "Preparation of Radioactive Standards" (M-001).

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 Instrument Calibration

Refer to EPI SOP "Gamma Spectrometer Operating Instructions" (I-001).

12.2 Instrument Performance Requirements

Refer to EPI SOPs "Gamma Spectrometer Operating Instructions" (I-001), and "Instrument Maintenance and Performance Checks" (I-010).

12.3 Analysis Procedures and Instrumental Operation

Refer to EPI SOP "Gamma Spectrometer Operating Instructions" (I-001).

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 Ion Exchange Separations

13.1.1 For charcoal samples prepare ion exchange column with 20 mL wet 1x8 resin. For water and swipe samples prepare ion exchange column with 10 mL wet 1x8 resin. Rinse with DI water. No other pre-treatment is required.

13.1.2 Pass the sample through the ion exchange resin at a flow rate of ≈ 20 mL/minute. Discard the effluent.

13.1.3 Transfer the resin from the ion exchange column to an appropriate container.

13.1.4 Proceed to Section 13.2 for sample counting

13.2 Sample counting

Count on a germanium detector capable of resolving gamma lines in the 20-50 KeV range using the appropiate counting geometry. Refer to EPI SOP "Gamma Spectrometer Operating Instructions" (I-001).

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to EPI SOP "Counting Room Instrumentation Maintenance and Performance Checks" (I-010) for instructions concerning Micro-VAX 3100 instrument maintenance.

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 The instrument will report sample pCi/unit according to the following equation:

$$\text{pCi / unit} = \frac{(S_{cpm} - B_{cpm}) * decay}{2.22 * E * V}$$

15.2 Counting uncertainty is propagated according to the following equation:

$$\text{pCi / unit} = Ac * 1.96\sqrt{\left(\frac{ef\_er}{E}\right)^2 + \left(\frac{pk\_er}{pk}\right)^2 + \left(\frac{ab\_er}{A}\right)^2 + \left(\frac{sy}{100}\right)^2 + (dk)^2}$$

15.3 The minimum detectable activity (MDA) is calculated according to the following equation:

$$MDA(pCi/unit) = \frac{2.71 + (4.65 * \sqrt{B_{cpm} * T_c}) * decay}{(2.22 * E * V * T_c)}$$

where:

$$decay = \frac{1}{e^{\left(\frac{-\ln(2)T_d}{T_{1/2}}\right)}}$$

$$dk = \frac{T_{1/2}err}{T_{1/2}} * \left(\frac{\lambda Tr}{1 - e^{-\lambda Tr}} - \lambda\left(T_c + T_r\right) - 1\right)$$

And where:

Scpm	=	Sample counts per minute
Bcpm	=	Background counts per minute
E	=	Counting efficiency (decimal form)
V	=	Volume in liters,g,cfm, etc.
ef_er	=	1 sigma efficiency error (decimal form)
pk_er	=	1 sigma peak error
ab_er	=	1 sigma isotopic abundance error (decimal form)
sy	=	1 sigma systematic error
pk	=	peak area
Tc	=	Sample count time in minutes
Td	=	Time interval for radioactive decay
Tr	=	Elapsed real time in minutes
Tcpm	=	Tracer counts per minute
Tdpm	=	Tracer known disintegrations per minute
Ac	=	Sample calculated activity
T1/2	=	Isotopic half life
T1/2err	=	Isotopic half life error
λ	=	Isotopic decay constant
e	=	exponential function
ln	=	natural log function

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Refer to EPI SOP "Analyst and Analytical Method Verification Requirements" (D-002).

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank should accompany each batch of samples. The reported value should be less than or equal to the CRDL.

16.2.2 A matrix spike (MS) should be run with every batch of samples. The recovery of the spikes should fall between 75% and 125%. The recovery is calculated as follows:

$$\%\,\mathrm{Rec} = \frac{\mathrm{Spike\,(pCi\,/\,ml)} - \mathrm{Sample\,(pCi\,/\,ml)}}{\mathrm{Spiked\ Amount\,(pCi\,/\,ml)}} * 100$$

16.2.3 A duplicate sample should be run with every batch of samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$\mathrm{RPD} = \frac{\mathrm{High\ Sample\ (pCi\,/\,mL)\ -\ Low\ Sample\ (pCi\,/\,mL)}}{\mathrm{Average\ (pCi\,/\,mL)}} * 100$$

16.2.4 A laboratory control sample (LCS) should be run with every batch of samples. The recovery of the LCS should fall between 75% - 125%. The recovery is calculated as follows:

$$\%\,\mathrm{Rec} = \frac{\mathrm{LCS\ (pCi\,/\,mL)}}{\mathrm{LCS\ Amount\ (pCi/mL)}} * 100$$

16.3 Actions required if the Quality Control requirements are not met.

If any of the QC criteria from 12.2.1 through 12.2.5 cannot be satisfied, the analyst should inform their group leader and initiate a Nonconformance Report as outlined in GEL SOP "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

NOTE: Some clients may have more or less stringent requirements.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

17.1 Data Recording.

Record the applicable information on the que sheet.

17.2 Data Review.

Refer to EPI SOP "Data Review and Validation Procedures" (D-003) for instructions concerning the validation of analysis and analytical data.

17.3 Data Reporting.

The data is automatically transferred to the LIMS at the completion of analysis.

18.0 RECORDS MANAGEMENT

All raw data printouts, calculations, spreadsheets, and batch checklists shall be filed with the sample data for archival and review.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

19.1 Radioactive samples and material waste shall be handled as outlined in SOP EPI "Radioactive Waste Handling Procedures" (S-005).

19.2 Refer to EPI SOP "EPI Laboratory Waste Disposal and Emergency Instructions" (S-011) for specific information on waste disposal.

20.0 REFERENCES

20.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

20.2 EML Procedures Manual, HASL-300, 1982.

STANDARD OPERATING PROCEDURE

FOR

UNCONTROLLED DOCUMENT

THE DETERMINATION OF RADON-222 IN WATER

Process Owner(s):	Russell L. Moser, EPI Group Leader
Technical Review by:	James B. Westmoreland, EPI Laboratory Manager
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	Heyward H. Coleman, EPI President

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERINATION OF RADON-222 IN WATER

2.0 METHOD OBJECTIVE AND APPLICABILITY

This standard operating procedure provides the necessary instructions to conduct the analysis for Radon-222 in water.

3.0 INTERFERENCES

Rn-222 has a half life of 3.82 days. The elapsed time from sampling to counting should be minimized to reduce the loss of radon via radioactive decay.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 Follow all safety procedures when handling any chemical substance.

4.2 All radioactive materials are handled according to EPI SOP "Radioactive Material Handling Procedure" (S-004).

5.0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment:

5.1.1 Liquid scintillation vials (20mL glass)

5.2 Reagents, Chemicals, and Standards:

5.2.1 Scintillation cocktail. Packard Optifluor O or equivalent

5.2.2 NIST traceable Ra-226 standard

5.2.3 10 mL graduated cylinders

5.3 Instrumentation:

5.3.1 Liquid scintillation analyzer.

6.0 SAMPLE COLLECTION & PRESERVATION

Samples should be collected with minimum aeration in two 40 mL volatile bottles. No headspace should exist in the sample vials. The samples should be kept at 4° ±2°C until analysis. Samples should be counted within 7 days of sampling

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to EPI SOP "Beckman LS-6000/6500 Operating Procedures" (I-004) for instructions concerning instrument maintenance.

8.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

8.1 All standards used in this method must be NIST traceable.

8.2 Dilution of standards should be noted in standard logbook in keeping with accepted procedure, EPI SOP "Preparation of Radioactive Standards" (M-001). The weight of the standard to be diluted and diluting solutions should be noted in the standards logbook. The weight should be taken on a balance of at least four places and calculation of the new standard activity carried out and noted in appropriate significant figures.

8.3 A "blank" water should be used as the batch blank.

8.4 A duplicate and spiked sample should be made on the same sample in a sample batch.

8.5 A laboratory control sample should be run with each batch.

9.0 OPERATING PROCEDURES

9.1 Sample Preparation Techniques

9.1.1 Add 10 mL of Optifluor-O cocktail to a 20 mL glass liquid scintillation vial. Slowly add 10 mLs of sample to the vial using a 10 mL graduated cylinder.

NOTE: It is important NOT to use an air displacement pipet to add 10 mL of sample to the liquid scintillation vial. Using an air displacement pipet may result in loss of Radon gas.

9.1.2 Shake the vial vigorously for several seconds and allow the vial to sit in the dark for 4 hrs.

9.1.3 Count the sample long enough to reach CRDL. See the operating manual for the Beckman LS-6000LL/6500 for analyzing samples for Rn-222.

9.2 Instrument Calibration

9.2.1 Prepare two blanks and seven Ra-226 standards of certified dpm. Carry the standards and blanks through the procedure (section VIII). Store the prepared Ra-226 standards for at least 25 days to achieve secular equilibrium with Rn-222 and daughters. Count the standards on the LSC and determine the method cpm/pCi conversion factor.

9.3 Instrument Performance Requirements

9.3.1 Instrument performance requirements: Refer to EPI SOP "Beckman LS-6000/6500 Operating Procedures" (I-004) for guidance concerning instrument performance requirements.

9.3.2 Documentation of instrument performance: Refer to EPI SOP "Operation of the Chemcheck Kinetic Laser Phosphorimeter" (I-010) for guidance concerning instrument performance documentation.

9.4 Analysis Procedures and Instrumental Operation

9.4.1 Refer to EPI SOP SOP "Beckman LS-6000/6500 Operating Procedures" (I-004) for guidance concerning analysis procedures and instrument operation for Rn-222.

10.0 CALCULATIONS AND DATA REDUCTION METHODS

10.1 The analyst will report sample pCi/ml according to the following equations:

10.1.1 $$\text{pCi/ml} = \frac{S_{cpm} - B_{cpm}}{2.22 * E * V * A_t}$$

10.1.2 Decay correction:

$$(A_t) = \frac{e^{-0.693(d)}}{3.82}$$

10.2 Rn-222 Factor = 2.22 * E

10.3 Counting uncertainty is calculated according to the following equation:

$$pCi / ml = \frac{1.96\sqrt{\frac{Scpm}{Tc} + \frac{Bcpm}{TcB}}}{2.22 * E * V}$$

10.4 The method detection limit (MDA) is calculated according to the following equation:

$$MDA = \frac{2.71 + 4.66\sqrt{cpm_{bkg} * Tc}}{2.22 * E * V * Tc}$$

where:

S_{cpm} = Sample counts per minute
B_{cpm} = Background counts per minute
E = cpm/dpm conversion factor
V = Sample volume (mL)
C = Sample count time (min)
d = Time in days from sampling to counting
A_t = Decay correction factor
T_C = Total count time

11.0 DATA RECORDING, REVIEW AND REPORTING

11.1 Data Recording

11.1.1 Record the information required on the que sheet (Appendix 1).

11.2 Data Review

11.2.1 Refer to EPI SOP "Data Review and Validation Procedures" (D-003) for instructions concerning the data review process.

11.3 Data Reporting.

11.3.1 The analyst will take the applicable Rn-222 spreadsheet and enter the data into LIMS. The following information should be included: analyst's initials, date and time the sample was counted, sample results and accuracy in pCi/mL.

12.0 QUALITY CONTROL REQUIREMENTS

12.1 Analyst and Method Verification Requirements

12.1.1 Refer to EPI SOP "Analyst and Analytical Methods Validation" (D-002) for information concerning analyst and method verification.

12.2 Method Specific Quality Control Requirements

12.2.1 A matrix spike(ms) should be run with every batch of samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows.

$$\text{Rec} = \frac{\text{Spike(pCi / ml)} - \text{Sample(pCi / ml)}}{\text{Nominal Concentration(pCi / ml)}} * 100$$

12.2.2 A duplicate should be run with every batch of samples. If both the sample and duplicate values (pCi/mL) are greater than 5 times the CRDL, the allowable RPD is less than or equal to 0%. If either the sample or the duplicate value (pCi/mL) is greater than or equal to the CRDL and less than 5 times the CRDL, the allowable RPD is less than or equal to 100%. The RPD is not applicable if either sample or duplicate values are less than the CRDL. The RPD is calculated as follows:

$$\text{Duplicate RPD} = \frac{\text{High Result} - \text{Low Result}}{\text{Average}} * 100$$

12.2.3 The reported value should be less than or equal to the CRDL.

12.2.4 The LCS recovery should fall between 75 and 125%. The recovery is calculated as follows:

$$\text{Recovery} = \frac{\text{LCS (pCi/mL)}}{\text{Nominal Concentration (pCi/mL)}} * 100$$

12.3 Actions Required if the Quality Control Requirements Are Not Met

12.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non-conformance report as outlined in GEL SOP " Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

NOTE: Clients may require more or less quality control requirements.

13.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

13.1 Raw data and associated documentation will be stored in binder books or in files and kept on hand for any subsequent reviews necessary. The records should be kept on hand for a length of time not less than 1 year.

13.2 Data generated by the instrument will be backed up during routine software backup.

13.3 Quality control charts are kept on the Micro-Vax 3100 as outlined in EPI SOP "VAX/VMS Quality Control Software Program" (I-008).

14.0 LABORATORY WASTE HANDLING AND DISPOSAL

Radioactive material is handled and disposed of as outlined in EPI SOP "Laboratory Waste Disposal and Emergency Instructions" (S-011).

15.0 REFERENCES

15.1 The Determination of Radon in Drinking Water. EPA EERF method.

15.2 Beckman LS-5000TD Liquid Scintillation System Operation Manual. May 1988.

APPENDIX 1

Radon-222 in Water Que Sheet

comment: none

Batch # 34652 Prep Date: Initials: _____
LCS Isotope:_____ LCS Code: Vol:_____
Nom Conc LCS:_____
Batched To: Alice Whitlock Instrument used: (circle one) LS6000 sn=7065155 LS6500 sn=7067083

Sample ID	*Sample Description*	*Sample Mass (mls)*	*Sampling Date*	*Sampling time*
9308009-01	2409-10824		7/27/93	1100
9308009-02	2409-10825		7/27/93	1200
9308009-03	2409-10826		7/27/93	1250
9308009-04	2409-10827		7/27/93	1445
9308009-05	2409-10828		7/27/93	1535
9308009-06	2409-10829		7/27/93	1730
9308009-07	2409-10830		7/27/93	1800
9308009-08	2409-10831		7/27/93	1825
9308009-09	2409-10832		7/27/93	2100
9308009-10	2416-10852		7/28/93	0940
9308009-11	2416-10853		7/28/93	1030
9308009-12	2416-10854		7/28/93	1210
9308009-13	2416-10855		7/28/93	1320
9308009-14	2416-10856		7/28/93	1420

Data Reviewed By: _____

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STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF

RADIUM-226

UNCONTROLLED DOCUMENT

(GL-EPI-A-008 Revision 3)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of the General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

Process Owner	9/29/99 (Sign and Date)	Tim C. Winters (Print Name)
Technical Review	10/12/99 (Sign and Date)	Richard M. Kinney (Print Name)
Quality Review	10/15/99 (Sign and Date)	Robert L. Pillano (Print Name)
Approval and Authorization	10/15/99 (Sign and Date)	James B. Westmoreland (Print Name)

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF RADIUM-226

2.0 METHOD OBJECTIVE AND PURPOSE

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for Radium-226 in various matrices.

2.2 General Engineering Laboratories, Inc. (GEL) utilizes methods that are derived from established sources. This method is based on the source procedures manual EPA 600 4-80-032 "Prescribed Procedures for Measurement of Radioactivity in Drinking water" August 1980 Method 903.1 and uses similar principles of radiochemical concentration and counting.

3.0 SUMMARY

Solid matrices are decomposed by digestion with hydrofluoric acid and nitric acid. The digestate is evaporated to dryness and diluted to known volume with nitric acid solution. A stream of helium gas is used to initially remove radon from a water sample or solid sample digestate. The sample is then sealed and radon is allowed to ingrow. The radon, which is supported entirely by Ra-226 in the sample, is then purged with helium and trapped on a liquid nitrogen cold trap. The trap is sealed and warmed. The radon is then transferred by vacuum to a Lucas cell and counted after three hours in the cell.

NOTE: The analysis of samples for Ra-226 content by Rn-222 emanation is very specific using this procedure, and the separation of radium from other elements is not required. Sample losses can occur only as the result of improper sample transfer. Due to the specific nature of Ra-226 measurement by this method, the use of stable barium carrier or radioactive Ba-133 tracer for yield monitoring is not required.

4.0 SAMPLE COLLECTION & PRESERVATION

Samples may be collected in plastic bottles and preserved with nitric acid to pH • 2.

5.0 INTERFERENCES

No interferences have been encountered.

6.0 APPARATUS

6.1 De-emanation system with cold trap

6.2 Liquid nitrogen Dewars (1 liter and 650 mL sizes)

6.3 1 liter plastic bottles

6.4 Aeration stones

6.5 Amber latex tubing

6.6 Small tubing clamps

6.7 Radon flask counter with scalar

6.8 Lucas cells

6.9 Teflon beakers, 100 mL

6.10 HI-pore diffusers

6.11 Graduated cylinder 500 mL

7.0 REAGENTS

7.1 Type II deionized water

7.2 NIST traceable Ra-226 standard

7.3 Concentrated nitric acid

7.4 Boric acid, granular, A.C.S. grade

8.0 PROCEDURE

8.1 Solid matrices

8.1.1 For analyses that require sample dissolution, digest solid samples as detailed in GL-EPI-E-A-015. Alternatively, Ra-226 analysis in solid matrices can be completed as detailed in the EPI Gamma Spectroscopy Procedure, GL-EPI-E-A-013.

8.1.2 Transfer sample digestate to a 100 mL Teflon beaker and evaporate to dryness on a hotplate. Add 5 mL of conc. nitric acid and add approximately 0.5 grams of solid boric acid. Evaporate to dryness on a hotplate.

8.1.3 Dissolve the sample residue in 5 mL conc. nitric acid and transfer to a labeled de-emanation bottle. Dilute to 500 mL with DI.

8.1.4 Proceed to Step 8.2 of this procedure.

8.2 Water samples

8.2.1 Determine the pH of each sample using a pH strip. If the pH is > 2, add conc. nitric acid to obtain a pH • 2 and store the sample for a minimum of 16 hours.

8.2.2 Transfer 500 mL of samples to a de-emanation bottle and proceed to Step 8.3 of this procedure.

8.3 Connect the inlet line (V-3 on Fig. 1) from the He regulator to the sample bottle and connect the outlet line (V-4) from the sample to the hood vent line. To remove radon from the sample, purge for 30 minutes with helium at a flow rate of 0.4 lpm by adjusting valve 1.

8.4 At the end of the 30 minute degassing, seal the sample by connecting the inlet and outlet lines together. Record the END date and time of the initial degassing on the sample Que sheet. Allow the sample to ingrow for a minimum of three days.

NOTE: Before proceeding to Step 8.5 it is advisable to begin acquiring daily source checks on the Lucas cells that will be used during the degassing process. Refer to "Ludlum Model 2000/1000 Lucas Cell Counter Operating Instructions" (GL-EPI-E-I007) for instrument operating instructions.

Counting times for samples and Lucas cell backgrounds should be optimized to achieve required detection limits. Typically, assuming a Lucas cell background of 0.200 cpm and a cell efficiency of 2.0 cpm/dpm, the following count times are required in order to meet the stated method detection limits:

Required Detection Limit	Sample Size (liters, grams)	Count Time
0.1 pCi/g	1.0 gram	200 minutes
0.3 pCi/g	1.0 gram	20 minutes
0.5 pCi/g	1.0 gram	15 minutes
1.0 pCi/g	1.0 gram	15 minutes
0.1 pCi/L (g)	0.5 liters/grams	400 minutes
0.3 pCi/L (g)	0.5 liters/grams	60 minutes
0.5 pCi/L (g)	0.5 liters/grams	30 minutes
1.0 pCi/L (g)	0.5 liters/grams	15 minutes
0.3 pCi/L (g)	0.25 liters/grams	200 minutes
0.5 pCi/L (g)	0.25 liters/grams	90 minutes
1.0 pCi/L (g)	0.25 liters/grams	30 minutes

8.5 Fill the 1 liter and 650 mL Dewars with liquid nitrogen. Lift the platform holding the 650 mL Dewars to completely submerge the cold trap in LN. Allow the cold trap to equilibrate before proceeding.

8.6 Connect the sample to lines V-3 and V-4 and ensure that the connections are secure. Turn valves V-3 and V-4 to the sample position. (i.e. pointing to the right). Bubbles should be visible as the helium is now purging the radon into the cold trap. Allow the helium to flow for 15 minutes. Afterwards, record the date and time of the sample degassing.

8.7 After 15 minutes, turn valves V-5 and V-6 to the closed position (i.e. pointing down). This will seal the cold trap that now contains the sample radon. Turn valves V-3 and V-4 to the bypass position.

8.8 Connect the Lucas cell to the system with the stop cock in the closed position. Pull a vacuum on the system (not including the Lucas cell) by turning on the vacuum pump and opening valve V-7. With the cold trap still under LN quickly open and close valve V-6 to remove excess helium.

8.9 Ensure that valves V-5 and V-6 are closed. Pull a vacuum on the system and the Lucas cell by turning on the vacuum pump, opening valve V-7 and opening the Lucas cell stop cock.

8.10 Close valve V-7 and turn off the vacuum pump. Check the system for leaks by observing the vacuum gauge for 30 seconds. The vacuum gauge should hold at the vacuum at the -25 to -30 vac range. If the vacuum does not hold, notify your supervisor.

8.11 Remove the LN Dewar and gently warm the trap with a hot air gun. The trap should feel warm to the touch before proceeding.

8.12 Open valve V-6 to the vacuum system. Place hands on valves V-5 and V-6. Keep eyes on the vacuum gauge. Open valve V-5 and close valve V-6 just before the vacuum pressure goes to zero.

NOTE: The pressure will drop fast so be alert to the clockwise direction required to close valve V-6.

NOTE: If the zero (atmospheric pressure) is slightly passed the sample may still be counted. The analyst is trying to avoid creating a large positive pressure within the Lucas cell, which may cause the cell to leak or rupture.

8.13 Allow the radon to equilibrate in the line for 30 seconds then close the stopcock on the Lucas cell. Disconnect the Lucas cell and allow the radon daughters to equilibrate for a minimum of three hours before counting the cell. Place the cell in the counter for 5 minutes before beginning the sample count.

8.14 Count each sample for a minimum of 15 minutes. Record the date and time the

count is started, the count time and the gross counts observed.

8.15 The Lucas cell should be cleaned as soon as possible after the sample count is completed. The Lucas cell cleaning procedure is detailed in Steps 8.15 through 8.19 of this procedure.

8.16 Turn on the helium flow with valves V-3 and V-4 in the bypass position. Open valves V-5 and V-6 and observe that helium is flowing through the system. Close valve 4.

8.17 Connect the Lucas cell to the de-emanation system. Connect the pump outlet line to the hood vent line and turn on the pump. Open the Lucas cell stop cock and evacuate the cell.

8.18 Close valve 6 and then open valve 4 to flush the cell with helium. Repeat this vacuum then flush cycle 20 times for low activity, 40 times for high activity counts.

8.19 Store the cell under a slightly positive helium pressure until the next use. The cell should be stored for a minimum of three hours prior to use for sample analysis.

9.0. QUALITY CONTROL

9.1 A matrix spike(MS) should be run with every batch of 20 samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\%\text{Rec} = \frac{spike(pCi/l) - sample(pCi/l)}{spike\ concentration(pCi/l)} * 100$$

9.2 A duplicate sample should be run with every batch of 20 samples. The relative percent difference (RPD) between the duplicate (dup) and the sample should be less than or equal to 20%. The RPD is calculated as follows:

$$\text{RPD} = \frac{high\ dup(pCi/l) - low\ dup(pCi/l)}{Average(pCi/l)} * 100$$

9.3 A method blank should accompany each batch of 20 samples. The reported value should be less than or equal to the CRDL (contract required detection limit).

9.4 A laboratory control sample (LCS) should be run with each batch of samples. The recover of the LCS should fall between 75-125%. The recovery is calculated as follows:

$$\%\text{Rec} = \frac{Observed(pCi/l)}{Known(pCi/l)} * 100$$

10.0 CALCULATIONS

The spreadsheet will calculate the Ra-226 activity in pCi/L by the following equation:

$$Ra-226(pCi/l) = \frac{cpm_s - cpm_b}{2.22 * V * E * I_1 * I_2 * I_3}$$

$$\text{Uncertainty} = \frac{1.96 * \sqrt{\frac{cpm_b + cpm_s}{ct}}}{2.22 * V * E * I_1 * I_2 * I_3}$$

Lower limit of detection (lld):

$$MDA = \frac{2.71 + 4.66 * \sqrt{cpm_{bkg} * T_c}}{(2.22 * E * V * I_1 * I_2 * I_3 * T_c)}$$

Where:
CPM_S= gross count rate (counts per minute) at the alpha detector voltage plateau.
CPM_b= background count rate for the cell used for counting.
V = volume of sample aliquot (l)
2.22 = conversion factor from dpm/pCi
E = counting efficiency of the cell, detector and de-emanation system.
$I_1 = 1-_E-\lambda+t_1$ where λ = decay constant for radon-222 ($0.181d^{-1}$) and T_1= time between initial and final de-emanation is days.
$I_2 = {}_E-\lambda+t_2$ where λ = decay constant for radon-222 in hours = 0.00755 hr. and ($0.181d^{-1}$) and t_2= delay time in hours between 2nd de-emanation and sample counting.
$I_3 = 1-_E-\lambda+ t_3/t_3$ where λ = decay constant for radon-222 ($0.181d^{-1}$) and T_3= sample count time in minutes.
Tc = Sample count time in minutes

11.0 CALIBRATION OF LUDLUM DETECTOR AND LUCAS CELLS

11.1 Ludlum Model 1000 and 2000 operating voltage and plateau generation and standard deviation:

11.1.1 Place a sealed lucas cell Ra-226 source of sufficient activity on the detector 5 minutes before counting. Set the front panel discriminator to 50 volts. Count the source for 0.1 minutes and record the counts.

11.1.2 Step the front panel discriminator up in 50 volt increments and acquire counts for 0.1 minutes at the increasing voltages up to 2000 volts and record counts. Plot the gross counts on the y axis and the voltage on the x axis and determine the "knee" of the plateau.

11.1.3 The operating voltage should be selected at 25 – 50 volts above the "knee".

11.1.4 Put a copy of the plateau for model 1000 and 2000 scaler/radon flask counter in the Ra-226 Calibration File.

11.1.5 To determine the control limits (standard deviation), place the sealed lucas cell that was used for the plateau on the detector. Do twenty 0.1 minute counts and record each count.

11.1.6 Put a copy of the counts and calculation of standard deviation in the Ra-226 Calibration File.

11.2 Calibration, cell constant, efficiency and verification of the lucas cell:

11.2.1 Determine what cells need to be calibrated. Either a year from the last calibration or new lucas cells. Give yourself at least 3 weeks before old calibration has expired.

11.2.2 Each lucas cell needs to be given a two or three digit number or old numbers can be reused. With the first number to each cell will be the detector it goes to. (For example, 120 will go into detector 1, 220 will go into detector 2.) Each lucas cell will have one detector it can be counted on.

11.2.3 A 15-minute background count will be performed on each cell before every calibration and verification run and each count will be recorded in the logbook.

11.2.4 Each counting cell is calibrated by spiking a 500 mL DI-water sample with a known dpm of Ra-226 activity. The sample is carried through the entire procedure (refer back to Section 8.3). This step is performed 3 separate times to each cell. Record each count.

11.2.5 Put information from the three runs in an excel spreadsheet to calculate cell constant, average and standard deviation. Standard deviation needs to be about 10 %. Put the Ra-226 cell constant spreadsheet in the Calibration File.

11.2.6 Each counting cell will be verified by spiking 500 mL of DI-water. Then refer back to Section 8.3. Acceptance criteria is 100% ± 10%.

11.2.7 After processing verification, put the spreadsheet into the Ra-226 Calibration File.

11.2.8 Go into CELLEFF File and change the old cell efficient to the new cell efficient.

12.0 REFERENCES

12.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, USEPA, Method 903.1, August 1980.

12.2 Friedlander, G., J. W. Kennedy, and J. Miller, Nuclear and Radiochemistry, John Wiley and Sons, Inc., New York, NY, 1964.

12.3 Youden, W. J. and F. J. Massey, Jr., Introduction to Statistical Analysis, 3rd edition, McGraw-Hill, 1969.

12.4 Mathieu, G.G., Biscaye, P.E., Lupton, R.A. "A System for Measurement of Rn-222 at Low Levels in Natural Waters." Health Physics, Vol. 55, No.6, pp. 989-992. 1988.

12.5 Key, R.M., Brewer, R.L., Stockwell, J.H., Guinasso, N.L., Schink, D.R., "Some Improved Techniques for Measuring Radcn and Radium in Marine Sediments and Seawater. Marine Chemistry, pp. 251-264. October 30, 1978.

12.6 Special thanks to Dr Bill Burnette and his associates at Florida State University for their help in building the radon de-emanation system.

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STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF RADIUM-228

IN WATER

UNCONTROLLED DOCUMENT

(GL-EPI-A-009 Revision 3)

HARD COPY IN ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories, Inc. and may not be specifically applicable to the activities of other organizations.

Heather G Anderson	7/13/99	Heather G. Anderson
Process Owner	(sign & date)	(print name)
Richard M. Kinney	7/29/99	Richard M. Kinney
Technical Review	(sign & date)	(print name)
Janie B Bost	7/29/99	Janie G. Bost
Quality Review	(sign & date)	(print name)
James B Westmoreland	7/29/99	James B. Westmoreland
Approval and Authorization	(sign & date)	(print name)

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Table of Contents

1.0 TITLE: STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF Ra-228 IN WATER

2.0 METHOD OBJECTIVE AND APPLICABILITY

This standard operating procedure provides the necessary instructions to conduct the analysis for Radium-228 in water.

3.0 INTERFERENCES

3.1 When converting barium sulfate to the carbonate it is important to remove excess sulfate or the potassium carbonate will be consumed by the reaction:

$$K_2CO_3 + H_2SO_4 \text{ --> } K_2SO_4 + H_2O + CO_2$$

3.2 Samples with high strontium-90 may cause interference in the Ac-228 beta count. This problem occurs because of Y-90 present that may follow the Ac-228 chemically. It is best to confirm high Ra-228 (i.e. greater than 5 pCi/L) by gamma spectroscopy.

3.3 Samples with elevated radio lead may cause interference in the Ac-228 beta count. It is best to confirm high Ra-228 (i.e. greater than 5 pCi/L) by gamma spectroscopy.

3.4 Th-234 would potentially interfere with Ac-228 beta counting. Its presence could be confirmed with a decay curve analysis.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

Care should be taken when handling concentrated acid as contact with fumes or liquid may cause severe burning of body tissue.

5.0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment

5.1.1 Pyrex watch glasses

5.1.2 200 mL beakers (8)

5.1.3 Centrifuge (capable of maintaining 2000 rpm with 8 ea 50 mL centrifuge tubes)

5.1.4 Pyrex 50 mL centrifuge tubes (29x113 mm)

5.1.5 Pyrex 1000 mL beakers

5.1.6 Pyrex stirring rods

5.1.7 700 watt microwave

5.1.8 Stainless steel planchets

5.1.9 Electric hot plates

5.1.10 Teflon Beakers

5.2 Reagents, Chemicals and Standards

5.2.1 Ba-133 standard

5.2.2 EPA or NIST traceable Ra-228 standard

5.2.3 Barium carrier (16 mg Ba/mL). Dissolve 30 grams reagent grade $BaCl_2$ x $2H_20$ in 1 L DI water.

5.2.4 Concentrated. sulfuric acid

5.2.5 0.5M nitric acid. Dilute 63 mL conc. nitric acid to 2000 mL of DI water.

5.2.6 0.5 M nitric acid

5.2.7 3M HCl. Dilute 500 mL of conc HCl to 2000 mL DI water.

5.2.8 50%(w/w) potassium carbonate

5.2.9 Concentrated nitric acid

5.2.10 .095M HNO_3. Dilute 3.0 mL con. HNO_3 to 500 mL DI water in volumetric flask

5.2.11 0.35 M HNO_3. Dilute 11.0 mL con. HNO_3 to 500 mL DI water in volumetric flask

5.2.12 Acetic acid, glacial

5.2.13 1M citric acid. Dissolve 192.13 g citric acid anhydrous powder in 1L DI water

5.2.14 1.5M Ammonium sulfate. Dissolve 200 g ammonium sulfate in 1L DI water

5.2.15 8M HNO_3. Add 500 mL conc. HNO_3 (16M HNO_3) to 400 mL DI water, allow to cool, then dilute to 1000 mL with DI water

5.2.16 NaOH 6M. Cautiously add 120 g NaOH pellets to approximately 300 mL water. When cool, dilute to 500 mL

5.2.17 Sodium Carbonate, 0.75M. Add 79.5g Na_2CO_3 to 1000 mL with DI water

5.2.18 Deionized water (DI water)

5.2.19 Phenolphthalein Solution

5.2.20 pH 10 DI water. Add 6M NaOH dropwise to DI water until a pH of 10 is achieved. Check with pH strip.

5.2.21 Conc. HF

5.3 Instrumentation

5.3.1 Gross Alpha/Beta Proportional Counting System

5.3.2 Gamma Spectrometer and associated electronics and data reduction package.

6.0 SAMPLE COLLECTION & PRESERVATION

6.1 Water samples should be collected in 1/2 gallon plastic bottles and preserved with concentrated nitric acid to pH $\leq$2.

6.2 If sample received w/pH>2, analyst should modify the sample to pH2, mix and allow to sit overnight before proceeding.

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to EPI SOP "Micro-Vax 3100 Gamma Spectrometer Operating Procedure" (I-001).

7.2 Refer to EPI SOP "HT-1000 Gross Alpha/Beta Counter Operating Procedure" (I-002).

7.3 Refer to EPI SOP "LB-4100 Gross Alpha/Beta Counter Operating Procedure" (I-006).

7.4 Refer to EPI SOP "Counting Room Instrument Maintenance and Performance Checks" (I-010).

8.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

Refer to EPI SOP "Preparation of Radioactive Standards" (M-001) for instructions concerning the preparation of standard solutions.

9.0 OPERATING PROCEDURES

9.1 Sample Preparation Techniques for Gas Flow Proportional Counting

9.1.1 Measure an appropriate aliquot of water sample into a beaker. Add 0.1 mL of Ba-133 tracer and 1.0 mL of stable barium carrier, 10 mL Acetic Acid and 5mL citric acid. Record the sample volume and standard spike information on the que sheet (Appendix 1).

9.1.2 Add 0.1 mL of Ba-133 tracer to 5 mL of 0.5M nitric solution in a 20mL liquid scintillation vial. This solution will serve as the reference for yield determination.

9.1.2.1 Add 0.1 mL Ba-133 tracer to 5 mL of 0.095 mL nitric solution in scintillation vial. This solution is another reference.

9.1.3 Add 10 mL ammonium sulfate then stir well. Cover with watchglass and let sit overnight.

9.1.4 Decant the clear supernate then add the remaining precipitate in a 50 mL pyrex centrifuge tube and centrifuge for 10 minutes at 4000 RPM.

9.1.5 Decant centrifuge tube and remove excess sulfate by washing the precipitate with DI water from a wash bottle.

9.1.6 Centrifuge and decant the DI water wash. Check the pH of the wash solution with a pH strip.

9.1.7 Repeat the washing as necessary until a pH of 6 is observed.

9.1.8 Add 1 mL of 50% K_2CO_3 to the precipitate making sure the precipitate is completely submerged in the solution.

9.1.9 Heat in water bath swirling occasionally for 4 hours.

9.1.10 Wash the dry slurry with 10-25 mLs DI water centrifuge and decant the supernate. Check the pH of the wash solution it should initially be around 12. If a high pH is not observed consult the appropriate group leader.

9.1.11 Wash the precipitate with 15 mLs of DI water, centrifuge and decant. Repeat washings until the pH of the wash solution is approximately 7. This will require 1-3 more washes.

9.1.12 Dissolve the precipitate in 5 mL of 0.095M HNO_3 or 0.5M HNO_3 solution. Gentle heating may be required. Centrifuge the solution and decant into a 20 mL scintillation vial.

9.1.13 Count the reference vial from step 9.1.2 or 9.1.2.1 for 5 minutes prior to counting samples on a gamma spectrometer.

9.1.14 Count the sample on the gamma spectrometer for 5 minutes. See the gamma VAX operating procedure for operating the gamma spectrometer (EPI SOP I-001). The samples and reference should be given a sample identification number, an identical reference date and a counting geometry of "can" or "2L_MB". Divide the sample result by the reference result to determine the chemical yield.

9.1.15 Allow the sample to ingrow for at least 30 hrs from the time the DI water was added to the centrifuge tubes after removing from bath or from time sodium carbonate was added in Step 9.3.3.

NOTE: The following steps should be performed within a 10 hour time frame to avoid decay of unsupported Ac-228 with a half-life of 6 hours.

9.1.16 Prerinse a TRU-SPEC^{tm} column with 10 mL of 0.5M nitric acid.

9.1.17 Load the sample (5 mL) onto the column. Rinse with an additional 8 mL of 0.5 M nitric acid. Add nitric acid in 2 mL increments allowing previous volume to completely drain. Note the date and time on the que sheet (Appendix 1). Collect the effluent (Ra-228) into labeled centrifuge tubes and save in the event reanalysis is required.

9.1.18 Elute the Ac-228 with 5 mL of 3M HCl into labeled plastic dispo cup or plastic centrifuge tube.

9.1.19 Add 200 µL of 500 ppm Ce solution to the samples and swirl.

9.1.20 Add 1 mL of concentrated HF, swirl and allow to stand for 30 minutes.

9.1.21 Place a 0.1 µm metricel filter on a 25 mm filtering apparatus.

9.1.22 Rinse the filter and funnel under vacuum with 5 mLs of 80% ethanol and then 5 mLs of type II water. With minimum delay, add the sample to the filtering apparatus and rinse the beaker several times into the funnel with type II water. Complete the filtering by adding 5 mLs of 80% ethanol.

9.1.23 Label a 29 mm flat planchet with the applicable laboratory number. Place glue on the planchet and carefully place the filter (precipitate side up) on the glue. Care should be taken to make the filter as flat as possible on the planchet. Dry the filter under a heat lamp.

9.1.24 Place the disk in a 2 inch planchet and count in a gas flow proportional counter for a time duration to meet the contract required detection limit and uncertainty.

9.1.25 Results above 5 pCi/L are reeluted through LN resin.

9.1.26 If result is above 5 pCi/L, the sample is reeluted on LN spec resin to verify the data. If the result of the reelution is above 5 pCi/L and within 40% of the first result, the second result is reported. If after the reelution the result is not less than 5 pCi/L and not within 40%, another reelution is performed and will repeat until results verify or the activity is less than 5 pCi/L.

9.1.27 Any sample labeled radioactive or has known interferences is loaded on a LN resin column.

9.2 Sample Preparation Techniques for LN Spec

9.2.1 Prerinse a LN Spec column w/10mL 0.095M HNO_3

9.2.2 Load 5mL of sample onto the column then add additional 5mL 0.095M HNO_3 and record time. Rinse with additional 12 mL 0.095M HNO_3 by adding in 3mL increments allowing previous volume to completely drain. Collect the effluent into a labeled centrifuge turbe and save in the event reanalysis is required. Sample in vial that is used to gamma count also has 5mL of 0.095M HNO_3

9.2.3 Elute the Ac-228 with 10mL of 0.35M HNO_3 into labeled plastic centrifuge tube

9.2.4 Follow same procedure from 9.1.21

9.3 Alternate Procedure for Sample Preparation

9.3.1 Transfer an aliquot of water sampe to an approprate beaker and record volume on the que sheet (Appendix 1). Add several drops of phenolphthalein solution and add M NaOH to a light purple color. Add 1.0 mL barium carrier, 0.1mL Ba-133 tracer and spike accordingly. Then cover with watch glass and heat to rapid boiling.

9.3.2 Remove the watch glass and allow the sample to partially cool. Slowly add 10 mL 0.75M Na_2CO_3 while stirring. Allow precipitate to settle and cool for at least one hour.

9.3.3 Allow 30 hours from the time the sodium carbonate was added before loading samples onto columns.

9.3.4 Aspirate the supernate, then rinse the precipitate into a centrifuge tube using pH 10 water. Centrifuge for 10 minutes at 4000 rpm.

9.3.5 Decant the supernate from the centrifuge tube and dissolve the remaining precipitate with approx. 10 mL 8M HNO_3.

9.3.6 Rinse the sides of the beaker with approx. 5 mL 8M HNO_3 and add to the centrifuge tube.

9.3.7 Poor sample from centrifuge tube to a teflon beaker and place on hot plate. Heat to dryness.

9.3.8 Add 5 mL 0.095M HNO_3 or 0.5M HNO_3 and decant into plastic scintillation vial.

9.3.9 Continue at Step 9.1.13.

9.4 Sample Preparation Techniques for Gamma Counting

9.4.1 Measure an appropriate aliquot of water sample into a beaker (s). In each beaker add 10 mL conc. nitric acid, 3 mL BaCL2 carrier, and add appropriate amount of Ba-133 tracer isotope equivalent to Ba-133 value used for calibration. Record the sample volume and standard spike information on the que sheet (Appendix 3).

9.4.2 Add 20 mL of concentrated sulfuric acid to samples, stir and allow precipitate to settle to bottom of beaker.

9.4.3 Filter sample through .45 µm Gelman DM-450 or equivalent filters.

9.4.4 Fold filter and place into plastic vial.

9.4.5 Count sample on calibrated gamma spectrometer for appropriate time to meet required MDA.

9.5 Instrument Calibration for Gas Flow Proportional Counters for Ra-228

9.5.1 Prerinse the same number of Tru-Spec columns as detectors that will be calibrated according to Step 9.1.18.

9.5.2 Add 4 mL of 0.5M nitric solution to each column. Catch the effluent in labeled dispo-cups or plastic centrifuge tubes.

9.5.3 Add 1 mL Ra-228 standard to each column as soon as possible. Rinse with an additional 8 mL of 0.5M nitric acid.

9.5.4 Repeat steps 9.1.20 through 9.1.25.

9.5.5 Count each prepared standard in each gas flow proportional detector, ensuring that each detector counts all of the standards at least once.

9.5.6 Determine the detector efficiencies utilizing a calculation spreadsheet according to the following equation:

$$\text{Detector Efficiency} = \frac{\text{Observed cpm} - \text{Background cpm}}{\text{Certified dpm}}$$

Where:

Obs cpm = cpm generated by the gas flow proportional counter
Bkg cpm = cpm generated by an unspiked standard blank
Known dpm = dpm decay corrected to the mid point of standard counting

9.6 Instrument Calibration for Gamma Spectrometer for Ra-228

Refer to EPI SOP "Microvax 3100 Gamma Spectroscopy System Operating Procedures" (I-001).

9.7 Instrument Performance Requirements

Refer to EPI SOP "Counting Room Instrumentation Maintenance and Performance Checks" (I-010) for instructions concerning instrument performance.

9.8 Analysis Procedures and Instrumental Operation

9.8.1 Refer to EPI SOP "Gamma Spectrometer Operating Instructions" (I-001) for instructions concerning the analysis of the Ba-133 tracer.

9.8.2 Refer to the appropriate gas flow proportional counting procedure (EPI SOP I-002, 003, 005, or 006) for instructions concerning the analysis of the Ac-228.

10.0 CALCULATIONS AND DATA REDUCTION

10.1 The analyst will use an Excel spreadsheet (Appendix 2), Access, or a client specified formula to calculate the sample Ra-228 pCi/L according to the following equations:

$$pCi/l = \frac{(S_{cpm} - B_{cpm}) * A_c}{2.22 * E * V * Y * A_t}$$

10.2 The counting uncertainty for Ra-228 is calculated according to the following equation:

$$pCi/l = \frac{Ac * 1.96\sqrt{((Scpm/T_S)+(Bcpm/T_B))}}{2.22 * E * V * Y * At}$$

10.3 The method detection limit (MDA) is calculated according to the following equation:

$$MDA = \frac{Ac(2.71 + 4.65\sqrt{Bcpm * Tc})}{(2.22 * E * V * Y * At * Tc)}$$

where:

Tc=Amount of time sample was counted
S_{cpm}=Sample counts per minute
B_{cpm}= background counts per minute
E=efficiency of counting
V=sample volume (liters)
C=sample count time (min)
t=time in minutes from separation to counting
A_t=Ac-228 decay correction($A_{t)} = e^{-0.00188*t}$
A_c=count duration decay correction ($A_{c)}$=$Tc*.00188/(1- e^{-0.00188*Tc})$
Y=Chemical yield

11.0 DATA RECORDING, REVIEW AND REPORTING

11.1 Data Recording

11.1.1 Record the information required on the Ra-228 que sheet (Appendix 1).

11.1.2 Transfer applicable data from Ra-228 Que Sheet to Ra-228 master spreadsheet in Excel (Appendix 2).

11.2 Data Review

Refer to EPI SOP "Data Review and Validation Procedures" (D-003) for instructions concerning the data review process.

11.3 Data Reporting

The analyst will take the applicable Ra-228 spreadsheet and enter the data into LIMS. The following information should be included: analyst's initials, date and time the sample was counted, sample results and accuracy in pCi/L.

12.0 QUALITY CONTROL REQUIREMENTS

12.1 Analyst and Method Verification Requirements

Refer to EPI SOP "Analyst and Analytical Methods Validation Procedures" (D-003) for instructions concerning the validation of analysts and analytical methods.

12.2 Method Specific Quality Control Requirements

12.2.1 A method blank should accompany each batch of samples. The reported value of the blank, should be less than or equal to the contract required detection limit (CRDL).

12.2.2 The Ba-133 tracer added to all samples in Step 9.1.1 is used to calculate the method recovery. The method recovery of all samples should be between 25-125% when compared to the reference standard. The method recovery is calculated as follows:

$$\text{Method Recovery} = \frac{\text{Sample pCi}}{\text{Ref pCi}}$$

Where:

Sample pCi = pCi of Ba-133 reported by gamma spectrometer of the sample.
Ref pCi = pCi of Ba-133 reported by gamma Spectrometer of the reference made in Step 9.1.2.

12.2.3 A matrix spike (MS) should be run with every batch of samples. The recovery of the ms should be between 75-125%. The MS recovery is calculated as follows:

$$\text{MS Recovery}(\%)=\frac{\text{Spike}(\text{pCi}/\text{l})-\text{Sample}(\text{pCi}/\text{l})}{\text{Spike Nominal Concentration}(\text{pCi}/\text{l})}*100\%$$

Where:

Spike = Results of matrix spike (MS)
Sample = Results of sample that MS was run on Spike Nominal Concentration Spike
Spike Nominal Concentration = Calculated concentration of Ra-228 in the MS solution as follows

$$\text{SNC}=\frac{\text{Ra}-228\text{dpm}}{2.22*\text{volume in liters}}*\text{ml of spike added}$$

12.2.4 A duplicate sample should be run with every batch. The relative percent difference (RPD) between the actual sample and the QC duplicate should be less than or equal to 20% if both the sample and the QC DUP results are greater than 5 times LLD or 100% if they are both less than 5 times LLD. The RPD should be calculated as follows:

$$\text{RPD}(\%)=\frac{\text{High Dup}(\text{pCi}/\text{l})-\text{Low Dup}(\text{pCi}/\text{l})}{[(\text{High Dup}(\text{pCi}/\text{l})+\text{Low Dup}(\text{pCi}/\text{l})]/2}*100\%$$

12.2.5 A Laboratory Control Sample (LCS) should be run with every batch. The recovery of the LCS should fall between 75-125%. The LCS recovery is calculated as follows:

$$\text{LCS REC}(\%)=\frac{\text{LCS Result}(\text{pCi}/\text{l})}{\text{Nominal Concentration of LCS}(\text{pCi}/\text{l})}*100\%$$

12.3 Actions required if the Quality Control Requirements Are Not Met

If any of the QC criteria from 12.2.1 through 12.2.5 cannot be satisfied, the analyst should inform their group leader and initiate a Nonconformance Report as outlined in GEL SOP "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

NOTE: Some clients may specify more or less stringent requirements.

13.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

All raw data printouts, calculations, spreadsheets, and batch checklists shall be filed with the sample data for archival and review.

14.0 LABORATORY WASTE HANDLING AND DISPOSAL

Radioactive samples and material shall be handled and disposed of as outlined in EPI SOP "EPI Laboratory Waste Disposal and Emergency Instructions" (S-011).

15.0 REFERENCES

15.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

15.2 EML Procedures Manual HASL-300, 1982.

15.3 Special thanks to Dr Bill Burnett and his associates for their development of this method at Florida State University.

APPENDIX 1

Radium-228 in Water Que Sheet

Comment: *none*

Batch # 34655 Prep Date:_____ Initials: _____
Spike Isotope: Ra-228 Spike Code:_____ Vol:_____
Tracer Isotope Ba-133 Tracer Code:_____ Vol:_____
LCS isotope Th-232 LCS Code:_____ Vol:_____
Instrument Used: (circle one) LB5100W HT1000 LB5100 Lb4110 Batched to:
s/n: 14740 10912771 203 8219 Russ Moser

Sample ID	*Sample Descrip.*	*Bkr #*	*Vol (mls)*	*Carrier or Det #*	*Ba yield*	*Ac-228 elution Date/Time*	*Start Beta Count Date/Time*	*Count Time (min)*
9308009-01								
9308009-02								
9308009-03								
9308009-04								
9308009-05								
9308009-06								
9308009-07								
9308009-08								
9308009-09								
9308009-10								
9308009-11								
9308009-12								
9308009-13								
9308009-14								
9308009-15								
9308009-16								
9308009-17								
9308009-18								
9308009-19								
9308009-20								
QC135382								
QC135383								
QC135384								
QC135385								
QC135386								
QC135387								

Data Reviewed By: _____

APPENDIX 2

RADIUM-228 CALCULATION SPREADSHEET

Analyst: DHC
Verified: 08/9/93
Batch # 34883 Ra-228 spk: 2610-1-2-B MS REC: 109%
Date: 8/13/93 129 pCi/l MSD REC: 125%
Alpha Crosstalk 0.01 RPD: 15% (< 5*LLD)
Beta Bkg Count Time: 480 min MS RPD: 13%
LCS: 118%

Lab I.D.	Sample Vol. (ml)	Beta cpm	Ac 228 eff	Ba yield (%)	ac decay con	cnt decay con	Ra bkg cpm	Beta cts	Count time	Ac-228 Elution Date/time	Starting count time	Ra 228.00 MDA	Ra-228 result (pCi/l)	Ra228 error (pCi/l)
9308092-10	500	2.54	0.4000	106	0.82	1.52	1.51	1223	480	8/25/93 15:30	8/25/93 17:15	1.05	4.1	0.7
9308092-11	800	2.22	0.4040	109	0.84	1.06	1.57	133	60	8/23/93 9:22	8/23/93 10:54	1.29	1.0	0.6
9308092-12	500	23.07	0.4070	40	0.83	1.01	1.66	346	15	8/26/93 12:30	8/26/93 14:09	11.71	144.9	16.5
9308092-13	500	24.53	0.3870	40	0.83	1.01	1.38	368	15	8/26/93 12:30	8/26/93 14:09	11.34	164.8	17.9
9308092-14	800	2.42	0.4000	91	0.84	1.06	1.51	145	60	8/23/93 9:22	8/23/93 10:54	1.53	1.8	0.8
9308092-15	800	2.27	0.4280	91	0.84	1.06	1.40	136	60	8/23/93 9:22	8/23/93 10:54	1.38	1.6	0.7
9308092-16	800	2.12	0.4070	96	0.84	1.06	1.40	127	60	8/23/93 9:22	8/23/93 10:54	1.37	1.3	0.7
9308092-17	800	2.05	0.3960	110	0.84	1.06	1.39	123	60	8/23/93 9:22	8/23/93 10:54	1.23	1.1	0.6
QC136375-B	800	0.95	0.4280	105	0.82	1.52	1.25	457	480	8/25/93 15:30	8/25/93 17:15	0.56	-0.7	0.3
QC-DUP	500	2.66	0.4280	98	0.87	1.52	1.40	1277	480	9/2/93 14:55	9/2/93 16:11	0.97	4.7	0.7
QC_MS	500	23.07	0.4070	40	0.83	1.01	1.66	346	15	8/26/93 12:30	8/26/93 14:09	11.71	144.9	16.5
QC_MSD	500	24.53	0.3870	40	0.83	1.01	1.38	368	15	8/26/93 12:30	8/26/93 14:09	11.34	164.8	17.9
QC_LCS	500	11.33	0.4610	93	0.83	1.01	1.24	170	15	9/1/93 10:05	9/1/93 11:44	3.91	25.9	4.4

Signature_____

Reviewed_____

APPENDIX 3

Ra-228 by Well Det. Que Sheet comment: **Radioactive**

Batch # 62793	Prep Date:	Initials:
Spike Isotope:	Spike Code:	Vol:
LCS Isotope:	LCS Code:	Vol:
Tracer Isotope:	Tracer Code:	Vol:
Nom. Conc. MS:		
Nom Conc. LCS:		

Sample ID	Sample Description	Sample Mass (Liters)	Sampling Date	Sampling Time	Prep Date
9502409-01	FSB 78		2/20/95	1020	
9502409-02	FSB 78C		2/20/95	1405	
9502409-02	FSB 110D		2/19/95	1147	
9502445-01	FSB 97C		2/21/95	0912	
9502445-02	FSB 97D		2/21/95	0950	
9502445-03	FSB 106C		2/21/95	1125	
9502445-04	FSB 112C		2/21/95	1025	
9502445-05	FSB 112D		2/21/95	1100	
9502445-06	FSB 119D		2/21/95	1010	
QC262205	blank				
QC262206	9502445-06				
QC262207	9502445-06				
QC262208	lcs				

Batch Assigned to: **IDXL** Automatic

Data Reviewed By:_____

APPENDIX 4

Ra-228 Calculations on the Well 22-Mar-95

Results in pCi/L **Batch Number** 62793 **Nominal Conc.:** Unknown 65.60

Nominal Conc.: QC262207 330.42

Nominal Conc.: QC262208 32.80

Sample ID	QC Type	Net Sample CPM	Yield	MDA	Result	Error
9502409-01		4.99	92.4%	3.58	56.89	4.58
9502409-02		1.11	90.6%	2.24	12.94	1.90
9502409-03		2.89	90.7%	3.65	33.55	3.69
9502445-01		3.66	88.5%	3.49	43.55	3.81
9502445-02		3.54	86.1%	4.66	43.36	4.62
9502445-03		0.63	88.5%	1.56	7.53	1.07
9502445-04		2.52	98.8%	2.42	26.84	1.91
9502445-05		4.11	93.1%	4.53	46.42	3.48
9502445-06		1.33	84.2%	5.25	33.31	4.71
QC262205	BLANK	-0.06	87.6%	1.35	-0.34	0.46
QC262206	DUP	1.32	89.9%	2.75	31.01	2.75
QC262207	SPIKE	1.62	88.5%	56.94	384.53	58.44
QC262208	LCS	1.66	85.9%	4.58	41.03	4.99
UNKNOWN		3.33	91.6%	9.16	76.97	11.22

REC % or RPD:QC262206 _____

REC % or RPD:QC262207 _____

REC % or RPD:QC262208 _____

REC %: _____

Submitted By: _____

Reviewed By: _____

STANDARD OPERATING PROCEDURE

FOR
THE DETERMINATION OF TOTAL ALPHA EMITTING RADIUM AND RADIUM-228 IN SOIL

UNCONTROLLED DOCUMENT

Process Owners:	Terri L. Reeves, Analyst	9/30/94
	Russell L. Moser, Group Leader Gas Flow Proportional Counter	9/30/94
Technical Review by:	James B. Westmoreland, Laboratory Manager	9/30/94
Quality Review by:	Robert L. Pullano, GEL Quality Manager	
Approved and Authorized by:	Howard H. Coleman, President	

SET 12

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1.0 STANDARD OPERATING PROCEDURE FOR DETERMINATION OF TOTAL ALPHA EMITTING RADIUM AND RADIUM-228 IN SOIL

2.0 METHOD OBJECTIVE AND APPLICABILITY

This standard operating procedure provides the necessary instructions to conduct the analysis for Total Alpha Emitting Radium and Ra-228.

3.0 INTERFERENCES

3.1 When converting barium sulfate to the carbonate it is important to remove excess sulfate or the potassium carbonate will be consumed by the reaction:

$$K_2CO_3 + H_2SO_4 \rightarrow K_2SO_4 + H_2O + CO_2$$

3.2 Samples with high strontium-90 may cause interference in the Ac-228 beta count. This problem occurs because of Y-90 present that may follow the Ac-228 chemically. It is best to confirm high Ra-228 (i.e. greater than 5 pCi/l) by gamma spectroscopy.

3.3 Samples with elevated radio lead may cause interference in the Ac-228 beta count. It is best to confirm high Ra-228 (i.e. greater than 5 pCi/l) by gamma spectroscopy.

3.4 Th-234 would potentially interfere with Ac-228 beta counting. It's presence could be confirmed with a decay curve analysis.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 Care should be taken when handling concentrated acids as contact with fumes or liquid may cause severe burning of body tissue.

5.0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment

5.1.1 Pyrex watch glasses

5.1.2 200 ml beakers (8)

5.1.3 Centrifuge: Capable of maintaining 2000 rpm with eight 50 ml centrifuge tubes.

5.1.4 Pyrex 50 ml centrifuge tubes. 29x113 mm.

5.1.5 Pyrex 1000 ml beakers

5.1.6 Pyrex stirring rods

5.1.7 700 watt microwave

5.1.8 Stainless Steel Planchets

5.1.9 Electric hot plates

5.1.10 Oven (103° C)

5.1.11 Glass fiber filters

5.1.12 0.1 um metricel filters

5.2 Reagents, Chemicals and Standards

5.2.1 Ba-133 standard.

5.2.2 EPA or NIST traceable Ra-226 and Ra-228 standard.

5.2.3 Barium chloride ($BaCl_2$) carrier. Dissolve 2.846 grams reagent grade $BaCl_2$ in DI water, add .5 ml 16 N HNO_3 and dilute to 100 ml.

5.2.4 Concentrated sulfuric acid.

5.2.5 0.5 M nitric acid. Dilute 63 ml concentrated nitric acid to 2000 ml of DI water.

5.2.6 3 M HCl. Dilute 500 ml of concentrated HCl to 2000 ml DI water.

5.2.7 50% (w/w) potassium carbonate.

5.2.8 Concentrated nitric acid.

5.3 Instrumentation

5.3.1 Gross Alpha Beta Proportional Counting System.

5.3.2 Gamma Spectrometer and associated electronics and data reduction package.

6.0 SAMPLE COLLECTION & PRESERVATION

A representative sample should be collected in a suitable container so that meaningful aliquots can be taken. No preservation is needed.

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to EPI SOP I-001 "Micro-Vax 3100 Gamma Spectrometer Operating Procedure".

7.2 Refer to EPI SOP I-002 "HT-1000 Gross Alpha/Beta Counter Operating Procedure".

7.3 Refer to EPI SOP I-006 "LB-4110 Gross Alpha/Beta Counter Operating Procedure".

7.4 Refer to EPI SOP I-010 Counting Room Instrument Maintenance and Performance Checks".

8.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

Refer to EPI SOP M-001 "Preparation of Radioactive Standards" for instructions concerning the preparation of standard solutions.

9.0 OPERATING PROCEDURES

9.1 Sample Preparation Techniques

9.1.1 Initially prepare the soil sample as outlined in EPI-SOP A-021 "Soil Sample Preparation for the Determination of Radionuclides".

9.1.2 Weigh an appropriate aliquot into a crucible and ash the sample in a muffle furnace at a temperature of 500° C for at least 3 hours. Record the aliquot weight of the que sheet (Appendix 1).

9.1.3 Transfer the sample into the Teflon cup of the digestion bomb. Add 0.1 ml of Ba-133 tracer . Record the sample volume and standard spike information on the que sheet (Appendix 1). Add 8 ml of concentrated HNO_3 to the samples. Refer to EPI SOP A-015, for digestion times and power as well as safe operation of the microwave digestion bombs.

Note: Between digestion phases and at completion the bomb should only be opened under a fume hood.

9.1.4 Add 0.1 ml of Ba-133 tracer to 5 ml of 0.5 M HNO_3 solution in a liquid scintillation vial. This solution will serve as the reference for yield determination.

9.1.5 Dilute the digestate to 500 ml with DI water in a beaker. Add 1.0 ml of stable barium carrier and 5 ml of concentrated nitric acid.

9.1.6 Add 10 ml of concentrated sulfuric acid, cover with a watch glass and warm the solution to 90° C. Occasionally stir while heating for 2 hours. Remove from heat and allow to cool overnight, or until a clear supernate is observed.

9.1.7 Decant the clear supernate then add the remaining precipitate in a 50 ml pyrex centrifuge tube and centrifuge for 5 minutes at 2000 RPM.

9.1.8 Decant centrifuge tube and remove excess sulfate by washing the precipitate with DI water from a wash bottle.

9.1.9 Centrifuge and decant the DI water wash. Check the pH of the wash solution with a pH strip.

9.1.10 Repeat the washing as necessary until a pH of 6 is observed.

9.1.11 Add 1 ml of 50% K_2CO_3 to the precipitate making sure the precipitate is completely submerged in the solution.

9.1.12 Place eight 200 ml glass beakers in a microwave and arrange one centrifuge tube in each beaker. Be certain that all of the tubes face outward and away from each other.

9.1.13 Heat the centrifuge tubes for 1 1/2 minutes on high power to form a thick slurry or paste.

9.1.14 Allow the tubes to cool then add 1-3 mls DI water and repeat the heating process twice. Clean the microwave after usage.

9.1.15 Wash the dry slurry with 15 mls DI water (gentle heating may be required to remove dry slurry from sides of centrifuge tube) centrifuge and decant the supernate. Check the pH of the wash solution it should initially be around 12. If a high pH is not observed consult the appropriate group leader.

9.1.16 Wash the precipitate with 15 mls of DI water, centrifuge and decant. Repeat washings until the pH of the wash solution is < 7. This will require 1-3 more washes.

9.1.17 Dissolve the precipitate in 5 ml of 0.5 M nitric/0.5 M HNO_3 solution. Gentle heating may be required. Centrifuge the solution and decant into a 20 ml scintillation vial.

9.1.18 Count the reference vial from step 9.1.2 for 5 minutes prior to counting samples on a gamma spectrometer.

9.1.19 Count the sample on the gamma spectrometer for 5 minutes. See the gamma VAX operating procedure for operating the gamma spectrometer (EPI SOP I-001). The samples and reference should be given a sample volume of 5 ml, an identical reference date and a counting geometry of "2L_MB". Divide the sample result by the reference result to determine the chemical yield.

9.1.20 Allow the sample to ingrow for at least 30 hours from the time the sample was added to the scintillation vial before proceeding.

Note: The following steps supporting the Ra-228 procedure should be performed within a 10 hour time frame to avoid decay of unsupported Ac-228 with a half-life of 6 hours.

9.1.21 Prerinse a TRU-SPEC™ column with 10 ml of 0.5 M nitric acid.

9.1.22 Place a plastic centrifuge tube under the column to collect the sample and rinsate that passes through the column. The collected sample is processed for Total Radium Analysis using starting with step 9.1.32.

9.1.23 Load the sample (5 ml) onto the column. Rinse with an additional 8 ml of 0.5 M nitric acid. Add nitric acid in 2 ml increments allowing previous volume to completely drain. Note the date and time on the que sheet (Appendix 1).

9.1.24 Elute the Ac-228 with 5 ml of 3 M HCl into labeled plastic dispo cup or plastic centrifuge tube.

9.1.25 Add 200 ul of 500 ppm Ce solution to the samples and swirl.

9.1.26 Add 1 ml of concentrated HF, swirl and allow to stand for 30 minutes.

9.1.27 Place a 0.1 um metricel filter on a 25 mm filtering apparatus.

9.1.28 Rinse the filter and funnel under vacuum with 5 mls of 80% ethanol and then 5 mls of type II water. With minimum delay, add the sample to the filtering apparatus and rinse the beaker several times into the funnel with type II water. Complete the filtering by adding 5 mls of 80% ethanol.

9.1.29 Label a 29 mm flat planchet with the applicable laboratory number. Place double stick tape on the planchet and carefully place the filter (precipitate side up) on the adhesive. Care should be taken to make the filter as flat as possible on the planchet. Dry the filter under a heat lamp.

9.1.30 Place the disk in a 2 inch planchet and count in a gas flow proportional counter for a time duration to meet the contract required detection limit and uncertainty.

9.1.31 Results above 5 pCi/l should be confirmed by gamma counting the Ac-228 gamma lines.

9.1.32 Total Alpha Emitting radium is prepared using sample and rinsate from step 9.1.22. Add 1 ml of concentrated HNO_3 to the centrifuge tubes and 1 ml BaCl carrier. Swirl to mix.

9.1.33 Add 2 ml concentrated H_2SO_4 and swirl to mix with the lid off. Care should be taken while mixing the sample, pressure may build up due to heating from the chemical reaction of the H_2SO_4.

9.1.34 Filter quantitatively through a 47 mm pre weighed glass fiber filter, rinsing the final precipitate out of the centrifuge tube with a fine jet of water.

9.1.35 Place the filters on aluminum foil and dry in an oven at 100° C for one hour.

9.1.36 Permanently fix the filters to a stainless steel planchet with glue stick or double stick tape.

9.1.37 Count the sample for an appropriate period of time to meet the contract required detection limit and uncertainty in a gross alpha beta counting system.

9.2 Instrument Calibration

9.2.1 Calibrate the glass flow proportional counters for Total Alpha Emitting Radium using EPI SOP A-010.

9.2.2 Calibrate the glass flow proportional counters for Radium-228 using EPI SOP A-009.

9.3 Instrument Performance Requirements

9.3.1 Refer to EPI SOP I-010 "Counting Room Instrumentation Maintenance and Performance Checks" for instructions concerning instrument performance.

9.4 Analysis Procedures and Instrumental Operation

9.4.1 Refer to EPI SOP I-001 "Gamma Spectrometer Operating Instructions" for instructions concerning the analysis of the Ba-133 tracer.

9.4.2 Refer to the appropriate gas flow proportional counting procedure (EPI SOP I-002, 003, 005, or 006) for instructions concerning the analysis of the Ac-228.

10.0 CALCULATIONS AND DATA REDUCTION

10.1 The analyst will use an excel spreadsheet (Appendix 2) to calculate the sample Ra-228 Ci/l according to the following equations:

$$pCi/l = \frac{(S_{cpm} - B_{cpm}) * A_c}{2.22 * E * V * Y * A_t}$$

10.2 The counting uncertainty for Ra-228 is calculated according to the following equation:

$$pCi/l = \frac{A_c \cdot 1.96\sqrt{(S_{cpm} + B_{cpm})}}{2.22 * E * V * Y * A_t}$$

10.3 The method detection limit for Ra-228 (MDA) is calculated according to the following equation:

$$MDA = \frac{A_c(2.72 + 4.66\sqrt{B_{cpm} * T_c})}{(2.22 * E * V * Y * A_t * T_c)}$$

where:
Tc=Amount of time sample was counted
S_{cpm}=Sample counts per minute
B_{cpm}= background counts per minute
E=efficiency of counting
V=sample volume (liters)
C=sample count time (min)
t=time in minutes from separation to counting
A_t=Ac-228 decay correction(A_t) = $e^{-0.00188*t}$

A_C=count duration decay correction (A_C)=Tc*.00188/(1- $e^{-0.00188*Tc}$)
Y=Chemical yield

10.4 The analyst will use an excel spreadsheet (Appendix 2) to calculate the sample Total Alpha Emitting Radium pCi/l according to the following equations:

$$pCi/l = \frac{(S_{cpm} - B_{cpm})}{2.22 * E * V * Y * I}$$

10.2 The counting uncertainty for Total Alpha Emitting Radium is calculated according to the following equation:

$$pCi/l = \frac{1.96\sqrt{(S_{cpm} + B_{cpm})}}{2.22 * E * V * Y * I}$$

10.3 The method detection limit for Total Alpha Emitting Radium(MDA) is calculated according to the following equation:

$$MDA = \frac{(2.72 + 4.66\sqrt{B_{cpm} * T_c})}{(2.22 * E * V * Y * I * T_c)}$$

where:
Tc=Amount of time sample was counted
S_{cpm}=Sample counts per minute
B_{cpm}= background counts per minute
E=efficiency of counting
V=sample volume (liters)
C=sample count time (min)
t=time in minutes from separation to counting
I=Ingrowth Factor
Y=Chemical yield
CF=Correction Factor for Efficiency Change due to ingrowth.
T= elapsed time (min) from $BaSO_4$ precipitation to sample counting.
I=1+3*(1-exp(-.0001259*T)+CF

11.0 DATA RECORDING, REVIEW AND REPORTING

11.1 Data Recording

11.1.1 Record the information required on the que sheet (Appendix 1).

11.1.2 Transfer applicable data from Que Sheet to master spreadsheet in Excel for the applicable test (Appendix 2).

11.2 Data Review

11.2.1 Refer to EPI SOP D-003 "Data Review and Validation Procedures" for instructions concerning the data review process.

11.3 Data Reporting

11.3.1 The analyst will take the applicable spreadsheet and enter the data into LIMS. The following information should be included: analyst's initials, date and time the sample was counted, sample results and accuracy in pCi/l.

12.0 QUALITY CONTROL REQUIREMENTS

12.1 Analyst and Method Verification Requirements

12.1.1 Refer to EPI SOP D-003 "Data Review and Validation Procedures" for instructions concerning the validation of analysts and analytical methods.

12.2 Method Specific Quality Control Requirements

12.2.1 A method blank should accompany each batch of samples. The reported value of the blank, should be less than or equal to the contract required detection limit (CRDL).

12.2.2 The Ba-133 tracer added to all samples in Step 9.1.1 is used to calculate the method recovery. The method recovery of all samples should be between 25-125% when compared to the reference standard. The method recovery is calculated as follows:

$$\text{Method Recovery} = \frac{\text{Sample pCi}}{\text{Ref pCi}}$$

Where:
Sample pCi =pCi of Ba-133 reported by gamma spectrometer of the sample.
Ref pCi=pCi of Ba-133 reported by gamma Spectrometer of the reference made in Step 9.1.2.

12.2.3 A matrix spike (ms) should be run with every batch of samples. The recovery of the ms should be between 75-125%. The ms recovery is calculated as follows:

$$MS\,Recovery(\%) = \frac{Spike(pCi\,/\,L) - Sample(pCi\,/\,L)}{Spike\,Nominal\,Concentration(pCi\,/\,L)} * 100\%$$

Where:
Spike=results of matrix spike
Sample=results of sample that ms was run on Spike Nominal Concentration
Spike Spike Nominal Concentration =Calculated concentration of Radium in the ms solution as follows

$$SNC = \frac{Radium dpm}{2.22 * volume\ in\ liters} * ml\ of\ spike\ added$$

12.2.4 A duplicate sample should be run with every batch. The relative percent difference (RPD) between the actual sample and the QC duplicate should be less than or equal to 20% if both the sample and the QC duplicate results are greater than 5 times LLD or 100% if they are both less than 5 times LLD. The RPD should be calculated as follows:

$$RPD(\%) = \frac{High\,Dup(pCi\ /\ L) - Low Dup(pCi\ /\ L)}{[(High\,Dup(pCi\ /\ L) + Low Dup(pCi\ /\ L)]\ /\ 2} * 100\%$$

12.2.5 A Laboratory Control Sample (LCS) should be run with every batch. The recovery of the LCS should fall between 75-125%. The LCS recovery is calculated as follows:

$$LCS\_REC = \frac{LCS Result(pCi\ /\ l)}{Nominal\ Concentrat\ ion\ of\ LCS(pCi\ /\ l)} * 100\%$$

12.3 Actions Required if the Quality Control Requirements Are Not Met

12.3.1 If any of the QC criteria from 12.2.1 through 12.2.5 cannot be satisfied, the analyst should inform their group leader and initiate a Nonconformance Report as outlined in GEL SOP GL-QS-E-004 "Nonconformance Identification, Control, Documentation, Reporting, and Dispositioning".

13.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

All raw data printouts, calculations, spreadsheets, and batch checklists shall be filed with the sample data for archival and review.

14.0 LABORATORY WASTE HANDLING AND DISPOSAL

Radioactive samples and material shall be handled and disposed of as outlined in EPI SOP S-005 "Radioactive Waste Handling Procedures".

15.0 REFERENCES

15.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

15.2 EML Procedures Manual HASL-300, 1982.

15.3 Special thanks to Dr Bill Burnett and his associates for their development of this method at Florida State University.

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APPENDIX 1

Radium-228 in Water Que Sheet

Comment: *none*

Batch # 34655 Prep Date:_____ Initials: _____

Spike Isotope: Ra-228 Spike Code:_____ Vol:_____

Tracer Isotope: Ba-133 Tracer Code:_____ Vol:_____

LCS isotope Th-232 LCS Code:_____ Vol:_____

Instrument Used:(circle one) LB5100WHT1000 LB5100 Lb4110 Batched to:

s/n: 1474010912771 203 8219 Russ Moser

Sample ID	*Sample Descrip.*	*Bkr #*	*Vol (mls)*	*Carrier or Det*	*Ba yield*	*Ac-228 elutio Date/Time*	*Start Beta Cou Date/Time*	*Count Time (min*
9308009-01								
9308009-02								
9308009-03								
9308009-04								
9308009-05								
9308009-06								
9308009-07								
9308009-08								
9308009-09								
9308009-10								
9308009-11								
9308009-12								
9308009-13								
9308009-14								
9308009-15								
9308009-16								
9308009-17								
9308009-18								
9308009-19								
9308009-20								
QC135382								
QC135383								
QC135384								
QC135385								
QC135386								
QC135387								

Data Reviewed By: _____

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APPENDIX 2

RADIUM-228 CALCULATION SPREADSHEET

Analyst: DHC
Verified: 08/9/93
Batch # 34883 Ra-228 spk: 2610-1-2-B MS REC: 109%
MSD REC: 125%
Date: 8/13/93 129 pCi/l RPD: 15% (< 5*LLD)
Alpha Crosstalk 0.01 MS RPD: 13%
Beta Bkg Count Time: 480min LCS: 118%

Lab I.D.	Sample Vol. (ml)	Beta cpm	Ac 228 eff	Ba yield (%)	ac decay con	cnt decay con	Ra bkg cpm	BetaCount cts	time	Ac-228 Elution Date/time	Starting count time	Ra 228.00 MDA	Ra-228 result (pCi/l)	Ra228 error (pCi/l)
9308092-10	500	2.54	0.4000	106	0.82	1.52	1.51	1223	480	8/25/93 15:30	8/25/93 17:15	1.05	4.1	0.7
9308092-11	800	2.22	0.4040	109	0.84	1.06	1.57	133	60	8/23/93 9:22	8/23/93 10:54	1.29	1.0	0.6
9308092-12	500	23.07	0.4070	40	0.83	1.01	1.66	346	15	8/26/93 12:30	8/26/93 14:09	11.71	144.9	16.5
9308092-13	500	24.53	0.3870	40	0.83	1.01	1.38	368	15	8/26/93 12:30	8/26/93 14:09	11.34	164.8	17.9
9308092-14	800	2.42	0.4000	91	0.84	1.06	1.51	145	60	8/23/93 9:22	8/23/93 10:54	1.53	1.8	0.8
9308092-15	800	2.27	0.4280	91	0.84	1.06	1.40	136	60	8/23/93 9:22	8/23/93 10:54	1.38	1.6	0.7
9308092-16	800	2.12	0.4070	96	0.84	1.06	1.40	127	60	8/23/93 9:22	8/23/93 10:54	1.37	1.3	0.7
9308092-17	800	2.05	0.3960	110	0.84	1.06	1.39	123	60	8/23/93 9:22	8/23/93 10:54	1.23	1.1	0.6
QC136375-B	800	0.95	0.4280	105	0.82	1.52	1.25	457	480	8/25/93 15:30	8/25/93 17:15	0.56	-0.7	0.3
QC-DUP	500	2.66	0.4280	98	0.87	1.52	1.40	1277	480	9/2/93 14:55	9/2/93 16:11	0.97	4.7	0.7
QC_MS	500	23.07	0.4070	40	0.83	1.01	1.66	346	15	8/26/93 12:30	8/26/93 14:09	11.71	144.9	16.5
QC_MSD	500	24.53	0.3870	40	0.83	1.01	1.38	368	15	8/26/93 12:30	8/26/93 14:09	11.34	164.8	17.9
QC_LCS	500	11.33	0.4610	93	0.83	1.01	1.24	170	15	9/1/93 10:05	9/1/93 11:44	3.91	25.9	4.4

Signature_____

Reviewed_____

STANDARD OPERATING PROCEDURE

FOR UNCONTROLLED DOCUMENT

TOTAL ALPHA RADIUM ISOTOPES

(GL-EPI-A-010 Revision 4)

PROPRIETARY INFORMATION

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

Process Owner	7/12/99 (sign and date)	Tim C. Winters (print name)
Technical Review	7/29/99 (sign and date)	Richard M. Kinney (print name)
Quality Review	7/27/99 (sign and date)	Gene G. Bost (print name)
Approval and Authorization (sign and date)	7/29/99	James B. Westmoreland (print name)

SET 2

GENERAL ENGINEERING LABORATORIES, INC.
P.O. Box 30712, Charleston, SC 29417

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TABLE OF CONTENTS

1.0 STANDARD OPERATING PROCEDURE TOTAL ALPHA RADIUM ISOTOPES

2.0 METHOD OBJECTIVE AND APPLICABILITY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for total alpha radium isotopes in soil and water.

2.2 This procedure combines two related procedures. The following standard operating procedure is canceled without replacement having served its purpose:

SOP "Total Alpha Radium Isotopes in Soil" (A-010b).

2.3 General Engineering Laboratories, Inc. utilizes methods that are derived from established sources. This method is based on the source procedures manual EPA 600 4-80-032 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water" August 1980 Method 903.0 and uses similar principles of radiochemical concentration and counting.

3.0 INTERFERENCES

This method is intended as a gross screening method for alpha emitting isotopes. Other alpha emitting isotopes that precipitate in barium sulfate may cause a positive bias.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

Care should be taken when handling/heating the acids used in this method (concentrated HCl and H_2SO_4).

5.0 APPARATUS AND MATERIAL

5.1 Ancillary Equipment

5.1.1 Stainless steel planchets

5.1.2 Electric hot plates

5.1.3 Drying Ovens

5.1.4 1000 mL beakers

5.1.5 Watch glasses

5.1.6 Glass fiber filters

5.1.7 Tuffryn filters

5.1.8 Glass stir rods

5.1.9 pH strips

5.1.10 1 mL pipette or equivalent

5.2 Reagents, Chemicals and Standards

5.2.1 Distilled or deionized water

5.2.2 Barium carrier, 16 mg/mL: Dissolve 28.46 grams $BaCl_2.H_2O$ in water; add 0.5 16 N HNO_3 and dilute to 1000 mL with DI water.

5.2.3 Hydrochloric acid, 12N: HCl (conc) sp.gr. 1.19, 37.2%.

5.2.4 Nitric acid, 1N: Mix 6.2 mL 16N HNO_3 (conc.) with water and dilute to 100 mL.

5.2.5 1:1 Sulfuric acid, (18N): Cautiously add 500 mL 36N H_2SO_4 (conc.) to 500 mL water, cool, add 500 mL 36N H_2SO_4, and dilute to 2L.

5.3 Instrumentation

Gas-flow proportional counting system.

6.0 SAMPLE COLLECTION & PRESERVATION

6.1 Soil samples should be collected in a suitable container so that a representative aliquot can be taken. No preservation is required.

6.2 The sample should be collected preserved at that time of collection with 1N HNO_3 to a pH of <2. The sample should be collected in plastic if possible. Analysis should be as soon after collection as possible. If the sample is found not to be preserved, then add a proper amount of concentrated HNO_3 to the aliquot until a pH of <2 is obtained. Allow the aliquot to equilibrate 24 hours prior to analysis.

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to GEL SOPs "LB5100 Gross Alpha/Beta Counter Operating Instructions" (GL-EPI-I-003), or "LB5100W Gross Alpha/Beta Counter Operating Instructions" (GL-EPI-I-005) for instructions concerning the gas flow proportional counter operation.

7.2 Refer to GEL SOP "Counting Room Instrument Maintenance and Performance Checks" (GL-EPI-I-010) for instructions concerning instrument maintenance.

8.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

Refer to GEL SOP "Preparation of Radioactive Standards" (GL-EPI-M-001) for instructions concerning the preparation of standard solutions.

9.0 OPERATING PROCEDURE

9.1 Sample Preparation Techniques

9.1.1 Initially prepare the soil sample as outlined in GEL-SOP "Soil Sample Preparation for the Determination of Radionuclides" (GL-EPI-A-021).

9.1.2 Weigh approximately 0.5 gram of the prepared soil into a crucible and ash the sample in a muffle furnace at a temperature of 500°C for at least 3 hours. Record the aliquot weight on the que sheet (Appendix 1). If the sample is a liquid, proceed with step 9.1.6.

9.1.3 Transfer the sample into the Teflon cup of the digestion bomb. Add 8 mL of concentrated HNO_3 to the samples. Refer to GEL SOP "Microwave Digestion by Soil and Sand" (GL-EPI-A-015) for digestion times and power as well as safe operation of the microwave digestion bombs.

NOTE: Between digestion phases and at completion the bomb should only be opened under a fume hood.

9.1.4 Rinse out the Teflon cups into Teflon beakers using type II water. Several rinses should be adequate.

9.1.5 Dilute the digestate to 500 mL with DI water in a beaker, add 10 mL 12N HCl mL and 1 mL of barium carrier. Cover the beakers with watch glasses and heat the samples to boiling.

9.1.6 For aqueous samples, transfer a known amount of sample to a beaker, ensure a pH of < 2, add 10 mL 12N HCl mL and 1 mL of barium carrier. Cover the beakers with watch glasses and heat the samples to boiling.

9.1. When the sample boils, remove the watch glass and slowly add 10 mL of 1:1 H_2SO_4 (18N) acid while stirring.

9.1.8 Adjust the hot plate to a medium setting and continue heating the samples for 1-2 hours.

9.1.9 Allow the samples to cool and settle at room temperature. Filter quantitatively through a 47 mm pre-weighed fiber filter, rinsing the final precipitate out of the beaker with a fine jet of water.

9.1.10 Place the filters in an oven to dry at $100^{\circ}C \pm 5^{\circ}C$.

9.1.11 Weigh the filter and record the weights.

9.1.12 Permanently fix the filters to a stainless steel planchet with glue stick or double stick tape.

9.1.12 Count the sample for an appropriate period of time to meet the contract required detection limit and uncertainty in a gross alpha beta counting system.

9.2 Instrument Calibration

9.2.1 Prepare radium standards using a known certified amount of spike solution. Add this solution to DI water in step 9.1.1 if soil and step 9.1.6 if a liquid.

9.2.2 Add varying amounts of barium carrier to simulate weight differences.

These weights should vary from about 10mg-150mg.

NOTE: Heating is not necessary for calibration samples.

9.2.3 Follow applicable steps in step 9.1 to prepare samples

9.2.4 Samples should be counted long enough to obtain at least 10,000 counts.

9.3 Instrument Performance Requirements

Refer to GEL SOP "Counting Room Instrument Maintenance and Performance Checks" (GL-EPI-I-010) for instructions concerning instrument maintenance.

9.4 Analysis Procedures and Instrumental Operation

Samples should be counted within 4 days to minimize errors due to radium daughter in growth.

10.0 CALCULATIONS AND DATA REDUCTION METHODS

10.1 The instrument will report sample pCi/g according to the following equation:

$$\text{Sample pCi/g} = \frac{\text{Scpm} - \text{Bcpm}}{2.22 * E * V * I}$$

10.2 Counting uncertainty is calculated according to the following equation:

$$\text{Sample pCi/g} = \frac{1.96\sqrt{\text{Scpm} - \text{Bcpm}/C}}{2.22 * E * V * I}$$

10.3 The method MDC is calculated according to the following equation:

$$MDC = \frac{2.71 + 4.66 * \sqrt{cpm_{bkg} * Tc}}{(2.22 * E * V * Tc * I)}$$

Where:

S_{cpm} = Sample counts per minute
B_{cpm} = Background counts per minute
E = Counting Efficiency
V = Sample volume (g)
C = Sample count time
I = In growth factor

$I = 1 + 3(1-\exp(-.0001259*T)$

Where: T = elapsed time (min.) from $BaSO_4$ precipitation to sample counting.

11.0 DATA RECORDING, REVIEW AND REPORTING

11.1 Data Recording

Record the applicable information on the que sheet (Appendix 1).

11.2 Data Review

Refer to GEL SOP "Data Review and Validation Procedures" (GL-EPI-D-003) for instructions concerning the validation of analysis and analytical.

11.3 Data Reporting

The analyst will take the applicable spreadsheet and enter the data into the LIMS. The following information should be included: analyst's initials, run date and time of the sample, results and accuracy in CPI/l.

12.0 QUALITY CONTROL REQUIREMENTS

12.1 Analyst and Method Verification Requirements

Refer to GEL SOP "Analyst and Analytical Methods Validation Procedures" (GL-EPI-D-002) for instructions concerning the validation of analysts and analytical methods.

12.2 Method Specific Quality Control Requirements

12.2.1 A matrix spike(ms) should be run with every batch of 20 or less samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\%\text{Rec}=\frac{\text{Spike (pCi/g)}-\text{Sample (pCi/g)}}{\text{Spike Concentration (pCi/g)}}*100$$

12.2.2 A duplicate of a sample should be run with every batch of 20 or less samples. The relative percent difference (RPD) between the duplicate (dup) and the sample should be less than or equal to 20%. The RPD is calculated as follows:

$$\text{RPD}=\frac{\text{High Dup (pCi/g)}-\text{Dup (pCi/g)}}{\text{Average (pCi/g)}}*100$$

12.2.3 A method blank should accompany each batch of 20 or less samples. The reported value should be less than or equal to the Contract Required Detection Limit (CRDL).

12.3 Actions required if the Quality Control Requirements Are Not Met

12.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non conformance report as outlined in GEL SOP "Documentation of Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

12.3.2 Some clients may specify more or less stringent requirements.

13.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

13.1 Each analysis that is performed on the instrument is documented in the run log according to GEL SOP "Run Logs" (GL-LB-E-009).

13.2 All raw data printouts, calculation spreadsheets, and batch checklists are filed with the sample data for archival and review.

14.0 LABORATORY WASTE HANDLING AND DISPOSAL

Radioactive material is handled and disposed of as outlined in GEL SOP "EPI Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-S-011).

15.0 REFERENCES

15.1 USEPA Method 900.0

15.2 EML Procedures Manual. HASL-300-Ed. 25. 1982.

APPENDIX 1

Total Radium Que Sheet

Total Radium Que Sheet Batch #: 140938 Analyst: IDXL Automatic Comments:

Spike isotope: Ra-226 Spike Code:_____ Vol:_____ Prep Date:_____
Initials :_____

LCS isotope: Ra-226 LCS Code: _____ Vol:_____ Minimum Duedate: 2/12/99

Sample ID	Client Description		RDL	Client	Matrix	BKR #	Vol (mL or g)	Carrier or Det #	ppt Date/Time	Initial Weight (g)	Final Weight (g)	Net Weight (mg)
9901764-02	ZBG 2		1 PCL	WSRC	GroundH2O							
9901810-01	6799		1 pCi/	SERC	GroundH2O							
QC579870	blank	BLA	1 pCi/	QC								
QC579871	9901764-02	DUP	1 pCi/	QC								
QC579872	9901764-02	MS	1 pCi/	QC								
QC579873	lcs	LCS	1 pCi/	QC								
QC579874	known	KNO	1 pCi/	QC								

Instrument Used: (Circle One) LB4110 S/N: 8219 HT1000 S/N: 10912771 Data Reviewed By:_____
LB5100 S/N: 203 LB5100W S/N: 14740

Page 1 of 1

STANDARD OPERATING PROCEDURE

UNCONTROLLED DOCUMENT

FOR

THE ISOTOPIC DETERMINATION

OF

AMERICIUM, CURIUM, PLUTONIUM AND URANIUM

(GL-EPI-A-011-Revision5)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of the General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

Robert D Timm 6/22/99	Robert D Timm
Process Owner (sign and date)	(print name)
Richard M. Kinney 6/17/99	Richard M. Kinney
Technical Review (sign and date)	(print name)
Gene D Bost 6/24/99	Gene G. Bost
Quality Review (sign and date)	(print name)
James B Westmoreland	James B. Westmoreland
Approval and Authorization (sign and date)	(print name)

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TABLE OF CONTENTS

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1.0 Title: Standard Operating Procedure for the Isotopic Determination of Americium, Curium, Plutonium, and Uranium.

2.0 Method Objective, Purpose, Code, and Summary

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic americium, curium, plutonium and uranium in a variety of matrices. This method also gives specific guidance on determining U-232 and Am-243, which are typically used as isotopic tracers.

2.2 A sample is digested if necessary and aliquoted. Transuranic elements are scavenged by coprecipitation with iron hydroxide. The precipitate is dissolved and separation of elements is accomplished through extraction chromatography and ion exchange resins. The elements are then prepared for the measurement of radioactive isotopes by coprecipitation with cerium fluoride. The cerium fluoride precipitate is trapped on a filter, mounted on a stainless steel disk and placed in a partially evacuated chamber for measurement of isotopic alpha emission.

2.3 General Engineering Laboratories (GEL) utilizes methods that are derived from established sources. This method is based on the source method from DOE EML Methods Manual HASL 300 E-U-04 and uses similar principles of radiochemical separation and counting. This method is also very similar in concept to the source method from the DOE Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP800: "Sequential Separation of Americium and Plutonium by Extraction Chromatography."

2.4 This revision combines several related procedures. The method for the determination of isotopic Plutonium originally in SOP "The Isotopic Determination of Thorium, Plutonium and Neptunium" (GL-EPI-E-A012) has been added to this procedure. The following standards operating procedures are canceled without replacement having served their purpose:

2.4.1 SOP GL-EPI-E-A012b, "The Determination of Isotopic Neptunium and Plutonium in Soil and Vegetation."

2.4.3 SOP GL-EPI-E-A012d, "The Determination of Isotopic Thorium, Plutonium and Neptunium and Plutonium in Air Filters."

3.0 Method Applicability

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for U/Am/Cm/Pu is 1 pCi/L or 0.1 pCi/g for all isotopes. For this procedure, MDAs as low as 0.01 pCi/L or 0.002 pCi/g for all isotopes can be obtained by appropriately increasing the sample size and/or count time.

3.2 Method Precision: Typical relative percent difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value. Analyst training records are kept on hand in the human resource department.

4.0 Definitions

4.1 National Institute of Standards and Technology (NIST). For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.2 Type II water: Deionized water.

4.3 LIMS: Laboratory Information Management System. The database system used to store and report data.

5.0 Method Variations

5.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Senior Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 Safety Precautions and Warnings

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals

in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the library and in the laboratory, respectively. Individual sample MSDS forms provided by the clients are kept in Login."

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are nitrile gloves for concentrated acids and bases, and potassium ferricyanide in neat form. Work under a hood when using concentrated acids and bases.

6.4 The handling of radioactive samples is outlined in SOP "Handling of Radioactive Samples" (GL-EPI-E-S004). General guidelines include:

6.4.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

6.4.2 A plastic apron may be worn over the lab coat for added protection from contamination when working with radioactive samples.

6.4.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.4.4 Prohibit admittance to immediate work area.

6.4.5 Post signs indicating radioactive samples are in the area.

6.4.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in the radiochemistry laboratory.

6.4.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management."

6.5 Refer to SOP "Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011) for instructions on how materials are disposed.

6.6 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the Group Leader prior to carrying out the rest of the procedure.

6.7 When handling biological samples protect the hands and forearms by wearing gloves and a laboratory coat to avoid contact of the biological material with the skin. Protect the eyes by wearing safety glasses and if desired a splash shield.

6.8 If cutting of meats or other tissue with cutlery is required, the analyst will wear a cutting glove of mesh steel over disposable gloves to avoid cuts, which could infect the analyst with pathogens.

6.9 Any procedure, which volatilizes biological substances such as drying or ashing, must be conducted in a hood or other suitable containment device.

6.10 Decontamination of work surfaces exposed to biological samples is performed by wiping the work area with a diluted (1:10) bleach solution and water as soon as possible following analytical operations.

6.11 Exterior protective clothing shall be removed prior to exiting the bioassay sample preparation area in order to prevent the inadvertent spread of biohazards to the rest of the laboratory.

6.12 Hands will be washed with an antibacterial soap directly after handling biological samples.

7.0 Interferences

7.1 Internal tracer standards may have ingrown daughters that may interfere with the analysis. For example Th-228 will be present in aged U-232 standard, Fr-221 will be present in Th-229 which will interfere with the curium analysis, and U-232 will be present in Pu-236. These problems are overcome by running separate aliquots of sample for thorium analysis.

7.2 Short lived radioactive progeny may ingrow on prepared filters. For example, the Ra-224 alpha peak will be present if the Th-228 parent is present. These interferences are minimized by counting samples as soon as possible after separation chemistry.

8.0 Apparatus, Materials, Reagents, Equipment, and Instrumentation

8.1 Ancillary Equipment

8.1.1 Silicon surface barrier detectors with associated electronics, vacuum chambers, and data reduction capabilities

8.1.2 Ion exchange columns. Eichrom TRU-Spec™ prepackaged column with 25 mL reservoir

8.1.3 Vacuum pump and filtration apparatus (25 mm)

8.1.4 Gelman metricel 25 mm filters with 0.1 µm pore size

8.1.5 Gelman polypropylene 25 mm support filter

8.1.6 Stainless steel disks. 29 mm diameter

8.1.7 Stainless steel tweezers

8.1.8 Polypropylene centrifuge tube (50 mL)

8.1.9 Sample drying and ashing apparatus

8.1.10 Sample homogenization apparatus

8.1.11 AG1X8 anion exchange resin 100-200 mesh

8.2 Reagents, Chemicals and Standards

8.2.1 Ammonium hydroxide concentrated (14 N)

8.2.2 Ascorbic acid (0.8M). Dissolve 14.1 g of ascorbic acid in 100 mL of DI water. This solution should be prepared weekly to maintain its effectiveness in reducing iron.

8.2.3 Cerium(III)nitrate hexahydrate. Dissolve 0.155g in 100 mL DI water. (500 µg Ce/mL).

NOTE: This is normally purchased in the correct concentration from an approved vendor such as High Purity Standards.

8.2.4 Ethyl alcohol (80%). Dilute 400 mL ethanol to 500 mL with DI water.

8.2.5 Hydrochloric acid 0.1N. Dilute 8.3 mL of concentrated HCl to 1 liter with DI water.

8.2.6 Hydrofluoric acid concentrated (48%)

8.2.7 Ion exchange resin. Bio-Rad AG 1X8, chloride form, 100-200 mesh

8.2.8 Iron Carrier. 10 mg/mL

8.2.9 KSCN indicator (0.1M). Dissolve 0.972 g of potassium thiocyanate in 100 mL of DI water.

8.2.10 NIST traceable standards: U-232, Am-241, Am-243, Cm-244, U-238, Pu-242, Pu-239, Pu-238, Pu-236

8.2.11 Nitric acid concentrated 16M

8.2.12 2M Nitric acid/0.5M aluminum nitrate. Dissolve 93.8g of $Al(NO_3)_3 * 9H_2O$ in 0.5 liter of 2M HNO_3.

8.2.13 Nitric acid (2M) Dilute 12.5 mL of concentrated nitric acid to 100 mL of deionized water.

8.2.14 Titanous chloride. 20% reagent

9.0 Sample Handling and Preservation

9.1 Samples should be preserved to approximately pH 2 with nitric acid and collected in a plastic bottle.

9.2 Before beginning an analysis the analyst should check the sample pH with a pH strip. If necessary, adjust the pH with nitric acid to a pH=1-2. If the sample was pH adjusted let the sample sit overnight before continuing the batch.

9.3 If the sample has exceeded the hold time the analyst should contact the Group Leader before continuing with the batch.

9.4 Soil samples require no preservation and may be shipped in any suitable container.

10.0 Sample Preparation

Soil Sample Preparation

10.1 If not already done, homogenize the sample by performing SOP "Preparation of Soils for the Determination of Radionuclides" (GL-EPI-E-A021).

10.2 It is recommended that the samples be ashed in a muffle furnace as specified in SOP "Soil Sample Ashing for the Determination of Radionuclides" (GL-EPI-E-A021b).

10.3 For uranium analysis, take an appropriate aliquot and digest as specified in SOP "Digestion for Soils and Sand" (GL-EPI-E-A015).

10.4 A separate Am/Cm/Pu aliquot is analyzed with an aggressive acid leach as described in the following steps. Uranium should not be run by this leaching technique.

10.4.1 Place the sample in a beaker and add 10 mL 10 M HCl per gram of sample, with a minimum of 10 mL. Add the appropriate tracers as described in section 10.6.

10.4.2 Heat the samples and cover with a watch glass, allow to leach for a minimum of 2 hours. Agitate the sample periodically to enhance the leaching process.

10.4.3 Allow the sample to partially cool and transfer to a centrifuge tube. Centrifuge to separate the solid and leached portions of the sample. Decant the leachate to a clean labeled beaker and rinse the solid phase with DI water. Centrifuge the sample, combine the leachates and dilute with DI water. Proceed to section 10.7.

Aqueous Sample Preparation

10.5 Add an appropriate aliquot of sample to a labeled beaker. Add a certified dpm of the following tracers to each of the samples:

- For isotopic uranium, U-232 is normally used
- For isotopic americium and curium, Am-243 is used
- For isotopic plutonium, Pu-242 is normally used

10.5.1 If the analysis of the sample calls for quantification of U-232 or Am-243, the following steps shall be taken:

10.5.1.1 The sample will be run normally with the tracer indicated in section 10.6.

10.5.1.2 Review of the data shall be undertaken to determine if there are any peaks in the spectrum with which a ratio can be setup with the tracer isotope.

10.5.1.3 If there is a peak with which a ratio can be setup, then a second run of the sample shall completed with no tracer addition, and the ratios of peaks used to make corrections as described in the equations in section 15.4.

10.5.1.4 If no suitable peaks are available to ratio, a second run of the sample shall be made with a different tracer isotope such as U-236 or Am-241. The quantification of the isotope that was normally the tracer can then be made. If there is any quantifiable activity a correction can be made to the initial run by calculating a correction ratio for the tracer recovery of the first run from the second run results and following the equations outlined in 15.5.

NOTE: Other sample matrices, such as vegetation, air filters, tissue etc. are run as outlined in SOP "Preparation of Special Matrices for the Determination of Radionuclides" (GL-EPI-E-A026). The analyst must ensure that the appropriate tracer(s) are added to these other matrices as discussed in section 10.6.1.1-10.6.1.4.

10.6 Add 1 mL of iron carrier (10 mG/mL).

NOTE: For soil samples iron carrier may not be needed.

10.7 Bring to a slight boil and add concentrated NH_4OH until turbidity persists, or pH>9. Heat to near boiling for 10 minutes and then allow to settle and cool.

10.8 Decant excess supernate and discard. Collect the remaining precipitate by centrifugation in a 50 mL centrifuge tube and discard the supernate.

NOTE: Exercise care in this step because finely divided material which contains the actinides may also be present in addition to the large iron hydroxide flocks.

10.9 Dissolve the precipitate in 10-15 mL of 9M HCl/H_2O_2 solution.

10.10 Slurry AG 1x8 anion resin (Cl form 100-200 Mesh) in a squirt bottle with DI water. Transfer the resin to a small column to obtain a settled resin bed of 2.5 mL.

10.11 Condition the column with 10 mL of 9 M HCl.

10.12 Pass the sample solution from step 10.9 through the column and catch the effluent in a labeled, disposable 50 mL centrifuge tube for americium/curium analysis.

10.13 Rinse the column with 5 mL of 9 M HCl and catch with the Am/Cm fraction. Proceed to step 10.17 for Am/Cm analysis.

10.14 Rinse the column with an additional 10 mL of 9 M HCl and catch in a drip pan for disposal.

10.15 Elute plutonium by adding 15 mL of 6M HCl/0.52 M HF solution, catching the Pu elute in a labeled, disposable 50 mL centrifuge tube. Proceed to step 10.25 for plutonium microprecipitation source preparation for alpha spectroscopy.

10.16 Elute uranium from the column by adding 15 mL of 0.1 M HCl, catching the U elute in a labeled, disposable 50 mL centrifuge tube. Proceed to step 10.26 for uranium microprecipitation source preparation for alpha spectroscopy.

10.17 To the column elute from step 10.13, add 25 mL of DI water and 0.5 mL of iron carrier (10 mG/mL). Add concentrated ammonium hydroxide solution to pH > 9 to coprecipitate iron and actinides

10.18 Centrifuge the samples and decant the supernate into a proper waste container for disposal.

10.19 Rinse the precipitate with 10 mL of DI water that has been adjusted to pH 10 with concentrated ammonium hydroxide. Centrifuge and decant the supernate into a proper waste container for disposal.

10.20 Dissolve the iron precipitate in 10 mL of 2M HNO3/0.5M aluminum nitrate solution. Add 1 drop of KSCN indicator solution and swirl to mix. The solution color should become a deep red color, indicating the presence of Fe^{+3}.

10.21 Add 0.8 M ascorbic acid dropwise to the sample to reduce the iron valence to +2.

10.22 Precondition a 2 mL TRU spec column with 10 mL of 2 M HNO3, catching the rinse in a drip pan for disposal.

10.23 Pass the sample solution from step 10.21 through the column, catching the load solution in a drip pan for disposal. Rinse the column twice with 5 mL of 2 M HNO3 and catch the rinses in a drip pan for disposal.

10.24 Place a labeled, disposable 50 mL centrifuge tube under each column. Elute Am and Cm from the column using 2 mL of 9 M HCl, followed by 10 mL of 4 M HCl. Proceed to step 10.27 for americium microprecipitation source preparation for alpha spectroscopy.

10.25 To the plutonium solution from step 10.15, add 10 mL of DI water and 0.1 mL of cerium carrier solution (250 uL/mL). Add 3 drops of 25% dihydrazine dihydrochloride solution and swirl to mix. Let the solution sit for 10 minutes, then add 1.5 mL of concentrated hydrofluoric acid. Swirl to mix. Allow the solution to sit for 30 minutes, then proceed to step 10.28 for source preparation..

10.26 To the uranium solution from step 10.16, add 1 mL of titanium trichloride solution and allow the sample to sit for 30 seconds. Add 0.1 mL of cerium carrier solution (500 uG/mL) and swirl to mix. Add 1.5 mL of concentrated hydrofluoric acid to precipitate fluorides. Allow the solution to sit for 30 minutes, then proceed to step 10.28 for source preparation.

10.27 To the americium/curium solution from step 10.24, add 0.1 mL of cerium carrier solution (500 uG/mL) and swirl to mix. Add 1.5 mL of concentrated hydrofluoric acid and swirl to precipitate fluorides. Allow the solution to sit for 30 minutes, then proceed to step 10.28 for source preparation.

10.28 Place a 0.1 µm metricel filter on the filter funnel base.

10.29 Rinse the filter and funnel under vacuum with 5 mL of 80% ethanol. With minimum delay, add the sample to the filtering apparatus and rinse the beaker several times into the funnel with type II water. Complete the filtering by adding 5 mL of 80% ethanol.

10.30 Dry the filter under a heat lamp in a labeled petri dish. Label a 29 mm flat planchet with the applicable laboratory number and desired radionuclide. Care should be taken to center the filter and make it as flat as possible on the planchet.

NOTE: Care should be taken not to touch the active area of the filter with forceps.

10.31 Count under vacuum on the alpha spectrometer long enough to reach requested MDA. Consult the operating manual for instruction on operating the alpha spectrometer.

11.0 Preparation of Standard Solutions and Quality Control Standards

11.1 Refer to SOP "Preparation of Radioactive Standards" (GL-EPI-E-M001).

11.2 All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in SOP "Preparation of Radioactive Standards" (GL-EPI-E-M001), Section 19.0.

11.3 Primary standards are kept in the laboratory in a secured cabinet. Secondary, working, standards are kept at the bench area in an enclosed plastic cabinet.

12.0 Instrument Calibration and Performance

12.1 For direction on calibration and instrument performance see SOP "Micro-VAX 4000 Alpha Spectroscopy System" (GL-EPI-E-I009).

13.0 Analysis and Instrument Operation

13.1 For analysis and instrument operation see SOP "Micro-VAX 4000 Alpha Spectroscopy System" (GL-EPI-E-I009).

14.0 Equipment and Instrument Maintenance

14.1 For maintenance of system see "Counting Room Maintenance and Performance Checks" (GL-EPI-E-I010).

15.0 Data Recording, Calculation, and Reduction Methods

15.1 The instrument will report sample pCi/unit according to the following equation:

$$\text{pCi / unit} = \frac{S_{cpm} - B_{cpm}}{2.22 * E * V * A * \text{decay} * R}$$

15.2 Counting uncertainty is propagated according to the following equation:

$$\text{pCi / unit} = Ac * 1.96 \sqrt{\left(\frac{ef\_er}{E}\right)^2 + \left(\frac{pk\_er}{pk}\right)^2 + \left(\frac{ab\_er}{A}\right)^2 + \left(\frac{sy}{100}\right)^2 + (dk)^2}$$

15.3 The minimum detectable activity (MDA) is calculated according to the following equation:

$$MDA(pCi / unit) = \frac{2.71 + 4.65 * \sqrt{B_{cpm} * T_c}}{(2.22 * E * V * R * A * decay * T_c)}$$

where:

$$decay = \frac{1}{e^{\left(\frac{-\ln(2) T_d}{T_{1/2}}\right)}}$$

$$R = \frac{T_{cpm} - B_{cpm}}{T_{dpm} * E}$$

$$dk = \frac{T_{v2}err}{T_{v2}} * \left(\frac{\lambda Tr}{1 - e^{-\lambda Tr}} - \lambda\left(T_c + T_r\right) - 1\right)$$

And where:

Scpm	=	Sample counts per minute
Bcpm	=	Background counts per minute
E	=	Counting efficiency (decimal form)
V	=	Volume in liters,g,cfm, etc.
A	=	Isotopic abundance (decimal form)
ef_er	=	1 sigma efficiency error (decimal form)
pk_er	=	1 sigma peak error
ab_er	=	1 sigma isotopic abundance error (decimal form)

sy	=	1 sigma systematic error
pk	=	peak area
Tc	=	Sample count time in minutes
Td	=	Time interval for radioactive decay
Tr	=	Elapsed real time in minutes
Tcpm	=	Tracer counts per minute
Tdpm	=	Tracer known disintegrations per minute
Ac	=	Sample calculated activity
T1/2	=	Isotopic half life
T1/2err	=	Isotopic half life error
•	=	Isotopic decay constant
e	=	exponential function
ln	=	natural log function

15.4 This section describes the calculation of U-232 by a ratio method. This process uses two analyses of the same sample, one run traced with U-232 and one run untraced. A limitation of this method is that sufficient activity of a non tracer isotope (such as U-238) must be present to ratio the two peaks. Because of this limitation, the preferred method is to use a two tracer approach as described in 15.5.

15.4.1 Ratio determination: To set up a ratio between the peaks of the untraced sample run, use the following equation.

$$\text{Ratio} = \frac{U_{232u}}{U_{238u}}$$

15.4.2 The corrected yield of the U-232 traced sample is calculated as follows:

$$U_{232s} = U_{238i} * \text{Ratio}$$

$$U_{232t} = U_{232i} - U_{232s}$$

$$Yield_{corrected} = \frac{U_{232t}}{E * T_{dpm} * T_c}$$

where:

U232s	=	U-232 counts in the traced sample run due to sample activity.
U238i	=	total U-238 counts in the traced sample run
U232i	=	total U-232 counts in the traced sample run
U232t	=	U-232 counts in the traced sample run due to the tracer addition
U232u	=	U-232 counts in the untraced sample run
U238u	=	U-238 counts in the untraced sample run
E	=	Efficiency for the detector used in analysis
Tdpm	=	Known dpm of the U-232 tracer added
Tc	=	Time interval of the sample count in minutes

15.4.3 The final results are then corrected by substituting the corrected yield into the equations listed in sections 15.1 through 15.3.

15.5 This section describes the calculation of U-232 by an alternate tracer method. This process uses two analyses of the same sample, one run traced with U-232 and one run traced with U-236 (or another suitable standard).

NOTE: No corrections are necessary, if there is no U-232 activity in the U-236 traced run. If U-232 activity is present in the U-236 traced run, then the yield of the U-232 must be corrected as follows:

$$U_{232s} = U_{232f} * \left(\frac{eff_i}{eff_f}\right) * \left(\frac{counttime_i}{counttime_f}\right) * \left(\frac{squant_i}{squant_f}\right)$$

$$U_{232t} = U_{232obs} - U_{232s}$$

$$Yield_{corr} = \frac{U_{232t}}{(Eff_i * Counttime_i * U_{232tadded})}$$

Where:

U232s	=	U-232 counts in the traced sample run due to sample activity.
U232t	=	U-232 counts in U-232 traced analysis due to tracer addition
U232f	=	U-232 counts in the U-236 traced analysis
U232added	=	U-232 dpm added as the tracer
U-232obs	=	Total U-232 counts observed in the U-232 traced analysis
Effi	=	Efficiency of detector used in U-232 traced analysis
Eff_f	=	Efficiency of detector used in U-236 traced analysis
$Counttime_i$	=	Count time of U-232 traced analysis
$Counttime_f$	=	Count time of U-236 traced analysis
$Squant_i$	=	Sample aliquot of U-232 traced analysis
$Squant_f$	=	Sample aliquot of U-236 traced analysis
$Yield_{corr}$	=	Corrected Yield of U-232 traced analysis

15.5.4 The final results are then corrected by substituting the corrected yield into the equations listed in sections 15.1 through 15.3:

15.6 Record the following information on the alpha que sheet: preparation date, analysts initials, spike isotope, spike code, spike volume, LCS isotope, LCS code, LCS volume, nominal concentration LCS, and nominal concentration MS. For each sample record the detector number, sample mass, sample date, and sample time.

16.0 Quality Control Requirements

16.1 Analyst and Method Verification

16.1.1 Refer to SOP "Analyst and Analytical Methods Validation Procedures" GL-EPI-E-D002) for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank will accompany each batch of 20 or less samples. The reported value should be less than or equal to the CRDL for all target isotopes.

16.2.2 A matrix spike (MS) should be run with every batch of 20 samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\%\text{Rec} = \frac{\text{spike(pCi/unit)} - \text{sample(pCi/unit)}}{\text{spiked amount(pCi/unit)}} * 100$$

16.2.3 A sample duplicate should be run with every batch of 20 or less samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$\text{RPD} = \frac{\text{high sample(pCi/unit)} - \text{low sample(pCi/unit)}}{\text{Average (pCi/unit)}} * 100$$

16.2.4 A laboratory control spike (LCS) should be run with every batch of 20 samples or less. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\text{LCS} = \frac{\text{observed\_pCi/unit}}{\text{known\_pCi/unit}} * 100$$

16.3 Actions Required if the Quality Control Requirements Are Not Met

16.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the Group Leader and initiate a non-conformance report as outlined GEL SOP "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

17.0 Data Review, Approval, and Transmittal

17.1 Refer to EPI SOP "Data Review and Validation Procedures" (D-003) for instructions concerning the data review process, approval, and transmittal.

18.0 Records Management

18.1 Each analysis that is performed on the instrument is documented in the run log according to GEL SOP "Run Logs" (GL-LB-E-009).

18.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

19.0 Laboratory Waste Handling And Waste Disposal

19.1 Radioactive samples and material shall be handled and disposed of as outlined in SOP "Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011).

20.0 References

20.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

20.2 EML Procedures Manual HASL-300, 1982.

20.3 Analytical Chemistry. Rapid Determination of Th-230 in Mill Tailings by alpha spectroscopy. UNC Geotech, Grand Junction Projects Office. Steve Donivan, Mark Hollenbach, and Mary Costello. Vol. 59, No. 21, 1987.

20.4 Los Alamos Health and Environmental Chemistry: Analytical Techniques. LA-10300-M Vol. 1, September 1987.

20.5 Special thanks to Dr. Bill Burnett and his associates for assistance in developing this method at Florida State University.

STANDARD OPERATING PROCEDURE

FOR

DETERMINATION OF THORIUM

(GL-EPI-E-A-012 Revision 5)

HARD COPY IN ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories, Inc. and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

	08/31/99	Robert J. Train	
Process Owner	(Sign & Date)	(Print Name)	
	8/10/99	Richard M. Kinney	
Technical Review	(Sign & Date)	(Print Name)	
	9/7/99	George Best	
Quality Review	(Sign & Date)	(Print Name)	
	9/7/99	James B. Whitmoreland	
Approval and Authorization	(Sign & Date)	(Print Name)	

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR THE ISOTOPIC DETERMINATION OF THORIUM

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic thorium and uranium in a variety of matrices.

2.2 A sample is digested if necessary and aliquoted. Transuranic elements are scavenged by coprecipitation with iron hydroxide. The precipitate is dissolved and separation of elements is accomplished through extraction chromatography and ion exchange resins. The elements are then prepared for the measurement of radioactive isotopes by coprecipitation with cerium fluoride. The cerium fluoride precipitate is trapped on a filter, mounted on a stainless steel disk and placed in a partially evacuated chamber for measurement of isotopic alpha emission.

2.3 GEL utilizes methods that are derived from established sources. This method is based on the source method from DOE EML Methods Manual HASL 300 PU-02, 03 and uses similar principles of radiochemical separation and counting. This method is also very similar in concept to the source method from the DOE Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP800: "Sequential Separation of Americum and Plutonium by Extraction Chromatography."

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for Thorium and Uranium is 1 pCi/L or 1 pCi/g for all isotopes.

3.2 Method Precision: Typical relative percent difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value. Analyst training records are kept on hand in the Human Resources department.

4.0 DEFINITIONS

4.1 National Institute of Standards and Technology (NIST). For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.2 Type II water: Deionized water.

4.3 LIMS: Laboratory Information Management System. The database system used to store and report data.

5.0 METHOD VARIATIONS

5.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Sr. Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the laboratory. Individual sample MSDS forms provided by the clients are kept in Login.

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are nitrile gloves for concentrated acids and bases, and potassium ferricyanide in neat form. Work under a hood when using concentrated acids and bases.

6.4 The handling of radioactive samples is outlined in SOP GL-EPI-M-001, "Handling of Radioactive Samples." General guidelines include:

6.4.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

6.4.2 A plastic apron may be worn over the lab coat for added protection from contamination when working with radioactive samples.

6.4.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.4.4 Prohibit admittance to immediate work area.

6.4.5 Post signs indicating radioactive samples are in the area.

6.4.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in the radiochemistry laboratory.

6.4.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

6.5 Refer to SOP GL-EPI-E-S-011, "Laboratory Waste Disposal and Emergency Instructions," for instructions on how materials are disposed.

6.6 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the group leader prior to carrying out the rest of the procedure.

6.7 When handling biological samples protect the hands and forearms by wearing gloves and a laboratory coat to avoid contact of the biological material with the skin. Protect the eyes by wearing safety glasses and if desired a splash shield.

6.8 If cutting of meats or other tissue with cutlery is required, the analyst will wear a cutting glove of mesh steel over disposable gloves to avoid cuts that could infect the analyst with pathogens.

6.9 Any procedure that volatilizes biological substances such as drying or ashing, must be conducted in a hood or other suitable containment device.

6.10 Decontamination of work surfaces exposed to biological samples is performed by wiping the work area with a diluted (1:10) bleach solution and water as soon as possible following analytical operations.

6.11 Exterior protective clothing shall be removed prior to exiting the bioassay sample preparation area in order to prevent the inadvertent spread of biohazards to the rest of the laboratory.

6.12 Hands will be washed with an antibacterial soap directly after handling biological samples.

7.0 INTERFERENCES

7.1 Internal tracer standards may have ingrown daughters that may interfere with the analysis. For example, TH-228 will be present in aged U-232 standard. These problems are overcome by running separate aliquots of sample for thorium analysis.

7.2 Short lived radioactive progeny may ingrow on prepared filters. For example, the Ra-224 alpha peak will be present if the Th-228 parent is present. These interferences are minimized by counting samples as soon as possible after separation chemistry.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 Silicon surface barrier detectors with associated electronics, vacuum chambers, and data reduction capabilities.

8.1.2 Ion exchange columns.

8.1.3 Vacuum pump and filtration apparatus (25 mm)

8.1.4 Gelman metricel 25 mm filters with 0.1 um pore size

8.1.5 Gelman polypropylene 25 mm support filter

8.1.6 Stainless steel disks. 29 mm diameter

8.1.7 Stainless steel tweezers

8.1.8 Polypropylene centrifuge tube (50 mL)

8.1.9 Sample drying and ashing apparatus

8.1.10 Sample homogenization apparatus

8.1.11 AGIX8 anion exchange resin 100-200 mesh

8.2 Reagents, Chemicals and Standards

8.2.1 Ammonium hydroxide concentrated (14N).

8.2.2 Cerium(III)nitrate hexahydrate. Dissolve 0.155g in 100 mL DI water. (500 ug Ce/mL).

NOTE: This is normally purchased in the correct concentration from an approved vendor such as High Purity Standards.

8.2.3 Ethyl alcohol (80%). Dilute 400 mL ethanol to 500 mL with DI water.

8.2.4 Hydrochloric acid concentrated. (12M)

8.2.5 Hydroflouric acid concentrated (48%)

8.2.6 Iron Carrier. 10 mg/mL

8.2.7 9 M HCl – 1500 mls con HCl diluted to 2 L

8.2.8 NIST traceable Standards: Th-232, Th-229.

8.2.9 Nitric acid concentrated 16 M

8.2.10 Nitric acid (8 M). Dilute 50 mL of conc. Nitric acid to 100 mL DI water.

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 Samples should be preserved to approximately pH 2 with nitric acid and collected in a plastic bottle.

9.2 Before beginning an analysis the analyst should check the sample pH with a pH strip. If necessary, adjust the pH with nitric acid to a pH=1-2. If the sample was pH adjusted let the sample sit overnight before continuing the batch.

9.3 If the sample has exceeded the hold time the analyst should contact the group leader before continuing with the batch.

9.4 Soil samples require no preservation and may be shipped in any suitable container.

10.0 SAMPLE PREPARATION

Soil Sample Preparation

10.1 If not already done, homogenize the sample by performing GL-EPI-E-A-021 "Preparation of Soils for the Determination of Radionuclides."

10.2 It is recommended that the samples be ashed in a muffle furnace as specified in GL-EPI-E-A-021b "Soil Sample Ashing for the Determination of Radionuclides."

10.3 For thorium and uranium analysis, take an appropriate aliquot and digest as specified in GL-EPI-E-A-015, "Digestion for Soil and Sand."

Aqueous Sample Preparation

10.4 Add an appropriate aliquot of sample to a labeled beaker. Add a certified dpm of the following tracers to each of the samples: for isotopic thorium, Th-229 is normally used.

NOTE: Other sample matrices, such as vegetation. air filters, tissue etc., are run as outlined in "Preparation of Special Matrices for the Determination of Radionuclides" (GL-EPI-E-A-021b).

10.5 Add 1 mL of iron carrier.

NOTE: Iron carrier may not be needed for soil samples.

10.6 Bring to a slight boil and add concentrated NH_4OH until turbidity persists, or pH>9. Heat to near boiling for 10 minutes and then allow to settle and cool.

10.7 Decant excess supernate and discard. Collect the remaining precipitate by centrifugation in a 50 mL centrifuge tube and discard the supernate.

NOTE: Exercise care in this step because finely divided material, which contains the actinides may also be present in addition to large iron hydroxide flocks.

10.8 Dissolve the precipitate in 15 mls of HNO_3.

10.9 Slurry AC 1X8 anion exchange resin in DI water. Add the resin to an empty plastic column up to line on column neck.

10.10 Pass 15 mL of 8M HNO_3 through the column and discard the effluent.

10.11 Load the sample on the column and discard the effluent, which will remove any americium, curium or uranium from the sample. For Thorium, complete each line of the following check list, in the order specified.

THORIUM
Rinse solution
___2 mls 8 M HNO_3
___3 mls 8 M HNO_3
___5 mls 8 M HNO_3

Th Elution: catch in a clean centrifuge tube
___5 mls 9 M HCL
___5 mls 9 M HCL
___10 mls 9 M HCL
___10 mls 9 M HCL
___10 mls 9 M HCL

Th precipitation

___transfer to a beaker. add 1 ml FE carrier and take dry
___dissolve the residue with 2 mls of 9M HCL
___transfer to a clean centrifuge tube and dilute to 20 mls with DI
___0.1 mL of Ce carrier (500 µg/mL)
___3 mls HF, wait 20 minutes
___filter

10.12 Place a 0.1 um metricel filter on the filter funnel base.

10.13 Rinse the filter and funnel under vacuum with 5 mL of 80% ethanol. With minimum delay, add the sample to the filtering apparatus and rinse the beaker several times into the funnel with type II water. Complete the filtering by adding 5 mL of 80% ethanol.

10.14 Dry the filter under a heat lamp in a labeled petri dish. Label a 29 mm flat planchet with the applicable laboratory number and desired radionuclide. Care should be taken to center the filter and make it as flat as possible on the planchet. Note: care should be taken not to touch the active area of the filter with forceps.

10.15 Count under vacuum on the alpha spectrometer long enough to reach requested MDA. Consult the operating manual for instruction on operating the alpha spectrometer.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to "Preparation of Radioactive Standards," (GL-EPI-E-M001).

11.2 All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in "Preparation of Radioactive Standards," GL-EPI-M-001, section 19.0.

11.3 Primary standards are kept in the laboratory in a secured cabinet. Secondary, working standards are kept at the bench area in an enclosed plastic cabinet.

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 For direction on calibration and instrument performance see "Micro-VAX 4000 Alpha Spectroscopy System," (GL-EPI-E-I-009).

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 For analysis and instrument operation see "Micro-VAX 4000 Alpha Spectroscopy System." (GL-EPI-E-I-009).

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 For maintenance of system see "Counting Room Maintenance and Performance Checks," (GL-EPI-E-I-010).

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 The instrument will report sample pCi/unit according to the following equation:

$$\text{pCi / unit} = \frac{S_{cpm} - B_{cpm}}{2.22 * E * V * A * \text{decay} * R}$$

15.2 Counting uncertainty is propagated according to the following equation:

$$\text{pCi / unit} = Ac * 1.96\sqrt{\left(\frac{ef\_er}{E}\right)^2 + \left(\frac{pk\_er}{pk}\right)^2 + \left(\frac{ab\_er}{A}\right)^2 + \left(\frac{sy}{100}\right)^2 + (dk)^2}$$

15.3 The minimum detectable activity (MDA) is calculated according to the following equation:

$$\text{MDA(pCi / unit)} = \frac{2.71 + 4.65 * \sqrt{B_{cpm}} * T_c}{(2.22 * E * V * R * A * \text{decay} * T_c)}$$

where:

$$\text{decay} = \frac{1}{e^{\left(\frac{-\ln(2)Tc}{T1/2}\right)}}$$

$$R = \frac{T_{cpm} - B_{cpm}}{T_{dpm} * E}$$

$$dk = \frac{T_{1/2}e^{\pi}}{T_{1/2}} * \left(\frac{\lambda T_r}{1-e^{-\lambda T_r}} - \lambda(T_c + T_r) - 1\right)$$

And where:

Scpm	=	Sample counts per minute
Bcpm	=	Background counts per minute
E	=	Counting efficiency (decimal form)
V	=	Volume in liters,g,cfm, etc.

A	=	Isotopic abundance (decimal form)
ef_er	=	1 sigma efficiency error (decimal form)
pk_er	=	1 sigma peak error
ab_er	=	1 sigma isotopic abundance error (decimal form)
sy	=	1 sigma sytematic error
pk	=	peak area
Tc	=	Sample count time in minutes
Td	=	Time interval for radioactive decay
Tr	=	Elapsed real time in minutes
Tcpm	=	Tracer counts per minute
Tdpm	=	Tracer known disintegrations per minute
Ac	=	Sample calculated activity
T1/2	=	Isotopic half life
T1/2err	=	Isotopic half life error
λ	=	Isotopic decay constant
e	=	exponential function
ln	=	natural log function

15.4 Record the following information on the alpha que sheet: preparation date, analysts initials, spike isotope, spike code, spike volume, LCS isotope, LCS code, LCS volume, nominal concentration LCS, and nominal concentration MS. For each sample record the detector number, sample mass, sample date, and sample time.

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification

16.1.1 Refer to SOP "Analyst and Analytical Methods Validation Procedures" (GL-EPI-E-D002) for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank will accompany each batch of 20 or less samples. The reported value should be less than or equal to the CRDL for all target isotopes.

16.2.2 A matrix spike (MS) should be run with every batch of 20 samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\%Rec = \frac{spike(pCi/unit) - sample(pCi/unit)}{spikedamount(pCi/unit)} * 100$$

16.2.3 A sample duplicate should be run with every batch of 20 or less samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$RPD = \frac{highsample(pCi/unit) - lowsample(pCi/unit)}{Average\ (pCi/unit)} * 100$$

16.2.4 A laboratory control spike (LCS) should be run with every batch of 20 samples or less. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$LCS = \frac{observed\_pCi/unit}{known\_pCi/unit} * 100$$

16.3 Actions Required if the Quality Control Requirements Are Not Met

16.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the Group Leader and initiate a non-conformance report as outlined in "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

17.0 DATA REVIEW, APPROVAL AND TRANSMITTAL

17.1 The analyst turns in the raw data, review checklist, que sheet, spreadsheet, data report, NCRs (if applicable), and other appropriate information in a batch to the Report Specialist/Data Validator.

17.2 Analysts' data go through the following process of review:

17.2.1 The first level of review is the analyst review. The analyst will perform the following review procedure:

17.2.1.1 Visually check the que sheet, spreadsheet, raw data and data report to make sure the information has been transcribed correctly.

17.2.1.2 Check to see that the required detection limit (RDL) is met if required.

17.2.1.3 Complete the batch checklist and review special requirements.

17.2.2 The second level review is performed by the Report Specialist/Data Validator, who reviews the batch checklist and reviews the transcription.

17.3 After the review process is complete, the data is transmitted from the laboratory personnel to the reporting personnel as outlined in "Data Review and Validation Procedures," (GL-EPI-E-D003).

18.0 RECORDS MANAGEMENT

18.1 Each analysis that is performed on the instrument is documented in the run log according to GEL SOP "Run Logs" (GL-LB-E-009).

18.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

19.1 Radioactive samples and material shall be handled and disposed of as outlined in SOP "Laboratory Waste Disposal and Emergency Instructions," (GL-EPI-E-S-011).

20.0 REFERENCES

20.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

20.2 EML Procedures Manual HASL-300, 1982.

20.3 Analytical Chemistry. Rapid Determination of Th-230 in Mill Tailings by alpha spectroscopy. UNC Geotech, Grand Junction Projects Office. Steve Donivan, Mark Hollenbach, and Mary Costello. Vol. 59, No. 21, 1987.

20.4 Los Alamos Health and Environmental Chemistry: Analytical Techniques. LA-10300-M Vol. 1, September 1987.

STANDARD OPERATING PROCEDURE

FOR

UNCONTROLLED DOCUMENT

THE DETERMINATION OF GAMMA ISOTOPES IN WATER AND SOIL

(GL-EPI-A-013 Revision 4)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Process Owner	(Sign and Date) 7/13/99	SCOTT C McRELAND (Print Name)
Technical Review	(Sign and Date) 7/14/99	Richard M. Kinney (Print Name)
Quality Review	(Sign and Date) 7/14/99	Gene G. Bost (Print Name)
Approval and Authorization	(Sign and Date)	James B Coleman (Print Name)

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF GAMMA ISOTOPES IN WATER AND SOIL

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for Gamma Isotopes in water and in soil.

2.2 The Department of Energy (DOE) EML Procedures Manual source method number for Gamma Pha in soils and sediments is HASL-300. For water the method code is EPA 901.1.

2.3 Water samples are counted in Marinelli beakers. Soil samples are sealed in aluminum cans which are counted immediately if Ra-226 is not desired. If Ra-226 is desired, the sealed can is set aside to allow secular equilibrium between Rn-222 and Bi-214. Quantification is done by the abundance of the 609 KeV Bi-214 line.

2.4 This revision combines two related procedures. The following standard operating procedure is canceled without replacement having served its purpose:

2.4.1 "The Determination of Gamma Isotopes in Soil" (GL-EPI-E-A-013b).

2.5 General Engineering Laboratories, Inc. (GEL) utilizes methods that are derived from established sources. This method is based on the source procedures manual EPA 600 4-80-032 "Prescribed Procedures for Measurement of Radioactivity in Drinking Water" August 1980 Method 901.1 and uses similar principles of radiochemical counting.

3.0 METHOD APPLICABILITY

3.1 Minimum Detectable Activity (MDA): The MDA is based upon sample volume, instrument background, instrument efficiency, count time and other statistical factors, as well as specific isotopic values such as abundance and half-life.

3.2 Method Precision: If the activity is greater than 5 times the RDL (Required Detection Limit) an allowed method precision of equal to or less than 20% is used. For activity between the MDA and 5 times the RDL, an allowed method precision of 100% is used. There are no requirements if the activity is less than the MDA.

3.3 Method Bias (Accuracy): The method accuracy requirements for gamma spectroscopy is ñ25% of the true value.

3.4 Analysts go through a partnered training program with an already certified analyst for gamma spectroscopy. The analyst receives training on reviewing of standard analytical requirement such as RPD, method bias and technical review of gamma spectra. The analyst can then become qualified to perform the analysis by passing an unknown sample analysis and correctly identifying the isotope(s). Documentation of training is kept on file in the Human Resources department.

4.0 DEFINITIONS

4.1 Clean Line: An energy line of an isotope with no known energy lines of other isotopes within 2 KeV. (This excludes daughters that use the same line for quantification.)

4.2 Interfered Line: an energy line of an isotope with one or more energy lines of one or more different isotopes within 2 KeV.

4.3 Single and Double Escape Interference Lines: When high energy gamma lines above 511 KeV have a large emission rate, it is possible to see single and double escape lines caused by electron capture (energy line - 511 is a single escape line, energy line - 1022 is a double escape line.) For example, for 10,000 gps at 1332, the single escape interference line can be seen at 1332-511=821, and the double escape interference line at 1332-1022=310.

4.4 Summation Interference: When high gamma emission rates are seen, sample summation can occur. Prominent in geometries close to detection and in low energy range (i.e., 10,000 gps at 88 KeV, 15,000 gps at 210 KeV), a summation interference can be seen at 88+88=176 KeV, 210+210=420 KeV, 210+88=298KeV.

4.5 False Positive: An isotope that has failed one or more of several tests including half-life, abundance, and energy tolerance (± 2 KeV)

4.6 Abundance Test: The test where the software calculates the total possible lines from the library and checks to see how many were actually seen. The cutoff for a positive identification is 75%.

4.7 Energy Tolerance: The test where the software checks the energy line in the spectrum to see if it is within the energy tolerance setting. (The standard setting is 2 KeV.) If it is within this setting then the line is associated with that nuclide. The energy line can be associated with more than one nuclide.

4.8 Half-Life Test: The test to determine if the half-life of the isotope is long enough not to have decayed away. The half-life of the sample is the time from sample date to analysis date plus 1/2 the count time. A limit of no more than eight half-life is the standard setting.

4.9 Key Line: the line chosen by the builder of the library to be the prominent line of the isotope. This line is used in the MDA table for purposes of calculating activity, error and MDA. For non-identified isotopes the key line is used as the basis for calculating a region around the key line and then calculating and activity error and MDA. Usually this line is the most abundant line on a line that is relatively free from interference.

4.10 Abundance: The branching ratio or ratio of disintegration of the isotope at a particular energy. For example, Cobalt-60 has an abundance, or branching ratio, of 99% at 1332 KeV.

4.11 Accuracy: The error of the reported result due to the counting statistics of the instrument used for quantification.

4.12 Back Scatter: The detection of a count that occurs when an event interacts with counting materials, changes direction, and scatters back to the detector.

5.0 METHOD VARIATIONS

Not applicable.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Keep hands free from moving parts of canning device and Gamma shields.

6.2 Wear eye protection with side shields while in the laboratory.

6.3 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. Environmental Physics, Inc. (EPI) maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS).

7.0 INTERFERENCES

7.1 Some Gamma isotopes emit gamma lines that may overlap with other isotopes. If the energies of the two isotopes are within 2 KeV, the peaks may not be resolvable and will give a positive bias to the result. This problem is minimized by careful review of the peak search.

7.2 Soil samples may vary in density from the standard used for calibration. This may bias the results due to self absorption of lower energy (<100 K).

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 110 mL aluminum cans with lids for soil and miscellaneous samples

8.1.2 Snap Falcon PETRI dish for soil and miscellaneous samples

8.1.3 2 L, 500 mL, and 1000 mL Marinelli beakers for water samples

8.1.4 Air displacement pipette. 1 mL

8.1.5 Can annealing tool

8.1.6 Graduated cylinder

8.2 Reagents, Chemicals and Standards

8.2.1 NIST traceable mixed gamma standard in 100cc aluminum can

8.2.2 NIST traceable 1.8 liter mixed gamma standard in 2 L Marinelli beaker

8.2.3 NIST traceable mixed gamma standard in 0.5 L Marinelli

8.2.4 NIST traceable mixed gamma standard in snap falcon PETRI dish

8.2.5 Standard soil blank

8.2.6 NIST traceable aqueous Cs-137 standard

8.3 Instrumentation

8.3.1 High purity germanium detector, with associated electronics and data reduction software

8.3.2 Top loader balance

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 For soil samples, 500g of sample should be collected, preferably in a plastic container to avoid breakage.

9.2 For water samples, 2 liters of sample should be collected in a plastic container and preserved to pH2 with Nitric acid.

10.0 SAMPLE PREPARATION

10.1 Soil sample preparation.

10.1.1 Pour the sample into an aluminum baking pan and record the initial net weight on the que sheet (Appendix 1). Place the labeled pan in an oven at 110 degrees and dry overnight.

10.1.2 Homogenize the sample by blending and/or crushing.

10.1.3 Mix and weigh an aliquot of sample into a 100 mL can or a 500 mL Marinelli beaker if Radium analysis is not required, using a top loader balance. Fill the container completely, attach the lid, and record the net sample weight on the Gamma que sheet (Appendix 1).

10.1.3.1 If Radium analysis is required, mix and weigh an appropriate aliquot into a 100 mL can, using a top loader balance. Fill can completely, seal, and record net sample weight, seal date, and seal time on Gamma que sheet (Appendix 1).

NOTE: It is recommended that ingrowth be allowed 14 days to quantify Ra-226. Shorter intervals can be used at the request of the client. However, shorter ingrowth periods may decrease the accuracy of the data.

10.2 Water sample preparation

10.2.1 Mix and measure an appropriate volume into a 2 L, 500 mL or 1 L Marinelli beaker and record the volume on the Gamma que sheet (Appendix 1)

10.2.1.1 If Radium analysis is required, measure 100 mL and seal in a 100 mL can. Record volume, sealed date, and sealed time on Gamma que sheet.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to "Preparation of Radioactive Standards" (GL-EPI-E-M-001) for instructions concerning the preparation of standard solutions.

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 The gamma spectrometer should be calibrated for the appropriate geometry every 12 months or when daily QC check standards indicate instrument problems. Refer to "Micro-VAX 4000 Gamma Spectroscopy System Operating Procedure" (GL-EPI-E-I-001) for calibration instructions.

12.2 Refer to EPI SOP "Micro-VAX 3100 Gamma Spectroscopy System Operating Procedure" (GL-EPI-E-I001) for instructions concerning the Gamma Spectrometer.

12.3 Refer to EPI SOP "Counting Room Instrument Maintenance and Performance Checks" (GL-EPI-E-I010) for instructions concerning instrument maintenance.

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 Prepare the sample as outlined in section 10.0

13.2 Place the sample on the detector and count the sample an appropriate amount of time in the gamma shield. See EPI SOP "Micro-VAX 3100 Gamma Spectroscopy System Operating Procedure" (GL-EPI-E-I0011) for specific instructions on operating the gamma spectrometers.

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 Refer to EPI SOP "Gamma Spectroscopy System Operating Procedure" (GL-EPI-E-I001) for instructions concerning the Gamma Spectrometer.

14.2 Refer to EPI SOP "Counting Room Instrument Maintenance and Performance Checks" " (GL-EPI-E-I001) for instructions concerning instrument maintenance.

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 Data Recording

15.1.1 Record the following information on the Gamma Que Sheet: preparation date, analysts initials, spike isotope, spike code, spike volume, LCS isotope, LCS code, LCS volume, nominal concentration LCS, and nominal concentration MS. For each sample record the detector number, sample mass, sample date, and sample time (Appendix 1).

15.2 The instrument will report sample pCi/g or pCi/L according to the following equations:

$$\text{Sample pCi/g} = \frac{A * d}{2.22 * E * V * B}$$

$$\text{Sample pCi/L} = \frac{A * d}{2.22 * E * V * B}$$

15.3 Counting uncertainty is calculated according to the following equation:

$$\text{pCi/unit} = Ac * 1.96 \sqrt{\left(\frac{ef - er}{E}\right)^2 + \left(\frac{pk - er}{pk}\right)^2 + \left(\frac{ab - er}{A}\right)^2 + \left(\frac{sy}{100}\right)^2 + (\text{Decay})}$$

Where:

$$\text{Decay} = \left(\frac{T_{1/2err}}{T_{1/2}}\right)^2 * \left[\frac{\lambda Er}{1 - e^{-\lambda Er}} - \lambda\left(T_s + Er\right) - 1\right]$$

15.4 The method MDA in pCi/g or pCi/L are calculated according to the following equations:

$$MDA\,(pCi/unit) = \frac{d * \left(2.71 + 4.66\sqrt{cpm_b * ct} \right)}{2.22 * E * V * B * ct}$$

Where:

A = net peak area (counts per minute)
ABS = relative absorption factor applied if Pb-210 or U-238 are requested
B = abundance (gammas/disintegration)
E = counting Efficiency (counts/gamma)
V = sample volume (grams or liters)
ct = sample count time (minutes)
d = decay factor

15.5 The absorption factor is calculated by the following equations:

$$I_1 = \frac{\ln((SScpm - Scpm)/ECcpm)}{(((SScpm - Scpm)/ECcpm) - 1}$$

$$I_0 = \frac{\ln((SSTcpm - STcpm)/ECcpm)}{(((SSTcpm - Scpm)/ECcpm) - 1)}$$

$$ABS = \frac{I_1}{I_0}$$

Where:

SScpm = sample plus the source cpm at the region of interest
Scpm = sample cpm at the region of interest
ECcpm = source cpm on the empty can at the region of interest
ln = natural logarithm
SStcpm = standard plus the source cpm at the region of interest
Stcpm = standard cpm at the region of interest

15.6 The VAX operating system will report the following information with each completed sample:

15.6.1 The nuclide identification report

15.6.2 The minimum detectable activity report

15.6.3 The peak search report.

15.7 The following criteria are used to accept a reported gamma isotope from the NID report:

15.7.1 The peak FWHM should be less than 2 KeV.

15.7.2 The activity of a non-target isotope will not be reported unless it is greater than the minimal detectable activity of a method blank with similar volume and count time.

15.7.3 The energy tolerance should be between 2 and 3 KeV.

15.7.4 The sensitivity setting should be between 0.1 and 3. The default setting is 3.

15.7.5 Start channel on peak search should be 50 and end channel should be 4096.

15.7.6 The confidence level setting should be 5.

15.7.7 These settings should not be changed without first checking with group leader.

15.8 The following guidelines are used to accept unidentified lines on the peak search after environmental background subtraction:

15.8.1 The line matches the natural fingerprint of the Uranium-238 or Thorium-232 decay chains (i.e. 63, 75, 93, 239, 295, 352, 511, 609, 1120, etc.).

15.8.2 The line matches as a summation peak from two other lines in the spectrum.

15.8.3 The line has a net area of less than 20.

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification

16.1.1 Refer to EPI SOP "Analyst and Analytical Methods Validation Procedures" (G-EPI-E-D003) for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank will accompany each batch of 20 or less samples. The reported value should be less than or equal to the CRDL for all target isotopes.

16.2.2 For water samples only, a matrix spike (MS) should be run with every batch of 20 samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\%\text{REC} = \frac{\text{spike(pCi/g)} - \text{sample(pCi/g)}}{\text{spikedamount(pCi/g)}} * 100$$

or:

$$\%\text{REC} = \frac{\text{spike(pCi/L)} - \text{sample(pCi/L)}}{\text{spikedamount(pCi/L)}} * 100$$

NOTE: Performing a matrix spike on a soil sample would result in direct contamination of the sample, therefore, only water samples require an MS.

16.2.3 A sample duplicate should be run with every batch of 20 or less samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$RPD = \frac{\text{high sample (pCi/g)} - \text{low sample (pCi/g)}}{\text{Average (pCi/g)}}$$

or:

$$RPD = \frac{\text{high sample (pCi/L)} - \text{low sample (pCi/L)}}{\text{Average (pCi/L)}}$$

16.2.4 A laboratory control spike (LCS) should be run with every batch of 20 samples or less. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\text{LCS} = \frac{\text{observed\_pCi/g}}{\text{known\_pCi/g}} * 100$$

or:

$$LCS = \frac{observed\_pCi/L}{known\_pCi/L} * 100$$

16.3 Actions Required if the Quality Control Requirements Are Not Met

16.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non conformance report as outlined in GEL SOP "Documentation of Nonconformance Reporting and Dispositioning. and Control of Nonconforming Items" (GL-QS-E-004).

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

17.1 The first level of review is the analyst review. The analyst will perform the following steps of review:

17.1.1 Visually check the que sheet, spreadsheet, raw data and data report to make sure the information has been transcribed correctly.

17.1.2 Review the raw data to see if there are any hits not on the requested list in LIMS. If there are, report to the client by adding the information into LIMS.

18.1.2.1 A true identification or a "hit" is any isotope greater than 10 pCi/L or 5 pCi/g on the identified nuclide list. The error must also be less than 40% of the result and not have interference by another isotope or have a very short half-life.

17.1.3 Check to see that the required detection limit (RDL) is met if required.

17.1.4 Check hits to see if they are true hits (see 18.1.2.1) and not an interference or a false positive.

17.1.4.1 Identifications are classified into two categories: false positives (interference), and true identification (hit). The false positives are rejected by checking the abundance test results for the isotope and by checking last results for the half-life. The result is considered interference and rejected by checking to see if there are any clean lines in sample spectrum for the isotope. If none exist. then the identification is rejected. If the key line has a possible interference and secondary lines do not confirm the activity calculation, the identification is rejected. Isotopes that

pass these criteria are accepted as true identifications. The above tests and criteria are standard and will be followed unless directed otherwise by contract, specification or instructions.

17.1.5 Complete the batch checklist.

17.2. The second level review is performed by the Data Validator or Report Specialist, who reviews the batch checklist, checks requested and non-requested hits, and reviews the transcription.

17.3 After the review process is complete, the data is transmitted from the laboratory personnel to the reporting personnel as outlined in "Data Review and Validation Procedures" (GL-LB-E-D-003).

18.0 RECORDS MANAGEMENT

18.1 Each analysis that is performed on the instrument is documented in the run log according to GEL SOP "Run Logs" (GL-LB-E-009).

18.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

19.1 All soil sample cans are opened and sample returned to original sample containers after completion of batch.

19.2 Radioactive waste is disposed of as outlined in "EPI Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S-011), and the GEL Laboratory Waste Management Plan (GL-LB-G-001).

20.0 REFERENCES

20.1 USEPA. Prescribed Procedures for Measurement of Radioactivity in Drinking Water. Method 901.1, August 1980.

20.2 Canberra Nuclear Genie System Spectroscopy, Applications and Display User's Guide. Vol. I and II, May 1991.

20.3 EML procedures manual. HASL-300-Ed.25, 1982.

Appendix 1

Gamma Spec Que Sheet comment: Radioactive

Batch # 34729 Prep Date:_____ Initials: _____
Spike Isotope:_____ Spike Code:_____ Vol:_____
LCS Isotope:_____ LCS Code:_____ Vol:_____
Nom Conc LCS:_____ Nom Conc MS:_____

gamma1	5933088	gamma3	12922955	gamma5	8932581
gamma2	11912863	gamma4	11912863	x-ray	12922782

Sample ID	*Sample Description*	*Detector #*	*Sample Mass (mls or g)*	*Sampling Date*	*Sampling time*
9307287-09	HSB101D			7/13/93	1303
9307411-04	HSB114D			7/20/93	1141
9307411-05	HSB115C			7/20/93	0925
9307411-06	HSB115D			7/20/93	0906
9307411-07	HSB112E			7/20/93	0822
9307411-08	HSB118A			7/20/93	1403
9307434-03	HSB116C			7/21/93	0858
9307434-04	HSB125D			7/22/93	1203
9307434-05	HSB129D			7/21/93	1229
9307434-06	HSB136C			7/22/93	0943
QC135727	blank				
QC135728	9307434-06				
QC135729	9307434-06				
QC135730	lcs				

Batched To: IDXL Automatic

Data Reviewed By: _____

Total Radioactivity in Contact Waste

Prepared by:	Signature:	James B. Westmoreland James B. Westmoreland
Technical Review by:	Signature:	Robert L. Pullano Robert L. Pullano
Sr. Management Review by:	Signature:	James T. Chambers James T. Chambers
Approved & Authorized by:	Signature:	Heyward H. Coleman Heyward H. Coleman

SET

1. Method Objective and Purpose

This standard operating procedure provides the neccessary instructions to conduct the analyses for total radioactivity in contact waste.

2. Method Summary

The majority of radioactive material received into the laboratory contains tritium. Because tritium is difficult to detect with conventional screening techniques, a method is needed to extract tritium and other radioisotopes into an aqueous phase for liquid scintillation counting. The method is summarized as follows: The contact waste is initially screened with a portable survey meter to detect any exposure above a background reading. A representative sample is removed from the contact waste stream, and is placed in a beaker. DI water is added to cover the sample, and the radioisotopes are leached in DI water with occasional stirring. The leachate is then decanted and brought to a measured volume. 4 mls of the leachate is added to 16 mls of scintillation cocktail in a scintillation vial. The vial is inverted several times and allowed to sit in the dark overnight. The vial is counted in a liquid scintillation counter.

3. Method Performance Characteristics

3.1) Method Detection Limit: The MDL is 0.1 nCi/g
3.2) Method Precision. Expected precision for the method is less than 20% on duplicated samples.
3.3) Method Bias. Expected recoveries for the method are 100% plus or minus 25%.

4. Method References

4.1) Prescribed Procedures for Measurement of Radioactivity in Drinking Water, USEPA, Method 906, Aug. 1980.
4.2) Standard Methods for the Examination of Water and Wastewater, 14th edition, American Public Health Association, Washington, DC (1976).

5. Method Interferences

Samples with color or chemical quenching agents may reduce the counting efficiency. Generally these problems are overcome by diluting the sample aliquot.

6. Safety Precautions

6.1) Care should be taken to avoid the spread of radioactive contamination that may be present within the trash bags.

6.2) A survey of the area should be performed upon completion of waste handling. Refer to the radioactive survey standard operating proceedure for guidance on radioactive survey.

7. Apparatus and Materials

7.1) Ancillary Equipment:

7.1.1) 20 ml plastic scintillation vials
7.1.2) 5 ml Eppendorf pipet
7.1.3) Whatman 41 filters.

7.2) Reagents, Chemicals and Standards

7.2.1) Type II deionized water.
7.2.2) Liquid scintilation cocktail. Beckman Ready Safe or equivalent.
7.2.3) NIST traceable tritium solution.

7.3) Instrumentation

7.3.1) GM survey meter. (Eberline E-120 or equivalent)
7.3.2) Liquid Scintillation Counter. (Beckman LS5000TD or equivalent)

8. Sample Handling and Preservation Requirements

Samples are collected without preservation in plastic bags.

9. Sample Preparation Procedure

9.1). Remove all the bags of contact waste from the drum and weigh to determine the gross weight of material. Record on the que sheet. Slowly(approximately 1 inch per second) pass the survey meter within an inch of the surface of the contact waste. Any waste that registers above 0.5 milli roentgen per hour should be separated and labeled as radioactive.

9.2) Remove a representative aliquot from each waste bag and mix in a suitable container. Homogenize the new aliquot by mixing the material in the container.

9.3) Weigh a representative aliquot into a beaker. Record the weight. Add enough water to saturate and cover the waste material. Stir the material intermittently for one hour.

9.4) Decant the liquid into a graduated cylinder and dilute to 250 mls. (Note: If the solution is high in particulates the liquid should be filtered through a whatman 41 filter.)

9.5) Place the solution in a plastic labeled bottle and hold for liquid scintillation counting.

9.6) Place 4.0 mls of the solution in 16 mls of cocktail. Cap the vial tightly and shake vigorously. Check for complete dissolution of sample in cocktail. (Note: a milky appearance is an indicator of cocktail loading. A volume size should be chosen so that the vial appears clear.)

9.7). Allow the samples to sit in the dark overnight and then count for 45 minutes in the LSC under the program "contact".

10. Equipment and Intrument Maintenance

Please refer to SOP I-004 for instructions concerning instrument maintenance.

11. Preparation of Standard Solution and Quality Control Samples

11.1) The standard solution is prepared by diluting a primary solution. The primary solution is weighed on an analytical balance and recorded in the standard log. The weighed aliquot is then diluted to 100mls in a volumetric flask. The working solution activity is calculated in dpm/ml according to the following equation:

Primary soln (uCi/g)*1e-6 (pCi/uCi)*(2.22 dpm/pCi)*(1/100mls)*($e^{-0.693*t/t_{1/2}}$)

where: t = time in days from the standard calibration to the current date
$t_{1/2}$ = half life of tritium in days (4490)
e = natural log (ln)

11.2) The matrix spike is prepared by adding a known amount of the working

solution to the sample aliquot.

12. Calibrations

12.1) Instrument calibration: Refer to SOP I-004 "Liquid Scintillation Counter Operating Instructions" for guidance concerning instrument calibration.

12.2) Initial Calibration: Refer to SOP I-004 "Liquid Scintillation Counter Operating Instructions" for guidance concerning instrument calibration.

12.3) Method Quench Calibration: The method is calibrated by spiking with a known dpm of tritium activity. The activity is added to samples of increasing quench. Quench is obtained by the addition of chloroform. A quench curve is derived by plotting percent efficiency versus the quench factor(H#).

13. Instrument Performance Requirements

13.1) Instrument Performance check (IPC). The instrument response is validated each day it is used by the IPC. A carbon-14 glass vial is counted under the tritium window to track the instrument response to a known activity. The carbon-14 vial is the IPC and the acceptance limits are determined by calculating a 3 sigma statistical range above and below the mean. Results of the daily IPC are recorded on a chart for review.

13.2) Instrument Blank: The instrument is checked for contamination each day it is used by the instrument blank. A water filled glass vial is counted under the tritium window to track the background counts. The instrument blank acceptance limits are determined by calculating a 3 sigma statistical range above and below the mean. Results of the daily instrument blank are recorded on a chart for review.

14. Analyst and Method Verification Requirements

Refer to SOP D-002 for information concerning analyst and method verification.

15. Analysis Procedure

The analysis of the data is performed using a spreadsheet created in Microsoft Excel. The analyst will enter the sample identification, the weighed aliquot, the sample counts per minute, and the final diluted volume into the appropriate cells. The

spreadsheet will automatically generate a pCi/g result.

16. Calculations

The spreadsheet uses the following equation to calculate the total radioactivity in pCi/g:

T-radioactivity = $(A * V) / 2.22 * C * g * 4$

Uncertainty= $1.96*V*[\underline{(bkg\ cpm/ct) + (sam\ cpm/ct)}]1/2/\ 2.22 * C * g * 4$

Where:
A = net beta count rate (gross LSC count rate minus the background count rate) at the 0-1000 channel window.
C = counting efficiency. (Based on tritium).
V = volume the sample aliquot was diluted to(mls).
g = grams of sample taken for analysis.
2.22 = conversion factor from dpm/pCi.
4 = volume added to the scintillation vial.

17) Quality Control

17.1) A matrix spike(ms) will be run with every batch of samples. The recovery of the spikes should fall between 75 and 125%. The recovery is calculated as follows.

$$REC = \frac{SPIKE\ (pCi/g) - SAMPLE\ (pCi/g)}{NOMINAL\ CONCENTRATION\ (pCi/g)} * 100$$

17.2) A duplicate will be run with every batch of samples. The relative percent difference (RPD) between the sample and duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$RPD = \frac{HIGH\ MS\ (pCi/g) - LOW\ MS\ (pCi/g)}{AVERAGE(pCi/g)} * 100$$

17.3) A method blank will accompany each batch of samples. The reported value should be less than or equal to 0.1 nCi/g.

STANDARD OPERATING PROCEDURE

FOR

MICROWAVE DIGESTION FOR SOIL AND SAND

Process Owner(s):	Tim C. Winters, EPI Team Task Leader
Technical Review by:	Barry D. Stewart, EPI Senior Technical Specialist
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Management Review by:	James B. Westmoreland, EPI Laboratory Manager
Approved and Authorized by:	Heyward H. Coleman, EPI President

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR MICROWAVE DIGESTION FOR SOIL AND SAND

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct microwave digestion on soil type matrices.

2.2 A soil sample is digested in a microwave digestion bomb under high pressure and temperature conditions using nitric acid in conjunction with hydrofluoric and hydrochloric acids. The digestate is transferred to a Teflon® beaker and evaporated to dryness to remove excess hydrofluoric acid and silicon tetrafluoride. The residue is boiled in nitric acid-boric acid to convert fluoride salts to nitrates. Since hydrofluoric acid and nitric have the same boiling point, i.e. 120°C, it is impossible to convert fluorides to nitrates through evaporation with nitric acid alone.

2.3 This procedure is applicable to soils for any analysis requiring complete solution. Sample aliquots of one gram or less can be accommodated by the equipment and amount of reagents specified. Sequential analysis of more than one radionuclide may be accomplished by the addition of carriers and tracers for each applicable analysis prior to digestion.

2.4 To minimize errors due to non-homogeneity of a sample, it is always desirable to use the largest possible aliquot of sample. The existence of "hot particles" may lead to questionable data when very small sample aliquots are used. In cases where radionuclide concentrations are "higher than normal environmental levels," and smaller aliquots are necessary it is preferable to dilute the digested 1-gram sample rather than select a smaller aliquot. The analyst should consult the group leader for guidance in these situations.

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit (MDL): See specific SOP for minimum detectable activity (MDA) values.

3.2 Method Precision: Typical relative percent difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is +/-25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within +/- 5% of true value. Analyst training records are kept on hand in the Human Resource Department.

4.0 DEFINITIONS

4.1 National Institute of Standards and Technology (NIST): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.2 Type II water: Deionized water.

4.3 LIMS: Laboratory Information Management System. The database system used to store and report data.

5.0 METHOD VARIATIONS

Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Senior Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 **WARNING: Under no circumstances should a sample containing volatile organic compounds be digested with this procedure.** These types of samples should be processed using EPI SOP "The Preparation of Special Matrices for the Determination of Radionuclides" (A-026).

6.2 Use caution when dealing with acids, and any other laboratory reagents. Safety procedures for these are set forth in the Handbook for Good Laboratory Practices, found in the library of the laboratory.

6.3 Microwave digestion bombs require the special attention of the analyst involved. They operate under pressures above 100 psi, combined with concentrated acids at high temperatures.

6.4 The analyst should ensure the rupture disk is in place prior to closure of vessel.

6.5 If there is any question regarding the safety of any laboratory practice, **stop immediately**, consult the group leader prior to carrying out the rest of the procedure.

6.6 Between digestion phases, microwave digestion bombs should only be opened under a fume hood.

6.7 Wear eye protection with side shields while in lab. Gloves are required when handling the chemicals in this procedure.

6.8 Refer to EPI SOP "Radioactive Waste Handling Procedures" (S-005) for instructions on how materials are disposed.

6.9 Refer to EPI SOP "Handling of Radioactive Samples" (M-001) for instructions on handling radioactive samples.

7.0 INTERFERENCES

Not Applicable.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Teflon® beakers.

8.2 Microwave Oven. 1.2 cu. ft., 700 watt with carousel.

8.3 Microwave Digestion Bombs. Teflon® lined available from CEM Inc.

8.4 Reagents, Chemicals and Standards

8.4.1 Nitric acid, reagent grade.

8.4.2 Hydrofluoric acid, 48%.

8.4.3 Hydrochloric acid, reagent grade.

8.4.4 1:1 Nitric acid, prepared by adding 500mL concentrated HNO_3 to 500 mL Type II water.

8.4.5 Type II water.

8.4.6 Boric acid, 5%. Dissolve 50 grams of $H_3 BO_3$ per liter of water.

9.0 SAMPLE HANDLING AND PRESERVATION

Soil and Sand require no preservation and may be shipped in any suitable container.

10.0 SAMPLE PREPARATION

10.1 For instructions on drying, grinding, homogenizing, and blending soil samples, refer to EPI SOP "Soil Sample Preparation for the Determination of Radionuclides" (A-021).

Transfer a weighed aliquot (three decimal places) of soil to a digestion bomb. Add nitric acid as prescribed in the appropriate section of the attached Microwave Digestion Table (Table 1).

10.2 Assemble the microwave digestion bombs.

10.2.1 Insert the Teflon® liner with the sample into the Kevlar® liner.

10.2.2 Place a new rupture disk into the Teflon® cap.

10.2.3 Tighten the Teflon® cover bolt into the Teflon® cap.

10.2.4 Place the Teflon® cap on the Teflon® liner with the Kevlar® liner.

10.2.5 Place the plastic lid over the Teflon® cap, and tighten to a snug fit

10.3 Repeat assembly for the desired number of samples to be digested (maximum of 10). Place the digestion bombs evenly spaced in the microwave carousel. Attach vent tubes to the bombs so any slow leakage can be directed into the carousel reservoir.

10.4 In the event a rupture disk gives way and is venting fumes, immediately shut off microwave. **Do not open the microwave.** Allow the vessel to finish venting, then the microwave door may be opened to ventilate it and prevent further damage to the unit. After the digestion bomb has been given enough time to cool down, remove it from microwave, and determine if sample loss was significant, annotate as necessary in run log or on batch que sheet. Inform Group Leader of the occurrence. Restart the digestion process from the point where the microwave was shut off if there is no further danger of over pressurization.

10.5 Utilize Table 1 to determine the operating parameters for microwave settings. (See Table 1, Note 2).

10.6 When the digestion process is completed remove the digestion vessels from the microwave. Cool for 10 minutes.

10.7 Open the bomb and transfer the sample to labeled Teflon® beaker. Evaporate to dryness.

10.8 Add 10 mL of 8M HNO_3 and 2 mL of 5% boric acid. Cover and boil for 15 minutes. Cool until the beaker can be handled and transfer the sample to a glass beaker.

10.9 Examine the solution carefully for the presence of undissolved material. Coarse particles of sand are likely to be present and should be disregarded. A trace of turbidity or white crystalline material is cause for concern since it may be due to rare earth fluorides which were not dissolved in Step 10.8. If no finely divided material is present, continue with Step 10.11.

NOTE: One gram of soil will contain sufficient rare earths to effectively scavenge non-oxidizable elements such as Am and Th, co-precipitating them with the rare earth fluorides. Pu can also be lost during this step if the digestion procedure did not oxidize Pu to the +6 oxidization state. If turbidity is present, it should be assumed that the insoluble material may contain most, if not all, of the trans-uranium elements. If the turbidity is due to undissolved silica, it should not adversely affect chemical yields, but it may partially clog the Eichrom columns used in subsequent analyses. In either event, the insoluble material must be dissolved.

10.10 If insoluble material is still present, transfer the sample back to the original Teflon® beaker. Add 0.5 mL of HF and 1 mL of HNO_3, and evaporate gently to dryness. Add 10 mL of 8M HNO_3 and 3 mL of 5% boric acid. Cover and boil for 15 minutes.

10.11 If much coarse material is present, it may be removed by centrifuging. Transfer the supernate back into the glass beaker and evaporate to dryness. Dissolve the residue in 10 mL of the reagent appropriate for the analysis to be performed. For example, for the determination of U, Am and Cm in soils by SOP A-011B, use 10 mL of 2M HNO_3 - 0.5M $Al(NO3)_3$, or 10mL of 8M HNO_3 for the determination of Sr-90 by SOP A-004.

TABLE 1

Microwave Digestion Table for Soils and Sands[1]
Power Rating of 700 watts

For 1-3 digestion bombs:

1. Phase I: Add 8 mL of HNO_3
   a. 2 min at 80% power
   b. 3 min at 50% power
   c. 10 min cool down in hood

2. Phase II: Add 10 mL HF & 5 mL HCL
   a. 3 min at 40% power
   b. 4 min at 30% power
   c. 10 min cool down in hood

For 4-5 digestion bombs:

1. Phase I: Add 8 mL of HNO_3
   a. 5 min at 80% power
   b. 8 min at 50% power
   c. 10 min cool down in hood

2. Phase II: Add 10 mL HF & 5 mL HCL
   a. 6 min at 50% power
   b. 7 min at 30% power
   c. 10 min cool down in hood

For 6-8 digestion bombs:

1. Phase I: Add 8 mL of HNO_3
   a. 6 min at 80% power
   b. 8 min at 60% power
   c. 10 min cool down in hood

2. Phase II: Add 10 mL HF & 5 mL HCL
   a. 6 min at 60% power
   b. 7 min at 40% power
   c. 10 min cool down in hood

TABLE 1 (CONT'D)

Microwave Digestion Table for Soils and Sands[1]
Power Rating of 700 watts

For 9-10 digestion bombs:

1. Phase I: Add 8 mL of HNO_3
   a. 7 min at 80% power
   b. 10 min at 60% power
   c. 10 min cool down in hood

2. Phase II: Add 10 mL HF & 5 mL HCL
   a 6 min at 70% power
   b 8 min at 50% power
   c 10 min cool down in hood

**NOTES:

1. Between digestion phases and at completion the bomb should only be opened under a fume hood.

2. Alterations to these parameters regarding additional time or power are completely acceptable if the analyst feels it is necessary to achieve complete sample digestion. Lowering power or time however, should rarely be necessary, but sample rupture may warrant lowering parameters in the event that lowering sample weight would be impractical.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to EPI SOP "Preparation of Radioactive Standards" (M-001).

11.2 All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in EPI SOP "Preparation of Radioactive Standards" (M-001), Section 19.0.

11.3 Primary standards are kept in the laboratory in a secured cabinet. Secondary, working standards are kept at the bench area in an enclosed plastic cabinet.

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

Not Applicable.

13.0 ANALYSIS PROCEDURES AND INSTRUMENT OPERATION

Not Applicable

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 The digestion vessel should be rinsed with type II water after sample transfer has been completed. The Teflon® cup may then be washed with a soft sponge and soap. The vessel can then be soaked in a low concentration nitric acid bath for 24 hours to reduce staining of cup walls.

14.2 The Teflon® cover should be disassembled and the rupture disk removed. The cover and bolt should then be rinsed with type II water then they may also be soaked in the nitric acid bath.

14.3 Please see operation instructions of microwave oven for maintenance instructions.

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Not Applicable.

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification

Refer to EPI SOP "Analyst and Analytical Methods Validation Procedures" (D-002) for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

See specific SOP for specific method quality control requirements.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

Not Applicable.

18.0 RECORDS MANAGEMENT

18.1 Each analysis that is performed on an instrument is documented in the run log according to GEL SOP "Run Logs" (GL-LB-E-009).

18.2 All raw data printouts, calculation spreadsheets and batch checklists are filedwith the sample data for archival and review.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Radioactive samples and material shall be handled and disposed of as outlined in EPI SOP "Radioactive Waste Handling Procedures" (S-005).

20.0 REFERENCES

20.1 "Standard Practice for the Microwave Digestion of Industrial Furnace Feed Sreams for Trace Element Analysis": ASTM

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1.0 The Determination of Polonium in Water

Process Owner:	Signature:	Wilbur B. Sigmon, III
Technical Review by:	Signature:	James B. Westmoreland
Quality Review by:	Signature:	Robert L. Pullano
Approved & Authorized by:	Signature:	Heyward H. Coleman

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2.0 Method Objective and Applicability

2.1) This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic alpha emitting polonium in water.

3.0 Interferences

3.1) Undissolved sample residue may result in quenching of alpha particles if the silver disk is left upright upon bottom of beaker. If undissolved residue is visible then the disk should be left upright facing in from the side of the beaker to prevent baking of residue onto the disk.

4.0 Safety Precautions and Hazard Warnings

4.1) Caution should be used when dealing with acids, and other any laboratory reagents. Safety procedures for these are set forth in the Handbook for Good Laboratory Practices, found in the library of the laboratory.

4.2) If there is any question regarding the safety of any laboratory practice, **stop immediately** and consult the group leader prior to carrying out the rest of the procedure.

5.0 Apparatus and Materials

5.1) Ancillary Equipment

5.1.1) Silver disks. 0.855 in. dia. 0.010 in thick.
5.1.2) Teflon beakers. 250 mL and 100 ml with graphite base.
5.1.3) 1" width electrical tape

5.2) Reagents, Chemicals, and Standards

5.2.1) Type II water.
5.2.2) Hydrochloric acid.
5.2.3) Nitric acid.
5.2.4) Ammonium hydroxide.
5.2.5) Methyl orange indicator
5.2.6.) Specific range pH paper encompassing pH=2
5.2.7) 20% w/v hydroxylamine-hydrochloride solution, prepared as needed..
5.2.8) 5% w/v sodium citrate solution.

5.2.9) Ascorbic acid
5.2.10) 3M HCL

5.3) Instrumentation

5.3.1) Micro-Vax 3100 workstation or equivalent
5.3.2) Silicon surface barrier detectors with associated electronics and vacuum chambers.

6.0 Sample Collection & Preservation

6.1) Samples are collected and preserved with HNO3 to an approximate pH of 2 in a container.

7.0 Equipment and Instrument Maintenance

7.1) Refer to EPI SOP I-010 for instructions concerning micro-Vax 3100 instrument maintenance

8.0 Preparation of Standard Solutions and Quality Control Samples

8.1) All standards used in this method must be NIST traceable.

8.2) Dilution of standards should be noted in standard logbook in keeping with accepted procedure of labeling and tracking of the standards. The weight of the standard to be diluted and diluting solutions should be noted in the standards logbook. The weight should be taken on a balance of at least four places and calculation of the new standard activity carried out and noted in appropriate significant figures.

8.3) A blank should be run and used as the batch blank. This may be similar to the batched matrix and predetermined by at least 10 analysis to be free of isotopes being analyzed.

8.4) A duplicate and spiked sample should be made on the same sample in a sample batch.

9.0 Operating Procedure

9.1) Sample Preparation Techniques

9.1.1) Remove representative aliquot from each sample container.

9.1.2) Concentrate the sample to the desired level by boiling in teflon beaker or in glass beaker then transfer to teflon beaker for plating.

9.1.3) Monitor the pH with a pH meter and probe. Adjust pH with ammonium hydroxide (c) and 3 M HCl. Add 3M HCl until the solution pH is between 2 and 2.5. If passed below 2, adjust pH with ammonium hydroxide until pH is between 2 and 2.5. Add 5 ml 20% hydroxylamine-hydrochloride, 2 ml of 5% sodium citrate, 0.1 gram of ascorbic acid, and mix with a stirring rod.

9.1.4) A silver disk should be taken and electrical tape applied to one side. The disk should then be cleaned from dust by taking a moist paper and wiping over the surface. The disk can then be placed in the bottom of the beaker face up. If there are any solids the disk should be placed face out along the side of the beaker.

9.1.5) The beaker should be heated from 80 - 90 degrees celcius for 3-6 hours. Care should be taken not to heat the sample above 90 degrees, driving off sample volume.

9.1.6) After heating the beaker for 3-6 hours, remove the disk and rinse with type II water. The tape is then removed from the back of the disk and the disk labeled with sample identification.

9.1.7) The disk is then dried under a heat lamp to drive off moisture.

9.1.8) The sample is then counted in an alpha spectrometer for determination of isotopic polonium. Refer to EPI SOP I-009 "Micro-Vax 3100 Alpha Spectroscopy System Operating Procedures".

9.2) Instrument Calibration

9.2.1) Refer to EPI SOP I-009 for guidance concerning instrument calibration.

9.3) Instrument Performance Requirements

9.3.1) Instrument performance requirements: Refer to SOP I-009 for guidance concerning instrument performance requirements.

9.3.2) Documentation of instrument performance: Refer to SOP I-009 for guidance concerning instrument performance documentation.

9.4) Analysis Procedures and Instrumental Operation

9.4.1) Refer to EPI SOP I-009 for guidance concerning analysis procedures and instrument operation for isotopic polonium.

10.0 Calculations and Data Reduction Methods

10.1) The uncorrected activity is calculated in pCi/l by the following equation:

$$ALPHA = \frac{(A - B)}{(2.22)(E)(T)(Ab)(V)}$$

$$ALPHA_{UNCERTAINTY} = \frac{(1.96)\sqrt{\frac{A}{T_c} + \frac{B}{T_b}}}{(2.22)(E)(Ab)(V)(T)}$$

10.2) Corrected activity can be calculated by the following equation:

$$ACTIVITY_{CORRECTED} = A * e^{((-.693 * Dt) / T_{1/2})}$$

10.3) Tracer recovery is calculated by the following equation:

$$YIELD(\%) = \frac{(\text{DPM of TRACER ADDED})(\text{DETECTOR EFFICIENCY})}{(\text{NET CPM of TRACER})}$$

10.4) $$MDA = \frac{2.71 + 4.66\sqrt{CPM_b * T_c}}{2.22 * E * V * Ab * T * T_c}$$

Where:
A = alpha count rate per minute
B = background cpm
E = counting efficiency.
V = volume of sample taken for analysis.

2.22 = conversion factor from dpm/pCi
T = tracer recovery, as a decimal
Ab = abundance of the alpha isotope
Tc=count time of sample
Tb=count time of blank
Dt=Time interval from sampling to midpoint of counting in days.
$T_{1/2}$=Half life of the isotope of interest in days.

11.0 Data Recording, Review and Reporting

11.1) Data Recording

11.1.1) The data is printed out from the micro-Vax 3100 is in the same form that it is to be reported. All values are in pCi/L.

11.1.2) Once the batch is complete the values from the alpha reports are entered into the LIMS.

11.2) Data Review

11.2.1) Refer to EPI SOP D-003 for instructions concerning data review.

11.3) Data Reporting

11.3.1) The analyst will enter the data into the LIMS. The following information should be included: Analyst initials, run time and date of the sample, results in pCi/l and the accuracy in pCi/l.

12.0 Quality Control Requirements

12.1) Analyst and Method Verification Requirements

12.1.1) Please refer to EPI SOP D-002 for information concerning analyst and method verification.

12.2) Method Specific Quality Control Requirements

12.2.1) A matrix spike(ms) and sample duplicate should be run with every batch of samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows.

$$MS(\%) = \frac{SPIKE(pCi/L) - SAMPLE(pCi/L)}{SPIKE\ CONCENTRATION(pCi/L)} * 100\%$$

12.2.2) The relative percent difference (RPD) between the sample and the sample duplicate should be less than or equal to 20% if the results are greater than 5 times MDA for sample and duplicate. If either or both are below 5 times MDA but above MDA, then RPD should be below 100%. If either result is below MDA then limits on RPD are not applicable. The RPD is calculated as follows.

$$RPD(\%) = \frac{HIGH\ DUP(pCi/L) - LOW\ DUP(pCi/L)}{AVERAGE(pCi/L)} * 100\%$$

12.2.3) A method blank should accompany each batch of samples. The reported value should be less than or equal to requested MDA. If the value of the blank activity is not below the requested MDA then an adequate explanation should be given.

12.2.4) Tracer recovery should be between 25 - 125 %.

12.3) Actions Required if the Quality Control Requirements Are Not Met

12.3.1) If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non conformance report as outlined in GL-QS-E-004 "Nonconformance Identification Control, Documentation, Reporting and Dispositioning".

13.0 Records Management and Document Control

13.1) Raw data and associated documentation will be stored in binder books or in files and kept on hand for any subsequent reviews necessary. The records should be kept on hand for a length of time not shorter than 1 year.

13.2) Data generated by the micro-Vax 3100 will be backed up during routine software backup.

13.3) Quality control charts for spike and blank results are kept on the Micro-Vax-3100. Relative percent difference charts are also kept on the Micro-Vax 3100.

14.0 Laboratory Waste Handling and Disposal

14.1) Radioactive material is handled and disposed as outlined in EPI SOP S-005 "Radioactive Waste Handling Procedure".

15.0 References

15.1 Pb-210 Dating of Sediment Samples via Po-210 Alpha Spectrometry, Dr. Bill Burnett: Florida State University 1992.

Attachment 1

Polonium Que Sheet

Batch Comment:

Batch # 34822 Prep Date:_____ Initials: _____

Spike Isotope:_____ Spike Code:_____ Vol:_____

Tracer Isotope:_____ Tracer Code:_____ Vol:_____

LCS Isotope:_____ LCS Code:_____ Vol:_____

Batched to: Charles Harris

Sample ID	*Sample Description*	*Samp Date*	*Samp Time*	*Samp Vol (mls)or (g)*	*Detector #*	*DET #'S*	*SN*
9307334-02		7/19/93	1020			1	13271
9307361-05		7/19/93	0912			2	13911
9307361-06		7/19/93	1203			3	13272
9307361-07		7/19/93	1346			4	13273
9307361-12		7/20/93	1250			5	13258
9307361-13		7/20/93	1400			6	13259
9307362-01		7/20/93	1010			7	13265
9307362-02		7/20/93	1040			8	13268
9307362-03		7/20/93	1340			9	13266
9307362-04		7/20/93	1105			10	13261
9307362-05		7/20/93	0930			11	13296
9307362-06		7/20/93	1105			12	13260
9307363-01		7/20/93	1450			13	17940
9307363-02		7/20/93	1540			14	13912
9307390-01		7/22/93	0900			15	13270
9307390-02		7/22/93	0920			16	13269
9307390-03		7/22/93	0950			17	13274
9307390-04		7/21/93	0800			18	13275
9307390-05		7/21/93	0815			19	17024
9307390-06		7/21/93	1210			20	16318
QC136113						21	16320
QC136114						22	13323
QC136115						23	16323
QC136116						24	16326

Data Review By:_____

1. The Determination of Polonium in Soil

Process Owners:	Signature:	Brian T. Morris
		Wilbur B. Sigmon, III
Technical Review by:	Signature:	James B. Westmoreland
Quality Review by:	Signature:	Robert L. Pullano
Approved & Authorized by:	Signature:	Heyward H. Coleman

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2.0 Method Objective and Applicability

2.1) This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic alpha emitting polonium in soil.

3.0 Interferences

3.1) Undissolved sample residue may result in quenching of alpha particles if the silver disk is left upright upon bottom of beaker. If undissolved residue (probable fluoride precipitates) is visible then the disk should be left upright facing in from the side of the beaker to prevent baking of residue onto the disk.

4.0 Safety Precautions and Hazard Warnings

4.1) Caution should be used when dealing with acids, and other any laboratory reagents. Safety procedures for these are set forth in the Handbook for Good Laboratory Practices, found in the library of the laboratory.

4.2) If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the group leader prior to carrying out the rest of the procedure.

5.0 Apparatus and Materials

5.1) Ancillary Equipment:

5.1.1) Silver disks. 0.855 in. dia. 0.010 in thick.
5.1.2) Teflon beakers.
5.1.3) Microwave Oven. 1.2 cu. ft ,700 watt with carousel or equivalent.
5.1.4) Microwave Digestion Bombs. 100ml teflon lined available from CEM Inc.
5.1.5) Electrical tape

5.2) Reagents, Chemicals and Standards

5.2.1) Type II water.
5.2.2) 48% hydrofluoric acid
5.2.3) Hydrochloric acid
5.2.4) Nitric acid
5.2.5) Ammonium hydroxide

5.2.6) Specific range pH paper encompassing pH=2 and/or pH meter
5.2.7) 20% w/v hydroxylamine-hydrochloride solution, prepared as needed.
5.2.8) 5% w/v sodium nitrate solution, prepared as needed.
5.2.9) Ascorbic acid.
5.2.10) 3M HCL

5.3) Instrumentation

5.3.1) Micro-Vax 3100 workstation or equivalent
5.3.2) Silicon surface barrier detectors with associated electronics, and vacuum chambers.

6.0 Sample Collection & Preservation

6.1) Samples are collected without preservation in plastic bags or bottles.

7.0 Equipment and Instrument Maintenance

7.1) Please refer to SOP I-001 for instructions concerning Micro-Vax 3100 instrument maintenance.

8.0 Preparation of Standard Solution and Quality Control Samples

8.1) All standards used in this method must be NIST traceable.

8.2) Dilution of standards should be noted in standard logbook in keeping with accepted procedure, EPI SOP M-001 "Preparation of Radioactive Standards". The weight of the standard to be diluted and diluting solutions should be noted in the standards logbook. The weight should be taken on a balance of at least four places and calculation of the new standard activity carried out and noted in appropriate significant figures.

8.3) A "blank" soil should be used as the batch blank. This soil may be any soil similar to the batched matrix and predetermined by at least 10 analysis to be free of isotopes being analyzed.

8.4) A duplicate and spiked sample should be made on the same sample in a sample batch.

9.0 Operating Procedures

9.1) Sample Preparation Techniques

9.1.1) Refer to EPI SOP A-015, "Microwave Digestion for Soil and Sands". Record the sample aliquot on the que sheet (attachment 1).

9.1.2) Dilute the remaining sample to ~100ml. Monitor the pH with a pH meter and probe. Adjust pH with ammonium hydroxide and 3 M HCl,. until pH is between 2 and 2.5. Add 5 ml 20% hydroxylamine-hydrochloride, 2 ml of 5% sodium citrate, 0.1 gram of ascorbic acid, and mix.

9.1.3) Apply electrical tape to one side of a silver disc. The disk should then be cleaned of dust by taking a moist paper and wiping over the surface. The disk can then be placed in the bottom of the beaker, face up. If there are any solids, the disk should be placed face out along the side of the beaker.

9.1.4) The beaker should be heated from 80 - 90 degrees celsius for 3-6 hours. Care should be taken not to overheat the sample so it does not go to dryness before the minimum 3 hr. plating time.

9.1.5) After heating the beaker for 3-6 hours the disk is removed and rinsed with type II water. The tape is then removed from the back of the disk and the disk labeled with sample identification.

9.1.6) The disk is then dried under a heat lamp to drive off moisture.

9.1.7) The sample is then counted in an alpha spectrometer for determination of isotopic polonium. See procedure I-001 for operation of the micro-Vax 3100.

9.2) Instrument Calibration

9.2.1) Instrument calibration: Refer to SOP I-001 "Operation of the micro-Vax 3100 Workstation" for guidance concerning instrument calibration.

9.2.2) Initial calibration: Refer to SOP I-001 "Operation of the micro-Vax 3100 Workstation" for guidance concerning instrument calibration.

9.3) Instrument Performance Requirements

9.3.1) Instrument performance requirements: Refer to SOP I-001 "Operation of the micro-Vax 3100 Workstation" for guidance concerning instrument performance requirements.

9.3.2) Documentation of instrument performance: Refer to SOP I-001 "Operation of the Micro-Vax 3100 Workstation" for guidance concerning instrument performance documentation.

9.4) Analysis Procedures and Instrumental Operations

9.4.1) Refer to EPI SOP I-001 "Operation of the Micro-Vax 3100 Workstation" for guidance concerning analysis procedures and instrument operation for isotopic polonium.

10.0 Calculations and Data Reduction Methods

10.1) Calculate the decay uncorrected activity in pCi/g by the following equation:

10.1.1) $$\text{ALPHA} = \frac{(A - B)}{(2.22)(E)(T)(Ab)(g)}$$

10.1.2) $$\text{ALPHA}_{\text{UNCERTAINTY}} = \frac{(1.96)\sqrt{\frac{B+A}{Tc}}}{(2.22)(E)(Ab)(g)(T)}$$

Where:
A = alpha count rate per minute
B = background cpm
E = counting efficiency.
g = grams of sample taken for analysis.
2.22 = conversion factor from dpm/pCi
T = tracer recovery, as a decimal
Ab = abundance of the alpha isotope
Tc=count time

10.2) Corrected activity can be calculated by the following equation:

10.2.1) $$ACTIVITY_{CORRECTED} = A * Exp\left(\frac{Dt * -\ln 2}{T_{1/2}}\right)$$

Where: A=uncorrected activity
Dt=decay time in years
$T_{1/2}$=isotopic half life in years

10.3) Tracer recovery is calculated by the following equation:

$$YIELD(\%) = \frac{(DPM\ of\ TRACER\ ADDED)(DETECTOR\ EFFICIENCY)}{(NET\ CPM\ of\ TRACER)}$$

10.4) $$MDA = \frac{2.71 + 4.66\sqrt{cpm_{bkg} * Tc}}{2.22 * E * Ab * g * T * Tc}$$

Where: cpm_{bkg}=detector background counts per minute
Tc=Sample counting time
Ab=isotopic alpha abundance
g=weight of sample
T=tracer recovery
E=detector efficiency

11.0 Data Recording, Review and Reporting

11.1) Data Recording

11.1.1) The data is printed out from the micro-Vax 3100 is in the same form that it is to be reported. All values are in pCi/g.

11.1.2) Once the batch is complete the values from the alpha reports are entered into the LIMS.

11.2) Data Review

11.2.1) Refer to EPI SOP D-003 "Data Review and Validation" for instructions concerning data review. After the data has been entered the entered numbers should be cross-checked by the analyst to ensure that there

are no transcription errors. A print-out of data should be generated and turned into the group leader or his assigned alternate with all batch que sheets, raw data and any calculation sheets, if applicable.

11.2.2) If the samples results or qc results are not acceptable, the sample or qc shall be rerun. An example of this might be a tracer recovery less than 25% or above 125% which would result in a rerun.

11.2.3) The reviewer should then check for transcription errors. The quality control samples should then be checked to ensure that they meet the above criteria. Raw data should also be checked for its accuracy, (i.e. correct sample weight, sample date, tracer activity, spike activity and so on). If any calculations were made, they should also be checked for accuracy. An example of this might be calculation of relative percent difference.

11.2.4) The reviewer should verify that the instrument was in conformance at the time of the analysis. See EPI SOP I-001 for instrument conformance criteria.

11.3) Data Reporting

11.3.1) The analyst will take the applicable spreadsheet and enter the data into the LIMS. The following information should be included: analyst's initials, run date and time of the sample, results and the accuracy in pCi/g.

12.0 Quality Control Requirements

12.1) Analyst and Method Verification Requirements

12.1.1) Refer to EPI SOP D-002 for information concerning analyst and method verification.

12.2) Method Specific Quality Control Requirements

12.2.1) A matrix spike(ms) should be run with every batch of samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows.

$$MS(\%) = \frac{SPIKE(pCi/g) - SAMPLE(pCi/g)}{SPIKE\ CONCENTRATION(pCi/g)} * 100\%$$

12.2.2) A duplicate sample should be run with every batch of samples. The relative percent difference (RPD) between the sample and the sample duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$RPD(\%) = \frac{\text{HIGH DUP(pCi / g)} - \text{LOW DUP(pCi / g)}}{\text{AVERAGE(pCi / g)}} * 100\%$$

12.2.3) A method blank should accompany each batch of samples. The reported value should be less than or equal to requested MDA. If the value of the blank activity is not below the requested MDA then an adequate explanation should be given.

12.2.4) Tracer recovery should be between 25 - 125 %.

12.3) Actions Required if the Quality Control Requirements Are Not Met

12.3.1) If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non conformance report as outlined in GEL SOP GL-QS-E-004 "Nonconformance Identification Control, Documentation, Reporting and Dispositioning".

13.0 Records Management and Document Control

13.1) Raw data and associated documentation will be stored in binder books or in files and kept on hand for any subsequent reviews necessary. The records should be kept on hand for a length of time not shorter than 1 year.

13.2) Data generated by the micro-Vax 3100 will be backed up during routine software backup, see procedure I-001 for instructions concerning how often the information is backed up and how long it is kept.

13.3) All charts are kept on the Micro-Vax-3100.

14.0 Laboratory Waste Handling Disposal

14.1) Radioactive material is handled and disposed of as outlined in EPI SOP S-005, "Radioactive Waste Handling Procedure".

15.0 References

15.1) Pb-210 Dating of Sediment Samples via Po-210 Alpha Spectrometry. Dr. Bill Burnett: Florida State University. 1992.

15.2) "Standard Practice for the Microwave Digestion of Industrial Furnace Feedstreams for Trace Element Analysis". ASTM.

Attachment 1

Polonium Que Sheet

Batch Comment

Batch # 34822 Prep Date:_____ Initials: _____

Spike Isotope:_____ Spike Code:_____ Vol:_____

Tracer Isotope:_____ Tracer Code:_____ Vol:_____

LCS Isotope:_____ LCS Code:_____ Vol:_____

Batched to: Charles Harris

Sample ID	*Sample Description*	*Samp Date*	*Samp Time*	*Samp Vol (mls)or (g)*	*Detector #*	*DET #'S*	*SN*
9307334-02		7/19/93	1020			1	13271
9307361-05		7/19/93	0912			2	13911
9307361-06		7/19/93	1203			3	13272
9307361-07		7/19/93	1346			4	13273
9307361-12		7/20/93	1250			5	13258
9307361-13		7/20/93	1400			6	13259
9307362-01		7/20/93	1010			7	13265
9307362-02		7/20/93	1040			8	13268
9307362-03		7/20/93	1340			9	13266
9307362-04		7/20/93	1105			10	13261
9307362-05		7/20/93	0930			11	13296
9307362-06		7/20/93	1105			12	13260
9307363-01		7/20/93	1450			13	17940
9307363-02		7/20/93	1540			14	13912
9307390-01		7/22/93	0900			15	13270
9307390-02		7/22/93	0920			16	13269
9307390-03		7/22/93	0950			17	13274
9307390-04		7/21/93	0800			18	13275
9307390-05		7/21/93	0815			19	17024
9307390-06		7/21/93	1210			20	16318
QC136113						21	16320
QC136114						22	13323
QC136115						23	16323
QC136116						24	16326

Data Review By:_____

1.0 The Determination of Iodine-131 in Water

UNCONTROLLED DOCUMENT

Process Owners:	Signature:	Keith S. Doran
		Russ L. Moser
Technical Review by:	Signature:	James B. Westmoreland
Quality Review by:	Signature:	Robert L. Pullano
Approved & Authorized by:	Signature:	Heyward H. Coleman

SE

2.0 Method Objective and Applicability

2.1) This standard operating procedure provides the necessary instructions to conduct the analysis for I-131 in water. I-131 may be done directly by EPI SOP A-013 if the sample is counted sufficiently to meet contract required detection limits.

3.0 Interferences

3.1) Samples which are acidified to pH of 2 at the time of collection may lose some Iodine as the gas.

4.0 Safety Precautions and Hazard Warnings

4.1) Caution should be used when dealing with acids, and other any laboratory reagents. Safety procedures for these are set forth in the Handbook for Good Laboratory Practices, found in the library of the laboratory.

4.2) If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the group leader prior to carrying out the rest of the procedure.

5.0 Apparatus and Materials

5.1) Ancillary Equipment

5.1.1) AG-1x8 ion exchange resin, 50-100 mesh, Cl^- form.
5.1.2) Ion exchange columns -- 2 cm ID by ~15 cm; use 10 ml wet resin in column (equivalent to 6.5 grams dry weight).
5.1.3) pH paper (general purpose 0-14 range).
5.1.4) Whatman 42 filter paper, 47 mm
5.1.5) Stainless steel planchet, 2" diameter, $^1/_8$" depth
5.1.6) Glass fiber filters, 47 mm

5.2) Reagents, Chemicals and Standards

5.2.1) NIST traceable standard I-131.
5.2.2) 1M hydroxylamine hydrochloride ($NH_2OH \cdot HCl$) solution (prepare by dissolving 69.49 g $NH_2OH \cdot HCl$ per liter DDW).
5.2.3) 1M sodium bisulfite ($NaHSO_3$) solution (prepare by dissolving 104.6 g $NaHSO_3$ per liter DDW).
5.2.4) 5% sodium hypochlorite (NaOCl) solution.

5.2.5) 50% NaOH solution.
5.2.6) 10% HNO_3 solution.
5.2.7) Pd carrier, 10,000 mg/ml
5.2.8) 4N HNO_3
5.2.9) Carbon tetrachloride
5.2.10) Iodine carrier, 10 mg/ml 1.18 NaI in 100 l DI H_2O

5.3) Instrumentation

5.3.1) Gas Flow Proportional Counter

6.0 Sample Collection & Preservation

6.1) Samples should be collected in a suitable container which will maintain its integrity during transportation. No preservation is required.

7.0 Equipment and Instrument Maintenance

7.1) Refer to EPI SOP I-010 for instructions concerning instrument maintenance.

8.0 Preparation of Standard Solutions and Quality Control Samples

8.1) All standards used in this method must be NIST traceable.

8.2) Dilution of standards should be noted in standard logbook in keeping with accepted procedure, EPI SOP M-001 "Preparation of Radioactive Standards". The weight of the standard to be diluted and diluting solutions should be noted in the standards logbook. The weight should be taken on a balance of at least four places and calculation of the new standard activity carried out and noted in appropriate significant figures.

8.3) A "blank" water should be used as the batch blank.

8.4) A duplicate and spiked sample should be made on the same sample in a sample batch.

9.0 Operating Procedure

9.1) Sample Preparation Techniques

Initial sample preparation

9.1.1) Transfer a known volume to a suitable container and record on the que sheet (attachment 1). Add 1.0 ml of I- carrier (10 mg/ml), and add 5 ml of 5% NaOCl. While stirring, allow to react for 2-3 minutes.

9.1.2) Add 5 ml 1M $NH_2OH \cdot HCl$ and 2 ml 1M $NaHSO_3$.

9.1.3) Adjust pH to 6.5 using 50% NaOH (about 4 drops has proven to be sufficient in most cases). Use 10% HNO_3 to lower pH if it goes too high. Continue to stir for several minutes.

9.1.4) Filter any suspended matter as necessary using a 47 mm glass fiber filter.

Ion exchange separations

9.1.5) Prepare ion exchange column with 10 ml wet AG1X8 resin, rinse with DDW. No other pretreatment required.

9.1.6) Pass the sample through the ion exchange resin at a flow rate of ~20 ml/min. Discard the effluent.

9.1.7) Wash the resin from the ion exchange column into a labeled 250 ml beaker. Allow the resin to settle and decant the water.

9.1.8) Add 50 ml of 5% NaOCl to the resin. Heat in a hot H_2O bath for 10 minutes, cool, centrifuge, filter the supernates by gravity through a Whatman No. 42 filter paper. Collect the filtrate in a 250 ml beaker. Add concentrated HNO_3 to make the sample filtrate normality between 2 and 3. The following table shows how much concentrated HNO_3 is required for 2-3N solution.

Eluate Volume	Needed mls of Concentrated Nitric Acid
50-60 mls	10 ml
60-70 mls	12 ml
70-80 mls	14 ml
80-90 mls	16 ml

9.1.9) Repeat step 9.1.8 twice, combining the filtrates. Discard the resin.

9.1.10) Note: This step must be performed in a hood due to the evolution of

chlorine gas. Carefully add 10 ml of HNO_3 for each 100 ml of NaOCl to the combined filtrates. Heat to boiling on a hot plate, cool, and transfer the sample to a 250 ml separatory funnel.

9.1.11) Add 50 ml of CCl_4, 1.5 g of hydroxylamine hydrochloride and shake for 2 minutes.

9.1.12) Drain the lower (organic) phase into a clean 250 ml separatory funnel and retain.

9.1.13) Add 25 ml of CCl_4 and 1 g of hydroxylamine hydrochloride to the aqueous phase in the first separatory funnel and re-extract. Combine the organic phases and discard the aqueous fraction.

9.1.14) Add 25 ml of H_2O and 10 drops of freshly prepared 1M $NaHSO_3$ to the separatory funnel containing the iodine extract and shake for 2 minutes. After phase separation, transfer the aqueous phase to a 50 ml centrifuge tube.

9.1.15) To the 50 ml centrifuge tube, add 2 mls of 1M HNO_3 and heat gently for 10 minutes, then stir.

9.1.16) Add 2 mls of Pd carrier. Wait for 30 minutes then filter through 47 mm glass fiber filter paper.

9.1.17) Dry the precipitate at 110° for 20 minutes. Cool in a dessicator and weigh to the nearest 0.1 mg. Record the weights on the que sheet (attachment 1).

9.1.18) Mount the filter paper in a counting planchet and transfer to the counting room for measurement on a gas flow proportional counter.

9.2) Instrument Calibration

9.2.1) Standardize the counter with known amounts of I-131. Prepare a self-absorption curve by adding a known amount of I-131 to various amounts of I^- carrier (5, 10, 15, 20 mg).

9.3) Instrument Performance Requirements

9.3.1) Instrument performance requirements: Refer to EPI SOP I-010 for guidance concerning instrument performance requirements.

9.3.2) Documentation of instrument performance: Refer to EPI SOP I-010 for

guidance concerning instrument performance requirements documentation.

9.4) Analysis Procedures and Instrumental Operations

9.4.1) Refer to EPI SOP I-002, I-003, I-005, or I-006 for guidance concerning instrument operation for gas flow proportional counters.

10.0 Calculations and Data Reduction Methods

10.1) The instrument will report sample pCi/l according to the following equation:

$$\text{Sample pCi / l} = \frac{S_{cpm} - B_{cpm}}{2.22 * E * V * A * D}$$

10.2) The counting uncertainty is calculated according to the following equation:

$$\text{pCi / l} = \frac{1.96 * \sqrt{\frac{S_{cpm}}{T_c} + \frac{B_{cpm}}{T_{cb}}}}{2.22 * E * V * A * D}$$

10.3) The method minimum detectable activity (MDA) is calculated according to the following equation:

$$MDA = \frac{2.71 + 4.66\sqrt{cpm_{bkg} * T_c}}{2.22 * E * V * A * D * T_c}$$

where:
S_{cpm} = Sample counts per minute
B_{cpm} = Background counts per minute
E= Counting efficiency
V= Sample volume (liters)
T_c= Sample count time(minutes)
A= Absorption correction factor
D= Decay factor for I-131
T_{cb}=Background count time (minutes)

11.0 Data Recording , Review and Reporting

11.1) Data Recording

11.1.1) The data is printed out from the gas flow proportional counter and reduced on the appropriate spreadsheet. All values are in pCi/l.

11.2) Data Review

11.2.1) Refer to EPI SOP D-003 "Data Review and Validation" for instructions concerning data review.

11.3) Data Reporting

11.3.1) The analyst will take the applicable spreadsheet and enter the data into the LIMS. The following information should be included: analyst's initials, run date and time of the sample, results and the accuracy in pCi/l.

12.0 Quality Control Requirements

12.1) Analyst and Method Verification Requirements

12.1.1) Refer to EPI SOP D-002 for information concerning analyst and method verification.

12.2) Method Specific Quality Control Requirements

12.2.1) A method blank should accompany each batch of samples. The reported value should be less than or equal to the CRDL.

12.2.2) A matrix spike(ms) should be run with every batch of samples. The recovery of the spikes should fall between 75 and 125%. The recovery is calculated as follows.

$$\%\,\mathrm{Rec} = \frac{\text{Spike pCi / l} - \text{Sample pCi / l}}{\text{Spiked Amount pCi / l}} * 100$$

12.2.3) A duplicate sample should be run with every batch of samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$RPD = \frac{\text{High Sample pCi / l} - \text{Low Sample pCi / l}}{\text{Average pCi / l}} * 100$$

12.2.4) A Laboratory Control Sample (LCS) should be run with each batch of samples. The recovery of the LCS should fall between 75-125%. The recovery is calculated as follows:

$$\%\,Rec = \frac{\text{Observed pCi / l}}{\text{Known pCi / l}} * 100$$

12.3) Actions Required if the Quality Control Requirements Are Not Met.

12.3.1) If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non conformance report as outlined in GEL SOP GL-QS-E-004 "Nonconformance Identification Control, Documentation, Reporting and Dispositioning".

13.0 Records Management and Document Control

13.1) Raw data and associated documentation will be stored in binder books or in files and kept on hand for any subsequent reviews necessary. The records should be kept on hand for a length of time not shorter than 1 year.

13.2) Data generated by the gas flow proportional counter will be backed up during routine software backup.

13.3) All charts are kept on the Micro-Vax-3100 or the gas flow proportional counter.

14.0 Laboratory Waste Handling Disposal

14.1) Radioactive material is handled and disposed of as outlined in EPI SOP S-005, "Radioactive Waste Handling Procedure".

15.0 References

15.1) EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples.

March, 1979.

15.2) EML Procedures Manual HASL-300, 1982.

Attachment 1

I-131 in Water Que Sheet

Comment: *none*

Batch # 37925 Prep Date:_____ Initials: _____

Spike Isotope:_____ Spike Code:_____ Vol:_____

LCS Isotope:_____ LCS Code:_____ Vol:_____

Instrument Used: (circle one) LB5100V HT1000 LB5100 Lb4110 Batched to:

s/n: 14740 1.1E+07 203 8219 Shane Mull

Sample ID	*Sample Descrip.*	*Bkr #*	*Vol (mls)*	*Carrier or Det #*	*Initial Weight (g)*	*Final Weight (g)*	*Net Weight (mg)*
9310151-23	ACB 4A						
9310151-24	QA 13D						
9310151-25	QA 14D						
9310233-01	HSB111E						
9310233-02	HSB112C						
9310233-03	HSB113C						
9310233-04	HSB125C						
9310285-16	HSB124AR						
9310285-17	HSB135C						
9310285-18	HSB135D						
QC149938	blank						
QC149939	9310285-18						
QC149940	9310285-18						
QC149941	lcs						

Data Reviewed By: _____

STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF LEAD-210

IN WATER

UNCONTROLLED DOCUMENT

Project Owner(s):	John D. Dornisch, Analyst I
Technical Review by:	Russell L. Moser, EPI Group Leader
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	James B. Westmoreland, EPI Laboratory Manager

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF LEAD-210

2.0 METHOD OBJECTIVE AND APPLICABILITY

This standard operating procedure provides the necessary instructions to conduct the analysis for Lead-210 in water and soil.

3.0 INTERFERENCES

No interferences have been encountered.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 Care should be taken when handling concentrated sulfuric acid as contact with fumes or liquid may cause severe burning of body tissue.

4.2 Lead is a cumulative poison which can cause brain damage; therefore, proper laboratory techniques are essential.

5.0 Apparatus and Materials

5.1 Ancillary Equipment

5.1.1 Aluminum foil

5.1.2 Filtration apparatus

5.1.3 Centrifuge

5.1.4 Centrifuge tubes

5.1.5 Beakers

5.1.6 Stirring rods

5.1.7 Sr Spec resin® EIChroM Industries, Inc

5.1.8 Hot plates

5.1.9 Gelman DM-450® or equivalent filters

5.1.10 2" stainless steel planchettes

5.1.11 Aluminum foil

5.1.12 Heat lamp or drying oven

5.1.13 Cellulose nitrate filters (47mm with .45µm pore size)

5.2 Reagents, Chemicals and Standards

5.2.1 Lead carrier ($Pb(NO_3)_2$ F.W.=331.21 g). Obtain ~10 mg/mL Pb. Dissolve 15.985 g $Pb(NO_3)_2$ in 900 mL DI water then dilute to 1000 mL with DI water. This carrier should be standardized as outlined in step 9.2.8.

5.2.2 NIST traceable Pb-210 standard

5.2.3 1:1 sulfuric acid. Carefully add 500 mL concentrated sulfuric acid to 500 mL DI water in a large beaker. Allow mixture to cool to room temperature then transfer it to a graduated cylinder to verify the total volume at 1000 mL.

5.2.4 1 M nitric acid. Carefully add 62.5 mL concentrated nitric acid (16 M) to 1000 mL DI water in a graduated cylinder.

5.2.5 0.1 M nitric acid. Carefully add 6.25 mL concentrated nitric acid (16 M) to 1000 mL DI water in a graduated cylinder.

5.2.6 0.1 M ammonium citrate (dibasic) (F.W.= 226.19 g). Dissolve 22.619 g ammonium citrate (dibasic) in 900 mL DI water then dilute to 1000 mL with DI water.

5.2.7 DI water

5.2.8 6 M NaOH. Carefully add 240 g NaOH pellets to approximately 600 mL of Deionized water. When cool dilute to 1000 mL with Deionized water.

5.2.9 Bromocresol purple pH indicator

5.3 Instrumentation

5.3.1 Gas flow proportional counter

6.0 SAMPLE COLLECTION AND PRESERVATION

Water samples should be collected in plastic bottles and preserved with concentrated nitric acid to pH~2.

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to EPI SOP "HT-1000 Gross Alpha/Beta Counter Operating Procedure" (GL-EPI-E-I002).

7.2 Refer to EPI SOP "LB-4100 Gross Alpha/Beta Counter Operating Procedure" (GL-EPI-E-I006).

7.3 Refer to EPI SOP "Counting Room Instrument Maintenance and Performance Checks" (GL-EPI-E-I010).

8.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

8.1 All standards used in this method must be traceable to a nationally accepted source, such as NIST or EPA.

8.2 Dilution of standards should be noted in the standard logbook as outlined in EPI SOP "Preparation of Radioactive Standards"GL-EPI-E-M001). The weight of the standard to be diluted and diluting solutions should be noted in the standards logbook. The weight should be taken on a balance of at least four places and calculation of the new standard activity carried out and noted in appropriate significant figures.

8.3 A reagent blank should be used as the batch blank. The blank matrix should resemble the sample matrix as closely as possible.

8.4 A duplicate and spiked sample should be made on the same sample in a sample batch. The spiked sample is made by adding a known quantity of analyte isotopes directly to the sample matrix.

8.5 A Laboratory Control Sample (LCS) should be made by spiking deionized water with a known quantity of analyte isotopes.

9.0 OPERATING PROCEDURES

9.1 Sample Preparation Techniques - (values in [] to be used for soils)

9.1.1 For water samples measure an appropriate aliquot of water sample into a beaker. For soils prepare as specified in EPI SOP "Preparation of Special Matrices for the Determination of Radionuclides" (GL-EPI-E-M001). Record the sample volume and standard spike information on the que sheet (Appendix 1).

9.1.2 Place soil sample into beaker containing 200 mL DI water.

9.1.3 Add 1.0 mL lead carrier ($Pb(NO_3)_2$. Add 1 mL of bromocresol purple for every 400 mL of samples aliquot used.

9.1.4 Heat samples to a slight boil, then remove from heat and allow to cool.

9.1.5 Slowly add 6 M NaOH until color just turns purpose (pH ~ 8.0).

9.1.5.1 For water samples proceed to step 9.1.11.

9.1.6 For soil samples, transfer precippitate from beaker to centrifuge tube and centrifuge, decant and discard supernate. If necessary, repeat to completely transfer the precipitate.

9.1.7 Rinse the sides of the beak with 9 mL of 1 M nitric acid into beaker and transfer to centrifuge tube. Vortex sample to allow to sit overnight.

9.1.8 Centrifuge the samples and collect the supernate.

9.1.9 Place a culture tube in a side arm flask. Place the funnel apparatus with the filter on the flask so the tip of the funnel is in the culture tube.

9.1.10 With no vacuum applied, filter supernate through a .45 µm cellulose nitrate filter. After 1 minute apply vacuum and add 6 mL of 1M nitric acid to rinse the funnel sides of any precipitate.

9.1.10.1 For soil samples proceed to Step 9.1.16.

9.1.11 For water samples, filter the sample from Step 9.1.5 through a .45 µm cellulose nitrate membrane filter. Transfer the remaining precipitate to the filter with a fine stream of DI water.

9.1.12 Place a culture tube in a side arm flask. Place the funnel apparatus with the filter on the flask so the tip of the funnel is in the culture tube.

9.1.13 Rinse the sides of the beaker with 6 mL of 1 M nitric acid and swirl.

9.1.14 Without vacuum applied, add the rinse from the beaker in Step 9.1.13.

9.1.15 Apply vacuum after 1 minute and add 4 mL of 1 M nitric acid to rinse the funnel sides of any precipitate.

9.1.16 Condition the Sr Spec resin® column with 10 [20] mL 1 M nitric acid.

9.1.17 Load the sample from the culture tube onto the column. Discard the eluent.

9.1.18 Rinse the column with 10 [20] mL 1 M nitric acid. Discard the eluent. Record the time and date on the que sheet. This rinse removes the iron and starts the bismuth ingrowth time.

9.1.19 Rinse the column with 10 [20] mL 0.1 M nitric acid. Discard the eluent. This rinse removes polonium and strontium.

9.1.20 Rinse the column with 2 [4] mL DI water. Discard the eluent.

9.1.21 Elute the Pb with 15 [30] mL 0.1 M ammonium citrate. Collect the eluent in a labeled centrifuge tube.

9.1.22 Add 10 [15] mL 1:1 sulfuric acid to the sample, cap and shake vigorously and/or vortex to precipitate lead sulfate. Allow the precipitate to settle for 1 hour.

9.1.23 Pre-weigh Gelman DM-450® or equivalent filters and record the initial weight on the que sheet.

9.1.24 Centrifuge the precipitate and decant the supernate. Add 20 mL DI water to wash the precipitate. Repeat this step 2 times to thoroughly wash the precipitate of residual ammonium citrate.

9.1.25 Filter the precipitate through the Gelman DM-450® or equivalent filters.

9.1.26 Dry the filter under a heat lamp.

9.1.27 Weigh the filters to obtain the precipitate weight and record the results on the que sheet.

9.1.28 Place the filter in a 2" stainless steel planchette and cover the filter with aluminum foil.

9.1.29 Allow the sample to ingrow for at least 30 hours before counting.

9.1.30 Count in a gas flow proportional counter for a time duration to meet the contract required detection limit and uncertainty.

9.2 Instrument Calibration

9.2.1 Add a known dpm of Pb-210 NIST traceable standard, with Bi-210 at equilibrium, to 10 separate centrifuge tubes. Record the information on the que sheet.

9.2.2 Add an increasing amount of lead carrier to each of the tubes.

9.2.3 Add 10 mL DI water to each of the tubes.

9.2.4 Add 10 mL 1:1 sulfuric acid to the standards, cap and shake vigorously to precipitate lead sulfate. Allow the precipitate to settle for 1 hour..

9.2.5 Pre-weigh the Gelman DM-450® filters, as needed, and record the information on the que sheet. Filter the standards through the filters.

9.2.6 Dry the filters under a heat lamp.

9.2.7 Weigh the filters to obtain the precipitate weight and record the information on the que sheet.

9.2.8 Place the filter in a 2" stainless steel planchette and cover with aluminum foil.

9.2.9 Count the standards for a duration long enough to obtain at least 10,000 total counts.

9.2.10 Determine the detector efficiencies utilizing a calculation spreadsheet according to the following equation.

$$\text{Detector Efficiency} = \frac{\text{observed cpm - bkg cpm}}{\text{certified dpm}}$$

where:

observed cpm = CPM generated by the standard.
bkg cpm = Method blank background or instrument background, as applicable.
certified dpm= standard DPM, decay corrected to the mid point of standard counting.

9.2.11 Plot a graph of detector efficiency versus precipitate weight. The plot should resemble a line with zero slope. If this is not the case, consult Group Leader for guidance.

9.2.12 Update the calibration spreadsheet to reflect the new instrument efficiency. Turn in all associated calibration material to the Group Leader for review.

9.2.13 Standardize any new lead carrier.

9.2.13.1 Pre-weigh 10 filters and record the data on the que sheet.

9.2.13.2 To 10 separate centrifuge tubes, add 10 mL DI water and 1.0 mL lead carrier.

9.2.13.3 To each of the tubes, add 10 mL 1:1 sulfuric acid.

9.2.13.4 Cap and shake the tubes vigorously. Allow the precipitate to settle for at least 1 hour.

9.2.13.5 A successful standardization will meet the following criteria:

9.2.13.5.1 Remaining sample population of at least seven.

9.2.13.5.2 Standard deviation of less than two.

9.2.13.5.3 An average value within 20% of the theoretical value. The theoretical value for 1 mL of 10 mg/mL Pb precipitated as $PbSO_4$ is 14.64 mg. The acceptable range is, therefore, 11.71-17.57 mg.

9.2.13.6 All successive chemical yield calculations will use the average standardized precipitated lead carrier weight, as calculated above, for the 100% chemical yield level.

9.2.13.7 Ensure the master spreadsheet is updated.

9.2.13.8 Calibrate the carrier every six months or when new carrier is made up.

9.3 Instrument Performance Requirements

9.3.1 Refer to EPI SOP "Counting Room Instrumentation Maintenance and Performance Checks" (GL-EPI-E-I010) for instructions concerning instrument performance.

9.4 Analysis Procedures and Instrumental Operation

9.4.1 Refer to the appropriate gas flow proportional counting procedures, GL-EPI-E-I002, ..I003, ..I005, or ..I006 for these instructions.

10.0 CALCULATIONS AND DATA REDUCTION METHODS

10.1 The analyst will use an Excel spreadsheet (Appendix 2) to calculate the sample pCi/L according to the following equations:

$$\text{Result } {}^{pCi}/_{L} = \frac{(S_{cpm} - B_{cpm})}{2.22 * E * I * V * Y}$$

10.2 The counting uncertainty is calculated according to the following equation:

$$\text{Uncertainty } {}^{pCi}/_{L} = \frac{1.96\sqrt{\frac{S_{cpm}}{Tc} + \frac{B_{cpm}}{T_{CB}}}}{2.22 * E * I * V * Y}$$

10.3 The method detection limit (MDA) is calculated according to the following equation:

$$\text{MDA } {}^{pCi}/_{L} = \frac{2.71 + 4.66\sqrt{B_{cpm} * T_c}}{2.22 * E * I * V * Y * T_c}$$

where:

T_C=Sample count duration
B_{CPM}=Background counts per minute
S_{CPM}=Sample counts per minute
E=Efficiency of counting
V=Sample volume (liters)
Y=Chemical yield
I=Bi-210 ingrowth = 1-EXP(-.138269934*Elapsed Time(Days))
T_{CB}=Background count time

11.0 DATA RECORDING, REVIEW AND REPORTING

11.1 Data Recording

11.1.1 Record the information required on the Pb-210 que sheet (Appendix 1).

11.1.2 Transfer applicable data from Pb-210 que sheet to Pb-210 master spreadsheet in Excel (Appendix 2).

11.2 Data Review

11.2.1 Refer to EPI SOP "Data Review and Validation Procedures" (GL-EPI-E-D003) for instructions concerning the data review process.

11.3 Data Reporting

11.3.1 The analyst will take the applicable Pb-210 spreadsheet and enter the data into LIMS. The following information should be included: analyst's initials, date and time the sample was counted, sample results and accuracy in pCi/L.

12.0 QUALITY CONTROL REQUIREMENTS

12.1 Analyst and Method Verification Requirements

12.1.2 Refer to EPI SOP "Analyst and Analytical Method Validation" (GL-EPI-E-D002) for information concerning analyst and method verification.

12.2 Method Specific Quality Control Requirements

12.2.1 A matrix spike (ms) should be run with every batch of samples. The recovery of the spike should fall between 75% and 125%. The recovery is calculated as follows.

$$\text{Rec} = \frac{\text{Spike(pCi/unit)} - \text{Sample(pCi/unit)}}{\text{Nominal Concentration(pCi/unit)}} * 100$$

12.2.2 A sample duplicate should be run with every batch of samples. The relative percent difference (RPD) between the sample and the sample duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$\%\text{RPD} = \frac{\text{High Activity(pCi / unit)} - \text{Low Activity(pCi / unit)}}{\text{Average(pCi / unit)}} * 100$$

12.2.3 A method blank should accompany each batch of samples. The reported value should be less than or equal to requested MDA. If the value of the blank activity is not below the requested MDA then an adequate explanation should be given.

12.2.4 A Laboratory Control Sample (LCS) should be run with each batch of samples. The recovery of the LCS should fall between 75% -125%. The recovery is calculated as follows:

$$\%\text{Rec} = \frac{\text{Observed pCi/l}}{\text{Known pCi/l}} * 100$$

12.3 Actions Required if the Quality Control Requirements Are Not Met

12.3.1 If any of the QC criteria from 12.2.1 through 12.2.5 cannot be satisfied, the analyst should inform their Group Leader and initiate a Non-conformance Report as outlined in GEL SOP "Documentatin of Non-conformance Reporting and Dispositioning, and Control of Non-conforming Items" (GL-QS-E-004).

NOTE: Some clients may have more or less quality control requirements.

13.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

All raw data printouts, calculations, spreadsheets, and batch checklists shall be filed with the sample data for archival and review.

14.0 LABORATORY WASTE HANDLING AND DISPOSAL

14.1 Radioactive samples and material shall be handled and disposed of as outlined in EPI SOP "EPI Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011).

14.2 Save ion exchange resin for regeneration.

15.0 REFERENCES

15.1 E. Philip Horwitz, Renato Chiarizia, and Mark L. Dietz. "A Novel Strontium-Selective Extraction Chromatographic Resin."

15.2 Special thanks to Dr. Bill Burnett and his associates for their development of this method at Florida State University.

Lead-210 in Water Que Sheet

Batch # Prep Date:_____ Comment:
Initials: _____
Spike Isotope: Pb-210 Spike Code:_____ Vol:_____

LCS isotope Pb-210 LCS Code:_____ Vol:_____

Instrument Used: (circle one)	LB5100W	HT1000	LB5100	Lb4110	Batched to:
s/n:	14740	10912771	203	8219	

Sample ID	*Sample Descrip.*	*Bkr #*	*Vol (mls)*	*Carrier or Det #*	*Filter Weight Initial (g)*	*Filter Weight Final (g)*	*Precipitate Weight (mg)*	*Bi-210 ingrowth Start Date/Time*

Data Reviewed By: _____

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APPENDIX 2

Pb-210 IN WATER CALCULATION SPREADSHEET

LB5100/W

Batch	1																
Date Analyzed:	9/23/93																
Initials:	BTM				Standard weight=	14.64	mg					Recovery					
Spike Value:	241	pCi/L			Spike=	225.2	pCi/L				Spike=	72.24%					
Beta Bkg	0.73	cpm			LCS=		pCi/L				LCS=	76.04%					
Bkg Count Time:	480	min															
Beta Cross talk:	0.0143																
	Sample	Solids	Chemical	Beta	Time	Time	Ingrowth	Carrier	Gross	Count	Net	Pb-210	Pb-210	Pb-210	Beta	ELASPED	
Lab I.D.	Volume	Weight	yield	Eff.	Sep.	Count	Factor	#	Beta	duration	Beta	MDA	Result	Uncert.	Background	TIME	
	(ml)	(mg)							(counts)	(min.)	(cpm)	(pCi/L)	(pCi/L)	(pCi/L)	(cpm)	(days)	
1	500	12.8	86%	0.3800	9/22/93 20:10	9/24/93 16:43	0.2264	69	282	20.00	13.37	12.482	162.7	20.0	0.73	1.8582	
1	500	13.0	89%	0.3800	9/22/93 20:10	9/24/93 16:43	0.2264	69	305	20.00	14.52	12.098	171.3	20.2	0.73	1.8582	
Analyst:																	
Secondary Review:																	

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1.0 The Determination of Phosphorus-32 in Soil and Water

Process Owners:	Signature:	Brian T. Morris
	Signature:	Wilbur B. Sigmon, III
Technical Review by:	Signature:	James B. Westmoreland
Quality Review by:	Signature:	Robert L. Pullano
Approved & Authorized by:	Signature:	Heyward H. Coleman

2.0 Method Objective and Applicability

2.1) This standard operating procedure provides the necessary instructions to conduct the analysis for Phosphorus-32 in soil and water.

3.0 Interferences

3.1) Quench number must fall within the quench calibration curve. Organic interferences may require dilution to reduce quench or addition of nitromethane to increase quench.

3.2) Phosphorus-32 has a half-life of 14.29 days therefore it must be analyzed as soon as possible after sample collection.

3.3) Samples with persistent color or chemical quenching agents not removed in the wash processes may reduce the counting efficiency. Generally these problems are overcome by diluting the sample aliquot.

4.0 Safety Precautions and Hazard Warnings

4.1) Caution should be used when dealing with acids, and other any laboratory reagents. Safety procedures for these are set forth in the Handbook for Good Laboratory Practices, found in the library of the laboratory.

4.2) If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the group leader prior to carrying out the rest of the procedure.

4.3) Nitromethane is a highly combustible substance, take special precautions to keep it away from excessive heat or open flames.

5.0 Apparatus and Materials

5.1) Ancillary Equipment

5.1.1) Plastic scintillation vials, 20 ml capacity.

5.1.2) Filtration apparatus.
5.1.3) Glass fiber filters, 47 mm diameter.
5.1.4) Centrifuge tubes.
5.1.5) Beakers.
5.1.6) Stirring rods.
5.1.7) Liquid scintillation counter and associated equipment

5.2) Reagents, Chemicals and Standards

5.2.1) Citric acid solution. Dissolve 50 g citric acid to 100 ml with DI water.
5.2.2) NIST traceable P-32 standard.
5.2.3) 4 M hydrochloric acid (HCl). Add 333 ml to 500 ml DI water, then dilute to 1000 ml.
5.2.4) 1% ammonium hydroxide (NH_4OH). Mix 10 ml concentrated ammonium hydroxide with 990 ml DI water.
5.2.5) Magnesia mixture. Mix 50 g magnesium chloride ($MgCl \cdot 6H_2O$) with 100 g ammonium chloride (NH_4Cl) in 250 ml DI water, then add 3-5 drops of concentrated hydrochloric acid (HCl) and dilute to 500 ml with DI water.
5.2.6) 0.1 M Nitric acid. Carefully add 6.25 ml concentrated nitric acid (16M) to 1000 ml DI water in a graduated cylinder.
5.2.7) Concentrated hydrogen peroxide 30% (H_2O_2)
5.2.8) DI Water.
5.2.9) Concentrated Ammonium Hydroxide (14.8 M)
5.2.10) Concentrated ammonium hydroxide 14.8 M (NH_4OH).
5.2.11) Scintillation Cocktail. Aquasol-2 or equivalent.
5.2.12) Nitromethane.

5.3) Instrumentation

5.3.1) Liquid Scintillation Counter and associated equipment.

6.0 Sample Collection & Preservation

6.1) Water samples should be collected in plastic bottles and preserved with concentrated nitric acid to pH~2. Soil samples are collected without preservation in suitable containers.

6.2) If the water sample is received with pH greater than 2, the analyst should adjust the sample pH to pH 2, mix and allow to sit overnight before proceeding.

7.0 Equipment and Instrument Maintenance

7.1) Refer to EPI SOP I-004 Liquid Scintillation Counter Operating Procedures for instrument maintenance.

8.0 Preparation of Standard Solutions and Quality Control Samples

8.1) All standards used in this method must be traceable to a nationally accepted source, such as NIST or EPA.

8.2) Dilution of standards should be noted in standard logbook as outlined in EPI SOP M-001 "Preparation of Radioactive Standards". The weight of the standard to be diluted and diluting solutions should be noted in the standards logbook. The weight should be taken on a balance of at least four places and calculation of the new standard activity carried out and noted in appropriate significant figures.

8.3) A reagent blank should be used as the batch blank.

8.4) A duplicate and spiked sample should be made on the same sample in a sample batch. The spiked sample is made by adding a known quantity of P-32 directly to the sample matrix.

8.5) A Laboratory Control Sample (LCS) should be made by spiking deionized water with a known quantity of Phosphorus-32.

9.0 Operating Procedures

9.1) Sample Preparation Techniques: Water.

9.1.1) Measure an appropriate aliquot of water sample into a beaker. Record the sample volume and standard spike information on the que sheet (attachment 1).

9.1.2) Add 10 ml Magnesia mixture and 2 ml Citric acid.

9.1.3) Add concentrated NH_4OH until a precipitate forms. Add an additional 5 ml concentrated NH_4OH as excess.

9.1.4) Add 10 ml 30% hydrogen peroxide.

9.1.5) Stir the sample occasionally while the precipitate collects for 30 min.

9.1.6) Prepare a filter funnel apparatus and filter the precipitate onto the glass fiber filter. Rinse the beaker with 1% NH_4OH.

9.1.7) Wash the filter paper five times with 1% NH_4OH.

9.1.8) Place the funnel apparatus, with the filter, over a centrifuge tube.

9.1.9) Rinse the beaker with 10 ml 4 M HCl and use the rinse to dissolve the precipitate from the filter paper into the centrifuge tube. Allow the rinse to completely drain into the centrifuge tube.

9.1.10) Rinse the funnel with an additional 10 ml 4 M HCl, allowing the rinse to completely drain into the centrifuge tube. Repeat this step again, using 5 ml instead of 10 ml.

9.1.11) Add 1 ml Magnesia mixture, 1 ml 30% H_2O_2 and 1 ml Citric Acid to the centrifuge tube.

9.1.12) Slowly add concentrated NH_4OH to reform the magnesium ammonium phosphate precipitate, then add 5 ml excess concentrated NH_4OH.

9.1.13) Cap and shake the centrifuge tube then allow it to cool for 30 min..

9.1.14) Centrifuge the precipitate, decant and discard the supernatant solution.

9.1.15) Rinse the precipitate with 1% NH_4OH, centrifuge decant and discard the supernatant solution. If the precipitate is colored, repeat this step until the color is eliminated.

9.1.16) Add 4 ml 4M HCl. shake or vortex to dissolve precipitate.

9.1.17) Add the contents of the centrifuge tube to a plastic scintillation vial and add 18 ml scintillation cocktail. Shake or vortex to obtain a homogenous mixture.

9.1.18) Proceed to sample counting, step 9.5.

9.2) Sample Preparation Techniques: Soil.

9.2.1) If not previously done, perform procedure A-021 "Soil Preparation for Radionuclide Analysis".

9.2.2) Weigh a representative aliquot from the homogenized sample and perform procedure A-021b "Sample Ashing, Preparation for Radionuclide Analysis". Record the aliquot information on the que sheet (attachment #1).

9.2.3) Perform procedure A-015 "Microwave Digestion of Soil and Sand".

9.2.4) Treat the digested soil as a liquid sample, proceed with step 9.1.2.

9.3) Instrument Calibration

9.3.1) Prepare the standards such that the matrix will be as similar as possible to actual sample matrices.

9.3.2) The LSC is calibrated by counting 7 to 10 standards with equal activity and different amounts of a quenching agent (nitromethane), and plotting efficiency verses quench number (H#). Refer to EPI SOP I-004 for instructions on establishing a quench curve.

9.3.3) Ensure the master spreadsheet and instrument setup program is updated with the new calibration information. Submit all data associated with the calibration to the group leader with the appropriate calibration checklist.

9.4) Instrument Performance Requirements

9.4.1) Refer to EPI SOP I-010 "Counting Room Instrumentation Maintenance and Performance Checks" for instructions concerning instrument performance.

9.5) Analysis Procedures and Instrumental Operation (Sample Counting)

9.5.1) Place samples in a scintillation rack with the proper user number. Note the rack number and position on the que sheet.

9.5.2) Place the rack in the scintillation counter, and allow it to dark adapt for 5-6 hr. before counting.

9.5.3) Count samples for a duration which is at least sufficient to meet the CRDL and uncertainty requirements.

9.5.4) Please refer to EPI SOP I-004 for guidance concerning analysis procedures and instrument operation.

10.0 Calculations and Data Reduction Methods

10.1) The analyst will use an excel spreadsheet (attachment 2) to calculate the sample pCi/l according to the following equations:

$$\text{Result}\left(\frac{\text{pCi}}{\text{l}}\right) = \frac{\left(S_{cpm} - B_{cpm}\right)}{2.22 * E * I * V}$$

10.2) The counting uncertainty is calculated according to the following equation:

$$\text{Uncertainty}\left(\frac{\text{pCi}}{\text{l}}\right) = \frac{1.96\sqrt{\frac{S_{CPM}}{T_C} + \frac{B_{CPM}}{T_{CB}}}}{(2.22 * E * I * V)}$$

10.3) The method detection limit (MDA) is calculated according to the following equation:

$$MDA\left(pCi/l\right) = \frac{\left(2.71 + 4.66\sqrt{B_{CPM} * T_C}\right)}{\left(2.22 * E * I * V * T_C\right)}$$

Where:
T_C=Sample count duration
T_{CB}=Background count duration
B_{CPM}=Background counts per minute
S_{CPM}=Sample counts per minute
E=Efficiency of counting
V=Sample volume (liters)
I=Decay factor for P-32.

$$I = e^{\frac{-\ln 2 * T_d}{T_{1/2}}}$$

Where:
$t_{1/2}$=Isotopic half life of P-32 (14.29 days).
T_D=Decay time of sample. Calculated from sampling time to midway through count duration.

11.0 Data Recording, Review and Reporting

11.1) Data Recording

11.1.1) Record the required information on the P-32 que sheet.

11.1.2) Transfer applicable data from P-32 que sheet and raw data to P-32 master spreadsheet in Excel (attachment 2).

11.2) Data Review

11.2.1) Refer to EPI SOP D-003 "Data Review and Validation Procedures" for instructions concerning the data review process.

11.3) Data Reporting

11.3.1) The analyst will take the applicable P-32 spreadsheet and enter the data into LIMS. The following information should be included: analyst's initials, date and time the sample was counted, sample results and accuracy in pCi/l.

12.0 Quality Control Requirements

12.1) Analyst and Method Verification Requirements

12.1.1) Refer to EPI SOP D-002 "Analyst and Analytical Methods Validation Procedures" for instructions concerning the validation of analysts and analytical methods.

12.2) Method Specific Quality Control Requirements

12.2.1) A method blank should accompany each batch of samples. The reported value of the blank, should be less than or equal to the contract required detection limit (CRDL).

12.2.2) A matrix spike (MS) should be run with every batch of samples. The recovery of the ms should be between 75-125%. The MS recovery is calculated as follows:

$$\text{MS Recovery } (\%) = \frac{\text{Spike}\left(\frac{\text{pCi}}{\text{unit}}\right) - \text{Sample}\left(\frac{\text{pCi}}{\text{unit}}\right)}{\text{Spike Nominal Concentration}\left(\frac{\text{pCi}}{\text{unit}}\right)} * 100\%$$

Where:
Spike=Results of matrix spike
Sample=Results of sample without added spike
Spike Nominal Concentration (SNC)=Concentration of P-32 in the spike, calculated as follows:

$$SNC = \frac{\text{standard dpm} * \text{ml of spike added}}{2.22 * \text{volume in liters}}$$

12.2.3) A duplicate sample should be run with every batch. The relative percent difference (RPD) between the actual sample and the QC duplicate should be less than or equal to 20% if both the sample and the QC duplicate results are greater than 5 times MDA or 100% if either is less than 5 times MDA but greater than MDA. If either result is less than MDA then limits are not applicable. The RPD should be calculated as follows:

$$RPD\ (\%) = \frac{ABS(DUP_1 - DUP_2)}{\left(DUP_1 + DUP_2/2\right)} * 100\%$$

12.2.4) A Laboratory Control Sample (LCS) should be run with every batch. The recovery of the LCS should fall between 75-125%. The LCS recovery is calculated as follows:

$$LCS_{RECOVERY} = \frac{\text{LCS Result}\left(\frac{pCi}{unit}\right)}{\text{Nominal Concentration of LCS}\left(\frac{pCi}{unit}\right)} * 100\%$$

12.3) Actions Required if the Quality Control Requirements Are Not Met

12.3.1) If any of the QC criteria from 12.2.1 through 12.2.5 cannot be satisfied, the analyst should inform their group leader and initiate a Non-conformance Report as outlined in GEL SOP GL-QS-E-004 "Non-conformance Identification, Control, Documentation, Reporting, and Dispositioning".

13.0 Records Management and Document Control

13.1) All raw data printouts, calculations, spreadsheets, and batch checklists shall be filed with the

sample data for archival and review.

14.0 Laboratory Waste Handling and Disposal

14.1) Radioactive samples and material shall be handled and disposed of as outlined in EPI SOP S-005 "Radioactive Waste Handling Procedures".

14.2) Segregate mixed wastes as appropriate for the type of scintillation cocktail used.

15.0 References

15.1) EML Procedures Manual, 25th ed., H. L. Volchok and G. de Planque, Eds.. Environmental Measurements Laboratory, New York, August 1982.

15.2) Health and Environmental Chemistry, "Phosphorus-32 in Urine", Los Alamos National Laboratory, January 1989.

Attachment 1

P-32 in Water Que Sheet

Batch #
Spike Isotope: P-32
LCS isotope P-32

Prep Date:_____
Spike Code:_____
LCS Code:_____

Comment:
Initials: _____
Vol:_____
Vol:_____

Instrument Used: (circle one) Beckman LS6000 7065155 Beckman LS6500 7067083
s/n:

Sample ID	*Sample Descrip.*	*Btr #*	*Rack position*	*Sample Mass (g) or (ml)*	*P-32 Sampling Date/Time*

Data Reviewed By: _____

STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF PROMETHIUM-147

IN SOIL AND WATER

UNCONTROLLED DOCUMENT

(GL-EPI-A-020 Revision 3)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Process Owner	11/19/99 (Sign and Date)	Frank Douglas Parik (Print Name)
Technical Review	11/19/99 (Sign and Date)	Richard M. Kinney (Print Name)
Quality Review	11/19/99 (Sign and Date)	Tim R. Winters (Print Name)
Approval and Authorization	11/19/99 (Sign and Date)	James B. Westmoreland (Print Name)

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF PROMETHIUM-147 IN SOIL AND WATER

2.0 METHOD OBJECTIVE AND APPLICABILITY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for Promethium-147 in soil and water.

3.0 INTERFERENCES

3.1 Quench number must fall within the quench calibration curve. Organic interferences may require dilutions to reduce quench or addition of carbon tetrachloride to increase quench.

3.2 Samples with persistent color or chemical quenching agents not removed in the wash processes may reduce the counting efficiency. Generally these problems are overcome by diluting the sample aliquot.

3.3 This method is based on the relationship between Pm and Am chemistry. Pm is discriminated from interferences by proper scintillation window settings.

3.4 Almost all of the light lanthanides Ce, Pr, Nd, Sm, and Eu are potential interferences. The process and counting is not specific for Pm. However, carefully adjusted window settings are helpful.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 If there is any question regarding the safety of any laboratory practice, stop **immediately** and consult the group leader prior to carrying out the rest of the procedure.

5.0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment

5.1.1 Ion exchange columns. EIchroM Industries Inc. TRU-Spec® prepacked column with 25 mL reservoir

5.1.2 Scintillation vials, glass

5.1.3 Centrifuge tubes

5.1.4 Beakers

5.1.5 Stirring rods

5.1.6 Membrane filters, Millipore HAWP or equivalent 25 mm diameter.

5.2 Reagents, Chemicals and Standards

5.2.1 Ammonium hydroxide, concentrated (14.8 M)

5.2.2 Calcium nitrate.(10 mg/mL). Dissolve 5.92g of $Ca(NO_3)_2$ in 100 mL of deionized water

5.2.3 NIST traceable standard Pm-147

5.2.4 Nitric acid, concentrated 16 M

5.2.5 Nitric acid (2 M). Dilute 12.5 mL of conc. nitric acid to 100 mL of deionized water

5.2.6 Phosphoric acid, concentrated

5.2.7 Hydrochloric acid (4M). Dilute 333 mL of hydrochloric acid to 1000 mL with deionized water.

5.2.8 1 M Oxalic acid. Dilute 126 g oxalic acid ($H_2C_2O_4*2H_2O$) to 1000 mL with deionized water.

5.2.9 Neodymium carrier, 1 mg Nd/mL. This standard is obtained from High Purity Standards. However, it must be standardized according to the sample process used in preparing samples for counting so that water of hydration or other factors that cause the empirical chemical formula to differ from the theoretical, may be taken into account. The carrier solution is standardized by precipitating three 5 mL aliquots of the standard as the oxalate at a pH of 2-3. The samples are filtered through tarred Gooch crucibles and dried under a heat lamp to constant weight. A satisfactory standardization is obtained when triplicate results give a relative standard deviation of $\leq$0.5%.

5.2.10 Scintillation Cocktail, Ready Gel

5.2.11 Hydrochloric acid (1.5 M). Dilute 12.5 mL concentrated HCL acid to 100 mL with deionized water.

5.2.12 Hydrochloric acid (6 M). Dilute 50 mL of concentrated HCL acid to 100 mL with deionized water.

5.2.13 Hydrochloric acid (10 M). Dilute 83.3 mL of concentrated HCL acid to 100 mL with deionized water.

5.2.14 Hydrochloric acid (12 M)

5.2.15 Ferric nitrate carrier (10 mg Fe/mL). Dissolve 14.5 g $Fe(NO_3)_3*9H_2O$ in 200 mL deionized water.

5.2.16 Methyl violet, pH indicator.

5.3 Instrumentation

5.3.1 Liquid Scintillation Counter and associated software.

6.0 SAMPLE COLLECTION AND PRESERVATION

6.1 Water samples should be collected in plastic bottles and preserved with concentrated nitric acid to pH~2.

6.2 If the sample is received with pH greater than 2, the analyst should modify the sample to pH 2, mix and allow to sit overnight before proceeding.

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to "Beckman LS 6000/6500 Series Operating Procedures" (GL-EPI-I-004) for instrument maintenance.

8.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

8.1 Refer to "Preparation of Radioactive Standards" (GL-EPI-M-001) for instructions concerning the preparation of standard solutions.

9.0 OPERATING PROCEDURES

9.1 Sample Preparation Techniques: Water.

9.1.1 Add an aliquot of sample to a labeled beaker. Add 1.0 mL Nd carrier and 1 mL of calcium carrier.

9.1.2 Heat the sample to a slow boil. Add 0.5 mL concentrated phosphoric acid.

9.1.3 While stirring slowly, add concentrated NH_4OH until turbidity persists, or pH>9. Continue heating for 10 minutes and then allow the precipitate to settle and cool.

9.1.4 Decant excess supernate and discard. Collect remaining precipitate by centrifugation in a centrifuge tube. Discard supernate.

9.1.5 Dissolve the precipitate in 10 mL of 2 M nitric acid.

9.1.6 Prepare the TRU-Spec® column by pre-rinsing it with 10 mL of 2 M nitric acid.

9.1.7 Load the sample onto the column and discard the column effluent.

9.1.8 Rinse with 3 mL of 2 M nitric acid and discard effluent. Repeat three times. Rinse with additional 2 M nitric acid if yellow color persists.

9.1.9 Add 2 mL of 10 M HCL, collect in clean centrifuge tube.

9.1.10 Elute Pm (and other light rare earth elements) with 10 mL 4 M HCL.

9.1.11 Add 2 mL 1 M oxalic acid to the sample in the centrifuge tube.

9.1.12 Add 5 - 10 drops methyl violet pH indicator.

9.1.13 Add concentrated NH_4OH dropwise to raise pH to > 2 but < 4 in order to form a precipitate. (A blue color is an indication of a pH approximately 2).

9.1.14 Allow the centrifuge tube to cool.

9.1.15 Filter the precipitate onto a preweighed membrane filter.

9.1.16 Wash the precipitate with 10 mL each of deionized water and 80% ethanol.

9.1.17 Dry the filter and weigh the precipitate to calculate yield. Record the data on the que sheet (Appendix 1).

9.1.18 Place filter in glass scintillation vial. Add 2 mls 8M HNO_3 to dissolve precipitant.

9.1.19 Pull filter out and evaporate to dryness.

9.1.20 Allow to cool and add 5 mls 0.05M HNO_3.

9.1.21 Add 15 mls of Ready Gel, then cap and shake vial well.

9.1.22 Proceed to step 9.5 for sample counting.

9.2 Sample Preparation Techniques: Soil.

9.2.1 If not previously done, perform procedure "Soil Sample Preparation for the Determination of Radionuclides," (GL-EPI-A-021).

9.2.2 Measure out an aliquot of the homogenized sample and add 1.0 mL Nd carrier.

9.2.3 Leach solid with an appropriate amount (approx. 4x sample aliquot) of 6 M HCL for 1-2 hours.

9.2.4 Centrifuge the leachate and collect the supernate.

9.2.5 Precipitate the hydroxides by the addition of concentrated NH_4OH to a pH > 7.

9.2.6 Separate and wash the precipitate by centrifuging. Then add as much 1.5 M HCL as is necessary to dissolve all of the precipitate.

9.2.7 Prepare a 13mm column by adding 10cm of AG50Wx4 (hydrogen form 100-200 mesh). Prerinse with 50 mL of 1.5 M HCL.

9.2.8 Load sample and discard eluant.

9.2.9 Rinse with 80 mL 1.5 M HCL.

9.2.10 Elute rare earth elements with 120 mL of 6 M HCL.

9.2.11 Add 1 mL Fe carrier to solution, precipitate FeOH by adding concentrated NH_4OH to a pH > 7.

9.2.12 Centrifuge and decant, then wash precipitate and centrifuge again.

9.2.13 Decant and redissolve precipitate in a minimum amount of 2 M HNO_3 (less than 20 mL).

9.2.14 The sample is now ready for column loading. Treat soil sample as a liquid sample and proceed with step 9.1.6.

9.3 Instrument Calibration

9.3.1 Establishing a quench curve

9.3.1.1 Prepare a set of standards consisting of 8 to 12 standards using the same matrix and cocktail as the samples to be measured. Add approximately 10,000 DPM to each standard, cap and shake.

NOTE: Do not add any quenching agent at this point.

9.3.1.2 Allow the standards to dark adapt for a minimum of 1 hour.

9.3.1.3 Using an available counting program, measure the observed CPM of each of the standards to verify accurate pipetting. All standards should agree within +/- 5% of the mean. Discard any standards that do not meet this criteria.

9.3.1.4 Add 0 to 220 ul of carbon tetrachloride in 20 ul increments to each of the standards.

9.3.1.5 Recount the standards for a period of 1 minute to determine if the range of H#'s covers the desired range of sample quench. If the range of the standards is not large enough to create more standards, adjust the amount of quenching agent appropriately. Record the amount of quenching agent used for each standard on the calibration source preparation sheet (Appendix 2).

9.3.1.6 Count the standard for a sufficient length of time to accumulate at least 10,000 counts in the highest quenched standard. Plot efficiency vs. H#.

9.3.2 Calibration verification

9.3.2.1 Count the standards against the newly established curves. The acceptance criteria being 100% +/- 10% accuracy measured against a known value.

9.4 Instrument Performance Requirements

9.4.1 Refer to "Counting Room Instrumentation Maintenance and Performance Checks," (GL-EPI-I-010) for instructions concerning instrument performance.

9.5 Analysis Procedures and Instrumental Operation

9.5.1 Refer to "Beckman LS-6000/6500 Operating Procedures" (GL-EPI-I-004) for guidance concerning analysis procedures and instrument operation.

10.0 CALCULATIONS AND DATA REDUCTION METHODS

10.1 The analyst will use an excel spreadsheet to calculate the sample pCi/unit according to the following equations:

$$\text{Result}\left(\text{pCi}/\text{unit}\right)=\frac{(\text{SCPM}-\text{BCPM})}{2.22*\text{E}*\text{I}*\text{V}*\text{Y}}$$

10.2 The counting uncertainty is calculated according to the following equation:

$$\text{Error}\left(\text{pCi}/\text{unit}\right)=\frac{\left(1.96\sqrt{\left(\frac{S_{CPM}}{T_C}+\frac{B_{CPM}}{T_{CB}}\right)}\right)}{(2.22*\text{E}*\text{I}*\text{V}*\text{Y})}$$

10.3 The method detection limit (MDA) is calculated according to the following equation:

$$\text{MDA}\left(\text{pCi}/\text{unit}\right)=\frac{(2.71+4.66\sqrt{B_{CPM}*T_C})}{(2.22*\text{E}*\text{I}*\text{V}*T_C*\text{Y})}$$

Where:

T_C=Sample count duration
T_{CB}=Background count duration
B_{CPM}=Background counts per minute
S_{CPM}=Sample counts per minute
E=Efficiency of counting
V=Sample volume (liters)
Y=Chemical yield
I=Decay factor for PM-147.

$$I=e^{\left(-\frac{\ln(2)*T_D}{t_{1/2}}\right)}$$

Where:

$t_{1/2}$=Isotopic half life of PM-147 (2.6234 years).
T_D=Decay time of sample. Calculated from sampling time to midway through count duration.

11.0 DATA RECORDING, REVIEW AND REPORTING

11.1 Data Recording

11.1.1 Record the information required on the PM-147 que sheet.

11.1.2 Transfer applicable data from PM-147 que sheet to PM-147 spreadsheet in Excel.

11.2 Data Review

11.2.1 Refer to "Data Review and Validation Procedures" (GL-EPI-D-003) for instructions concerning the data review process.

11.3 Data Reporting

11.3.1 The analyst will take the applicable PM-147 spreadsheet and enter the data into LIMS. The following information should be included: analyst's initials, date and time the sample was counted, sample results and accuracy.

12.0 QUALITY CONTROL REQUIREMENTS

12.1 Analyst and Method Verification Requirements

12.1.1 Refer to "Analyst and Analytical Methods Validation Procedures" (GL-EPI-D-003) for instructions concerning the validation of analysts and analytical methods.

12.2 Method Specific Quality Control Requirements

12.2.1 A method blank should accompany each batch of samples. The reported value of the blank, should be less than or equal to the contract required detection limit (CRDL).

12.2.2 A matrix spike (ms) should be run with every batch of samples. The recovery of the ms should be between 75-125%. The ms recovery is calculated as follows:

$$\text{MS Recovery (\%)} = \frac{\text{Spike}\left(\text{pCi}/\text{unit}\right) - \text{Sample}\left(\text{pCi}/\text{unit}\right)}{\text{Spike Nominal Concentration}\left(\text{pCi}/\text{unit}\right)} * 100\%$$

Where:

Spike=Results of matrix spike
Sample=Results of sample without added spike
Spike Nominal Concentration (SNC)=Concentration of PM-147 in the spike, calculated as follows:

$$\text{SNC} = \frac{\text{standard dpm} * \text{ml of spike added}}{2.22 * \text{volume in liters}}$$

12.2.3 A duplicate sample should be run with every batch. The relative percent difference (RPD) between the actual sample and the QC duplicate should be less than or equal to 20% if both the sample and the QC duplicate results are greater than 5 times MDA or 100% if they are both less than 5 times MDA. The RPD should be calculated as follows:

$$\text{RPD (\%)} = \frac{\text{ABS}(\text{DUP}_1 - \text{DUP}_2)}{\left[\left(\text{DUP}_1 + \text{DUP}_2\right)/2\right]} * 100\%$$

12.2.4 A Laboratory Control Sample (LCS) should be run with every batch. The recovery of the LCS should fall between 75-125%. The LCS recovery is calculated as follows:

$$\text{LCS}_{\text{RECOVERY}} = \frac{\text{LCS Result}\left(\text{pCi}/\text{unit}\right)}{\text{Nominal Concentration of LCS}\left(\text{pCi}/\text{unit}\right)} * 100\%$$

NOTE: Some clients may have more or less stringent requirements.

12.3 Actions required if the Quality Control Requirements Are Not Met

12.3.1 If any of the QC criteria from 12.2.1 through 12.2.4 cannot be satisfied, the analyst should inform their Group Leader and initiate a Non-conformance Report as outlined in GEL SOP "Documentation of Non-conformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

13.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

13.1 All raw data printouts, calculations, spreadsheets, and batch checklists shall be filed with the sample data for archival and review.

14.0 LABORATORY WASTE HANDLING AND DISPOSAL

14.1 Radioactive samples and material shall be handled and disposed of as outlined in "Laboratory Waste Disposal and Emergency Instructions," (GL-EPI-S-011).

14.2 Segregate mixed wastes as appropriate for the type of scintillation cocktail used.

15.0 REFERENCES

15.1 EML Procedures Manual, 25th ed. "Radiological Determination of Promethium-147", H. L. Volchok and G. de Planque, Eds. Environmental Measurements Laboratory, New York, August 1982.

15.2 Special thanks to Andy Rollins, Ph.D. at EiChroM Industries Inc. for his assistance in developing this method.

15.3 Special thanks to Bill Burnette, Ph.D. at Florida State University, Department of Oceanography for his assistance in developing this method.

APPENDIX 1

Pm-147 Que Sheet Batch #: 93162 Analyst: IDXL Automatic Comments:

Witness:_____

Pm-147 Spike Code:_____ Vol:_____ Balance #:_____ Prep-Date:_____

Pm-147 LCS Code(s):_____ Vol:_____ Pipet #:_____ Initials :_____

Minimum Duedate: 11/15/96

Sample ID	Client Description		Min RDL	Client	Sample Date/Time	Blk #	Aliquot (mL or g)	LCS Rack #	Initial Weight	Final Weight (mg)	Net Weight (mg)
9610624-03	109088		10 PCL	WSRC	10/29/96 1030						
QC397571	blank	LANK	10 pCi/	QC	11/1/96						
QC397572	9610624-03	DUP	10 pCi/	QC	10/29/96 1030						
QC397573	9610624-03	MS	10 pCi/	QC	10/29/96 1030						
QC397574	lcs	LCS	10 pCi/	QC	11/1/96						

Instruments Used (circle as appropriate):

LS6000 (Red) 7065155 LS6500 (Black) 7069123

LS6500 (Blue) 7067083 WALLAC (Yellow) 404012

LS6500 (Green) 7067404 WALLAC (Pink) Z200682

Data Reviewed By:_____

Page 1 of 1

APPENDIX 2

General Engineering Laboratories
Calibration Source Preparation Sheet

Applicable SOP Number _____ Isotope _____

Date Standards Prepared _____ Cocktail Type Used _____

Standard ID _____ Mass of [illegible] _____

Amount Used (g or ml) _____

Standard Activity (DPM/g or mL) _____ Type of Scintillation Vial _____

Reference Date _____ Pipette ID Used _____

Expiration Date _____ Balance ID Used _____

Residue/Carrier Agent _____ Quenching Agent _____

Sample number	Standard Number	Quenching Vol (mL)/ (Residue Volume/mL)	Initial Wt. (g)	Final Wt. (g)	Net Wt. (avg)

Prepared By: _____ Date _____

Reviewed By: _____ Date _____

Rev 1 RLM 8/13/97

STANDARD OPERATING PROCEDURE

FOR

UNCONTROLLED DOCUMENT

SOIL SAMPLE PREPARATION

FOR

THE DETERMINATION OF RADIONUCLIDES

(GL-EPI-E-A-021 Revision 3)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

Process Owner	10/14/99 (Sign and Date)	Tim C. Winters (Print Name)
Technical Review	10/14/99 (Sign and Date)	Richard M. Kinney (Print Name)
Quality Review	10/15/99 (Sign and Date)	Robert L. Pillano (Print Name)
Approval and Authorization (Sign and Date)	10/15/99	James B. Westmoreland (Print Name)

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR SOIL SAMPLE PREPARATION FOR THE DETERMINATION OF RADIONUCLIDES

2.0 METHOD OBJECTIVE AND APPLICABILITY

2.1 This standard operating procedure provides the necessary instructions to conduct the preparation of soil samples for radionuclide determination.

3.0 INTERFERENCES

3.1 This procedure involves drying the soil at 110°C. If that temperature would volatalize any component for which an analysis has yet to be run, a separate aliquot must be set aside for such analyses.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 Handle samples carefully to avoid dust, clean equipment thoroughly to prevent subsequent sample cross contamination.

4.2 Equipment involved in this procedure is of industrial use. Keep clear of moving parts, including any loose clothing.

4.3 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the Group Leader prior to carrying out the rest of the procedure.

5.0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment

5.1.1 Metal cans ~ quart size

5.1.2 Steel balls ~1" diameter

5.1.3 Drying oven or segmented muffle furnace, able to maintain ±2°C

5.1.4 Sieve Screens (500µm or 32 mesh)

5.1.5 Paper funnels

5.1.6 100cc aluminum cans

5.2 Reagents, Chemicals and Standards

5.2.1 N/A

5.3 Instrumentation

5.3.1 Paint can shaker, heavy duty

5.3.2 Analytical balance

6.0 SAMPLE COLLECTION & PRESERVATION

6.1 Soil samples should be collected and maintained in a sealed container except for sample preparation and removal of aliquots for subsequent analysis.

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to the technical manual provided with the paint shaker for information regarding equipment maintenance.

7.2 The analytical balance should be cleaned after use, and especially when weighing soils.

8.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

8.1 Method blanks should be run through the entire soil preparation process for each analysis bi-annually to ensure contamination buildup does not occur.

9.0 OPERATING PROCEDURES

9.1 Sample Preparation Techniques

9.1.1 Label a clean metal container with the laboratory sample number.

9.1.2 If % moisture is required on the sample being prepared, enter the container weight into the computerized soil prep balance log.

9.1.3 Transfer an appropriate aliquot from the field collection container to the labeled container.

9.1.4 If % moisture is required on the sample being prepared, enter the pre-oven sample weight into the computerized soil prep balance log.

9.1.5 Place the container in a drying oven set at 110° C until the sample has reached a constant weight, normally a period of ~ 24 hours.

9.1.6 Using heat resistant gloves, remove the sample from the oven and allow to cool.

9.1.7 If % moisture is required on the sample being prepared, enter the post-oven sample weight into the computerized soil prep balance log.

9.1.8 Homogenize the sample. This is normally accomplished by placing a lid on the container and placing the container in the industrial paint shaker. Depending on the matrix of the soil, it may be necessary to add several stainless steel balls inside the container to assist in homogenizing the sample. The length of time the sample remains on the shaker is dependent on the matrix of the sample, and normally ranges from 5 – 30 minutes.

9.1.9 Remove the metal container from the shaker and allow to settle for several minutes.

9.1.10 Place the container in front of the sample preparation hood and remove the lid. If stainless steel balls were added to the container, they should now be removed.

9.1.11 Determine an appropriate aliquot based on the analysis required. Normally, depending upon the required analysis, the sample will be passed through a sieve screen or placed directly into a labeled sample container.

9.1.12 Return the unused portion of prepared sample back into the original field container, if no other test is required on the sample.

9.1.13 Seal the container. The soil sample is now ready for radiochemical analysis.

9.2 Instrument Calibration

9.2.1 N/A

9.3 Instrument Performance Requirements

9.3.1 N/A

9.4 Analysis Procedures and Instrumental Operation

9.4.1 N/A

10.0 Calculations and Data Reduction Methods

10.1 The electronic balance program provides documentation of all necessary raw data. The raw data can be used to provide clients with sample results of wet weights by using ratios.

12.0 Quality Control Requirements

12.1 Analyst and Method Verification Requirements

12.1.1 Refer to GL-EPI-D-002 for information concerning analyst and method verification.

12.2 Method Specific Quality Control Requirements

12.2.1 See the specific isotope operating procedure for instructions concerning method quality control requirements.

12.3 Actions required if the Quality Control Requirements are not met

12.3.1 If any of the quality criteria cannot be satisfied, the analyst should inform the Group Leader and initiate a non-conformance report as outlined in GL-QS-E-004 "Nonconformance Identification Control, Documentation, Reporting, and Dispositioning".

13.0 Records Management and Document Control

13.1 The que sheets shall be sequentially numbered and kept in a binder for archival and review.

14.0 Laboratory Waste Handling and Disposal

14.1 Radioactive samples and material shall be handled and disposed of as outlined in GL-EPI-S-005 "Radioactive Waste Handling Procedures".

14.2 When all analyses have been successfully completed, the remaining sample can be transferred back to the original field container for return. This will also allow for disposal of the metal can.

15.0 References

15.1 Storer, Roberta A. Director, Ed. Svcs. 1993 Annual Book of ASTM Standards Volume 12.01 C999-90 pp. 477-478 Philadelphia PA ASTM 1993

STANDARD OPERATING PROCEDURE

FOR

UNCONTROLLED DOCUMENT

SOIL SAMPLE ASHING

FOR

THE DETERMINATION OF RADIONUCLIDES

(GL-EPI-E-A-021b Revision 1)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Chris C. Martin 11/5/99		Christopher C. Martin	
Process Owner	(Sign and Date)	(Print Name)	
Tim C. Winters 11/5/99		Tim C. Winters	
Technical Review	(Sign and Date)	(Print Name)	
Robert J. Pellegrino 11/8/99		Robert C. Pellegrino	
Quality Review	(Sign and Date)	(Print Name)	
James B. Westmoreland 11/8/99		James B. Westmoreland	
Approval and Authorization	(Sign and Date)	(Print Name)	

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR SOIL SAMPLE ASHING FOR THE DETERMINATION OF RADIONUCLIDES

2.0 METHOD OBJECTIVE AND APPLICABILITY

2.1 This standard operating procedure provides the necessary instructions to conduct the ashing of soil/organic samples for radionuclide determination.

3.0 INTERFERENCES

3.1 This procedure involves ashing the soil at 550°C. If that temperature would volatalize any component for which an analysis is to be run, this procedure should not be carried out. Refer to the source procedure for alternate ashing procedures, when applicable.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 Handle samples carefully to avoid dust, clean equipment thoroughly to prevent subsequent sample cross contamination.

4.2 Equipment involved in this procedure is of industrial use. Do not attempt to open the furnace when it is above 200°C. Allowing air to flow in as the door is open may allow enough oxygen entry to cause an explosion.

4.3 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the group leader prior to carrying out the rest of the procedure.

4.4 Wear special protective gloves for heat resistance when removing hot samples from the furnace. The analyst should use tongs when removing samples from the furnace.

5.0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment

5.1.1 Borosilicate glass vials.
5.1.2 High temperature resistant gloves.
5.1.3 Tongs.

5.2 Reagents, Chemicals and Standards

5.2.1 Not Applicable

5.3 Instrumentation

5.3.1 Segmented muffle furnace.

6.0 SAMPLE COLLECTION & PRESERVATION

6.1 Refer to the applicable source method for information regarding sample collection and preservation.

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to the technical manual provided with the furnace for information regarding equipment maintenance.

8.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

8.1 Refer to the applicable source method for preservation of standard solutions and quality control samples.

9.0 OPERATING PROCEDURES

9.1 Sample Preparation Techniques

9.1.1 Preweigh empty glass vials and enter into Balance Program.

9.1.2 Place aliquot into a glass vial, weigh and enter into Balance Program.

9.1.3 Place the vials in the furnace with even spacing. A removable shelf is provided, which can be used as necessary.

9.2 Instrument Calibration

9.2.1 Refer to the technical manual provided with the furnace for information regarding instrument calibration.

9.3 Instrument Performance Requirements

9.3.1 The furnace has been programmed to maintain the specified temperatures for the predetermined times. Holdback, high temperature, and low temperature alarms provide adequate protection of furnace continuity. Should an alarm setpoint be reached, consult the technical manual and a group leader.

9.3.2 The pre-programmed ashing routine proceeds as follows:

9.3.2.1 Ramp from ambient (room temperature) to 300°C at 15°C per minute.

9.3.2.2 Hold at 300°C for 3 hours.

9.3.2.3 Ramp from 300°C to 550°C at 15°C per minute.

9.3.2.4 Hold at 550°C for 8 hours.

9.3.2.5 End of program, return to ambient.

9.3.3 The total time for completion of this routine is ~12 hours.

9.4 Analysis Procedures and Instrumental Operation

9.4.1 Pre-programmed operation (normal):

9.4.1.1 Press the [PAR] button until reaching the display "Prog".

9.4.1.2 Use the [⇧] or [⇩] button to change the display to "Run".

9.4.1.3 The furnace will automatically begin the ashing cycle.

9.4.1.4 If the analyst desires a longer hold time at a particular temperature, they may do so by entering the "Prog" display and using the arrow buttons to place it into "Hold". When the additional time has been met, return the mode to run and the furnace will resume the ashing cycle. Note any deviation from the set program on the method que sheet.

9.4.1.5 If the analyst desires to stop the ashing cycle, enter the "Prog" display, and switch the setting to "Idle" with the arrow buttons. Note this action on the method que sheet.

9.4.1.6 Use tongs and/or wear protective gloves and remove the samples when the furnace temperature has decreased below 100°C.

9.4.2 Manual operation:

9.4.2.1 Use the [⇧] or [⇩] button to change the furnace temperature setting to the desired level.

9.4.2.2 The furnace will stay at the set level until further changes are made.

10.0 Calculations and Data Reduction Methods

10.1 A que sheet provides documentation of all necessary raw data.

11.0 Data Recording, Review and Reporting

11.1 Data Recording

11.1.1 Record the information required on the applicable method que sheet.

11.2 Data Review

11.2.1 Refer to GL-EPI-D-003 "Data Review and Validation Procedures" for instructions concerning the data review process.

11.3 Data Reporting

11.3.1 The furnace temperature log (Attachment 1) should be completed each day the furnace is used.

12.0 Quality Control Requirements

12.1 Analyst and Method Verification Requirements

12.1.1 Refer to GL-EPI-D-002 for information concerning analyst and method verification.

12.2 Method Specific Quality Control Requirements

12.2.1 See the specific method operating procedure for instructions concerning method quality control requirements.

12.3 Actions required if the Quality Control Requirements are not met

12.3.1 If any of the quality criteria cannot be satisfied, the analyst should inform the group leader and initiate a non-conformance report as outlined in GL-QS-E-004 "Nonconformance Identification Control, Documentation, Reporting, and Dispositioning."

13.0 Records Management and Document Control

13.1 Data generated from this method is recorded on the applicable method que sheet, and controlled as described in applicable method standard operating procedure.

13.2 The furnace temperature log (Attachment 1) should be completed each day the furnace is used.

14.0 Laboratory Waste Handling and Disposal

14.1 Radioactive samples and material shall be handled and disposed of as outlined in GL-EPI-S-005 "Radioactive Waste Handling Procedures".

15.0 REFERENCES

15.1 Margaret A Gautier, Ed. Health and Environmental Chemistry: Analytical Techniques, Data Management, and Quality Assurance (LA-10300-M) Volume II, pp. ER110-7, March 1992, revised July, 1993.

15.2 Philip W. Krey, Acting Director. EML Procedures Manual (HASL300) 27th Edition, Volume I, pp. 2-78 through 2-87, Issued November 1990, revised February 1992.

15.3 Robert Lieberman, Ed. Eastern Environmental Radiation Facility Radiochemistry Procedures Manual, (EPA) pp. P-01-2 and Sr-01-3, June 1984

15.4 Louis Z. Bodnar and Donald R. Percival, Editors. RESL Analytical Chemistry Branch Procedures Manual (DOE) pp. SP-1-9 through SP-1-11, 1982.

Attachment 1

General Engineering Laboratories, Inc.

MUFFLE FURNACE LOG

Page ______ of ______

MUFFLE FURNACE #004 Location: Radiochemistry

Thermocouple ID: 927247

QC Ranges: METHOD SPECIFIC

For ____________ 19____
(MONTH)

PLEASE USE BLACK INK

OUT OF RANGE CONDITIONS REQUIRE ACTION

Date	Time	Temp	Target	W/in QC?	Initials	Method	Action Taken

QUALITY REVIEW AND APPROVAL:______________________ DATE:______________

IN THE EVENT OF FAILURE CONTACT: Mike Barnes Ext: Page

geiqmf053

12.1.2 Refer to GL-EPI-E-D002 for information concerning analyst and method verification.

12.2 Method Specific Quality Control Requirements

12.2.1 A matrix spike (ms) should be run with every batch of samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows.

$$\text{Rec} = \frac{\text{Spike}(\text{pCi / unit}) - \text{Sample}(\text{pCi / unit})}{\text{Nominal Concentration}(\text{pCi / unit})} * 100$$

12.2.2 A sample duplicate should be run with every batch of samples. The relative percent difference (RPD) between the sample and the sample duplicate should be less than or equal to 20%. If both results are greater than 5X MDA. If either result is between 5X MDA and MDA limit is ≤ 100. If either result is less than MDA then limits are not applicable. The RPD is calculated as follows.

$$\%\text{RPD} = \frac{\text{High Activity}(\text{pCi / unit}) - \text{Low Activity}(\text{pCi / unit})}{\text{Average}(\text{pCi / unit})} * 100$$

12.2.3 A method blank should accompany each batch of samples. The reported value should be less than or equal to requested MDA. If the value of the blank activity is not below the requested MDA then an adequate explanation should be given.

12.2.4 A Laboratory Control Sample (LCS) should be run with each batch of samples. The recovery of the LCS should fall between 75-125%. The recovery is calculated as follows:

$$\%\,\text{Rec} = \frac{\text{Observed pCi / l}}{\text{Known pCi / l}} * 100$$

12.3 Actions Required if the Quality Control Requirements Are Not Met

12.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non conformance report as

outlined in GEL-SOP GL-QS-E-004 "Nonconformance Identification Control, Documentation, Reporting and Dispositioning".

13.0 Records Management and Document Control

13.1 Raw data and associated documentation will be stored in binder books or in files and kept on hand for any subsequent reviews necessary. The records should be kept on hand for a length of time not shorter than 1 year.

13.2 Data generated by the micro-Vax 3100 will be backed up during routine software backup.

13.3 Quality control charts for spike and blank results are kept on the micro-Vax 3100.

14.0 Laboratory Waste Handling and Disposal

Radioactive material is handled and disposed of as outlined in GL-EPI-E-S005, "Radioactive Waste Handling Procedure".

15.0 References

15.1 Babcock & Wilcox, "Determination of Fe-55 in Water and Soil", 1985.

Attachment 1

Fe-55 Que Sheet

Batch #
Prep Date:_______________
Spike Isotope:__________
Spike Code:_______________
LCS Isotope:__________
LCS Code:_______________
Instrument Used: (circle one) LS6500 LS6000

Comment: *none*
Initials: ________
Vol:__________
Vol:__________
Batched to:

Sample ID	*Sample Descrip.*	*Vol (mls or g)*	*Initial Weight (g)*	*Final Weight (g)*	*Net Weight (mg)*

Data Reviewed By: ______________________

STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF NI-59 and NI-63 IN SOIL AND WATER

(GL-EPI-E-A-022 Revision 3)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Process Owner	10/11/99 (Sign and Date)	Tim C. Winters (Print Name)
Technical Review	10/12/99 (Sign and Date)	Richard M. Kinney (Print Name)
Quality Review	10/15/99 (Sign and Date)	Robert L. Pollard (Print Name)
Approval and Authorization (Sign and Date)	10/15/99	James B. Westmoreland (Print Name)

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TABLE OF CONTENTS

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1.0 THE DETERMINATION OF NICKEL-59 AND NICKEL-63 IN SOIL AND WATER

2.0 METHOD OBJECTIVE AND APPLICABILITY

This standard operating procedure provides the necessary instructions to conduct the analysis of Ni-59 and/or Ni-63 in soil or water.

3.0 INTERFERENCES

3.1 Organic complexing agents, such as EDTA must be destroyed to attain a quantitative precipitation of nickel as nickel dimethylglyoxime.

3.2 Palladium can be a source of interference when present in large quantities due to an interference in the gravimetric recovery.

4.0 SAFETY PRECAUTIONS AND HAZARD WARNINGS

4.1 Wear eye protection with shields while in the laboratory.

4.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory, as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the laboratory. Individual sample MSDS forms provided by the clients are kept in Login.

4.3 Gloves are required when handling the chemicals in this procedure.

4.4 Work under a hood when using concentrated acids and bases.

4.5 The handling of radioactive samples is outlined in "Handling of Radioactive Samples," GL-EPI-E-S-004.

4.6 Refer to "Laboratory Waste Disposal and Emergency Instructions," GL-LB-E-011, for instructions on how materials are disposed.

4.7 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the Group Leader prior to carrying out the rest of the procedure.

5.0 APPARATUS AND MATERIALS

5.1 Ancillary Equipment

5.1.1 Glass beakers

5.1.2 Disposable transfer pipet

5.1.3 Disposable centrifuge tubes

5.1.4 Hot plate

5.1.5 Tuffryn filters or equivalent

5.1.6 2 inch stainless steel planchet

5.1.7 0.25 mil mylar xray film

5.1.8 20 mL glass liquid scintillation vial

5.1.9 Drying oven

5.1.10 Analytical balance

5.2 Reagents, Chemicals and Standards

5.2.1 Deionized water.

5.2.2 Fe Carrier (~10 mg/mL). Dissolve 48.4 g $FeCl_2$ in 1 liter of 1 M HCl.

5.2.3 Ni Carrier (10 mg/mL). This standard is obtained from High Purity Standards. However, it must be standardized according to the sample process used in preparing samples for counting. The carrier solution is standardized by precipitating three 5 mL aliquots of the standard. The samples are filtered through tared Gooch crucibles and dried under a heat lamp to a constant weight. A satisfactory standardization is obtained when triplicate results give a relative standard deviation of less than 0.5%.

5.2.4 Concentrated Ammonium hydroxide.

5.2.5 1 M Ammonium citrate.

5.2.6 0.125 M dimethyl glyoxime in methanol (pH adjusted to 8 with NH_4OH). Dissolve 14.52 g DMG in 1 liter of methanol.

5.2.7 Methanol.

5.2.8 Concentrated Nitric Acid.

5.2.9 Hydrogen Peroxide, 30%.

5.2.10 0.7 M Hydrochloric acid. Dilute 58.5 mL of conc. HCl to 1 liter with deionized water.

5.2.11 Ready gel liquid scintillation cocktail (or equivalent).

5.2.12 1 M HNO_3. Dilute 62.5 mL of conc. HNO_3 to 1000 mL with deionized water.

5.2.13 6 M HCl. Dilute 500 mL of conc. HCl to 1000 mL with deionized water.

5.3 Instrumentation

5.3.1 Low Energy germanium detector capable of measuring emissions at 6 keV, with a data acquisition and analysis system.

5.3.2 Liquid Scintillation Counter.

6.0 SAMPLE COLLECTION & PRESERVATION

Samples are collected without preservation in plastic bags or bottles.

6.1 Aqueous samples should be collected in plastic containers and preserved with concentrated nitric acid to a pH <2.

NOTE: Certain high alkaline samples such as saltstone solution are not preserved with acid. These samples are run "as received."

6.2 If the sample is received with a pH greater than 2, the analyst should preserve the sample, mix and allow to sit overnight before proceeding (at least 16 hours).

7.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

7.1 Refer to "Counting Room Instrumentation Maintenance and Performance Checks," GL-EPI-E-I-010, for instructions concerning micro-Vax 3100 instrument maintenance.

8.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL SAMPLES

8.1 All standards used in this method must be traceable to a national acceptable source such as NIST or EPA.

8.2 Refer to GL-EPI-E-M-001 for the accepted procedure for labeling and tracking the standards.

8.3 A "blank" will be analyzed with each batch. The blank shall consist of an aliquot of DI water for both the soil and water analyses.

8.4 A duplicate and spiked sample should be made on the same sample in a sample batch.

8.5 A laboratory control sample should be made by spiking deionized water with a known quantity of analyte isotope.

9.0 OPERATING PROCEDURE

9.1 Sample Preparation Techniques

9.1.1 For solid matrices, refer to GL-EPI-E-A-021, "Soil Sample Preparation for the Determination of Radionuclides." Weigh an appropriate aliquot into a beaker. Record the weight. Add 25 mL of 8 M HNO_3, 0.5 mL of nickel carrier and 1 mL of iron carrier if the sample is suspected to be low in iron. Spike the appropriate samples.

9.1.2 Allow the sample to leach by covering with a watch glass and gently heat on a hot plate for 1 to 2 hours with occasional stirring. Transfer the slurry to a centrifuge tube. Centrifuge and decant the leachate into a clean beaker.

9.1.3 Wash the sample with DI water and centrifuge. Transfer the wash to the leachate beaker. At this point, the sample may be treated as a liquid. Continue with Step 9.1.5.

9.1.4 For aqueous matrices, aliquot a sample into a glass beaker, add 1mL of iron carrier, 0.5 mL of nickel carrier, and 3 mL of concentrated nitric acid. Spike the appropriate samples.

9.1.5 Add concentrated ammonium hydroxide dropwise until the iron hydroxide precipitate begins to form, then add 2 mL in excess. Heat the sample for 5 to 10 minutes on low heat.

9.1.6 Transfer sample to a centrifuge tube. Centrifuge and save the supernate in a clean glass beaker. Wash the precipitate with DI water, centrifuge, and combine with supernate.

9.1.7 Add 5 mL 1.0 M ammonium citrate (at this point the sample should be very basic in order for the nickel-DMG complex to form).

9.1.8 Add 5 mL 0.125 M DMG (a red nickel precipitate should form). Cover the beaker with a watch glass while the precipitation occurs.

9.1.9 Preweigh a Tuffryn filter and record weight on que sheet. Filter the nickel-DMG precipitate through the Tuffryn filter. Use 80% ethanol to effect the transfer.

9.1.10 Completely dry the filter in an oven at approximately 110 degrees Celsius for about 30 minutes, cool in a dessicator, weigh and record the weight on the que sheet. If NI-59 analysis is desired, proceed with Step 9.1.16; otherwise, proceed with Set 9.1.11.

9.1.11 Transfer the filter to a small beaker and add 3 mL of DI water and 2 mL of conc. nitric acid. Swirl and let soak to dissolve the precipitate.

9.1.12 Remove the filter from the beaker, rinsing both sides. Transfer the solution to a labeled scintillation vial.

9.1.13 Place the scintillation vial on a hot plate and evaporate the sample to dryness. Add 4-5 drops of hydrogen peroxide (30%) and heat the sample to dryness to destroy DMG and any other organics. Repeat this step as necessary to destroy organic material. The final product should be dark green in color.

9.1.14 Heat the sample to dryness and the formation of a black nickel oxide. Remove the sample from the hot plate and add 10 mL of 0.7 M HC1 to the vial. Swirl to dissolve the nickel oxide. Cool and add 10 mL of Ready GEL cocktail (or equivalent). Cap and shake well.

9.1.15 Count the samples on the liquid scintillation counter in accordance with GL-EPI-E-I-004, "Beckman LS6000 Series Liquid Scintillation System Operation."

9.1.16 Cover the filter with a mylar tape and fasten the filter to the outside bottom of a flat-bottom planchet. Count the sample on an x-ray detector for the length of time necessary to meet the client specified MDA.

NOTE: Samples need to be counted using the same tape as the one that was used during the calibration.

9.2 Instrument Calibration

9.2.1 Gamma spectroscopy

9.2.1.1 The gamma spectroscopy system is calibrated in accordance with GL-EPI-E-I-001, "Micro VAX 3100 Gamma Spectroscopy System Operation."

9.2.2 Liquid scintillation

9.2.2.1 Establishing a quench curve

9.2.2.1.1 Prepare a set of standards consisting of 8 to 12 standards using the same matrix and cocktail as the samples to be measured. Add approximately 10,000 DPM to each standard. Cap and shake.

NOTE: Do not add any quenching agent at the point.

9.2.2.1.2 Allow the standards to dark adapt for a minimum of 1 hour.

9.2.2.1.3 Using an available counting program, measure the observed CPM of each of the standards to verify accurate pipetting. All standards should agree within +/- 5% of the mean. Discard any standards that do not meet this criteria.

9.2.2.1.4 Add 0 to 220 ul of carbon tetrachloride in 20 ul increments to all but the first standard.

9.2.2.1.5 Recount the standards for a period of 1 minute to determine if the range of H#s covers the desired range of sample quench. If the range of the standards is not large enough, create more standards, adjusting the amount of quenching agent appropriately. Record the amount of quenching agent used for each standard on the standard preparation sheet. (Appendix 2).

9.2.2.1.6 Count the standards for a sufficient length of time to accumulate at least 10,000 counts in the highest quenched standard. Plot efficiency vs. H#.

9.2.2.2 Calibration verification

9.2.2.2.1 Count the standards against the newly established curves. The acceptance criteria being 100% +/- 10% accuracy measured against a known value.

9.3 Instrument Performance Requirements

9.3.1 The gamma spectroscopy system operation shall be verified in accordance with GL-EPI-E-I-001, "Micro VAX 3100 Gamma Spectroscopy System Operation."

9.3.2 The liquid scintillation counting system operation shall be verified in accordance with GL-EPI-E-I-004, "Beckman LS6000 Series Liquid Scintillation System Operation."

10.0 CALCULATIONS AND DATA REDUCTION METHODS

10.1 Calculate the activity in pCi/unit by the following equations:

$$\text{Activity} = \frac{\text{CPMs} - \text{CPMb}}{2.22 * E * Ab * R * g}$$

$$\text{Uncertainty} = \frac{1.96 * \sqrt{\frac{CPM_b}{C_b} + \frac{CPM_s}{C_s}}}{2.22 * E * g * Ab * R}$$

The method minimum detectable activity is calculated by the following equation.

$$MDA = \frac{2.71 + 4.66 * \sqrt{B_{cpm} * Cs}}{2.22 * E * g * R * Ab * Cs}$$

Where:

CPMs = Sample gross count rate per minute
CPMb = Background count rate per minute
C_b = Count time of the background in minutes
C_s = Count time of the sample in minutes

E = counting efficiency.
g = weight or volume of sample aliquot.
2.22 = conversion factor from dpm/pCi
R = Chemical recovery
Ab = isotopic abundance

10.2 Chemical recovery is calculated by:

$$R\ (\%\text{Recovery}) = \frac{Wf}{Wc} * 100\%$$

Where:

Wf = final weight of the nickel DMG precipitate.
Wc = calibrated weight of the nickel DMG precipitate.

11.0 DATA RECORDING , REVIEW AND REPORTING

11.1 Data Recording

11.1.1 The gamma spectroscopy data is corrected for chemical recovery and used to report the Ni-59 activity in pCi/g or pCi/l.

11.1.2 The liquid scintillation data is corrected for chemical recovery and used to report the Ni-63 activity in pCi/g or pCi/l.

11.1.3 The completed data is entered into LIMS and the data forwarded to the Group Leader for review.

11.2 Data Review

11.2.1 Refer to GL-EPI-E-D003, "Data Review and Validation Procedures," for instructions concerning the data review process.

11.3 Data Reporting

11.3.1 The analyst will take the applicable spreadsheet and enter the data into the LIMS. The following information should be included: Analyst initials, Run date and time of the sample, results, accuracy and the nominal concentration of spike and LCS.

12.0 QUALITY CONTROL REQUIREMENTS

12.1 Analyst and Method Verification Requirements

12.1.2 Refer to GL-EPI-E-D-002 for information concerning analyst and method verification.

12.2 Method Specific Quality Control Requirements

12.2.1 A matrix spike (ms) should be run with every batch of samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows.

$$Rec = \frac{Spike(pCi/unit) - Sample(pCi/unit)}{Nominal\ Concentration(pCi/unit)} * 100$$

12.2.2 A sample duplicate should be run with every batch of samples. The relative percent difference (RPD) between the sample and the sample duplicate should be less than or equal to 20% if both results are greater than 5X MDA. If either result is between 5X MDA and MDA, the limit is 100%. If either result is less than MDA then limits are not applicable. The RPD is calculated as follows.

$$\%RPD = \frac{High\ Activity(pCi/unit) - Low\ Activity(pCi/unit)}{Average(pCi/unit)} * 100$$

12.2.3 A method blank should accompany each batch of samples. The reported value should be less than or equal to requested MDA. If the value of the blank activity is not below the requested MDA then an adequate explanation should be given.

12.2.4 A Laboratory Control Sample (LCS) should be run with each batch of samples. The recovery of the LCS should fall between 75-125%. The recovery is calculated as follows:

$$\%Rec = \frac{Observed\ pCi/l}{Known\ pCi/l} * 100$$

12.3 Actions Required if the Quality Control Requirements Are Not Met

12.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the Group Leader and initiate a non conformance report as outlined in SOP GL-QS-E-004, "Nonconformance Identification Control, Documentation, Reporting and Dispositioning."

13.0 RECORDS MANAGEMENT AND DOCUMENT CONTROL

13.1 Raw data and associated documentation will be stored in binder books or in files and kept on hand for any subsequent reviews necessary. The records should be kept on hand for a length of time not shorter than 1 year.

13.2 Data generated by the micro-Vax 3100 will be backed up during routine software backup.

14.0 LABORATORY WASTE HANDLING AND DISPOSAL

Radioactive material is handled and disposed of as outlined in GL-EPI-E-S005, "Radioactive Waste Handling Procedure".

15.0 REFERENCES

15.1 USDOE, RESL/ID, Analytical Chemistry Branch, "Nickel-63 in Water by Scintillation", Revision 11/18.

15.2 Babcock & Wilcox, "Determination of Ni-59 and Ni-63 in Water and Soil", 1985.

Ni-59 and 63 Que Sheet

Batch #: 105049 **Analyst:** William Bennett **Comments:** RAD

Ni-59 Spike Code:_____ Vol:_____ Witness:_____ Prep Date:_____
Ni-63 Spike Code:_____ Vol:_____ Initials :_____
Ni-59/63 LCS Code(s):_____/_____ Vol(s):_____/_____ Minimum Duedate: *8/8/97*

Sample ID	Client Description		Min RDL	Client	Tests	Sample Date/Time	Bkr #	Aliquot (mL or g)	LCS Rack #	Initial Weight (g)	Final Weight (g)	Net Weight (mg)
9707363-02	SSQ297		3+03 PCL	WSRC	63 63	7/8/97						
QC443713	BLANK	BLANK	10 pCi/L	QC	63 63	7/17/97						
QC443714	9707363-02	DUP	10 pCi/L	QC	63 63	7/8/97						
QC443715	9707363-02	MS	10 pCi/L	QC	63 63	7/8/97						
QC443716	LCS	LCS	10 pCi/L	QC	63 63	7/17/97						

APPENDIX 1

Instruments Used (circle as appropriate):
LS6000 (Red) 7065155
LS6500 (Blue) 7067083
LS6500 (Green) 7067404
X-ray1:12922782
X-ray2:1954119

Data Reviewed By:_____

APPENDIX 2

General Engineering Laboratories
Calibration Source Preparation Sheet

Applicable SOP Number _____ Isotope _____

Date Standards Prepared _____ Cocktail Type Used _____

Standard ID _____ Matrix of Vial/Planchett _____

Amount Used (g or ml) _____

Standard Activity (DPM/g or mL) _____ Type of Scintillation Vial _____

Reference Date _____ Pipette ID Used _____

Expiration Date _____ Balance ID Used _____

Residue/Carrier Agent _____ Quenching Agent _____

Sample number	Standard Number	Quenching Vol (uL)/ Residue Volume(mL)	Initial Wt. (g)	Final Wt. (g)	Net Wt. (mg)

Prepared By: _____ Date _____

Reviewed By: _____ Cate _____

Rev 1 RLM 9/10/97

STANDARD OPERATING PROCEDURE

FOR

TOTAL URANIUM IN ENVIRONMENTAL AND BIOASSAY SAMPLES

BY

KINETIC PHOSPHORESCENCE

(GL-EPI-E-A-023-Revision 5)

PROPRIETARY INFORMATION

HARD COPY ORIGINAL REPOSITORY:

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Process Owner	10/4/99 (Sign and Date)	Tim P. Winters	(Print Name)
Technical Review	10/4/99 (Sign and Date)	Michael Schultz	(Print Name)
Quality Review	10/4/99 (Sign and Date)	Robert L. Pillano	(Print Name)
Approval and Authorization	10/4/99 (Sign and Date)	James B. Westmoreland	(Print Name)

GENERAL ENGINEERING LABORATORIES, INC
P.O. Box 30712, Charleston, SC 29417

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TABLE OF CONTENTS

SECTION Page

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1.0 TOTAL URANIUM IN ENVIRONMENTAL AND BIOASSAY SAMPLES BY KINETIC PHOSPHORESCENCE

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

This standard operating procedure provides the necessary instructions to conduct the analysis for total uranium in environmental and bioassay samples. Specific guidelines are provided for sample preparation for urine, soil, water and filters. Consult the Senior Analyst for guidance on preparation of other matrices.

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit and Practical Quantitation Limit: This method is capable of achieving sub-ppm detection limits.

3.2 Method Precision: Typical Relative Percent Difference (RPD) between sample duplicates is less than 20%.

3.3 Method Bias (Accuracy): Typical recoveries are between 90% and 110%.

4.0 DEFINITIONS

Not applicable

5.0 METHOD VARIATIONS

Variations may be necessary to handle unusual sample types.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. General Engineering Laboratories, Inc. (GEL) maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the library and in the laboratory, respectively. Individual sample MSDS forms provided by the clients are kept in Login.

6.3 Gloves are required when handling the chemicals in this procedure.

6.4 Instructions on the handling of radioactive samples is outlined in "Handling of Radioactive Samples" (GL-EPI-M-001).

6.5 Refer to "Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-S-011) for instructions on how materials are properly disposed.

6.6 When handling biological samples protect the hands and forearms by wearing gloves and a laboratory coat to avoid contact of the biological material with the skin. Protect the eyes by wearing safety glasses and if desired a splash shield.

6.7 If cutting of meats or other tissue with cutlery is required, the analyst will wear a cutting glove of mesh steel over disposable gloves to avoid cuts that could infect the analyst with pathogens.

6.8 Any procedure that volatilizes biological substances such as drying or ashing, must be conducted in a hood or other suitable containment device.

6.9 Decontamination of work surfaces exposed to biological samples is performed by wiping the work area with a diluted (1:10) bleach solution and water as soon as possible following analytical operations.

6.10 Exterior protective clothing shall be removed prior to exiting the bioassay sample preparation area in order to prevent the inadvertent spread of biohazards to the rest of the laboratory.

6.11 Hands will be washed with an antibacterial soap directly after handling biological samples.

7.0 INTERFERENCES/LIMITATIONS

Halides such as chlorides, are a source of interference in that they allow the complexed solution to decay by physical vibration and not through phosphorescence. Undissolved particles in the sample, such as organics, can also give this interference. Short phosphorescence lifetimes and non-linear graphs (<.99) of the complexed solution are an indication of interferences.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Apparatus and equipment:

8.1.1 Hot Plate

8.1.2 Aluminum heating block to accept scintillation vials (optional)

8.1.3 Glass scintillation vials, 20 mL capacity

8.1.4 Muffle furnace

8.1.5 Plastic or quartz cells

8.1.6 Calibrated pipettes

8.1.7 Acid and uraplex dispensers

8.2 Reagents, chemicals, and standards: All chemicals should be of reagent grade or equivalent whenever they are commercially available.

8.2.1 Nitric acid, concentrated (16.0 M)

8.2.2 Stilbene 420 (lasing compound)

8.2.3 NIST traceable standard natural uranium

8.2.4 Uraplex (proprietary complexing solution)

8.2.5 Nitric acid (1 M). Dilute 12.5 mL of concentrated nitric acid to 200 mL of deionized water

8.2.6 Hydrogen peroxide 30%

8.2.7 Hydrofluoric acid 48%

8.3 Instrumentation:

8.3.1 Chemcheck Kinetic Laser Phosphorimeter system.

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 Water samples should be collected in plastic bottles and preserved with concentrated nitric acid to pH 2.

9.2 If the sample is received with pH greater than 2, the analyst will add nitric acid to acidify the sample to pH 2, mix and allow to sit overnight before proceeding.

9.3 Urine samples will be refrigerated at 4° 2°C until analysis.

9.4 Soil samples may be collected in plastic or glass without preservation.

10.0 SAMPLE PREPARATION

Sample Preparation Techniques: Water

10.1 Add an appropriate aliquot (2-10 mL) of sample to the glass scintillation vial.

NOTE: Samples must be homogenized, because uranium concentrates in solids.

10.2 Add 2-3 mL concentrated nitric acid with swirling and approximately 0.5 mL of 30% hydrogen peroxide.

10.3 Place the scintillation vials on hot plate and heat to near boiling and allow the samples to go to dryness.

10.4 Repeat step (10.2) increase heat and take samples to dryness. Repeat addition of nitric acid and hydrogen peroxide until the residue is pale yellow to white.

NOTE: Allow the samples to cool partially before adding the hydrogen peroxide to avoid spattering hot chemicals.

10.5 Allow the vials to cool, dissolve the residue in 1 M nitric acid and dilute to desired volume with a calibrated pipette. Swirl the contents of the vial to mix.

10.6 Analyze resulting solutions with the KPA as described in "Operation of the Chemcheck Kinetic Laser Phosphorimeter" (GL-EPI-I-011).

Sample Preparation Techniques: Soil.

10.8 If not previously done, perform digestion in accordance with SOP GL-EPI-A-015.

10.9 Treat the digested soil as a liquid sample, proceed with step 10.2.

Sample Preparation Techniques: Urine

10.10 Arrange an appropriate number of liquid scintillation vials in an aluminum brick. Arrange sample bottles and que sheet in the order of analysis by sample number. Obtain independent check by another analyst, technician or Group Leader that brick number que sheet and sample numbers are correct (witness shall initial que

sheet next to brick number). Next, shake each sample to ensure the sample is homogenized, then add 5 mL of sample to the liquid scintillation vials.

10.11 Add 5 mL of concentrated nitric acid to each vial. Then place the aluminum brick on the hotplate. Heat the samples at setting #3 to complete dryness. Cool.

10.12 Place the aluminum brick into the muffle furnace at 500°C for 30 minutes. Take samples out of the muffle furnace and allow to cool.

10.13 Place the aluminum brick with samples back on the hotplate, and add 5 mL of concentrated nitric acid to each sample. Then go back and add ~ 4-5 drops of 30% hydrogen peroxide dropwise to each sample. Take the samples to complete dryness. Cool.

NOTE: The hydrogen peroxide should be added to each sample, one drop at a time to avoid the splattering of the sample due to the reaction of the nitric acid and the hydrogen peroxide.

10.14 Repeat steps 10.12-10.13 until a pale yellow to pure white residue is obtained.

10.15 Add 10 mL of 1 M nitric acid to each sample and swirl.

10.16 Analyze resulting solutions with the KPA as described in "Operation of the Chemcheck Kinetic Laser Phosphorimeter" (GL-EPI-I-011).

Sample Preparation Techniques: Filters

10.17 Take each sample filter and carefully place in glass scintillation vial.

10.18 Place vials in muffle furnace and ramp temperature with the following sequence:

30 minutes	150° C
30 minutes	300° C
60 minutes	500° C

Allow to cool.

10.19 Add 5 ml concentrated nitric and evaporate to dryness at medium heat. Allow to cool.

10.20 Add 2 ml dropwise 30% H_2O_2 taking great care to avoid splattering. Evaporate to complete dryness and allow to cool.

10.21 Repeat Steps 10.19 and 10.20 until oxidation is complete (i.e., little or no whitish colored residue should remain).

10.22 Dissolve residue in 1 M nitric acid and dilute to desired volume with a calibration pipette. Swirl the contents of the vial to mix.

10.23 Analyze resulting solutions with the KPA as described in SOP GL-EPI-I-011.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

Refer to "Preparation of Radioactive Standards" (GL-EPI-M-001).

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

Refer to "The Operation of the Chemchek Kinetic Laser Phosphorimeter" (GL-EPI-I-011) for the calibration of the Chemcheck Laser Phosphorimeter.

13.0 ANALYSIS AND INSTRUMENT OPERATION

Refer to "The Operation of the Chemchek Kinetic Laser Phosphorimeter" (GL-EPI-I-011).

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

Refer to "Counting Room Instrumentation Maintenance and Performance Checks" (GL-EPI-I-010).

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 The Chemcheck Laser Phosphorimeter system analyzes the sample concentration as compared to a linear least squared fit of the standard curve calibration.

15.2 Record the information required on the que sheet.

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification Requirements

16.1.1 Refer to "Analyst and Analytical Methods Validation Procedures" (GL-EPI-D-003) for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank should accompany each batch of samples. The reported value of the blank, should be less than or equal to the contract required detection limit (CRDL).

16.2.2 A matrix spike (MS) should be run with every batch of samples. The recovery of the MS should be between 75-125%. The MS recovery is calculated as follows:

$$\text{MS Recovery (\%)} = \frac{\text{Spike } (\mu g/\text{unit}) - \text{Sample } (\mu g/\text{unit})}{\text{Spike Nominal Concentration } (\mu g/\text{unit})} * 100\%$$

where:

Spike = Results of matrix spike
Sample = Results of sample without added spike
Spike Nominal Concentration (SNC) = Concentration of standard in the spike calculated as follows:

$$\text{SNC} = \frac{\text{Standard } \mu g * \text{ml of Spike Added}}{222 * \text{Volume in Grams}}$$

16.2.3 A duplicate sample should be run with every batch. The relative percent difference (RPD) between the actual sample and the QC duplicate should be less than or equal to 20% if both the sample and the QC duplicate results are greater than 5 times MDA or 100% if they are both less than 5 times MDA.

The RPD should be calculated as follows:

$$\text{RPD (\%)} = \frac{\text{ABS}(DUP_1 - DUP_2)}{(DUP_1 + DUP_2)/2} * 100\%$$

16.2.4 A Laboratory Control Sample (LCS) should be run with every batch. The recovery of the LCS should fall between 75-125%. The LCS recovery is calculated as follows:

$$LCS_{recovery} = \frac{\text{LCS Result } (\mu g/unit)}{\text{Nominal Concentration of LCS } (\mu g/unit)} * 100\%$$

16.2.5 Actions Required if the Quality Control Requirements Are Not Met.

If any of the QC criteria cannot be satisfied, the analyst should inform their group leader and initiate a Nonconformance Report as outlined in GEL SOP "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

NOTE: Some clients contractually override these limits and may be more or less restrictive.

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

17.1 The analyst turns in the raw data, review checklist, que sheet, spreadsheet, data report, NCR's (if applicable), and other appropriate information in a batch to the data review specialist.

17.2 After the review process is complete, the data is transmitted from the laboratory personnel to the reporting personnel as outlined in "Data Review and Validation Procedures" (GL-EPI-D-003).

18.0 RECORDS MANAGEMENT

18.1 The following records are retained to document the analytical process:

18.1.1 A runlog that lists the sequence the analyses that were performed

18.1.2 A maintenance log which describes the routine and non-routine maintenance

18.1.3 A batch que sheet and spreadsheet which contains applicable dates, times, detectors used, etc.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

Radioactive samples and material shall be handled and disposed of as outlined in "Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-S-011).

20.0 REFERENCES

20.1 Chemchek Instruments, Inc.: KPA-11 Operation and Service Manual20.2 ASTM D-5174

1.0 The Determination of Radium-223 and Radium-224 Isotopes in Water

Process Owner(s):	Signature:	Robert D. Timm
	Signature:	Wilbur B. Sigmon, III
Technical Review by:	Signature:	James B. Westmoreland
Quality Review by:	Signature:	Robert L. Pullano
Approved & Authorized by:	Signature:	Heyward H. Coleman

SET

2.0 Method Objective and Applicability

2.1) This standard operating procedure provides the necessary instructions to conduct the analysis for Radium-223 and 224 in water matrices. Radium-226 can be determined by this method if desired, however the preferred operating method for Radium-226 is EPI A-008 "The Determination of Radium-226 in Water".

3.0 Interferences

3.1) Care must be taken when mounting the barium sulfate precipitate for counting. Any excess precipitate can degrade the resolution of the alpha spectroscopy.

4.0 Safety Precautions and Hazard Warnings

4.1) Caution should be used when dealing with acids, and other any laboratory reagents. Safety procedures for these are set forth in the Handbook for Good Laboratory Practices, found in the library of the laboratory.

4.2) If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the group leader prior to carrying out the rest of the procedure.

5.0 Apparatus and Materials

5.1) Ancillary Equipment

5.1.1) Centrifuge
5.1.2) 25mm filtering apparatus
5.1.3) 25mm Metricel 0.1 um filters

5.2) Reagents, Chemicals and Standards

5.2.1) Barium chloride carrier (0.75mg/ml of Ba). Prepare by dissolving 338mg reagent grade BaCl2 - 2H2O in 250ml DI water.
5.2.2) ^{133}Ba tracer. Dilute a stock solution to give about 1X105 cpm/ml in 0.2% nitric acid.
5.2.3) Lead perchlorate (10mg/ml of Pb). Dissolve 1.0g of granulated lead by boiling with 2ml of 72% perchloric acid and dilute to 100 ml.
5.2.4) Ra-226 tracer. Dilute a standard Ra-226 solution volumetrically to give a standard solution containing about 0.01 uCi/ml in 0.5% HNO_3.

5.2.5) Sodium sulfate, 40%. Dissolve 220g of anhydrous sodium sulfate in 460ml of DI water at room temperature and filter.
5.2.6) Lead sulfate wash. Dissolve 25g of potassium sulfate and 2ml of concentrated sulfuric acid in 250ml of water, filter, and add 250ml of water.
5.2.7) Alkaline diethylenetriaminepentaacetic acid, 0.1M and 0.5M. Suspend 20.0g of DTPA in 460ml of water. Dissolve 9.5g of sodium hydroxide in 25ml of water, cool and add to the DTPA suspension. When the acid has dissolved, filter the solution and adjust the pH to 10.6 with filtered 10M sodium hydroxide. Prepare the stronger solution in the same way except suspend the DTPA in only 60ml of water.
5.2.8) Sodium hydrogen sulfate, 70%. Dissolve 104g of anhydrous sodium sulfate in a mixture of 45ml of concentrated sulfuric acid and 180ml of water and filter.
5.2.9) Seeding suspension, 0.125mg/ml of Ba. Place 11.3mg of $BaCl_2$-$2H_20$, 10ml of the 70% sodium hydrogen sulfate, and 1 drop of 72% perchloric acid into a 250ml Erlenmeyer flask. Evaporate the solution carefully over a burner with continuous swirling to prevent bumbing until the barium sulfate has dissolved and a pyrosulfate fusion is obtained. Cool the flask to room temperature and dissolve the cake in a solution of 25ml each of 40% sodium sulfate and water. The resulting suspension can be used for a few weeks.
5.2.10) Concentrated H_2SO_4.
5.2.11) Concentrated HCl.
5.2.12) 1:1 acetic acid.

5.3) Instrumentation

5.3.1) Alpha spectrometer with associated data reduction capabilities.

5.3.3) Gamma spectrometer with associated data reduction capabilities.

6.0 Sample Collection & Preservation

6.1) Samples are collected without preservation in a plastic bottle.

7.0 Equipment and Instrument Maintenance

7.1) Refer to EPI SOP I-009 "Alpha Spectrometer Operating Instructions" for instructions concerning instrument maintenance.

7.2) Please refer to EPI I-001 "Gamma Spectrometer Operating Instructions" for

instructions concerning the gamma spectrometer.

8.0 Preparation of Standard Solutions and Quality Control Samples

8.1) All standards used in this method must be NIST traceable.

8.2) Dilution of standards should be noted in standard logbook in keeping with accepted procedure, EPI SOP M-001 "Preparation of Radioactive Standards". The weight of the standard to be diluted and diluting solutions should be noted in the standards logbook. The weight should be taken on a balance of at least four places and calculation of the new standard activity carried out and noted in appropriate significant figures.

8.3) A "blank" water should be used as the batch blank.

8.4) A duplicate and spiked sample should be made on the same sample in a sample batch.

9.0 Operating Procedure

9.1) Sample Preparation Techniques

9.1.1) Measure 500ml of a filtered water sample in a graduated cylinder containing 2ml concentrated HCl. Transfer to a large glass beaker. Add 1ml ^{133}Ba tracer.

9.1.2) Add 3ml 1:1H_2SO_4, 5g potassium sulfate and 2g recrystallized sodium sulfate. Stir and heat if necessary to completely dissolve salts. Cool.

9.1.3) Add 3-1ml portions of the lead perchlorate solution while swirling the solution rapidly. Allow solution to stand for 5 minutes between each addition. Allow precipitate to settle and decant. Note: If it is found to be difficult to separate this precipitate from 500ml volume, an evaporation step can be included after step 9.1.1 to reduce the volume to less than 50ml so the entire volume can be centrifuged.

9.1.4) Transfer precipitate to a glass centrifuge tube and rinse the beaker walls with two small portions of the wash solution. Centrifuge at 2000 rpm for 5 minutes and decant. Discard the supernate.

9.1.5) Rinse the centrifuge tube and precipitate with 10ml wash solution, mix,

centrifuge, and discard supernate.

9.1.6) Rinse tube with DI water to remove excess acid and sulfate ions.
Precipitation and Mounting of Barium Sulfate

9.1.7) Add 3ml of the 0.1 M alkaline DTPA, mix thoroughly and place the tube in a beaker of boiling water for 1-2 minutes or until the turbidity clears completely. If the burbidity does not clear up, add 0.5 M DTPA in 50 ul aliquots until it clears on further standing for 1 minute in the hot water bath.

9.1.8) Place the centrifuge tube in a beaker of cool water to cool to below room temperature.

9.1.9) Add 100 ul of the 0.75mg/ml barium carrier solution.

9.1.10) Add 3ml of filtered 40% sodium sulfate and 3 drops of 1:1 acetic acid while swirling the solution rapidly. The pH should be between 4 and 5.

9.1.11) As quickly as possible after the addition of acetic acid, inject 200 ul of the seeding suspension and swirl rapidly.

9.1.12) Allow centrifuge tube to stand in a beaker of cold water for 30 minutes.

9.1.13) Mount barium sulfate by filtering through a 25mm Gelman Metricel 0.1-micron polypropylene filter. Rinse throughly with 80% ethanol. Dry the precipitate for 5 minutes under a heat lamp. Record the time of filtration to calculate decay for ^{223}Ra and ^{224}Ra. Note: The filtering apparatus should be cleaned consecutively in hot alkaline DTPA, dilute acid, and water after every use to prevent contamination.

9.1.14) Place the filter in a petri dish and count the ^{133}Ba on a gamma detector and calculate the barium (radium) yield by reference to standard.

9.2) Instrument Calibration

9.2.1) Refer to EPI SOP I-009, "Alpha Spectrometer Operating Instructions" for guidance concerning instrument calibration.

9.3) Instrument Performance Requirements

9.3.1) Please refer to EPI I-009 "Alpha Spectrometer Operating Instructions" for instructions concerning the alpha spectrometer.

9.3.2) Please refer to EPI I-001 "Gamma Spectrometer Operating Instructions" for instructions concerning the gamma spectrometer.

9.3.3) Please refer to EPI I-010 "Counting Room Instrument Maintenance and Performance Checks" for instructions concerning instrument maintenance.

9.4) Analysis Procedures and Instrumental Operation

9.4.1) Mount filter on a stainless steel disc and count for approximately 500 minutes.

9.4.2) Please refer to EPI I-009 "Alpha Spectrometer Operating Instructions" for instructions concerning the alpha spectrometer.

9.4.3) Please refer to EPI I-001 "Gamma Spectrometer Operating Instructions" for instructions concerning the gamma spectrometer.

10.0 Calculations and Data Reduction Methods

10.1) Calculate Ra-223 by use of the following equation:

$$Ra-223(pCi/l)=\frac{A}{2.22*E*Y*(e^{-\lambda t_1})*V}*\frac{\lambda_{t2}}{1-(e^{-\lambda t_2})}$$

where:

A=net counts per minute
2.22=dpm/pCi
E=counting efficiency expressed as a fraction
Y=133Ba(Ra) yield expressed as a fraction
t1=decay time of 223Ra, measured from filtration until start of counting (min.)
t2=counting time min.
λ=decay constant of 223Ra($4.209*10^{-5}$ min^{-1})

10.2) Calculate Ra-224 in the same way except for the change in the decay constant

($\lambda=1.31*10^{-4}$ min^{-1}).

10.3) Calculate Ra-226 in the same manner but neglect the decay correction.

11.0 Data Recording , Review and Reporting

11.1) Data Recording

11.1.1) Record the information required on the que sheet (attachment 1).

11.2) Data Review

11.2.1) Refer to EPI SOP D-003 "Data Review and Validation Procedures" for instructions concerning the data review process.

11.3) Data Reporting.

11.3.1) The analyst will take the applicable tritium spreadsheet and enter the data into LIMS. The following information should be included: analyst's initials, date and time the sample was counted, sample results and accuracy in pCi/l.

12.0 Quality Control Requirements

12.1) Analyst and Method Verification Requirements

12.1.1) Refer to EPI SOP D-002 for information concerning analyst and method verification.

12.2) Method Specific Quality Control Requirements

12.2.1) A matrix spike(ms) should be run with every batch of samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows.

$$\text{Rec} = \frac{\text{Spike(pCi / ml)} - \text{Sample(pCi / ml)}}{\text{Nominal Concentration(pCi / ml)}}$$

12.2.2) A duplicate should be run with every batch of samples. If both the sample and duplicate values (pCi/mL) are greater than 5 times the CRDL, the allowable RPD is less than or equal to 0%. If the sample and the duplicate values (pCi/ml) is greater than or equal to the CRDL and less than 5x the CRDL, the allowable RPD is less than or equal to 100%. The RPD is not applicable if either sample or duplicate values are less than the CRDL. The RPD is calculated as follows:

$$\%RPD=\frac{\text{High Result}-\text{Low Result}}{\text{Average}}*100$$

12.2.3) The reported value should be less than or equal to the CRDL.

12.2.4) The spike recovery should fall between 75 and 125%. The recovery is calculated as follows.

$$Rec=\frac{\text{Spike}(pCi/ml)-\text{Sample}(pCi/ml)}{\text{Nominal Concentration}(pCi/ml)}$$

12.3) Actions Required if the Quality Control Requirements Are Not Met

12.3.1) If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non-conformance report as outlined in GEL SOP GL-QS-E-004 "Nonconformance Identification Control, Documentation, Reporting, and Dispositioning".

13.0 Records Management and Document Control

13.1) Raw data and associated documentation will be stored in binder books or in files and kept on hand for any subsequent reviews necessary.

13.2) Data generated by the instrument will be backed up during routine software backup.

13.3) Quality control charts are kept on the micro-Vax 3100 as outlined in EPI SOP I-008.

14.0 Laboratory Waste Handling and Disposal

14.1) Radioactive material is handled and disposed of as outlined in EPI SOP S-005 "Radioactive Waste Handling Procedures".

15.0 References

15.1) Sill, C.W. "Determination of Radium-226 in Ores, Nuclear Wastes and Environmental Samples." Nuclear Engineering 1987 pp. 239-256.

Attachment 1

Radium-223 and 224 in Water Que Sheet

Batch Comment: *Radioactive*

Batch # 34684 Prep Date:_____ Initials: _____

Spike Isotope:_____ Spike Code:_____ Vol:_____

Spike Isotope:_____ Spike Code:_____ Vol:_____

Tracer Isotope:_____ Tracer Code:_____ Vol:_____

Tracer Isotope:_____ Tracer Code:_____ Vol:_____

LCS Isotope:_____ LCS Code:_____ Vol:_____

LCS Isotope:_____ LCS Code:_____ Vol:_____

Batched to: Genie
Gibson

Sample ID	*Sample Description*	*Label #*	*Sample Mass (mls)*	*Detector # 223*
9307411-03	HSB114C			
9307411-04	HSB114D			
9307411-05	HSB115C			
9307411-06	HSB115D			
9307411-07	HSB112E			
9307411-08	HSB118A			
9307434-03	HSB116C			
9307434-04	HSB125D			
9307434-05	HSB129D			
9307434-06	HSB136C			
QC135534	blank			
QC135535	9307434-06			
QC135536	9307434-06			
QC135537	lcs			

DET #'S	*SN*
1	13271
2	13911
3	13272
4	13273
5	13258
6	13259
7	13265
8	13268
9	13266
10	13261
11	13296
12	13260
13	17940
14	13912
15	13270
16	13269
17	13274
18	13275
19	17024
20	16318
21	16320
22	13323
23	16323
24	16326

Data Review By:_____

The Determination of Fe-55 in Soil and Water

Process Owners:	Signature:	Wilbur B. Sigmon, III
Technical Review by:	Signature:	James B. Westmoreland
Quality Review by:	Signature:	Robert L. Pullano
Approved & Authorized by:	Signature:	Heyward H. Coleman

SET

2.0 Method Objective and Applicability

This standard operating procedure provides the necessary instructions to conduct the analysis of Fe-55 in soil or water.

3.0 Interferences

The presence of large quantities of elemental iron will bias the chemical recovery.

4.0 Safety Precautions and Hazard Warnings

4.1 Caution should be used when dealing with acids and any other laboratory reagents. Safety procedures for these are set forth in the Handbook for Good Laboratory Practices, found in the library of the laboratory.

4.2 Microwave digestion bombs require the special attention of the analyst involved. The analyst should insure the rupture disk is in place prior to closure of vessel. The lid of the digestion bomb should not be over tightened. A good indicator of the properly sealed vessel is that the teflon cover should just be able to be turned by rotating the teflon bolt in the center of the teflon cover.

4.3 If there is any question regarding the safety of any laboratory practice, **stop immediately** and consult the group leader prior to carrying out the rest of the procedure.

5.0 Apparatus and Materials

5.1 Ancillary Equipment

5.1.1 Glass beaker

5.1.2 Disposable transfer pipet

5.1.3 50 mL disposable centrifuge tube

5.1.4 Hot plate

5.1.5 47mm, 0.45 um cellulose nitrate filter

5.1.6 2 inch stainless steel planchet

5.1.7 0.25 mil mylar xray film

5.1.8 Ion exchange column

5.1.9 Drying oven

5.1.10 Analytical balance

5.2 Reagents, Chemicals and Standards

5.2.1 DI water.

5.2.2 Fe Carrier (~10 mg/mL)

5.2.3 Co Carrier (~5 mg/mL)

5.2.4 Cs Carrier (~5 mg/mL)

5.2.5 Mn Carrier (~5 mg/mL)

5.2.6 Concentrated HCl

5.2.7 Concentrated ammonium hydroxide

5.2.8 8N HCl

5.2.9 6N HCl:0.5 N HF

5.2.10 4N HCl:0.06 N HF

5.2.11 0.1N HCL

5.2.12 Biorad AG MP-1, 200-400 Chloride form resin (or equivalent)

5.3 Instrumentation

5.3.1 Low Energy germanium detector capable of measuring emissions at 6 keV, with a data acquisition and analysis system.

6.0 Sample Collection & Preservation

Samples are collected preserved with nitric acid in plastic or bottles.

7.0 Equipment and Instrument Maintenance

7.1 Refer to GL-EPI-E-I010 for instructions concerning micro-Vax 3100 instrument maintenance.

7.2 Refer to GL-EPI-E-A015 for instructions for operation of microwave oven maintenance.

7.3 The digestion vessel should be rinsed with type II water after sample transfer has been completed. The teflon cup may then be washed with a soft sponge and soap. The vessel can then be soaked in a low concentration nitric acid bath for 24 hours to reduce staining of cup walls.

7.4 The teflon cover should be disassembled and the rupture disk removed. The cover and bolt should then be rinsed with type II water then they may also be soaked in the nitric acid bath.

8.0 Preparation of Standard Solutions and Quality Control Samples

8.1 All standards used in this method must be traceable to a national acceptable source such as NIST or EPA.

8.2 Refer to GL-EPI-E-M001 for accepted procedure of labeling and tracking of the standards.

8.3 A "blank" will be analyzed with each batch. The blank shall consist of an aliquot of DI water for both the soil and water analyses.

8.4 A duplicate and spiked sample should be made on the same sample in a sample batch.

8.5 A laboratory control sample (LCS) should be made by spiking deionized water with a known quantity of analyte isotope.

9.0 Operating Procedure

9.1 Sample Preparation Techniques

9.1.1 If the sample is a liquid proceed with step 9.1.2. Refer to GL-EPI-E-A021, "Soil Sample Preparation for the Determination of Radionuclides". Then add 1mL of iron carrier, and proceed with the microwave digestion in accordance with GL-EPI-E-A-015, "Microwave Digestion for Soils and Sand" then proceed with step 9.4.1. Record the aliquot on que sheet (attachment 1).

9.1.2 Aliquot a sample into a glass beaker and add 1mL of iron carrier Record the aliquot on the que sheet. Prepare all required QC samples in the same manner.

9.1.3 Add concentrated ammonium hydroxide dropwise until the iron hydroxide precipitate begins to form, then add 2 mL in excess. Heat the sample for 5 minutes on low heat.

9.1.4 Proceed with step 9.4.2 of the iron separation procedure.

9.2 Instrument Calibration

9.2.1 The gamma spectroscopy system is calibrated in accordance with GL-EPI-E-I001 "Micro VAX 3100 Gamma Spectroscopy System Operation".

9.2.3 The iron carrier shall be calibrated by aliquoting five 1.0 mL aliquots of iron carrier to 20 mL of DI water in a 50 mL centrifuge tube. Perform steps 9.4.4 through 9.4.9. Calculate the average weight of the iron (III) hydroxide precipitate. Record in carrier calibration book.

9.3 Instrument Performance Requirements

9.3.1 The gamma spectroscopy system operation shall be verified in accordance with GL-EPI-E-I001 "Micro VAX 3100 Gamma Spectroscopy System Operation".

9.4 Analysis Procedures and Instrumental Operation

9.4.1 Transfer the digestate to a beaker using DI water to effect the transfer. Precipitate the $Fe(OH)_3$ by carefully adding concentrated NH_4OH

dropwise until the iron precipitate begins to form, then adding approximately 2 mL in excess. Heat the sample on low heat for 5 minutes.

9.4.2 Cool and filter the sample through a 0.45 µm cellulose nitrate filter and save the filter and precipitate for the iron analysis. Discard the filtrate as radioactive waste.

9.4.3 Transfer the filter to a labeled 50 mL centrifuge tube. Add 30 mL of concentrated HCL, 1 mL of cobalt carrier, 1 mL of cesium carrier, and 1mL of manganese carrier.

9.4.4 Prepare an ion exchange column using Bio-Rad AG MP-1 200-400 mesh, which has been soaked in concentrated HCl for 15 minutes. The resin bed should be approximately 10 cm in length. Place a small ball of glass wool on top of the resin.

9.4.5 Wash the resin with 30 mL of concentrated HCl.

9.4.6 Quantitatively transfer the dissolved sample to the ion exchange column, using 10 N HCl to effect the transfer.

NOTE: The iron fraction can be seen as the yellow band on top of the ion exchange column and the cobalt fraction is the blue band directly below the iron.

9.4.7 Wash the column with 30 mL of 8 N HCl.

9.4.8 Wash the column with 20 mL of 6 N HCl:0.5 N HF.

9.4.9 Wash the column with 20 mL of 4 N HCl:0.06 N HF. At this point the cobalt band should begin migrating down the column.

9.4.10 Wash the column with 20 mL of 0.1 NHCl to elute the iron fraction. When the blue band has disappeared, begin collecting the iron fraction in a labeled 100 mL beaker.

9.4.11 Precipitate the iron by adding concentrated ammonium hydroxide dropwise until the iron hydroxide begins to precipitate, then add 3 mL in excess.

9.4.12 Collect the precipitate on a tared 47mm, 0.45 um nitrate cellulose filter paper. Dry the paper in an oven at ~110 degrees Celsius for 15 minutes. Cool, weigh, and record the weight on the que sheet. The weight of the precipitate will be used to calculate the chemical recovery.

9.4.13 Mount the filter paper on a 2" stainless steel counting planchet, using glue stick to adhere the filter. Cover the planchet with mylar xray film and bring the sample to the counting room for analysis.

9.4.14 Count the sample in accordance with GL-EPI-E-I001 "Operation of the VAX/VMS Gamma Spectroscopy System" on the XRAY detector (low energy germanium), measuring the Fe-55 K x-rays at 5.9 keV.

10.0 Calculations and Data Reduction Methods

10.1 Calculate the activity in pCi/unit by the following equation:

$$\text{Activity} = \frac{Cs - Cb}{2.22 * E * R * V * Ab}$$

$$\text{Uncertainty} = \frac{1.96 * \sqrt{\frac{CPM_b}{C_b} + \frac{CPM_s}{C_s}}}{2.22 * E * V * Ab * R}$$

10.2 Chemical recovery is calculated by:

$$\%\,\text{Recovery} = \frac{Wf}{Wc} * 100\%$$

Where:
Wf = Final weight of the iron hydroxide precipitate.
Wc = Calibrated weight of the iron hydroxide precipitate.

10.3 Minimum detectable activity is calculated as follows:

$$MDA = \frac{2.71 + 4.66\sqrt{Cb * T}}{2.22 * E * V * Ab * R}$$

Where:
Cs = Sample gross count rate per minute
Cb = Background count rate per minute
E = Counting efficiency.
V = Weight or volume of sample aliquot.
2.22 = Conversion factor from dpm/pCi
R = Chemical recovery
Ab = Isotopic abundance
T = Sample count time in minutes

11.0 Data Recording , Review and Reporting

11.1 Data Recording

11.1.1 The gamma spectroscopy data is corrected for chemical recovery and used to report the Fe-55 activity in pCi/g or pCi/l.

11.1.2 The completed data is entered into LIMS and the data package forwarded to the group leader for review.

11.2 Data Review

11.2.1 Please refer to GL-EPI-E-D003 for specifics on data review.

11.2.2 If the samples results or qc results are not acceptable, the sample and/or applicable qc shall be re analyzed.

11.3 Data Reporting

11.3.1 The analyst will enter the run sequence in the liquid scintillation counter run log as outlined in GEL-SOP-GL-LB-E009.

11.3.2 The analyst will take the applicable spreadsheet and enter the data into the LIMS. The following information should be included: Analyst initials, run date and time of the sample, results in pCi/g, the accuracy in pCi/g and the nominal concentration of spike and LCS.

12.0 Quality Control Requirements

12.1 Analyst and Method Verification Requirements

12.1.2 Refer to GL-EPI-E-D002 for information concerning analyst and method verification.

12.2 Method Specific Quality Control Requirements

12.2.1 A matrix spike (ms) should be run with every batch of samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows.

$$\text{Rec} = \frac{\text{Spike}(\text{pCi / unit}) - \text{Sample}(\text{pCi / unit})}{\text{Nominal Concentration}(\text{pCi / unit})} * 100$$

12.2.2 A sample duplicate should be run with every batch of samples. The relative percent difference (RPD) between the sample and the sample duplicate should be less than or equal to 20%. If both results are greater than 5X MDA. If either result is between 5X MDA and MDA limit is ≤ 100. If either result is less than MDA then limits are not applicable. The RPD is calculated as follows.

$$\%\text{RPD} = \frac{\text{High Activity}(\text{pCi / unit}) - \text{Low Activity}(\text{pCi / unit})}{\text{Average}(\text{pCi / unit})} * 100$$

12.2.3 A method blank should accompany each batch of samples. The reported value should be less than or equal to requested MDA. If the value of the blank activity is not below the requested MDA then an adequate explanation should be given.

12.2.4 A Laboratory Control Sample (LCS) should be run with each batch of samples. The recovery of the LCS should fall between 75-125%. The recovery is calculated as follows:

$$\%\,\text{Rec} = \frac{\text{Observed pCi / l}}{\text{Known pCi / l}} * 100$$

12.3 Actions Required if the Quality Control Requirements Are Not Met

12.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non conformance report as

outlined in GEL-SOP GL-QS-E-004 "Nonconformance Identification Control, Documentation, Reporting and Dispositioning".

13.0 Records Management and Document Control

13.1 Raw data and associated documentation will be stored in binder books or in files and kept on hand for any subsequent reviews necessary. The records should be kept on hand for a length of time not shorter than 1 year.

13.2 Data generated by the micro-Vax 3100 will be backed up during routine software backup.

13.3 Quality control charts for spike and blank results are kept on the micro-Vax 3100.

14.0 Laboratory Waste Handling and Disposal

Radioactive material is handled and disposed of as outlined in GL-EPI-E-S005, "Radioactive Waste Handling Procedure".

15.0 References

15.1 Babcock & Wilcox, "Determination of Fe-55 in Water and Soil", 1985.

Attachment 1

Fe-55 Que Sheet

Comment: *none*

Batch # Prep Date:_____ Initials: _____

Spike Isotope:_____ Spike Code:_____ Vol:_____

LCS Isotope:_____ LCS Code:_____ Vol:_____

Instrument Used: (circle one) LS6500 LS6000 Batched to:

Sample ID	*Sample Descrip.*	*Vol (mls or g)*	*Initial Weight (g)*	*Final Weight (g)*	*Net Weight (mg)*

Data Reviewed By: _____

STANDARD OPERATING PROCEDURES

FOR

RADIUM-226 IN DRINKING WATER

BY EPA METHOD 903.1

Process Owner(s):	Patrick E. Konen, Technician III
Technical Review by:	Wilbur B. Sigmon, III, Group Leader
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	James B. Westmoreland, EPI Laboratory Manager

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR RADIUM-226 IN DRINKING WATER BY EPA METHOD 903.1

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This method covers the measurement of radium-226 in a drinking water sample and would be employed after the gross alpha or the gross radium alpha screening technique had indicated possible non-compliance with the alpha radioactivity limits set forth in the Safe Drinking Water Act, PL 93-523. 40 FR 34324. This method is specific for radium-226, and is based on the emanation and scintillation counting of radon-222, a daughter product of radium-226. The detection limit for this method assures measuring radium-226 concentrations as low as 0.1 pCi/l.

2.2 The radium-226 in the drinking water sample is concentrated and separated by coprecipitation on barium sulfate. The precipitate is dissolved in EDTA reagent, placed in a sealed bubbler and stored for ingrowth of radon-222. After ingrowth, the gas is purged into a scintillation cell. When the short-lived radon-222 daughters are in equilibrium with the parent (~4h), the scintillation cell is counted for alpha activity. The absolute measurement of radium-226 is effected by calibrating the scintillation cell system with a standard solution of this nuclide.

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit and Practical Quantitation Limit: This method is capable of achieving 0.1 pCi/L on routine samples

3.2 Method Precision: Typical Relative Percent Difference (RPD) between samples duplicates are less than 20%.

3.3 Method Bias (Accuracy): Typical recovery of the Radium-226 is between 75 and 125%.

3.4 An analyst is required to run a duplicate, spike, and a blind sample spiked with a known quantity of Ra-226 to show proficiency in the method as specified in EPI SOP "Standard Operating Training Procedure" (D-001).

4.0 DEFINITIONS

4.1 Lucas cell. See Appendix 3. An airtight instrument that is coated internally with a zinc phosphor compound. The cell functions as an emitter of photons proportional to the alpha activity in the cell.

4.2 Radon bubbler. See Appendix 2. Glassware used to purge radon from a sample and trap in the Lucas cell.

5.0 METHOD VARIATIONS

No variations are currently being used.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. Environmental Physics, Inc. (EPI) maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the library and in the laboratory, respectively. Individual sample MSDS forms provided by the clients are kept in Login.

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are:

6.3.1 Nitrile gloves for concentrated acids and bases.

6.4 Never leave gas cylinders unchained or untied, including when they are on a moving cart.

6.5 Instructions on the handling of radioactive samples is outlined in EPI SOP "Handling of Radioactive Samples" (M-001). The following general guidelines are applicable:

6.5.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling.

6.5.2 Wear a plastic apron over lab coat when working with radioactive samples.

6.5.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.5.4 Prohibit admittance to immediate work area.

6.5.5 Post signs indicating radioactive samples are in the area.

6.5.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in EPI.

6.5.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

6.6 Refer to EPI SOP "Radioactive Waste Handling Procedures" (S-005) for instructions on proper disposal of radioactive materials.

7.0 INTERFERENCES/LIMITATIONS

There are no radioactive interferences in this method.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Apparatus and equipment:

8.1.1 Electric hot plate

8.1.2 Analytical balance

8.1.3 Centrifuge

8.1.4 Misc. Glassware

8.1.5 Radon emanation apparatus (Appendix 1)

8.1.6 Radon bubbler (Appendix 2)

8.1.7 Scintillation cell (Appendix 3)

8.2 Reagents, chemicals, and standards: All chemicals should be of reagent grade or equivalent whenever they are commercially available.

8.2.1 Distilled or deionized water.

8.2.2 Ammonium hydroxide, 15N: NH_4OH (conc.), sp. gr. 0.90, 56.6%.

8.2.3 Ascarite, drying reagent: B-20 mesh.

8.2.4 Barium carrier, 16 mg/mL, standardized:

Carrier Standardization: (in triplicate)

Pipette 2.0 mL carrier solution into a centrifuge tube containing 15 mL water. Add 1 mL 18N H_2SO_4 with stirring and digest precipitate in a water bath for 10 minutes. Cool, centrifuge and decant the supernatant. Wash precipitate with 15 mL water. Transfer the precipitate to a tared

stainless steel planchet with a minimum of water. Dry under infra-red lamp, store in desiccator and weigh as $BaSO_4$.

8.2.5 EDTA reagent, basic, (0.25M): Dissolve 20g NaOH in 750 mL water, heat and slowly add 93g disodium ethylenedinitriloacetate dihdydrate, ($Na_2C_{10}H_{14}O_8N_2*2H_2O$) while stirring. After the salt is in solution, filter through coarse filter paper and dilute to 1 liter.

8.2.6 Helium gas

8.2.7 Hydrochloric acid, 12N: HCl (conc.), sp. gr. 1.19, 37.2%.

8.2.8 Magnesium Perchlorate, $Mg(ClO_4)_2$: reagent grade.

8.2.9 Sodium hydroxide, 10N: Dissolve 40g NaOH in 50 mL water and dilute to 100 mL.

8.2.10 Standard radium-226 tracer solution: preferably purchased from National Bureau of Standards, Special Publication 260, 1978, SRM 4960. Prepare stock dilution equivalent to 50 pCi radium-226 per mL.

8.2.11 Sulfuric acid, 18N: Carefully mix 1 volume 36N H_2SO_4 (conc.) with 1 volume of water.

8.2.12 Sulfuric acid, 0.1N: Mix 1 volume 18N H_2SO_4 with 179 volumes of water.

8.3 Instrumentation:

8.3.1 A scalar such as a Ludlum Model 1000 is needed to count data.

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 It is recommended that samples be preserved with HCl to pH 2 at the time of collection. A sample size of at least 1 liter should be collected for analysis.

9.2 The pH of the sample is checked by the analyst prior to analysis using a pH indicator strip.

9.3 If a sample was not correctly preserved, the analyst will preserve the sample and allow it to stand overnight prior to analyzing.

9.4 If the sample was not preserved correctly, a nonconformance report is filled out noting the problem with preservation as outlined in GEL SOP "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

10.0 SAMPLE PREPARATION

10.1 To a 1000 mL drinking water sample, add 20 mL 12N HCl and 2.0 mL barium carrier and heat to boiling.

10.2 NOTE: If there is solid matter in the sample, do not filter before starting analysis. Follow procedure steps through 9.1.5, then filter solution into a clean centrifuge tube. Add 1 mL $(NH_4)_2SO_4$ (200 mg/mL) and stir thoroughly. Add glacial (17.4N) acetic acid (CH_3COOH) until barium sulfate precipitates, then add 2 mL excess. Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate. Repeat step 10.5 and continue with radium analysis.

10.3 Cautiously and with vigorous stirring, add 20 mL 18N H_2SO_4. Digest 5 to 10 minutes and let precipitate settle overnight. Decant and discard supernate.

10.4 Slurry the precipitate and transfer to a centrifuge tube with a minimum amount of 0.1N H_2SO_4. Centrifuge and discard supernate. Wash twice with 0.1N H_2SO_4. Centrifuge and discard washes.

10.5 Add 20 mL basic EDTA reagent, heat in a water bath and stir well. Add a few drops 10N NaOH if the precipitate does not readily dissolve.

10.6 Transfer the solution to a radon bubbler (Appendix 2). Open both the upper and lower stopcocks and de-emanate the solution by slowly passing helium gas through the bubbler for about 20 minutes.

10.7 NOTE: The volume of these bubblers is usually greater than 20 mL allowing for at least a 1 cm air space between the bubbler and the stopper. In those instances where the solution volume exceeds the capacity of the bubbler, it will be necessary to continue the boiling in the water bath until the volume is reduced.

10.8 Close the two stopcocks, and record time. Store the solution for 4 to 8 days for ingrowth of radon-222 (Appendix 4). At the end of the storage period, fill the upper half of an absorption tube with magnesium perchlorate and the lower half with ascarite.

10.9 NOTE: For minimizing corrections that would be required in subsequent calculations, the voids above the bubbler must be kept very small. Capillary tubing should be used whenever possible, and the drying tube volume with the ascarite and magnesium perchlorate must be kept to a minimum. A typical system consists of a drying tube 10 cm x 1.0 cm (I.D.), with each of the drying agents occupying 4 cm and being separated by small glass wool plugs. The column can be reused several times before the chemicals need to be replaced.

10.10 Attach the tube to the radon bubbler and then attach the evacuated scintillation cell (Appendix 3) to the tube. Open the stopcock on the cell and check the assembly for leaks. Gradually open the outlet stopcock in the bubbler, and when the stopcock is fully open and no further significant bubbling takes place, close the stopcock.

10.11 Adjust the helium gas pressure so that the gas flows at slightly above atmospheric pressure.

10.12 Connect the hose to the bubbler inlet and gradually open the inlet stopcock using the bubbling as a guide. When the stopcock can be fully opened without a significant amount of bubbling, the bubbler is essentially at atmospheric pressure again.

10.13 Open the outlet stopcock very slightly and allow bubbling to proceed at a rate, determined by experience, such that 15 to 20 minutes are required to complete de-emanation.

10.14 Toward the end of the de-emanation, when the vacuum is no longer effective, gradually increase the helium gas pressure. When the system is at atmospheric pressure, shut off the helium gas, disconnect the tubing from the bubbler inlet and close the inlet and outlet stopcocks of the cell and bubbler, and record time. This is the beginning of radon-222 decay and ingrowth of radon-222 daughters.

10.15 Store the scintillation cell for at least 4 hours to ensure equilibrium between radon and radon daughters. Count the alpha scintillations from the cell in a radon counter with a light-tight enclosure that protects the photomultiplier tube. Record the counting time to correct for the decay of radon-222.

10.16 NOTE: After each analysis, flush the cell three times by evacuation and filling with helium, and store filled with helium at atmospheric pressure. This procedure removes radon from the cell and prevents the build-up of radon daughter products. Before each analysis, the scintillation cell should be evacuated, filled with helium and counted to ascertain the cell background. For drinking water samples, the cell background count time must be 100 minutes or 20 counts.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to EPI SOP "Preparation of Radioactive Standards" (M-001).

11.2 Describe the source of the calibration and internal standard materials. Describe the step-by-step preparation procedures. Include proper storage, shelf-life, and concentration.

11.3 Source materials are ordered from any of four main suppliers listed below. All four companies are called for a quote, and the most economical price for the laboratory needs is selected. All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in Section 19.0 of EPI SOP "Preparation of Radioactive Standards" (M-001).

11.3.1 National Institute of Standards and Technology (NIST), Gaithersburg, Maryland.

11.3.2 Isotope Products Laboratories, Burbank, California.

11.3.3 Amersham Corporation, Arlington Heights, Illinois.

11.3.4 North American Scientific Products and Services, North Hollywood, California.

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 Instrument Calibration. The calibration constant of each scintillation cell must be determined using a standardized radium-226 solution with a labeled cell and a specific photon counter. This is determined as follows:

12.1.1 Place 50 pCi of the radium-226 standard solution in a bubbler (50 pCi of radium-226 will produce about 6 pCi radon-222 in 18 hours). Attach the bubbler to the radon assembly. (Appendix 1.)

12.1.2 With the scintillation cell disconnected, bubble helium gas through the solution for 20 minutes to remove all radon-222.

12.1.3 Close both stopcocks on the bubbler to establish zero time for ingrowth of radon-222. Set aside for approximately 18 hours.

12.1.4 Evacuate the scintillation cell and attach to the column and bubbler.

12.1.5 Proceed with steps 10.11-10.17, Radon Emanation Technique.

12.1.6 The calibration constant is determined from the radium-226 activity in the bubbler and the ingrowth time of radon-222.

12.1.7 The calibration constant includes the de-emanation efficiency of the system, the counting efficiency of the cell, and the alpha activity contributed by polonium-218 and polonium-214, which will be in equilibrium with radon-222 when the sample is counted 4 hours after the de-emanation. A 100-minute counting time will be sufficient for the standard and will eliminate the need to correct for decay of radon-222, which occurs during counting.

12.1.8 The bubbler used for the radium-226 standardization should not be used for sample analysis. It should be set aside to be retained for future calibrations. Each scintillation cell should be calibrated periodically with the radium-226 standard to ensure instrument quality control.

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 Refer to EPI SOP "Ludlum Lucas Cell Counter Operating Instructions" (I-007) for guidance concerning analysis procedures and instrument operation.

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 Refer to EPI SOP "Counting Room Instrumentation Maintenance and Performance Checks" (I-010).

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 Calculate the sample pCi/L according to the following equations:

$$\text{Result (pCi / L)} = \frac{C}{2.22 * E * V} * \frac{1}{1 - e^{-\lambda t 1}} * \frac{1}{1 - e^{-\lambda t 2}} * \frac{1}{1 - e^{-\lambda t 3}}$$

15.2 The counting uncertainty is calculated according to the following equation:

$$\text{Error (pCi / L)} = \frac{1.96\sqrt{\frac{S_{cpm}}{T_c} + \frac{B_{cpm}}{T_{cb}}}}{(2.22 * E * R * V)}$$

15.3 The method detection limit (MDA) is calculated according to the following equation:

$$\text{MDA (pCi / L)} = \frac{(2.71 + 4.66\sqrt{B_{CPM} * T_C})}{(2.22 * E * V * T_C)} * \frac{1}{1 - e^{-\lambda t 1}} * \frac{1}{1 - e^{-\lambda t 2}} * \frac{1}{1 - e^{-\lambda t 3}}$$

Where:

C = net count rate, cpm
E = calibration constant for the de-emanation system and the scintillation cell in counts per minute/disintegrations per minute of radon-222, (see 9.2),
V = liters of sample used,

t1 = the elapsed time in days between the first and second de-emanations (steps 8.6 and 8.12) and λ is the decay constant of radon-222 (0.181 d-1),
t2 = the time interval in hours between the second de-emanation and counting, and λ is the decay constant of radon-222 (0.00755 hr-1),
t3 = the counting time in minutes and λ is the decay constant of radon-222 (1.26 x 10-4 min-1)
2.22 = conversion factor from dpm/pCi.
Bcpm=Backgound cpm
Scpm= Sample cpm
Tc= Sample count time

15.4 Record the information required on the que sheet.

15.5 Transfer applicable data from the que sheet to a verified spreadsheet.

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification Requirements

16.1.1 Refer to EPI SOP "Analyst and Analytical Methods Validation Procedures" (D-003) for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank should accompany each batch of samples. The reported value of the blank, should be less than or equal to the contract required detection limit (CRDL).

16.2.2 A matrix spike (MS) should be run with every batch of samples. The recovery of the MS should be between 75-125%. The MS recovery is calculated as follows:

$$\text{MS Recovery (\%)} = \frac{\text{Spike (pCi / L)} - \text{Sample(pCi / L)}}{\text{Spike Nominal Concentration (pCi / L)}} * 100\%$$

where:

Spike= Results of matrix spike
Sample= Results of sample without added spike
Spike Nominal Concentration (SNC)= Concentration of Ra-226 in the spike, calculated as follows:

$$\text{SNC} = \frac{\text{standard dpm} * \text{ml of spike added}}{2.22 * \text{volume in Liters}}$$

16.2.3 A duplicate sample should be run with every batch. The relative percent difference (RPD) between the actual sample and the QC duplicate should be less than or equal to 20% if both the sample and the QC duplicate results are greater than 5 times MDA or 100% if they are both less than 5 times MDA. The RPD should be calculated as follows:

$$RPD\ (\%) = \frac{ABS\ (DUP_1 - DUP_2)}{(DUP_1 + DUP_2)/2} * 100\%$$

16.2.4 A Laboratory Control Sample (LCS) should be run with every batch. The recovery of the LCS should fall between 75-125%. The LCS recovery is calculated as follows:

$$^{LCS}RECOVERY = \frac{\text{LCS Result(pCi/L)}}{\text{Nominal Concentration of LCS (pCi/L)}} * 100\%$$

16.2.5 Actions Required if the Quality Control Requirements Are Not Met. If any of the QC criteria cannot be satisfied, the analyst should inform their group leader and initiate a Nonconformance Report as outlined in GEL SOP "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004)

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

17.1 The analyst turns in the raw data, review checklist, que sheet, spreadsheet, data report, NCR's (if applicable), and other appropriate information in a batch to the data review specialist.

17.2 After the review process is complete, the data is transmitted from the laboratory personnel to the reporting personnel as outlined in EPI SOP "Data Review and Validation Procedures" (D-003).

17.3 The Data Reveiw/Quality Group Leader is responsible for reporting the data.

18.0 RECORDS MANAGEMENT

18.1 The following records are retained to document the analytical process

18.1.1 A runlog that lists the sequence of the analyses that were performed.

18.1.2 A maintenance log which describes the routine and non-routine maintenance.

18.1.4 A batch que sheet and spreadsheet which contains applicable dates, times, detectors used, etc.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

19.1 Radioactive samples and material shall be handled and disposed of as outlined in EPI SOP "Radioactive Waste Handling Procedures" (S-005).

20.0 REFERENCES

20.1 Krieger, H.L., Whittaker, E.L. Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 7, Method 903.1, EPA 600 4-80-032. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH. August 1980.

20.2 Krieger, H.L. Interim Radiochemical Methodology for Drinking Water, Ra-226 in Drinking Water, EPA 600/4-75-008 (Revised). Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH. September 1975.

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APPENDIX 1

Radon Emanation Apparatus with Scintillation Cell

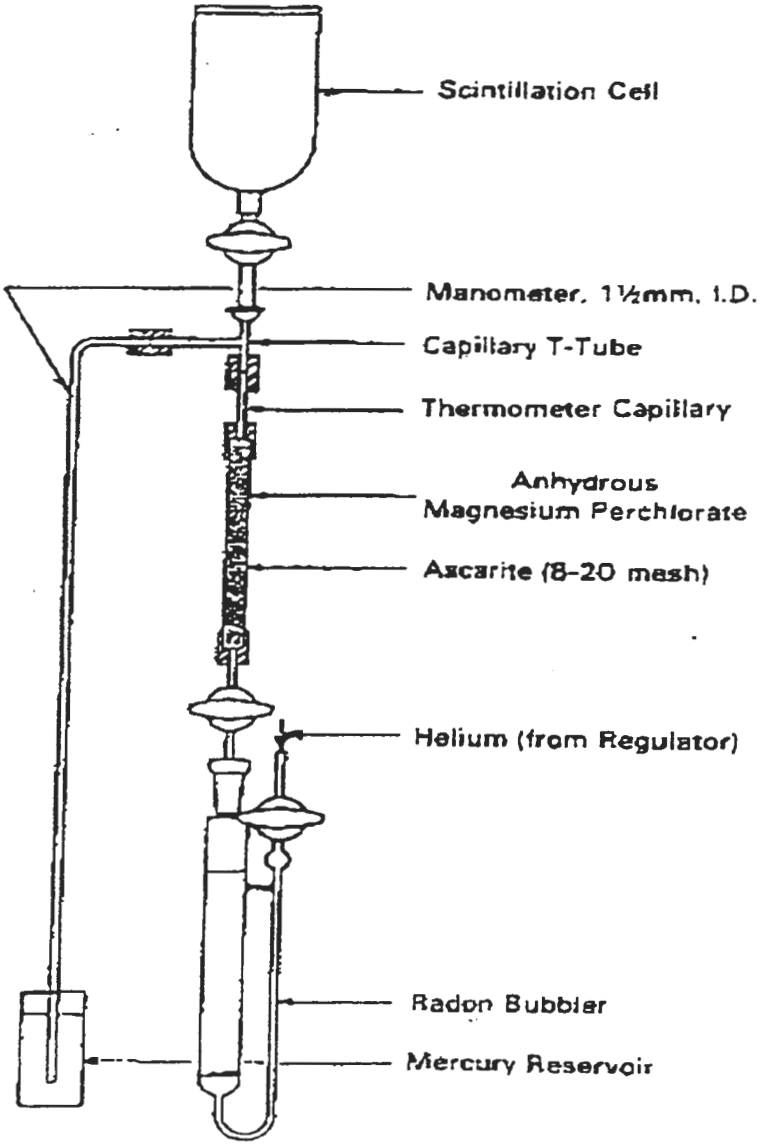

Figure 1. Radon emanation apparatus with scintillation cell

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APPENDIX 2

Radon Bubbler

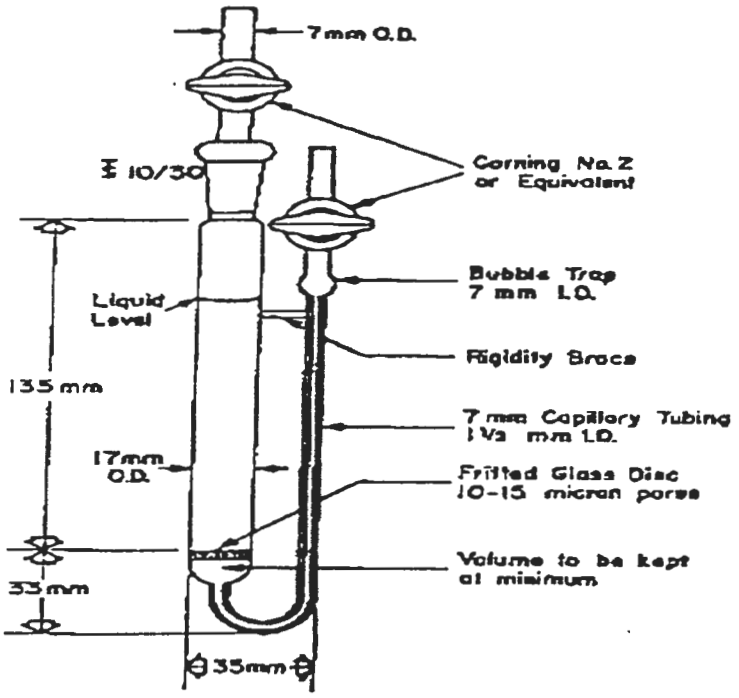

Figure 2. A typical radon bubbler

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APPENDIX 3

Radon Scintillation Cell

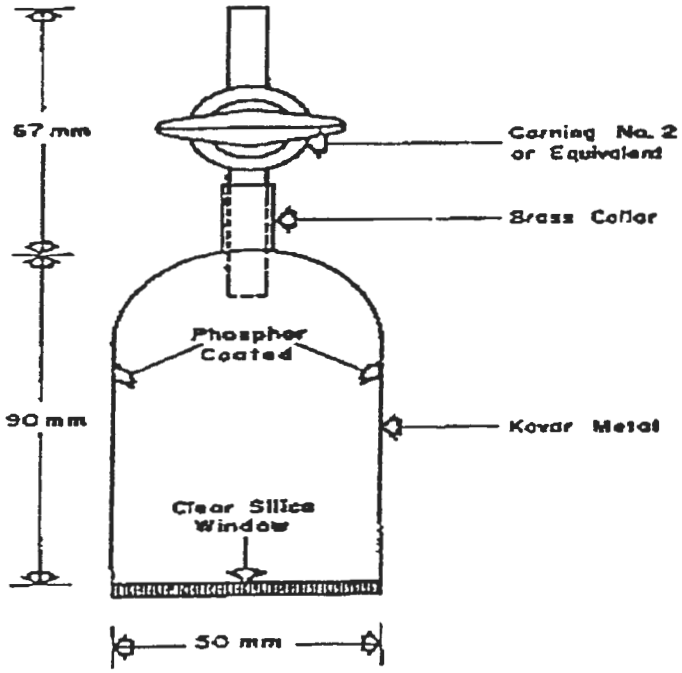

Figure 3. A typical scintillation cell for radon counting

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APPENDIX 4

Ingrowth of Radon-222 from Radium-226

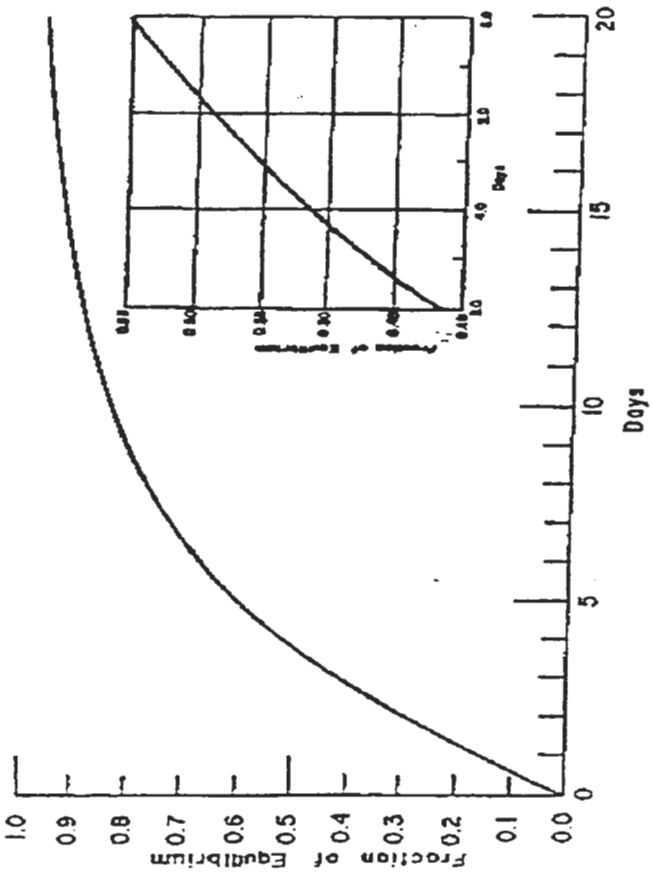

Figure 4. The growth of radon-222 from radium-226

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STANDARD OPERATING PROCEDURE

FOR

DETERMINATION OF STRONTIUM 89 AND 90 IN DRINKING WATER BY METHOD 905.0

UNCONTROLLED DOCUMENT

Process Owner(s):	Russell L. Moser, EPI Group Leader
Technical Review by:	Barry D. Stewart, Senior Technical Specialist
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	James B. Westmoreland, EPI Laboratory Manager

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TABLE OF CONTENTS

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1.0 DETERMINATION OF STRONTIUM 89 AND 90 IN DRINKING WATER BY METHOD 905.0

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This method covers the measurement of total strontium and soluble strontium-89 and strontium-90 in drinking water. Some naturally insoluble (and probably suspended) forms of strontium-89 and strontium-90 would also be measured by this method when samples of such drinking water supplies are acid-preserved before analysis.

2.2 The Drinking Water Regulations under the Safe Drinking Water Act set maximum contaminant concentrations for radionuclides in drinking water based on a 2 liter per day drinking water intake using the 168 hour data listed in Handbook 69, National Bureau of Standards. The maximum contaminant concentration for strontium-89 and strontium-90 are 80 pCi/l and 8 pCi/l, respectively, the critical organ being bone marrow. If other radionuclides are also present in the drinking water, the sum of their annual dose equivalent must not exceed 4 mrem per year. The Regulations also give a required sensitivity of measurement which is defined in terms of a detection limit. The required detection limits given for strontium-89 and strontium-90 are 10 pCi/l and 2 pCi/l, respectively. Appendix C has equations for calculating the counting time necessary to meet the required detection limit.

2.3 Stable strontium carrier is added to the drinking water sample and strontium-89 and strontium-90 are precipitated from the solution as insoluble carbonates. Interferences from calcium and some radionuclides are removed by one or more precipitations of the strontium carrier as strontium nitrate. Barium and radium are removed as the chromate. The yttrium-90 daughter of strontium-90 is removed by a hydroxide precipitation step and the separated combined strontium-89 and strontium-90 are counted for beta particle activity. The counting results, immediately ascertained, represents the total strontium activity (strontium-90 + strontium-89) plus an insignificant fraction of the yttrium-90 that has grown into the separated strontium-90. The yttrium-90 daughter grows in again and is then separated with stable yttrium carrier as hydroxide and finally precipitated as oxalate and beta counted. The strontium-90 concentration is determined by the yttrium-90 activity and the strontium-89 concentration is then determined by difference.

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for Sr-89 & Sr-90 is 1 pCi/L.

3.2 Method Precision: Typical relative percent difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value. Analyst training records are kept on hand in the human resource department.

4.0 DEFINITIONS

4.1 National Institute of Standards and Technology (NIST). For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.2 Type II water: Deionized water.

4.3 LIMS: Laboratory Information Management System. The database system used to store and report data.

5.0 METHOD VARIATIONS

5.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Senior Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. Environmental Physics, Inc. (EPI) maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the library and in the laboratory, respectively. Individual sample MSDS forms provided by the clients are kept in Login."

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are nitrile gloves for concentrated acids

and bases, and potassium ferricyanide in neat form. Work under a hood when using concentrated acids and bases.

6.4 The handling of radioactive samples is outlined in EPI SOP M-001, "Handling of Radioactive Samples." General guidelines include:

6.4.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

6.4.2 A plastic apron may be worn over the lab coat for added protection from contamination when working with radioactive samples.

6.4.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.4.4 Prohibit admittance to immediate work area.

6.4.5 Post signs indicating radioactive samples are in the area.

6.4.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in EPI.

6.4.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management."

6.5 Refer to EPI SOP S-005 "Radioactive Waste Handling Procedures" for instructions on how materials are disposed.

6.6 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the group leader prior to carrying out the rest of the procedure.

6.7 When handling biological samples protect the hands and forearms by wearing gloves and a laboratory coat to avoid contact of the biological material with the skin. Protect the eyes by wearing safety glasses and if desired a splash shield.

6.8 If cutting of meats or other tissue with cutlery is required, the analyst will wear a cutting glove of mesh steel over disposable gloves to avoid cuts which could infect the analyst with pathogens.

6.9 Any procedure which volatilizes biological substances such as drying or ashing, must be conducted in a hood or other suitable containment device.

6.10 Decontamination of work surfaces exposed to biological samples is performed by wiping the work area with a diluted (1:10) bleach solution and water as soon as possible following analytical operations.

6.11 Exterior protective clothing shall be removed prior to exiting the bioassay sample preparation area in order to prevent the inadvertent spread of biohazards to the rest of the laboratory.

6.12 Hands will be washed with an antibacterial soap directly after handling biological samples.

7.0 INTERFERENCES

7.1 Radioactive barium and radium will be carried down with radioactive strontium as carbonate. This method includes steps to separate strontium from barium and radium.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 Gas flow proportional counter with associated electronics and data reduction capabilities.

8.1.2 Centrifuge and 50 ml centrifuge tubes

8.1.3 Drying oven

8.1.4 Hot water bath

8.1.5 Electric hot plate

8.1.6 Analytical balance

8.1.7 pH meter

8.1.8 Desiccator, aluminum and/or glass

8.1.9 Stainless steel planchets, 2-inch diameter by 1/4-inch deep

8.1.10 Sintered-glass (fine) crucibles

8.1.11 Plastic ring and disc mounts

8.1.12 Mylar film

8.1.13 Teflon filter holder

8.1.14 Drying lamps

8.1.15 Glassware

8.2 Reagents, Chemicals and Standards

8.2.1 Distilled or deionized water is to be used, and all chemicals should be of "reagent-grade" or equivalent whenever they are commercially available.

8.2.2 Strontium carrier (10 mg/ml): Dissolve 24.16g Sr(NO3)2 in water and dilute to 1 liter in a volumetric flask with water. Mix thoroughly.

Standardization: (In triplicate).

8.2.3 Carefully pipet 10.0-ml portions of the strontium carrier solution into separate 50-ml centrifuge tubes. Add I ml 6N NAOH and heat in a water bath. Slowly, and with stirring, add 15 ml of 2N Na2CO3 solution.(see sodium carbonate solution below) and-continue digesting for 15 io 20 minutes. Allow to ccol and filter the SrCO3 precipitate through a tared sintered-glass (fine) crucible. Wash the precipitate and the crucible walls with three 5-ml portions of distilled water adjusted to pH 8 with 6N $NH_4$0H, and with three 5-ml portions of acetone. Dry the crucible for 30 minutes in a 105 C oven. Cool the crucibles in a desiccator and weigh.

$$\text{Strontium mg / ml} = \frac{\text{mg of SrCO3} * 0.5935}{10\text{mL}}$$

8.2.4 Yttrium carrier (10 mg/ml): Dissolve 43g $Y(NO_3)_3$*6H20 in water plus 5 ml 16N HN03 (conc.) and dilute to 1 liter in a volumetric flask with water. Mix thoroughly.

Standardization: (In triplicate).

8.2.5 Carefully pipet 10.0-ml portions of the yttrium carrier solution into separate 50-ml centrifuge tubes. Add 30 ml saturated $(NH_4)_2C_2O_4.H_2O$ to each centrifuge tube and stir. Digest in a hot water bath (near boiling) for 30 minutes. Cool in an ice bath. Filter

the precipitate onto a Whatman #42 filter paper, then ignite in a tared crucible at 800°C for 1 hour to convert the oxalate to the oxide. Cool and weigh the crucible and calculate the yttrium concentration from the following equations.

$$\frac{Y}{Y_2O_3} = \frac{2 * 88.92}{225.84} = 0.7875$$

$$\text{yttrium, mg / ml} = \frac{\text{mg } Y_2O_3 * 0.7875}{10\text{ml}}$$

8.2.6 Acetic acid, 5.BN: Mix 1 volume 17.4N CH_3COOH (glacial) with 2 volumes of water.

8.2.7 Acetone, $(CH_3)_2CO$: anhydrous.

8.2.8 Ammonium acetate buffer: Dissolve 154g $NH_4C_2H_3O_2$ in 800 ml of water, add 57 ml 17.4N CH_3COOH (glacial), adjust the mixture to pH 5.5 using CH_3COOH or NH_4OH. Dilute to 1 liter.

8.2.9 Ammonium hydroxide, 15N: NH_4OH (conc.), sp. gr. 0.90, 56.6%.

8.2.10 Ammonium hydroxide, 6N: Mix 2 volumes 15N NH_4OH (conc.) with 3 volumes of water.

8.2.11 Ammonium hydroxide, 0.1N: Mix 1 volume 15N NH_4OH (conc.) with 150 volumes of water.

8.2.12 Ammonium oxalate, saturated: Into 100 ml boiling water, dissolve 10g $(NH_4)_2C_2O_4.H_2O$ cool.

8.2.13 Barium carrier, (10 mg/ml): Dissolve 19.0g $Ba(NO_3)_2$ in water and dilute to 1 liter with water.

8.2.14 Hydrochloric acid, 6N: Mix 1 volume 12N HCl (conc.) with 1 volume of water.

8.2.15 Methyl red indicator, 0.1%: Dissolve 0.1g of methyl red in 100 ml ethanol.

8.2.16 Nitric acid, 16N: HNO_3 (conc.) sp. gr. 1.42, 70.4%.

8.2.17 Nitric acid, 6N: Mix 3 volumes 16N HNO_3 (conc.) with 5 volumes of water

8.2.18 Nitric acid, 1N: Mix 1 volume 6N HNO_3 with 5 volumes of water.

8.2.19 Phenolphthalein indicator, 1%: Dissolve lg phenolphthalein in 50 ml ethanol and add 50 ml water.

8.2.20 Sodium carbonate, 2N: Dissolve 124g $Na_2CO_3.H_20$ (or 106g Na_2CO_3) in water and dilute to 1 liter with water.

8.2.21 Sodium chromate, 0.5M: Dissolve 117g $Na_2CrO_4.4H_20$ in water and dilute to 1 liter with water.

8.2.22 Sodium hydroxide, 6N: Dissolve 240g NAOH in water and dilute to 1 liter with water.

8.2.23 Wetting agent solution: e.g. Photo-Flo, Eastman Kodak Co.

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 Samples should be preserved to approximately pH 2 with nitric acid and collected in a plastic bottle.

9.2 Before beginning an analysis the analyst should check the sample pH with a pH strip. If necessary, adjust the pH with nitric acid to a pH=1-2. If the sample was pH adjusted let the sample sit overnight before continuing the batch.

9.3 If the sample has exceeded the hold time the analyst should contact the group leader before continuing with the batch.

10.0 SAMPLE PREPARATION

10.1 Transfer 1-liter water sample aliquots to 2-liter beakers. Add 2.0 ml each of strontium and barium carrier solutions to each sample and blank beakers. Heat the samples to boiling and add 6N NAOH while stirring, to the phenolphthalein end point (red color), and add 50 ml 2N Na_2CO_3 solution. Continue heating to near boiling for 1 hour with occasional stirring. Then set the beakers aside for at least 2 hours, allowing the carbonate precipitate to settle.

10.2 Decant most of the clear supernate.and discard it. With the remainder of the-supernate and necessary water washes (adjusted to pH 8 with 6N NH_40H), quantitatively transfer the precipitate to a 50-ml centrifuge tube. Centrifuge and discard the supernate. This precipitate will contain the strontium and barium carriers.

10.3 Dissolve the precipitate by the dropwise addition of 4 ml 16N HNO_3.

10.4 Add 20 ml 16N HNO_3 to the centrifuge tube, cool in an ice bath and stir. Centrifuge and discard the supernate which will contain a significant fraction of the calcium present in the sample.

10.5 Add 20 ml 16N HN03 to the centrifuge tube, cool in an ice bath and stir. Centrifuge and discard supernate.

NOTE: If drinking water samples contain much calcium (hardness), it will be necessary to repeat step 8.5.

10.6 Dissolve the strontium and barium nitrate precipitate in 25 ml water, add 2 drops methyl red indicator, neutralize to yellow color with 6N NH_4OH, then adjust the pH back to red color by adding 5.8N CH_3COOH dropwise.

10.7 Add 5 ml ammonium acetate buffer solution, and heat in a hot water bath. Add, with stirring, 2 ml 0.5M Na_2CrO_4 and digest in the hot water bath for 15 minutes. Cool the reaction mixture and centrifuge. Transfer the supernate to a clean 50-ml centrifuge tube, and discard the barium chromate residue.

NOTE: This residue can be saved if radioactive barium, radium, or lead analysis is desired.

10.8 To the buffered chromate supernate add 2 ml 15N NH_4OH and heat in a hot water bath. Add 5 ml 2N Na_2CO_3 solution and digest for 15 minutes. Cool, centrifuge, and discard the supernate.

NOTE: In the next step, the strontium-89 and strontium-90 are separated from yttrium-90 with a yttrium carrier scavenge to start a specific ingrowth period and to get a separate radiostrontium count in the following steps.

10.9 Add a few drops 16N HN03 tO the carbonate precipitate, then add 25 ml water and 1 ml yttrium carrier. Add 1 drop of wetting agent solution (such as "Photo-Flo," an Eastman Kodak Company film processing product) and 5 ml 15N NH_4OH. Heat in a hot water bath for 15 minutes with occasional stirring. Centrifuge and transfer the supernate to a clean 50-ml centrifuge tube. Wash the yttrium hydroxide precipitate with 5 ml water, centrifuge and add this wash to the supernate. Note the time of this yttrium hydroxide precipitation which marks the beginning of the yttrium-90 ingrowth period. From this point on it is important to proceed without delay to the final separation and count of the strontium-89 and strontium-90 activity to minimize ingrowth of yttrium-90.

NOTE: Concentrated NH_4OH sometimes contains CO_2 in solution which will cause precipitation of some of the strontium carrier in this step. If low carrier recoveries are obtained in step 8.11, then for subsequent strontium analyses, anhydrous NH_3 gas may be substituted for concentrated NH_4OH in step 10.9 by bubbling NH_3 gas in the sample solution until the phenolphthalein end point is reached, and then 5 minutes more. The same precaution might be taken in step 10.14 to prevent carrydown of the strontium-90 as the carbonate precipitate in that step.

10.10 Add 5 ml 2N Na_2CO_3 to the supernate from step 8.9, heat in a hot water bath for about 10 minutes, centrifuge and discard the supernate.

10.11 Slurry the strontium carbonate precipitate with a few ml water and transfer quantitatively to a tared glass fiber filter. Wash the precipitate with three 10-ml portions of water adjusted to pH 8 with NH_4OH, then with three 10-ml portions of acetone. During filtration and washes of the strontium carbonate, minimize the time of air flow through the filter to avoid collection of radon daughters. Dry the filter at 105°C for 10 minutes, then weigh, mount and count (within 2 hours). This count gives the total of strontium-89 and strontium-90 activities, plus the ingrown yttrium-90. Note the time of this count as it must be corrected for yttrium-90 ingrowth (time between steps 10.9 and 10.11).

NOTE A: An alternative to step 8.11 involves the collection and counting of the strontium carbonate precipitate on a tared stainless steel planchet. For this, the approach is as follows:

10.11.1 Slurry the strontium carbonate precipitate with a few ml water and transfer quantitatively to a tared stainless steel planchet. Dry under infrared lamp.

10.11.2 Cool, weigh, and beta count (within 2 hours).

NOTE B: The calculation of the total strontium activity D, in the sample at this point in time can be made as follows:

$$D = \frac{C}{2.22 * EVR}$$

where:

C = net count rate, cpm
E = counter efficiency, for strontium-90
V = liters of sample used

R = fractional chemical yield, and
2.22 = conversion factor from dpm/pCi.

Strontium-90 (By Yttrium-90)

10.12 After counting the strontium carbonate for strontium-89 and strontium-90 activity, store the filter or the planchet for a measured period of ingrowth, then proceed with the following steps for yttrium-90 separation. A 2-week or longer ingrowth period is recommended for samples with very low strontium-90 activity. Step 10.9 was the beginning of this ingrowth period.

10.13 Undo the mylar covering from the nylon ring and disc, and transfer the filter to a small funnel which has been placed to drain into a 50 ml centrifuge tube. Dissolve the strontium precipitate by carefully wetting the filter with 5 ml of 6N HNO_3. Wet the filter with 2.0 ml yttrium carrier. Rinse the strontium and yttrium into the centrifuge tube by washing the filter with four 5-ml portions of 1N HNO_3. Remove the funnel from the centrifuge tube, discard the filter, and add 1 drop of wetting agent solution to the centrifuge tube. Swirl the tube to mix the contents thoroughly.

NOTE: In the case of the stainless steel planchet, do the following:

10.13.1 After the period for yttrium-90 ingrowth, slurry the precipitate on the planchet with 2 ml water and transfer to a centrifuge tube with the aid of a rubber policeman. To make the transfer quantitative, wash the residue from the planchet with a small amount of 1N HNO_3. Dissolve the precipitate in the tube with sufficient 1N HNO_3, and dilute with water to 10 ml.

10.13.2 Add 2.0 ml yttrium carrier and stir.

10.13.3 Boil to expel dissolved carbon dioxide, cool to room temperature.

10.14 Precipitate the yttrium as hydroxide by adding 10 ml 15N NH_4OH to the centrifuge tube, stirring and heating for 10 minutes in a hot water bath. Cool, centrifuge and decant supernate into a 100-ml beaker. Note time of last precipitation; this is the end of yttrium-90 ingrowth and the beginning of yttrium-90 decay.

10.15 Dissolve precipitate in 1 ml 1N HNO_3 and dilute with water to 10 ml.

10.16 Reprecipitate yttrium by dropwise addition of 15N NH_4OH.

10.17 Centrifuge and combine supernate with solution in the 100-ml beaker (step 10.14).

10.18 Repeat steps 10.15, and 10.16. Save the combined supernates in the beaker for strontium gravimetric yield determination, step 10.22.

NOTE: Steps 10.22 to 10.25 are a repeat of the strontium carbonate precipitation to determine chemical yield after the yttrium has been removed.

10.19 Dissolve the precipitate in 15 ml water containing 2 ml 6N HCl. Precipitate the yttrium as oxalate by adding 20 ml saturated $(NH_4)_2C_2O_4$ and heating for 30 minutes in a hot water at (near boiling). Cool in an ice bath and then filter the yttrium oxalate onto a Whatman #42 filter (4.25 cm diameter). Wash the precipitate with three 5-ml portions of water, then with three 5-ml portions of acetone. Air dry the filter for about 1 hour.

NOTE: A pH of 1.7-1.9 in the solution from which yttrium oxalate is being precipitated is necessary to get a uniform $9H_20$ hydrate precipitate. This is necessary if the analyst prefers and is going to weigh the yttrium oxalate for chemical yield. Also, the analyst may then prefer to use a tared glass fiber filter instead of a Whatman #42 paper filter. The filter plus oxalate precipitate is weighed to determine chemical yield (recovery). See note following 15.1 for calculations. If this procedure is followed, step 10.21 can be eliminated.

10.20 Mount filter on a plastic ring and disc, and count for yttrium-90 activity. Record the time of the counting for decay correction (time between 10.14 and count time).

10.21 Undo the mylar covering, and transfer the filter to a tared crucible. Ignite at 800°C for 1 hour in a muffle furnace to convert the oxalate to the oxide. Cool and weigh the crucible. Determine the yttrium recovery (see Section 15.1).

10.22 Warm the combined supernates from step 10.14, add 5 ml 2N Na_2CO_3, and digest for 10 minutes. Cool, centrifuge, and discard supernate.

10.23 Wash the $SrCO_3$ with 15 ml water and discard wash solution.

10.24 Slurry with a few ml water and transfer quantitatively to a tared stainless-steel planchet. Dry under infrared lamps.

10.25 Cool and weigh the planchet. Determine the strontium recovery section 15.0.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to EPI SOP M-001, "Preparation of Radioactive Standards."

11.2 All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in EPI SOP M-001, "Preparation of Radioactive Standards," section 19.0.

11.3 Primary standards are kept in the laboratory in a secured cabinet. Secondary, working, standards are kept at the bench area in an enclosed plastic cabinet.

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 For direction on calibration and instrument performance see GL-EPI-I-006, "LB4100 Gross Alpha/Beta Counter Operating Instructions."

12.2 Counting Efficiencies - Separate counting efficiencies should be determined for strontium-89 and strontium-90 using known amounts of the respective radioactive standards and 20.0 mg of strontium carrier, precipitated as carbonate and counted. A strontium-90 precipitate is prepared after separation of the yttrium-90 daughter by the following procedure. Add a known amount of strontium-90 standard, in the order of 1000 disintegrations per minute (dpm), and 20 mg of strontium carrier to a 50-ml centrifuge tube, add 20 ml of water and proceed as in steps 10.9 through 10.11. Then for the yttrium-90 counting efficiency, continue with steps 10.12 through 10.16.

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 For direction on calibration and instrument performance see GL-EPI-I-006, "LB4100 Gross Alpha/Beta Counter Operating Instructions."

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 For maintenance of system see GL-EPI-I-010, "Counting Room Instrumentation Maintenance and Performance Checks."

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 Determine the strontium method yield according to the following equation: (20.0 mg of strontium is equivalent to 33.7 mg $SrCO_3$)

$$Rs = \frac{\text{mg } SrCO_3 \text{ recovered}}{\text{mg Sr carrier added as carbonate}}$$

15.2 Determine the yttrium method yield according to the following equation. 20.0 mg of yttrium is equivalent to 25.4 mg Y_2O_3.

$$Ry = \frac{\text{mg } Y_2O_3 \text{ recovered}}{\text{mg Y carrier added as oxide}}$$

15.3 Determine the yttrium method yield according to the following equation if yttrium oxalate precipitation is used. 20.0 mg of yttrium is equivalent to 67.9 mg $Y_2(C_2O_4)_3$.

$$Ryo = \frac{\text{mg } Y_2(C_2O_4)_3 \text{ recovered}}{\text{mg Y carrier added as oxalate}}$$

15.4 The instrument will report sample pCi/unit according to the following equation:

$$Y\text{-}90 \text{ dpm} = \frac{Y\text{-}90_{cpm}}{abefi} = Sr - 90dpm$$

$$Sr\text{-}90 \text{ cpm} = Y\text{-}90_{dpm} * c$$

Total Sr-89/90 cpm = total cpm ($SrCO_3$)-ingrown Y-90 cpm.

Ingrown Y-90 cpm = Sr-90 cpm *e*g

$$Sr\text{-}89 \text{ cpm} = \frac{\text{Total } Sr_{cpm}}{a} - Sr90cpm - Sr90dpm * e * g$$

$$Sr\text{-}89 \text{ dpm} = \frac{Sr\text{-}89_{cpm}}{d}$$

$$\text{Sr-90 pCi / L} = \frac{\text{Sr-90}_{dpm}}{2.22 * V}$$

$$\text{Sr-89 pCi / L} = \frac{\text{Sr-89}_{dpm}}{2.22 * V}$$

Where:

c	=	strontium-90 counting efficiency
d	=	strontium-89 counting efficiency
e	=	yttrium-90 counting efficiency
f	=	yttrium-90 decay factor
g	=	yttrium-90 ingrowth factor, for unwanted yttrium- 90 in total strontium-89, strontium-90 count
h	=	strontium-89 decay factor
V	=	volume of sample analyzed, in liters
i	=	yttrium-90 ingrowth factor for strontium-90 by yttrium-90 determination, and
2.22	=	conversion factor from dpm/pCi.

15.2 Counting uncertainty is propagated according to the following equation:

$$\text{Sr-90 pCi / unit} = \frac{1.96 * D_2 * 1000}{2.22 * \text{sbefiV}}$$

$$\text{Sr-89 pCi / unit} = \frac{1.96 * 1000}{2.22 * \text{adV}} \sqrt{(D_1)^2 + \left(\frac{c+e+g}{befi}\right)^2 * (D_2)^2}$$

Where:

1.96	=	95% confidence factor
1000	=	ml/liter
2.22	=	conversion factor from disintegrations/minute to picocuries
a	=	strontium recovery factor
b	=	yttrium recovery factor
c	=	strontium-90 counting efficiency
d	=	strontium-89 counting efficiency
e	=	yttrium-90 counting efficiency
f	=	yttrium-90 decay factor
g	=	ingrowth factor for unwanted yttrium-90 in total radiostrontium count
I	=	ingrowth factor for yttrium-90 for strontium-90 determination

15.3 The minimum detectable activity (MDA) is calculated according to the following equation:

$$MDA(pCi\ /\ unit) = \frac{2.71 + 4.65 * \sqrt{B_{cpm} * T_c}}{(2.22 * E * V * R * A * decay * T_c)}$$

Where:

$$decay = \frac{1}{e^{\left(\frac{-\ln(2) T_d}{T_{1/2}}\right)}}$$

$$R = \frac{T_{cpm} - B_{cpm}}{T_{dpm} * E}$$

$$dk = \frac{T_{1/2}err}{T_{1/2}} * \left(\frac{\lambda Tr}{1 - e^{-\lambda Tr}} - \lambda\left(T_c + T_r\right) - 1\right)$$

And where:

Scpm	=	Sample counts per minute
Bcpm	=	Background counts per minute
E	=	Counting efficiency (decimal form)
V	=	Volume in liters,g,cfm, etc.
A	=	Isotopic abundance (decimal form)
Tc	=	Sample count time in minutes
Td	=	Time interval for radioactive decay
Tr	=	Elapsed real time in minutes
T1/2	=	Isotopic half life
T1/2err	=	Isotopic half life error
λ	=	Isotopic decay constant
e	=	exponential function
ln	=	natural log function

15.4 Record the following information on the que sheet: preparation date, analysts initials, spike isotope, spike code, spike volume, LCS isotope, LCS code, LCS volume, nominal concentration LCS, and nominal concentration MS. For each sample record the detector number, sample mass, sample date, and sample time.

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification

16.1.1 Refer to EPI SOP D-002, "Analyst and Analytical Methods Validation Procedures" for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank will accompany each batch of 20 or less samples. The reported value should be less than or equal to the CRDL for all target isotopes.

16.2.2 A matrix spike (MS) should be run with every batch of 20 samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\%\,\mathrm{Rec} = \frac{\mathrm{spike(pCi/g)} - \mathrm{sample(pCi/g)}}{\mathrm{spiked\,amount\,(pCi/g)}} * 100$$

16.2.3 A sample duplicate should be run with every batch of 20 or less samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$\mathrm{RPD} = \frac{\mathrm{high\,sample(pCi/g)} - \mathrm{low\,sample(pCi/g)}}{\mathrm{Average\ (pCi/g)}} * 100$$

16.2.4 A laboratory control spike (LCS) should be run with every batch of 20 samples or less. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\mathrm{LCS} = \frac{\mathrm{observed\_pCi/unit}}{\mathrm{known\_pCi/unit}} * 100$$

16.3 Actions Required if the Quality Control Requirements Are Not Met

16.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non conformance report as

outlined in GL-QS-E-004, "Nonconformance Identification Control, Documentation, Reporting and Dispositioning".

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

17.1 The analyst turns in the raw data, review checklist, que sheet, spreadsheet, data report, NCR's (if applicable), and other appropriate information in a batch to the data review specialist.

17.2 Analyst's data go through the following process of review:

17.2.1 The first level of review is the analyst review. The analyst will perform the following review procedure:

17.2.1.1 Visually check the que sheet, spreadsheet, raw data and data report to make sure the information has been transcribed correctly.

17.2.1.2 Check to see that the required detection limit (RDL) is met if required.

17.2.1.3 Complete the batch checklist.

17.2.2 The second level review is performed by the Data Review Specialist who reviews the batch checklist, checks requested and non-requested hits, and reviews the transcription.

17.2.3 The third level review is completed by the Quality Group Leader who reviews the checklist and checks requested hits.

17.3 After the review process is complete, the data is transmitted from the laboratory personnel to the reporting personnel as outlined in EPI SOP D-003, "Data Review and Validation Procedures."

17.4 The Data Review/Quality Group Leader is responsible for reporting the data.

18.0 RECORDS MANAGEMENT

18.1 Each analysis that is performed on the instrument is documented in the run log according to GEL SOP GL-LB-E-009, "Run Logs."

18.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

19.1 Radioactive samples and material shall be handled and disposed of as outlined in EPI SOP S-005, "Radioactive Waste Handling Procedures."

20.0 REFERENCES

20.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

UNCONTROLLED DOCUMENT

STANDARD OPERATING PROCEDURE

FOR

DETERMINATION OF RADIUM-228

IN DRINKING WATER

BY METHOD 904.0

Process Owner(s):	Russell L. Moser, EPI Group Leader
Technical Review by:	Barry D. Stewart, Senior Technical Specialist
Quality Review by:	Genie G. Bost, EPI Group Leader, Quality
Approved and Authorized by:	James B. Westmoreland, EPI Laboratory Manager

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TABLE OF CONTENTS

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1.0 DETERMINATION OF RADIUM-228 IN DRINKING WATER BY METHOD 904.0

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This method covers the measurement of radium-228 in drinking water and, if desired, the determination of radium-226 on the same sample. The Interim Primary Drinking Water regulations state that if the alpha screening test reveals a gross alpha activity above 5 pCi/l, a radium-226 analysis must also be performed. If the level of radium-226 is above 3 pCi/l, the sample must also be measured for radium-228.

2.2 This technique is devised so that the beta activity from actinium-228 which is produced by decay of radium-228, can be determined and related to the radium-228 that is present in the sample.

2.3 To quantify actinium-228 and thus determine radium-228, the efficiency of the beta counter for measuring the very short half-lived actinium-228 (avg. beta energy-0.404 keV) is to be calibrated with a beta source of comparable average beta energy.

2.4 The radium in the drinking water sample is collected by coprecipitation with barium and lead sulfate, and purified by reprecipitation from EDTA solution. Both radium-226 and radium-228 are collected in this manner. After a 36-hour ingrowth of actinium-228 from radium-228, the actinium-228 is carried on yttrium oxalate, purified and beta counted. If radium-226 is also desired, the activity in the supernate can be reserved for method 903.1 by coprecipitation on barium sulfate, dissolving in EDTA and storing for ingrowth in a sealed radon bubbler.

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for Ra-228 is 1 pCi/L.

3.2 Method Precision: Typical relative percent difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value.

Analyst training records are kept on hand in the Human Resource department.

4.0 DEFINITIONS

4.1 National Institute of Standards and Technology (NIST). For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.2 Type II water: Deionized water.

4.3 LIMS: Laboratory Information Management System. The database system used to store and report data.

5.0 METHOD VARIATIONS

5.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Senior Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. Environmental Physics, Inc. (EPI) maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the library and in the laboratory, respectively. Individual sample MSDS forms provided by the clients are kept in Login."

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are nitrile gloves for concentrated acids and bases, and potassium ferricyanide in neat form. Work under a hood when using concentrated acids and bases.

6.4 The handling of radioactive samples is outlined in EPI SOP "Handling of Radioactive Samples" (M-001). General guidelines include:

6.4.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

6.4.2 A plastic apron may be worn over the lab coat for added protection from contamination when working with radioactive samples.

6.4.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.4.4 Prohibit admittance to immediate work area.

6.4.5 Post signs indicating radioactive samples are in the area.

6.4.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in EPI.

6.4.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management."

6.5 Refer to EPI SOP "Radioactive Waste Handling Procedures" (S-005) for instructions on how materials are disposed.

6.6 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the group leader prior to carrying out the rest of the procedure.

7.0 INTERFERENCES

7.1 As evidenced from the results of the performance studies, the presence of strontium-90 in the water sample gives a positive bias to the radium-228 activity measured.

7.2 As in the case of method 903.0, excess barium in the drinking water sample might result in a falsely high chemical yield.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 Gas flow proportional counter with associated electronics and data reduction capabilities. (<3 cpm beta background)

8.1.2 Centrifuge and 50 mL centrifuge tubes

8.1.3 Drying oven

8.1.4 Hot water bath

8.1.5 Electric hot plate

8.1.6 Analytical balance

8.1.7 Stainless steel planchets, 2-inch diameter by 1/4-inch deep

8.1.8 Drying lamps

8.1.9 Glassware

8.1.10 Membrane filters, metricel 47 mm.

8.2 Reagents, Chemicals and Standards

8.2.1 Distilled or deionized water is to be used, and all chemicals should be of "reagent-grade" or equivalent whenever they are commercially available.

8.2.2 Acetic acid, 17.4N: glacial CH3COOH (conc.), sp. gr. 1.05, 99.8%

8.2.3 Ammonium hydroxide, 15N: NH_4OH (conc.), sp. gr. 0.90, 56.6%.

8.2.4 Ammonium oxalate, 5%: Dissolve 5g $(NH_4)_2C_2O_4*H_2O$ in water and dilute to 100 mL.

8.2.5 Ammonium sulfate, 200 mg/mL: Dissolve 20g $(NH_4)_2SO_4$ in water and dilute to 100 mL.

8.2.6 Ammonium sulfide, 2%: Dilute 10 mL $(NH_4)_2S$, (20-24%), to 100 mL with water.

8.2.7 Barium carrier, 16 mg/mL, standardized: (see Sec. 6, Method 903.0).

8.2.8 Citric acid, 1M: Dissolve 19.2g $C_6H_8O_7*H_2O$ in water and dilute to 100 mL.

8.2.9 EDTA reagent, basic (0.25M): Dissolve 20g NaOH in 750 mL water, heat and slowly add 93g disodium ethylenedinitriloacetate dehydrate, $(Na_2C_{17}H_{14}O_8N_2*2H_2O)$ while stirring. After the salt is in solution, filter through coarse filter paper and dilute to 1 liter.

8.2.10 Lead carrier, 15 mg/mL: Dissolve 2.3979 $Pb(NO_3)_2$ in water, add 0.5 mL 16N HNO_3 and dilute to 100 mL with water.

8.2.11 Lead carrier, 1.5 mg/mL: Dilute 10 mL lead carrier, (15 mg/mL), to 100 mL with water.

8.2.12 Methyl orange indicator, 0.1%: Dissolve 0.1g methyl orange indicator in 100 mL water.

8.2.13 Nitric acid, 16N: HNO_3 (conc.), sp. gr. 1.42, 70.4%.

8.2.14 Nitric acid, 6N: Mix 3 volumes 16N HNO_3 (conc.) with 5 volumes of water.

8.2.15 Nitric acid, 1N: Mix 1 volume 6N HNO_3 with 5 volumes of water.

8.2.16 Sodium hydroxide, 18N: Dissolve 72g NaOH in water and dilute to 100 mL.

8.2.17 Sodium hydroxide, 10N: Dissolve 40g NaOH in water and dilute to 100 mL.

8.2.18 Strontium carrier, 10 mg/mL: Dissolve 24.16 g $Sr(NO_3)_2$ in water and dilute to 1 liter.

8.2.19 Sulfuric acid, 18N: Cautiously mix 1 volume 36N H_2SO_4 (conc.) with 1 volume of water.

8.2.20 Yttrium carrier 18 mg/mL: Add 22.85g Y_2O_3 to an Erlenmeyer flask containing 20 mL water. Heat to boiling and continue stirring with a magnetic stirring hot plate while adding 16N HNO_3 in small amounts. Usually about 30 mL 16N HNO_3 is necessary to dissolve the Y_2O_3- Small additions of water may be required to replace that lost by evaporation. After total dissolution add 70 mL 16N HNO_3 and dilute to 1 liter with water.

8.2.21 Yttrium carrier, 9 mg/mL: Dilute 50 mL yttrium carrier, (18 mg/mL), to 100 mL with water.

8.2.22 Strontium-yttrium mixed carrier, 0.9 mg/mL Sr^{+2} 0.9 mg/mL Y^{+3}:

8.2.22.1 Solution A: Dilute 10.0 mL yttrium carrier, (18 mg/mL), to 100 mL.

8.2.22.2. Solution B: Dissolve 0.4348g $Sr(NO_3)_2$ in water and dilute to 100 mL.

8.2.23 Combine solutions A and B and label.

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 Samples should be preserved to approximately pH 2 with nitric acid and collected in a plastic bottle.

9.2 Before beginning an analysis the analyst should check the sample pH with a pH strip. If necessary, adjust the pH with nitric acid to a pH=1-2. If the sample was pH adjusted let the sample sit overnight before continuing the batch.

9.3 If the sample has exceeded the hold time the analyst should contact the group leader before continuing with the batch.

10.0 SAMPLE PREPARATION

10.1 For each liter of drinking water, add 5 mL 1M $C_6H_8O_7*H_2O$ and few drops methyl orange indicator. The solution should be red.

NOTE: At the time of sample collection add 2 mL 16N HN03 for each liter of water.

10.2 Add 10 mL lead carrier (15 mg/mL), 2 mL strontium carrier (10 mg/mL) 2.0 mL barium carrier (16 mg/mL), and 1 mL yttrium carrier (18 mg/mL); stir well. Heat to incipient boiling and maintain at this temperature for 30 minutes.

10.3 Add 15N NH_4OH until a definite yellow color is obtained, then add a few drops excess. Precipitate lead and barium sulfates by adding 18N H_2SO_4 until the red color reappears, then add 0.25 mL excess. Add 5 mL $(NH_4)_2SO_4$ (200 mg/mL) for each liter of sample. Stir frequently and keep at a temperature of about 900°C for 30 minutes.

10.4 Cool slightly, then filter with suction through a 47-mm metricel membrane filter (GA-6,0.45 pore size). Make a quantitative transfer of precipitate to the filter by rinsing last particles out of beaker with a strong jet of water.

10.5 Carefully place filter with precipitate in the bottom of a 250 mL beaker. Add about 10 mL 16N HNO_3 and heat gently until the filter completely dissolves. Transfer the precipitate into a polypropylene centrifuge tube with additional 16N HNO_3. Centrifuge and discard supernate.

10.6 Wash the precipitate with 15 mL 16N HNO_3, centrifuge, and discard supernate.

10.7 Repeat step 10.6.

10.8 Add 25 mL basic EDTA reagent, heat in a hot water bath, and stir well. Add a few drops 10N NaOH if the precipitate does not readily dissolve.

10.9 Add 1 mL strontium-yttrium mixed carrier and stir thoroughly. Add a few drops 10N NaOH if any precipitate forms.

10.10 Add 1 mL $(NH_4)_2SO_4$ (200 mg/mL) and stir thoroughly. Add 17.4N CH_3COOH until barium sulfate precipitates, then add 2 mL excess. Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate.

10.11 Add 20 mL basic EDTA reagent, heat in a hot water bath, and stir until precipitate dissolves. Repeat steps 10.9 and 10.10. (Note time of last barium sulfate precipitation; this is the beginning of the actinium-228 ingrowth time.)

10.12 Dissolve the precipitate in 20 mL basic EDTA reagent as before, then add 1.0 mL yttrium carrier (9 mg/mL) and 1 mL lead carrier (1.5 mg/mL). If any precipitate forms, dissolve by adding a few drops 10N NaOH. Cap the polypropylene tube and age at least 36 hours.

10.13 Add 0.3 mL $(NH_4)_2S$ and stir well. Add 10N NaOH dropwise with vigorous stirring until lead sulfide precipitates, then add 10 drops excess. Stir intermittently for about 10 minutes. Centrifuge and decant supernate into a clean tube.

10.14 Add 1 mL lead carrier (1.5 mg/mL), 0.1 mL $(NH_4)_2S$, and a few drops 10N NaOH. Repeat precipitation of lead sulfide as before. Centrifuge and filter supernate through Whatman #42 filter paper into a clean tube. Wash filter with a few mL water. Discard residue.

10.15 Add 5 mL 18N NaOH, stir well and digest in a hot water bath until yttrium hydroxide coagulates. Centrifuge and decant supernate into a beaker. Save for barium yield determination, step 10.20 (Note time of yttrium hydroxide precipitation; this is the end of the actinium-228 ingrowth time and beginning of actinium-228 decay time.)

10.16 Dissolve the precipitate in 2 mL 6N HNO_3. Heat and stir in a hot water bath about 5 minutes. Add 5-mL water and reprecipitate yttrium hydroxide with 3 mL 10N NaOH. Heat and stir in a hot water bath until precipitate coagulates. Centrifuge and discard supernate.

10.17 Dissolve precipitate with 1 mL 1N HNO_3 and heat in hot water bath a few minutes. Dilute to 5 mL and add 2 mL 5% $(NH_4)_2C_2O_4*H_2O$. Heat to coagulate, centrifuge and discard supernate.

10.18 Add 10 mL water, 6 drops 1N HNO_3 and 6 drops 5% $(NH_4)_2C_2O_4.*H_2O$. Heat and stir in a hot water bath a few minutes. Centrifuge and discard supernate.

10.19 To determine yttrium yield, transfer quantitatively to a tared stainless steel planchet with a minimum amount of water. Dry under an infra-red lamp to a constant weight and count in a low-background beta counter.

10.20 To the supernate from step 10.15, add 4 mL 16N HNO_3 and 2 mL $(NH_4)_2SO_4$ (200 mg/mL), stirring well after each addition. Add 17.4N CH_3COOH until barium sulfate precipitates, then add 2 mL excess. Digest on a hot plate until precipitate settles. Centrifuge and discard supernate.

10.21 Add 20 mL basic EDTA reagent, heat in a hot water bath, and stir until precipitate dissolves. Add a few drops 10N NaOH if precipitate does not readily dissolve.

10.22 Add 1 mL $(NH_4)_2SO_4$ (200 mg/mL) and stir thoroughly. Add 17.4N CH3COOH until barium sulfate precipitates, then add 2 mL excess. Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate.

10.23 Wash precipitate with 10 mL water. Centrifuge and discard supernate.

10.24 Transfer precipitate to a tared stainless steel planchet with a minimum amount of water. Dry under an infra-red lamp and weigh for barium yield determination.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to EPI SOP "Preparation of Radioactive Standards" (M-001).

11.2 All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in EPI SOP "Preparation of Radioactive Standards" (M-001), Section 19.0.

11.3 Primary standards are kept in the laboratory in a secured cabinet. Secondary, working, standards are kept at the bench area in an enclosed plastic cabinet.

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 For direction on calibration and instrument performance see EPI SOP "LB4100 Gross Alpha/Beta Counter Operating Instructions" (I-006).

12.2 Counting Efficiencies: It is not practical to calibrate the beta counter with actinium-228, since its' half-life is only six hours. Standard strontium-89 (tl/2 = 51d) may be substituted. Strontium-89 has an average beta energy of 0.589 KeV, while the average beta energy for actinium-228 is 0.404 KeV. A standard strontium-89 tracer solution can be used to determine beta efficiencies over a range of precipitate weights on the stainless steel planchet.

12.3 If radium-226 analyses are also required, see Sec. 7, Method 903.1.

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 For direction on calibration and instrument performance see EPI SOP "LB4100 Gross Alpha/Beta Counter Operating Instructions" (I-006).

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 For maintenance of system see EPI SOP "Counting Room Instrumentation Maintenance and Performance Checks" (I-010).

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 Determine the radium-228 pCi/unit according to the following equation:

$$\text{Ra-228 pCi / L} = \frac{C}{2.22 * EVR} * \frac{\lambda t_2}{(1-e^{-\lambda t_2})} * \frac{1}{(1-e^{-\lambda t_3})} * \frac{1}{(e^{-\lambda t_1})}$$

Where:

C = average net count rate, cpm
E = counter efficiency, for actinium-228, or comparable beta energy nuclide
V = volume of sample analyzed, in liters,
R = fractional chemical yield of yttrium carrier (step 10.19) multiplied by fractional chemical yield of barium carrier (step 10.24)
2.22 = conversion factor from dpm/pCi.
λ = decay constant for actinium-228 (0.001884 min^{-1})

15.2 Record the following information on the que sheet: preparation date, analysts initials, spike isotope, spike code, spike volume, LCS isotope, LCS code, LCS volume, nominal concentration LCS, and nominal concentration MS. For each sample record the detector number, sample mass, sample date, and sample time.

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification

16.1.1 Refer to EPI SOP "Analyst and Analytical Methods Validation Procedures" (D-002) for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank will accompany each batch of 20 or less samples. The reported value should be less than or equal to the CRDL for all target isotopes.

16.2.2 A matrix spike (MS) should be run with every batch of 20 samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\%\,\mathrm{Rec} = \frac{\mathrm{spike(pCi/g)} - \mathrm{sample(pCi/g)}}{\mathrm{spiked\ amount\,(pCi/g)}} * 100$$

16.2.3 A sample duplicate should be run with every batch of 20 or less samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$\mathrm{RPD} = \frac{\mathrm{high\,sample(pCi/g)} - \mathrm{low sample(pCi/g)}}{\mathrm{Average\ (pCi/g)}} * 100$$

16.2.4 A laboratory control spike (LCS) should be run with every batch of 20 samples or less. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\mathrm{LCS} = \frac{\mathrm{observed\_pCi/unit}}{\mathrm{known\_pCi/unit}} * 100$$

16.3 Actions Required if the Quality Control Requirements Are Not Met

16.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non conformance report as outlined in GEL SOP "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

17.1 The analyst turns in the raw data, review checklist, que sheet, spreadsheet, data report, NCR's (if applicable), and other appropriate information in a batch to the data review specialist.

17.2 Analyst's data go through the following process of review:

17.2.1 The first level of review is the analyst review. The analyst will perform the following review procedure:

17.2.1.1 Visually check the que sheet, spreadsheet, raw data and data report to make sure the information has been transcribed correctly.

17.2.1.2 Check to see that the required detection limit (RDL) is met if required.

17.2.1.3 Complete the batch checklist.

17.2.2 The second level review is performed by the Data Review Specialist who reviews the batch checklist, checks requested and non-requested hits, and reviews the transcription.

17.2.3 The third level review is completed by the Quality Group Leader who reviews the checklist and checks requested hits.

17.3 After the review process is complete, the data is transmitted from the laboratory personnel to the reporting personnel as outlined in EPI SOP "Data Review and Validation Procedures" (D-003).

17.4 The Data Review/Quality Group Leader is responsible for reporting the data.

18.0 RECORDS MANAGEMENT

18.1 Each analysis that is performed on the instrument is documented in the run log according to GEL SOP "Run Logs" (GL-LB-E-009).

18.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

19.1 Radioactive samples and material shall be handled and disposed of as outlined in EPI SOP "Radioactive Waste Handling Procedures" (S-005).

20.0 REFERENCES

20.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

20.2 Krieger, H.L. Interim Radiochemical Methodology for Drinking Water, Ra-228 in Drinking Water, EPA 600/4-75-008 (Revised). Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, OH. September 1975.

STANDARD OPERATING PROCEDURE

FOR

THE DETERMINATION OF

SELENIUM AND TELLURIUM

UNCONTROLLED DOCUMENT

Process Owner(s):	Tim C. Winters, Radioanalytical Technician
	Russell L. Moser, EPI Group Leader
Technical Review by:	Barry D. Stewart, Senior Technical Specialist
Management Review by:	James B. Westmoreland, EPI Laboratory Manager
Quality Review by:	Genie G. Bost, EPI Quality Group Leader
Approved and Authorized by:	Heyward H. Coleman, EPI President

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR THE DETERMINATION OF SELENIUM AND TELLURIUM

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This standard operation procedure provides necessary instructions to conduct analysis for Selenium-79 by liquid scintillation counting and Tellurium-125m by low energy gamma counting.

2.2 This procedure is applicable to water soil, and swipes. Selenium (Se) and Tellurium (Te) are separated from interfering radionuclides by TEVA chromatography resin. Chemical yield is determined gravimetrically by precipitating elemental Se and Te from solution and weighing. The Se precipitate and filter are dissolved in nitric acid and counted in a liquid scintillation counter. The Te precipitate is covered with mylar tape and counted on a low energy photon detector. Since standards of Se-79 are not readily available, counting efficiency is determined from C-14, which has an equivalent beta particle energy.

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for Se-79 is 50 pCi/L or 5 pCi/g for all isotopes. Typical minimum detectable activity (MDA) for samples analyzed for Te-125m is 10 pCi/L or 5 pCi/g for all isotopes.

3.2 Method Precision: Typical relative percent difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value. Analyst training records are kept on hand in the human resource department.

4.0 DEFINITIONS

4.1 NIST (National Institute of Standards and Technology): For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.2 Type II water: Deionized water.

4.3 LIMS (Laboratory Information Management System): The database system used to store and report data.

5.0 METHOD VARIATIONS

5.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Sr. Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. Environmental Physics, Inc. (EPI) maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS).

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are nitrile gloves for concentrated acids and bases, and potassium ferricyanide in neat form. Work under a hood when using concentrated acids and bases.

6.4 The handling of radioactive samples is outlined in EPI SOP "Handling of Radioactive Samples" (M-001). General guidelines include:

6.4.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

6.4.2 A plastic apron may be worn over the lab coat for added protection from contamination when working with radioactive samples.

6.4.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.4.4 Prohibit admittance to immediate work area.

6.4.5 Post signs indicating radioactive samples are in the area.

6.4.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in EPI.

6.4.7 Segregate radioactive wastes from non radioactive wastes. Waste containers are obtained from the waste coordinator.

6.5 Refer to EPI SOP "EPI Laboratory Waste Disposal and Emergency Instructions" (S-011), and GEL/EPI Laboratory Waste Management Plan (GL-LB-G-001) for instructions on how materials are disposed in the laboratory.

6.6 If there is any question regarding the safety of any laboratory practice, stop immediately, and consult the group leader prior to carrying out the rest of the procedure.

6.7 When handling biological samples protect the hands and forearms by wearing gloves and a laboratory coat to avoid contact of the biological material with the skin. Protect the eyes by wearing safety glasses and if desired a splash shield.

6.8 If cutting of meats or other tissue with cutlery is required, the analyst will wear a cutting glove of mesh steel over disposable gloves to avoid cuts which could infect the analyst with pathogens.

6.9 Any procedure which volatilizes biological substances such as drying or ashing, must be conducted in a hood or other suitable containment device.

6.10 Decontamination of work surfaces exposed to biological samples is performed by wiping the work area with a diluted (1:10) bleach solution and water as soon as possible following analytical operations.

6.11 Exterior protective clothing shall be removed prior to exiting the bioassay sample preparation area in order to prevent the inadvertent spread of biohazards to the rest of the laboratory.

6.12 Hands will be washed with an antibacterial soap directly after handling biological samples.

7.0 INTERFERENCES

7.1 This procedure is subject to the usual quenching interferences typical of liquid scintillation measurements. The quench curve used for Se-79 is the same one used for C-14 and has been found adequate for this procedure.

7.2 High concentrations of radionuclides emitting high energy beta or alpha particles can cause tailing into the Se-79 counting window. The final Se precipitate from step 10.4.11 is counted on a gas-flow proportional counter (prior to LS counting) to determine if significant alpha contamination remains. Spectra of samples with high count rates should be examined carefully to ensure such interferences are not present. Reducing the width of the counting window will not completely remove the effects of tailing from interferences. If significant alpha activity is detected during the gross alpha screen of the Se precipitate, the chemical separations must be repeated.

7.3 The spike used for MS and LCS is Se-75, which has a different LS spectrum from Se-79 (C-14). Counting efficiency for MS and LCS is determined from a standard of Se-75 prepared with the same quench as MS and LCS. Alternatively, a quench curve for Se-75 would have to be prepared.

7.4 To prevent volatility losses, solutions of Se (and also Te) should not be evaporated to complete dryness. Even in the presence of nitric acid, Se can be volatilized if it is allowed to reach complete dryness. It is therefore critical that the analyst constantly attend the evaporation of samples so that they can be removed from the hotplate while still moist. The most frequent cause of low yields is overheating sample residues.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 150 mL beakers

8.1.2 250 mL beakers

8.1.3 400 mL beakers

8.1.4 100 mL Teflon beakers

8.1.5 "Medium" disposable plastic ion exchange columns, 7 cm length

8.1.6 Hot plate

8.1.7 20 mL glass scintillation vials

8.1.8 47 mm filtration apparatus

8.1.9 47 mm glass fiber filters

8.1.10 47 mm cellulose nitrate filters, 0.45µ pore size

8.1.11 47 mm Tuffryn HT-450 filters

8.2 Reagents, chemicals, and standards

8.2.1 Scintillation cocktail, Ready-Gel or equivalent

8.2.2 Hydrochloric Acid, concentrated

8.2.3 Se carrier, 10 mg Se/mL

8.2.4 Te carrier, 10 mg Te/mL

8.2.5 Se-75 standard, (~500 dpm/mL)

8.2.6 Te-123m standard, (~500 dpm/mL)

8.2.7 TEVA resin

8.2.8 10M NaOH. Dissolve 400 grams of NaOH pellets in 900 mL of water in a glass beaker. Cool and dilute to 1 liter with DI water.

8.2.9 Ethanol, 80%. Dilute 400 mL of ethanol to 500 mL with water

8.2.10 Nitric acid, 1.25M. Dilute 158 mL of concentrated HNO_3 to 2 liters with DI water.

8.2.11 Nitric acid, concentrated

8.2.12 Boric acid, 5%. This is almost a saturated solution. Add 50 grams of boric acid to 1L of water and stir until dissolved. The presence of undissolved material has no adverse effect.

8.2.13 Sulfurous Acid, concentrated

8.2.14 HCl, 6M. Mix equal volumes of concentrated HCl and DI water.

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 Aqueous samples should be collected in a plastic container and preserved with nitric acid to pH ~2.

NOTE 1: Certain highly alkaline samples such as saltstone solution are not preserved with acid. These samples are run "as received" without pretreatment.

9.2 Swipes are collected without preservation. The sample will generally have been digested and dissolved to prepare a stock solution that is used for a number of other tests in addition to Se-79 and Te-125m.

9.3 Soils are dried, ground, and homogenized according to EPI SOP "Soil Sample Preparation for the Determination of Radionuclides" (A-021).

10.0 SAMPLE PREPARATION

10.1 Aqueous Samples

10.1.1 Transfer the sample aliquot (50-500 mL) to a suitable beaker. Record the sample volume on the que sheet. Add 2.0 mL of Se carrier and 2.0 mL of Te carrier. (Add Te-123m and Se-75 tracers to the appropriate MS and LCS samples.)

10.1.2 Evaporate to 100 mL using the approximate graduations on the beaker as a guide, and add HCl to bring the volume to 150 mL. Allow the solution to cool. The optimal HCl concentration is 3-6M.

NOTE 2: Highly alkaline samples such as the saltstone solution should have the pH verified after adding HCl.

10.1.3 Add 25 mL of sulfurous acid and stir. Allow the precipitate to stand at least 45 minutes to ensure complete precipitation. A precipitate which slowly fades indicates that the sample contains oxidizing agents, and more sulfurous acid is needed. Should this condition be noted, add 25 mL additional sulfurous acid and 15 mL HCl.

NOTE 3: Samples with high nitrate concentration such as saltstone solution may not behave as expected. If the HCl concentration is low (i.e. 3M), black Te may precipitate first. It is then difficult to determine if Se has been precipitated. If Te precipitates first, add a volume of HCl equal to one third of the solution volume. Also, if the precipitate fades, continue adding 25 mL portions of sulfurous acid followed by 15 mL portions of HCl until the precipitate stabilizes.

10.1.4 Continue with "Determination," beginning with Step 10.4.

10.2 Swipes

10.2.1 Swipes have generally been previously wet-ashed with nitric acid and diluted to 500 mL to prepare a solution for gamma spectroscopic analysis and serve as a stock solution for other required tests. Obtain a 50 mL aliquot of the stock solution, note the volume on the que sheet, and transfer to a 250 mL beaker. Add 2.0 mL of Se carrier, 2.0 mL of Te carrier, and 40 mL of HCl. (Add Te-123m and Se-75 tracers to the appropriate MS and LCS samples.)

10.2.2 Add 25 mL of sulfurous acid and stir. A red precipitate will develop almost immediately, gradually turning black as Te precipitates. Allow the precipitate to stand at least 45 minutes for complete precipitation.

NOTE 4: If the precipitate is not visible, it is due to either improper HCl concentration, insufficient reducing agent, or both. Add 25 mL of sulfurous acid and stir. If the precipitate still does not form, check the HCl concentration as described above in Note 2 and repeat 10.2.2.

10.2.3 Continue with "Determination," beginning with Step 10.4.

10.3 Soil

10.3.1 Transfer 1-5 grams of soil to a 150 mL beaker. Note the sample weight on the que sheet. Add 2.0 mL of Se carrier, and 2.0 mL of Te carrier. (Add Te-123m and Se-75 tracers to the appropriate MS and LCS samples.)

10.3.2 Add 25 mL of 8M HNO_3, cover, and heat to near boiling for 30 minutes. Cool.

10.3.4 Transfer to a 50 mL centrifuge tube and centrifuge. Decant the supernate into a 100 mL Teflon beaker.

10.3.5 Add 5 mL of HF and evaporate to moist dryness. DO NOT OVERHEAT.

10.3.6 Dissolve the residue in 20 mL of HCl and 10 mL of 5% boric acid. Warm gently, but do not boil. Cool and transfer to a 150 mL beaker.

10.3.7 Add 15 mL of sulfurous acid and stir. A red precipitate of Se will develop almost immediately, gradually turning black as Te precipitates. Allow the precipitate to stand at least 45 minutes for complete precipitation.

10.3.8 Continue with "Determination," beginning with Step 10.4

10.4 Determination

10.4.1 Filter through a 47-mm 0.45-um cellulose nitrate filter. The precipitate will not be weighed, so the tare weight of the filter is not required. Rinse the precipitate and filter with water and 80% alcohol and place the filter on a Kimwipe or paper towel outside the hood for about 5 minutes to allow most of the alcohol to evaporate.

10.4.2 Place the filter and precipitate in a clean 150 mL beaker, add 5 mL of HNO_3, cover with a watch glass and heat until the filter is completely destroyed and the precipitate is dissolved. Continue heating, adding HNO_3 if necessary, until brown fumes are no longer visible. Remove the lid and evaporate to moist dryness. DO NOT OVERHEAT. Cool.

10.4.3 Dissolve the residue in 10 mL of 6M HCl and dilute to 25 mL with water. Cover and let stand 15 minutes so that Se and Te are reduced from +6 to +4 oxidation states.

10.4.4 For each sample, fill a 7 cm column with TEVA resin to the 6 cm mark. Allow 15-20 minutes for the resin to settle completely, then insert a small plug of glass wool (or a frit) to prevent disturbing the resin. Condition the column with 20 mL of 6M HCl. Discard the effluent.

10.4.5 Place a clean 150 mL glass beaker under each column. Using 6M HCl from a wash bottle, quantitatively transfer the sample to the TEVA column. The column should turn yellow as Te is loaded. Rinse with 15 mL of 6M HCl, collecting the effluent in the same beaker as the load effluent. Reserve for Se determination.

10.4.6 Place a 150 mL beaker under the column. Elute Te with 20 mL of 1.25M HNO_3. Reserve for Te determination.

10.4.7 To the Se portion from 10.4.5, add 5 mL of sulfurous acid and mix well. Se will precipitate instantaneously. Allow the precipitate to stand for 30 minutes.

10.4.8 Filter through a tared 47 mm glass fiber filter. Rinse with 25 mL portions of water and then rinse the funnel walls and filter with 25 mL of 80% alcohol.

10.4.9 With vacuum still connected, remove the filter chimney. Rinse the filter copiously with 80% alcohol, paying particular attention to the edges of the filter. This is necessary to remove the last traces of HCl from the filter and precipitate.

10.4.10 Place the filter on a clean paper towel to remove most of the moisture and then dry for 10 minutes at 105 degrees Celsius. Cool and weigh.

10.4.11 Count the Se precipitate in a gas-flow conter for 5 minutes. If the gross alpha activity is greater than 20 times background, the sample will have to be re-processed to remove the alpha contamination which would cause a spectral interference. If the gross alpha count rate is less than 20 times background, proceed with step 10.4.12.

10.4.11.1 Place the Se precipitate and filter in a teflon beaker and add 5 mL of HNO3 and 2 mL of HF. Heat to dissolve the filter and drive off SiF4 and excess HF, and then evaporate to moist dryness. DO NOT OVERHEAT.

10.4.11.2 Dissolve the residue in 10 mL of 6M HCl. Let stand at least 15 minutes. Meanwhile, perform step 10.4.4 to prepare a new chromatography column.

10.4.11.3 Place a 150 mL beaker under each column. Using 6M HCl from a wash bottle, quantitatively transfer the sample to the TEVA column. The column should turn yellow as Te is loaded. Rinse with 15 mL of 6M HCl, collecting the effluent in the same beaker as the load effluent.

10.4.11.4 Add 5 mL of sulfurous acid. Allow the precipitate to stand for 30 minutes. Resume the analysis with step 10.4.8.

10.4.12 Transfer the Se precipitate and filter to a teflon beaker. Add 5 mL of HNO_3 and 2 mL HF. Heat to dissolve the filter and drive off SiF_4. Continue evaporation to moist dryness. DO NOT OVERHEAT.

10.4.13 Dissolve the residue in 1 mL of 6M HCl and 1 mL of 5% boric acid. Transfer to a glass LSC vial, keeping the total volume less than 9 mL. Add 0.5 mL of 10M NaOH to neutralize most of the acid, and then dilute to 10 mL. (An empty vial with a mark at the 10 mL level can be used as a convenient guide).

10.4.14 Add 10 mL of Ready-Gel to the vials. Cover the vial with a small piece of Parafilm and screw on the cap. Shake vigorously to mix the contents.

NOTE: LSC vials with Al foil-lined caps will reduce Se +4 to elemental Se, causing a reddish color to appear in the vial. The color will cause quenching, but it also causes separation of Se from the counting fluid.

10.4.15 Check the H# of the sample to ensure that it is within the current C-14 calibration range. If the H# is below the minimum level, add CCL_4 to increase the amount of quenching. The H# will be increased roughly two units for each uL of CCL_4 added.

10.4.16 Allow the sample to dark adapt in the LSC for an hour and then count for the length of time necessary to meet client specific MDA. This will usually be 30-60 minutes.

10.4.17 The remainder of the procedure is for the determination of Te. To the effluent from step 10.4.6 add 20 mL of HCl and 25 mL of sulfurous acid. Allow the precipitate to stand for 30 minutes.

10.4.18 Prepare a Tuffryn HT-450 filter for each sample by placing it on the filter base, connecting the vacuum, and rinsing thoroughly with 80% ethanol. Dry in the oven at 105 degrees Celsius for 10 minutes, cool, and weigh. This pretreatment is necessary to bring the filter to constant weight.

10.4.19 Filter the samples from 10.4.17 through the tared Tuffryn HT-450 filters. Rinse copiously with water and finally with 80% alcohol. (Avoid dislodging the precipitate from the filter during rinsing because counting efficiency is affected by the size and thickness of the precipitate).

NOTE: Tuffryn HT-450 filters are used because the Te precipitate varies in particle size and is not always quantitatively retained by glass fiber filters. In addition, with glass fiber filters, there is considerable penetration into the filter medium by the precipitate particles, which increases self-absorption.

10.4.20 With vacuum still connected, remove the filter funnel and gently continue rinsing the precipitate and filter with 80% alcohol, paying particular attention to the edges of the filter. This is necessary to remove the last traces of HCl from the filter and precipitate.

10.4.21 Place the filter on a clean paper towel to remove most of the moisture and then dry for 10 minutes at 105 degrees Celsius. Cool and weigh.

10.4.22 Cover with 3M #3690 mylar tape, and fasten the filter to the outside bottom of a flat-bottom planchet. Count on an x-ray detector for the length of time necessary to meet the client specific MDA.

NOTE: The use of any other grade or type of tape to cover the filter will affect the efficiency calibration. If the specified grade tape is not available, prepare new calibration standards covered with the available tape.

10.5 Cleanup of glassware

10.5.1 All glassware which has contained Se or Te precipitates must be rinsed with hot nitric acid before the usual cleaning procedure. This is necessary to dissolve the trace amount of precipitate that invariably remains in the beakers. A convenient way to do this is to add 10 mL of HNO_3, cover with a watch glass, and heat on a hotplate until a good reflux of HNO_3 is observed. Cool and dilute the acid with water, and discard as liquid rad waste. Follow up with the usual cleaning process.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to EPI SOP "Preparation of Radioactive Standards" (M-001).

11.2 Source materials are ordered from any of four main suppliers listed below. All four companies are called for a quote, and the most economical price for the laboratory needs is selected. All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate infomation. Source preparation data and certificate inventory is described in EPI SOP "Preparation of Radioactive Standards" (M-001), Section 19.0.

11.2.1 National Institute of Standards and Technology (NIST), Gaithersburg, MD

11.2.2 Isotope Products Laboratories, Burbank, CA

11.2.3 Amersham Corporation, Arlington Heights, IL

11.2.4 North American Scientific Products and Services, North Hollywood, CA.

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 Refer to EPI SOP "Beckman LS 6000-6500 Operating Procedure" (I-004) for instructions concerning instrument calibration.

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 Refer to EPI SOP "Beckman LS 6000-6500 Operating Procedure" (I-004) for instructions concerning instrument operation.

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 Refer to EPI SOP "Beckman LS 6000/6500 Operating Procedure" (I-004) for instructions concerning the scintillation counters.

14.2 Refer to EPI SOP "Counting Room Instrumentation Maintenance and Performance Checks" (I-010) for instructions concerning instrument maintenance.

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 Calculation of Se-79 activity. The activity in the sample is calculated by Excel spreadsheet according to the following equation:

$$pCi/L = \frac{1000\left(C_B - C_S\right)}{2.22\,E\,Y\,V}\,\frac{F_{Dil}}{F_{Sample}}$$

where:

1000 = conversion from mL to L
C_B = Sample cpm
C_S = Background cpm
2.22 = Conversion to pCi
E = Counting efficiency
Y = Selenium yield
V = Aliquot of sample taken for analysis, mL
F_{Dil} = Dilution factor = volume of stock solution prepared from ashed swipe
F_{Sample} = Fraction of the total swipe ashed in preparing stock solution

15.2 Se-79 MDA

$$MDA, pCi/L = \frac{1000\left(2.71+4.65\sqrt{C_B T}\right)}{2.22 E Y V T} \frac{F_{Dil}}{F_{Sample}}$$

where variables are the same as defined in 15.1, and:

T = Sample count length, minutes

15.3 Counting uncertainty, pCi/L, is based only on counting errors unless specified otherwise by a specific client requirement:

$$Error = A\left(\frac{1.96\sqrt{\frac{C_S}{T}+\frac{C_B}{T_B}}}{C_S - C_B}\right)$$

where variables are the same as defined in 15.1 and 15.2, and:

A = Se-79 activity, pCi/unit
T_B = Background count length, minutes

15.4 Te-123M calculations are performed by the gamma analysis software using expressions equivalent to those given in 15.1-15.3.

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification Requirements

16.1.1 Refer to EPI SOP "Analyst and Analytical Methods Validation Procedures" (D-003) for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank should accompany each batch of samples. The reported value of the blank should be less than or equal to the contract required detection limit (CRDL).

16.2.2 A matrix spike (MS) should be run with every batch of samples. The recovery of the MS should be between 75-125%. The MS recovery is calculated as follows:

$$MS\,Recovery,\% = \frac{Spike - Sample}{SNC} 100$$

where:

Spike = Se-79 activity in the MS,
Sample = Se-79 activity in the sample without added spike, and

$$SNC = \frac{1000(Se - 75\ dpm)(ml\ added)}{2.22\left(\frac{1000\ V\ F_{Sample}}{F_{Factor}}\right)}$$

16.2.3 A duplicate sample should be run with every batch. The relative percent difference (RPD) between the actual sample and the QC duplicate should be less than or equal to 20% if both the sample and the QC duplicate results are greater than 5 times MDA, or 100% if they are both less than 5 times MDA. The RPD is calculated as follows.

$$RPD,\% = 100 \times \left(\frac{|DUP_1 - DUP_2|}{DUP_1 + DUP_2 / 2}\right)$$

16.2.4 A Laboratory Control Sample (LCS) should be run with every batch. The recovery of the LCS should fall between 75 - 125%. The LCS recovery is calculated as follows:

$$LCS = \frac{observed\_pCi/unit}{known\_pCi/unit} * 100$$

16.3 If any of the QC criteria in Section 12.2 cannot be satisfied, the analyst should inform the group leader and initiate a Non-conformance report as outlined in GEL SOP "Documentation of Nonconformance Reporting, and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

17.0 ANALYSIS PROCEDURES

17.1 Refer to EPI SOP "Beckman LS 6000-6500 Operating Procedure" (I-004) for instructions concerning instrument operation.

17.2 Refer to EPI SOP "Microvax Gamma Spectroscopy Operating Procedure" (I-001) for instructions concerning instrument operation for Te-125m.

18.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

18.1 The analyst turns in the raw data, review checklist, que sheet, spreadsheet, data report, NCR's (if applicable), and other appropriate information in a batch to the data review specialist.

18.2 Analysts data go through the following process of review:

18.2.1 The first level of review is the analyst review. The analyst will perform the following review procedure:

18.2.1.1 Visually check the que sheet, spreadsheet, raw data and data report to make sure the information has been transcribed correctly.

18.2.1.2 Check to see that the required detection limit (RDL) is met, if required.

18.2.1.3 Complete the batch checklist.

18.2.2 The second level review is performed by the Data Review Specialist who reviews the batch checklist, and reviews the data for transcription errors.

18.2.3 The third level review is completed by the Quality Group Leader who reviews the checklist.

18.3 After the review process is complete, the data is transmitted from the laboratory personnel to the reporting personnel as outlined in EPI SOP "Data Review and Validation Procedures" (D-003).

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

19.1 Radioactive samples and material shall be handled and disposed of as outlined in EPI SOP "Radioactive Waste Handling Procedures" (S-011).

20.0 REFERENCES

20.1 Hillebrand, W. F., and Lundell, G. E. F. Applied Inorganic Analysis. John Wiley & Sons, Inc., New York: 1955. pp 258-267.

20.2 Goheen, S. C., et. al., Eds. "Determination of Se-79 in Aqueous Samples," RP530, from DOE Methods for Evaluating Environmental and Waste Management Samples, Oct. 1994, Battelle Pacific Northwest Laboratory, Richland, WA.

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STANDARD OPERATING PROCEDURE

FOR

THE ISOTOPIC

DETERMINATION OF NEPTUNIUM

(GL-EPI-A-032-Revision 1)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

N/A		10/15/99	
Process Owner	(Sign and Date)		(Print Name)
Richard M. Kinney	10/15/99	Richard M. Kinney	
Technical Review	(Sign and Date)		(Print Name)
Robert C. Pullano	11/2/99	Robert C. Pullano	
Quality Review	(Sign and Date)		(Print Name)
James B Westmoreland	11/2/99	James B Westmoreland	
Approval and Authorization	(Sign and Date)		(Print Name)

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TABLE OF CONTENTS

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1.0 ISOTOPIC DETERMINATION OF NEPTUNIUM

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic neptunium (Np) in a variety of matrices.

2.2 A sample is digested if necessary and aliquoted. Transuranic elements are scavenged by coprecipitation with iron. The precipitate is dissolved and separation of elements is accomplished through ion exchange resins. The elements are then prepared for the measurement of radioactive isotopes by coprecipitation with cerium fluoride. The cerium fluoride precipitate is trapped on a filter, mounted on a stainless steel disk and placed in a partially evacuated chamber for measurement of isotopic alpha emission.

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for Np is 1 pCi/L or 1 pCi/g.

3.2 Method Precision: Typical relative percent difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value. Analyst training records are kept on hand in the human resource department.

4.0 DEFINITIONS

4.1 Disintegrations per minute (dpm), unit of radioactive measurement. One radioactive decay event is one disintegration.

4.2 NIST: National Institute of Standards and Technology.

4.3 pH: Unit of measurement of hydrogen ion concentration.

4.4 Supernate: The clear solution remaining after precipitation has been completed.

4.5 Eluate/eluant: Reagent solution used to strip selected analyte(s) from a resin column.

4.6 Type II water: Deionized water.

4.7 LIMS: Laboratory Information Management System. The database system used to store and report data.

4.8 pCi: Unit of radioactive measurement. One pCi is equal to 2.22 dpm.

4.9 CRDL: Contract Required Detection Limit

5.0 METHOD VARIATIONS

5.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Sr. Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. General Engineering Laboratories, Inc. (GEL) maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the library and in the laboratory, respectively. Individual sample MSDS forms provided by the clients are kept in Login.

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are nitrile gloves for concentrated acids and bases, and potassium ferricyanide in neat form. Work under a hood when using concentrated acids and bases.

6.4 The handling of radioactive samples is outlined in GL-EPI-M-001, "Handling of Radioactive Samples." General guidelines include:

6.4.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

6.4.2 Wear a plastic apron over lab coat when working with radioactive samples.

6.4.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.4.4 Prohibit admittance to immediate work area.

6.4.5 Post signs indicating radioactive samples are in the area.

6.4.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in the radiochemistry lab.

6.4.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

6.5 Refer to GL-EPI-S-005, "Radioactive Waste Handling Procedures," for instructions on how materials are disposed.

6.6 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the group leader prior to carrying out the rest of the procedure.

6.7 When handling biological samples protect the hands and forearms by wearing gloves and a laboratory coat to avoid contact of the biological material with the skin. Protect the eyes by wearing safety glasses and if desired a splash shield.

6.8 If cutting of meats or other tissue with cutlery is required, the analyst will wear a cutting glove of mesh steel over disposable gloves to avoid cuts that could infect the analyst with pathogens.

6.9 Any procedure that volatilizes biological substances such as drying or ashing, must be conducted in a hood or other suitable containment device.

6.10 Decontamination of work surfaces exposed to biological samples is performed by wiping the work area with a diluted (1:10) bleach solution and water as soon as possible following analytical operations.

6.11 Exterior protective clothing shall be removed prior to exiting the bioassay sample preparation area in order to prevent the inadvertent spread of biohazards to the rest of the laboratory.

6.12 Hands will be washed with an antibacterial soap directly after handling biological samples.

7.0 INTERFERENCES

7.1 Internal tracer standards may have ingrown daughters that may interfere with the analysis.

7.2 Short-lived radioactive progeny may ingrow on prepared filters.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 Silicon surface barrier detectors with associated electronics, Vacuum chambers, and data reduction capabilities.

8.1.2 Vacuum pump and filtration apparatus (25 mm)

8.1.3 Gelman metricel 25 mm filters with 0.1 um pore size

8.1.4 Gelman polypropylene 25 mm support filter

8.1.5 Stainless steel disks. 29 mm diameter

8.1.6 Double stick tape

8.1.7 Stainless steel tweezers

8.1.8 Polypropylene centrifuge tube (50 mL)

8.1.9 Sample drying and ashing apparatus

8.1.10 Sample homogenization apparatus

8.1.11 Glass beakers

8.1.12 Teflon beakers

8.1.13 Flat planchets, 2 inches in diameter

8.1.14 LB5100W or another appropriate gas flow proportional counter

8.2 Reagents, Chemicals and Standards

8.2.1 Ammonium hydroxide concentrated (14 N)

8.2.2 Cerium(III)nitrate hexahydrate. Dilute 500 mL of 1000µg/mL Cerium standard to 1000 mL with DI water.

8.2.3 Ethyl alcohol.(80%) Dilute 400 mL ethanol to 500 mL with DI water.

8.2.4 Hydrofluoric acid concentrated (48%).

8.2.5 TEVA pre-packed

8.2.6 NIST traceable standards: Am-243 and Np-237.

8.2.7 Nitric acid concentrated 16N

8.2.8 3 M HNO_3/ 1 M $AL(NO_3)_3$. Add 191 mL of concentrated HNO_3 (sp gr 1.42) to 700 mL of water. Dissolve 375 g $AL(NO_3)_3$ $9H_2O$ and dilute to 1 L with water.

8.2.9 0.6 M ferrous sulfamate. Add 285 g of sulfuric acid (NH_2SO_2H) to 600 ml of water, heat to 70° C, add 35 g of iron, in small increments until dissolved, filter (Whatman #4 suggested), transfer to flask and dilute to 1000 mL with water. Prepare fresh weekly (very reactive).

8.2.10 3 M HNO_3. Add 188 mL of concentrated HNO_3 to 700 mL of water. Dilute to 10 L with DI water.

8.2.11 2.5 M HNO_3/0.1 M FeS. Add 159 mL of concentrated HNO_3 (sp gr 1.42) to 166 mL of 0.6 M ferrous sulfamate. Dilute to 1 L with water. Prepare fresh daily.

8.2.12 9 M HC1. Add 750 mL of concentrated HCl (sp gr 1.19) to 100 mL of water and dilute to 1 L with water.

8.2.13 6 M HC1. Add 500 mL of concentrated HC1 to 400 mL of water and dilute to 1L with water.

8.2.14 0.02 M HNO_3/0.02 M HF. Add 0.64 mL concentrated HNO_3 and 0.36 mL of concentrated HF to 400 mL of water and dilute to 500 mL with water.

8.2.15 Hydrochloric acid concentrated (12N).

9.0 SAMPLE HANDLING AND PRESERVATION

Liquid Samples

9.1 Samples should be preserved to approximately pH 2 with nitric acid and collected in a plastic bottle.

9.2 Before beginning an analysis the analyst should check the sample pH with a pH strip. If necessary, adjust the pH with nitric acid to a pH=1-2. If the sample was pH adjusted let the sample sit overnight before continuing the batch. Note: some clients must be informed prior to doing this step.

9.3 If the sample has exceeded the hold time the analyst should contact the Group Leader before continuing with the batch.

Soil Samples

9.4 Samples should be collected in a suitable container that will maintain its integrity during transportation. No preservation is required.

10.0 SAMPLE PREPARATION

Soil Sample Preparation

10.1 If not already done, homogenize the sample by performing the GL-EPI-E-A-021, "Preparation of Soils for the Determination of Radionuclides".

10.2 It is recommended that the samples be ashed in a muffle furnace as specified in GL-EPI-E-A-021b, "Soil Sample Ashing for the Determination of Radionuclides".

10.3 A Np aliquot is analyzed with an aggressive acid digestion as described in the following steps.

10.3.1 Weigh an appropriate aliquot (0.5-1.0 grams) of ashed soil sample into a Teflon beaker. Correct the sample mass for losses due to ashing and record the corrected weight on the Que sheet.

10.3.2 Add appropriate tracers and record tracer IDs and tracer volumes on the Que sheet.

10.3.3 Add 10 mL of concentrated nitric acid and 10 mL of 48% HF to each Teflon beaker.

10.3.4 Cover beakers with a Teflon cover and place on hotplate on medium heat for 30 minutes. Remove lids and evaporate solution to dryness.

10.3.5 Add 10 mL of concentrated HCl and 5 mL of 48% HF to each sample, cover with the Teflon lid and heat for 30 minutes on the hotplate. Remove the lid and evaporate to dryness on the hotplate.

10.3.6 Add 10 mL concentrated HCl and 10 mL concentrated nitric acid to each Teflon beaker and evaporate to dryness on a hotplate.

10.3.7 Add 10 mL concentrated HCl and 1 mL saturated boric acid to each sample and evaporate to dryness on a hotplate set on medium heat.

10.3.8 Dissolve the sample residue in 15 mLs of 9 M hydrochloric acid and transfer to a disposable centrifuge tube using DI water as a rinse. Dilute to 25 mls with DI water. Add concentrated ammonium hydroxide until iron hydroxide precipitates, then add 2 mL excess NH4OH. Centrifuge sample and discard supernate.

10.3.9 Wash precipitate with 20 mL of DI water that has been adjusted to pH 10 with ammonium hydroxide. Centrifuge sample and discard supernate.

10.3.10 Dissolve the ppt with 20 mls 3 M HNO_3/1 M AL $(NO_3)_3$, 4 mls 0.6 M ferrous sulfamate, 1 ml ascorbic acid

Aqueous Sample Preparation

10.4 Add an appropriate aliquot of sample to a labeled beaker. Add a certified dpm of an appropriate tracer to each of the samples.

Note: Other sample matrices, such as vegetation, air filters, tissue, etc., are run as outlined in GL-EPI-E-A-026, "Preparation of Special Matrices for the Determination of Radionuclides".

10.5 Add 1 mL of Fe carrier to the sample. This step may be omitted for soils if enough Fe already exists in the sample. This is evident by a rich yellow color in the sample.

10.6 Add concentrated NH4OH until turbidity persists. Then add 5 mL excess. Continue heating for 10 minutes and then allow to settle and cool.

10.7 Decant excess supernate and discard. Collect the remaining precipitate by centrifugation in a 50 mL centrifuge tube and discard the supernate. Slurry the precipitate in 25 mL of DI water. Centrifuge, decant and discard supernate.

10.8 See load solution on NP checklist (Appendix 1).

10.9 Prepare the TEVA resin column by pre-rinsing with 5 mL of 3 M HNO_3.

10.10 Load the sample from step 10.8 onto the column and discard the initial eluant. See NP checklist for column work, separation time and elution.

10.11 Add 100 ul of 500 ppm cerium solution to the samples and swirl.

10.12 Add 3 mL of concentrated HF, swirl and allow to stand for 30 minutes.

10.13 Place a 0.1 um metricel filter on the filter funnel base.

10.14 Rinse the filter and funnel under vacuum with 5 mL of 80% ethanol. With minimum delay, add the sample to the filtering apparatus and rinse the beaker several times into the funnel with type II water. Complete the filtering by adding 5 mL of 80% ethanol.

10.15 Dry the filter under a heat lamp in a labeled petri dish. Label a 29 mm flat planchet with the applicable laboratory number and desired radionuclide. Cover the planchet with double stick tape and carefully place the filter (precipitate side up) on the adhesive. Care should be taken to center the filter and make it as flat as possible on the planchet.

NOTE: care should be taken not to touch the active area of the filter with forceps.

10.16 Place the Np samples in a two inch planchet and cover with aluminum foil. This will inhibit any alpha particles from interfering with the beta count. Count the filter in a gas flow proportional counter to determine the Np yield.

10.17 Count under vacuum on the alpha spectrometer long enough to reach requested MDA. Consult the operating manual for instruction on operating the alpha spectrometer.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to GL-EPI-M-001, "Preparation of Radioactive Standards."

11.2 All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in GL-EPI-M-001, "Preparation of Radioactive Standards," section 19.0.

11.3 Primary standards are kept in the laboratory in a secured cabinet. Secondary, working, standards are kept at the bench area in an enclosed plastic cabinet.

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 For direction on calibration and instrument performance see GL-EPI-I-009, "Micro-VAX 3100 Alpha Spectroscopy System."

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 For analysis and instrument operation see GL-EPI-I-009, "Micro-VAX 3100 Alpha Spectroscopy System."

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 For maintenance of system see GL-EPI-I-010,-"Counting Room Maintenance and Performance Checks."

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 The instrument will report sample pCi/unit according to the following equation:

$$\text{pCi / unit} = \frac{S_{cpm} - B_{cpm}}{2.22 * E * V * A * \text{decay} * R}$$

15.2 Counting uncertainty is propagated according to the following equation:

$$\text{pCi / unit} = Ac * 1.96 \sqrt{\left(\frac{ef\_er}{E}\right)^2 + \left(\frac{pk\_er}{pk}\right)^2 + \left(\frac{ab\_er}{A}\right)^2 + \left(\frac{sy}{100}\right)^2 + (dk)^2}$$

$$dk = \frac{T_{1/2}err}{T_{1/2}} * \left| \frac{\lambda Tr}{1 - e^{-\lambda Tr}} - \lambda * \left| T_c + T_r \right| - 1 \right|$$

And where:

Scpm	=	Sample counts per minute
Bcpm	=	Background counts per minute
E	=	Counting efficiency
V	=	Volume in liters,g,cfm, etc.
A	=	Isotopic abundance
ef_er	=	1 sigma efficiency error
pk_er	=	1 sigma peak error
ab_er	=	1 sigma isotopic abundance error
sy	=	1 sigma sytematic error
pk	=	peak area
Tc	=	Sample count time in minutes
Td	=	Time interval for radioactive decay
Tr	=	Elapsed real time in minutes
Tcpm	=	Tracer counts per minute
Tdpm	=	Tracer known disintegrations per minute
Ac	=	Sample calculated activity
T1/2	=	Isotopic half life
T1/2err	=	Isotopic half life error
λ	=	Isotopic decay constant
e	=	exponential function
ln	=	natural log function

15.3 The minimum detectable activity (MDA) is calculated according to the following equation:

$$MDA(pCi / unit) = \frac{2.71 + 4.65 * \sqrt{B_{cpm}} * T_c}{(2.22 * E * V * R * A * decay * T_c)}$$

where:

$$decay = \frac{1}{\left(e^{(-\ln 2)} * \frac{T_d}{T_{1/2}}\right)}$$

$$R = \frac{T_{cpm} - B_{cpm}}{T_{dpm} * E}$$

15.4 Record the following information on the alpha que sheet: preparation date, analysts initials, spike isotope, spike code, spike volume, LCS isotope, LCS code, LCS volume, nominal concentration LCS, and nominal concentration MS. For each sample record the detector number, sample mass, sample date, and sample time.

16.0 QUALITY CONTROL REQUIREMENTS

NOTE: Some clients may specify more or less stringent requirements.

16.1 Analyst and Method Verification

16.1.1 Refer to GL-EPI-D-003, "Analyst and Analytical Methods Validation Procedures," for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank will accompany each batch of 20 or less samples. The reported value should be less than or equal to the CRDL for all target isotopes.

16.2.2 A matrix spike (MS) should be run with every batch of 20 samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\%\text{Rec} = \frac{\text{spike(pCi/g)} - \text{sample(pCi/g)}}{\text{spikedamount(pCi/g)}} * 100$$

16.2.3 A sample duplicate should be run with every batch of 20 or less samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$\text{RPD} = \frac{\text{highsample(pCi/g)} - \text{lowsample(pCi/g)}}{\text{Average (pCi/g)}} * 100$$

16.2.4 A laboratory control spike (LCS) should be run with every batch of 20 samples or less. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\text{LCS} = \frac{\text{observed\_ pCi / unit}}{\text{known\_ pCi / unit}} * 100$$

(NOTE: Some clients may specify more or less stringent requirements.)

16.3 Actions required if the Quality Control Requirements are not met

16.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a non conformance report as outlined in GL-QS-E-004, "Nonconformance Identification Control, Documentation, Reporting and Dispositioning."

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

17.1 The analyst turns in the raw data, review checklist, que sheet, spreadsheet, data report, NCR's (if applicable), and other appropriate information in a batch to the Report Specialist/Data Validator.

17.2 Analyst's data go through the following process of review:

17.2.1 The first level of review is the analyst review. The analyst will perform the following review procedure:

17.2.1.1 Visually check the que sheet, spreadsheet, raw data and data report to make sure the information has been transcribed correctly.

17.2.1.3 Check to see that the required detection limit (RDL) is met if required.

17.2.1.4 Complete the batch checklist and check the special requirements page.

17.2.2 The second level review is performed by the Data Report Specialist/Data Validator, who reviews the batch checklist, checks requested and non-requested hits, and reviews the transcription.

17.3 After the review process is complete, the data is transmitted from the laboratory personnel to the reporting personnel as outlined in GL-EPI-D-003, "Data Review and Validation Procedures."

18.0 RECORDS MANAGEMENT

18.1 Each analysis that is performed on the instrument is documented in the run log according to GL-LB-E-009.

18.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

19.1 Radioactive samples and material shall be handled and disposed of as outlined in GL-EPI-S-011, "Radioactive Waste Handling Procedures."

20.0 REFERENCES

20.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

20.2 EML Procedures Manual HASL-300 1982.

20.3 Analytical Chemistry. Rapid Determination of Th-230 in Mill Tailings by alpha spectroscopy. UNC Geotech, Grand Junction Projects Office. Steve Donivan, Mark Hollenbach, and Mary Costello. Vol 59, No 21 1987.

20.4 Los Alamos Health and Environmental Chemistry: Analytical Techniques. LA-10300-M Vol. 1 Sept. 1987.

APPENDIX 1

NEPTUNIUM

Use a 2.5 cm3 column, TEVA resin

LOAD SOLUTION

_____20 mls 3 M HNO3/1M A1(NO3)3 *(IT IS OK FOR THE SAMPLE TO SIT IN THIS OVERNIGHT)*
_____40 mls 0.6 M Ferrous Sulfamate, swirl *(MAKE WEEKLY, SEE PROCEDURE FOR RECEIPT)*
_____1 ml 1 M Ascorbic Acid, swirl, wait 5 minutes before loading

COLUMN WORK

_____5 mls 3M HNO3 (conditioning)
_____Load Solution onto column
____5 mls 2.5 M HNO3/0.1 M Ferrous Sulfamate *(THIS NEEDS TO MADE DAILY)*
159 mls HNO3, 166 mls 0.6 M Ferrous Sulfamate, dilute to 1 liter with DI
____10 mls 2.5 MHNO3/0.1 M Ferrous Sulfamate (rinse)
_____10 mls 2.5 MHNO3/0.1 M Ferrous Sulfamate (rinse)
_____10 mls 2.5 MHNO3/0.1 M Ferrous Sulfamate (rinse)
_____10 mls 9 M HC1
_____10 mls 9 M HC1
_____5 mls 6 M HC1

NP Elution *(NOTE TIME AND DATE)*
CATCH THIS IN A CLEAN CENTRIFUGE TUBE
_____10 mls 0.02 M HNO3/0.02 M HF

PRECIPITATION

_____0.1 ml Ce (500 mg), swirl
_____3 mls HF, swirl, wait 20 minutes
_____filter

STANDARD OPERATING PROCEDURE

FOR

DETERMINATION OF CHLORINE-36

IN AQUEOUS MATRICES

UNCONTROLLED DOCUMENT

(GL-EPI-E-A-033 Revision)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Process Owner	8/30/99 (Sign and Date)	Tim P. Winters (Print Name)
Technical Review	8/26/99 (Sign and Date)	Richard M Kinney (Print Name)
Quality Review	8/31/99 (Sign and Date)	Gene Bost (Print Name)
Approval and Authorization (Sign and Date)	8/30/99	James B. Westmoreland (Print Name)

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TABLE OF CONTENTS

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1.0 STANDARD OPERATING PROCEDURE FOR DETERMINATION OF CHLORINE-36 IN AQUEOUS MATRICES

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for chlorine-36 in aqueous matrices.

2.2 Aqueous samples are equilibrated with potassium chloride carrier and silver nitrate and adjusted to pH > 9 with concentrated ammonium hydroxide. Samples are adjusted to pH < 4 with concentrated nitric acid to precipitate silver chloride. The chloride precipitate is filtered through a tared filter, dried in an oven, and weighed to determine chemical yield. Samples are counted on a gas flow proportional counting system to determine beta activity.

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for chlorine-36 is 100 pCi/L.

3.2 Method Precision: Typical relative percent differences (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value. Analyst training records are kept in the human resource department.

4.0 DEFINITIONS

4.1 National Institute of Standards and Technology (NIST). For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.2 Type II water: Deionized water.

4.3 LIMS: Laboratory Information Management System – The database system used to store and report data.

5.0 METHOD VARIATIONS

5.1 Some variations in procedure may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group

Leader or Senior Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard, and exposure to these chemicals must be reduced to the lowest level possible. General Engineering Laboratories, Inc., (GEL), maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the library and in the laboratory, respectively. Individual sample MSDS forms provided by the clients are kept in Login.

6.3 Gloves are required when handling the chemicals in this procedure.

6.4 The handling of radioactive samples is outlined in GL-EPI-S-004 "Handling of Radioactive Samples."

6.5 Refer to GL-EPI-S-011, "Laboratory Waste Disposal and Emergency Instructions" for instructions on how materials are disposed.

6.6 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the group leader possible prior to carrying out the rest of the procedure.

6.7 When handling biological samples, protect the hands and forearms by wearing gloves and a laboratory coat to avoid contact of the biological material with the skin. Protect the eyes by wearing safety glasses and, if desired, a splash shield.

6.8 If cutting of meats or other tissue with cutlery is required, the analyst will wear a cutting glove of mesh steel over disposable gloves to avoid cuts that could infect the analyst with pathogens.

6.9 Any procedure that volatilizes biological substances, such as drying or ashing, must be conducted in a hood or other suitable containment device.

6.10 Decontamination of work surfaces exposed to biological samples is performed by wiping the work area with a diluted (1:10) bleach solution following analytical operations.

6.11 Exterior protective clothing shall be removed prior to exiting the bioassay sample preparation area in order to prevent the inadvertent spread of biohazards to the rest of the laboratory.

6.12 Hands will be washed with an antibacterial soap directly after handling biological samples.

7.0 INTERFERENCES

7.1 Chloride is abundantly present in natural matrices. Stable chloride present in the sample matrix may bias chemical recovery results. Steps are included to account for the bias resultant from stable chloride that may be present in the sample matrix.

7.2 Radioactive silver and/or iodine present in the sample will bias the analytical results.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 Vacuum pump and filtration apparatus (47 mm)
8.1.2 47 mm filters with 0.450 um pore size

8.1.3 Stainless steel planchettes, 2-inch diameter

8.1.4 Double-stick tape

8.1.5 Stainless steel tweezers

8.1.6 Polypropylene centrifuge tubes, 50 mL

8.1.7 Sample drying apparatus

8.1.8 Glass beakers, various sizes

8.2 Reagents, Chemicals and Standards

8.2.1 Ammonium hydroxide, concentrated (14 N)

8.2.2 Nitric acid (HNO3), concentrated (16 N)

8.2.3 Silver chloride solution (1 N). Dissolve 17 g solid silver nitrate in DI water and dilute to 100 mL with DI.

8.2.4 Potassium chloride solution (20 mg Cl/mL). Dissolve 10.51 g solid potassium chloride in DI water and dilute to 250 mL with DI water.

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 Aqueous samples should **not** be preserved and are received in plastic bottles.

9.2 If the sample has exceeded the hold time, the analyst should contact the group leader before continuing with the batch.

10.0 SAMPLE PREPARATION

10.1 For large (> 30 mL) water samples, label an appropriate size glass beaker with each sample number to be processed. For small (≤ 30 mL) water samples label a 50 mL disposable centrifuge tube.

10.2 Transfer an appropriate volume of sample to a labeled sample container. Record the sample volume to be analyzed on the applicable lab sheet.

10.3 Add 1 mL potassium chloride carrier (20 mg Cl/mL) and 1 mL of 1 M silver nitrate solution to each sample and stir with a glass rod to ensure complete mixing.

10.4 Adjust the sample to approximately pH 9 by adding concentrated ammonium hydroxide dropwise. Precipitate silver chloride by adding concentrated nitric acid to each sample to obtain a pH of approximately 4.

10.5 Allow the samples to cool slightly. Centrifuge each sample and discard the supernate to waste. Suspend the precipitate in 15 mL of DI water and add concentrated ammonium hydroxide dropwise to dissolve. Reprecipitate the silver chloride by adding concentrated nitric acid to obtain a pH of 4.

10.6 Allow the samples to cool slightly. Centrifuge each sample and discard the supernate to waste.

10.7 Label and weigh a 47 mm DM-450® Tuffryn filter for each sample. Slurry the chloride precipitate in DI water and filter though the tared filter, rinsing the final precipitate out of the tube with a fine jet of water.

10.8 Dry the filters in an oven at 100°C. Move the filters to a desiccator for 10 minutes and weigh. Record the weight on the queue sheet. Mount the filters on the bottom of a two-inch stainless steel counting planchette using double-stick tape.

10.9 Dry the filters in the low background gas flow alpha/beta counter for a duration that will meet the CRDL and uncertainty.

10.10 Enter all necessary data into the calculation spreadsheet. Update method control charts and enter final results into the LIMS.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to GL-EPI-M-001, "Preparation of Radioactive Standards."

11.2 All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in GL-EPI-M-001, "Preparation of Radioactive Standards," section 19.0

11.3 Primary standards are kept in the laboratory in a secured cabinet. Secondary, working, standards are kept at the bench area in an enclosed plastic cabinet.

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 Standardization of Cl carrier

12.1.1 Pipet four 5.0 mL aliquots of Cl carrier (nom. Conc. 20 mg Cl/mL) into tared 50 mL centrifuge tubes. Record the weight of each carrier aliquot.

12.1.2 Add 10 mL of DI water and 5 mL of 1 N silver nitrate solution to each centrifuge tube.

12.1.3 If a precipitate forms, add concentrated ammonium hydroxide dropwise until the precipitate dissolves. Add concentrated nitric acid dropwise to obtain a pH of 2. Allow the precipitate to settle, then centrifuge. Discard the supernate to waste.

12.1.4 Wash the chloride precipitate with 20 mL of DI water and centrifuge. Discard the supernate to waste.

12.1.5 Dry the precipitate in each c-tube at 100°C in an oven for 30 minutes. Remove the tubes from the oven and allow to cool. Reweigh each tube to determine the weight of the precipitate.

12.1.6 Calculate the standard weight of Cl carrier in mg/mL and mg/g of solution as follows to obtain the standard weight for 1.00 mL or 1.00 gram of Cl carrier solution:

$$Cl.\ Std.\ Wt.\ Mg\ /\ mL\ of\ carrier\ solution = \frac{Net.\ Wt.,\ mg\ of\ AgCl}{5.00}$$

$$Cl.\ Std.\ Wt.\ Mg\ /\ mL\ of\ carrier\ solution = \frac{Net.\ Wt.,\ mg\ of\ AgCl}{Net.\ Wt.\ G\ of\ 5-mL\ aliquot}$$

12.1.7 Acceptable precision is a relative standard deviation of 0.5%. Record the standardization results in the lab notebook. Label the carrier solution with the standardization results. The solution should be restandardized every six months.

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 Refer to GL-EPI-E-I006, "LB4110 Gross Alpha/Beta Counter Operating Instructions" for instrumentation operation procedures.

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 For maintenance of system see GL-EPI-I-010, "Counting Room Maintenance and Performance Checks."

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

Refer to client-specific Statement of Works for specific instructions regarding calculations, otherwise the following calculations should be used.

15.1 The spreadsheet will calculate Cl-36 in pCi/unit according to the following equation:

$$result\ (pCi\ /\ unit) = \frac{(S_{cpm} - B_{cpm})}{2.22 * E * R * V}$$

15.2 Counting uncertainty is calculated according to the following equation:

$$error\ (pCi\ /\ unit) = \frac{1.96\sqrt{\frac{S_{cpm}}{T_c} + \frac{B_{cpm}}{T_{cb}}}}{2.22 * E * R * V}$$

15.3 The minimum detectable activity (MDA) is calculated according to the following equation:

$$MDA(pCi/unit) = \frac{\left(2.71 + 4.66\sqrt{B_{CPM} * T_C}\right)}{2.22 * E * V * R * T_C}$$

Where:
T_c = sample count duration
T_{cb} = background count duration
B_{CPM} = background counts per minute
S_{CPM} = sample counts per minute
E = efficiency of counting
V = sample volume
R = chemical recovery factor

16.0 QUALITY CONTROL REQUIREMENTS

16.1 Analyst and Method Verification

16.1.1 Refer to GL-EPI-D-022, "Analyst and Analytical Methods Validation Procedures" for instructions concerning the validation of analysts and analytical methods.

16.2 Method-Specific Quality Control Requirements

16.2.1 A method blank will accompany each batch of 20 or fewer samples. The reported value should be less than or equal to the CRDL for all target isotopes.

16.2.2 A matrix spike (MS) should be run with every batch of 20 samples. The recovery of the spike should fall between 75% and 125%. The recovery is calculated as follows:

$$\%rec = \frac{spike\,(pCi/unit) - sample\,(pCi/unit)}{spiked\ amount\,(pCi/unit)} * 100$$

16.2.3 A sample duplicate should be run with every batch of 20 or fewer samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20%. The RPD is calculated as follows:

$$RPD = \frac{high\ sample\,(pCi/unit) - low\ sample\,(pCi/unit)}{Average\,(pCi/unit)} * 100$$

16.2.4 A laboratory control spike (LCS) should be run with every batch of 20 samples or fewer. The recovery of the spike should fall between 75% and 125%. The recovery is calculated as follows:

$$LCS = \frac{observed\_pCi/unit}{known\_pCi/unit} * 100$$

16.3 Actions Required if the Quality Control Requirements Are Not Met

16.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the group leader and initiate a nonconformance report as outlined in GL-QS-E-004, "Nonconformance Identification Control, Documentation, Reporting and Dispositioning."

17.0 DATA REVIEW, APPROVAL, AND TRANSMITTAL

17.1 The analyst turns in the raw data, review checklist, que sheet, spreadsheet, data report, NCRs (if applicable), and other appropriate information in a batch to the data reviewer.

17.2 Analyst's data go through the following process of review:

17.2.1 The first level of review is the analyst review. The analyst will perform the following review procedure:

17.2.1.1 Visually check the queue sheet, spreadsheet, raw data and data report to make sure the information has been transcribed correctly.

17.2.1.2 Check to see that the required detection limit (RDL) is met if required.

17.2.1.3 Complete the ~~batch~~ checklist and review the special requirements summary report.

17.2.2 The second level review is performed by the Data Validator or trained designee who reviews the batch checklist, checks requested and nonrequested hits, and reviews the data for transcription errors.

17.3 After the review process is complete, the data is transmitted from the laboratory personnel to the reporting personnel as outlined in GL-EPI-D-003, "Data Review and Validation Procedures."

18.0 RECORDS MANAGEMENT

18.1 Each analysis that is performed on the instrument is documented in the run log according to GL-LB-E009.

18.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

19.0 LABORATORY WASTE HANDLING AND WASTE DISPOSAL

19.1 Radioactive samples and material shall be handled and disposed of as outlined in GL-EPI-S-011, "Laboratory Waste Disposal and Emergency Instructions."

20.0 REFERENCES

20.1 National Academy of Sciences, National Research Council, Nuclear Science Series, "The Radiochemistry of Silver," D.N. Sunderman and C.W. Townley, November 1961.

STANDARD OPERATING PROCEDURE

FOR

THE ISOTOPIC DETERMINATION

OF

PLUTONIUM –241

(GL-EPI-A-035 Revision 0)

UNCONTROLLED DOCUMENT

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

Robert D Tiuum 6/22/99	Process Owner (sign & date)	Robert D Tiuum (print name)
Richard M. Kinney 6/29/99	Technical Review (sign & date)	Richard M. Kinney (print name)
Genie Bost 6/30/99	Quality Review (sign & date)	Genie G. Bost (print name)
James B Wilsmore 6/30/99	Approval and Authorization (sign & date)	James B. Weston (print name)

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TABLE OF CONTENTS

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1.0 Title: Standard Operating Procedure for the Isotopic Determination Plutonium - 241.

2.0 Method Objective, Purpose, Code, and Summary

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic plutonium-241 in a variety of matrices.

2.2 A sample is leached (if necessary) and aliquoted. Transuranic elements are scavenged by coprecipitation with iron hydroxide. The precipitate is dissolved and separation of elements is accomplished through extraction chromatography and ion exchange resins. The elution is aliquoted into two equal parts. One to be prepared for liquid scintillation and the other for alpec spectroscopy detectors.

2.3 General Engineering Laboratories (GEL) utilizes methods that are derived from established sources. This method is based on the source method from DOE EML Methods Manual HASL 300 E-U-04 and uses similar principles of radiochemical separation and counting. This method is also very similar in concept to the source method from the DOE Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP800: "Sequential Separation of Americium and Plutonium by Extraction Chromatography."

2.4 This revision combines several related procedures. The method for the determination of isotopic Plutonium originally in SOP "The Isotopic Determination of Thorium, Plutonium and Neptunium" (GL-EPI-E-A012) has been added to this procedure. The following standards operating procedures are canceled without replacement having served their purpose:

2.4.1 SOP GL-EPI-E-A012b, "The Determination of Isotopic Neptunium and Plutonium in Soil and Vegetation."

2.4.3 SOP GL-EPI-E-A012d, "The Determination of Isotopic Thorium, Plutonium and Neptunium and Plutonium in Air Filters."

3.0 Method Applicability

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for Pu-241 is 10 pCi/L or 10 pCi/g. For this procedure, MDAs as low as 0.5 pCi/L or 0.5 pCi/g for Pu-241 can be obtained by appropriately increasing the sample size and/or count time.

3.2 Method Precision: Typical relative percent difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value. Analyst training records are kept on hand in the human resource department.

4.0 Definitions

4.1 National Institute of Standards and Technology (NIST). For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.2 Type II water: Deionized water.

4.3 LIMS: Laboratory Information Management System. The database system used to store and report data.

5.0 Method Variations

5.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Senior Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 Safety Precautions and Warnings

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the library and in the laboratory, respectively. Individual sample MSDS forms provided by the clients are kept in Login."

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are nitrile gloves for concentrated acids and bases,

and potassium ferricyanide in neat form. Work under a hood when using concentrated acids and bases.

6.4 The handling of radioactive samples is outlined in SOP "Handling of Radioactive Samples" (GL-EPI-E-S004). General guidelines include:

6.4.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

6.4.2 A plastic apron may be worn over the lab coat for added protection from contamination when working with radioactive samples.

6.4.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.4.4 Prohibit admittance to immediate work area.

6.4.5 Post signs indicating radioactive samples are in the area.

6.4.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in the radiochemistry laboratory.

6.4.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management."

6.5 Refer to SOP "Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011) for instructions on how materials are disposed.

6.6 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the Group Leader prior to carrying out the rest of the procedure.

6.7 When handling biological samples protect the hands and forearms by wearing gloves and a laboratory coat to avoid contact of the biological material with the skin. Protect the eyes by wearing safety glasses and if desired a splash shield.

6.8 If cutting of meats or other tissue with cutlery is required, the analyst will wear a cutting glove of mesh steel over disposable gloves to avoid cuts which could infect the analyst with pathogens.

6.9 Any procedure which volatilizes biological substances such as drying or ashing, must be conducted in a hood or other suitable containment device.

6.10 Decontamination of work surfaces exposed to biological samples is performed by wiping the work area with a diluted (1:10) bleach solution and water as soon as possible following analytical operations.

6.11 Exterior protective clothing shall be removed prior to exiting the bioassay sample preparation area in order to prevent the inadvertent spread of biohazards to the rest of the laboratory.

6.12 Hands will be washed with an antibacterial soap directly after handling biological samples.

7.0 Apparatus, Materials, Reagents, Equipment, and Instrumentation

7.1 Ancillary Equipment

7.1.1 Silicon surface barrier detectors with associated electronics, vacuum chambers, and data reduction capabilities

7.1.2 Vacuum pump and filtration apparatus (25 mm)

7.1.3 Gelman metricel 25 mm filters with 0.1 µm pore size

7.1.4 Gelman polypropylene 25 mm support filter

7.1.5 Stainless steel disks. 29 mm diameter

7.1.6 Stainless steel tweezers

7.1.7 Polypropylene centrifuge tube (50 mL)

7.1.8 Sample drying and ashing apparatus

7.1.9 Sample homogenization apparatus

7.1.10 AG1X8 anion exchange resin 100-200 mesh

7.2 Reagents, Chemicals and Standards

7.2.1 Ammonium hydroxide concentrated (14 N)

7.2.2 Cerium(III)nitrate hexahydrate. Dissolve 0.155g in 100 mL DI water. (500 µg Ce/mL).

NOTE: This is normally purchased in the correct concentration from an approved vendor such as High Purity Standards.

7.2.3 Ethyl alcohol (80%). Dilute 400 mL ethanol to 500 mL with DI water.

7.2.4 Hydrochloric acid 0.1N. Dilute 8.3 mL of concentrated HCl to 1 liter with DI water.

7.2.5 Hydrofluoric acid concentrated (48%)

7.2.6 Ion exchange resin. Bio-Rad AG 1X8, chloride form, 100-200 mesh

7.2.7 Iron Carrier. 10 mg/mL

7.2.8 NIST traceable standards: Pu-242, Pu-239, Pu-238, Pu-236

7.2.9 Nitric acid concentrated 16M

7.2.10 8M HNO_3. Dilute500mL concentrated HNO_3 with 500 mL DI water.

7.2.11 9M HCl. Dilute 750mL concentrated HCl with 500 mL DI water

7.2.12 1M Sodium Nitrate. Dilute 6.9g $NaNO_2$ with 100 mL DI water.

7.2.13 20% Sodium Sulfate. Dilute 20g Sodium Sulfate with 80 mL DI water.

7.2.14 Phosphoric Acid (H_3PO_4) (85.9%

7.2.15 Ready Safe-Liquid Scintillation Cocktail

7.2.16 25% Hydrazine dihydrochloride. Dilute 25g hydrazine dihidrochloride with 75 mL DI water.

7.2.17 6 mL HCL. Dilute 500 mL concentrated HCl with 500 mL DI water.

8.0 Sample Handling and Preservation

8.1 Samples should be preserved to approximately pH 2 with nitric acid and collected in a plastic bottle.

8.2 Before beginning an analysis the analyst should check the sample pH with a pH strip. If necessary, adjust the pH with nitric acid to a pH=1-2. If the sample was pH adjusted let the sample sit overnight before continuing the batch.

8.3 If the sample has exceeded the hold time the analyst should contact the Group Leader before continuing with the batch.

8.4 Soil samples require no preservation and may be shipped in any suitable container.

9.0 Sample Preparation

Soil Sample Preparation

9.1 If not already done, homogenize the sample by performing SOP "Preparation of Soils for the Determination of Radionuclides" (GL-EPI-E-A021).

9.2 It is recommended that the samples be ashed in a muffle furnace as specified in SOP "Soil Sample Ashing for the Determination of Radionuclides" (GL-EPI-E-A021b).

9.3 A Pu-241 aliquot is analyzed with an aggressive acid leach as described in the following steps.

9.3.1 Place the sample in a beaker and add 30 mL 6 M HCl per gram with a minimum of 10 mL. Add the appropriate tracers as described in section 10.6.

9.3.2 Heat the samples and cover with a watch glass, allow to leach for a minimum of 2 hours. Agitate the sample periodically to enhance the leaching process.

9.3.3 Allow the sample to partially cool and transfer to a centrifuge tube. Centrifuge to separate the solid and leached portions of the sample. Decant the leachate to a clean labeled beaker and rinse the solid phase with DI water. Centrifuge the sample, combine the leachates and dilute with DI water. Proceed to section 10.7.

Aqueous Sample Preparation

9.5 Add an appropriate aliquot of sample to a labeled beaker. Add a certified dpm of the following tracers to each of the samples:

- For isotopic plutonium, Pu-236 is normally used

9.6 Add 1 mL of iron carrier (10 mG/mL).

NOTE: For soil samples iron carrier may not be needed.

9.7 Bring to a slight boil and add concentrated NH_4OH until turbidity persists, or pH>9. Heat to near boiling for 10 minutes and then allow to settle and cool.

9.8 Decant excess supernate and discard. Collect the remaining precipitate by centrifugation in a 50 mL centrifuge tube and discard the supernate.

NOTE: Exercise care in this step because finely divided material which contains the actinides may also be present in addition to the large iron hydroxide flocks.

9.10 Dissolve the precipitate in 10 mL of 8 M HNO_3 and 0.1 mL 1 M $NaNO_2$ solution.

9.11 Slurry AG 1x8 anion resin (Cl form 100-200 Mesh) in a squirt bottle with DI water. Transfer the resin to a small column to obtain a settled resin bed of 2.5 mL.

9.11 Condition the column with 20 mL of 8 M HNO_3.

9.12 Pass the sample (in 8 m $HNO_3/NaNO_2$) through the column.

9.13 Rinse the column with 20 mL 8 M HNO_3.

9.14 Elute Th w/40 mL 9M HCl

9.15 Elute Pu w/20 mL 0.1 M HCl

9.16 Split in Half

9.16.1 Pu-242 Portion

9.16.1.1 Put in labeled glass scintillation vial.

9.16.1.2 Add 0.2 mL 20% Sodium Sulfate

9.16.1.3 Evaporate to dryness.

9.16.1.4 Add 2 mL DI water.

9.16.1.5 When ppt. dissolves, add 0.5 mL H_3PO_4,, mix well.

9.16.1.6 Add 17 mL Ready Safe™.

9.16.1.7 Count on LSC.

9.16.2 Alpha-spec portion

9.16.2.1 Add 3-4 drops hydrazine dihyrdochloride, swirl, let stand 10 min.

9.16.2.2 Add 0.1 mL Ce, swirl.

9.16.2.3 Add 1 mL HF, swirl and let stand 30 min before mounting.

9.17 Place a 0.1 µm metricel filter on the filter funnel base.

9.18 Rinse the filter and funnel under vacuum with 5 mL of 80% ethanol. With minimum delay, add the sample to the filtering apparatus and rinse the beaker several times into the funnel with type II water. Complete the filtering by adding 5 mL of 80% ethanol.

9.19 Dry the filter under a heat lamp in a labeled petri dish. Label a 29 mm flat planchet with the applicable laboratory number and desired radionuclide. Care should be taken to center the filter and make it as flat as possible on the planchet.

NOTE: Care should be taken not to touch the active area of the filter with forceps.

9.20 Count under vacuum on the alpha spectrometer long enough to reach requested MDA. Consult the operating manual for instruction on operating the alpha spectrometer.

9.21 Beckman LS 6500/6600 operating procedure (GL-EPI-EI-004).

10.0 Preparation of Standard Solutions and Quality Control Standards

10.1 Refer to SOP "Preparation of Radioactive Standards" (GL-EPI-E-M001).

10.2 All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in SOP "Preparation of Radioactive Standards" (GL-EPI-E-M001), Section 19.0.

10.3 Primary standards are kept in the laboratory in a secured cabinet. Secondary, working, standards are kept at the bench area in an enclosed plastic cabinet.

11.0 Instrument Calibration and Performance

11.1 For direction on calibration and instrument performance see SOP "Micro-VAX 4000 Alpha Spectroscopy System" (GL-EPI-E-I009).

11.2 Beckman LS6500/6600 operating procedure (GL-EPI-EI-004).

12.0 Analysis and Instrument Operation

12.0 For analysis and instrument operation see SOP "Micro-VAX 4000 Alpha Spectroscopy System" (GL-EPI-E-I009).

12.1 Beckman LS 6500/6600 (GL-EPI-EI-004) operating procedure.

13.0 Equipment and Instrument Maintenance

13.1 For maintenance of system see "Counting Room Maintenance and Performance Checks" (GL-EPI-E-I010).

14.0 Data Recording, Calculation, and Reduction Methods

14.1 The instrument will report sample pCi/unit according to the following equation:

14.2 Counting uncertainty is propagated according to the following equation:

$$\text{pCi / unit} = Ac * 1.96\sqrt{\left(\frac{ef\_er}{E}\right)^2 + \left(\frac{pk\_er}{pk}\right)^2 + \left(\frac{ab\_er}{A}\right)^2 + \left(\frac{sy}{100}\right)^2 + (dk)^2}$$

14.3 The minimum detectable activity (MDA) is calculated according to the following equation:

$$\text{MDA(pCi / unit)} = \frac{2.71 + 4.65 * \sqrt{B_{cpm} * T_c}}{(2.22 * E * V * R * A * decay * T_c)}$$

where:

$$decay = \frac{1}{e^{\left(\frac{-\ln(2)T_d}{T_{1/2}}\right)}}$$

$$R = \frac{T_{cpm} - B_{cpm}}{T_{dpm} * E}$$

$$dk = \frac{T_{v2}err}{T_{v2}} * \left(\frac{\lambda Tr}{1 - e^{-\lambda Tr}} - \lambda\left(T_c + T_r\right) - 1\right)$$

And where:

Scpm	=	Sample counts per minute
Bcpm	=	Background counts per minute
E	=	Counting efficiency (decimal form)
V	=	Volume in liters,g,cfm, etc.
A	=	Isotopic abundance (decimal form)
ef_er	=	1 sigma efficiency error (decimal form)
pk_er	=	1 sigma peak error
ab_er	=	1 sigma isotopic abundance error (decimal form)
sy	=	1 sigma systematic error
pk	=	peak area
Tc	=	Sample count time in minutes

Td	=	Time interval for radioactive decay
Tr	=	Elapsed real time in minutes
Tcpm	=	Tracer counts per minute
Tdpm	=	Tracer known disintegrations per minute
Ac	=	Sample calculated activity
T1/2	=	Isotopic half life
T1/2err	=	Isotopic half life error
•	=	Isotopic decay constant
e	=	exponential function
ln	=	natural log function

14.4 Record the following information on the alpha que sheet: preparation date, analysts initials, spike isotope, spike code, spike volume, LCS isotope, LCS code, LCS volume, nominal concentration LCS, and nominal concentration MS. For each sample record the detector number, sample mass, sample date, and sample time.

15.0 Quality Control Requirements

15.1 Analyst and Method Verification

15.1.1 Refer to SOP "Analyst and Analytical Methods Validation Procedures" GL-EPI-E-D002) for instructions concerning the validation of analysts and analytical methods.

15.2 Method Specific Quality Control Requirements

15.2.1 A method blank will accompany each batch of 20 or less samples. The reported value should be less than or equal to the CRDL for all target isotopes.

15.2.2 A matrix spike (MS) should be run with every batch of 20 samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\%\text{Rec} = \frac{\text{spike(pCi/ unit)} - \text{sample(pCi/ unit)}}{\text{spiked amount(pCi/ unit)}} * 100$$

15.2.3 A sample duplicate should be run with every batch of 20 or less samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$\text{RPD} = \frac{\text{high sample(pCi / unit)} - \text{low sample(pCi / unit)}}{\text{Average (pCi / unit)}} * 100$$

15.2.4 A laboratory control spike (LCS) should be run with every batch of 20 samples or less. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\text{LCS} = \frac{\text{observed\_pCi / unit}}{\text{known\_pCi / unit}} * 100$$

15.3 Actions Required if the Quality Control Requirements Are Not Met

15.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the Group Leader and initiate a non-conformance report as outlined GEL SOP "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

16.0 Data Review, Approval, and Transmittal

16.1 Refer to EPI SOP "Data Review and Validation Procedures" (D-003) for instructions concerning the data review process, approval, and transmittal.

17.0 Records Management

17.1 Each analysis that is performed on the instrument is documented in the run log according to GEL SOP "Run Logs" (GL-LB-E-009).

17.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

18.0 Laboratory Waste Handling And Waste Disposal

18.1 Radioactive samples and material shall be handled and disposed of as outlined in SOP "Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011).

19.0 References

19.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

19.2 EML Procedures Manual HASL-300, 1982.

19.3 Analytical Chemistry. Rapid Determination of Th-230 in Mill Tailings by alpha spectroscopy. UNC Geotech, Grand Junction Projects Office. Steve Donivan, Mark Hollenbach, and Mary Costello. Vol. 59, No. 21, 1987.

19.4 Los Alamos Health and Environmental Chemistry: Analytical Techniques. LA-10300-M Vol. 1, September 1987.

19.5 Special thanks to Dr. Bill Burnett and his associates for assistance in developing this method at Florida State University.

STANDARD OPERATING PROCEDURE

FOR

THE ISOTOPIC DETERMINATION OF AMERICUM, CURIUM, AND PLUTONIUM IN LARGE SOIL SAMPLES

(GL-EPI-A-036 Revision 0)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Process Owner	9/10/99 (Sign & Date)	JENNIFER A LABBE (Print Name)
Technical Review	9/10/99 (Sign & Date)	Richard M. Kinney (Print Name)
Quality Review	9/10/99 (Sign & Date)	Robert L. Pullano (Print Name)
Approval and Authorization	(Sign & Date)	James B Westmoreland (Print Name)

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TABLE OF CONTENTS

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1.0 Title: Standard Operating Procedure for the Isotopic Determination of Americum, Curium and Plutonium in Large Soil Samples.

2.0 Method Objective, Purpose, Code, and Summary

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic americum, curium and plutonium in 5 – 10 gram soil samples. This method also gives specific guidance on determining U-232 and Am-243, which are typically used as isotopic tracers

2.2 Plutonium and americum are leached from soil samples using a combination of hydrochloric acid and nitric acid. Sample leachates are evaporated to dryness and re-dissolved in column load solution. Plutonium is separated using ion-exchange chemistry. Americum is separated using extraction chromatography. The purified elements are then prepared for the measurement of radioactive isotopes by coprecipitation with cerium fluoride. The cerium fluoride precipitate is trapped on a filter, mounted on a stainless steel disk and placed in a partially evacuated chamber for measurement of isotopic alpha emission.

2.3 General Engineering Laboratories (GEL) utilizes methods that are derived from established sources. This method is based on the source method from DOE EML Methods Manual HASL 300 E-U-04 and uses similar principles of radiochemical separation and counting. This method is also very similar in concept to the source method from the DOE Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP800: "Sequential Separation of Americium and Plutonium by Extraction Chromatography."

3.0 Method Applicability

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for U/Am/Cm is 1 pCi/L or 0.1 pCi/g. For this procedure, MDAs as low as 0.002 pCi/g can be obtained by appropriately increasing the sample size and/or count time.

3.2 Method Precision: Typical relative percent difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value. Analyst training records are kept on hand in the human resource department.

4.0 Definitions

4.1 National Institute of Standards and Technology (NIST). For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.2 Type II water: Deionized water.

4.3 LIMS: Laboratory Information Management System. The database system used to store and report data.

5.0 Method Variations

5.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Senior Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 Safety Precautions and Warnings

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the library and in the laboratory, respectively. Individual sample MSDS forms provided by the clients are kept in Login.

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are nitrile gloves for concentrated acids and bases, and potassium ferricyanide in neat form. Work under a hood when using concentrated acids and bases.

6.4 The handling of radioactive samples is outlined in SOP "Handling of Radioactive Samples" (GL-EPI-E-S004). General guidelines include:

6.4.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

6.4.2 A plastic apron may be worn over the lab coat for added protection from contamination when working with radioactive samples.

6.4.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.4.4 Prohibit admittance to immediate work area.

6.4.5 Post signs indicating radioactive samples are in the area.

6.4.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in the radiochemistry laboratory.

6.4.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management."

6.5 Refer to SOP "Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011) for instructions on how materials are disposed.

6.6 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the Group Leader prior to carrying out the rest of the procedure.

6.7 When handling biological samples protect the hands and forearms by wearing gloves and a laboratory coat to avoid contact of the biological material with the skin. Protect the eyes by wearing safety glasses and if desired a splash shield.

6.8 If cutting of meats or other tissue with cutlery is required, the analyst will wear a cutting glove of mesh steel over disposable gloves to avoid cuts that could infect the analyst with pathogens.

6.9 Any procedure that volatilizes biological substances such as drying or ashing, must be conducted in a hood or other suitable containment device.

6.10 Decontamination of work surfaces exposed to biological samples is performed by wiping the work area with a diluted (1:10) bleach solution and water as soon as possible following analytical operations.

6.11 Exterior protective clothing shall be removed prior to exiting the bioassay sample preparation area in order to prevent the inadvertent spread of biohazards to the rest of the laboratory.

6.12 Hands will be washed with an antibacterial soap directly after handling biological samples.

7.0 Interferences

7.1 When present. TH-228 alpha energies interfere with the proper quantification of Pu-238. Steps are taken to ensure that the final plutonium counting sources are free from thorium interferences.

7.2 Am-241 and Pu-238 alpha energies cannot be separated using alpha spectroscopy. Steps are taken to ensure that proper separation of Am and Pu is obtained prior to final source preparation and counting.

8.0 Apparatus, Materials, Reagents, Equipment, and Instrumentation

8.1 Ancillary Equipment

8.1.1 Silicon surface barrier detectors with associated electronics, vacuum chambers, and data reduction capabilities

8.1.2 Ion exchange columns. Eichrom TRU-Spec™ prepackaged column with 25 mL reservoir

8.1.3 Vacuum pump and filtration apparatus (25 mm)

8.1.4 Gelman metricel 25 mm filters with 0.1 µm pore size

8.1.5 Gelman polypropylene 25 mm support filter

8.1.6 Stainless steel disks. 29 mm diameter

8.1.7 Double stick tape

8.1.8 Stainless steel tweezers

8.1.9 Polypropylene centrifuge tube (50 mL)

8.1.10 Sample drying and ashing apparatus

8.1.11 Sample homogenization apparatus

8.2 Reagents, Chemicals and Standards

8.2.1 Ammonium hydroxide concentrated (14 N)

8.2.2 Ammonium thiocyanate, (NH_4SCN) 6 M. Dissolve 456.72 grams of solid ammonium thiocyanate, ACS grade in DI and dilute to 1.0 liter with DI.

Filter through a 47 mm 0.45 um membrane filter and store in 1.0 liter Nalgene bottle.

8.2.3 1 M Ammonium thiocyanate/0.1 M Formic acid solution. Add 167 mL of 6 M ammonium thiocyanate solution to 100 mL of 1M formic acid solution and dilute to 1.0 liter with DI water.

8.2.4 3 M Ammonium thiocyanate/0.1 M Formic acid solution. Add 500 mL of 6 M ammonium thiocyanate solution to 100 mL of 1M formic acid solution and dilute to 1.0 liter with DI water.

8.2.5 Ascorbic acid (0.8M). Dissolve 14.1 g of ascorbic acid in 100 mL of DI water. This solution should be prepared weekly to maintain its effectiveness in reducing iron.

8.2.6 Calcium carrier solution. Dissolve 71.7 grams of calcium nitrate in DI water and dilute to 200 mL with DI.

8.2.7 Cerium(III)nitrate hexahydrate. Dissolve 0.155 g in 100 mL DI water. (500 µg Ce/mL).

NOTE: This is normally purchased in the correct concentration from an approved vendor such as High Purity Standards.

8.2.8 Ethyl alcohol (80%). Dilute 400 mL ethanol to 500 mL with DI water.

8.2.9 Formic acid, HCOOH, 1 M. Dilute 40.7 mL of concentrated formic acid (24.6 M) to 1.0 liter with DI.

8.2.10 Hydrochloric acid, concentrated 12 N.

8.2.11 Hydrochloric acid 9 N. Dilute 750 mL of concentrated HCl to 1 liter with DI water.

8.2.12 Hydrochloric acid 2 N. Dilute 167 mL of concentrated HCl to 1 liter with DI water.

8.2.13 Hydrochloric acid/Peroxide solution. To 1.0 liter of 9 M HC1, add 8 drops of 30% peroxide solution.

8.2.14 Hydrochloric acid/Ammonium Iodide solution. Dissolve 7.26 grams of Ammonium Iodide in 1 L of 9 M HCl.

8.2.15 9 M Hydrochloric acid/0.52 M Hydrofluoric acid. Dilute 18.6 mL of concentrated HF in 1 liter of 9 M HC1.

8.2.16 Hydrochloric acid 4.0 N. Dilute 333 mL of concentrated HC1 to 1.0 L with DI water.

8.2.17 Hydrofluoric acid concentrated (48%)

8.2.18 Hydrogen peroxide (30%)

8.2.19 Ion exchange resin. Bio-Rad AG 1X8, chloride form, 100-200 mesh

8.2.20 Iron Carrier. 10 mg/mL

8.2.21 KSCN indicator (0.1 M). Dissolve 0.972 g of potassium thiocyanate in 100 mL of DI water

8.2.22 NIST traceable standards: Am-241, Am-243, Cm-244, Pu-242, Pu-239, Pu-238, Pu-236

8.2.23 Nitric acid concentrated 16M

8.2.24 2 M Nitric acid/1 M aluminum nitrate. Dissolve 375.1 g of $A1(N0_3)_3$* $9H_2O$ in 500 mL of DI water, add 62.5 mL of concentrated nitric acid, and dilute to 1.0 with DI water.

8.2.25 Nitric acid (2M). Dilute 125 mL of concentrated nitric acid to 1.0 L with DI water

8.2.26 Titanous chloride. 20% reagent.

9.0 Sample Handling and Preservation

9.1 If the sample has exceeded the hold time, the analyst should contact the Group Leader before continuing with the batch.

9.2 Soil samples require no preservation and may be shipped in any suitable container.

10.0 Sample Preparation

Soil Sample Preparation

10.1 If not already done, homogenize the sample by performing SOP "Preparation of Soils for the Determination of Radionuclides" (GL-EPI-E-A021).

10.2 Samples must be ashed in a muffle furnace as specified in SOP "Soil Sample Ashing for the Determination of Radionuclides" (GL-EPI-E-A021b).

10.3 Following ashing, weigh an appropriate aloquot of soil sample (5 – 10 grams) into a 400 mL glass beaker. Add Am-243 and Pu-242 (or Pu-236) tracers.

10.4 Add 40 mL of concentrated nitric acid and 40 mL of concentrated hydrochloric acid. Cover sample with a watch glass and heat on medium heat for one hour.

10.5 Remove samples from the hotplate and allow to cool.

10.6 Without disturbing the sample solids, decant as much of the sample leachate as possible into a labeled disposable 50 mL centrifuge tube/

10.7 Centrifuge, then decant the leachate into a second 400 mL glass beaker. Repeat step 10.6 to collect the remaining leachate, centrifuge, then decant leachate into the second glass beaker. Transfer the sample solids in the c-tube back to the original beaker using concentrated nitric acid as a rinse.

10.8 Repeat steps 10.4 through 10.7 until the sample solids color stabilizes to the neutral color of sand.

10.9 Evaporate the sample leachates to dryness. Dissolve the sample residue in 20 mL of concentrated hydrochloric acid and evaporate to dryness.

10.10 Dissolve the sample residue in 50 mL of 9M HCl/H_2O_2 solution and warm gently to effect dissolution. Remove the samples from the hotplate and allow to cool.

10.11 Condition a 5 mL AG 1 x 8 resin column (100-200 mesh) with 40 mL of 9 M HCl. Load the 9 M HCl/H_2O_2 sample solution through the column and catch the sample load in a labeled glass beaker for Am analysis.

10.12 Rinse the column with 50 mL of 9 M HCl/ H_2O_2 solution and catch the rinse in the glass beaker for Am analysis. Proceed to step 10.17 for Am analysis.

10.13 Rinse the columns with 25 mL of 9 M HCl and catch the rinses in a drip pan for disposal.

10.14 Rinse the column with 75 mL of 9 M HCl and catch the rinse in a drip pan for disposal.

10.15 Elute Pu from the column into a labeled glass beaker using 40 mL of 9 M HCl/0.05 M NH_4I solution.

10.16 To the Pu sample, add 4 drops of iron carrier solution, 1 mL of 30% H_2O_2, and 10 mL of concentrated nitric acid. Evaporate to dryness. Dissolve residue in 5 mL of 3 M HCl and transfer to a disposable centrifuge tube using DI water as a rinse.

Proceed to step 10.37 for microprecipitation source preparation for alpha spec analysis.

10.17 To the Am sample fraction from step 10.12, add 1 mL of calcium carrier and evaporate to dryness on a hotplate. Dissolve the sample residue in 50 mL of 1 M HCl. Dilute the sample to 150 mL with DI water. Add 1 mL of 30% H_2O_2 and approximately 5 grams of Oxalic acid Dihydrate to the sample and heat to dissolve. Precipitate calcium Oxalate from the sample by adjusting the pH to 2 – 3 using concentrated ammonium hydroxide solution. Allow the precipitate to settle.

10.18 Decant the supernate and transfer the precipitate to a disposable centrifuge tube using 0.1 % Oxalic acid wash solution. Centrifuge and decant with supernate. Dissolve the precipitate in 10 mL of concentrated nitric acid and transfer the solution to a 150 mL glass beaker. Evaporate to dryness.

10.19 Muffle the sample at 550° C for a minimum of 6 hours. Cool the sample and add 10 mL of concentrated nitric acid and 1mL of 30% H_2O_2. Evaporate to dryness and continue with step 10.20.

10.20 Dissolve the precipitate in 10 mL of 2 M nitric acid/0.5 M aluminum nitrate.

10.21 Add 1 drop of KSCN indicator and swirl. Add 8 drops of ascorbic acid solution. This process ensures that no trivalent iron is present, which may inhibit the retention of americum and curium by the resin.

10.22 Prepare the TRU-Spec column by pre-rinsing with 5mL of 2 M nitric acid.

10.23 Load the sample from step 10.21 onto the column and discard the initial effluent.

10.24 Rinse the column three times with 5 mL increments of 2 M HNO_3. Discard the rinses to waste.

10.25 Add 2 mL of 9 M HCl and collect the effluent in a clean, labeled 50 mL disposable centrifuge tube.

10.26 Elute Am and Cm with 10 mL 4 M HCl into the tube from previous step. Proceed to Am/rare earth separation in step 10.27.

10.27 Evaporate the Am/TRU elute from step 10.26 to a minimum volume in a small labeled glass beaker. Add 10 mL of concentrated nitric acid and warm gently on a hotplate. Add 30% H_2O_2 solution dropwise to oxidize any TRU extractant that may have bled from the TRU column. Add a maximum of 2 mL of 30% H_2O_2. Evaporate the solution to near dryness.

10.28 Repeat step 10.27.

10.29 Add 10 mL of concentrated hydrochloric acid and evaporate just to dryness, being careful not to bake the residue.

10.30 Add 2 ml concentrated formic acid and heat to dryness. Then dissolve the chloride residue in 10 mL of 3 M ammonium thiocyanate/0.1 M formic acid solution. Heat gently to effect dissolution, then remove to cool.

10.31 Condition a 2.5 mL TEVA column with 10 mL of 3 M ammonium thiocyanate/0.1 M formic acid solution. Pass the cooled sample through the column and catch the load solution in a waste pan for disposal.

10.32 Rinse the column three times with 1 M ammonium thiocyanate/0.1 M formic acid solution using increments of 2 mL, 3 mL and 5 mL. Discard the rinses to waste.

10.33 Elute Am from the column using 15 mL of 2 M HCl, collecting the elute into a disposable centrifuge tube. Proceed to step 10.34 for microprecipitation source preparation for alpha spec analysis.

10.34 Add 100 µl of 500 ppm cerium solution to the Am/Cm samples and swirl. For plutonium samples, add 4 drops of 20% dihydrazine dihydrochloride solution and swirl to mix.

10.35 Add 2 mL of concentrated HF for Am/Cm analysis or 3 mL of HF for Pu, swirl, and allow to stand for 30 minutes.

10.36 Place 0.1µm metricel filter on the filter funnel base.

10.37 Rinse the filter and funnel under vacuum with 5 mL of 80% ethanol. Rinse the filter and funnel with 5 mL of type II water. With minimum delay, add the sample to the filtering apparatus and rinse the beaker several times into the funnel with type II water. Complete the filtering by adding 5 mL of 80% ethanol after the sample has completely run through the filter.

10.38 Allow the filters to air dry. Label a 29 mm flat planchet with the applicable laboratory number and desired radionuclide (i.e., Pu/Am/Cm). Cover the planchet with double stick tape and carefully place the filter (precipitate side up) on the adhesive. Care should be taken to center the filter and make it as flat as possible on the planchet.

NOTE: Care should be taken not to touch the active area of the filter with forceps.

10.39 Count under vacuum on the alpha spectrometer long enough to reach requested MDA. Consult the operating manual for instruction on operating the alpha spectrometer.

11.0 Preparation of Standard Solutions and Quality Control Standards

11.1 Refer to SOP "Preparation of Radioactive Standards" (GL-EPI-E-M001).

11.2 All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in SOP "Preparation of Radioactive Standards" (GL-EPI-E-M001), Section 19.0.

11.3 Primary standards are kept in the laboratory in a secured cabinet. Secondary, working, standards are kept at the bench area in an enclosed plastic cabinet.

12.0 Instrument Calibration and Performance

12.1For direction on calibration and instrument performance see SOP "Micro-VAX 3100 Alpha Spectroscopy System" (GL-EPI-E-I009).

13.0 Analysis and Instrument Operation

13.0 For analysis and instrument operation see SOP "Micro-VAX 3100 Alpha Spectroscopy System" (GL-EPI-E-I009).

14.0 Equipment and Instrument Maintenance

14.1 For maintenance of system see "Counting Room Maintenance and Performance Checks" (GL-EPI-E-I010).

15.0 Data Recording, Calculation, and Reduction Methods

15.1 The instrument will report sample pCi/unit according to the following equation:

$$\text{PCi/unit} = \frac{S_{cpm} - B_{cpm}}{2.22 * E * A * \text{decay} * R}$$

15.2 Counting uncertainty is propagated according to the following equation:

$$\text{pCi / unit} = Ac * 1.96 \sqrt{\left(\frac{ef\_er}{E}\right)^2 + \left(\frac{pk\_er}{pk}\right)^2 + \left(\frac{ab\_er}{A}\right)^2 + \left(\frac{sy}{100}\right)^2 + (dk)^2}$$

15.3 The minimum detectable activity (MDA) is calculated according to the following equation:

$$MDA(pCi/unit) = \frac{2.71 \div 4.65 * \sqrt{B_{cpm} * T_c}}{(2.22 * E * V * R * A * decay * T_c)}$$

where:

$$decay = \frac{1}{e^{\left(\frac{-\ln(2)T_d}{T_{1/2}}\right)}}$$

$$R = \frac{T_{cpm} - B_{cpm}}{T_{dpm} * E}$$

$$dk = \frac{T_{ef\_er}}{T_{pk\_er}} * \left(\frac{\lambda Tr}{1 - e^{-\lambda Tr}} - \lambda\left(T_c + T_r\right) - 1\right)$$

And where:

Scpm	=	Sample counts per minute
Bcpm	=	Background counts per minute
E	=	Counting efficiency (decimal form)
V	=	Volume in liters,g,cfm, etc.
A	=	Isotopic abundance (decimal form)
ef_er	=	1 sigma efficiency error (decimal form)
pk_er	=	1 sigma peak error
ab_er	=	1 sigma isotopic abundance error (decimal form)

18.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

19.0 Laboratory Waste Handling And Waste Disposal

19.1 Radioactive samples and material shall be handled and disposed of as outlined in SOP "Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011).

20.0 References

20.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

20.2 EML Procedures Manual HASL-300, 1982.

20.3 Analytical Chemistry. Rapid Determination of Th-230 in Mill Tailings by alpha spectroscopy. UNC Geotech, Grand Junction Projects Office. Steve Donivan, Mark Hollenbach, and Mary Costello. Vol. 59, No. 21, 1987.

20.4 Los Alamos Health and Environmental Chemistry: Analytical Techniques. LA-10300-M Vol. 1, September 1987.

20.5 Special thanks to Dr. Bill Burnett and his associates for assistance in developing this method at Florida State University.

STANDARD OPERATING PROCEDURE

FOR

UNCONTROLLED DOCUMENT

DETERMINATION OF THORIUM/URANIUM

(GL-EPI-E-A-038 Revision 0)

HARD COPY IN ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of General Engineering Laboratories, Inc. and may not be specifically applicable to the activities of other organizations.

PROPRIETARY INFORMATION

Process Owner	8/31/99 (sign & date)	Robert D Timms (print name)
Technical Review	8/10/99 (sign & date)	Richard M. Kinney (print name)
Quality Review	9/7/99 (sign & date)	Gene Bost (print name)
Approval and Authorization	9/7/99 (sign & date)	James B. Westmoreland (print name)

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510

TABLE OF CONTENTS

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1.0 ISOTOPIC DETERMINATION OF THORIUM/URANIUM

2.0 METHOD OBJECTIVE, PURPOSE, CODE AND SUMMARY

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic thorium and uranium in a variety of matrices.

2.2 A sample is digested if necessary and aliquoted. Transuranic elements are scavenged by coprecipitation with iron hydroxide. The precipitate is dissolved and separation of elements is accomplished through extraction chromatography and ion exchange resins. The elements are then prepared for the measurement of radioactive isotopes by coprecipitation with cerium fluoride. The cerium fluoride precipitate is trapped on a filter, mounted on a stainless steel disk and placed in a partially evacuated chamber for measurement of isotopic alpha emission.

2.3 GEL utilizes methods that are derived from established sources. This method is based on the source method from DOE EML Methods Manual HASL 300 PU-02, 03 and uses similar principles of radiochemical separation and counting. This method is also very similar in concept to the source method from the DOE Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP800: "Sequential Separation of Americum and Plutonium by Extraction Chromatography."

3.0 METHOD APPLICABILITY

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for Thorium and Uranium is 1 pCi/L or 1 pCi/g for all isotopes.

3.2 Method Precision: Typical relative percent difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value. Analyst training records are kept on hand in the Human Resources department.

4.0 DEFINITIONS

4.1 National Institute of Standards and Technology (NIST). For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.2 Type II water: Deionized water.

4.3 LIMS: Laboratory Information Management System. The database system used to store and report data.

5.0 METHOD VARIATIONS

5.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Sr. Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 SAFETY PRECAUTIONS AND WARNINGS

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the laboratory. Individual sample MSDS forms provided by the clients are kept in Login.

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are nitrile gloves for concentrated acids and bases, and potassium ferricyanide in neat form. Work under a hood when using concentrated acids and bases.

6.4 The handling of radioactive samples is outlined in SOP GL-EPI-M-001, "Handling of Radioactive Samples." General guidelines include:

6.4.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

6.4.2 A plastic apron may be worn over the lab coat for added protection from contamination when working with radioactive samples.

6.4.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.4.4 Prohibit admittance to immediate work area.

6.4.5 Post signs indicating radioactive samples are in the area.

6.4.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in the radiochemistry laboratory.

6.4.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management.

6.5 Refer to SOP GL-EPI-E-S-005, "Radioactive Waste Handling Procedures," for instructions on how materials are disposed.

6.6 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the group leader prior to carrying out the rest of the procedure.

6.7 When handling biological samples protect the hands and forearms by wearing gloves and a laboratory coat to avoid contact of the biological material with the skin. Protect the eyes by wearing safety glasses and if desired a splash shield.

6.8 If cutting of meats or other tissue with cutlery is required, the analyst will wear a cutting glove of mesh steel over disposable gloves to avoid cuts that could infect the analyst with pathogens.

6.9 Any procedure that volatilizes biological substances such as drying or ashing, must be conducted in a hood or other suitable containment device.

6.10 Decontamination of work surfaces exposed to biological samples is performed by wiping the work area with a diluted (1:10) bleach solution and water as soon as possible following analytical operations.

6.11 Exterior protective clothing shall be removed prior to exiting the bioassay sample preparation area in order to prevent the inadvertent spread of biohazards to the rest of the laboratory.

6.12 Hands will be washed with an antibacterial soap directly after handling biological samples.

7.0 INTERFERENCES

7.1 Internal tracer standards may have ingrown daughters that may interfere with the analysis. For example, TH-228 will be present in aged U-232 standard. These problems are overcome by running separate aliquots of sample for thorium analysis.

7.2 Short lived radioactive progeny may ingrow on prepared filters. For example, the Ra-224 alpha peak will be present if the Th-228 parent is present. These

interferences are minimized by counting samples as soon as possible after separation chemistry.

8.0 APPARATUS, MATERIALS, REAGENTS, EQUIPMENT, AND INSTRUMENTATION

8.1 Ancillary Equipment

8.1.1 Silicon surface barrier detectors with associated electronics, vacuum chambers, and data reduction capabilities.

8.1.2 Ion exchange columns.

8.1.3 Vacuum pump and filtration apparatus (25 mm)

8.1.4 Gelman metricel 25 mm filters with 0.1 um pore size

8.1.5 Gelman polypropylene 25 mm support filter

8.1.6 Stainless steel disks. 29 mm diameter

8.1.7 Stainless steel tweezers

8.1.8 Polypropylene centrifuge tube (50 mL)

8.1.9 Sample drying and ashing apparatus

8.1.10 Sample homogenization apparatus

8.1.11 AGIX8 anion exchange resin 100-200 mesh

8.2 Reagents, Chemicals and Standards

8.2.1 Ammonium hydroxide concentrated (14N).

8.2.2 Con. Nitric Acid (HNO_3)

8.2.3 Cerium(III)nitrate hexahydrate. Dissolve 0.155g in 100 mL DI water. (500 ug Ce/mL).

NOTE: This is normally purchased in the correct concentration from an approved vendor such as High Purity Standards.

8.2.4 Ethyl alcohol (80%). Dilute 400 mL ethanol to 500 mL with DI water.

8.2.5 Hydrochloric acid concentrated. (12M)

8.2.6 6 M HCl/0.52 M HF

8.2.7 0.1 M HCl – 8.3 mls conc. HCl diluted to 1 L with DI

8.2.8 Hydroflouric acid concentrated (48%)

8.2.9 Iron Carrier. 10 mg/mL

8.2.10 9 M HCl – 1500 mls con HCl diluted to 2 L

8.2.11 NIST traceable Standards: Th-232, Th-229,.U-232, U-238

8.2.12 Nitric acid concentrated 16 M

8.2.13 Nitric acid (8 M). Dilute 50 mL of conc. Nitric acid to 100 mL DI water.

8.2.14 Titanous chloride. 20% reagent

9.0 SAMPLE HANDLING AND PRESERVATION

9.1 Samples should be preserved to approximately pH 2 with nitric acid and collected in a plastic bottle.

9.2 Before beginning an analysis the analyst should check the sample pH with a pH strip. If necessary, adjust the pH with nitric acid to a pH=1-2. If the sample was pH adjusted let the sample sit overnight before continuing the batch.

9.3 If the sample has exceeded the hold time the analyst should contact the group leader before continuing with the batch.

9.4 Soil samples require no preservation and may be shipped in any suitable container.

10.0 SAMPLE PREPARATION

Soil Sample Preparation

10.1 If not already done, homogenize the sample by performing GL-EPI-E-A-021 "Preparation of Soils for the Determination of Radionuclides."

10.2 It is recommended that the samples be ashed in a muffle furnace as specified in GL-EPI-E-A-021b "Soil Sample Ashing for the Determination of Radionuclides."

10.3 For thorium and uranium analysis, take an appropriate aliquot and digest as specified in GL-EPI-E-A-015, "Digestion for Soil and Sand."

Aqueous Sample Preparation

10.4 Add an appropriate aliquot of sample to a labeled beaker. Add a certified dpm of the following tracers to each of the samples: for isotopic thorium, Th-229 is normally used; and for Uranium, U-232 is normally used.

NOTE: Other sample matrices, such as vegetation, air filters, tissue etc., are run as outlined in "Preparation of Special Matrices for the Determination of Radionuclides" (GL-EPI-E-A-021b).

10.5 Add 1 mL of iron carrier.

NOTE: Iron carrier may not be needed for soil samples.

10.6 Bring to a slight boil and add concentrated NH_4OH until turbidity persists, or pH>9. Heat to near boiling for 10 minutes and then allow to settle and cool.

10.7 Decant excess supernate and discard. Collect the remaining precipitate by centrifugation in a 50 mL centrifuge tube and discard the supernate.

NOTE: Exercise care in this step because finely divided material, which contains the actinides may also be present in addition to large iron hydroxide flocks.

10.8 Dissolve the precipitate in 8 M HNO_3.

10.9 Slurry AC 1X8 anion exchange resin in DI water. Add the resin to an empty plastic column up to line on column neck.

10.10 Pass 25 mL of 8M HNO_3 through the column and discard the effluent. For sequential Uranium/Thorium determination, complete each line of the following checklist, in the order specified.

URANIUM/THORIUM

COLUMN WORK: Catch in clean centrifuge tube for U

NOTE: *Save the columns to run the U through them again.*

HINT: *Place 15 mls of NH4OH in the centrifuge tube to begin with to assist with the U ppt later*

___Load solution

___2 mls 8 M HNO_3
___3 mls 8 M HNO_3
___5 mls 8 M HNO_3
___ppt elution with NH4OH and spin, use this ppt for 2nd set of columns

Th Elution: catch in a clean centrifuge tube
___5 mls 9 M HCL
___5 mls 9 M HCL
___10 mls 9 M HCL
___10 mls 9 M HCL
___10 mls 9 M HCL

Th precipitation
___transfer to a beaker, add 1 ml FE carrier and take dry
___dissolve the residue with 2 mls of 9M HCL
___transfer to a clean centrifuge tube and dilute to 20 mls with DI
___0.1 Ce carrier (500 mg)
___add 0.1 mL of cerium carrier (500 µg/mL)
___3 mls HF, wait 20 minutes
___filter

URANIUM Column Work (2ND Time): catch in drip plan
___dissolve the ppt with 15 mls 9 M HCl
___conditioning 10 mls 9 M HCL (if the columns are being ran in the same day this step is not necessary)
___load solution
___5 mls 9 M HCL (rinse)
___5 mls 9 M HCL (rinse)
___5 mls 6 M HC1/0.52 M HF (rinse) [18 mls HF diluted to 1 L with 9 M HCl)
___5 mls 6 M HC1/0.52 M HF (rinse)

URANIUM Elution: catch in clean centrifuge tube
___5 mls 0.1 M HC1
___5 mls 0.1 M HC1
___5 mls 0.1 M HC1
___5 mls 0.1 M HC1

U precipitation
___0.1 Ce carrier (500 mg)
___1 ml TiC1, swirl, wait 5 minutes
___3 mls HF, swirl, wait 20 minutes
___filter

10.11 Place a 0.1 um metricel filter on the filter funnel base.

10.12 Rinse the filter and funnel under vacuum with 5 mL of 80% ethanol. With minimum delay, add the sample to the filtering apparatus and rinse the beaker several times into the funnel with type II water. Complete the filtering by adding 5 mL of 80% ethanol.

10.13 Dry the filter under a heat lamp in a labeled petri dish. Label a 29 mm flat planchet with the applicable laboratory number and desired radionuclide. Care should be taken to center the filter and make it as flat as possible on the planchet. Note: care should be taken not to touch the active area of the filter with forceps.

10.14 Count under vacuum on the alpha spectrometer long enough to reach requested MDA. Consult the operating manual for instruction on operating the alpha spectrometer.

11.0 PREPARATION OF STANDARD SOLUTIONS AND QUALITY CONTROL STANDARDS

11.1 Refer to "Preparation of Radioactive Standards," (GL-EPI-E-M001).

11.2 All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in "Preparation of Radioactive Standards," GL-EPI-M-001, section 19.0.

11.3 Primary standards are kept in the laboratory in a secured cabinet. Secondary, working standards are kept at the bench area in an enclosed plastic cabinet.

12.0 INSTRUMENT CALIBRATION AND PERFORMANCE

12.1 For direction on calibration and instrument performance see "Micro-VAX 4000 Alpha Spectroscopy System," (GL-EPI-I-009).

13.0 ANALYSIS AND INSTRUMENT OPERATION

13.1 For analysis and instrument operation see "Micro-VAX 4000 Alpha Spectroscopy System," (GL-EPI-I-009).

14.0 EQUIPMENT AND INSTRUMENT MAINTENANCE

14.1 For maintenance of system see "Counting Room Maintenance and Performance Checks," (GL-EPI-I-010).

15.0 DATA RECORDING, CALCULATION, AND REDUCTION METHODS

15.1 The instrument will report sample pCi/unit according to the following equation:

$$\text{pCi / unit} = \frac{S_{cpm} - B_{cpm}}{2.22 * E * V * A * \text{decay} * R}$$

15.2 Counting uncertainty is propagated according to the following equation:

$$\text{pCi / unit} = Ac * 1.96\sqrt{\left(\frac{ef\_er}{E}\right)^2 + \left(\frac{pk\_er}{pk}\right)^2 + \left(\frac{ab\_er}{A}\right)^2 + \left(\frac{sy}{100}\right)^2 + (dk)^2}$$

15.3 The minimum detectable activity (MDA) is calculated according to the following equation:

$$MDA(\text{pCi / unit}) = \frac{2.71 + 4.65 * \sqrt{B_{cpm}} * T_c}{(2.22 * E * V * R * A * decay * T_c)}$$

where:

$$decay = \frac{1}{\left(e^{(-\ln 2)} * \frac{T_d}{T_{1/2}}\right)}$$

$$R = \frac{T_{cpm} - B_{cpm}}{T_{dpm} * E}$$

$$dk = \frac{T_{1/2}err}{T_{1/2}} * \left| \frac{\lambda Tr}{1 - e^{-\lambda Tr}} - \lambda * \left| T_c + T_r \right| - 1 \right|$$

And where:

Scpm	=	Sample counts per minute
Bcpm	=	Background counts per minute
E	=	Counting efficiency (decimal form)
V	=	Volume in liters,g,cfm, etc.
A	=	Isotopic abundance (decimal form)
ef_er	=	1 sigma efficiency error (decimal form)
pk_er	=	1 sigma peak error
ab_er	=	1 sigma isotopic abundance error (decimal form)
sy	=	1 sigma sytematic error
pk	=	peak area
Tc	=	Sample count time in minutes
Td	=	Time interval for radioactive decay
Tr	=	Elapsed real time in minutes
Tcpm	=	Tracer counts per minute
Tdpm	=	Tracer known disintegrations per minute
Ac	=	Sample calculated activity
T1/2	=	Isotopic half life
T1/2err	=	Isotopic half life error