473-01

Defense Environmental Restoration Program (DERP)

Quality Assurance Project Plan for Closure of Nine Burning Pads Seneca Army Depot ROMULUS, NY

JANUARY 1988
Revised: APRIL 1988

Prepared for

DEPARTMENT OF THE ARMY
KANSAS CITY DISTRICT, CORPS OF ENGINEERS
700 FEDERAL BUILDING
KANSAS CITY, MISSOURI 64106-2896

Prepared by

Metcalf & Eddy
METCALF & EDDY ENGINEERS
10 HARVARD MILL SQUARE
WAKEFIELD, MA 01880

DEFENSE ENVIRONMENTAL RESTORATION PROGRAM
(DERP)

QUALITY ASSURANCE PROJECT PLAN

FOR

CLOSURE OF NINE BURNING PADS

SENECA ARMY DEPOT, ROMULUS, NY

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Metcalf & Eddy
Hazardous Waste
Management Division

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J-3161

April 22, 1988

LTC David L. Franklin
U.S. Army Corps of Engineers
Kansas City District
MRKED-SD, Federal Plaza
601 East 12th Street
Kansas City, MO 64106

Attention: Mr. Doug Bennett, Project Manager, DERA Section

Dear LTC Franklin:

Please find enclosed six (6) copies each of Metcalf & Eddy's Final Sampling and Analysis Quality Assurance Project Plan for the Closure of Nine Burning Pads at Seneca Army Depot, Romulus, NY, Contract DACW41-86-D-0112.

Each plan has been submitted to the offices listed below.

If you have any further questions or comments, do not hesitate to contact me.

Very truly yours,

METCALF & EDDY, INC.
Hazardous Waste Management Division

Deborah M. Simone

Deborah M. Simone
Project Engineer

cc: USCE Huntsville, AL - (4 copies)
USCE Aberdeen Proving Ground, MD - (2 copies)
USCE New York, NY - (1 copy)
USCE Rock Island, IL - (1 copy)
USCE Alexandria, VA - (1 copy)
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PREFACE

The objective of Metcalf & Eddy's quality assurance program is to ensure that all measurement, data gathering, and data generation activities yield data that are of adequate quality for the intended use. The key to achieving this objective is the successful implementation of a project specific Quality Assurance Project Plan (QAPP) for all such activities.

This document constitutes a site specific QAPP for the Closure of Nine Burning Pads at the Seneca Army Depot (SEAD) in Romulus, NY and specifically addresses Quality Assurance (QA) issues for field sampling at the SEAD site. This report outlines the procedures and methods anticipated to be used during the sampling episode by Metcalf & Eddy, Inc. and its subcontractors. This site specific QAPP supplements Metcalf & Eddy Standard Operating Procedures (SOP's) by providing QA objectives, specific sampling procedures, sample custody protocol and analytical procedures that are unique to the sampling effort at the SEAD site.

This QAPP has been prepared according to guidelines in Contract No. DACW41-86-D-0112, Closure of Nine Burning Pads, Seneca Army Depot, Romulus, NY, and in the COE document

ER-1110-1-263, "Engineering and Design, Chemical Quality Management -- Toxic and Hazardous Wastes"; December 30, 1985, and "Sample Handling Protocol for Low, Medium and High Concentration Samples of Hazardous Waste"; October, 1986.

2.0

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3.0 Project Description

The U.S. Army Corps of Engineers (ACOE) has contracted Metcalf & Eddy, on behalf of Seneca Army Depot (SEAD), to prepare a Closure and Post Closure Plan for permanent closure of nine open burning pads located in the Demolition Ground of SEAD.

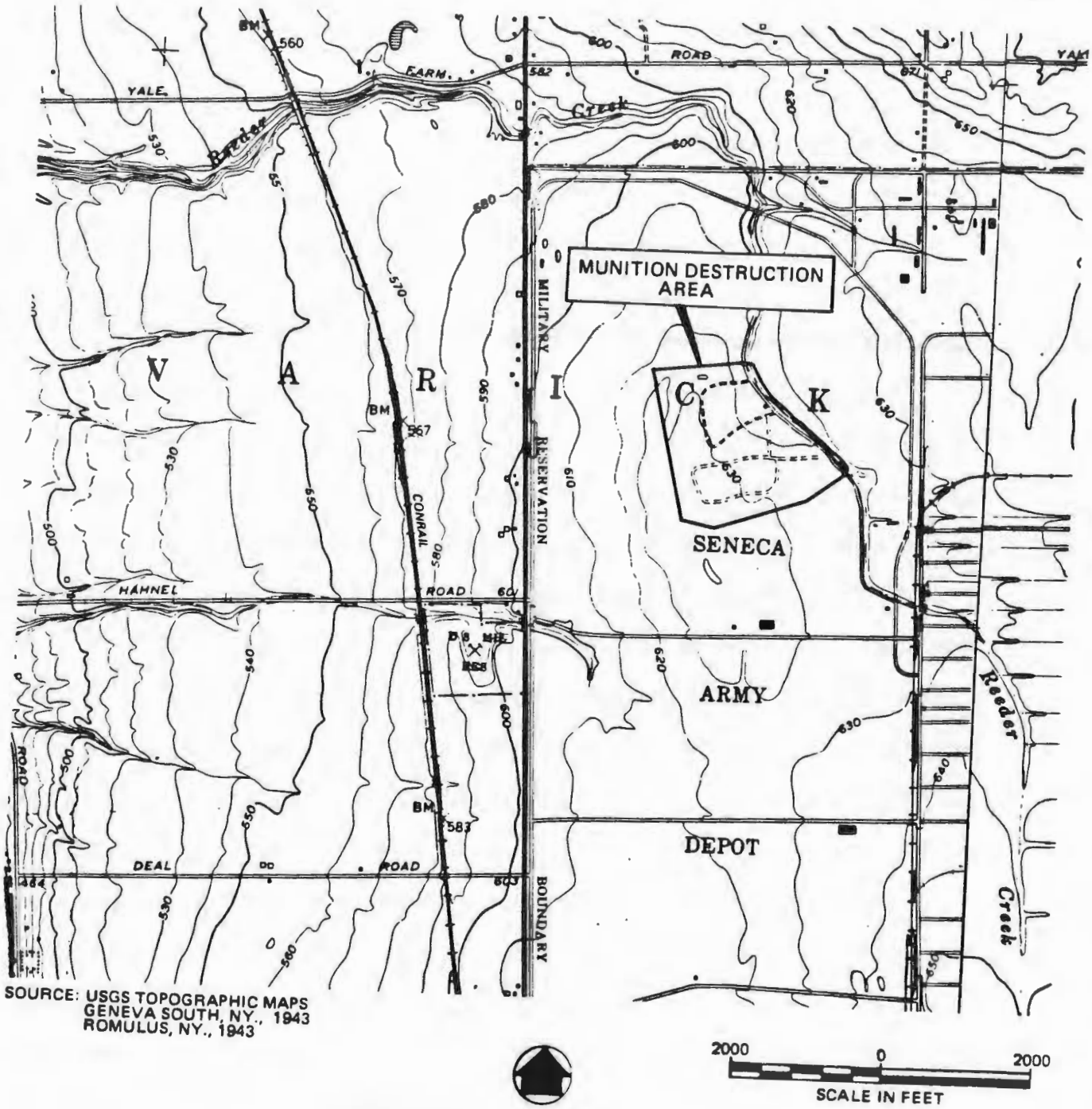
This plan presents procedures which will be followed by Metcalf & Eddy while performing on-site investigations as part of the Closure and Post Closure Plans for SEAD. The evaluation falls under the Defense Environmental Restoration Program (DERP).

The Seneca Army Depot is located near the town of Romulus, New York, east of Seneca Lake, west of Cayuga Lake and approximately fifty miles southeast of Rochester (see Figure 3.1). The ninety-acre Demolition Ground is located in the northwest section of the installation. The burning pads are part of this area and have been in use since 1941 (see Figure 3.2).

Seven of the nine pads are used for the open burning of pyrotechnics, explosives and propellants (PEP) that have been declared obsolete or off-specification. The other two pads are used for the burning of explosives-contaminated packing material.

The U.S. Army Environmental and Hygiene Agency (USAEHA) has supervised several studies pertaining to soil and water quality at

FIGURE 3.1
SITE LOCUS MAP



SOURCE: USGS TOPOGRAPHIC MAPS
GENEVA SOUTH, NY., 1943
ROMULUS, NY., 1943

FIGURE 3.1. SITE LOCATION MAP, MUNITION DESTRUCTION AREA, SENECA ARMY DEPOT, ROMULUS, NEW YORK

three pads; B, F, and H. Since seven of the pads have had identical operations, it is assumed that results from these three pads would be representative of conditions on the other four pads. Similar, but reduced levels of contaminants would be expected at pads G and I where only explosives-contaminated containers have been burned.

The open burning area is currently operated under RCRA Interim status. It is the intent of this project that the pads be permanently closed. Containment and closure-in-place is the Army's method of choice.

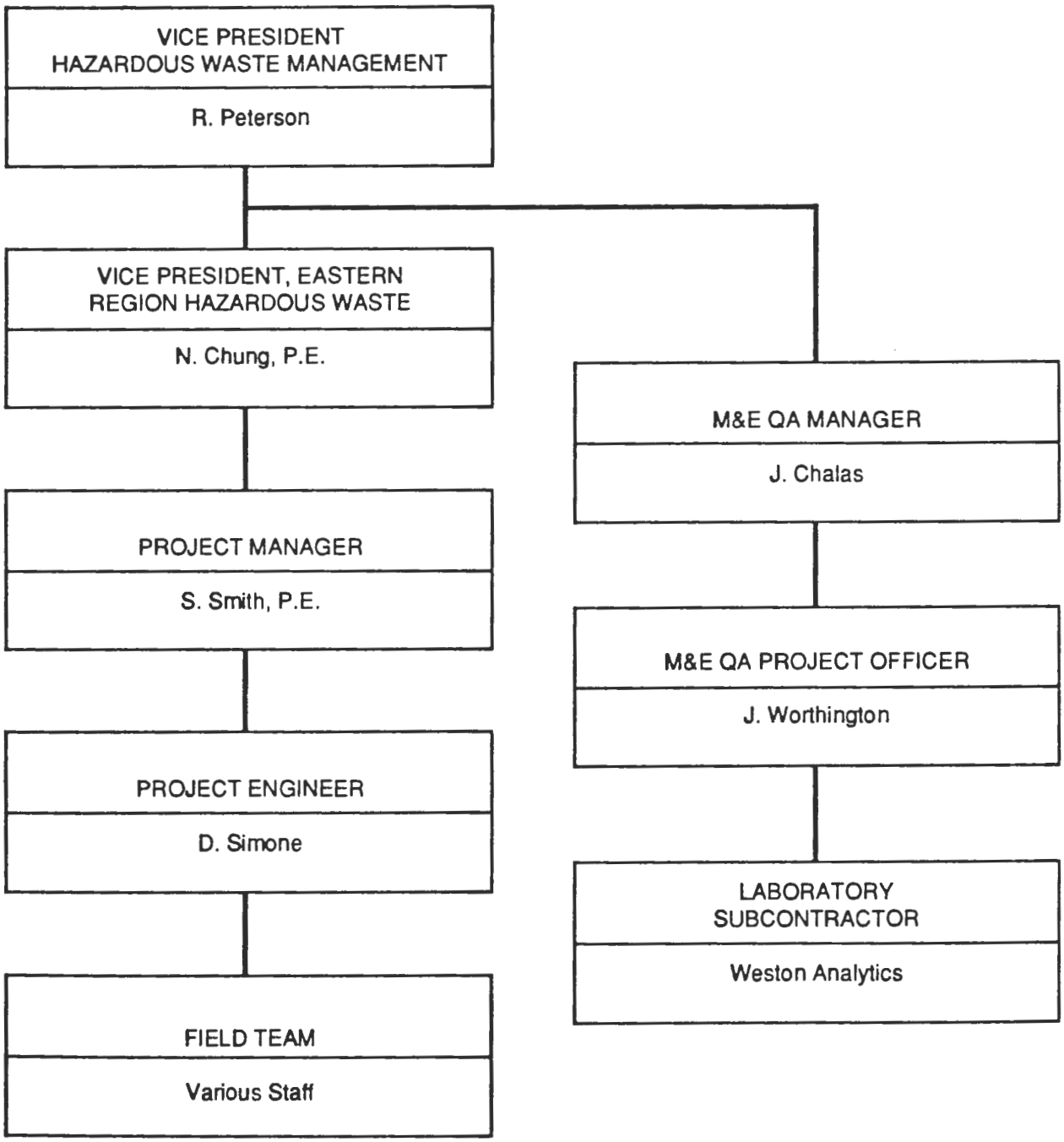
The Closure and Post Closure Plan will be supported by existing soil and water quality data and additional data provided by Metcalf & Eddy's field investigation. The field investigation will focus on the installation of six new shallow groundwater wells and the collection and analysis of groundwater samples. The six new wells will be used later for post-closure groundwater monitoring.

4.0 Project Organization and Responsibility

Figure 4.1 presents the QA Organization Chart for this project and identifies the individuals responsible for each element of the overall program. The key individual responsible for Quality Assurance is the QA Project Officer, a full-time professional reporting directly to the Corporate QA Manager who is the Senior Vice President of Metcalf & Eddy with Corporate QA responsibility. Mr. Jeffrey Worthington, Senior Chemist in Metcalf & Eddy's Chemical Waste Management Group, will be QA Project Officer for this study. Analytical results are reported directly to the QA Project Officer who reviews the data and provides it to the Project Manager and the project engineer. The Project Manager for this evaluation is Mr. Steven Smith, P.E., Hazardous Waste Management Division.

The Project Engineer will be responsible for overseeing the implementation of the QAPP objectives while conducting the field investigations and sampling episode and reports directly to the Project Manager. For the specific sampling episode at the Seneca Army Depot, Ms. Deborah Simone, project engineer in Metcalf & Eddy's Hazardous Waste Management Group, will be charged with overseeing all on-site activities.

FIGURE 4.1
PROJECT ORGANIZATION CHART



Various professional staff members will work on-site during the field sampling portion of this project. At this time, the exact staff members to be assigned to this work cannot be identified. However, at the request of the ACOE, names and resumes of the staff members to be involved can be provided for review approximately 14 days before site-work is scheduled to begin.

The subcontract laboratory, which will provide analytical services during the Closure and Post Closure Plan field investigation, will be Weston Analytics of Lionville, Pennsylvania. Weston is to provide Metcalf & Eddy with environmental analysis services together with technical support in the application of the chemical data produced.

Figures 4.2a and 4.2b depict the reporting responsibilities of key individuals within the Weston laboratory.

Resumes of key individuals that will take part in analysis of samples generated by this study are contained in Appendix A-1. Example data sheets and a descriptive list of Weston facilities and equipment are included in Appendices A-2 and A-3.

FIGURE 4.2a
WESTON ANALYTICS ORGANIZATION

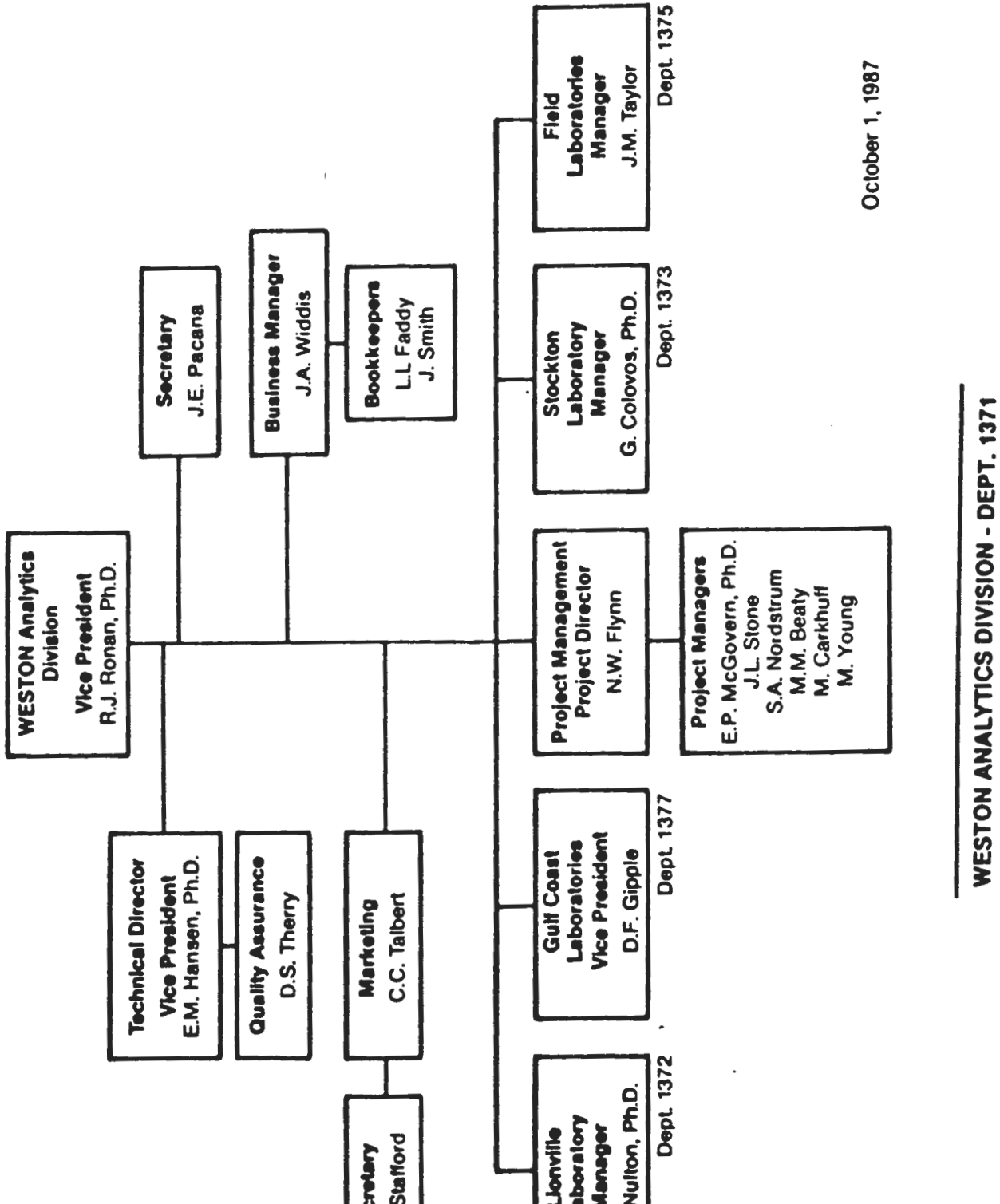
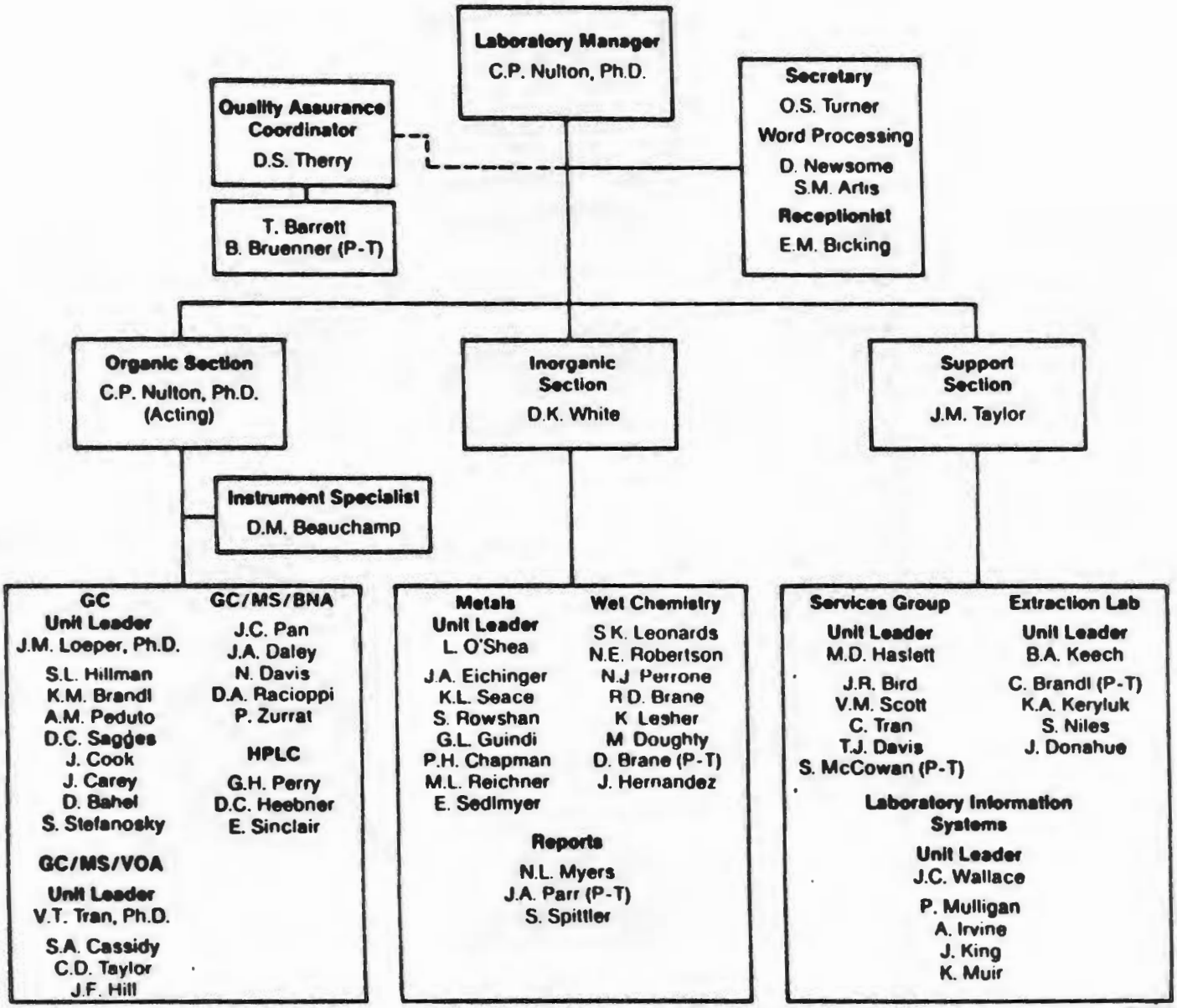


FIGURE 4.2b
WESTON ANALYTICS ORGANIZATION



October 1, 1987

LIONVILLE LABORATORY - DEPT. 1372

The key individuals responsible for implementation of Quality Assurance procedures and their specific QA responsibilities are as follows:

QA Project Officer - Jeffrey Worthington, Senior Chemist, Metcalf & Eddy, Inc.

- Reports to Metcalf & Eddy, Inc. Corporate QA Officer
- Reviews and approves QAPP
- Performs performance and systems audits of field sampling and chemical analysis activities
- Reviews and approves all laboratory measurement data
- Responsible for preparation of QA reports to management

Project Manager- Steven Smith, Metcalf & Eddy, Inc.

- Reports to Vice President, Hazardous Waste Management Division, Eastern Region
- Reviews and approves field operating procedures
- Assures that approved procedures meet project objectives
- Coordinates field activities with QA Project Officer
- Responsible for implementation of recommendations made by QA Project Officer

Site Engineer - Deborah Simone, Metcalf & Eddy, Inc.

- Reports to Project Manager, Metcalf & Eddy, Inc.
- Responsible for collection of representative samples

- Assures that samples are collected in accordance with approved QA/QC procedures
- Assures that all documentation of field sampling activities is complete and accurate
- Records and reports any problems or changes associated with field sampling activities to the QA project officer

Weston QA Project Manager - D.S. Therry, Weston Analytics

- Reports to QA Project Officer, Metcalf & Eddy, Inc.
- Approves all Weston Quality Assurance documents
- Performs audits of Weston laboratories to verify compliance with the Quality Assurance Program
- Verifies completion of corrective action cited in audits
- Acts as the collection point for proposed changes in the Quality Assurance Program and initiates changes in the program
- Maintains current Weston distribution lists for QA documents

Weston Section Managers - Weston Analytics

- Reports to QA Project Officer, Metcalf & Eddy, Inc., and Weston Project Manager
- Implements the Quality Assurance Program within the Weston Laboratory
- Periodically determines effectiveness of the Quality Assurance Program in the Weston Laboratory

- Supervises Quality Control activities in the Weston Laboratory
- Approves laboratory-specific attachments to the Quality Assurance Project Plan
- Recommends to the Quality Assurance Manager changes in the Quality Assurance Program
- Approves all reports issued by Weston laboratories
- Serves as the "focal point" for the reporting and disposition of all nonconformances
- Maintains current laboratory organization charts.

Weston Quality Control Coordinator

- Supervise the log-in of all samples received, completes chain of custody, and maintains sample log books
- Prepare Quality Control standards, insert Quality Control samples into the laboratory sample stream, and analyze results
- Perform statistical analyses utilizing results of QC sample analyses
- Inform the Laboratory Manager and/or his designated representatives of data which lies outside of acceptable limits
- Notify the Laboratory Manager and/or his designated representatives of out-of-control situations
- Report nonconformances to the Quality Assurance Manager if the situation is not corrected within the laboratory

- Conducts and evaluates results of system audits
- Train Analysts in Quality Control procedures
- Oversee instrument preventive maintenance schedule
- As necessary, discuss with the Laboratory Manager unresolved nonconformances brought to the Quality Assurance Manager's attention by a Quality Control Coordinator
- Reviews program plans for consistency with organizational and contractural requirements and will advise personnel of inconsistencies.

5.0 Sampling

All sampling methods described in this section are standard Metcalf & Eddy project-specific SOP's based on recognized ACOE protocols. USEPA procedures are used if ACOE protocols do not cover specific activities.

5.1 Selection of Sampling Locations

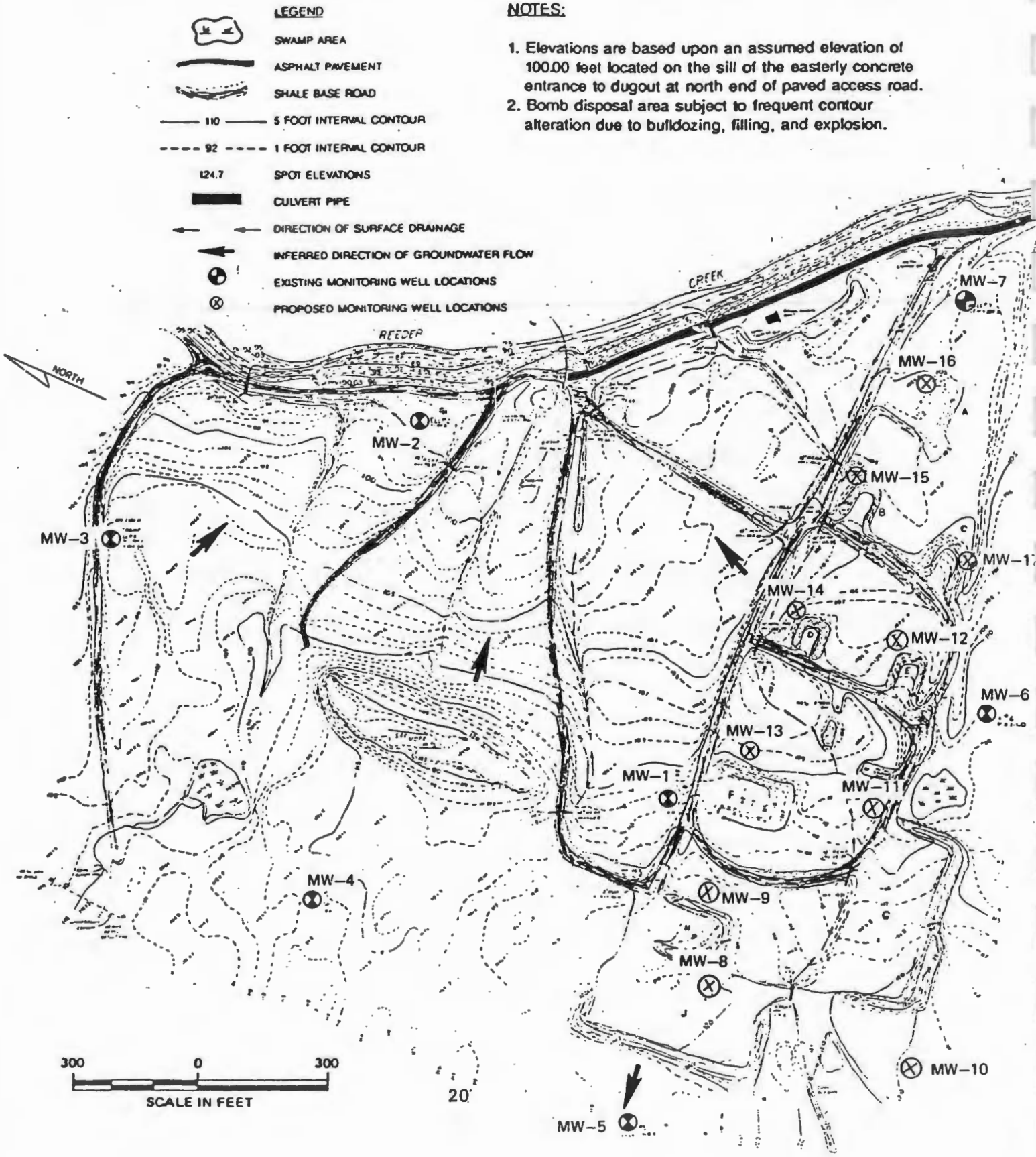
Sampling locations for groundwater samples are shown in Figure 5.1. These samples include:

- Seven (7) groundwater samples from existing monitoring wells
- Ten (10) groundwater samples from newly installed monitoring wells

These locations were selected after a review of site background data, geologic maps, and an initial site visit by Metcalf & Eddy, Inc. personnel on November 9, 10, 1987.

The wells were sited to determine general site groundwater flow directions, characterize background groundwater quality at the site and assess groundwater quality downgradient from the open burning pads. Additionally, the ten newly installed wells were sited for post closure groundwater monitoring. All but one of these wells will be down gradient from the open burning pads.

FIGURE 5.1
MONITORING WELL AND SAMPLING LOCATIONS



The rationale for each sampling location is presented below.

Ten (10) groundwater monitoring wells will be installed within the Munition Destruction Area at the Seneca Army Depot. The wells are to be used for continued groundwater monitoring during and after permanent closure of the burning pads. The proposed locations of these monitoring wells (Figure 5.1), pending the results of the geophysical surveys, are based on: (1) the particular uses of each of the nine burning pads, (2) previous investigations of waste sources conducted at the site, and (3) groundwater flow direcions based on the existing data. The wells are described in Table 5.1.

TABLE 5.1. PROPOSED MONITORING WELL LOCATIONS

Well Name	Depth	Type	Location
MW-8	15'	Monitor	Directly west of pad H.
MW-9	21'	Monitor	Within 100' east of pad H.
MW-10	21'	Monitor	200' west of the southwest corner of pad G.
MW-11	21'	Monitor	Directly north of the pavement adjacent to pad G.
MW-12	21'	Monitor	Within 100' northeast of pad E.
MW-13	15'	Monitor	Outside of the east berm of pad F.

TABLE 5.1. (Continued) PROPOSED MONITORING WELL LOCATIONS

Well Name	Depth	Type	Location
MW-14	15'	Monitor	Within 100' northeast of pad D.
MW-15	15'	Monitor	Between the norhteast berm of pad B and the adjacent depression.
MW-16	15'	Monitor	Directly northeast of pad A.
MW-17	15'	Monitor	Between the southwest berm of pad C and the adjacent drainage ditch.

A review of available documents and information pertaining to the SEAD site has been completed. An initial site visit was conducted by Deborah Simone and Tim Llewellyn (M&E) on November 9,and 10 1987 in order to assess existing site conditions. In addition, available information on the local hydrogeology was collected and reviewed to assist in locating proposed monitoring wells. The following information was obtained from U.S. Geological Survey reports and maps, New York State reports, and other sources listed in the references.

Based on the Phase 2 and Phase 4 OB/OD Hazardous Waste Management Studies performed by USAEHA, the distribution and concentration of contaminants is highly variable. Although significant levels of contaminants were found at only three burning pads, this should not preclude the possibility that

further sampling would reveal contamination within the other pads. Burning pad J could be considered to present less environmental risk, because its use was limited to the burning of PEP contaminated rubbish. It should be noted that six monitoring wells may be an insufficient number for closure purposes as set forth by RCRA regulations for the placement of detection monitoring wells. The six proposed monitoring wells should detect any contaminants originating from burning pads A, B, C, D, F, and H. Metcalf & Eddy, Inc. recommends the installation of 4 additional monitoring wells to be used to directly monitor pads E, G, J, and the area east of pad H.

Groundwater monitoring has been performed since 1982 with seven wells. Monitoring results showed no groundwater contamination originating from any of the burning pads. Approximate groundwater flow directions have been determined from groundwater elevation measurements made from these wells. A general northeast flow pattern towards Reader Creek exists beneath burning pads A through F. Hydraulic gradients are locally altered by surface water drainage patterns. Drilling revealed shallow bedrock beneath pad H, which probably divides the groundwater such that flow beneath pad J moves to the west.

Groundwater flow through the till and bedrock beneath the

burning pads is expected to be slow. Therefore, in order to detect any migration of contaminants, the proposed well locations are close to the monitored pads. Monitoring Well No. 8 (MW-8) will monitor the possible westward flow of groundwater from beneath burning pad H whereas MW-9 will monitor any eastward flow of groundwater from pad H. MW-10 will provide background water quality data, and information on water table elevation and groundwater flow direction in the southwest section of the OB area. MW-11 will monitor the expected northeast groundwater flow from burning pad G. MW-12, MW-13, MW-14, MW-15, and MW-16 are located just downgradient (northeast) of burning pads E, F, D, B, and A, respectively. Groundwater flow near pad C is expected to move southeast towards the adjacent local surface water drainage. MW-17 is located between pad C and this drainage ditch.

In the selection of monitoring well locations, physiographic and topographic conditions of the site were considered in terms of drilling accessibility and obtaining the most representative samples. The final selection of monitoring well locations will be determined as a result of site-specific field conditions during drilling and sampling operations.

5.2 Samples to be Analyzed

Table 5.2 summarizes the samples to be collected from the SEAD site, and Table 5.3 presents the specific parameters to be analyzed for each sample.

TABLE 5.2. SAMPLES TO BE ANALYZED

Description of Sample	Total Samples Collected	Analyzed by Weston	Analyzed by COE QA Lab
Groundwater			
17 Monitoring Wells; 1 Triplicate	19	18	1
Sampling/Equipment Blank	12	6	6
Travel/Trip Blank	12	6	6

TABLE 5.3 PARAMETERS TO BE MEASURED METHOD REFERENCES

Sample Type	Parameter Description	Method	Title	Method Reference
Water	Total Recoverable Metals:			
	Arsenic	3020/7060	Graphite Furnace Atomic Adsorption Spectroscopy	Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., U.S. EPA, Nov. 1986
	Barium	6010	Inductively Coupled Plasma Atomic Emission Spectroscopy	Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., U.S. EPA, Nov. 1986
	Cadmium	6010	Inductively Coupled Plasma Atomic Emission Spectroscopy	Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., U.S. EPA, Nov. 1986
	Chromium	6010	Inductively Coupled Plasma Atomic Emission Spectroscopy	Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., U.S. EPA, Nov. 1986
	Lead	6010	Inductively Coupled Plasma Atomic Emission Spectroscopy	Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., U.S. EPA, Nov. 1986
	Mercury	7470	Mercury in Liquid Waste (Manual Cold-Vapor Technique)	Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., U.S. EPA, Nov. 1986
	Selenium	3020/7740	Graphite Furnace Atomic Absorption Spectroscopy	Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., U.S. EPA, Nov. 1986
	Silver	6010	Inductively Coupled Plasma Atomic Emission Spectroscopy	Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., U.S. EPA, Nov. 1986

TABLE 5.3 PARAMETERS TO BE MEASURED METHOD REFERENCES (CONTINUED)

Sample Type	Parameter Description	Method	Title	Method Reference
	Digestion	3020	Digestion for metals, GFAA	Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., U.S. EPA, Nov. 1986
	Digestion	3005	Digestion for metals, ICP	Test Methods for Evaluating Solid Waste, SW-846, 3rd ed., W.S. EPA, Nov. 1 1986
	Petroleum Hydrocarbons	418.1	Petroleum Hydrocarbons, Total Recoverable	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, 1983
	Explosives: RDX, HMX, PETN, 2,4-DNT 2,6-NDT, 2,4,6-TNT	86	Explosives in water	USATHAMA Method 86 Explosives in Water by HPLC, 12/27/82
All Travel Trip Blanks	Petroleum Hydrocarbons	418.1	Petroleum Hydrocarbons, Total Recoverable	Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020 1983

5.3 Sample Collection Methods

Other sections of this report presents specific requirements for the types, numbers, locations and analysis of samples to be collected as part of this study. Project-specific standard operating procedures for the collection of samples both for laboratory and field analysis are outlined below.

5.3.1 Groundwater Monitoring Wells

Groundwater monitoring wells will be installed as specified in the Site Specific Well Installation Plan for closure of nine burning pads at the Seneca Army Depot, Romulus, NY.

Each of the newly installed groundwater monitoring wells will be allowed to stand at least three days after development. Then, each well (the six newly installed wells and the seven existing wells) will be sampled once during this field sampling episode. As part of that sampling, certain field generated quality control samples will be collected and submitted to the laboratory as specified.

Before a sample is collected from a well, the standing water level as well as the total well depth will be measured and recorded to the nearest 0.01 foot. The well will then be pumped or bailed with cleaned equipment to remove a quantity of water

equal to at least five times the submerged volume of well casing. If the well does not recharge fast enough to permit removing five casing volumes, the well shall be pumped to dryness and subsequently sampled as soon as sufficient recharge has occurred.

A standard operating procedure for well sampling has been prepared for this project and is presented below.

Sampling Procedures - Groundwater Monitoring Wells

The equipment required for sampling includes:

1. Stainless steel weighted tape (or electronic measuring tape) to measure the water level. "Chalking" of the tape will not be allowed during this project.

2. Bladder, Fultz, or positive displacement hand pump to purge the well. Entire pump and discharge lines to be constructed of non-contaminating materials.

3. Dedicated Teflon bailers to collect the sample. Teflon bailers are closed top, 1.66" O.D., three feet in length, with ball valves at top and bottom.

4. Dedicated Teflon coated stainless steel leader line for bailers will be used at each well.

5. The laboratory will be instructed to provide 1L, amber, narrow-mouth bottles. 40 ml VOC vials filled so that no headspace is present will be used for the collection of volatile organic samples.

6. Sample bottles prepared by standard lab washing procedure, provided with all required preservatives in separate dropper bottles. The standard bottle preparation procedure is provided in Section 6.0.

7. Disposable polystyrene cups for collection of field monitoring aliquots. Temperature, pH, and conductivity will be monitored from these aliquots.

8. Thermometer meeting ASTM specifications.

9. pH meter.

10. Conductivity meter.

11. Fisher Scientific "buffer pak®" containing certified, dated, pH standards of 4.0, 7.0, and 10.0 pH units.

12. Single use KCl calibration standard for calibration of conductivity meter.

General: Sampling will proceed beginning with the well located in the area suspected of least contamination (up gradient) and proceeding to areas of most contamination.

Procedure

1. Calibrate pH meter with two buffers before each use. Select standard buffers which will bracket the pH of the sample to be measured. Calibrate conductivity meter with standard KCl solution according to manufacturer's instructions. Record in field book.

2. Unlock protective casing on well.

3. Using the decontaminated measuring tape (refer to SOP for decontamination, Section 6), measure to the nearest .01 foot and record in the field book the static groundwater level in the well in addition to the depth to the bottom of the well.

4. Using the measured depth of standing water and a well volume table for that diameter well, note and record the volume of well water corresponding to the measured depth of standing water.

5. Using a decontaminated purging pump or bailer, purge a minimum of five well volumes from the well. The volumes will be estimated by discharging all purge water to a calibrated container. During the purging, the temperature, conductivity, and pH will be measured and recorded after each well volume is removed from the well. Purging will be stopped after five well volumes have been pumped providing pH and conductivity readings have stabilized. (i.e., any given pH or conductivity reading demonstrates no more than a 10% change from the previous reading). A minimum of five readings is required. The discharged water is to be disposed of as described in the health and safety plan.

6. Using a decontaminated bailer and nylon rope, collect groundwater samples from the well. Record time at beginning and end of bailing. During sample collection, the rope and bailer should not touch the ground or any objects except the well casing. Personnel filling sample bottles must wear new PVC gloves.

7. Immediately transfer the groundwater sample directly from the bailer to the appropriate sample containers. Fill 40 ml VOC vials directly from the bailer so that no headspace is present. A decontaminated glass funnel may be used to facilitate sample transfer to all other bottles. DO NOT FILTER the samples.

8. Collect a final sample aliquot in a disposable container and immediately measure and record pH, temperature, and conductivity, for that sample.

9. Immediately label, tag, and place all sample bottles in iced coolers. Preserve samples as required, referring to Table 8.1. Record sampling details in field log book and complete chain of custody forms.

10. Repeat Steps 7 through 10 to satisfy any field duplicate sample requirements.

11. Remeasure the standing water levels in the well after sampling. Record measurements in field log book.

12. Decontaminate all sampling equipment according to Standard Operating Procedures. Obtain a sampling/field (Decon) blank before collection of the samples from the first well as follows:

    a. Use fresh, deionized, analyte-free water supplied in glass containers by the subcontract laboratory.

    b. Pour the deionized water through all decontaminated sampling equipment as in field sampling procedure.

    c. Dispense into proper sample containers; preserve as required.

    This sampling/field blank will verify that proper field equipment decontamination has been performed.

13. After all samples have been taken from a given well, decontaminate the pH, conductivity, and temperature monitoring equipment. The used bailer shall be placed in an appropriate contaminated equipment storage bag for transportation. Discard all contaminated gloves.

14. Replace protective cap and lock well.

15. Ship samples to lab according to Standard Operating Procedures for sample packaging and shipping.

5.4 Field Sampling/Monitoring Procedures

5.4.1 HNu Photoionization Detector

During the course of this investigation, the HNu will provide two services; field monitoring of organic vapors and field screening of soil samples for relative levels of volatile organic compounds. Field screening of soil samples will assist

in characterization of the vertical extent of contamination as well as indicate if special disposal of well cuttings is needed.

Instrument Operation

Each HNu will be prepared for use each day according to the following procedure:

1. Warm-up: 5-10 minutes

2. Battery check: Turn function switch to battery check position. The needle should reach within or above the green battery area on the scaleplate. If the needle is in the lower position of the battery arc, the instrument must be recharged prior to making any measurements. If the red LED comes on, the battery must be recharged.

3. Check UV Light Source: Turn the function switch to the "on" position. In this position, the UV light source should be on. Check by looking quickly into the end of the probe to see purple glow of the lamp.

4. Zero Instrument: Turn the function switch to the standby position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation of the zero potentiometer produces an upscale deflection while counterclockwise rotation yields a downscale deflection. If the span adjustment setting is changed after the zero is set, the zero should be rechecked and adjusted, if necessary. Wait 15 or 20 seconds to ensure that the zero reading is stable. If necessary, readjust the zero. The zero must be checked periodically during the day to check for zero drift and must be recorded or repaired as necessary.

5. Calibrate Instrument: Turn function switch to proper measurement range (0-20, 0-200, 0-2000 ppm). Attach regulator to HNu calibration gas cylinder. Attach tygon tubing from regulator to HNu probe. Open the valve of the pressurized container until a flow is indicated on the cylinder gauge. Adjust the span pot knob until the instrument is reading the exact value of the calibration gas concentration noted on the cylinder. (If the instrument span setting is changed, the instrument must be turned back to the standby position and the electronic zero readjusted as necessary).

6. Recharging Instrument: All HNu's must be recharged overnight after each day's use. To charge the battery, place the mini phone plug into the jack on left side of the bezel prior to plugging charger into 120 VAC. When disconnecting charger, remove from 120 VAC before removing mini phone plug. The battery is completely recharged overnight (14 hours). To ensure that the charger is functioning, turn the function switch to the battery check position, place phone plug into jack and plug charger into AC outlet. The meter should go upscale if charger is working and is correctly inserted into the jack.

5.4.2 Soil Sample Screening

Soil samples will be screened with the HNu by the inspecting hydrogeologist immediately after the split-spoon is opened during well installation. Screening involves passing the tip of the probe within approximately 1/4 inch of the soil surface (without touching) along the length of the split spoon. Peak values of the HNu must be recorded by depth in the inspecting geologist's field notebook.

6.0 Decontamination Procedures

The cleaning procedures outlined in this section are to be used by all M&E personnel to clean sampling and other field equipment as well as sample containers prior to field use. Sufficient clean equipment and sample containers should be transported to the field so that the entire investigation can be conducted without the need for cleaning equipment in the field. Since this will not always be possible when using specialized field equipment, field cleaning procedures are included to cover these special problem areas.

These procedures are the standard operating procedures (SOP) for this project; any deviation from them must be documented in field records and investigative reports.

6.1 Cleaning Materials

The cleaning materials referred to throughout this section are defined in the following paragraphs.

The laboratory detergent shall be a standard brand of laboratory detergent such as Sparkleen® or Liquinox®. The use of any other detergent must be justified and documented in the field log books.

The nitric acid solution shall be made from ACS reagent-grade nitric acid and deionized water.

The standard cleaning solvent shall be pesticide-grade isopropanol. The use of any solvent other than pesticide-grade isopropanol for equipment cleaning purposes must be justified and its use must be documented in field log books and inspection or investigation reports.

Tap water may be used from any municipal water treatment system. The use of an untreated or non-potable water supply is not an acceptable substitute for tap water.

Deionized water of "Type II Quality" must be used and is defined as tap water that has been distilled with a resulting conductivity of 1.0 μmho/cm or less and subsequently treated by passing it through a deionizing resin column. The Type II deionized water should contain no heavy metals or other inorganic compounds.

Organic-free water is defined as tap water that has been treated with activated carbon and deionizing units. Usually, commercial units utilize a 5-micron prefilter, activated carbon unit, two mixed bed deionizing units (in series), a 0.2 micron post filter, and a postcarbon filter to produce organic-free water.

Organic-free water should contain no detectable pesticides, herbicides, extractable organic compounds or metals, and less than 20 ug/l of common laboratory solvents. All other organic compounds should be below detection limits as measured by a low level GC/MS analysis. Distilled water purchased from local supply stores (e.g., supermarket) is not Type II reagent water or organic free. The field team will retain the analytical data or manufacturer's certification which verifies the quality of the Type II Reagent water used by the field team during this project.

The brushes used to clean equipment as outlined in the various sections of this procedure shall not be of the wire-wrapped type.

The solvent, nitric acid solution, laboratory detergent, and rinse waters used to clean equipment shall not be reused, except as specifically permitted in the footnote for Step 3, Section 6.3.3.

6.1.1 Marking of Cleaned Sampling Equipment and Containers

All equipment and sample containers that are cleaned utilizing these procedures shall be labeled or marked with the date that the equipment was cleaned. Also, if there was a

deviation from the standard cleaning procedures outlined in this section, this fact should be noted on the label.

When sample containers are cleaned and prepared, they should be cleaned in standard sized lots to facilitate the quality control procedures outlined in Section 6.2.

6.1.2 Marking and Segregation of Used Field Equipment

Field or sampling equipment that needs to be repaired shall be identified with a tag. Any problems encountered with the equipment and needed repairs shall be noted on this tag. Field equipment or reusable sample containers needing cleaning or repairs shall not be stored with clean equipment, sample tubing, or sample containers. Field equipment, reusable sample containers, disposable sample containers, and sample tubing that are not used during the course of this investigation may not be replaced in storage without being recleaned if these materials have been transported to the study site and herbicides, pesticides, organic compounds, or other toxic materials are suspected of being present.

6.1.3 Decontamination of Equipment Used to Collect Samples of Toxic or Hazardous Waste

Equipment that is used to collect samples of known hazardous materials or toxic wastes or materials from the project site shall be decontaminated before being returned from the field. At a minimum, this decontamination procedure shall consist of washing with laboratory detergent and rinsing with tap water.

6.1.4 Use of Safety Procedures to be Utilized During Cleaning Operations

The materials used to implement the cleaning procedures outlined in this SOP can be dangerous if improperly handled. Caution must be exercised by all personnel and all applicable safety procedures shall be followed. At a minimum, the following precautions shall be taken in the lab and in the field during these cleaning operations:

1. Safety glasses with splash shields or goggles, neoprene gloves, and a neoprene laboratory apron will be worn during all cleaning operations.

2. All solvent rinsing operations will be conducted under a fume hood or in the open (never in a closed room).

3. No eating, smoking, drinking, chewing, or any hand to mouth contact shall be permitted during cleaning operations.

6.2 Specific Quality Control Procedures for Cleaning Operations

6.2.1 Sampling Equipment Cleaned in the Field

The effectiveness of field cleaning procedures shall be monitored by rinsing field cleaned equipment with organic-free water and submitting the rinse water in standard sample containers to the laboratory for analysis (sampling/field or Decon blank). Any time equipment is cleaned in the field, at least one such quality control sample should be collected per day of sampling.

6.3 Cleaning Procedures for Teflon Equipment Used for the Collection of Samples for Trace Organic Compounds and/or Metals Analyses(1)

1. Equipment will be washed thoroughly with laboratory detergent and tap water using a brush to remove any particulate matter or surface film.

2. The equipment will be rinsed thoroughly with tap water.

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(1) When this sampling equipment is used to collect samples that contain oil, grease or other hard to remove materials, it may be necessary to rinse the equipment several times with pesticide-grade acetone or hexane to remove the materials and steam clean the field equipment before proceeding with Step 1. If the field equipment cannot be cleaned utilizing these procedures, it should be discarded.

3. Rinse equipment with at least a 10 percent nitric acid solution.(2)

4. Rinse equipment thoroughly with deionized water.

5. Rinse equipment twice with solvent and allow it to air dry.

6. Wrap equipment completely with solvent rinsed aluminum foil to prevent contamination during storage and/or transport to the field.

7. Rinse the Teflon sampling equipment thoroughly with tap water in the field as soon as possible after use.

6.4 Cleaning Procedures for Stainless Steel Equipment Used for the Collection of Samples for Trace Organic Compounds and/or Metals Analyses(1)

1. Wash equipment thoroughly with laboratory detergent and tap water using a brush to remove any particulate matter or surface film.

2. Rinse equipment thoroughly with tap water.

3. Rinse equipment thoroughly with deionized water.

4. Rinse equipment twice with solvent and allow to air dry.

5. Wrap equipment completely with solvent rinsed aluminum foil to prevent contamination during storage and/or transport to the field.

6. Rinse the stainless steel or metal sampling equipment thoroughly with tap water in the field as soon as possible after use.

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(2) Small and awkward equipment such as bottle lid inserts and well bailers may be soaked in the nitric acid solution instead of being rinsed with it. Fresh nitric acid solution should be prepared for each cleaning session.

6.5 Miscellaneous Equipment Cleaning Procedures

6.5.1 Well Sounders or Tapes Used to Measure Ground Water Levels

1. Wash with laboratory detergent and tap water.
2. Rinse with tap water.
3. Rinse with deionized water.
4. Draw tape through solvent soaked "Kimwipe" towel while rewinding.
5. Equipment should be wrapped with aluminum foil to prevent contamination during storage or transit.

6.5.2 Ice Chests and Shipping Containers

All ice chests and reusable containers will be washed with laboratory detergent (interior and exterior) and rinsed with tap water and air dried before storage. In the event that an ice chest becomes severely contaminated with concentrated waste or other toxic material, it shall be cleaned as thoroughly as possible and disposed of properly.

6.5.3 Vehicles

All vehicles utilized by M&E should be washed at the conclusion of each field trip. This routine maintenance should minimize any chance of contamination of equipment or samples due to contamination of vehicles. A thorough interior and exterior

cleaning is mandatory at the conclusion of such investigations. It shall be the responsibility of the Project Engineer to see that this procedure is followed.

6.6 Preparation of Sample Containers

6.6.1 General

No sample container will ever be reused. All sample containers will be stored in their original packing cartons. When packages of uncapped sample containers are opened, they will be placed in new plastic garbage bags and sealed to prevent contamination during storage. Specific precleaning instructions for sample containers are given in the following sections.

6.6.2 One-Half and One-Liter Amber Glass Bottles (Water Samples), 8; 16; and 32-Ounce Clear Widemouth Jars (Soil, Sediment), with Teflon® Lined Caps for Organic Compounds (Excluding Purgeables) and Metals Analysis

1. Wash bottles and jars, Teflon® liners, and caps in hot tap water and laboratory detergent.
2. Rinse three times with tap water.
3. Rinse with at least 10% nitric acid solution.
4. Rinse three times with deionized water.
5. Rinse bottles, jars, and liners (not caps) with solvent.

6. Oven dry bottles, jars, and liners at 125°C overnight, allow to cool.

7. Place liners in caps and cap containers without touching interior surfaces.

8. Store containers in contaminant-free area.

6.6.3 40 ml Glass Vials for Water Samples (Purgeable Organic Compounds Analysis) and 4-Ounce (120 ml) Clear Widemouth Glass Jars with Teflon® Liner for Soil Samples (Purgeable Organic Compounds Analysis)

1. Wash vials, bottles and jars, Teflon® liners and septa, and caps in hot tap water and laboratory detergent.

2. Rinse all items with deionized water.

3. Oven dry at 125°C overnight.

4. Allow all vials, bottles, jars, liners, and septa to cool in an enclosed contaminant-free environment.

5. Seal vials, bottles, and jars with liners or septa as appropriate and cap.

6. Store vials, bottles, and jars in a contaminant free area.

6.6.4 One Liter Polyethylene Bottle for Metals

1. Wash Polyethylene bottles and caps in hot water with laboratory detergent.

2. Rinse both with at least 10% nitric acid solution.

3. Rinse three times with deionized water.

4. Invert bottles and dry in contaminant free environment.

5. Cap bottles.

6. Store in contaminant free area.

6.6.5 One Liter Polyethylene Bottle for General Inorganics (nitrate)

1. Wash Polyethylene bottles and caps in hot water with laboratory detergent.

2. Rinse three times with deionized water.

3. Invert bottles and dry in contaminant free environment.

4. Cap bottles.

5. Store in contaminant free area.

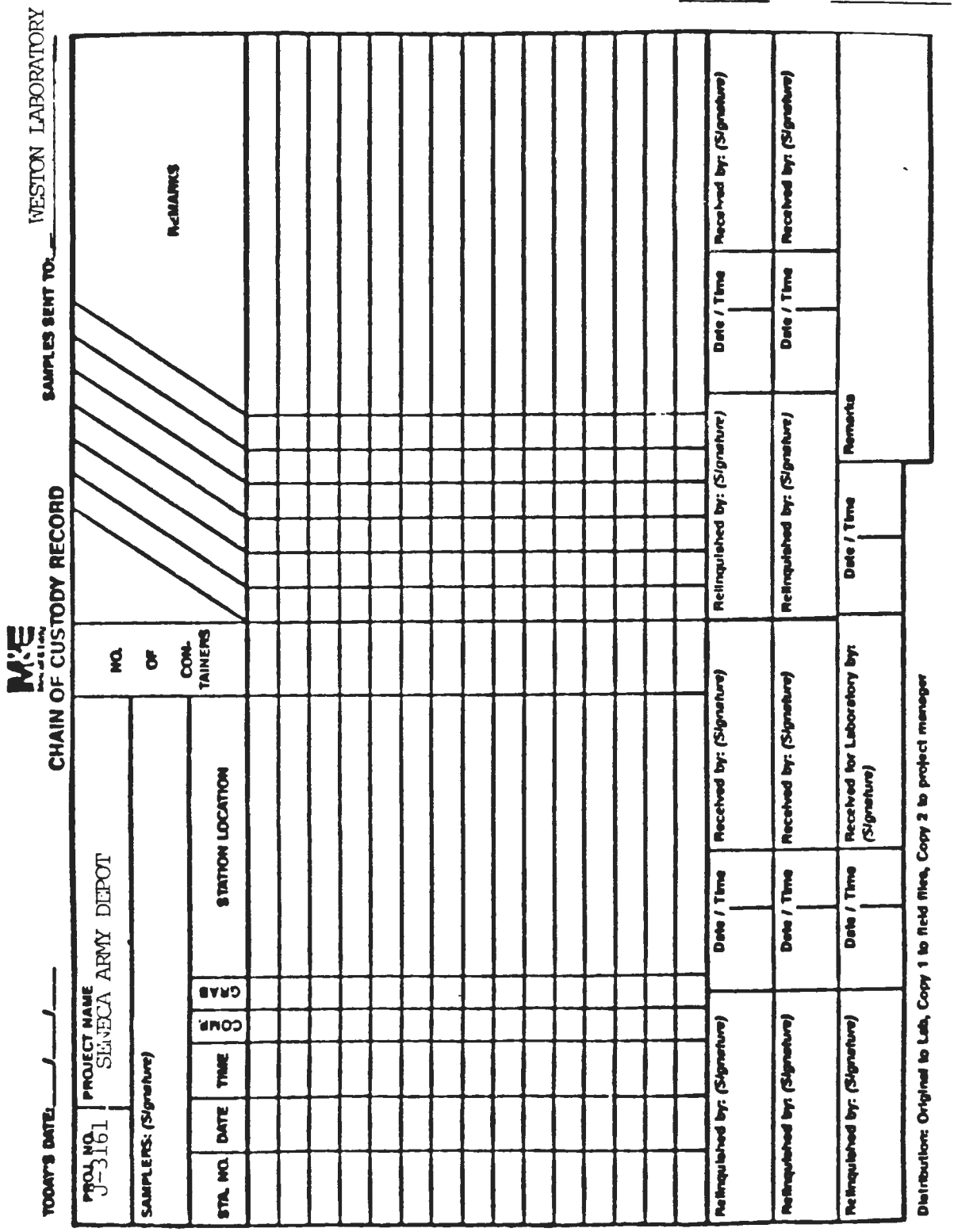
7.0 Sample Handling and Chain of Custody

An overriding consideration for environmental measurement data is the ability to demonstrate that samples have been obtained from the locations stated and that they have reached the laboratory without alteration. Evidence of collection, shipment, laboratory receipt and laboratory custody until disposal must be documented to accomplish this. Documentation is accomplished through a chain of custody record that records each sample and the individuals responsible for sample collection, shipment, and receipt. A sample is considered in custody if it is:

- In a person's actual possession.
- In view after being in physical possession.
- Locked so that no one can tamper with it after having been in physical custody.
- In a secured area, restricted to authorized personnel.

Sample custody will be initiated by Metcalf & Eddy field personnel upon collection of samples. Documents specifically prepared for such purposes will be used for recording pertinent information about the types and numbers of samples collected and shipped for analysis. An example chain of custody form is included as Figure 7.1. The samples collected will first be brought to an on-site location for batching and paperwork

FIGURE 7.1
CHAIN OF CUSTODY FORM



checks. This task includes the matching of like sample types (solids, liquids) with similar sample types from all sample locations. Labels, tags and log information are checked to be sure there is no error in identification. Samples are packaged to prevent breakage or leakage, and labeled according to DOT regulations for transport by air as laboratory samples. These procedures are outlined in Section 9. Copies of forms will be maintained for the project record. Storage of samples by the laboratory will be under conditions specified for the analyses to be performed. Samples used for analysis will be held for 60 days following report of the data before disposal. Samples are handled by the laboratories as described in the following paragraphs.

7.1 Chain of Custody Record Form

Figure 7.1 is the chain of custody form to be used by Metcalf & Eddy personnel in collecting and shipping samples associated with this study.

The chain of custody form shall be signed by each individual who has had the samples in their possession. Preparation of the chain of custody form shall be as follows:

- The chain of custody record shall be initiated for every sample by the person collecting the sample. All chain of custody form entries will be made with indelible ink. Every sample shall be assigned a unique identification number that is entered on the chain of custody form. Samples can be grouped for shipment using a single form.

- The record shall be completed in the field to indicate project, sampling team, etc.

- The record shall indicate how samples were transported to the lab (i.e., Federal Express, Purolator, etc.).

- Because the samples are to be shipped to the laboratory by commercial carrier, the chain of custody form shall be sealed in a watertight envelope, placed in the shipping container, and the shipping container sealed prior to being given to the carrier.

- The commercial carrier's airbill shall serve as an extension of the chain of custody record between the final field custodian and receipt in the laboratory.

- Upon receipt in the laboratory, the Quality Control Coordinator, or representative, shall open the chain of custody record, and sign and date the record. Any discrepancies shall be noted on the chain of custody form.

- If discrepancies occur, the samples in question shall be segregated from normal sample storage and the field personnel immediately notified.

- Chain of custody records shall be maintained with the specific project files, becoming part of the permanent project documentation.

7.2 Field Collection and Shipment

In addition to initiating the chain of custody form, field personnel are responsible for uniquely identifying, (required on the chain of custody form) labeling, tagging, providing proper preservation, and packaging samples to preclude breakage during shipment.

Every sample shall be labeled:

- Project number and site name.
- Unique sample number.
- Sample description
- Sampling date and time.
- Initials of person obtaining the sample.
- Method of sample preservation/conditioning, if any.

Every sample tag shall include:

- Sampler's initials
- Project number and site name.
- Unique sample number.
- Sampling date and time.
- Method of sample preservation, if any.
- Sample description

Prior to sample collection, labels will be affixed to sample containers using transparent tape. Indelible waterproof ink will be used for all logbook, sample label, and sample tag entries.

Samples must be placed in containers compatible with the intended analysis and properly preserved. Requirements for various analytical parameters with respect to the type of container, preservation method, and maximum holding time between collection and analysis have been presented in other sections.

Shipping containers are to be sealed prior to shipment, both during direct transport via field personnel as well as when commercial carrier is used. The only exception to this is if sufficient holding time exists so that the samples can be held in the field and it is necessary to re-ice the containers prior to or during transport.

As soon as field personnel are ready to transport samples from the field to the laboratory, they shall notify the laboratory by telephone of the shipment. The estimated time of arrival at the laboratory should be given.

7.3 Laboratory Sample Receipt

Upon sample receipt, the QC Coordinator or his designee shall:

- Examine all samples and determine if proper temperature has been maintained during shipment. If samples have been damaged during shipment, the remaining samples shall be carefully examined to determine whether they were affected. Any samples affected shall also be

considered damaged. It will be noted on the chain of custody record that specific samples were damaged and that the samples were removed from the sampling program. Field personnel will be notified as soon as possible that samples were damaged and that they must be resampled, or the testing program changed.

- Compare samples received against those listed on the chain of custody.
- Verify that sample holding times have not been exceeded.
- Sign and date the chain of custody form and attach the airbill to the chain of custody.
- Assign unique sequential weston sample numbers which will identify each sample in the laboratory's internal tracking system.
- List the samples in the laboratory sample master log-in book which contains the following information:
  - Project identification number
  - Sample numbers
  - Type of samples
  - Date received in laboratory
- Notify the Laboratory Manager of sample arrival.
- Place the completed chain of custody records in the project file.

7.4 Laboratory Storage of Samples.

The primary considerations for sample storage are:

- Maintenance of prescribed temperature. Typically four degrees celcius is required.
- Extracting and/or analyzing samples within the prescribed holding time for the parameters of interest.

Placement of samples in a secure and proper storage environment is the responsibility of the sample custodian.

7.5 Sample Retention and Disposal

Samples will be retained in the refrigerator for thirty (30) calendar days after the date of the invoice accompanying the analytical results. Unless a written requrest is received for retaining the sample beyond the thirty (30) days, the samples will be disposed of in an appropriate manner.

8.0 Sample Containers and Preservation Requirements

Table 8.1 presents container requirements, preservation specifications and laboratory holding times to be adhered to during all sampling and analysis activities associated with this project.

TABLE 8.1
SAMPLE CONTAINER AND PRESERVATION REQUIREMENTS

Parameter	Container(1)	Preservation(2)	Holding Time(3)
	AQUEOUS SAMPLES		
Petroleum Hydrocarbons	1L, G, teflon-lined lid	1:1 HCL, 5ml/L Cool 4°C	Unspecified
Metals	1L, P, teflon-lined lid	HNO_3, pH<2	6 Months(4)
Explosives	500 mL, G, amber	None	7 Days

(1) G, glass; P, High density Polyethylene with unlined polyethylene cap.

(2) Sample preservation will be performed in the field immediately upon sample collection. Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Some samples may not be stable for the maximum time period given in the table. A laboratory is obligated to hold the samples for a shorter time if knowledge exists to show that this is necessary to maintain sample stability.

(3) As presented in SW 846, 3rd edition, Table 2-16 and 4-1.

(4) 28 day holding time for mercury.

9.0 Sample Packaging and Shipping

In order to ensure safe, secure delivery of all collected samples to the Weston laboratories, the following packaging, labeling and shipping procedures have been prepared for this project. All procedures presented below are written to comply with applicable DOT regulations for transportation by surface and air.

All samples will be surveyed visually as well as with the on-site monitoring equipment to determine the likelihood of sample contamination. These instruments will include air monitoring devices such as HNu PI-101 total hydrocarbon analyzers and Draeger stain detector tubes, which can be used to survey monitoring well and sample container headspace for organic vapors. Conductivity and pH meter readings will be taken from liquid samples to further evaluate the hazardous nature of these samples. This data will be recorded in the field logbook as documentation of the shipping procedures used.

Unless such field collected information indicates otherwise, all environmental samples collected at the SEAD site will be treated as non-hazardous aqueous liquids and non-hazardous soil.

Because of the expected non-hazardous nature of the collected samples, packaging and shipping criteria have been

designed only to maintain chain-of-custody protocol as well as prevent breakage of the sample containers.

9.1 Non-hazardous Packaging and Shipping - Field Procedure

1. Wrap properly labeled and secured glass sample bottles and purgeable vials with plastic bubble wrap. Place the wrapped containers into a watertight zip lock bag. Seal.

2. Put a layer of cushioning material (e.g., vermiculite) in the bottom of the watertight metal or equivalent strength plastic shipping containers.

3. Place sample bottles, tops up, in the shipper. Arrange bottles such that glass bottles are surrounded by plastic bottles.

4. Use pieces of rigid styrofoam as necessary to ensure that there will be no shifting of bottles during transport.

5. Fill void space around and on top of the sample bottles with ice cubes or chips sealed in plastic bags.

6. Seal chain of custody forms in a zip-lock plastic bag and tape securely to the inside of the cooler lid.

7. Close and lock or latch the shippers. Seal the space between the container body and lid with waterproof tape.

8. Apply several wraps of pre-printed chain of custody tape around the shipping containers perpendicular to the seal to assure that the lid will remain closed if the latch is accidentally released or damaged and to prevent tampering during shipment.

9. If the shipping container used is a picnic cooler, tape the drain plug closed so it will not open.

10. Place a completed Federal Express Airbill on the lid of the cooler, including name, address and phone number of the receiving laboratory and the return address and phone number of the shipper.

11. Place a "This End Up" label on the lid and on all four sides of the shipper.

Samples will be shipped to the laboratory via Federal Express Priority 1.

Samples will be shipped from the Federal Express Office which is closest to the site or at the closest airport. Samples will be shipped directly to the Weston laboratory. One of the M&E field team members will deliver the properly labeled sample packages to the Federal Express Office or the appropriate airport. Office hours are normally 8:00 am to 6:00 pm, Monday through Friday and 8:00 am to 5:00 pm on Saturday. Federal Express may provide sample pick-up on-site for this project. The field team will investigate this possibility at the time of sample collection.

10.0 Data Quality Objectives for Measurement Data

The U.S. EPA specifies five major characteristics of data quality which must be addressed in environmental sampling and analytical projects. These are:

1. Accuracy: The degree of agreement of a measurement (or measurement average) with an accepted reference or true value. It is a measure of system bias and is usually expressed as the difference of measured from true values, or as a percentage of the difference.

2. Precision: A measure of agreement among individual measurements of the same property under similar conditions. It is expressed in terms of relative percent difference between replicates or in terms of the standard deviation when three or more replicate analyses are performed.

3. Completeness: A measure of the amount of valid data obtained compared to the amount expected to be collected under normal correct conditions. It is usually expressed as a percentage. The completeness objective will be calculated on those samples reaching the laboratory intact, not the total number of samples collected, since breakage during transit can occur.

4. Representativeness: Expresses the degree to which data accurately and precisely represents a characteristic of a data population, process condition, a sampling point, or an environment. For this project, grab samples are taken from surface and groundwater sources. Soil samples are to be collected, together with sediment samples. Grab samples are by definition representative of only the conditions at the point in time collected, within sampling and analysis error.

5. Comparability: Expresses the confidence with which one data set can be compared to another. To achieve comparability in this project, the data generated will

be reported using units of ug/l and mg/kg. Analytical results will be comparable to that produced from similar labs using the same instrumentation and methodology. Standard Reference Materials will be used to document traceability of calibration standards, and allow comparison of data across laboratories performing analyses.

The QA objectives for this project in terms of precision, accuracy, and completeness are listed in Table 10.1.

Method quantitation limits expected to be achieved during this work are shown in Table 10.2.

QA objectives for the explosives in water are defined as averages expected for the group of compounds measured. Any spike and surrogate compounds used will be those specified in the analytical methods used.

QA objectives for metals are defined as averages for groups of spiked compounds when analyzed by ICP and by individual metal for atomic absorption furnace analysis. EPA inorganic acceptance limits will be applied to samples that are split with another lab.

Metals include arsenic, barium, cadmium, chromium, lead, mercury, selenium and silver. The explosives in water include: RMX, HDX, PETN, 2,6-DNT, 2,4-DNT and 2,4,6-TNT. "Petroleum hydrocarbons" is a measure of non-polar freon extractable materials.

The QA objective for this work is that all measurements be representative of the actual site conditions and, that all data resulting from sampling and analysis activities be comparable. The use of accepted, published sampling and analysis methods as well as standard reporting units will aid in ensuring the comparability of the data.

TABLE 10.1. PRECISION, ACCURACY, COMPLETENESS OBJECTIVES
AQUEOUS SAMPLES

Measurement Parameter	Method No.(1)	Precision(2)	Accuracy(3)	Completeness(4)
Arsenic	7060	20%	80%	90%
Barium	6010	20%	80%	90%
Cadmium	6010	20%	80%	90%
Chromium	6010	20%	80%	90%
Lead	6010	20%	80%	90%
Mercury	7470	20%	80%	90%
Selenium	7740	20%	80%	90%
Silver	6010	20%	80%	90%
Petroleum Hydrocarbons	418.1	30%	(not applicable)	90%
HMX	8G	20%	85%	90%
RDX	8G	20%	85%	90%
PETN	colorimetric	20%	85%	90%
2,4,6-TNT	8G	20%	85%	90%
2,6-DNT	8G	20%	85%	90%
2,4-DNT	8G	20%	85%	90%

(1) 100-500 METHODS
METHODS FOR CHEMICAL ANALYSIS OF WATER AND WASTES
U.S. EPA
Environmental Monitoring and Support Laboratory
Cincinnati, OH 45268
EPA 600/4-79-020

6000-9000 METHODS
TEST METHODS FOR EVALUATING SOLID WASTE
U.S. EPA
Office of Solid Waste Management and Emergency Response
Washington, DC 20460
November, 1986; Third Edition

USATHAMA
Method 8G
Explosives in Water by HPLC, 12/27/82

TABLE 10.1. PRECISION, ACCURACY, COMPLETENESS OBJECTIVES
AQUEOUS SAMPLES (CONTINUED)

(2) Expressed as Relative Percent Difference (RPD) (or Average RPD for a group of compounds analyzed together during a single analysis such as metals by ICP) of duplicate measurements made on a single laboratory sample.

(3) Expressed as Percent Recovery of analyte added to actual samples as part of a matrix spike and matrix spike-duplicate (MS/MSD) regime.

(4) Completeness is a measure of the amount of valid data obtained from a measurement process compared to the amount of valid data that was planned to be taken to achieve a particular statistical level of confidence in the data resulting from that measurement process. This value is usually presented as a percent.

TABLE 10.2. PROJECT-SPECIFIC QUANTITATION LIMITS

Metals Analyses	Water (ug/l)
1. Arsenic	10
2. Barium	200
3. Cadmium	5
4. Chromium	30
5. Lead	50
6. Mercury	2
7. Selenium	10
8. Silver	30

Miscellaneous Wet Chemistry	Water (mg/l)
Petroleum Hydrocarbons	0.2

Explosives in Water	Water (ug/l)
1. RDX	0.63
2. HMX	1.3
3. PETN	0.66
4. 2,4-DNT	0.60
5. 2,6-DNT	0.55
6. 2,4,6-TNT	0.78

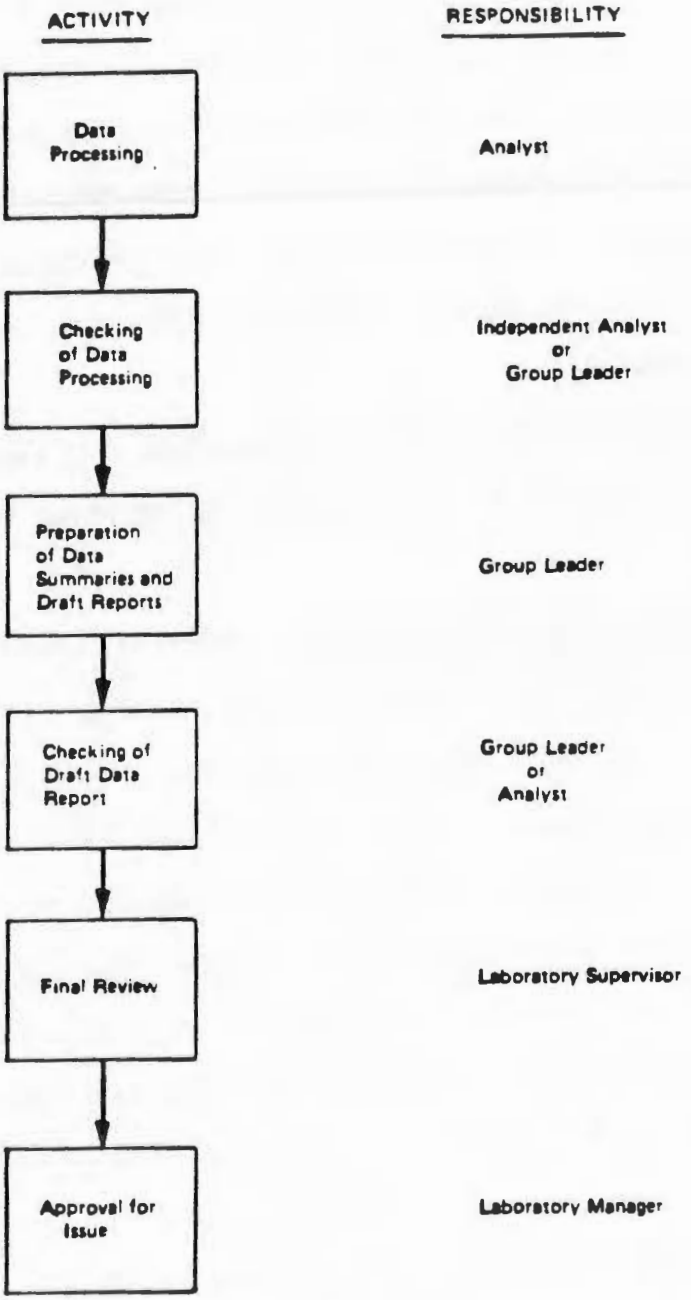
11.0 Data Reduction, Validation, and Reporting

This section describes data reduction, validation, and reporting procedures which will be used at the Weston laboratory participating in this project. Primary responsibility for implementation of these procedures within each laboratory will reside with the Laboratory Manager. The principal points of contact between the Metcalf & Eddy Project Manager and QA Project Officer will be the Weston Laboratory Manager and the Quality Assurance Manager.

Final responsibility for validation and reporting of data will reside with the Metcalf & Eddy QA Project Officer.

11.1 Laboratory Data Validation. Data validation begins with the processing of data and continues through review of the data and the reporting of analytical results. Data processing can be performed by the Analyst who obtained the data or another Analyst. Data review starts with an Analyst independent of the data acquisition and processing, or the Group Leader, reviewing (validating) that data processing has been correctly performed and continues through verifying that the reported analytical results correspond to the data acquired and processed. Final review of the data to be reported is by the Laboratory Supervisor. The procedure is outlined in Figure 11.1

Figure 11.1 Data Validation Flowchart



As stated, the first step in validation is data processing. In general, data will be processed by an Analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets.
- Input of raw data for computer processing.
- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an Analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors, dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, calculations should be done on standard Weston calculation paper and attached to the data sheets. The Analyst shall sign (full signature) and date in ink each page of calculations.

For data that are input by an Analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed shall be evident and the input signed and dated by the Analyst.

If data are directly acquired from instrumentation and processed, the Analyst shall verify that the following are

correct: project and sample numbers, calibration constants and response factors, output parameters such as units, and numerical values used for detection limits (if a value is reported as less than). The Analyst shall sign and date the resulting output.

11.1.1 Review of Data Processing. Following is a discussion of the method to be used for reviewing (checking) data processing. At least 20 percent of all data shall be checked in this manner. If, during the checking process, errors are determined, checking shall be completely (100 percent) performed for the data set.

- The Analyst performing the data processing shall give an Analyst independent of the work the data package. The package shall include, as appropriate, raw data, data sheets, strip charts, computer input/output, calculations, sources for input parameters such as response factor, etc.
- The independent Analyst (checker) shall review the data for:
  - Appropriateness of equations used.
  - Correctness of numerical input.
  - Numerical correctness of all calculations. This should be done by reperforming 100% of all numerical computations.
  - Correct interpretation of strip charts, etc.

- All entries and calculations that the checker reviews shall be marked in ink with a check mark. The checking process must be thorough enough to validate that the results are correct. If the checker disagrees with any part of the computations, the checker shall mark through the number with a single line and place the revised number above it.

- Any changes made by the checker shall be backchecked by the originator. If the originator agrees with the change, no action is necessary. If the originator disagrees, the originator and checker must resolve the difference so they agree with the result presented. Actions taken to resolve such discrepancy must be noted.

- The checker shall sign originals and date in ink all pages of the data package (except for groups of printout such as chromatograms). Signing and dating indicates that reviewer agrees with the calculations and that any changes made have been agreed to by the originator.

- If the data have been processed by computer, the reviewer shall check every input entry. Agreement should be indicated by a check mark for every line. If the checker disagrees with the input, the number should be marked through with a line and corrected number indicated above it. Corrections must be backchecked by the originator as discussed above.

- If an input error is identified and the data have been processed, it will be necessary to reprocess the data. In this event, the checker shall mark the second set of input to indicate agreement with the input changes. The checker shall sign and date in ink the computer input to indicate agreement.

- Raw data that are automatically acquired and processed do not require any validation at this point beyond that previously discussed.

11.1.2 Review of Data Reporting. Review of data reports is required to verify that information reported by the laboratory corresponds with processed analytical results. Review is only required of the data as it is presented for issuance. Intermediate steps performed after the processed data are checked to prepare the data report (such as data summaries) do not require validation. Preparation of the report is the responsibility of the Laboratory Manager or his designated representative.

After the draft data report is prepared, the reported results should be checked against the reviewed processed data so that transcription errors do not occur. The checking process follows:

- Using the draft report, all data entries are checked by an Analyst. The checker is not required to be independent of the work because only the transcription from the reviewed data to the data report is being checked.
- The draft data report should be checked so that the items cited for data presentation in Section 11.2 are complete and correct. As the reviewer checks the entries on the draft report, an ink check mark is placed beside each correct entry. Corrected entries are marked through with a single line and the correct entry provided. The reviewer will indicate that corrections have been made in the report by placing a second check mark by the correction after comparing the change with the revised copy. The checker shall sign and data every page of the data report in ink.

- Use of the draft data report results in a checkprint which should be maintained as a record to demonstrate the review.
- If data printouts, such as chromatograms or GC/MS data processing, are included in the data report, review is not required for the data printout.
- If computer output is used directly as the data report without further transcription, only the input requires review as discussed in Section 11.1.1.

After checking of the data report is complete, it is given to the Laboratory Manager or his designated representative for final review. This step is not intended to verify the reported data. This review is intended to determine that the report meets project requirements. The data report is approved for issue by the Laboratory Manager.

11.2 Laboratory Data Reporting. The following are applicable to data presentation:

- The final presentation shall be checked in accordance with data verification requirements of Section 11.1.2 and approved by the Laboratory Manager.
- Data will be presented in a tabular format whenever possible.
- Data will be formatted as a standard lab report such as shown in the example attached to this document as Appendix A-2.

- Each page of the data will be identified with the project number and name; date of issue; and project-site name.
- As a minimum, data presentation will include:
  - Sample identification number used by the Weston laboratory and/or the sample identification provided to the laboratory, if different than identification used in the laboratory.
  - Chemical parameters analyzed, reported values, and units of measurement.
  - Detection limit of the analytical procedure if the reported value is less than the detection limit.
  - Data for a chemical parameter are reported with consistent significant figures for all samples.
  - Results of Quality Control sample analysis if appropriate.
  - Achieved accuracy, precision, and completeness of data.
  - Footnotes referenced to specific data if required to explain reported values.
- Data will be transmitted from the laboratory only by the Laboratory Manager.

If explanatory text is not issued with the analytical results, a letter of transmittal will be included.

The letter of transmittal will include:

- Person(s) receiving the data
- Person transmitting the data

- Document the chain of custody, specifically if it was not provided or correct, if any samples were damaged in shipment, if sample containers were inappropriate for analysis, or samples not preserved, and if volume provided was inadequate for proper analysis
- Brief discussion of samples analyzed and the analytical program
- Discussion of any apparent data anomalies
- Discussion of any analytical difficulties and follow-up actions taken to remedy the difficulty.

11.3 Field Data Quality Reviews

A simple program designed to ensure field performed analyses yield valid, useful data is summarized below. In all cases, the Field Team Leader will maintain a concise, detailed field logbook containing accounts of all field activities and actions taken.

Objective	Action	Responsible Person
1. Sample and location information conforms to conditions and requirements in Table 5.1	Review of labeled samples and in-process samples using daily sample inventory	Field Sampling team
2. Verify incoming field data and sample completeness	Daily count of incomplete items	Field Sampling team

Objective	Action	Responsible Person
3. Verify completeness of field log books	Review Daily	Field Team Leader
4. Field calibration criteria reviewed and test calibration acceptance recorded	As Necessary	Field Team Leader
5. All data forms are properly completed	Review and check off during each sample collection	Field Team Leader
6. All field generated QC samples collected as required	Review requirements and confirm	Field Team Leader

12.0 Calibration Procedures and Frequencies

This section describes calibration procedures and policies pertinent to this project.

Instruments and equipment used in the Weston laboratory are controlled by a formal calibration program. The program verifies that equipment is of the proper type, range, accuracy, and precision to provide data compatible with specified requirements. All instruments and equipment which measure a quantity, or whose performance is expected at a stated level, are subject to calibration. Calibration may be performed by Weston personnel using reference standards, or externally by calibration agencies or equipment manufacturers.

Implementation of the laboratory calibration program is the responsibility of the Laboratory Manager and Analysts. The Quality Control Coordinator shall review the implementation of the program.

Two types of calibration are discussed in this section:

- Operational calibration which is routinely performed as part of instrument usage, such as the development of a standard curve for use with an atomic absorption spectrophotometer. Operational calibration is generally performed for instrument systems.
- Periodic calibration which is performed at prescribed intervals for equipment, such as balances and ovens. In general, equipment which can be calibrated periodically

is a distinct, single purpose unit and is relatively stable in performance.

12.1 Calibration Procedures

Whenever possible, recognized procedures, such as those published by ASTM or the USEPA, or procedures provided by manufacturers shall be utilized.

At a minimum, the procedures shall include:

- Equipment to be calibrated
- Reference standards used for calibration
- Calibration technique and sequential actions
- Acceptable performance tolerances
- Frequency of calibration
- Calibration documentation format.

12.1.1 Equipment Identification. Equipment that is subject to calibration shall be uniquely identified so that calibration records can be associated with a specific instrument.

12.1.2 Calibration Frequency. Instruments and equipment shall be calibrated at prescribed intervals and/or as part of the operational use of the equipment. Frequency shall be based on the type of equipment, inherent stability, manufacturer's recommendations, values provided in recognized standards, intended use, effect of error upon the measurement process, and prior experience.

12.1.3 Calibration Reference Standards. Two types of reference standards are used within Weston laboratories for calibration:

- Physical standards, such as weights for calibrating balances and certified thermometers for calibrating working thermometers and ovens, which are generally used for periodic calibration
- Chemical standards such as Standard Reference Materials (SRMs) provided by the National Bureau of Standards (NBS) which are primarily used for operational calibration.

Whenever possible, physical and chemical reference standards shall have known relationships to nationally recognized standards (e.g., NBS) or accepted values of natural physical constants. If national standards do not exist, the basis for the reference standard shall be documented.

12.1.4 Calibration Failure. Equipment that fails calibration or becomes inoperable during use shall be removed from service and segregated to prevent inadvertent use, or shall be tagged to indicate it is out of calibration. Such equipment shall be repaired and satisfactorily recalibrated before reuse.

12.1.5 Calibration Records. Records shall be prepared and maintained for each piece of equipment subject to calibration. Records demonstrating accuracy of reference standards shall also be maintained.

For instruments and equipment that are calibrated on an operational basis, calibration generally consists of determining instrumental response against compounds of known composition and concentration or the preparation of a standard response curve of the same compound at different concentrations. Records of these calibrations can be maintained in several ways:

- The calibration data can be kept with analytical sample data
- A log book can be prepared for each instrument which contains all calibration data.

The former method provides response factor information, etc., directly with analytical data so that the analytical data can be readily processed and verified. Also, the raw data package is complete as a unit.

The latter method provides an ongoing record of the calibration undertaken for a specific instrument; however, to process and verify the analytical data the log must be used in conjunction with the raw data.

For operational calibration of instrumentation used for this project, the following procedures will be followed:

- Calibration data will be included with the raw analytical data and maintained in project files.

12.2 Operational Calibration.

Operational calibration is generally performed as part of the analytical procedure. Included may be the analysis of a method blank and the preparation of a standard response (standard calibration) curve.

Following is a brief discussion of the analysis of method blanks and preparation of standard curves.

12.2.1 Method Blank. After determining the individual reagent or solvent blanks, the Analyst defines the method blank to determine if the cumulative blank interferes with the analysis. The method blank is defined by following the procedure step by step, including the addition of all of the reagents and solvents in the quantity required by the method during an analysis of "lab-pure", analyte-free water." If the cumulative blank interferes with the determination, steps must be taken to eliminate or reduce the interference to a level that will permit the combination of solvents and reagents to be used. If the blank interference cannot be eliminated, the magnitude of the interference must be considered when calculating the concentration of specific constituents in the samples analyzed.

A method blank should be determined whenever an analysis is made. The number of blanks is determined by the method of analysis and the number of samples analyzed at a given time.

12.2.2 Preparation of Standard Calibration Curve. Concurrent with preparation of reagent and method blanks, a standard calibration curve is prepared for the instrumentation. Preparation of a standard calibration curve is accomplished by using calibration standards. The process may be summarized as follows:

- Preparation of a standard calibration curve is accomplished by using calibration standards prepared by mixing the species to be analyzed into the solvent that is to be introduced into the instrument.
- The concentration of the calibration standards are chosen to cover the working range of the instrument.
- All sample measurements are made within this working range.
- The calibration curve is prepared by plotting instrument response versus concentration of the species analyzed.
- Concentrations of the sample prepared with the same procedure are read directly from the calibration curve or determined by interpolation.

12.3 Calibration of High Performance Liquid Chromatograph (HPLC)

Prior to the analysis of samples and after tuning criteria have been met, the HPLC system must be initially calibrated to

verify peak separation and retention times. The calibration standard concentration will be tabulated versus the peak height response for each calibration standard.

Working calibration standards will be prepared fresh daily. The working standards will include a blank and a minimum of five (5) concentrations to cover the anticipated range of measurement. At least one of the calibration standards will be at or below the desired instrument detection limit. The correlation coefficient must be at least 0.996 in order to consider the responses linear over a range. If a correlation coefficient of 0.996 cannot be obtained, additional standards must be analyzed to define the calibration curve. A midpoint calibration check standard will be analyzed each shift to confirm the validity of the initial calibration curve. The check standard must be within twenty (20) percent of the initial response curve to demonstrate that the initial calibration curve is still valid. For multi-analyte methods, the check standard may contain a representative number of target analytes Calibration data, to include the correlation coefficient, will be entered into laboratory notebooks to maintain a permanent record of instrument calibration.

12.4 Calibration of the Inductively Coupled Argon Plasma Spectrometer (ICAP) or the Atomic Absorption Spectrophotometer (AAS) or the Spectrophotometer, Infrared

The ICAP, AAS, and spectrophotometer are standardized for the analytes of interest by analyzing a set of calibration standards prepared by diluting a stock solution of known concentration. Three to five working standards are prepared by dilution of the stock standard. The concentration of the calibration standards is chosen so as to cover the working range of the instrument. Subsequently, all sample measurements are made within this working range. Once the working standards are prepared, they are analyzed on the ICAP, AAS, or spectrophotometer and the instrument response is calibrated to provide a direct readout in micrograms of analytes per milliliter of digestate or parts per million.

The calibration is accomplished by inputting the analyte concentration equivalent to the readout in absorbance/emission units during analysis of the working standards.

Once the instrument has been initially calibrated, the analysis of the working standards is repeated during sample analysis to confirm expected instrument response during analysis as well as to confirm the calibration settings. A typical analysis sequence is presented below:

- Working standards are prepared by dilution of a stock standard solution of the metal of interest.
- A calibration curve within the working range of the instrument is established by analysis of three to five working standards.
- The working standards are re-analyzed to confirm the calibration settings. If the calibration settings are not confirmed, the instrument is recalibrated.
- The samples are analyzed for the metal of interest.
- During sample analysis, a midpoint standard is analyzed to monitor instrument stability. If the analysis indicates the instrument calibration has changed, the instrument is recalibrated and the analysis is repeated.
- Following completion of the sample analyses, the working standards are re-analyzed to confirm calibration settings. If calibration settings are confirmed, the analysis is completed. However, if the calibration settings are not confirmed, the problem is corrected, and the analyses are repeated.
- Analysis data may be input (if available) into a computer data file for later calculation and normalization for matrix effects.

12.5 Periodic Calibration

Periodic calibration shall be performed for equipment such as balances, thermometers, ovens, and furnaces that are required in analytical methods, but which are not routinely calibrated as part of the analytical procedure. Documentation of calibration shall be kept for each equipment item.

Calibration requirements are determined within each CompuChem laboratory depending upon the equipment used and its operating function.

13.0 Internal Quality Control Checks

All analyses performed in support of this program will be done using standardized laboratory procedures. The QC program developed by Metcalf & Eddy will make use of QC samples which are both known and unknown, or "blind", to the laboratory, calibration check samples, method blanks, field blanks, trip blanks, ambient conditions blanks, and replicate aliquot analyses (duplicates). The various types of both field and laboratory generated QC samples are described below.

FIELD GENERATED QC SAMPLES

a. One (1) trip blank with every batch of VOC samples (both soils and water) sent to the laboratory. **Definition of trip blank:** a sample bottle is filled with ASTM Type II Reagent water, transported to the site, handled like a sample, and returned to the laboratory for analysis (trip blanks are not to be opened in the field). The trip blank for soils is Type II Reagent water, just as in the case of water samples.

b. One (1) set of equipment blanks for every day of sampling (all parameters analyzed). **Definition of Equipment Blank:** Type II Reagent water is poured into the sampling device, or pumped through it (in the case of sampling pumps), transferred to the sample bottle, and then transported to the laboratory for analysis. (Also called De-Con blanks).

c. Ten (10) percent field duplicates (all parameters analyzed) for all sample types. **Definition of Duplicate:** two samples collected independently at a sampling location during a single episode of sampling. Field duplicates shall be indistinguishable from other analytical samples so that personnel performing the analyses are not able to determine which samples are duplicates.

LABORATORY GENERATED QC SAMPLES

d. One (1) Laboratory Control Standard (LCS) for every batch of samples analyzed for all parameters. **Definition of LCS:** Solutions prepared by adding known quantities of EMSL-Cincinnati or NBS Standard Reference or independently prepared stock materials to deionized water. The LCS are routinely used to establish that an instrument or procedure is in Control before analysis of samples begins. The analyst notes that LCS result in the instrument logbook and on the Control chart; the result must be within Control limits before sample analysis begins. An LCS is normally carried through the entire sample preparation and analysis procedure.

e. One (1) calibration check sample (CCS) analyzed for each 10 samples analyzed sequentially for inorganic parameters. **Definition of CCS:** one of the working calibration standards is periodically re-analyzed and the subsequent values used to demonstrate that the original calibration is still valid.

f. One (1) method/reagent blank analyzed with each sample batch analyzed. **Definition of method Blank:** laboratory-pure, analyte-free water carried through the entire preparation and analysis procedure. Analytical results are corrected for the method blank values before they are reported.

g. One (1) holding blank is analyzed for each batch of volatile organic samples received. **Definition of Holding Blank:** Laboratory pure organic-free water is placed into four (4) VOA vials by laboratory personnel. Two of the four vials are sent with the empty sample containers into the field and are referred to as "Trip Blanks" as explained in item a. above. The other two vials remain in the laboratory sample bank. Upon return of the samples to the lab from the field, the samples are held in the sample bank with the holding blanks. If contamination found in the samples is suspected to have originated in the laboratory sample bank rather than in the field, the holding blanks can be analyzed to demonstrate if any contamination of samples by the lab sample bank environment occurred.

h. Ten (10) percent lab replicates (all parameters analyzed) for all samples. **Definition of Replicate:** a single

sample (e.g., one particular aliquot) is collected, then divided into two equal parts for the purpose of determining analytical precision. Replicate samples are often called "Splits" if each half of the replicate is sent to separate labs for independent analysis. Lab replicates can be indistinguishable from other analytical samples so that personnel performing the analyses are not able to determine which samples are replicates.

The type and frequency of analysis of each QC sample is shown in Table 13.1.

Field as well as laboratory generated blanks, duplicates, and standards are analyzed alongside samples to provide continuous quality control during the determination of trace constituents. The blanks are analyzed to provide data on possible carry-over contamination of samples by the collection, extraction or digestion process and also provide background concentration levels in the reagents used during sample preparation and analysis. The replicate analyses provide laboratory precision data while the certified standards provide a measure of accuracy. Field duplicate data provides insight into sample variability concerns.

TABLE 13.1 - QC SAMPLE TYPES, CRITERIA AND CORRECTIVE ACTIONS

Type	Purpose	Frequency	Criteria	Corrective Action
Trip Blank	Verifies no contamination during sample transport/storage	1/batch of VOC samples shipped	No compound of interest >5XDL	Data Qualified or resample
Holding Blank	Verifies no contamination during lab storage	1/batch of VOA VOC samples received	No compound of interest >5XDL	Data Qualified or resample
Method Blank	Verifies clean reagents and instrument systems and lab environment	1/batch of samples analyzed	No compound of interest >5XDL	Clean system, data qualified or reanalyze affected samples
Performance Evaluation Samples	Verifies analyst proficiency with method and instrumentation	>1/analyst during training	<20% RSD, >90% recovery	Additional training; check system
Laboratory Control Samples	System performance check	When sample matrix interference is suspected.	>90% recovery	Check system
Laboratory Replicate	Precision check	10% of samples received by lab	± 85%	Compare with field duplicates Check for matrix interferences
Calibration Check Sample	Verifies drift-free calibration	≥ 1/analysis day or as specified by method	± 10% original curve	Recalibrate, check system
Matrix Spikes and Duplicates (MS/MSD)	Checks recovery from real matrix	10% of samples unless specified otherwise by method	Specified in analytical methods	Check for matrix interferences

TABLE 13.1 - QC SAMPLE TYPES, CRITERIA AND CORRECTIVE ACTIONS

(Continued)

Type	Purpose	Frequency	Criteria	Corrective Action
Standard Reference Materials	Determine accuracy vs. "true value", show comparability	1/sample batch and matrix type	± 10%	Recalibrate, correct values
Internal Standards	Monitor instrument response; used for quantitation	Every GC and GC/MS sample and standard	± 50% average of original curve	Check system, matrix interferences, recalibrate
Surrogate Standards	Determine recoveries, control limits, matrix effects	All GC and GC/MS samples	Specified in analytical methods	Check for matrix interferences; reanalyze samples
Field Duplicate	Submit "blind" to lab to determine sample variability	10% of samples collected	± 20%	Compare to lab replicates; Check systems, matrix interferences possible
Equipment Blank (Decon blank)	Verifies effective decon procedures used in field	1/day/sampling team/matrix	BDL	Data Qualified or resample

14.0 Performance and Systems Audits

An audit is a systematic check to determine the quality of operation of some function or activity. There are two basic types of audits: (1) laboratory performance audits in which quantitative data are independently obtained for comparison with routinely obtained data in the measurement system; and (2) system audits of a qualitative nature that consists of on-site review of conformance with quality assurance/control procedures in all laboratory and field sampling chemical analysis activities.

Each Weston Laboratory participating in this project will receive at least one performance audit during the analysis of project samples. The Weston QA Manager will collect the information and reports from these audits conducted by the respective Weston Quality Control Coordinators for project files and followup as necessary. In addition, the Weston QA Manager will conduct at least one system audit at each lab during the period of performance. He may be accompanied by Metcalf & Eddy personnel and Corps of Engineers personnel, or the latter may conduct their own independent system audits.

The content and conduct of the audits is discussed below.

14.1 Performance Audits

The Quality Control Coordinator is responsible for the preparation of Quality Control samples, insertion into the sample stream, and analysis of the results. The samples are analyzed on a daily, ongoing basis and provide the means for demonstrating data quality by statistical analyses.

To complete internal laboratory reviews, the Quality Control Coordinator shall provide ongoing monitoring of laboratory operations. The review is conducted on behalf of the Laboratory Manager to verify that the laboratory Quality Assurance Program is implemented and functioning on a daily basis. The review is intended to be a spot check and should include:

- Sample maintenance
  - Are stated temperatures for sample storage provided?
- Calibration
  - Is calibration data documented in instrument log books, and as part of project data as required?
  - Do calibration results indicate a trend in instrument performance?
- Preventive maintenance
  - Are adequate spare parts available?
  - Do specific instruments have repeated maintenance problems?

  - Is preventive maintenance performed and properly documented?

- Receipt and storage of standards, chemicals, and gases

  - Are all reagents, chemicals, and gases purchased for use in the laboratory of adequate grade for the intended use?

  - Are certifications of material compositions provided when required?

  - Are materials adequately stored to prevent degradation?

  - Are materials kept beyond stated shelf life?

  - Are internal standards kept beyond stated shelf life?

- Data verification

  - Are data processed and validated as prescribed?

- Records management

  - Are the records of analyses complete and properly identified?

  - Are documents submitted to the record system in a timely manner and are they properly maintained?

Nonconformances observed by the Quality Control Coordinator shall be reported to the Laboratory Manager or Quality Assurance Manager, if necessary, for corrective action to be taken. The Quality Control Coordinator shall keep a log of nonconformances observed. The log shall document the nonconformance; date of

occurrence; reason for occurrence, if known; date of corrective action; and the corrective action taken.

14.2 System Audits

System audits may be conducted by the Quality Assurance Manager. These audits shall provide a thorough overview of implementation of the Quality Assurance Program within the laboratory. The audit will focus only on the performance of the laboratory for the project.

System audits will review operation of the laboratory and resulting documentation, including all items reviewed by the Quality Control Coordinator. Particular emphasis will be placed upon implementation of the Quality Control sample program and nonconformance log. Review of these aspects of the laboratory Quality Assurance Program should indicate trends adverse to data quality.

Audits by the Quality Assurance Manager shall be performed in the following manner:

- An audit plan shall be prepared which considers the activities of the specific laboratory. The audit plan shall be the basis for the audit and should define:
  - participating auditors, applicable documents, schedule, scope of laboratory activities.

A lead auditor will be responsible for the audit planning and performance. As necessary, technical specialists will assist the audit team in preparation and conduct of the audit. All persons participating in the audit team will be independent of the laboratory audited.

- Based on the audit plan, detailed checklists of questions to be asked during the audit will be prepared. The checklists will provide adequate means for indicating whether or not the question is satisfactorily answered, or if it is not applicable, and for comments.

- The audit team will meet at the beginning of the audit with the Laboratory Manager to discuss the laboratory operations to be audited. The Quality Control Coordinator should be available to the audit team throughout the audit.

- At the close of the audit, the audit team will meet with the Laboratory Manager to discuss the audit findings. As necessary, other laboratory staff and the Quality Control Coordinator should attend the audit closure to discuss the findings. The lead auditor can close a finding during this discussion if the laboratory staff can satisfactorily demonstrate that the finding is inappropriate.

  Also during the audit-closure meeting, the means for corrective action and verifying correction will be discussed. If corrective action can be initiated immediately after the audit closure, the action should be taken.

- An audit report will be prepared by the lead auditor which discusses the following:

  - Date and location of audit

  - Audit team members and persons contacted in the laboratory

- Laboratory operations audited
- Description of items requiring corrective action and, if possible, the means for correction
- Due date for completion of corrective actions
- Means for verifying completion of corrective action
- Review of the Quality Assurance Program

The audit report shall be issued as soon as possible after completion of the audit (required within 30 days).

- The Laboratory Manager is responsible for responding to the audit report. Response shall be in writing to the lead auditor and shall state the corrective action or the action underway. If correction can be verified through correspondence, the Laboratory Manager shall attach documentation of corrective action to the audit response.

  Upon receipt of the audit response, the lead auditor must verify completion of the corrective action.

- After verification of corrective action, the lead auditor will issue a closure statement stating that all corrective action has been completed and the audit is closed. All audits must be closed.

During system audits, the Quality Assurance Manager and/or lead auditor should be cognizant of recurring nonconformances in a laboratory or trends which will affect quality. Recurring nonconformances and trends should be addressed in the audit report. Correction for such events may require a review of the adequacy of the Quality Assurance Program. If the inherent problems lies within the Program, the Program shall be amended through appropriate revision of Quality Assurance documents.

15.0 Preventive Maintenance

Table 15.1 summarizes requirements for the preventive maintenance of analytical instrumentation associated with this project.

Written Maintenance records are maintained for all laboratory equipment and kept on file. Also, the listed analytical equipment in Table 15.1 is maintained under maintenance contracts with the manufacturers.

TABLE 15.1. PREVENTIVE MAINTENANCE REQUIREMENT

	Items Checked/Serviced	Frequency	Documentation	Reference
lance	Internal weight train, gears, electronics	Annual service	Log book, service sticker	Manufacturer's Manual
	Electronics checked Electrolyte changed	Each analysis day Checked weekly, changed when low	Log book	Manufacturer's Manual
	Check optics Check electronics Check torch	Daily	Log book	Manufacturer's Manual
tion eter	Check optics, Check electronics Check vapor tightness of CVAA System	Daily	Log book	Manufacturer's Manual
eter	Check electronics Check optics Check response using standard solutions	Each analysis day Monthly Monthly	Log book	Manufacturer's Manual
	Check electronics Replace column	Each analysis day As required	Log book Manual	Manufacturer's Manual

16.0 Corrective Action Procedures

Corrective action procedures for this program will be initiated by the analytical personnel and their supervisors directly involved with implementing the procedures presented in the QAPP. Quality control charts for daily instrument calibration and replicate analyses will be utilized to indicate the necessity for corrective action. Control charts will be established for each procedure indicating upper and lower limits of three standard deviations as the acceptability ranges. Warning ranges are established at two standard deviations. At the point when the control charts show a determination outside the warning ranges, investigation as to the cause will be initiated. Any of the following events that occurs on the quality control chart will trigger corrective action:

- Two consecutive determinations fall outside the upper or lower control limits.
- Runs up -- (seven consecutive determinations increasing in value) -- or runs down (seven consecutive determinations decreasing in value).
- Three consecutive values fall above or below the warning limits (two standard deviations).

Corrective actions will also be initiated as a result of other QA activities which include performance audits, systems audits, and laboratory comparison studies.

The corrective action relative to the control charts is related more to precision than to accuracy. These charts give clues as to when some factor, generally of a procedural nature, is causing the results to drift or when an unexpected difference beyond the control limits occurs. Data within the upper and lower control limits of the control charts are well within the precision, accuracy, and completeness criteria.

These routine analytical corrective action procedures within the laboratory will be documented and may result in the reanalysis of samples or recalibration of analytical instrumentation. Routine corrective action will take place as necessary and will not require the approval of the Metcalf & Eddy QA Project Officer. However, should significant other events occur such as sample breakage or loss, exceeding sample holding times, extensive instrumentation downtime, or changes or additions to sample clean-up for removal of interferences, the laboratory will immediately report these events to Metcalf & Eddy.

Corrective Action Reporting

1. Follow-up Corrective Action reports will also be submitted, in writing, within 48 hours to the CO by the laboratory. Information justifying poor recovery or

precision will be documented when limits are exceeded. The Corps QA Laboratory will then decide what further corrective action, if any, need be taken. Personnel responsible for initiating and executing corrective action shall be indicated in the Corrective Action Reports. Those individuals responsible for the corrective actions shall sign the bound laboratory log books and all documentation citing the corrective action.

2. As required by this contract, Metcalf & Eddy shall prepare Design A-E/Construction Contractor Daily Quality Control (DQC) Reports. The DQC reports shall be prepared per contract specifications and submitted to the CO during ongoing field activities. The daily report shall contain, at a minimum the following:

   - Location of Work
   - Weather information
   - Work performed
   - Specific inspections performed and results
   - Problems identified
   - Corrective actions, if any
   - Verbal or written instructions from government personnel for retesting
   - Type of tests performed, samples collected, personnel involved, and results of tests
   - General remarks
   - Calibration procedures and recordings
   - Design A-E's/construction contractor's certification

3. To summarize project-specific QA/QC activities, a Design A-E/Construction Contractor Quality Control Summary Report will be prepared by the QA Project Officer upon conclusion of this project for CO approval.

17.0 Procedures for Assessing Precision, Accuracy and Completeness

Following are the procedures recommended for evaluating the precision and accuracy of all environmental measurement data generated by this project. Quality control sample analyses are performed as appropriate for organic or inorganic sample analyses as discussed in Section 13. The protocol used will be in accordance with specific analytical procedures if QC requirements are stated in the procedure.

17.1 Review of QC Samples Data

When the analysis of a sample set is completed, the results will be reviewed and evaluated to assess the validity of the data set. Review is based on the criteria in Table 13.1, applied as follows:

- Reagent Blank Evaluation - The reagent and/or method blank results are evaluated for high readings characteristic of background contamination. If high blank values are observed, laboratory glassware and reagents will be checked for contamination and the analysis halted until the system is brought under control before further sample analysis proceeds. A high background is defined as a background value greater than 20 µg/L for common laboratory solvents and greater than detection limits for all other analytes.

- Field Blank Evaluation - Field blank results are evaluated for high readings similar to the reagent and/or method blanks described above. If high field blank readings are encountered, the procedure for sample collection, shipment, and laboratory analysis should be reviewed. If both the reagent and/or method blanks and the field blanks exhibit significant background contamination, the source of contamination is probably within the laboratory.

- Matrix Spike Evaluation - The observed recovery of the spike versus the theoretical spike recovery is used to calculate accuracy as defined by the percent recovery. The average accuracy value, (the average percent recovery), may be plotted on a control chart for the parameter determined. If the average accuracy value exceeds the warning limit for the given parameters the Quality Control Coordinator is notified. If the average accuracy value exceeds the control limit, the sample set may be reanalyzed for the parameter in question.

- Calibration Standard Evaluation - The calibration curve is evaluated to determine linearity through its full range, and to verify that sample values are within the range defined by the low and high standards. If the curve is not linear, (correlation coefficient <0.995) sample values must be corrected for nonlinearity by deriving sample concentrations from a graph or by using an appropriate algorithm to fit a nonlinear curve to the standards.

- Replicate Sample Evaluation - Duplicate sample analysis for the sample set is used to determine the precision of the analytical method for the sample matrix. The duplicate results are used to calculate the precision as defined by the relative percent difference (RPD). If the precision value exceeds the warning limit for the given parameter, Quality Control Coordinator is If the precision value exceeds the control limit, the sample set must be reanalyzed for the parameter in question. Attainable precision limits will be specified by the Quality Control Coordinators and updated periodically following review of data.

- Replicate Analysis Evaluation - The replicate analyses are evaluated in the same manner as described above for the duplicate sample analysis and are treated as duplicate results for purposes of evaluating the precision of the analytical method. This evaluation is performed independently by the laboratory analyst, and may involve the use of Matrix Spike/Matrix Spike Duplicates for organics, and Matrix Spike/Duplicate Analysis for inorganics.

- Reference Standard Evaluation - Standard Reference Material analyses are compared with true values and acceptable ranges. Values outside the acceptable ranges require corrective action to determine the source of error and provide corrective action. All sample analyses should be halted pending this evaluation. Following correction of the problem, the Standard Reference Material should be reanalyzed.

- Check Standard Evaluation - The results of check standard analyses are compared with the true values and the percent recovery of the check standard is calculated. If correction is required, the check standard should be reanalyzed to demonstrate that the corrective action has been successful.

- Surrogate Standard Evaluation - The results of surrogate standard determinations are compared with the true values spiked into the sample matrix prior to extraction and analysis and the percent recoveries of the surrogate standards are determined. Average recovery values for surrogates in each GC/MS fraction will be plotted on control charts.

17.2 Evaluation of Data Using Control Charts

Weston Laboratories will apply precision and accuracy criteria to each parameter that is analyzed. When analysis of a sample set is completed, the quality control data are reviewed

and evaluated through the use of control charts to validate the data set.

Control charts will be established for all major analytical parameters. A minimum of ten measurements of precision and accuracy are required before control limits can be established. Once established, control limits will be updated (by the Quality Control Coordinator) as additional precision and accuracy data become available.

17.3 Evaluation of Analytical Precision

Because the referenced analytical methods present precision acceptance criteria as "advisory" only, acceptable precision will be defined as: when at least ½ of the compounds analyzed per parameter group (i.e. metals, VOA, etc.) meet advisory acceptance criteria.

For replicate results D_1 and D_2, the RPD is calculated from Equation 17-1:

$$RPD\% = \frac{D_1 - D_2}{\frac{D_1 + D_2}{2}} \times 100 \qquad (17\text{-}1)$$

When the RPD is obtained for at least ten replicate pairs, the average RPD and the standard deviation are calculated using:

$$\bar{m} = \frac{\sum_{i=1}^{n} m_i}{n} \qquad (17\text{-}2)$$

and

$$Sm = \sqrt{\frac{\sum_{i=1}^{n} (m-\bar{m})^2}{n-1}} \qquad (17\text{-}3)$$

where

m = the RPD of a replicate pair,

$\bar{m}$ = the average of the Relative Percent Difference determinations,

Sm = the standard deviation of the data set of RPD determinations, and

n = the number of RPD determinations.

When constructing a control chart for a specific parameter, the Warning and Control Limits are then calculated from the following:

Upper Control Limit = $\bar{m}$ + 3 Sm
Lower Control Limit = $\bar{m}$ - 3 Sm
Upper Warning Limit = $\bar{m}$ + 2 Sm
Lower Warning Limit = $\bar{m}$ - 2 Sm

A control chart is established by plotting the RPD of each replicate pair on a graph generated as follows:

- The calculated RPD of each replicate pair is plotted on the graph to determine whether the RPD is within the Warning and Control Limits of the Control Chart.

- If the RPD plots between the Warning and Control Limits, the Quality Control Coordinator will be notified for a decision as to how to proceed.

- If the RPD plots outside the Control Limits, the data set is invalid and the analysis will be halted until the source of error has been determined and corrective action taken. Once the error source has been resolved, the data set will be reanalyzed.

17.4 Evaluation of Analytical Accuracy

Because the referenced analytical methods present Accuracy acceptance criteria as "advisory" only, acceptable accuracy will be defined as: when at least ½ of the compounds analyzed per parameter group (i.e. metals, VOA, etc.) meet advisory acceptance criteria.

To determine the accuracy of an analytical method and/or the Laboratory Analyst, a periodic program of sample spiking is conducted. The results of sample spiking will be used to calculate the quality control parameter for accuracy evaluation, the Percent Recovery (%R).

The %R is defined as 100 times the observed concentration, minus the sample concentration, divided by the true concentration of the spike.

$$\%R = \frac{O_i - O_s}{T_i} \times 100\% \qquad (17\text{-}4)$$

where

$\%R$ = the Percent Recovery
O_i = the Observed Spiked Sample Concentration,
O_s = the Sample Concentration, and
T_i = the True Concentration of the Spike.

The true Concentration is calculated from the Equation below:

$$T_i = \frac{\text{Spike Concentration [c] (mg/L) x Volume of Spike (in mL)}}{\text{Volume of Sample [in mL] + Volume of Spike [in mL]}} \qquad (17\text{-}5)$$

When the Percent Recovery is obtained for at least ten spiked samples, the mean percent recovery and the standard deviation are calculated using the formulae:

$$\%\bar{R} = \frac{\sum_{i+1}^{n} \%R_i}{n} \qquad (17\text{-}6)$$

and

$$S_R = \sqrt{\frac{\sum_{i=1}^{n} (\%R_i - \%\bar{R})^2}{n-1}} \qquad (17\text{-}7)$$

where

$\%\bar{R}$ = the Mean Percent Recovery,

$\%R_i$ = The Percent Recovery of a Single Spiked Sample,

n = the number of results, and

S_R = the Standard Deviation of the data set of Percent Recovery determinations.

The Warning Control Limits are then calculated from the following equations:

$$\text{Upper Control Limit} = \%\bar{R} + 3\ S_R$$

$$\text{Lower Control Limit} = \%\bar{R} - 3\ S_R$$

$$\text{Upper Warning Limit} = \%\bar{R} + 2\ S_R$$

$$\text{Lower Warning Limit} = \%\bar{R} - 2\ S_R$$

A control chart is generated by plotting the Percent Recovery data on a graph as follows:

- The average of the Percent Recovery determinations for the original data set is established as the midpoint on the Y axis above the mean of the Percent Recovery on the graph.
- The Upper Warning and Control Limits calculated above are plotted as solid horizontal lines across the graph at their respective points on the Y axis above the mean of the Percent Recovery determinations.
- The Lower Warning and Control Limits calculated above are plotted as solid horizontal lines.
- The calculated Percent Recovery of each spiked sample is plotted on the graph to determine whether the Percent Recovery is within the Warning and Control Limits of the Control Chart.
- If the Percent Recovery plots between the Warning and Control Limits, the Quality Control Coordinator is notified for a decision as to how to proceed.

- If the Percent Recovery plots outside the Control Limits, the data set is invalid and the analysis will be halted until the source of error has been determined and corrective action taken. Once the error source has been resolved, the data set will be reanalyzed.
- When an additional ten Percent Recoveries have been determined, the Warning and Control Limits will be recalculated for the entire data set and the Control Chart for the corresponding parameter is updated.

All control charts are maintained by the Quality Control Coordinator.

17.5 Evaluation of Completeness

Completeness is calculated as the percentage of total usable data points out of the set of total data points collected and analyzed and available. Data points may not be usable if samples exceeded holding times, or if quality control sample criteria were not met and reanalysis of samples is not possible, or if samples were broken in the lab.

18.0 Quality Assurance Reports to Management

18.1 Metcalf & Eddy, Inc.

At monthly intervals for the duration of the field sampling and chemical analysis activities of this project, the Metcalf & Eddy QA project officer will prepare summary reports on the performance of measurement systems and data quality. These reports will address, at a minimum, the following:

- Results of performance audits of all field sampling and laboratory analysis activities performed during the subject reporting period
- Results of system audits
- Assessment of measurement data accuracy, precision, and completeness, including review of all Weston laboratory measurement data
- Significant QA problems and recommended solutions

Information provided in the periodic QA reports will be summarized for inclusion in the final project report.

18.2 Weston Laboratories

18.2.1 Performance Audit Reporting. The internal laboratory review activities of the Quality Control Coordinator shall be summarized in a monthly report to be submitted to the M&E QA project officer.

The report may be in the form of a checklist, with brief narratives as required, describing the activities reviewed. A copy of the nonconformance log for the month shall be attached. The monthly report shall emphasize ongoing or recurring problems. To demonstrate review, the Laboratory Supervisors affected and the Laboratory Manager shall sign and date the report. The signed copy shall be maintained by the Quality Control Coordinator for a period of two years after the date of the report.

18.2.2 System Audit Reporting. Audit reports prepared from the system audit shall be distributed to the Director, Analytical Services; Laboratory Manager; and the Quality Assurance Manager (if the lead auditor is not the Quality Assurance Manager). The Vice President and Quality Assurance Manager shall sign and date their copies to demonstrate review, and the copies shall be maintained by the Quality Assurance Manager for two years after the date of the report audit.

18.2.3 Nonconformance/Corrective Action Resolution from System Audits. The Laboratory Manager and Quality Assurance Manager shall seek to resolve directly all differences concerning

cited nonconformances, requests for corrective action, or the completion of corrective action. If the differences cannot be resolved, the Vice President, Environmental Services shall be the arbiter. The decision of the Vice President is final.

18.2.4 Management Review of the Quality Assurance Program. Review of the appropriateness and adequacy of the Quality Assurance Program is ongoing. At anytime, the Laboratory Manager should present recommended changes to the Quality Assurance Manager.

During system audits, the Quality Assurance Program should be discussed. The audit report will document recommendations made by either the Laboratory Manager or the audit team for revision.

In addition to these reviews, the Vice President, Environmental Services, shall conduct an annual review of the Quality Assurance Program considering:

- Results of system audit reports, i.e., are undesirable trends occurring?
- Is the present status of Quality Assurance documents adequate? Should manuals be revised; are new manuals needed?
- Are audits fulfilling their purposes?

The Environmental Services Director will consult with the Laboratory Managers and the Quality Assurance Manager during the review as deemed appropriate. To document the review, the Vice President of Environmental Services will issue a memorandum to the Quality Assurance Manager and Laboratory Managers stating the extent of the review and recommendations.

19.0 References

USACE, 1986, Sample Handling Protocol for Low, Medium and High Concentration Samples of Hazardous Waste.

USACE, 1985, Engineering and Design, Chemical Quality Management, Toxic and Hazardous Wastes, ER 1110-1-263.

"Test Methods for Evaluating Solid Wastes," USEPA Publ. No. SW-846, September 1986.

"Methods for Chemical Analysis of Water and Wastes," U.S. Environmental Protection Agency, Publ. No. EPA/600/4-79-020.

"Code of Federal Regulations," Volume 40, Parts 260 through 265 plus 270, July 1986.

APPENDICES

A-1 Resumes of Key Weston Personnel

Norman W. Flynn

Fields of Competence

Laboratory and program/project management relating to the environmental industry; trace analyses of organic compounds using gas chromatographic and mass spectrometric technologies; sample collection and field determinations, especially involving volatile organic compounds found in process gas streams.

Experience Summary

Fourteen years of technical and management experience in commercial environmental laboratories, health care and university institutions. Laboratory management experience is complemented by a strong background in advanced chemical instrumentation, especially in gas chromatography/mass spectrometry. This foundation is supplemented with numerous project/program management roles, including major technical and management activities in the Rocky Flats CEARP program, U.S. EPA hazardous waste incineration and 301(h) analytical programs, and Gas Research Institute landfill gas recovery programs. Additional experience includes involvement in marketing environmental chemistry capabilities to commercial and government clients.

Credentials

B.S., Animal Sciences—University of Arkansas (1970)

M.S., Animal Nutrition/Biochemistry—University of Arkansas (1971)

Employment History

1986-Present	WESTON
1986	Thermo Analytical Inc.
1983-1985	McKesson Environmental Services
1980-1983	Science Applications, Inc.
1978-1980	Acurex Corporation
1977-1978	University of California, San Francisco
1976-1977	Stanford Research Institute
1972-1976	University of Tennessee Center for Health Sciences

Key Projects

Analytical program direction and coordination of three WESTON and three subcontract laboratories in support of Rocky Flats CEARP activities. Responsible for coordination of sampling schedules with analytical laboratories, work flow management at WESTON on-site laboratory, data deliverables from the laboratories to the program office, and interfacing with program data management section.

Program management responsibilities for analytical portion of Gas Research Institute, Environmental Health and Safety Division programs involving trace constituent identification and removal from landfill gas. Developed gas chromatography/mass spectrometry methods for determination of individual volatile trace constituents using cryogenic capillary column techniques. Developed portable detector systems for determination of total halogenated and sulfonated compounds at the parts-per-million level. Evaluated major treatment technologies for removal of trace constituents from landfill gas.

Program management responsibilities for California Air Resource Board program designed to characterize hazardous wastes generated in California and supplement historical characterization (manifest) data with laboratory analyses from selected waste processes.

Technical Manager for gas chromatographic analyses of industrial effluents in the Puget Sound. Program per-

Professional Profile

Earl M. Hansen, Ph.D.

Fields of Competence

Trace organic and inorganic analysis using U.S. EPA, ASTM, AIHA methodology; analytical methods development; collection and analysis of environmental samples including ambient air, stationary source discharges, water, wastewater, biological tissue, biological fluids, soils, sediments and hazardous waste; development and implementation of laboratory quality assurance and quality control programs.

Experience Summary

Fourteen years experience in the following areas:

Preparation and analysis of environmental samples for inorganic and organic analytes using GC, GC/MS, AA, ICP, HPLC and wet chemical techniques. Method development for selected priority pollutant analytes in chemical process wastewater as part of U.S. EPA BAT program.

Development of methods for analysis of tetrachlorinated dibenzo-dioxin (TCDD) isomers in organic liquids and commercial chlorinated phenols using GC/MS selected ion monitoring techniques.

Methods validation for use of volatile organic sampling train (VOST) to collect and analyze volatile organic emissions from hazardous waste incinerators. Sampling and analysis of selected analytes in multimedia emissions from Refuse Derived Fuel (RDF) Waste-to-Energy processes.

Credentials

B.A., Chemistry—Wittenberg University (1963)

Ph.D., Chemistry—Michigan State University (1970)

Employment History

1984-Present	WESTON
1982-1984	Envirodyne Engineers, Inc.
1977-1982	Midwest Research Institute
1973-1977	Snell Environmental Group
1972-1973	Clyde E. Williams and Associates
1969-1972	Notre Dame University

Key Projects

Managed a program to analyze environmental samples for 2,3,7,8,-TCDD for the U.S. EPA. This program required the analysis of over 2,000 environmental samples in 1983.

Managed a sampling and analysis contract for U.S. EPA at Research Triangle Park, North Carolina. This program focused on the evaluation of a volatile organic sampling train (VOST) for the collection of volatile organic compounds from the gaseous effluents of hazardous waste incinerators. Directed the construction of two VOST trains and developed a protocol for the use of VOST to evaluate the performance of hazardous waste incinerators.

Managed five laboratory tasks as part of a contract with United States Army Toxic and Hazardous Materials Agency (USATHAMA) for contamination survey of Army installation. This included development and validation of methods for selected analytes using the USATHAMA Quality Assurance Procedure.

Participated in the design and preliminary evaluation of a laboratory-scale thermal destruction system to be used to evaluate the feasibility of incineration of liquid and solid hazardous wastes. Directed a multi-task program which required quick response methods evaluation and analysis of groundwaters and soils from hazardous waste disposal sites. Samples received in this program were analyzed for substituted phenols and polynuclear aromatic hydrocarbons (PAH's) using GC/MS and HPLC.

Managed a program to analyze process wastewaters from six organic chemical manufacturing plants. This program was conducted for the U.S. EPA to identify and quantify the presence of organic and inorganic priority pollutants in these wastewaters. The project required design of sampling plans, development and evaluation of analytical methods, and collection and analysis of over 250 samples. These data were incorporated into the database which, is to be used by U.S. EPA to establish Best Available Treatment Technology (BAT) regulations for the organic chemical manufacturing industry.

Led the evaluation, selection, and recommendation of an inductively-coupled plasma (ICP) spectrophotometer which was purchased as an addition to MRI's atomic spectroscopy instrumentation in 1981.

Professional Profile

Margaret M. Beaty

Fields of Competence

Laboratory management. Analytical chemistry methods development, evaluation and analysis, with extensive experience in foods, soils and environmental samples.

Experience Summary

Twenty years experience in university and industrial laboratories, conducting analytical research and methods development, and supervision of laboratory personnel. Variety of practical experience in sample preparation, instrumental analysis, analytical quality control, and troubleshooting.

Credentials

B.S., Biological Science—Oklahoma State University (1971)

M.S., Food Science—Louisiana State University (1977)

Institute of Food Technologists

Louisiana Association of Agronomy

Employment History

1986-Present	WESTON
1977-1981	Perkin-Elmer Corporation
1972-1977	Louisiana State University Soil Testing Laboratory
1967-1972	Oklahoma State University Animal Science, Biochemistry and Dairy Science Laboratories

Key Projects

Managed field laboratory in preparation and analysis of hazardous waste samples. Monitored data and analytical quality control to verify results.

Wrote product specifications and performed evaluation and testing of new laboratory instrumentation for atomic absorption spectrophotometry.

As Applications Chemist, performed methods and applications development for atomic spectroscopy. Provided technical support and training for clients and in-house personnel.

Conducted research and development on soil digestion and extraction techniques. Work included chemical analysis, quality control and evaluation of results.

Laboratory Supervisor, responsible for personnel management, work assignments, and technical training for laboratory technicians.

Conducted meat and protein research, including proximal analysis, enzyme assays, protein extraction, separation and gel electrophoresis. Gained experience in small-scale food production and taste panel.

Dairy science laboratory work, including milk enzyme separation, analysis and electrophoresis, and research on dairy waste products.

Professional Profile

Carter P. Nulton

Field of Competence

His graduate and post-doctoral research was directed toward the development and application of analytical methods for the study of small molecule metabolism. He was involved with the design and construction of a combined, computerized radio-gas chromatograph/mass spectrometer (RGC/MS) and its application to metabolic studies in fungi, plants and algae. For seven years at Southwest Research Institute he worked on developing methods for analysis of trace levels of organic pollutants in a variety of environmental matrices, characterizing potentially toxic organic constituents resulting from combustion processes and developing approaches to analyzing hazardous wastes. As manager of the GC/MS facility at Southwest Research Institute he also supported research in organic synthesis, fuel characterization, electronic component failure analysis and biochemistry.

Credentials

B.S., Chemistry - Geneva College (1969)

Ph.D., Biochemistry - University of Pittsburgh (1975)

Employment History

1984-Present	WESTON Organic Laboratory Manager
1978-1984	Southwest Research Institute Manager, Mass Spectrometry
1975-1978	University of Pittsburgh Research Associate

Key Projects

Development of GC methods for the analysis of industrial process waters and effluents using a wide variety of detectors (ECD, Hall, PID, FID, NPD, TCD, FPD).

Characterization of organic pollutants in municipal sludges using GC/MS.

Analysis of biota and sediments from an oil producing area in the Central Gulf of Mexico to determine the presence and extent of contamination of petrogenic hydrocarbons.

Characterization of organic wastes generated by the organobromine industry.

Studies to elucidate the mechanism(s) of sediment formation in diesel fuel using pyrolysis capillary GC/MS and FT-IR.

Sampling and analysis of feedstocks emissions and wastes from a coal/refuse co-fired power plant with emphasis on determining if chlorinated pollutants (particularly dioxins) were evolved.

Analysis of combustion products arising from halocarbon polymers.

Publications

C.P. Nulton. Secondary Metabolism in *Pennicillium brevicompactum.* Ph.D. Thesis, University of Pittsburgh, Pittsburgh, Pennsylvania, 1975.

C.P. Nulton, J.D. Naworal, I.M. Campbell and (in part) E.W. Grotzinger, Combined Radio Gas Chromatography/Mass Spectrometry Detects Intermediates in Mycophenolic Acid Biosynthesis. Analytical Biochemistry, 75:219-233, 1976.

C.P. Nulton and I.M. Campbell. Mycophenolic Acid is Produced During Balanced Growth of *Pennicillium brevicompactum.* Cand. J. Microbiol., 23:20-27, 1977.

I.M. Campbell, D.L. Doerfler, S.A. Donahey, R. Kadlec, E.L. McGandy, J.D. Naworal, C.P. Nulton, M. Venza-Raczka, and F. Wimberly. A Software Package to Collect and Process Radiogas Chromatographic Data. Analytical Chem., 49:1726-734, 1977.

C.P. Nulton and I.M. Campbell. Labelled Acetone and Levulinic Acid Are Formed. When C-Acetate is Being Converted to Mycophenolic Acid in *Pennicillium brevicompactum.* Cand. J. Microbiol., 24:199-201, 1978.

D.L. Doerfler, C.P. Nulton, C.D. Bartman, F.J. Gottlieb, and I.M. Campbell. Spore Germination, Colony Development, and Secondary Metabolism in *Pennicillium brevicompactum:* A Radiogas Chromatbgraphic and Morphological Study. Cand. J. Microbiol., 24:1490-1501, 1978.

Professional Profile

George H. Perry

Fields of Competence

Environmental sampling and analysis. Gas chromatography and HPLC. Data compliance with EPA and USATHAMA requirements. Instrument automation and technical writing. Mobile lab operation. Method development.

Experience Summary

Residue analysis of environmental, industrial, and bioassay samples for hydrocarbons, pesticides, priority pollutants and explosives.

Baseline oceanographic sampling and analysis. Damage assessment in herbicide application cases. Implementation of U.S. EPA protocol for pesticides by gas chromatography. Explosives analysis for USATHAMA on soil and water samples by HPLC. Method development for laboratory and field use.

Credentials

B.S., Chemistry and Marine Science—Southampton College (1976)

Student Intern—Woods Hole Oceanographic Institute

American Chemical Society

Employment History

1983-Present	WESTON
1977-1983	Energy Resources Company, Inc.
1975-1977	New York Ocean Science Laboratory

Key Projects

As contractor to U.S. EPA in Kansas City, reviewed data assessing dioxin distribution in Missouri.

Developed methodology for rapid field screening of soil and water samples for total PAH content.

Developed methodology for rapid field screening of soil samples for PCB and DOT.

Outfitted mobile trailer as field laboratory to support sampling with field analysis by rapid extraction and gas chromatography.

Professional Profile

Richard J. Ronan, Ph.D.

Fields of Competence

Laboratory operations management; direction of product development and engineering; development of new analysis methods.

Experience Summary

More than 17 years experience in laboratory research, operations and management in academia, government and consulting. Accomplishments have included: development of laboratory management team, reorganization of professional staff, implementation of laboratory expansion, development of broad-based laboratory certifications, implementation of control procedures regarding product development and efficiency.

Credentials

A.B., Chemistry/Mathematics—Franklin College of Indiana (1965)

M.S., Inorganic Chemistry—University of Hawaii (1968)

Ph.D., Inorganic Chemistry—University of Hawaii (1970)

Employment History

1987-Present	WESTON
1978-1987	Versar, Inc.
1976-1978	Fisher Scientific, Jarrell-Ash Division
1974-1976	U.S. EPA, Region V
1973-1974	Iowa State University, Ames Laboratory
1970-1973	Simpson College

Key Projects

Developed and managed first model of final test approach for chemically contaminated buildings.

Obtained national acceptance of new metals analysis technique for Region V NPDES permit work. Resulted in annual savings ranging from $100,000 to $300,000.

Designed and developed a computer-based Data Management System, which saved five man-years effort per year.

Worked out analytical quality control procedures, which are now standard, for lead in gas for both field and laboratory.

Developed several new procedures for analysis of dredged material.

Professional Profile

James M. Taylor

Fields of Competence

Ambient, source and industrial hygiene air pollution monitoring; analytical wet chemistry using ASTM and U.S. Environmental Protection Agency (U.S. EPA) methodology; microbiological analysis of water, wastewater, air and humans; collection and analysis of drinking water, industrial process streams and hazardous wastes.

Experience Summary

Thirteen years experience in the following areas:

Method development for U.S. EPA ambient and stationary source monitoring methods. Team leader for numerous air program evaluations and compliance tests for industry and government. Method development for detecting pesticides and PCB's in air by use of solid sorbent media. Sampling and analysis of pesticides and PCB's in indoor atmospheres and at hazardous waste facilities using polyurethane foam sampling cartridges. Method development for concentrating and collecting biological and viral organisms around sewage treatment facilities and sewage spray irrigation operations to determine aerosolization characteristics.

Method development for U.S. EPA water and wastewater methods for nitrosamines, phthalate esters, and pesticide/PCB's. Conducted collaborative testing program for nitrosamine and pesticide/PCB methods. Experience in wet chemical techniques for inorganic and organic constituents in liquid and solid matrices.

Familiar with U.S. EPA and standard method procedures for the analysis of metals, inorganics, herbicides, pesticides, PCB's and acid/base/neutral extractables for GC/MS analysis.

Method development for a variety of environmental matrices from hazardous waste to analyze for trace-level organic pollutants.

Credentials

B.A., Microbiology—University of Texas at Austin (1971)

Employment History

1985-Present	WESTON
1973-1985	Southwest Research Institute
1972-1973	Kelsey - Seybold at NASA
1971-1972	Southwestern Laboratories

Key Projects

Conducted sampling and analysis of potentially hazardous industrial waste from plants in several industrial categories. The purpose of the study was to characterize wastes in accordance with U.S. EPA's May 19, 1980 regulations.

Participated in sampling and analysis of industrial wastewaters from numerous plants in the plastic and organic chemicals industrial categories for priority pollutants under a program for U.S. EPA-EGD. This effort was for both screening and verification analysis of industrial wastewater. One of the methods developed was a microextraction procedure that was later adopted by the U.S. EPA-EGD for use in the verification programs.

Participated in sampling and analysis program in the environmental monitoring effort of Love Canal program sponsored by U.S. EPA-EMSL.

Participated in method development and collaborative studies of U.S. EPA Stationary Source Methods 2,3,4,5,6,7,8,9 and 17. Also participated in collaborative testing of Methods 2, 3 and 5 using paired particulate sampling trains.

Participated in two programs jointly funded by U.S. EPA and U.S. Army Medical R&D to evaluate health implications of sewage treatment facilities and to evaluate health effects associated with the application of wastewater to land.

Participated in a U.S. EPA program to develop and validate analytical methods for pesticides, PCB's, nitrosamines, and phthalate esters in using non - GC/MS techniques. These methods were later proposed as Federal Register methods 606, 607, and 608 for quantitative analysis of drinking, surface, and wastewater samples.

Participated in a comprehensive seven-year monitoring and ecological analysis of a coastal power plant con-

Professional Profile

Dianne S. Therry

Fields of Competence

Laboratory QA/QC development and implementation; data management coordination, including quality assurance and quality control procedures; identification and quantification of chemicals through the use of instrumental and wet methods of analysis; chemical and microbiological analysis of potable, surface, and wastewater.

Audit of laboratory fate-and-effect studies to ensure conformance to EPA-Good Laboratory Practices for regulated substances. Data validation of organic/inorganic analyses for hazardous substance list compounds. Technical audits of the analytical laboratory to evaluate laboratory performance facilities, and conformance to client program requirements. Development, implementation, and management of laboratory quality assurance and quality control programs.

Experience Summary

Documentation of major contract laboratory projects for government agencies, including USATHAMA and EPA. Data validation for the U.S. EPA - Contract Laboratory Program. Client/agency audits of the WESTON laboratories and required audit response with laboratory management. Coordinate functions for application/maintenance of laboratory licenses and certifications, including participation in interlaboratory performance evaluation audit studies. Internal QA/QC audits of WESTON laboratories to monitor and evaluate ongoing QA programs.

Laboratory QA/QC requirements including compilation of analytical lab SOP's and maintaining and updating the lab QA/QC Manual; conducting laboratory audits, maintenance of certification records and requirements.

As Data Management Coordinator: track sample status from log-in through final reporting and sample disposal; prepare lab QA samples and subsequent performance reports; interface with clients and regulatory agencies for monitoring/auditing purposes.

Analysis of process and industrial waters; quality control checks of water treatment chemicals; chemical and microbiological analysis of streams, drinking water, and domestic and industrial wastewater using EPA and APHA Standard Methods of Analysis. Methodology includes wet methods of analysis, AA, GC, Auto Analyzer, and TOC.

Courtroom experience at local and Federal levels with regard to laboratory analyses.

Credentials

B.S., Chemistry Education—West Chester State College (1974), ACS Accredited Program.

Post-graduate courses in priority pollutants techniques of analysis; gas chromatography; water microbiology; toxicology; geochemistry; geological field studies; physics of the atom. Refresher courses in AA and GC.

Certified by EPA and the State of Pennsylvania to perform and supervise water microbiology.

American Chemical Society, Philadelphia Local Section, Division of Environmental Chemistry

Remedial Response Health and Safety Training Course (1985)

Project Management Course (1985)

Employment History

1982-Present	WESTON
1977-1982	Chester County Health Department, Public Health Laboratory
Winter-Summer 1977	Nalco Chemical Company
1974-1976	West Windsor-Plainsboro High School

Key Projects

Developed, implemented, and coordinated a system for the assembly and audit of QA deliverable packages for the EPA Contract Laboratory Program, including compilation of a standard practices manual and training of personnel.

Professional Profile

Debra K. White

Fields of Competence

Trace inorganic analysis using U.S. EPA methodology; development of laboratory quality assurance/quality control programs; management of analytical services; analytical protocol development; data quality/useability assessment.

Experience Summary

Laboratory/project management. Technical staff training. Laboratory QA/QC, laboratory audits, documentation of analytical standard operating procedures. Data validation/useability assessment of CLP inorganic data. Preparation and analysis of environmental samples for inorganic analytes using atomic absorption, ICP and wet chemical techniques.

Credentials

B.A., Chemistry—Cedar Crest College (1978)

Graduate Studies, Environmental/Analytical Chemistry

University of Maryland (1978-80)

Employment History

1986-Present	WESTON
1985-1986	U.S. EPA Headquarters, Washington, D.C.
1984-1985	U.S. EPA Region III, Central Regional Laboratory
1980-1984	JTC Environmental Consultants, Inc.

Key Projects

Program management for the U.S. EPA Contract Laboratory Program (CLP). Technical/contractual oversight of 16 inorganic CLP laboratories. Analytical method/protocol development. Onsite laboratory evaluations. Oversight of interlaboratory performance evaluation studies. Coordination of technical review caucuses.

Principle author of U.S. EPA manual for inorganic data validation under the Contract Laboratory Program.

Publications

White, D.K., "Inorganic Analytical Methods—General Description and Quality Control Considerations." *Quality Control in Remedial Site Investigation: Hazardous and Industrial Solid Waste Testing, Fifth Volume, ASTM STP 925, C.L. Perket, Ed.* American Society for Testing and Materials, 1986.

Fowler, J.W., G. Ward, and D.K. White, et al., "Inorganic Analytical Methodology in the United States Environmental Protection Agency Contract Laboratory Program." Proceedings of the Water Pollution Control Federation Conference, Denver, Colorado, May 1986.

Professional Profile

A-2 Example Laboratory Reports - Weston

WESTON Analytics
RFW Batch:

The following qualifiers/codes are used on the data summary:

U- Indicates that the compound was analyzed for but not detected. The detection limit for the sample (not the method detection limit) is reported with the U (e.g.,10u).

MB- Method Blank- consists of deionized, distilled water processed through each sample preparation procedure performed. The analysis of method blanks provides a means of assessing the existence and magnitude of contamination introduced via the analytical scheme. The reported sample results are not corrected for the blank results.

LCS- Indicates laboratory control sample in which reagent grade water is spiked with the USEPA CLP LCS solution and carried through all the steps in the method. The LCS is designed to serve as a monitor of the efficiency of the digestion procedure. Percent recoveries are reported.

The subscripts S and R are utilized to denote matrix spikes and matrix replicates respectively. A matrix spike analysis is designed to provide information concerning the effect of the sample matrix on the recovery of a specific analyte. A matrix replicate analysis will provide information on the precision of the method utilized as well as the sample homogeneity.

The methods utilized by this laboratory, unless otherwise requested, are for all ICP analysis USEPA method 200.7; the furnace analysis of Arsenic (206.2), Selenium (270.2), Lead (239.2), Thallium (279.2) and Antimony (204.2), as well as the Flame Emission method for Sodium (273.1) and Potassium (258.1) are taken from Methods for Chemical Analysis of Water and Wastes (USEPA 600/4-79-020).

NA- Not Applicable
NR- Not Required
NC- Not Calculable, result below detection limit.

Approved by _____ 9/3/87
Debra K. White
Inorganic Section Manager
WESTON Analytical Laboratories

WESTON ANALYTICS

INORGANICS DATA SUMMARY REPORT 08/03/87

CLIENT: WESTON BATCH #:

SAMPLE	SITE ID	ANALYTE	RESULT		UNITS	DETECTION LIMIT
-0010	MW7	ARSENIC, TOTAL	23.8		UG/L	
		ARSENIC, TOTAL	10.0	u	UG/L	10.0
		BARIUM, TOTAL	200	u	UG/L	200
		CALCIUM, TOTAL	105000		UG/L	10000
		CADMIUM, TOTAL	5.0	u	UG/L	5.0
		CHROMIUM, TOTAL	10.0	u	UG/L	10.0
		IRON, TOTAL	100	u	UG/L	100
		MERCURY, TOTAL	0.2	u	UG/L	0.2
		POTASSIUM, TOTAL	1570		UG/L	500
		MAGNESIUM, TOTAL	20400		UG/L	500
		MANGANESE, TOTAL	141		UG/L	15.0
		SODIUM, TOTAL	4900		UG/L	500
		LEAD, TOTAL	5.0	u	UG/L	5.0
-0020	MW9	ARSENIC, TOTAL	19.6		UG/L	
		ARSENIC, TOTAL	10.0	u	UG/L	10.0
		BARIUM, TOTAL	200	u	UG/L	200
		CALCIUM, TOTAL	54500		UG/L	10000
		CADMIUM, TOTAL	5.0	u	UG/L	5.0
		CHROMIUM, TOTAL	10.0	u	UG/L	10.0
		IRON, TOTAL	3910		UG/L	100
		MERCURY, TOTAL	0.2	u	UG/L	0.2
		POTASSIUM, TOTAL	17400		UG/L	5000
		MAGNESIUM, TOTAL	33400		UG/L	500
		MANGANESE, TOTAL	1460		UG/L	15.0
		SODIUM, TOTAL	150000		UG/L	10000
		LEAD, TOTAL	5.0	u	UG/L	5.0

WESTON ANALYTICS

INORGANICS DATA SUMMARY REPORT 08/03/87

CLIENT: WESTON BATCH #:

SAMPLE	SITE ID	ANALYTE	RESULT		UNITS	DETECTI LIMIT
-0030	MW10	ARSENIC, TOTAL	16.7		UG/L	
		ARSENIC, TOTAL	10.0	u	UG/L	10.0
		BARIUM, TOTAL	200	u	UG/L	200
		CALCIUM, TOTAL	384000		UG/L	10000
		CADMIUM, TOTAL	5.0	u	UG/L	5.0
		CHROMIUM, TOTAL	10.0	u	UG/L	10.0
		IRON, TOTAL	4270		UG/L	100
		MERCURY, TOTAL	0.2	u	UG/L	0.2
		POTASSIUM, TOTAL	19700		UG/L	5000
		MAGNESIUM, TOTAL	23700		UG/L	500
		MANGANESE, TOTAL	2760		UG/L	300
		SODIUM, TOTAL	148000		UG/L	10000
		LEAD, TOTAL	5.0	u	UG/L	5.0
-0040	MW11	ARSENIC, TOTAL	18.7		UG/L	
		ARSENIC, TOTAL	10.0	u	UG/L	10.0
		BARIUM, TOTAL	200	u	UG/L	200
		CALCIUM, TOTAL	76800		UG/L	10000
		CADMIUM, TOTAL	5.0	u	UG/L	5.0
		CHROMIUM, TOTAL	10.0	u	UG/L	10.0
		IRON, TOTAL	726		UG/L	100
		MERCURY, TOTAL	0.2	u	UG/L	0.2
		POTASSIUM, TOTAL	1600		UG/L	500
		MAGNESIUM, TOTAL	12500		UG/L	500
		MANGANESE, TOTAL	121		UG/L	15.0
		SODIUM, TOTAL	2400		UG/L	500
		LEAD, TOTAL	5.0	u	UG/L	5.0

WESTON ANALYTICS

INORGANICS DATA SUMMARY REPORT 08/03/87

CLIENT: WESTON BATCH #:

SAMPLE	SITE ID	ANALYTE	RESULT		UNITS	DETECTI(LIMIT
BLANK1		ARSENIC, TOTAL	10.0	u	UG/L	10.(
		LEAD, TOTAL	5.0	u	UG/L	5.(
BLANK1		BARIUM, TOTAL	200	u	UG/L	200
		CALCIUM, TOTAL	500	u	UG/L	500
		CADMIUM, TOTAL	5.0	u	UG/L	5
		CHROMIUM, TOTAL	10.0	u	UG/L	10
		IRON, TOTAL	100	u	UG/L	100
		POTASSIUM, TOTAL	500	u	UG/L	500
		MAGNESIUM, TOTAL	500	u	UG/L	500
		MANGANESE, TOTAL	15.0	u	UG/L	15.(
		SODIUM, TOTAL	500	u	UG/L	500

WESTON ANALYTICS

INORGANICS ACCURACY REPORT 08/03/87

CLIENT: WESTON BATCH #:

SAMPLE	SITE ID	ANALYTE	SPIKED SAMPLE	SPIKED AMOUNT	UNITS	%REC(
-0080	MW12D	ARSENIC, TOTAL	33.0	40.0	UG/L	82.5

WESTON ANALYTICS

INORGANICS PRECISION REPORT 08/03/87

CLIENT: WESTON BATCH #:

SAMPLE	SITE ID	ANALYTE	INITIAL RESULT	REPLICATE	% DIFF
-0080	MW12D	ARSENIC, TOTAL	0.6	15.7	185
		ARSENIC, TOTAL	10.0 u	10.0 u	NC

WESTON ANALYTICS

INORGANICS LABORATORY CONTROL STANDARDS REPORT 08/03/87

SAMPLE	SITE ID	ANALYTE	SPIKED SAMPLE	SPIKED AMOUNT	UNITS	%REC
LCS1		ARSENIC, LCS	30.2	30.0	UG/L	101
		LEAD, LCS	24.2	30.0	UG/L	80.
LCS1		BARIUM, LCS	9860	10000	UG/L	98.
		CALCIUM, LCS	49500	50000	UG/L	99.
		CADMIUM, LCS	80.0	80.0	UG/L	100
		CHROMIUM, LCS	487	500	UG/L	97.
		IRON, LCS	4890	5000	UG/L	97.
		POTASSIUM, LCS	25400	25000	UG/L	102
		MAGNESIUM, LCS	49400	50000	UG/L	98.
		MANGANESE, LCS	728	750	UG/L	97.
		SODIUM, LCS	27000	25000	UG/L	108

WESTON ANALYTICS

INORGANICS LABORATORY CONTROL STANDARDS REPORT 08/03/87

SAMPLE	SITE ID	ANALYTE	SPIKED SAMPLE	SPIKED AMOUNT	UNITS	%REC
LCS1		MERCURY, LCS	8.6	8.0	UG/L	107

WESTON

WESTON Analytics
RFW Batch:

The following qualifiers/codes are used on the data summary:

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MB- Method Blank- consists of deionized, distilled water processed through each sample preparation procedure performed. The analysis of method blanks provides a means of assessing the existence and magnitude of contamination introduced via the analytical scheme. The reported sample results are not corrected for the blank results.

The method used for the analysis of petroleum hydrocarbons is EPA method 418.1 (USEPA 600/4-79-020). Solid Samples are extracted using method 3540 (USEPA SW 846) then analyzed by EPA method 418.1.

NA- Not Applicable
NR- Not Required
NC- Not Calculable, result below detection limit.

Approved by for Beth Keech 9/4/87
James Michael Taylor Date
Department Manager
WESTON Analytical Laboratories

WESTON ANALYTICS

ORGANICS DATA SUMMARY REPORT 09/02/87

CLIENT: WESTON BATCH #:

SAMPLE	SITE ID	ANALYTE	RESULT		UNITS	REPORTING LIMIT
-001	U-MW-1-HST	PETROLEUM HYDROCARBONS	0.8		MG/L	0
-002	D-MW-2-HST	PETROLEUM HYDROCARBONS	0.2	u	MG/L	0
-003	D-MW-2A-HST	PETROLEUM HYDROCARBONS	0.2	u	MG/L	0
-004	D-MW-3-HST	PETROLEUM HYDROCARBONS	0.2		MG/L	0.
-007	D-MW-3-GCP	PETROLEUM HYDROCARBONS	0.2	u	MG/L	.
-010	D-MW-2-GCP	PETROLEUM HYDROCARBONS	0.2	u	MG/L	0.
-011	D-MW-2A-GCP	PETROLEUM HYDROCARBONS	0.2	u	MG/L	
-012	U-MW-1-GCP	PETROLEUM HYDROCARBONS	0.2	u	MG/L	

WESTON ANALYTICS

ORGANICS METHOD BLANK DATA SUMMARY PAGE 09/02/87

CLIENT: WESTON BATCH #:

SAMPLE	SITE ID	ANALYTE	RESULT	UNITS	REPORTI. LIMIT
BLANK10	87IR963A-MB1	PETROLEUM HYDROCARBONS	0.2 u	MG/L	0.2

WESTON ANALYTICS

ORGANICS ACCURACY REPORT 09/02/87

CLIENT: WESTON BATCH #:

SAMPLE	SITE ID	ANALYTE	SPIKED SAMPLE	INITIAL RESULT	SPIKED AMOUNT	%REC
BLANK10	87IR963A-MB1	PETROLEUM HYDROCARBONS	37	0.2 u	42	88.1
		PETROLEUM HYDROCARBONS	37	0.2 u	42	88.1

WESTON ANALYTICS

ORGANICS DUPLICATE SPIKE REPORT 09/02/87

CLIENT: WESTON BATCH #:

SAMPLE	SITE ID	ANALYTE	SPIKE#1 %RECOV	SPIKE#2 %RECOV	%DIFF
BLANK10	87IR963A-MB1	PETROLEUM HYDROCARBONS	88.1	88.1	0.2

WESTON

ROY F. WESTON, INC.

RFW #
W.O.#

1. The following qualifiers are used on the data summary:

   U - Indicates that the compound was analyzed for but not detected. The minimum detection limit for the sample (not the method detection limit) is reported with the U (e.g., 10U).

   J - Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or where the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero (e.g., 10J). If the limit of detection is 10 ug/L and a concentration of 3 ug/L is calculated, it is reported as 3J.

   BS - Indicates blank spike in which reagent grade water is spiked with the CLP matrix spiking solutions and carried through all the steps in the method. Spike recoveries are reported.

   BSD - Indicates blank spike duplicate.

   MS - Indicates matrix spike.

   MSD - Indicates matrix spike duplicate.

   DL - Indicates that surrogate recoveries were not obtained because the extract had to be diluted for analysis.

   NA - Not applicable.

   DF - Dilution factor.

   NR - Not required.

   NRP - Not reported.

2. Samples Collected: 6-3,4,5-87
   Extraction Date: NA
   Analysis Date: 6-8,9,10,11,12-87

7-1-87
DATE

Carter P. Nulton, Ph.D.
Organic Section Manager
WESTON Analytical Laboratories

tch Number: Client:

Cust ID:	MWT1		MWT2		MWT2 DUP		MWT4		MWT4A	
RFW#:	0030		0040		0050		0060		0070	
		fl		fl		fl		fl		fl
hloroethene.....	5 U		22 J		16 J		5 U		5 U	
2-Tetrachloroethane.....	5 U		50 U		50 U		5 U		5 U	
e.....	5 U		50 U		50 U		5 U		5 U	
benzene.....	5 U		50 U		50 U		5 U		5 U	
enzene.....	5 U		50 U		50 U		5 U		5 U	
e.....	5 U		50 U		50 U		5 U		5 U	
Xylenes.....	5 U		99		68		5 U		5 U	
chlorobenzene.....	5 U		50 U		50 U		5 U		5 U	
chlorobenzene.....	5 U		50 U		50 U		5 U		5 U	
chlorobenzene.....	5 U		50 U		50 U		5 U		5 U	
orofluoromethane.....	5 U		2800		2200		5 U		5 U	

s:

U=Analyzed, not detected. B=Present in blank. NRP=Not Reported
J=Present at less than detection limit. NR=Not requested.

WESTON ANALYTICS
GC/MS DATA SUMMARY
VOLATILE HAZARDOUS SUBSTANCE LIST COMPOUNDS

ch Number: Client:

	Cust ID:	MWT1	MWT2	MWT2 DUP	MWT4	MWT4A
tion	RFW#:	0030	0040	0050	0060	0070
	Matrix:	Water	Water	Water	Water	Water
	D.F.:	1	10	10	1	1
	Units:	ug/l	ug/l	ug/l	ug/l	ug/l
te	Toluene-d8:	102 %	107 %	100 %	100 %	98 %
y	Bromofluorobenzene:	96 %	103 %	98 %	104 %	100 %
	1,2-Dichloroethane-d4:	102 %	102 %	98 %	102 %	102 %
		fl	fl	fl	fl	fl
ethane.....		10 U	100 U	100 U	10 U	10 U
thane.....		10 U	100 U	100 U	10 U	10 U
hloride.....		10 U	100 U	100 U	10 U	10 U
thane.....		10 U	100 U	100 U	10 U	10 U
ne Chloride.....		1 J	22 J	16 J	1 J	2 J
.....		2 J	22 J	12 J	2 J	2 J
Disulfide.....		5 U	50 U	50 U	5 U	5 U
hloroethene.....		5 U	50 U	50 U	5 U	5 U
hloroethane.....		5 U	50 U	50 U	5 U	5 U
,2-Dichloroethene.....		5 U	50 U	50 U	5 U	5 U
orm.....		5 U	50 U	50 U	5 U	1 J
hloroethane.....		5 U	50 U	50 U	5 U	5 U
one.....		10 U	100 U	100 U	10 U	10 U
richloroethane.....		5 U	50 U	50 U	5 U	5 U
Tetrachloride.....		5 U	50 U	50 U	5 U	5 U
cetate.....		10 U	100 U	100 U	10 U	10 U
chloromethane.....		5 U	50 U	50 U	5 U	5 U
hloropropane.....		5 U	50 U	50 U	5 U	5 U
,3-Dichloropropene.....		5 U	50 U	50 U	5 U	5 U
roethene.....		5 U	50 U	50 U	5 U	5 U
chloromethane.....		5 U	50 U	50 U	5 U	5 U
richloroethane.....		5 U	50 U	50 U	5 U	5 U
.....		5 U	50 U	50 U	5 U	5 U
-Dichloropropene.....		5 U	50 U	50 U	5 U	5 U
roethylvinylether.....		10 U	100 U	100 U	10 U	10 U
orm.....		5 U	50 U	50 U	5 U	5 U
yl-2-pentanone.....		10 U	100 U	100 U	10 U	10 U
none.....		10 U	100 U	100 U	10 U	10 U

WESTON ANALYTICS
GC/MS DATA SUMMARY
VOLATILE HAZARDOUS SUBSTANCE LIST COMPOUNDS

ch Number:	Client:	
	Cust ID:	BLANK
	RFW#:	BLANK
tion	Matrix:	Water
	D.F.:	1
	Units:	ug/l
te	Toluene-d8:	100 %
Y	Bromofluorobenzene:	102 %
	1,2-Dichloroethane-d4:	100 %

ethane.....	10	U
thane.....	10	U
hloride.....	10	U
thane.....	10	U
ne Chloride.....	1	J
.....	10	U
Disulfide.....	5	U
hloroethene.....	5	U
hloroethane.....	5	U
2-Dichloroethene.....	5	U
orm.....	5	U
hloroethane.....	5	U
one.....	10	U
richloroethane.....	5	U
Tetrachloride.....	5	U
cetate.....	10	U
chloromethane.....	5	U
loropropane.....	5	U
3-Dichloropropene.....	5	U
roethene.....	5	U
chloromethane.....	5	U
richloroethane.....	5	U
.....	5	U
-Dichloropropene.....	5	U
ethylvinylether.....	10	U
rm.....	5	U
-2-pentanone.....	10	U
one.....	10	U

tch Number: Client:

	Cust ID:	BLANK				
	RFW#:	BLANK				
	fl	fl	fl	fl	fl	fl
hloroethene	5 U					
2-Tetrachloroethane	5 U					
e	5 U					
benzene	5 U					
enzene	5 U					
e	5 U					
Xylenes	5 U					
chlorobenzene	5 U					
chlorobenzene	5 U					
chlorobenzene	5 U					
orofluoromethane	5 U					

s:

U=Analyzed, not detected. B=Present in blank. NRP=Not Reported
J=Present at less than detection limit. NR=Not requested.

WESTON Analytics

RFW #
W.O.#

1. The following qualifiers are used on the data summary:

   U - Indicates that the compound was analyzed for but not detected. The minimum detection lilmit for the sample (not the method detection limit) is reported with the U (e.g., 10U).

   J - Indicates an estimated value. This flag is used in cases where a target analyte is detected at a level less than the lower quantification level. If the limit of quantification is 10 ug/l and a concentration of 3 ug/l is calculated, it is reported as 3J.

   BS - Indicates blank spike in which reagent grade water is spiked with the CLP matrix spiking solutions and carried through all the steps in the method. Spike recoveries are reported.

   BSD - Indicates blank spike duplicate.

   MS - Indicates matrix spike.

   MSD - Indicates matrix spike duplicate.

   DL - Indicates that surrogate recoveries were not obtained because the extract had to be diluted for analysis.

   NA - Not applicable.

   DF - Dilution factor.

   NR - Not required.

   NRP - Not reported.

2. Samples Collected: 10-18,20-87
   Extraction Date: 10-21-87
   Analysis Date: 10-27,28-87

Carter P. Nulton, Ph. D. DATE
Organic Section Manager
WESTON Analytical Laboratories

WESTON ANALYTICS
PCB's

tch Number: Client:

ation	Cust ID:	PBW-1-PO	PBW-2-PO	PBW-1-CD	PBW-1-CD	PBW-1-CD	E
	RFW#:	001	002	003	003 DUP	003 MS	
	Matrix:	Water	Water	Water	Water	Water	
	D.F.:	10	1	1	1	1	
	Units:	ug/l	ug/l	ug/l	ug/l	ug/l	
ate: Di-n-butylchlorendate:		NA % fl	NA % fl	NA % fl	NA % fl	NA % fl	fl
e:							
r-1016		5.0 U	0.5 U	0.5 U	0.5 U	0.5 U	
r-1221		5.0 U	0.5 U	0.5 U	0.5 U	0.5 U	
r-1232		5.0 U	0.5 U	0.5 U	0.5 U	0.5 U	
r-1242		5.0 U	0.5 U	0.5 U	0.5 U	0.5 U	
r-1248		5.0 U	0.5 U	0.5 U	0.5 U	0.5 U	
r-1254		5.2 J	3.3	1.0 U	1.0 U	78 %	
r-1260		10.0 U	1.0 U	0.53 J	0.12 J	1.0 U	

alyzed,not detected. J=Present below detection limit. B=Present in blank. NR=Not requ
rcent recovery. NS=Not spiked.

WESTON

ROY F. WESTON, INC.

RFW #
W.O.#

1. The following qualifiers are used on the data summary:

   U - Indicates that the compound was analyzed for but not detected. The minimum detection limit for the sample (not the method detection limit) is reported with the U (e.g., 10U).

   J - Indicates an estimated value. This flag is used either when estimating a concentration for tentatively identified compounds where a 1:1 response is assumed or where the mass spectral data indicate the presence of a compound that meets the identification criteria but the result is less than the specified detection limit but greater than zero (e.g., 10J). If the limit of detection is 10 ug/L and a concentration of 3 ug/L is calculated, it is reported as 3J.

   BS - Indicates blank spike in which reagent grade water is spiked with the CLP matrix spiking solutions and carried through all the steps in the method. Spike recoveries are reported.

   BSD - Indicates blank spike duplicate.

   MS - Indicates matrix spike.

   MSD - Indicates matrix spike duplicate.

   DL - Indicates that surrogate recoveries were not obtained because the extract had to be diluted for analysis.

   NA - Not applicable.

   DF - Dilution factor.

   NR - Not required.

   NRP - Not reported.

2. Samples Collected: 8-13-87
   Extraction Date: 8-17-87
   Analysis Date: 9-11,12-87

Carter P. Nulton, Ph.D. — 9-18-87 DATE
Organic Section Manager
WESTON Analytical Laboratories

WESTON ANALYTICS
GC/MS DATA SUMMARY
SEMI-VOLATILE HAZARDOUS SUBSTANCE LIST COMPOUNDS

tch Number: Client:

tion :D.F.,except :,(3)=20x	Cust ID: RFW#: Matrix: D.F.: Units:	BLANK(906) WBLK Water 1 ug/l	B.S. B.S. Water 1 ug/l	LAB #4111 0010 Water 1 ug/l	LAB #4111 0010 DUP Water 1 ug/l	LAB #4111 0010 MS Water 1 ug/l
	2-Fluorophenol:	82 %	67 %	29 %	52 %	56 %
te	Phenol-d5:	56 %	44 %	27 %	41 %	60 %
y (%)	2,4,6-Br3-Phenol:	104 %	80 %	41 %	65 %	41 %
	Nitrobenzene-d5:	104 %	62 %	62 %	72 %	46 %
	2-Fluorobiphenyl:	106 %	70 %	68 %	66 %	52 %
	p-Terphenyl-d14:	114 %	82 %	62 %	58 %	58 %
		fl	fl	fl	fl	fl
.....		10 U	40 %	10 U	10 U	51 %
hloroethyl)Ether.....		10 U	10 U	10 U	10 U	10 U
ophenol.....		10 U	96 %	10 U	10 U	53 %
hlorobenzene.....		10 U	10 U	10 U	10 U	10 U
hlorobenzene.....		10 U	72 %	10 U	10 U	56 %
Alcohol.....		10 U	10 U	10 U	10 U	10 U
hlorobenzene.....		10 U	10 U	10 U	10 U	10 U
lphenol.....		10 U	10 U	10 U	10 U	10 U
hloroisopropyl)Ether.....		10 U	10 U	10 U	10 U	10 U
lphenol.....		10 U	10 U	2 J	5 J	10 U
so-di-n-propylamine.....		10 U	58 %	10 U	10 U	52 %
loroethane.....		10 U	10 U	10 U	10 U	10 U
nzene.....		10 U	10 U	10 U	10 U	10 U
rone.....		10 U	10 U	10 U	10 U	10 U
phenol.....		10 U	10 U	10 U	10 U	10 U
ethylphenol.....		10 U	10 U	10 U	10 U	10 U
Acid(2).....		50 U	50 U	50 U	50 U	50 U
hloroethoxy)Methane.....		10 U	10 U	10 U	10 U	10 U
hlorophenol.....		10 U	10 U	10 U	10 U	10 U
richlorobenzene.....		10 U	58 %	10 U	10 U	38 %
lene.....		10 U	10 U	10 U	10 U	10 U
roaniline.....		10 U	10 U	10 U	10 U	10 U
lororbutadiene.....		10 U	10 U	10 U	10 U	10 U
ro-3-methylphenol.....		10 U	86 %	10 U	10 U	46 %
ylnaphthalene.....		10 U	10 U	10 U	10 U	10 U
lorocyclopentadiene.....		10 U	10 U	10 U	10 U	10 U

atch Number: Client:

	Cust ID: BLANK(906)	B.S.	LAB #4111	LAB #4111	LAB #4111
	RFW#: WBLK	B.S.	0010	0010 DUP	0010 MS
-Trichlorophenol.....	10 U	10 U	10 U	10 U	10 U
-Trichlorophenol(2).....	50 U	50 U	50 U	50 U	50 U
oronaphthalene.....	10 U	10 U	10 U	10 U	10 U
roaniline(2).....	50 U	50 U	50 U	50 U	50 U
yl Phthalate.....	10 U	10 U	10 U	10 U	10 U
hthylene.....	10 U	10 U	10 U	10 U	10 U
roaniline(2).....	50 U	50 U	50 U	50 U	50 U
hthene.....	10 U	76 %	10 U	10 U	56 %
nitrophenol(2).....	50 U	50 U	50 U	50 U	50 U
rophenol(2).....	50 U	30 %	50 U	50 U	52 %
cofuran.....	10 U	10 U	10 U	10 U	10 U
nitrotoluene.....	10 U	68 %	10 U	10 U	54 %
nitrotoluene.....	10 U	10 U	10 U	10 U	10 U
l Phthalate.....	10 U	10 U	10 U	10 U	10 U
rophenyl-phenylether.....	10 U	10 U	10 U	10 U	10 U
ne.....	10 U	10 U	10 U	10 U	10 U
roaniline(2).....	50 U	50 U	50 U	50 U	50 U
nitro-2-methylphenol(2).....	50 U	50 U	50 U	50 U	50 U
rosodiphenylamine(1).....	10 U	10 U	10 U	10 U	10 U
ophenyl-phenylether.....	10 U	10 U	10 U	10 U	10 U
lorobenzene.....	10 U	10 U	10 U	10 U	10 U
hlorophenol(2).....	50 U	71 %	50 U	50 U	0 %
threne.....	10 U	10 U	10 U	10 U	10 U
cene.....	10 U	10 U	10 U	10 U	10 U
utyl Phthalate.....	10 U	NR	2 J	2 J	NR
nthene.....	10 U	10 U	10 U	10 U	2 J
.....	10 U	94 %	10 U	10 U	70 %
Benzyl Phthalate.....	10 U	10 U	10 U	10 U	10 U
ichlorobenzidine(3).....	20 U	20 U	20 U	20 U	20 U
a)Anthracene.....	10 U	10 U	10 U	10 U	10 U
Ethylhexyl)Phthalate.....	19	10 U	10 U	27	1 J
ne.....	10 U	10 U	10 U	10 U	10 U
ctyl Phthalate.....	1 J	10 U	22	17	1 J
b)Fluoranthene.....	10 U	10 U	10 U	10 U	10 U
k)Fluoranthene.....	10 U	10 U	10 U	10 U	10 U
a)Pyrene.....	10 U	10 U	10 U	10 U	10 U
(1,2,3-cd)Pyrene.....	10 U	10 U	10 U	10 U	10 U
(a,h)Anthracene.....	10 U	10 U	10 U	10 U	10 U
g,h,i)Perylene.....	10 U	10 U	10 U	10 U	10 U

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CASE NARRATIVE

Samples have been prepared and analyzed according to USATHAMA Method UW01.

According to USATHAMA protocol, the following QA/QC control samples have been analyzed concurrently with each extraction batch. Abbreviations noted below have been used in the data summary.

Abbreviation	Description
Blank -	USATHAMA standard matrix (soil or water) analyzed to provide an indication of lab contamination and it's effect on reported analytical data.

Standard matrix samples (soil or water) are spiked with nitroaromatic target compounds to provide precision and accuracy data. Standard matrix is spiked at two levels:

2XSS -	2X spike represents levels at or near twice the reported detection limit.
10XSS and 10XSSD -	10X spike represents levels at ten times the reported detection limits. Two 10X standard spikes are analyzed.
NS -	Natural spike sample(s) selected from each sample batch, analyzed at 10% frequency and spiked at ten times the detection limit.
D -	Indicates duplicate analysis of a sample.
DL -	Diluted below calibration range.
NOTE:	Spikes have been reported as result (% recovery).

Data Qualifiers:

< - Less than > - Greater than

Analysis Summary:

Weston Analytical Batch:	8710-599
Samples Collected:	10-29-87
Samples Prepared:	11-04-87
Samples Analyzed:	11-04-87

APPROVED BY _____

Carter P. Nulton, Ph.D
Lab/Organic Section Manager
WESTON Analytical Laboratories

WESTON ANALYTICS
WATER EXPLOSIVES DATA

atch Number: CLIENT:

e mation	Client ID : RFW#: D.F.: Units:	G-50 001 1 ug/L	G-53 002 1 ug/L	G-52 003 1 ug/L	AW-1 004 1 ug/L	---- BLANK 1 ug/L
.....		< 1.30	< 1.30	< 1.30	< 1.30	< 1.30
.....		< 1.26	< 1.26	< 1.26	< 1.26	< 1.26
-TNB.....		< 0.56	< 0.56	< 0.56	< 0.56	< 0.56
NB.....		< 0.61	< 0.61	< 0.61	< 0.61	< 0.61
l.....		< 0.66	< 0.66	< 0.66	< 0.66	< 0.66
-TNT.....		< 0.78	< 0.78	< 0.78	< 0.78	< 0.78
NT.....		< 0.55	< 0.55	< 0.55	< 0.55	< 0.55
NT.....		< 0.60	< 0.60	< 0.60	< 0.60	< 0.60

e mation	Client ID : RFW#: D.F.: Units:	---- 10XSS 1 ug/L	---- 10XSSD 1 ug/L	G-50 001 NS 1 ug/L
.....		12.8(98.5%)	12.1(93.1%)	12.3(94.6%
.....		5.79(91.9%)	4.68(74.3%)	6.21(98.6%
-TNB.....		5.79(103%)	5.26(93.9%)	5.10(91.1%
NB.....		6.23(102%)	5.82(95.4%)	4.96(81.3%
l.....		6.20(93.9%)	5.91(89.5%)	5.67(85.9%
-TNT.....		7.62(97.7%)	6.97(89.4%)	6.51(83.5%
NT.....		5.94(108%)	5.06(92.0%)	4.29(78.0%
NT.....		6.37(106%)	5.75(95.8%)	5.52(92.0%

A-3 Facilities and Equipment - Weston

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EQUIPMENT LIST

Analytical Instrumentation Available at WESTON Analytics
Lionville Laboratory

Type and Model	Quantity
High Pressure Liquid Chromatograph - Purchased in 1980	2
Perkin-Elmer Model LC-75, Series 211, continuously variable wave-length detector	
Ion Chromatograph - Purchased in 1984	1
Dionex Model 2020i	
Atomic Absorption Apparatus	
Perkin-Elmer Model 503 with cold vapor mercury attachment, background correction - Purchased in 1975	1
Perkin-Elmer Model 5000 with graphite furnace and background correction - Purchased in 1975	1
Perkin-Elmer Model 5000 flame/graphite furnace with Zeeman background correction, auto sampler - Purchased in 1985	2
Technicon Automatic Analyzer - Purchased in 1984	1
Gas Chromatograph/Mass Spectrophotometers	8
Finnigan Model 5100, equipped with electron ionization and chemical ionization source; varian 8000 autosampler; capillary column system INCOS data system; positive and negative ions electron multiplier detector; all glass jet separator and direct transfer line interface for capillary column system; with stand alone computer for data reduction and reporting - Purchased in 1983, 1984, 1985	4
Finnigan OWA 1020 GC/MS - electron impact quadrupole mass spectrometer, equipped with packed and capillary column injectors; INCOS data system; EPA, NIH, and special EPA-priority pollutant mass spectral library - Purchased in 1980, 1986	2

Type and Model	Quantity
Finnigan Model 4610B GC/MS - electron impact quadruple, varian 8000 autosampler, equipped with packed and capillary column capability; INCOS data system; EPA/NIH, mass spectral library - Purchased in 1985	1
Finnigan Model INCOS 50 GC/MS - electron impact quadrupole, equipped with capillary column injectors; INCOS data system; EPA, NIH and special IEPA - priority pollutant mass spectral library - Purchased 1986	1
Total Organic-Halide Analyzer - Purchased in 1984	1
Dohrmann DX-20 Analyzer System equipped with microcoulometric analyzer	
Inductively Coupled Plasma Apparatus	3
Perkin-Elmer ICP/5000 system equipped with automatic ignition system, automatic tuning, data system and automatic sampler - Purchased in 1978	1
Perkin-Elmer ICP/6500 system equipped with automatic ignition system, automatic tuning, auto sampler, and 7300 microprocessor - Purchased in 1986	1
Thermo Jarrel Ash ICP 61 S equipped with data system and random access autosampler - Purchased in 1987	1
Organic and Total Carbon Analyzers	2
Ionics Model 1270 TOC and TOC analyzer equipped with automatic sampler and infrared analyzer - Purchased in 1975	
Dohrmann Envirotech Analyzer Model DC-50 - Purchased in 1984	

WESTON

Type and Model	Quantity
Finnigan Model 4610B GC/MS - electron impact quadruple, varian 8000 autosampler, equipped with packed and capillary column capability; INCOS data system; EPA/NIH, mass spectral library - Purchased in 1985	1
Finnigan Model INCOS 50 GC/MS - electron impact quadrupole, equipped with capillary column injectors; INCOS data system; EPA, NIH and special IEPA - priority pollutant mass spectral library - Purchased 1986	1
Total Organic-Halide Analyzer - Purchased in 1984	1
Dohrmann DX-20 Analyzer System equipped with microcoulometric analyzer	
Inductively Coupled Plasma Apparatus	3
Perkin-Elmer ICP/5000 system equipped with automatic ignition system, automatic tuning, data system and automatic sampler - Purchased in 1978	1
Perkin-Elmer ICP/6500 system equipped with automatic ignition system, automatic tuning, auto sampler, and 7300 microprocessor - Purchased in 1986	1
Thermo Jarrel Ash ICP 61 S equipped with data system and random access autosampler - Purchased in 1987	1
Organic and Total Carbon Analyzers	2
Ionics Model 1270 TOC and TOC analyzer equipped with automatic sampler and infrared analyzer - Purchased in 1975	
Dohrmann Envirotech Analyzer Model DC-50 - Purchased in 1984	

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Type and Model	Quantity
Gas Chromatographs	13
Hewlett-Packard Model 5880 with level 4 programmable keyboard, alkaline flame ionization NP detector, 2 FID detectors, and electron capture detector. Model 7672A auto-sampler for continuous sample analysis - Purchased in 1982	1
Hewlett-Packard Model 5880 with level 4 programmable keyboard, flame photometric, FID, and 2 electron capture detectors. Model 7672A auto-sampler for continuous sample analysis - Purchased in 1982	1
Hewlett-Packard Model 5880 with level 4 programmable keyboard, Hall Model 700A electrolytic conductivity detector, FID, PID and ECD, Tekmar LSC-2 Purge and Trap concentrator with Tekmar Model ALS 10 sample auto-sampler - Purchased in 1983	1
Hewlett-Packard Model 5840A gas chromatograph equipped with flame ionization detector; electron capture detector; automatic sampler; capillary column systems; and automatic integrator, Model 7672A auto-sampler - Purchased in 1980	2
Hewlett-Packard Model 5880 with level 4 programmable keyboard, Hall Model 700A electrolytic conductivity detector, and FID, Tekmar LSC-2 Purge and Trap concentrator with Tekmar Model ALS 10 sample auto-sampler - Purchased in 1984	1
Perkin-Elmer SIGMA 2000 completely automated headspace analyzer with flame ionization and electron detectors - Purchased in 1984	1
Analytical Instrument Development Portable Model 511 with flame ionization detector; dual column; and sampling, column switching, and backflush valves - Purchased in 1983	1

Type and Model	Quantity
Perkin-Elmer Model SIGMA 3B isothermal and temperature programmed auto sampler for headspace analysis, packed and capillary column capability, automatic integrator - Purchased in 1978	1
Varian Model 3400 equipped with 2 electron capture detectors; Model 8000 autosampler and integrator	2
Varian Model 3400 equipped with a Hall and a photoionization detectors; Automatic Integrator, Tekmar LSC-2 Purge and Trap Concentrator with Tekmar MOdel ALS 10 port autosampler - Purchased in 1986	1
Carlo-Erba Model 5300 Maga Series, temperature programmed, packed, and capillary column, FID - Purchased in 1986	1
Gas Chromatograph Detectors	
Flame Ionization Detectors - Purchased in 1980 Hewlett-Packard NO. 18801B	2
Electron Capture Detectors - Purchased in 1980 Hewlett-Packard NO. 18803B Analog Technology Corporation Model 140A	2
Hall Electrolytic Conductivity Detectors - Purchased in 1983, 1984 Tracor Model 700A	2
Photoionization Detector - Purchased in 1984 HNV Model PI-52-02	1
Flame Photometric Detector - Purchased in 1983 Tracor Model 12003	1
Nitrogen Phosphorus Detector - Purchased in 1983 Hewlett Packard Model 18847A/8A	1
Spectrophotometers - Purchased at various times	4
pH Instruments - Purchased at various times	4

Type and Model	Quantity
Specific Ion Analyzer - Purchased in 1978	1
Orion Model 901 pH/ion meter with Model 658 switch and various specific ion electrodes	
Liquid Scintillation Counter - Purchased in 1983	
Tracor Analytic Beta Trac 6895 microprocessor base. It features automatic DPM, ESR, SCR, and is complete with printer and an internal memory	

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Analytical Instrumentation Available at WESTON Analytics
Stockton Laboratory

Type and Model	Quantity
Atomic Absorption	
Perkin-Elmer, ICP/6500 System	1
Ignition System, Automatic Tuning, with Automatic Autosampler and Microprocessor - Purchased in 1986	1
Perkin-Elmer, Model 3030, Flameless Atomic Absorption Spectrophotometer with Zeeman Background Correction - Purchased in 1987	1
Instrumentation Laboratory Model 457, single-channel double beam, with automatic sampler, graphite furnace, deuterim background correction, atomic vapor accessory and printer - Purchased in 1986	1
Ion Chromatograph	
Dionex Model 2020:	1
Technicon Automatic Analyzer II	
Sampler IV Module	1
Proportioning Pump, Model III	1
Manifolds for:	
Sulfate	1
Nitrate	1
Nitrite	1
Ortho-phosphate	1
Total phosphate	1
Kjeldahl Nitrogen	1
Ammonia	1
Colorimeter, single-channel	1
Recorder, single-channel	1
pH/Specific Ion Meters	
Fisher Accumet Model 142 pH Meter	1
Fisher Accumet Model 620 pH/Specific Ion Meter	1
Orion Ion Analyzer Model 399a pH/Specific Ion Meter	1

Type and Model	Quantity
Specific Conductivity	
YSI Model 32 Conductance Meter	2
Turbidity	
H.F. Instruments Model DRT-100	1
Gas Chromatographs	
Varian Model 3700, one with capillary column system	4
Hewlett-Packard Model 5710 with Capillary Column	1
Perkin-Elmer, Sigma 2	1
Gas Chromatograph Detectors	
Electron Capture	5
Flame Ionization	3
Sulfur - Phosphorus	4
Nitrogen - Phosphorus	2
Thermal Conductivity	1
Hall Electrolytic Conductivity	2
Photoionization	2
Gas Chromatograph Automatic Samplers	
Varian Model 8040 Automatic Samplers	6
Gas Chromatograph Data Systems	
Varian Model 604 equipped with a HP inkjet plotter	1
Varian Vista 401, each with three channels and two floppy disk drives. One with remote printer-plotter	2
Hewlett-Packard Model 3390A Integrators	2
Liquid Scintillation	
Unilux III	1

Type and Model	Quantity
Purge and Trap	
Tekmar Model LSC-2	4
Tekmar Model ALS (Automatic Liquid Sampler)	4
Centrifuge	
IEC (International Equipment Company) Model HN-42	2
Balances	
Metler HL 52, 5-place analytical	1
Metler H 80, 4-place analytical	1
Ohaus Brainweigh B 3000D, top-loader	1
Fisher Titralyzer II Titration System	
Model 387 Rotary Multi-Sampler	1
Model 395 Digital Barette/Dispenser	2
Model 385 Titrate Stirrer	1
Model 380 Electrometers	2
Model 381 FEP (Fixed Endpoint) Titrate Demand Module	1
Model 383 AEP (Automatic Endpoint) Titrate Demand Module	1
Model 382 Titrate Control Module	1
Model 386 Printer Module	1
Spectrophotometer	
Bausch and Lomb Spectromic 2000, double-beam, with X-Y recorder and printer (UV-Visible)	1
Perkin-Elmer, Model 467, Double Beam (IR)	1
Gas Chromatograph/Mass Spectrophotometer	
Finnegan, Model 1020C with Autosampler and Incos Software	1
Finnegan, Model 5100C with Autosampler and Super Incos Software	1

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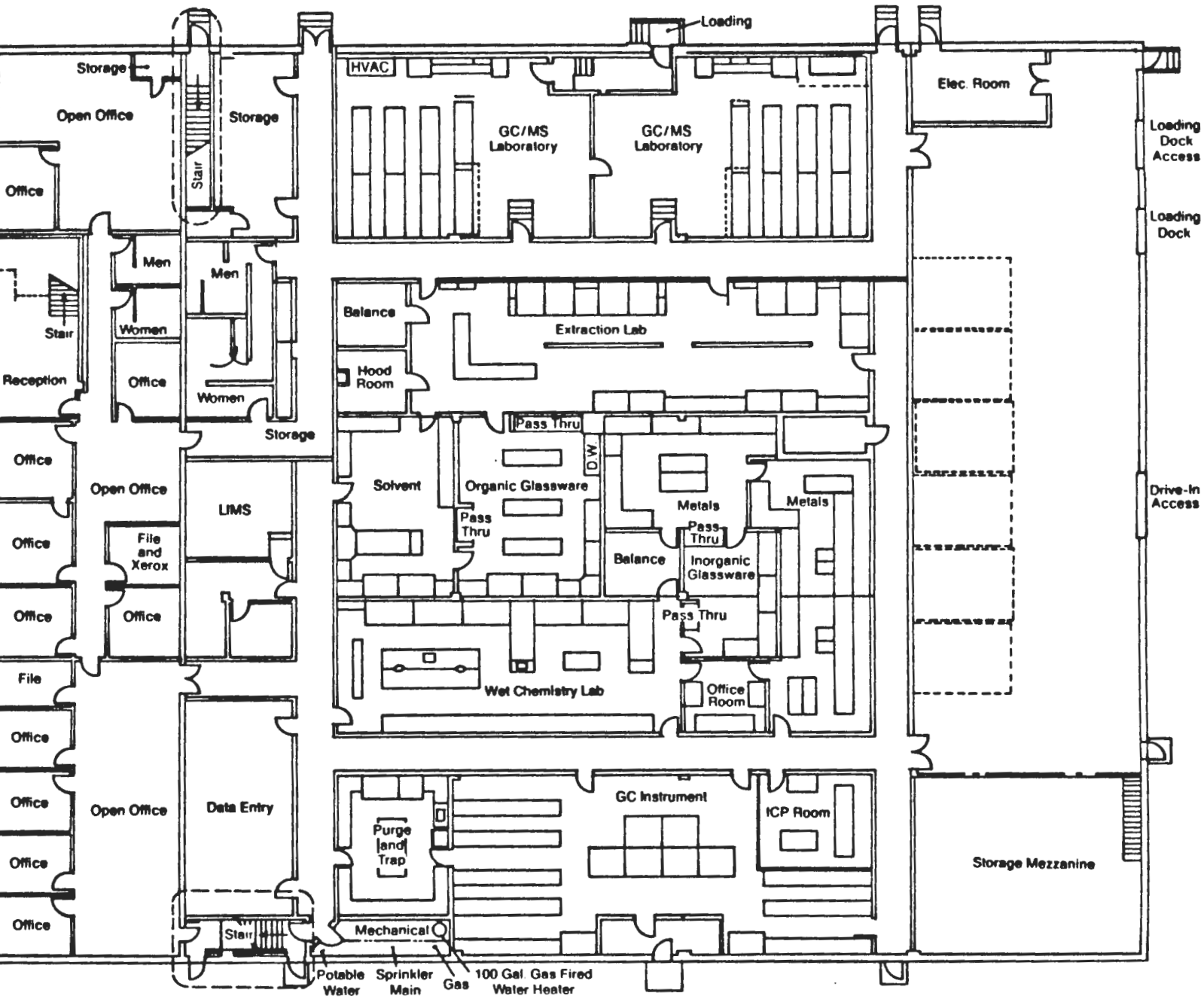
Type and Model	Quantity
Miscellaneous Equipment	
Rotavaporators	5
Ultrasonic Cleaners	2
Ovens	3
Incubator	1
Refrigerator/Freezers	12
Labcenco Micro-Kjeldahl Digestor	1
Magnetic Stirring Units, single	2
Magnetic Stirring Units, 4-place	3
Heating Plates	5
Heating/Stirring Units, single	3
Desiccators	3
Water Bath and Circulator	3
Milli-Q Water Purification System	1
Thermolyne Type 1500 Furnace	1



LIONVILLE LABORATORY
208 WELSH POOL ROAD, LIONVILLE, PA

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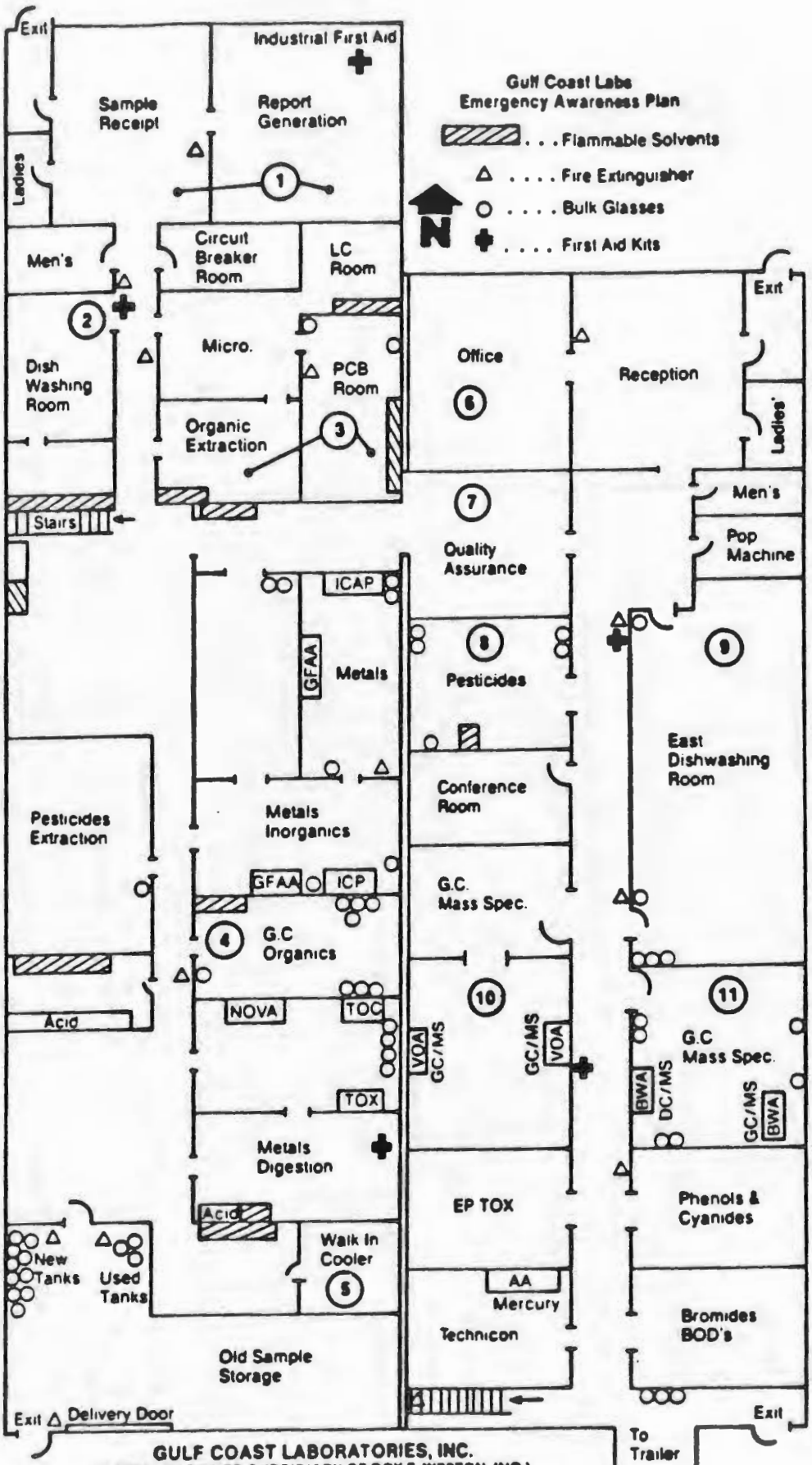

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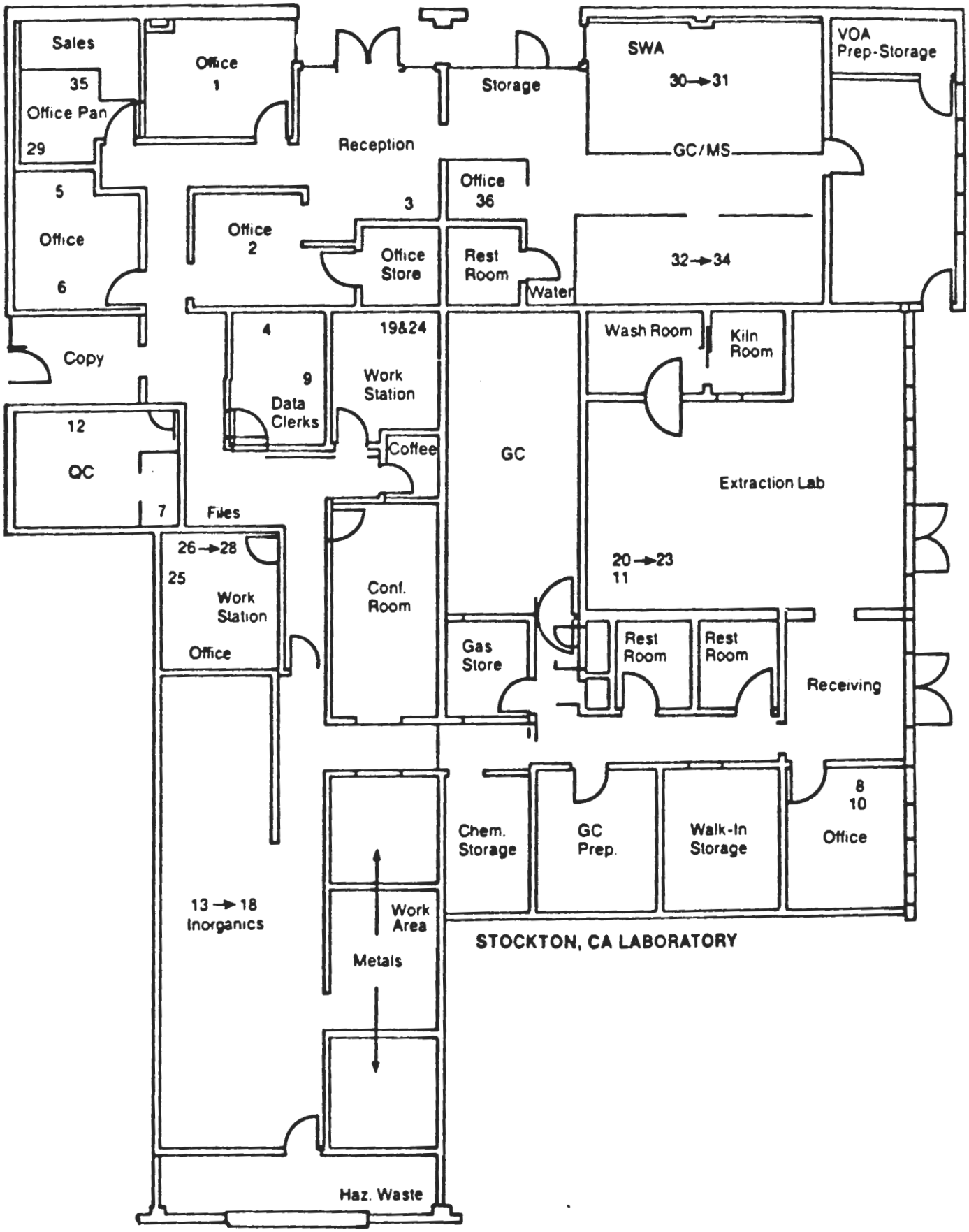
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Office Pan
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Reception
Storage
SWA
30→31
GC/MS
VOA
Prep-Storage
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Office
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3
Office
36
Office
Store
Rest
Room
Water
32→34
Copy
4
9
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Clerks
19&24
Work
Station
Coffee
GC
Wash Room
Kiln
Room
Extraction Lab
12
QC
7
Files
26→28
25
Work
Station
Office
Conf.
Room
20→23
11
Gas
Store
Rest
Room
Rest
Room
Receiving
Chem.
Storage
GC
Prep.
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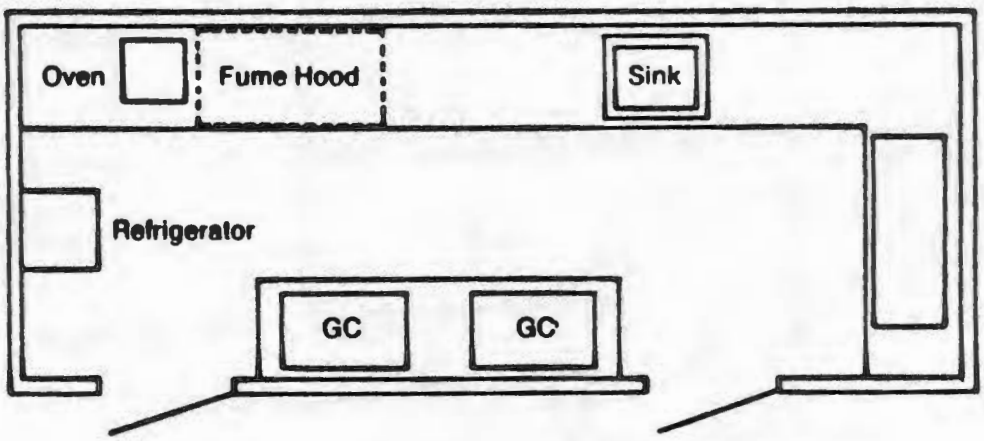
Inorganics
Work
Area
Metals
Haz. Waste
STOCKTON, CA LABORATORY



MOBILE LABORATORY

GULF COAST LABORATORIES, INC.
2417 Bond St., University Park, Illinois 60466
Phones (312) 534-5200 (219) 885-7077 (815) 723-75

MAJOR LABORATORY EQUIPMENT

Atomic Absorption/Emission Spectrophotometer - Instrumentation Laboratories Video 12E equipped with Model 188 Graphite Furnace and printer

Atomic Absorption/Emission Spectrophotometer - Instrumentation Laboratories Model 357 equipped with Model 755 Graphite Furnace and Model 254 auto sampler

Atomic Absorption/Emission Spectrophotometer - Perkin Elmer Model 306 equipped with flameless mercury cold vapor system

Atomic Absorption/Emission Spectrophotometer-Perkin Elmer Model 370

Atomic Absorption/Emission Spectrophotometer-Perkin Elmer Model 5000

Atomic Absorption/Emission Spectrophotometer - Varian Spectr AA-20 with PSC-56 programmable sample changer and arsenic-selenium hydride system

Inductively Coupled Argon Plasma (ICAP) Emission Multi-Element Spectrometer - Instrumentation Laboratories Plasma 100 with Model 254 auto sampler

Inductively Coupled Argon Plasma (ICAP) Emission Multi-Element Spectrometer - Thermo Jarrell Ash ICAP 1100 equipped with a DEC Micro PDP-11 Computer and Autosampler

GC/MS (2) - Hewlett Packard 5890A capillary gas-liquid chromatograph with 5970B mass selective detector

GC/MS (2) - Hewlett Packard 5995 benchtop utlizing either packed or capillary columns in conjunction with Tekmar Model LSC-2 purge and trap APD Model ALS auto sampler supported by HP/RTE-1000 data base

Gas Chromatograph (2) - Varian 3700 equipped with dual electron capture detectors. One unit is equipped with a Varian 8000 auto sampler

Gas Chromatograph - Tracor 565 equipped with flame photometric and flame ionization detectors

Gas Chromatograph - Tracor 565 equipped with Hall Electrolytic and flame ionization detectors with a Hitachi D-2000 Integrater

Gas Chromatograph - Tracor MT 222 equipped with dual electron capture and dual flame ionization detectors

GULF COAST LABORATORIES, INC.
2417 Bond St., University Park, Illinois 60466
Phones (312) 534-5200 (219) 885-7077 (815) 7[illegible]-7

MAJOR LABORATORY EQUIPMENT CONTINUED

Gas Chromatograph - Carle AGC Series 100 equipped with a dual thermal conductivity detector

Gas Chromatograph - Perkin-Elmer Model 8410 equipped with a flame ionization detector and GP-100 graphics printer

Gas Chromatograph - Perkin Elmer Sigma 3B equipped with flame ionization detector and Wilkes-Foxboro variable wavelength infrared detector and flame ionization detector

ABC GPC Auto Prep 1002A - Gel permeation unit equipped with chromatographic column for sample preparation

High Pressure Liquid Chromatograph - Waters Model 440 equipped with ultraviolet and refractive index detectors

High Pressure Liquid Chromatograph - Waters Model 450 equipped with variable wavelength detector and gradient elution

Technicon Autoflow Analyzer II equipped for nitrate, nitrite, phenol, cyanide, ammonia, total kjeldahl nitrogen, chloride, sulfate, phosphorus, phosphate, and alkalinity parameters

Infrared Spectrophotometer - Perkin Elmer Model 337

Total Organic Carbon (TOC) Analyzer - Xertex-Dohrmann Model DC-80

Total Organic Halogen (TOX) Analyer Xertex-Dohrmann Model DX20-A

Nova ISE Analytical auto analyer for ammonia, nitrate, and chloride parameters

UV Visible Spectrophotometer - Bausch and Lomb Spectronic 1001 with auto sampler and flow through cell

UV Visible Spectrophotometer - Perkin Elmer Lambda 1A

UV Visible Spectrophotometer - Perkin Elmer Coleman 295

Nitrogen Carbon Analyzer - Carlo Erba Model 1500

2 Tekmar Purge and Trap Concentrator Model LSC-3 for volatile and purgeable organics

2 Tekmar Liquid Sample Concentrator Model LSC-2 for organic analysis

Labconco rapid kjeldahl digestion/distillation system

Hach Colorimeter DR/1A for COD

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MAJOR LABORATORY EQUIPMENT CONTINUED

Balances (analytical):
- Mettler H72
- Mettler AC-100
- Mettler AE-200 (2)
- Mettler AE-100
- Mettler PE-2000
- Mettler PE-3000
- Ainsworth Type 10
- Cahn Model 28 Automatic Electrobalance

Balances (pan) - Sartoreous 1205 (2)

Digital pH Meters:
- Beckman Model 40 digital pH meter
- Beckman Model 21 digital pH meter (2)
- Corning Model 135 digital pH meter with ion selective electrodes for fluoride and chloride parameters
- Corning Model 140 pH meter with dissolved oxygen probe

Computers:
- Radio Shack TRS-80 Model 16B with 50 MB hard disc storage and 4 remote terminals and printer
- Radio Shack TRS-80 Model 6000 with printer
- Radio Shack TRS-80 Model II with printer
- Radio Shack TRS-80 Model 4 (2) with printers
- Perkin Elmer Sigma 10 Data Handling system with remote terminal and BASIC programming capabilities
- Commodore B032 with graphic capabilities for chromatographic data collection and manipulation
- 2 Hewlett Packard HP-1000 minicomputers with NBS Spectral Library
- Hewlett Packard 7974 9 Track Magnetic Tape
- Tandy 1000 Model SI with dual disc drive
- Commodore CDM Model 1612 HPLC control and data module with Model 8050 dual drive floppy disc

Centrifugal Grinding Mill Brinkman Instruments for sample preparation

Fisher Scientific Coal Analyer with printer

Various sizes Linear Instruments strip chart recorders

Ultrasonic probe (sonicator) - Heat Systems Ultrasonics

Fisher Isotemp Muffle Furnace Model 184A (3)

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MAJOR LABORATORY EQUIPMENT CONTINUED

Fisher Isotemp Series 200 Drying Oven

Lab Line LC Drying Oven

VWR 1660 Drying Oven

Blue M Gravity Oven

Thelco 600 Watt Oven

Market Forge Sterilmatic Autoclave

Buchi Rotary Evaporators (3) - Brinkmann Instruments

Ericsen Karl Fisher Titrator

Parr Adiabatic Colorimeter

Viscometer - Brookkfield Model RVF

Milli-Q Reagent Water System

General Metal Works high volume air sampler

Vehicles for sampling, pickups, and deliveries (6)

Field Laboratory: Sampling equipment includes pumps and bailers (PVC, Stainless Steel, Teflon), generators, control units for dedicated pumps, filtering apparatus, water level indicators, compressors, pH and Sc meters, methane gas testing equipment, personal air samplers, ISCO composite samplers and flow measuring devices.

Sampling Boats - Sea Sprite 17 foot power boat and Jon Boat - 17 foot flat bottom boat.

A-4 Special Analytical Methods

PETN, HMX, AND RDX IN WATER SAMPLES

1. APPLICATION

This method is applicable to the quantitative analysis of environmental water samples for PETN, HMX, and RDX.

A. TESTED CONCENTRATION RANGE

The tested concentration ranges in natural and standard water are listed below:

Analyte	Range (ug/L)
PETN	1.58 to 31.6
HMX	0.43 to 8.5
RDX	1.26 to 25.2

B. SENSITIVITY

The normalized responses (integrator counts) at the natural water detection limits designated in Section 1(C) are listed below:

Analyte	Integrator Counts	Nanograms
PETN	37700	281.1
HMX	121000	143.7
RDX	173000	256.2

The normalized responses (integrator counts) at the standard water detection limits designated in Section 1(C) are listed below:

Analyte	Integrator Counts	Nanograms
PETN	27179	213.1
HMX	96096	110.4
RDX	68495	91.1

C. DETECTION LIMIT

The detection limits in natural water, calculated according to Hubaux and Vos (1970), are listed below:

Analyte	Detection Limit (ug/L)
PETN	4.5
HMX	2.3
RDX	4.1

The detection limits in standard water, calculated according to Hubaux and Vos (1970), are listed below:

Analyte	Detection Limit (ug/L)
PETN	3.4
HMX	1.8
RDX	1.5

D. INTERFERENCES

This method may be subject to interferences from nonvolatile organic compounds which absorb light at 215 nm and are extractable from water with methylene chloride.

E. ANALYSIS RATE

After instrument calibration, one analyst can analyze 10 extracts in an 8-hour day. One analyst can perform approximately eight extractions in an 8-hour day.

2. CHEMISTRY

A. ALTERNATE NOMENCLATURE AND CHEMICAL ABSTRACT SERVICE (CAS) REGISTRY NUMBER

Analyte	Alternate Nomenclature	CAS Registry Number
PETN	Pentaerythrite tetranitrate Pentaerythritol tetranitrate 2,2-Bis[(nitrooxy)-methyl]-1,3-Propanediol dinitrate (ester) Nitropentaerythritol Pentrit	78-11-5
HMX	Cyclotetramethylenetetranitramine Octahydro-1,3,5,7-tetrazocine 1,3,5,7-Tetranitro-1,3,5,7-tetrazacyclooctane Octogen	2691-41-0
RDX	Cyclotrimethylenetrinitramine Hexogen, T-4, Cyclonite, Hexahydro-1,3,4-trinitro-s-triazine	121-84-4

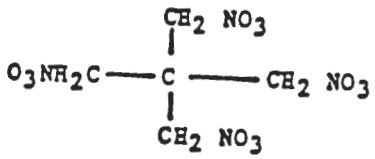
B. PHYSICAL AND CHEMICAL PROPERTIES OF ANALYTE

Analyte	Formula	Melting Point (°C)	Boiling Point	Density (g/ml)
PETN	$C_5H_8N_4O_{12}$	141	180 at 50 torr	1.77
HMX	$C_4H_8N_8O_8$	276	—	1.77-1.96*
RDX	$C_3H_6N_6O_6$	204.1	—	1.816

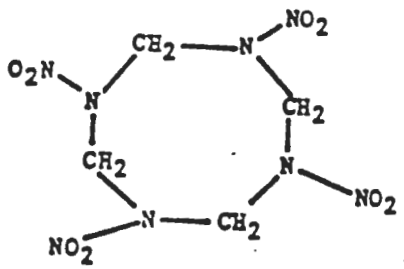
* There are four polymorphic forms of HMX with this range of densities.

Chemical Structures

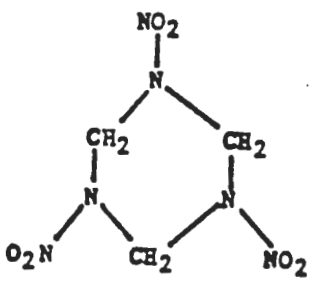
PETN



HMX



RDX



C. CHEMICAL REACTIONS

All of these compounds are highly explosive, and caution should be used in handling. Each compound is subject to alkaline hydrolysis in aqueous solution.

3. APPARATUS

A. INSTRUMENTATION

Altex Model 322 dual-pump liquid chromatograph equipped with a Perkin-Elmer LC-75 variable-wavelength detector interfaced to a Spectra Physics Model 4100 computing integrator.

B. HPLC INSTRUMENTAL PARAMETERS

1. Detector: Perkin-Elmer LC-75 variable-wavelength detector (λ = 215 nm)
2. Column: Zorbax-CN (4.6-mm ID x 25 cm)
   Particle size: 7-8 um
3. Flow Rate/Mobile Phase: 1 ml/min/35% H_2O/65% methanol
4. Temperature: 22°C
5. Injection Volume: 250 ul, fixed loop
6. Retention Times:

Analyte	Retention Time (Minutes)
RDX	7.8
HMX	11.8
PETN	13.9

C. HARDWARE/GLASSWARE

1. 1-liter separatory funnel (Teflon® or glass) (8).
2. 500-ml K-D flask (8).
3. 15-ml K-D receiver (8).
4. 3-ball Snyder column (8).
5. 2-ball micro-Snyder column (8).
6. 10-ml graduated centrifuge tubes (8).
7. Disposable glass pipettes.

D. CHEMICALS

1. Nanograde methylene chloride--J.T. Baker Company.
2. HPLC-grade acetonitrile--J.T. Baker Company.
3. HPLC-grade water--J.T. Baker Company.
4. Anhydrous sodium sulfate--reagent grade.
5. HPLC-grade methanol.

4. STANDARDS

A. CALIBRATION STANDARDS

Separate calibration stock solutions are prepared for each analyte. A composite working calibration standard is prepared from these solutions.

1. The RDX stock calibration standard (6,310 ug/ml) is prepared by weighing 63.1 mg of RDX in a 10-ml volumetric flask, dissolving the RDX in a few ml of acetonitrile, and diluting to the mark with acetonitrile. An intermediate RDX stock calibration standard is prepared by pipetting 1 ml of the RDX stock calibration standard into a 100-ml volumetric flask and diluting to the mark with methanol to give a solution containing 63.1 ug/ml of RDX.
2. The HMX stock calibration standard (5,320 ug/ml) is prepared by weighing 53.2 mg of HMX in a 10-ml volumetric flask, dissolving the HMX in a few ml of acetonitrile (a drop of acetone is added to aid in solubilization), and diluting to the mark with acetonitrile. An intermediate HMX stock calibration standard is prepared by pipetting 1 ml of the HMX stock calibration standard into a 50-ml volumetric flask and diluting to the mark with methanol to give a solution containing 106.4 ug/ml of HMX.
3. The PETN stock calibration standard (3,950 ug/ml) is prepared by weighing 39.5 mg of PETN in a 10-ml volumetric flask, dissolving the PETN in a few ml of acetonitrile (a drop of acetone is added to aid in solubilization), and

diluting to the mark with acetonitrile. An intermediate PETN stock calibration standard is prepared by pipetting 1 ml of the PETN stock calibration standard into a 50-ml volumetric flask and diluting to the mark with methanol to give a solution containing 79.0 ug/ml of PETN.

4. Prepare a series of composite working calibration standards by making dilutions of the intermediate calibration standards with 50% methanol/50% water as follows:

Working Calibration Standard	Intermediate Standard Diluted	Volume of Standard Used (ml)	Final Volume (ml)
B	RDX	5	50
	HMX	1	
	PETN	5	
C	RDX	5	100
	HMX	1	
	PETN	5	
D	Standard B	5	25
E	Standard B	5	50
F	Standard B	5	100

Working Calibration Standard	Concentration (ug/ml)		
	RDX	HMX	PETN
B	6.31	2.13	7.90
C	3.15	1.06	3.95
D	1.26	0.426	1.58
E	0.631	0.213	0.790
F	0.315	0.106	0.395

B. CONTROL SPIKES

1. The working control spike solutions are prepared in the same manner as the working calibration standards using the same letter designations for the different solutions; therefore,

the Working Control Spike Solution B has the same concentration as the Working Calibration Standard B.

2. Pipette 2 ml of the corresponding working control spike solutions into 500 ml of standard or natural water. The solutions used are selected to provide a concentration range of 0.5 to 10 times the desired detection limit.
3. Determine the precision, accuracy, and detection limits for each analyte.

Working Control Spike Used	Analyte	Analyte Concentration in the Working Control Spike Solution (ug/ml)	Spiked Analyte Concentration in Water (ug/L)
--		--	0.0
B	RDX	6.31	25.2
	HMX	2.13	8.5
	PETN	7.90	31.6
C	RDX	3.15	12.6
	HMX	1.06	4.26
	PETN	3.95	15.8
D	RDX	1.26	5.04
	HMX	0.426	1.70
	PETN	1.58	6.32
E	RDX	0.631	2.52
	HMX	0.213	0.851
	PETN	0.790	3.16
F	RDX	0.315	1.26
	HMX	0.106	0.426
	PETN	0.395	1.58

5. PROCEDURE

A. EXTRACTION

1. Measure 500 ml of the water sample into a 1-L separatory funnel.
2. Check the pH of the sample with pH paper, and adjust the pH to neutral, if necessary.

3. Extract the sample sequentially with three 100-ml portions of methylene chloride. After each portion has been added, shake the funnel vigorously for at least 5 minutes.
4. Let the layers separate for about 2 minutes after each extraction.
5. Draw off the methylene chloride and pass through a glass funnel filled with a small plug of glass wool and about 1 inch of anhydrous sodium sulfate into a 500-ml K-D flask fitted with a 10-ml K-D receiver.
6. After the third extract has been transferred to the K-D flask, rinse the sodium sulfate in the funnel with approximately 20 ml of methylene chloride.
7. Add a boiling chip (Hengar) to the methylene chloride extract in the flask and attach a 3-ball Snyder column to the apparatus.
8. Concentrate the methylene chloride extract by placing the K-D apparatus in an 80°C water bath. Immerse the receiver of the K-D nearly up to the joint.
9. The balls of the Snyder column should actively chatter when the solvent is evaporating.
10. When the apparent volume of the solution remaining in the receiver is about 1 ml, remove the apparatus from the water bath and allow to cool. After about 1 ml of methylene chloride has drained into the receiver, remove the receiver from the K-D flask.
11. Add approximately 2 ml of HPLC methanol to the receiver. Attach a 2-ball micro-Snyder column and reconcentrate. When the apparent volume in the receiver reaches 0.5 ml, remove the receiver from the water bath.
12. Repeat Step 11 two times.
13. Detach the micro-Snyder column from the receiver. Transfer the extract into a 10-ml graduated centrifuge tube rinsing quantitatively with HPLC acetonitrile. Raise the extract

volume to 1.0 ml in the centrifuge tube with HPLC methanol. Dilute to 2 ml with HPLC water.

14. Transfer to a 5-ml amber, septum-sealed vial for storage at 4°C.
15. The extract is now ready for chromatography by HPLC.

B. CALIBRATION

1. Inject Working Calibration Standards G, F, E, D, C, and B and a blank singly at the beginning of the analytical run. Inject Working Calibration Standard D at the conclusion of the analytical run to verify constant instrument response.
2. Plot the normalized integrator areas versus nanograms/microliter of each standard to obtain a working curve.

C. ANALYSIS

1. Inject 250 ul of the extract onto the HPLC column.
2. Perform the analysis of the sample according to the conditions given in Section 3(B).
3. Measure the response of the sample for the components of interest.

6. CALCULATIONS

Determine the concentration of RDX according to the following formula:

$$\text{Concentration (ug/L)} = \frac{(A)(V_t)}{V_s}$$

where: A = Concentration (ug/ml) of analyte found in the sample by comparison with the appropriate standard curve (ug/ml),

V_t = Volume of total extract (ml), and

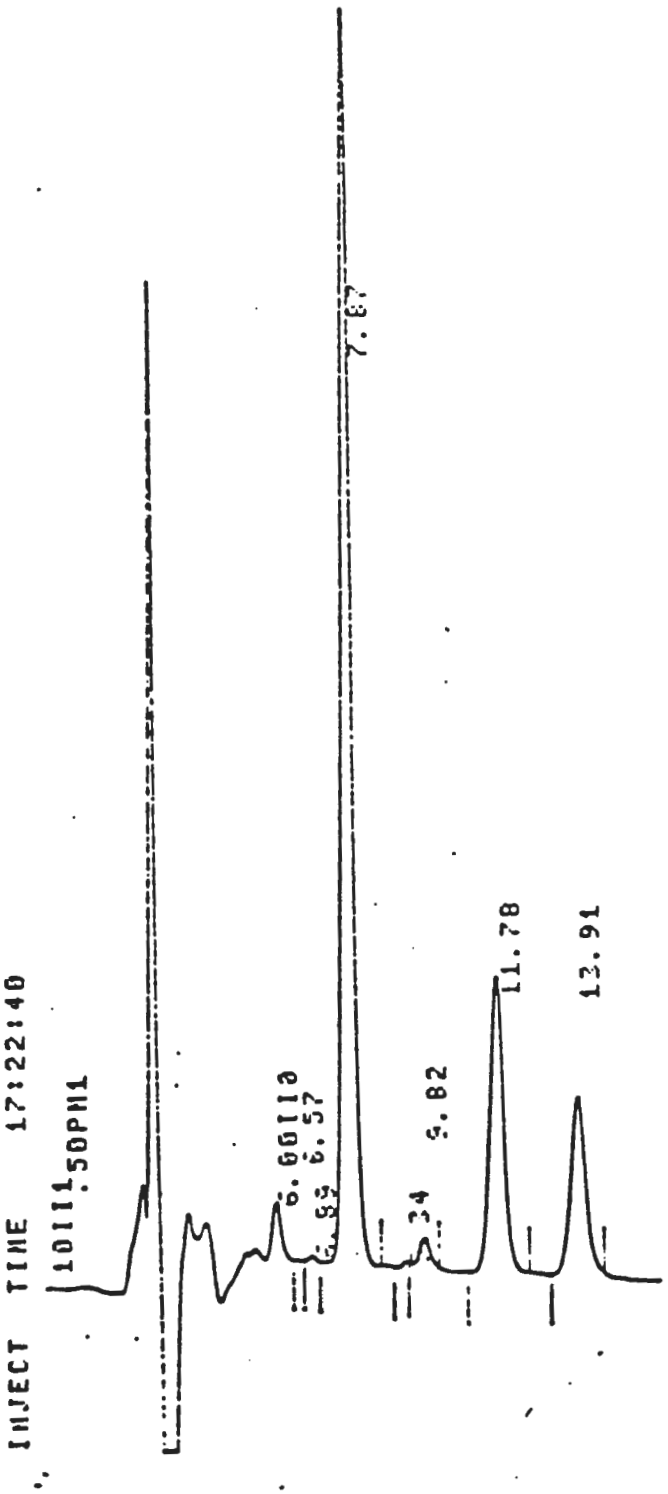
V_s = Volume of initial sample extracted (L).

7. REFERENCES

None found.

8. DATA

See attached data sheets.



Chromatogram of Standard Water Spiking Experiment

Analyte	Amount Spiked	Retention Time
RDX	12.6 ug/L	7.87 min
HMX	4.3 ug/L	11.78 min
PETN	15.8 ug/L	13.91 min

EXPLOSIVES IN WATER

I. SUMMARY

A. Analytes:

24DNT	2,4-Dinitrotoluene
26DNT	2,6-Dinitrotoluene
246TNT	2,4,6-Trinitrotoluene

B. Matrix: WATER

C. General Method: A water sample is filtered, 2% NaCl added, and injected onto the HPLC for analysis.

II. APPLICATION

A. Tested Concentration Range:

24DNT	0.60 - 60.0 ug\L
26DNT	0.55 - 54.8 ug/L
246TNT	0.78 - 75.3 ug\L

B. Sensitivity:

Peak Height in mm at an Attenuation of 2^3

24DNT	16 mm for 7.5 ug\L
26DNT	9 mm for 7.5 ug\L
246TNT	11 mm for 12.5 ug\L

C. Detection Limits:

24DNT	0.60 ug/L
26DNT	0.55 ug/L
246TNT	0.78 ug/L

D. Interferences:

Any compound that is water soluble and gives a retention time similar to the nitro-compounds and absorbs at 250 nm.

E. Analysis Rate:

After instrument calibration, one analyst can analyze two samples in one hour. One analyst can conduct sample preparation at a rate of three samples per hour. One analyst doing both sample preparation and the HPLC analysis can run 16 samples in an 8-hour day.

F. Safety information:

Work in well-ventilated areas. Wear adequate protective clothing to avoid skin contact. Wash skin with soap and water thoroughly immediately after contact.

TNT is classified as Explosives A by DOT. Avoid extreme temperatures and pressures.

III. APPARATUS AND CHEMICALS

A. Glassware/Hardware

1. Syringes: 10 uL, 50 uL, 100 uL, 1 mL syringe (Hamilton 1005 TEFLL)

2. Vials with Teflon-lined caps or septa. Nominal volume of 1.8 m L, 4.0 mL and 12 mL.

3. B-D Glaspak disposable syringes, 10 mLs, with frosted tip

4. 0.45 micron nylon filters

5. 10 mL pippette

B. Instrumentation

1. Perkin-Elmer Series 4 High Performance Liquid Chromatograph (HPLC) equipped with a Micromeritics variable wavelength detector 786. A Hewlett-Packard 3390 recording integrator in peak height mode was used to record data output.

2. Rheodyne 10-port manual injector

3. Analytical balance capable of weighing 0.01 grams for sample preparation and 0.1 mg for standard preparation. Mettler AE 163 or equivalent.

4. Parameters

a. Columns:

1) DuPont Zorbax® ODS 4.6 mm i.d. x 25 cm HPLC column with a particle size of 5-6 microns.

2) DuPont Permaphase® ODS guard column. (optional)

b. Mobile Phase: The water/methanol ratio must be adjusted as described in the calibration Section V.A.5.c to obtain optimum peak separation.

44-55% water
28-34% methanol
16-22% acetonitrile

c. Flow: 1.6 mL/min with a pressure of approximately 2860 psig.

d. Detector: 250 nm

e. Injection Volume: 10 mls

f. Retention Times:

	Minutes
24DNT	13.70-14.10
26DNT	12.80-13.50
246TNT	11.50-12.80

C. Analytes

1. Chemical Abstracts Registry Numbers

24DNT	121-14-2
26DNT	606-20-2
246TNT	118-96-7

2. Chemical Reactions

None. Explosives are measured directly on the HPLC.

3. Physical Properties

	Formula	Mol. Wt.	M.P. (°C)	B.P. (°C)
24DNT	$C_7H_6N_2O_4$	182.14	71	300 (decomposes)
26DNT	$C_7H_6N_2O_4$	182.14	66	-
246TNT	$C_7H_5N_3O_6$	227.13	82	240 (decomposes)

D. Reagents and SARMs:

1. Acetonitrile, distilled in glass for HPLC use

2. Methanol, distilled in glass for HPLC use

3. Water, distilled in glass for HPLC use

4. Sodium Chloride ACS Reagent grade

5. Standard Analytical Reference Materials

24DNT SARM No. 1147(PA 1298)
26DNT SARM No. 1148(PA 1299)
246TNT SARM No. 1129(PA 1297)

IV. CALIBRATION

A. Initial Calibration

1. Preparation of Standards:
   a. Stock calibration solutions containing approximately 10,000 ug/L of a nitro-compound are prepared by accurately weighing ca. 50 mg of a SARM into a 5 mL serum bottle and dissolving the nitro-compound in 5 mL of acetonitrile pipetted into the bottle. All stock solutions prepared in this manner and stored in a freezer (0°- 4° C) have remained stable for a period of 6 months.

b. Intermediate Calibration Standards: All compounds appear to be stable for at least 3 months.

1) Intermediate Calibration Standard A (high level): Combine the appropriate volumes of stock calibration standard as shown below. Dilute to 5 mL with acetonitrile and seal with a Teflon-lined cap. Store in the dark at 0°-4°C. The resulting solution will have the concentrations indicated in the following table.

Nitro-compound	uL of Stock Cal Std	Resulting concentration (ug/mL)
24DNT	75	150
26DNT	75	150
246TNT	125	250

2) Intermediate Calibration Standard B (low level): 1:10 dilution of the Intermediate Calibration Standard A is made in acetonitrile. Seal with a Teflon-lined cap and store in the dark at 0-4°C. The resulting solution will have the following concentrations:

Nitro-Compound	Resulting conc. (ug/mL)
24DNT	15.0
26DNT	15.0
246TNT	25.0

c. Working Calibration Standards: Using the following table, prepare a series of nine calibration standards. Pipette 10 mLs HPLC grade water into a 10 mL vial prepared with 0.2 grams NaCl. Inject the indicated volumes of intermediate calibration standard A or B into the 10 mLs of 2% NaCl with a microliter syringe. Seal the vial with a teflon-lined septum and cap. Mix well. These solutions are prepared immediately prior to injection.

WORKING CALIBRATION STANDARDS

Conc.	Amt. (uL) Interaed. Cal. Std. to Add A	B	Amt. (mL) Mobile 2.0% Phase NaCl	Resulting Concentration (ug/L) 246TNT	26DNT 24DNT
0	-	0.0	10.0	0.0	0.0
0.5 X	-	0.25	10.0	0.625	0.375
1.0 X	-	0.50	10.0	1.25	0.75
2.0 X	-	1.0	10.0	2.50	1.50
5.0 X	-	2.5	10.0	6.25	3.75
10 X	-	5.0	10.0	12.5	7.50
20 X	-	10.0	10.0	25.0	15.0
50 X	2.5	-	10.0	62.5	37.5
100 X	5.0	-	10.0	125	75.0

2. Instrument Calibration

   a. Set up the instrument according to the manufacturer's recommendations.

   b. 2% NACl water is analyzed as a blank to verify a stable baseline.

   c. Analyze the medium calibration standard (10X) to verify peak separation and retention times.

   d. Analyze the calibration standards prepared in Section IV.A.1.

3. Analysis of Calibration Data

   a. Tabulate the calibration standard concentration versus the peak height response for each calibration standard.

   b. Perform a linear regression analysis on the calibration data plotting peak height vs. concentration in ug/l.

4. Calibration Checks

   a. After completion of analyses of samples, a calibration standard at the highest concentration is analyzed. The response must

agree within 25% for that concentration from the first seven calibration curves. Thereafter, the response must agree within two standard deviations of the mean response for that concentration. If it does not, the calibration standard will be reanalyzed. If the calibration standard fails this test, initial calibration must be performed, and all samples analyzed since the last acceptable calibration must be reanalyzed.

b. No certified calibration check standards are available for these compounds.

B. Daily Calibration

1. Prior to analyses each day, a high calibration standard will be analyzed. For the first seven determinations at this concentration, the response must agree within 25% of the mean of all previous responses. After seven determinations, the response must agree within ± two standard deviations (± 2σ) of the mean response for previous determinations at this concentration.

2. If the calibration standard fails this test, it will be reanalyzed. If the calibration standard fails the second test, the system will have failed daily calibration, and initial calibration will be performed.

3. After completion of sample analyses each day, the high calibration standard will be analyzed again. The response for this calibration standard will be subjected to the criteria discussed in Section IV.B.1, above. If the response fails the criteria, the standard will be reanalyzed. If the second response fails the test, the system will have failed calibration, and initial calibration will be performed. All samples analyzed since the last acceptable calibration must be reanalyzed.

V. Certification Testing

Control spikes are prepared the same way as the working calibration standards in section IV.A.1.c.

VI. SAMPLE HANDLING STORAGE

A. Sampling Procedure: The stability of explosives in water is not fully known. Precautions should be taken to avoid prolonged exposure to light and heat.

B. Containers: 1 L amber glass bottles with teflon-lined lids.

C. Storage Conditions: Samples should be maintained at 4° C from the time of collection until the time of analysis. No chemical preservatives are necessary.

D. Holding Time Limits: Since no extractions are performed, aqueous samples must be analyzed within seven days.

E. Solution Verification: No certified check standards are available.

VII. PROCEDURE

A. Separations

1. Accurately weigh 0.2 grams of NaCl into a 10 mL vial.

2. Using a 10 mL syringe fitted with a 0.45 micron nylon disposable filter, pour the sample into the syringe barrel.

3. Force at least 10 mLs of the filtrate into a 10 mL vial prepared with 0.2 g NaCl.

4. The filtrate is ready for analysis by HPLC.

B. Chemical Reactions: None. Compounds are read directly on the HPLC.

C. Instrumental Analysis:

1. Set the chromatographic conditions as follows:

	Time (minutes)	Flow (mls/min.)	MeCN %	MeOH %	HOH %
Equilibrium	2	1.6	16	34	50
Analysis Run	20	1.6	16	34	50

2. Using the manual injector manufacturer's recommended procedure, introduce 10 mL of the medium level calibration standard into the chromatographic system. Check the chromatogram to ensure separation of the nitrated toluenes. If necessary, adjust the water/methanol ratio of the mobile phase until separate peaks are distinguished. As the column ages, less methanol is required. Generally , the column ages rapidly the first 24 hours, after which it is fairly stable.

3. Once good peak separation is obtained, introduce 10 mL of each working calibration standard, samples, and spikes into the chromatographic system using the manual injector manufacturer's recommended procedure.

VIII. CALCULATIONS

No calculations are necessary. Target compound concentration vs. response is plotted directly into a linear regression program. Sample response is entered and concentration is provided from the linear regression.

IX. DAILY QUALITY CONTROL

A. Control Samples

1. Daily control samples are prepared in a manner identical to that described in Section V. A total of three control spikes are required on a daily basis: two at 10X and one at 2X. They will have the following concentrations.

Conc.	Amt (uL) Intermed. Spiking B to add to 2.0 mLs Acetonitrile	246TNT	24DNT 26DNT
2X	10	2.0	1.2
10X	50	10.0	6.0

2. At least one method blank using ASTM Type II water is also analyzed with each analytical lot.

3. At least one matrix spike (actual sample) at 10X is analyzed for each analytical lot or at a frequency of 10%, whichever is more frequent.

B. Control Charts:

1. Average Percent Recovery (X)

   a. Percent recoveries for the 10X certification spikes from days 1 and 2 are averaged to obtain the first value to be plotted.

   b. Percent recoveries for the 10X certification spikes from days 3 and 4 are averaged to obtain the second value to be plotted.

   c. Percent recoveries for the method spikes closest to the certification 10X from the first day of analyses are averaged to obtain the third value to be plotted.

   d. Values from a, b, and c are averaged to determine the central line of the control chart.

e. Differences in percent recoveries for each pair of values in a, b, and c are averaged to obtain R.

f. The upper and lower warning limits are ± 1.25 R from the central line.

g. The upper and lower control limits are ± 1.88 R from the central line.

2. Difference in percent recoveries (R)

a. The value for R obtained in Section IX.B.1.e, above, is the base line of the control chart.

b. The warning limit is 2.511 R.

c. The control limit is 3.627 R.

3. Three Point Moving Average X

a. The average percent recovery from the 2X concentration from the first three days of certification testing is the first point to be plotted.

b. Subsequent points to be plotted are the average percent recoveries from the 2X concentration from the next group of three determinations (e.g., certification days 2. 3. and 4; certification days 3 and 4 and the first day of analysis; certification day 4, day 1 of analysis, and day 2 of analysis; etc.)

c. The central point on the control chart is the average of the plotted points and changes with each added point.

d. The range for each point is the difference between the highest and lowest values in each group of three determinations. The average range (MAR) is used to define the warning and control limits.

e. The upper and lower warning limits are ± 0.682 MAR, respectively.

f. The upper and lower control limits are ± 1.023 MAR, respectively.

4. Three Point Moving Average R:

a. The base line is the MAR.

b. The warning limit is 2.858 MAR.

STANDARD DIRECTORY

CORN 01	ANNE	03/19/87	00/00/00	2	CORNHUSKER - Quals & Experience
02	M1383E0100	02/27/87	02/16/87	66	CORNHUSKER-Resume
03	M1383E0102	02/16/87	02/16/87	4	CORNHUSKER - Lurie Resume
04	M1383E0103	02/16/87	02/16/87	5	CORNHUSKER
05	M1383E0104	02/18/87	02/16/87	10	CONRHUSKER
06	M1383L0101	02/23/87	02/23/87	2	CORNHUSKER - Letter to U.S. Army
07	M1383R0100	02/16/87	02/16/87	2	CORNHUSKER - Executive Summary
08	M1383R0101	02/16/87	02/16/87	32	CORNHUSKER-Vol I Operation Plan
09	M1383R0102	02/12/87	02/04/87	11	CORNHUSKERS - SamAnalQualCont/DataM m
CORN 10	M1383R0104	02/14/87	02/11/87	3	CORNHUSKER-Remaining text from commin
11	M1383R0105	02/16/87	02/16/87	47	CORNHUSKER-Vol I (Cont) Work Plan
LOUS 01	M1383R0201	02/12/87	02/05/87	28	LOUISIANA - Sections 1 & 2
CORN 12	M1383R0202	02/16/87	02/16/87	9	CORNHUSTER-Contractor QA/QC
LOUS 02	M1383R0203	02/15/87	02/11/87	22	LOUISIANA - Oper Plan (Obsolete)
LOUS 03	M1383R0204	02/16/87	02/16/87	51	LOUISIANA-Vol I (Cont) Work Plan
LOUS 04	M1383R0205	02/16/87	02/16/87	31	LOUISIANA - Operation Plan
CORN 13	M1383R0301	02/16/87	02/16/87	20	CORNHUSKER-Corp Experience
14	M1383R0302	02/12/87	01/22/87	2	CORNHUSKER-EXPERIENCE
15	M1383R0305	02/16/87	02/16/87	6	CORNHUSKERS - Advanced Agreements
16	M1383R0306	02/16/87	02/16/87	3	Cornhusker Subcont. Mgmt. Plan
17	M1383R0307	02/16/87	02/16/87	3	CORNHUSKERS - Resource Util. Plan
18	M1383R0308	02/16/87	02/16/87	2	CORNHUSKERS - 5. Home Office Suppor
19	M1383R0309	02/16/87	02/16/87	1	CORNHUSKERS - 1. Experience
20	M1383R0400	02/15/87	02/15/87	1	CORNHUSKER - TofC, Vol 4
21	M1383R0401	02/15/87	02/15/87	1	CORNHUSKER - Vol. 4 Army Amm. Plant
22	M1383R0COM	02/15/87	02/05/87	22	CORNHUSKER - Sections 1 & 2
23	M1383T0301	02/15/87	02/15/87	2	Cornhusker - Table
24	M1383X0100	02/16/87	02/16/87	1	CORNHUSKER - TOC Volumes 1-3
25	M1383X0101	02/12/87	01/26/87	8	CORNHUSKER SHERP
26	M1383X0103	03/16/87	03/12/87	19	CORNHUSKERS - Company Experience Form
27	M1383X0104	02/12/87	02/12/87	2	CORNHUSKER - ThermALL Qualifications
28	M1383X0105	02/16/87	03/12/87	4	CORNHUSKERS - Corporate Commitments
29	M1383X0106	03/16/87	03/12/87	25	CORNHUSKER - Extra Experience
30	M1383X0107	02/16/87	02/16/87	1	CORNHUSKER - Mobilization for PRASA
31	M1383X0303	02/13/87	02/13/87	24	CORNHUSKER CO. EXP. - EXTRA COPY