PARSONS ENGINEERING SCIENCE, INC.
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November 22, 1999

Mr. Julio Vazquez
USEPA Region II
Emergency & Remedial Response Division
290 Broadway, 18th Floor
New York, NY 10007-1866

Mr. James Quinn
New York State Department of Environmental Conservation
Bureau of Eastern Remedial Action
Division of Hazardous Waste Remediation
50 Wolf Road
Albany, NY 12233-7010

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SUBJECT: Sampling & Analysis Plan for Re-Sampling of Pu-239/240 of Soil and Sediment at SEAD-12, Seneca Army Depot Activity, Romulus, NY

Dear Mr. Vazquez/Mr. Quinn:

The purpose of this sampling and analysis plan is to (1) provide background information supporting the rationale for performing additional sampling in soil and sediment at SEAD-12 for Plutonium 239/240; (2) establish re-sampling objectives; (3) propose re-sampling locations in sediment and soil at SEAD-12; and (4) propose a sampling schedule.

1.0 Background Information

A total of 516 soil samples (38 background), 68 sediment samples (20 off-site), 68 surface water samples (21 off-site) and 50 (9 background) groundwater samples have been collected to date at SEAD-12 and analyzed for radionuclides of concern that includes Pu-239/240. Soil samples were collected in two phases during the Fall of 1997 and the Fall of 1998. Sediment and surface water samples were collected in Fall of 1997 and the first round of groundwater samples were collected this Spring, 1999. Detected results for Pu-239/240 are presented in Tables 1 and 2 for soil and sediment. No Pu-239/240 was detected in surface water or groundwater.

Radionuclide results for background samples were presented to USEPA and NYSDEC in a submittal entitled "Background Sample Analyses for Plutonium-239/240 at SEAD-12, Seneca Army Depot Activity" on July 2, 1999. Although all background results were presented in the submittal, Pu-239/240 results were its focus. Comments were received on October 27, 1999 from NYSDEC and on November 2, 1999 from USEPA concerning the results from background samples. A follow-up conference call was held on November 4, 1999 to discuss a plan to resolve USEPA and NYSDEC concerns over the quality of laboratory results received for Pu-239/240 during the initial sampling event. Responses to specific USEPA and NYSDEC comments are addressed separately. However, this sampling and analysis plan is focused on resolving questions over the quality of the Pu-239/240 data from the site.

Of the samples collected, 61 surface soil samples (three background), thirteen subsurface soil samples (five background), and 26 sediment samples (2 background) reported validated detections of Pu-239/240. These samples are the subject of concern and are targeted for re-sampling. No validated detections of Pu-239/240 were reported for surface water and the first round of groundwater.

The majority of detections of Pu-239/240 in both sediment and soil were detected between 0.1 and 0.2 pCi/g. The lower limit of detection for these samples was 0.1 pCi/g. One sample collected from outside Building 804 (SS12-139) detected Pu-239/240 at 1.0 pCi/g. Figures 1.1, 1.2 and 1.3 show the locations where Pu-239/240 was detected in soil. The error associated with these results was typically between 0.1 and 0.2 pCi/g. Figures 2.1 and 2.2 show the locations where Pu-239/240 was detected in sediment. Tables 1 and 2 summarize the detections of Pu-239/240from these two media.

In Parsons ES' submittal to you on July 2, 1999, we explained that due to the frequency of detections of Pu-239/240 at the detection limit, the error associated with the results, and the frequency of detections of Pu-239/240 in the method blanks, we believed that the "hits" of Pu-239/240 at the detection limit were due to analytical error. In order to support our verify that this is the case, we propose to re-collect and re-sample those locations where Pu-239/240 was detected by the laboratory.

The NYS Department of Health has raised concerns over the level of Pm-147 and Ra-226 that has been detected in samples from both background and site samples. These concerns are addressed under separate cover in responding to their comments dated 10/27/99. No additional sampling is proposed in addressing NYSDOH's concerns about Pm-147 and Ra-226 at this time.

2.0 Study Objective

The purpose of the re-sampling effort is to verify that the activity of Pu-239/240 reported by the laboratory in the initial sampling event are not present at the reported values. In order to do this, samples where detections were reported (upon data validation) will be re-collected and re-analyzed. Re-analysis will be for Pu-239/240 at a detection limit ten times less than that originally requested. A detection limit of 0.01 pCi/g will be requested. Results from the initial sampling event will be compared to the re-sampled results. If the re-analysis shows that Pu-239/240 is non-detect or detected at a level significantly lower than the initial sampling result, then the initial sampling result was due to analytical error, not the actual presence of Pu-239/240 at the level reported. If the re-analysis confirms that Pu-239/240 is present, then the associated margin of error will be low enough to verify the presence or absence of Pu-239/240 in the sample.

The action level for Pu-239/240 in soil is on the order of 1 to 2 pCi/g (NUREG 1500). Although the detection limits requested during the initial sampling event were ten times below this number, NYSDEC and USEPA have requested that the presence of Pu-239/240 be verified by using analytical methods that look more closely at background levels. Specifically, NYS Department of Health has requested a detection limit of 0.01 pCi/g. The Army has agreed to re-sample and re-analyze samples for Pu-239/240 at the requested detection limit. However, the data collected is not intended for use in conducting a background study for Pu-239/240 in soil and sediment. The re-sampling plan, as it is outlined below, does not include a statistically significant number of background samples in soil and sediment to ensure a representative background population. A significantly larger sampling effort would be required to

establish background levels at the site. We know of no published studies of background levels of Pu-239/240 in New York State. According to a recent sampling effort performed for Brookhaven National Laboratories in Suffolk County, New York, background levels of Pu-239/240 were established for sediments at an average activity of 0.03 pCi/g. However, no background levels were established for soil at Brookhaven (personal communication, Andrew Rapiejko, Suffolk County Health Department and Jacqueline Travers, Parsons ES, 11/9/99). In order to establish a representative background dataset at SEAD-12 using the lower detection limits, approximately 40 to 50 additional samples would need to be collected.

Since the objective of the re-sampling event is to provide support that the Pu-239/240 results from the initial round of sampling were not actually detected at the levels reported, establishment of background levels during the re-sampling event is not necessary. The Army has affirmed that the background area established in the North End of the Depot was not impacted by activities at the site. Data from the initial round of sampling will be compared to the re-analysis results. Where Pu-239/240 was detected in background soil or sediment samples, a sample will be re-collected from this location and submitted for re-analysis at the lower detection limit to show that the original reported activity was due to analytical error.

3.0 Proposed Re-Sampling Plan

Only those media where a detection of Pu-239/240 was reported after data validation will be subject to re-sampling. Soil and sediment were the only two media where Pu-239/240 was detected.

3.1 Soil Re-Sampling

A total of 61 surface soil locations will be re-sampled for Pu-239/240. The surface soil locations are highlighted in Figures 3.1. 3.2 and 3.3 and listed in Table 3. These locations correlate with locations where Pu-239/240 was detected in the surface soil, except for four locations. Four samples corresponding with sample locations SS12-2, SS12-9, SS12-12, and SS12-14 in the North End will also be re-sampled. Although Pu-239/240 was not detected in the original samples from these locations, NYSDOH has requested that some samples from the area in North End be re-collected and re-analyzed to verify the results from the previous sampling event (non-detect). To off-set these four additional locations, four locations where Pu-239/240 was detected will not be re-collected and re-analyzed. These are SS12-044, SS12-054, SS12-059, and SS12-235.

Surface soil samples will be collected in accordance with Section 3.4.4 of Appendix A of the Generic Installation RI/FS Work Plan with one exception. Rather than collecting a sample from 0-2 inches, a core sample will be collected from 0-6 inches. The upper two inches of this sample (less organic matter) will be prepared as discussed in Section 3.4.4 of the RI/FS Work Plan and sent to the laboratory for analysis. The remainder of the sample (interval from 2 to 6 inches) will be prepared and collected as the sample from 0-2 inches. However, this sample will be logged on a separate chain of custody and archived. If the data from the 0 to 2-inch interval indicates that Pu-239/240 has impacted the soil, it may be necessary to analyze samples collected from the deeper interval to determine the extent of impacted soil.

3.2 Sediment Re-Sampling

A total of 26 sediment locations will be re-sampled for Pu-239/240. The sediment locations are highlighted in Figures 4.1 and 4.2 and listed in Table 4. These locations correlate with locations where Pu-239/240 was detected in the sediment. Two of the locations are off-site locations (SD12-60 and SD12-63).

Sediment samples will be collected in accordance with Section 3.7.3 of Appendix A of the Generic RI/FS Work Plan. No sediment samples will be collected for the purposes of archiving.

4.0 Quality Assurance Project Plan

As requested by EPA in their comments dated November 2, 1999, compliance with current EPA Quality Assurance System requirements must be demonstrated. The majority of these elements are addressed in Appendices A and C of the Generic Installation RI/FS Work Plan, Appendix F of the SEAD-12 RI/FS Scoping Plan, this letter sampling and analysis plan, and the laboratories SOP for the Isotopic Determination of Plutonium (provided in Attachment A). Due to the change in laboratories used for radiochemical analyses on this project, certain information required in the quality assurance project plan is still being gathered from the newly selected laboratory. When all information has been gathered, Attachment B, Quality Assurance Project Plan Elements, will be provided for agency review.

5.0 Sampling Schedule

Re-collection of samples for the analysis of Pu-239/240 is proposed to commence on Monday, December 13, 1999.

Parsons ES appreciates the opportunity to provide you with this letter sampling and analysis plan. Should you have any questions, please do not hesitate to call me at (781) 401-2535. We look forward to discussing this plan with you on a conference call to be scheduled in early December.

Sincerely,

PARSONS ENGINEERING SCIENCE, INC.

Jacqueline Travers

Jacqueline Travers, P.E.
Task Order Manager

cc: Mr. Tom Enroth, USACOE
Mr. Stephen Absolom, SEDA
Ms. Dorothy Richards, CEHNC-PM-ND
Mr. Keith Hoddinott, USACHPPM (Prov.)
Mr. John Buck, USAEC

TABLES

Table 1
Pu-239/240 Results for Soil Samples Where Detected
SEAD-12, Seneca Army Depot Activity

Sample Location	Top Depth (ft)	Bottom Depth (ft)	Laboratory Result	Qualifier	Result Error +/-	Background?
MW12-1	0	0.2	0.2 pCi/g	J	0.3	yes
MW12-3	0	0.2	0.2 pCi/g	J	0.2	yes
SB12-3	0	0.2	0.2 pCi/g	J	0.2	
SB12-4	0	0.2	0.2 pCi/g	J	0.2	
SS12-68	0	0.2	0.1 pCi/g	J	0.1	
SS12-21	0	0.2	0.1 pCi/g	J	0.1	
SS12-24	0	0.2	0.1 pCi/g	J	0.1	
SS12-26	0	0.2	0.1 pCi/g	J	0.1	
SS12-34	0	0.2	0.1 pCi/g	J	0.1	
SS12-36	0	0.2	0.2 pCi/g	J	0.1	
SS12-39	0	0.2	0.1 pCi/g	J	0.1	
SS12-42	0	0.2	0.1 pCi/g	J	0.1	
SS12-44	0	0.2	0.1 pCi/g	J	0.1	
SS12-46	0	0.2	0.2 pCi/g	J	0.1	
SS12-48	0	0.2	0.2 pCi/g	J	0.1	
SS12-49	0	0.2	0.1 pCi/g	J	0.1	
SS12-87	0	0.2	0.1 pCi/g	J	0.1	
SS12-90	0	0.2	0.2 pCi/g	J	0.2	
SS12-99	0	0.2	0.2 pCi/g	J	0.2	
SS12-136	0	0.2	0.1 pCi/g	J	0.1	
SS12-51	0	0.2	0.1 pCi/g		0.1	
SS12-52	0	0.2	0.1 pCi/g		0.1	
SS12-53	0	0.2	0.1 pCi/g		0.1	
SS12-54	0	0.2	0.1 pCi/g		0.1	
SS12-58	0	0.2	0.1 pCi/g		0.1	
SS12-59	0	0.2	0.1 pCi/g		0.1	
SS12-94	0	0.2	0.1 pCi/g		0.2	
SS12-103	0	0.2	0.2 pCi/g		0.2	
SS12-112	0	0.2	0.1 pCi/g		0.1	
SS12-127	0	0.2	0.1 pCi/g		0.1	
SS12-129	0	0.2	0.1 pCi/g		0.1	
SS12-134	0	0.2	0.1 pCi/g		0.1	
SS12-135	0	0.2	0.1 pCi/g		0.1	
SS12-137	0	0.2	0.1 pCi/g		0.1	
SS12-139	0	0.2	1 pCi/g		0.6	
SS12-140	0	0.2	0.1 pCi/g		0.1	
SS12-141	0	0.2	0.2 pCi/g		0.1	
SS12-145	0	0.2	0.1 pCi/g		0.1	
SS12-148	0	0.2	0.1 pCi/g		0.1	
SS12-155	0	0.2	0.1 pCi/g		0.1	
SS12-158	0	0.2	0.1 pCi/g		0.1	
SS12-170	0	0.2	0.1 pCi/g		0.1	
SS12-172	0	0.2	0.1 pCi/g		0.1	
SS12-175	0	0.2	0.1 pCi/g		0.1	
SS12-176	0	0.2	0.1 pCi/g		0.1	
SS12-179	0	0.2	0.1 pCi/g		0.1	

Table 1
Pu-239/240 Results for Soil Samples Where Detected
SEAD-12, Seneca Army Depot Activity

Sample Location	Top Depth (ft)	Bottom Depth (ft)	Laboratory Result	Qualifier	Result Error +/-	Background?
SS12-183	0	0.2	0.1 pCi/g		0.1	
SS12-187	0	0.2	0.1 pCi/g		0.1	
SS12-188	0	0.2	0.1 pCi/g		0.1	
SS12-197	0	0.2	0.1 pCi/g		0.1	
SS12-199	0	0.2	0.1 pCi/g		0.1	
SS12-207	0	0.2	0.2 pCi/g		0.1	
SS12-218	0	0.2	0.2 pCi/g		0.1	
SS12-228	0	0.2	0.1 pCi/g		0.1	
SS12-233	0	0.2	0.2 pCi/g		0.1	
SS12-201	0	0.2	0.1 pCi/g		0.1	
SS12-167	0	0.2	0.1 pCi/g		0.1	
SS12-235	0	0.2	0.1 pCi/g		0.1	
SS12-236	0	0.2	0.2 pCi/g		0.1	
MW12-4	0	0.2	0.2 pCi/g		0.1	yes
MW12-1	0.2	2	0.2 pCi/g		0.2	yes
TP12-5A	0.5	0.5	0.1 pCi/g		0.1	
TP12-5A	0.5	0.5	0.1 pCi/g		0.1	
SB12-3	1	4	0.1 pCi/g	J	0.1	
TP12-7BA	1	1	0.1 pCi/g		0.1	
TP12-8A	1	1	0.1 pCi/g		0.1	
TP12-8B	3	3	0.1 pCi/g		0.1	
MW12-4	4	5.4	0.1 pCi/g		0.1	yes
MW12-1	4	6	0.1 pCi/g		0.1	yes
MW12-4	6	8	0.1 pCi/g	J	0.1	yes
MW12-3	6	8	0.2 pCi/g		0.2	yes
TP12-5C	8	8	0.1 pCi/g	J	0.1	
SB12-2	8	10	0.2 pCi/g		0.1	

Table 2
Pu-239/240 Results for Sediment Samples Where Detected
SEAD-12, Seneca Army Depot Activity

Sample Location	Laboratory Result	Qualifier	Result Error +/-	Background?
SD12-26	0.1 pCi/g		0.2	
SD12-25	0.1 pCi/g		0.2	
SD12-41	0.1 pCi/g		0.2	
SD12-40	0.1 pCi/g		0.2	
SD12-24	0.1 pCi/g		0.1	
SD12-27	0.1 pCi/g		0.1	
SD12-10	0.1 pCi/g		0.1	
SD12-19	0.1 pCi/g		0.1	
SD12-46	0.2 pCi/g		0.2	
SD12-42	0.1 pCi/g		0.1	
SD12-63	0.1 pCi/g		0.1	yes
SD12-18	0.1 pCi/g		0.1	
SD12-20	0.1 pCi/g		0.1	
SD12-32	0.2 pCi/g	J	0.2	
SD12-6	0.2 pCi/g	J	0.2	
SD12-22	0.1 pCi/g	J	0.1	
SD12-36	0.2 pCi/g	J	0.2	
SD12-36	0.1 pCi/g	J	0.1	
SD12-14	0.2 pCi/g	J	0.2	
SD12-5	0.1 pCi/g	J	0.1	
SD12-9	0.1 pCi/g	J	0.1	
SD12-43	0.1 pCi/g	J	0.1	
SD12-60	0.1 pCi/g	J	0.1	yes
SD12-47	0.1 pCi/g	J	0.2	
SD12-28	0.2 pCi/g	J	0.3	
SD12-7	0.1 pCi/g	J	0.1	

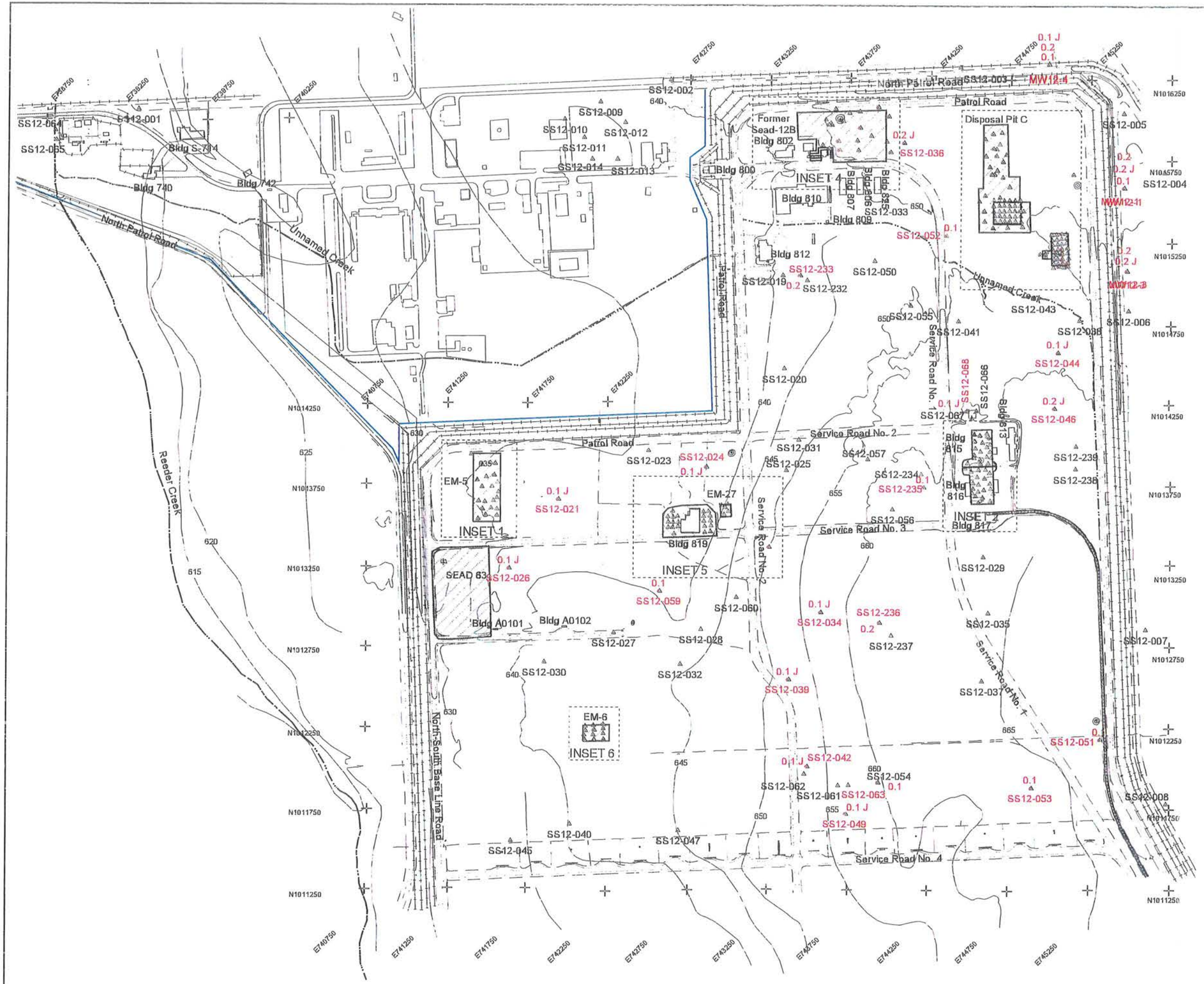
Table 3
Soil Re-sample Locations
for Pu-239/240
SEAD-12, Seneca Army Depot Activity

Sample Location	Northing	Easting	Background?
SS12-2	1,016,253.00	742,629.60	yes - North end
SS12-9	1,016,116.15	742,191.97	yes - North end
SS12-12	1,015,989.84	742,350.79	yes - North end
SS12-14	1,015,764.35	742,143.73	yes - North end
MW12-1	1,015,591.70	745,456.80	yes
MW12-3	1,015,079.90	745,477.00	yes
MW12-4	1,016,353.40	744,983.60	yes
SB12-3	1,015,184.30	744,969.00	
SB12-4	1,015,183.20	744,938.56	
SS12-103	1,013,809.85	741,548.69	
SS12-112	1,013,600.08	741,475.68	
SS12-127	1,016,086.99	743,922.58	
SS12-129	1,015,959.29	743,808.27	
SS12-134	1,016,029.52	743,988.33	
SS12-135	1,015,809.38	744,000.49	
SS12-136	1,016,058.66	743,804.09	
SS12-137	1,016,001.24	743,699.70	
SS12-139	1,015,831.61	743,612.45	
SS12-140	1,015,821.49	743,679.11	
SS12-141	1,015,878.38	743,670.31	
SS12-145	1,015,978.22	743,610.82	
SS12-148	1,015,448.48	744,589.37	
SS12-155	1,015,925.36	744,639.73	
SS12-158	1,015,753.82	744,703.72	
SS12-167	1,015,199.14	744,973.74	
SS12-170	1,015,108.14	745,052.63	
SS12-172	1,015,110.04	745,106.06	
SS12-175	1,015,126.72	745,077.18	
SS12-176	1,015,131.64	745,106.00	
SS12-179	1,015,156.01	745,074.56	
SS12-183	1,015,186.60	745,072.58	
SS12-187	1,015,216.37	745,070.01	
SS12-188	1,015,221.77	745,103.53	
SS12-197	1,015,303.13	745,007.48	
SS12-199	1,015,308.85	745,067.66	
SS12-201	1,015,516.88	744,811.52	
SS12-207	1,015,479.49	744,646.80	
SS12-21	1,013,661.59	741,950.70	
SS12-218	1,015,451.31	744,811.62	
SS12-228	1,015,386.65	744,748.76	
SS12-233	1,015,047.02	743,442.05	
SS12-236	1,012,900.73	743,948.82	
SS12-24	1,013,863.73	742,869.33	
SS12-26	1,013,234.90	741,644.56	
SS12-34	1,012,966.03	743,580.48	
SS12-36	1,015,868.37	744,087.07	
SS12-39	1,012,549.94	743,383.63	
SS12-42	1,012,010.55	743,500.98	
SS12-46	1,014,229.59	745,033.13	
SS12-48	1,015,672.94	745,138.47	
SS12-49	1,011,719.90	743,742.24	
SS12-51	1,012,181.53	745,325.13	
SS12-52	1,015,296.11	744,355.07	
SS12-53	1,011,882.17	744,901.58	
SS12-58	1,013,370.13	743,261.37	
SS12-68	1,014,212.92	744,488.58	
SS12-87	1,013,487.18	742,658.92	
SS12-90	1,012,270.30	742,231.79	
SS12-94	1,012,234.34	742,177.90	
SS12-99	1,012,174.08	742,118.14	

Table 4
Sediment Re-sample Locations
for Pu-239/240
SEAD-12, Seneca Army Depot Activity

Sample Location	Northing	Easting	Background?
SD12-10	1,014,003.70	742,626.90	
SD12-14	1,014,509.60	745,272.40	
SD12-18	1,015,439.70	745,226.00	
SD12-19	1,016,123.30	745,093.50	
SD12-20	1,015,726.70	743,091.40	
SD12-22	1,013,548.00	742,654.80	
SD12-24	1,013,737.30	742,692.10	
SD12-25	1,013,650.60	742,853.00	
SD12-26	1,013,393.70	742,449.70	
SD12-27	1,013,401.70	742,718.20	
SD12-28	1,013,557.30	744,759.10	
SD12-32	1,014,152.40	744,608.40	
SD12-36	1,014,520.90	744,366.90	
SD12-36	1,014,520.90	744,366.90	
SD12-40	1,015,354.20	743,300.40	
SD12-41	1,015,171.90	743,087.40	
SD12-42	1,015,833.10	743,614.30	
SD12-43	1,015,757.40	743,685.40	
SD12-46	1,015,901.30	743,518.90	
SD12-47	1,015,895.70	743,389.60	
SD12-5	1,013,162.80	743,108.80	
SD12-6	1,013,516.70	744,048.90	
SD12-60	1,007,774.30	742,682.80	yes
SD12-63	1,005,327.90	742,792.20	yes
SD12-7	1,012,186.10	744,951.60	
SD12-9	1,014,025.70	742,199.40	

FIGURES



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PARSONS ENGINEERING SCIENCE, INC.

SENECA ARMY DEPOT ACTIVITY

RI / FS

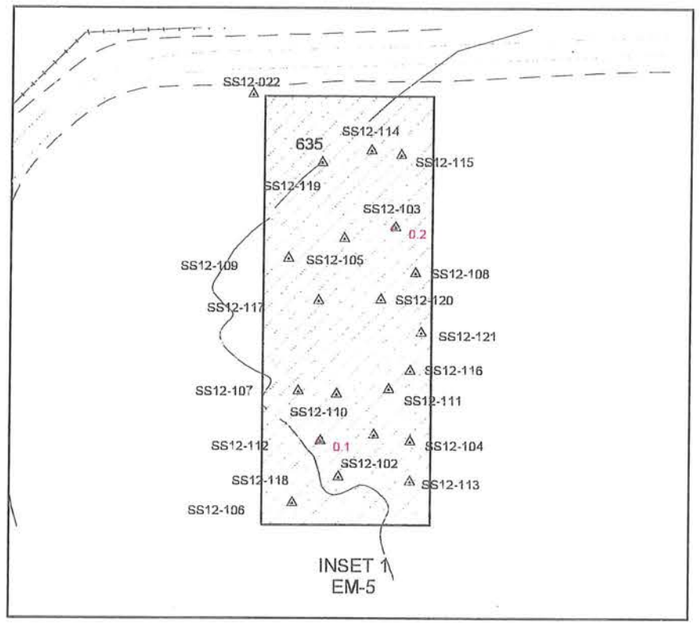
SEAD-12

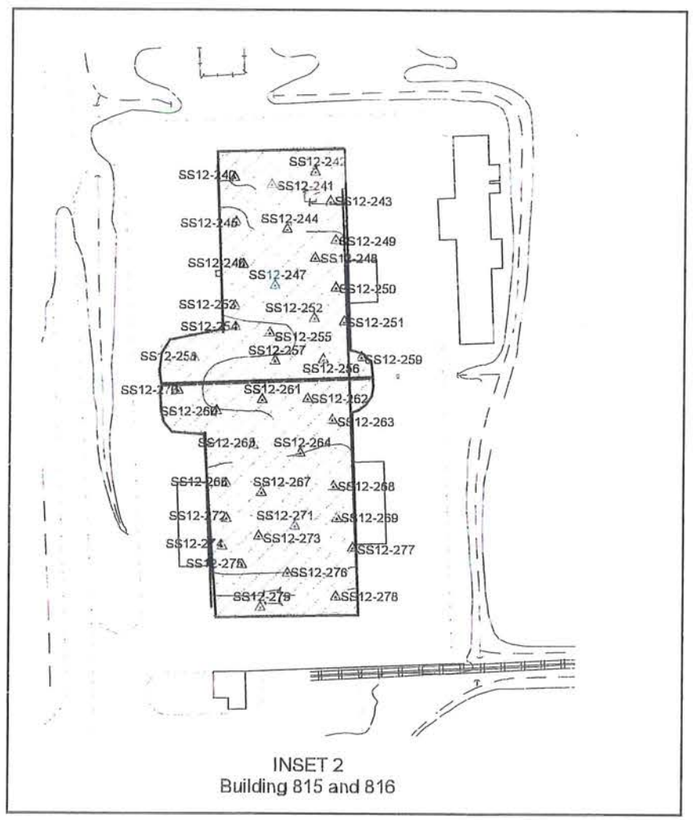
FIGURE 1.1

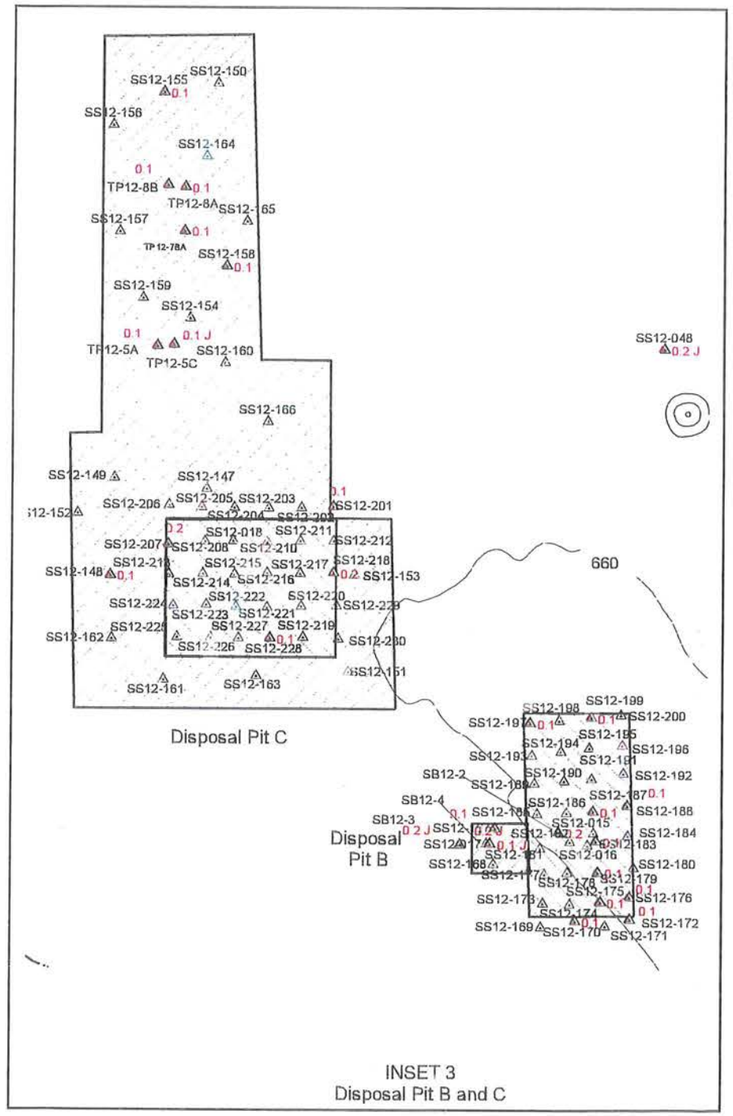
PU-239/240 DETECTIONS IN SOIL SAMPLES

NOV, 1999 | SHEET 1 OF 1

O:\AV_GIS30\SENECA\SEAD12\SAMPLELO\SS_LOCS







N

SS12-15
SOIL SAMPLE LOCATION
WITH LABEL

.1 J
SS12-15
LOCATION WITH PU239/240
DETECTION AND DETECTED
VALUE (pCi/g)

RADIATION
CLASSIFICATION AREAS

- CLASS 1
- CLASS 2
- CLASS 3

100 0 100 200 Feet

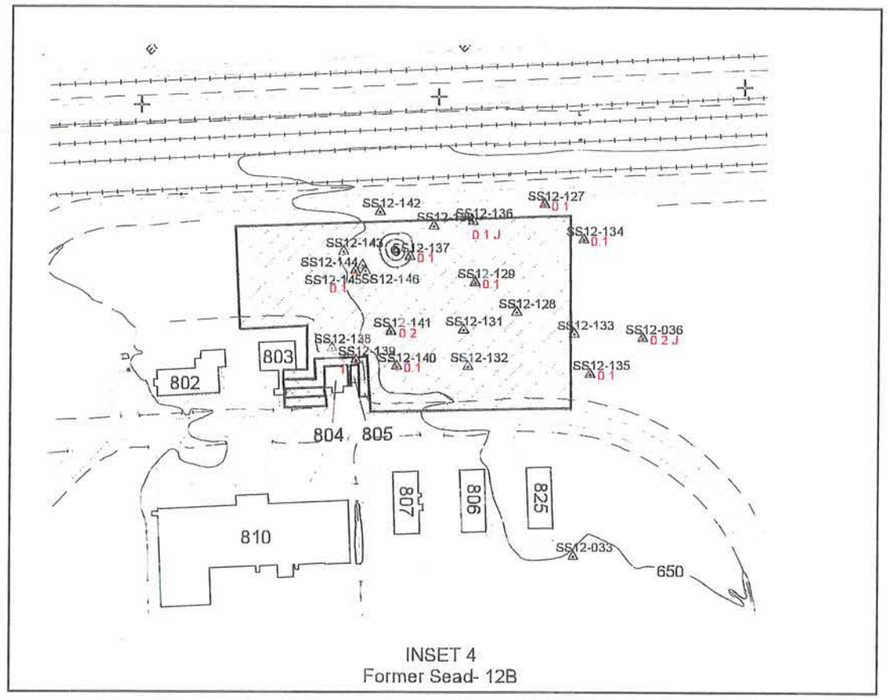
PARSONS
PARSONS ENGINEERING SCIENCE, INC.

SENECA ARMY DEPOT ACTIVITY
RI / FS
SEAD-12

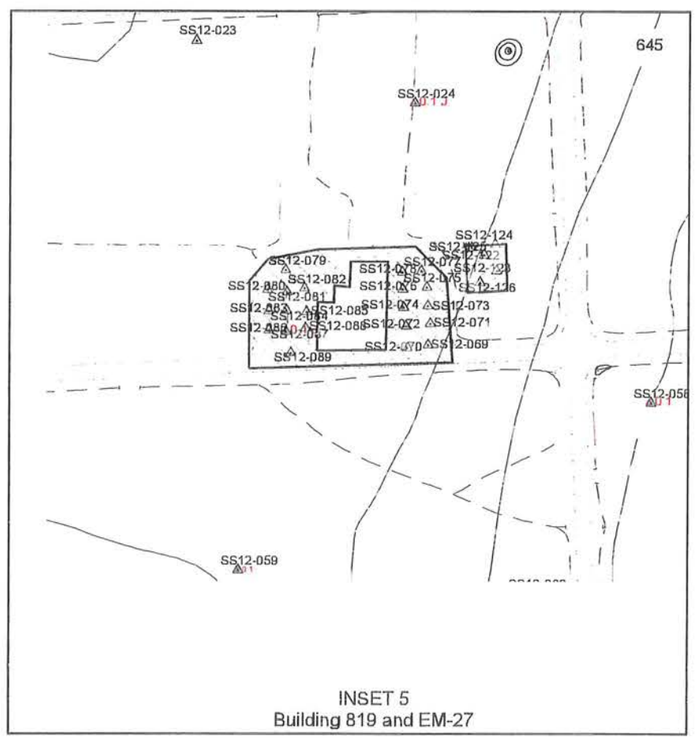
FIGURE 1.2
PU239/240 DETECTIONS IN
SOIL SAMPLES

NOV, 1999 | SHEET 1 OF 1

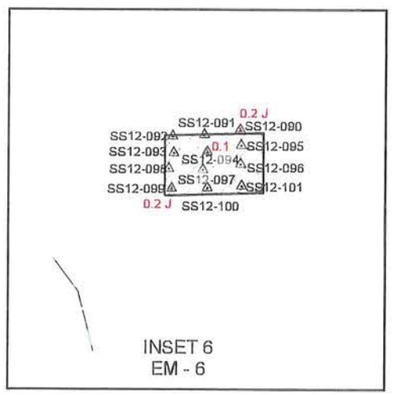
O:\AV_GIS30\SENECA\SEAD12\SAMPLELO\SS_LOCS



INSET 4
Former Sead- 12B



INSET 5
Building 819 and EM-27



INSET 6
EM - 6

N

△ SS12-15
SOIL SAMPLE LOCATION
WITH LABEL

.1 J SS12-15
△ LOCATION WITH PU-239/240
DETECTION AND DETECTED
VALUE (pCi/g)

RADIATION
CLASSIFICATION AREAS

- CLASS 1
- CLASS 2
- CLASS 3

150 0 150 300 Feet

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SENECA ARMY DEPOT ACTIVITY
RI / FS
SEAD-12

FIGURE 1 3
PU-239/240 DETECTIONS IN
SOIL SAMPLES

NOV. 1999 | SHEET 1 OF 1

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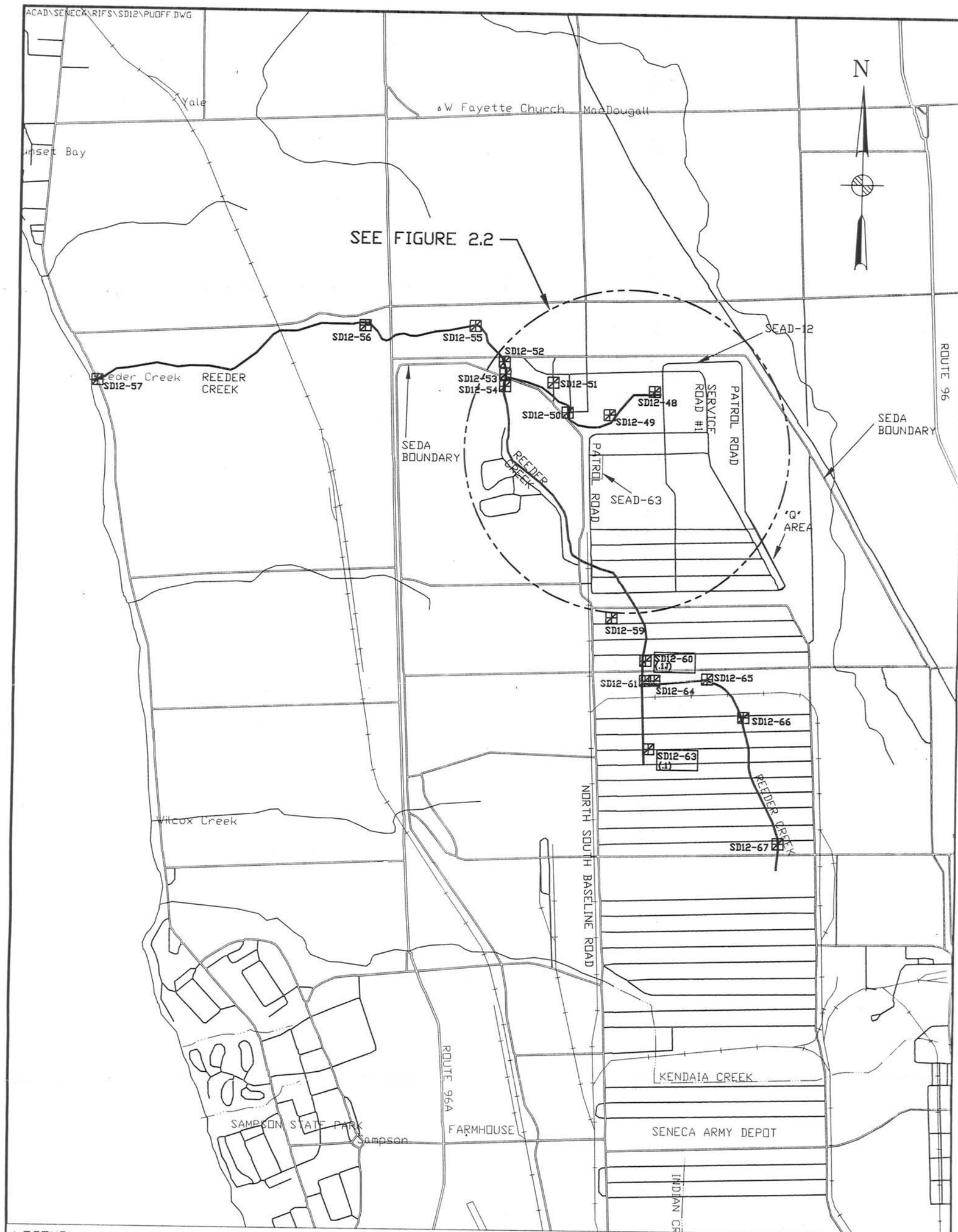

LEGEND

CREEK OR TRIBUTARY

SD12-65 — SEDIMENT LOCATION

SD12-60 (.17) — LOCATION WHERE Pu-239/240 WAS DETECTED (pCi/g)

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DEPT. ENVIRONMENTAL ENGINEERING

Dwg. No.

FIGURE 2.1

OFF-SITE SEDIMENT LOCATIONS

(PU-239/240 DETECTIONS)

SCALE NA

DATE DECEMBER 1999

REV NA

APPROXIMATE BOUNDARY OF NORTH AREA

SD12-11
SEDIMENT SAMPLE
LOCATION WITH LABEL

.1 J
SD12-11
LOCATION WITH PM-147
DETECTION AND DETECTED
VALUE (pCi/g)

N

300 0 300 600 900 Feet

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RI / FS
SEAD-12

FIGURE 2.2
PU-239/240 DETECTIONS IN
SEDIMENT SAMPLES

NOV, 1999 | SHEET 1 OF 1

O:\AV_GIS30\SENECA\SEAD12\SAMPLELO\LOCATION

N

— APPROXIMATE BOUNDARY OF NORTH END

△ SS12-15
SURFACE SOIL SAMPLE LOCATION WITH LABEL

△ SS12-023
SURFACE SOIL SAMPLE TO BE RECOLLECTED

RADIATION CLASSIFICATION AREAS

- CLASS 1
- CLASS 2
- CLASS 3

NOTE: SEE FIGURE 1.5 FOR DETAILED SURFACE SOIL SAMPLE LOCATIONS

600 0 600 Feet

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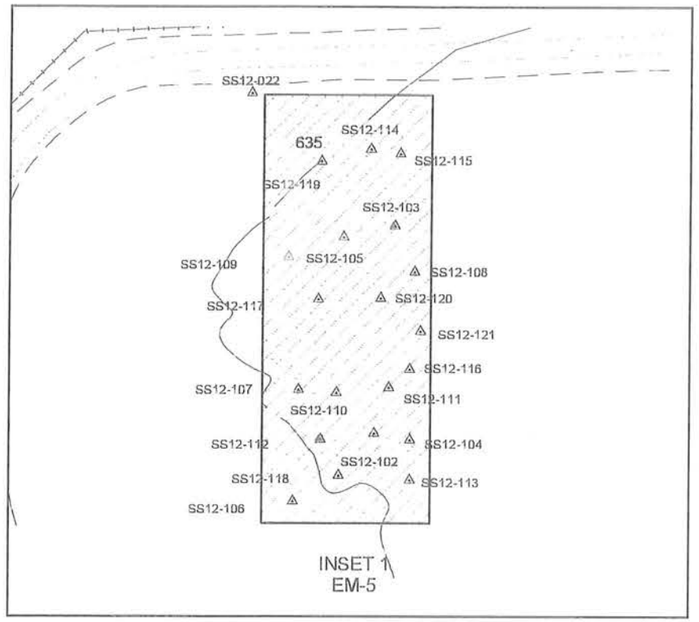
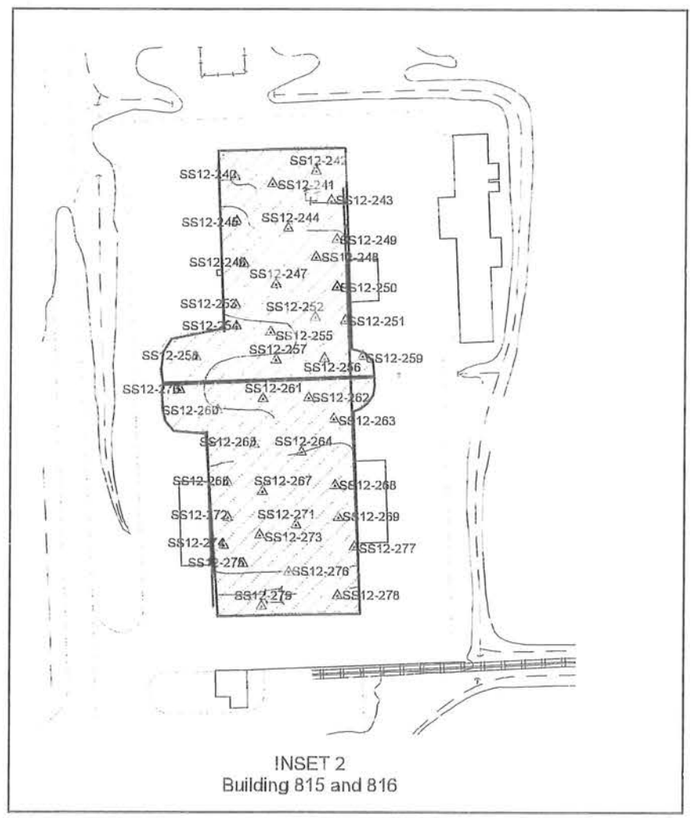
RI/FS
SEAD-12

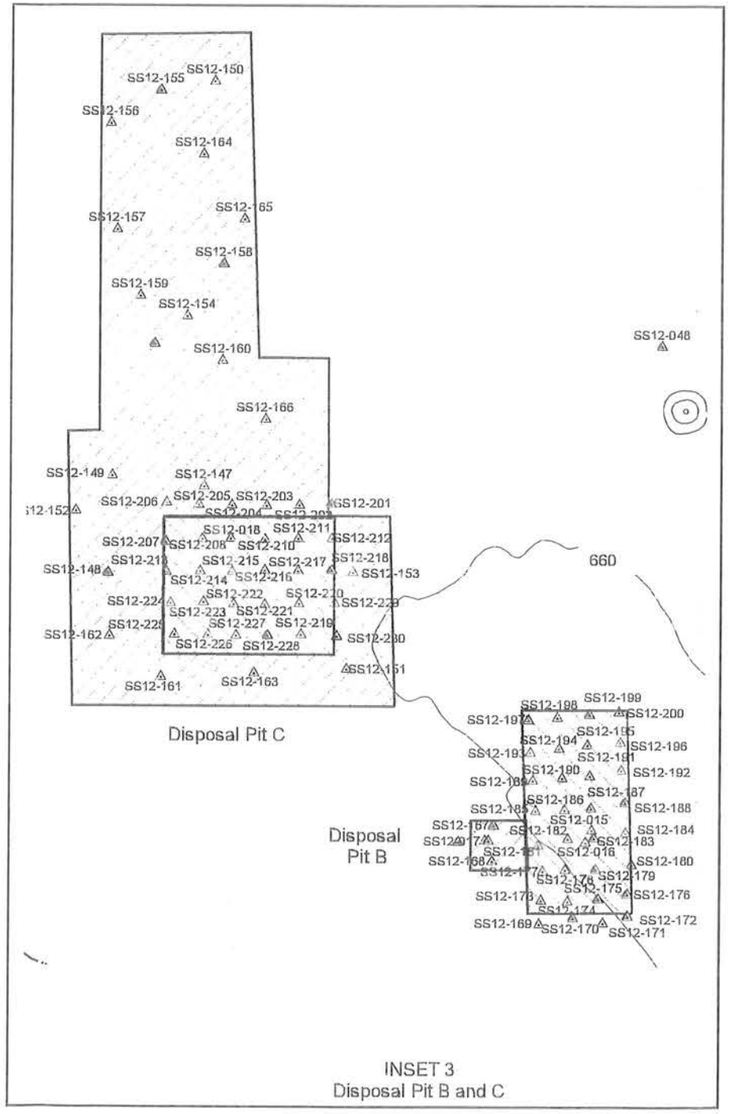
FIGURE 3.1
LOCATION OF SURFACE SOIL SAMPLES TO BE RECOLLECTED

NOV, 1999 | SHEET 1 OF 1

O:\AV_GIS30\SENECA\SEAD12\SAMPLELO\SS_LOCS







N

▲ SS12-15
SURFACE SOIL SAMPLE
LOCATION WITH LABEL

▲ SS12-15
SURFACE SOIL SAMPLE TO
BE RECOLLECTED

RADIATION
CLASSIFICATION AREAS

- CLASS 1
- CLASS 2
- CLASS 3

150 0 150 Feet

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RI / FS
SEAD-12

FIGURE 3.2
LOCATION OF SURFACE SOIL
SAMPLES TO BE RECOLLECTED

NOV. 1999 | SHEET 1 OF 1

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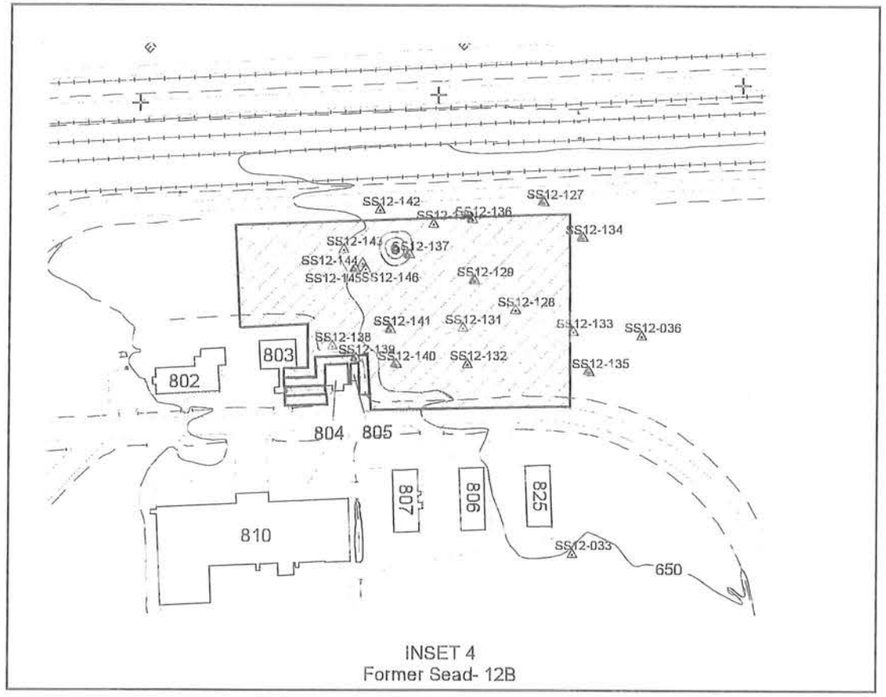
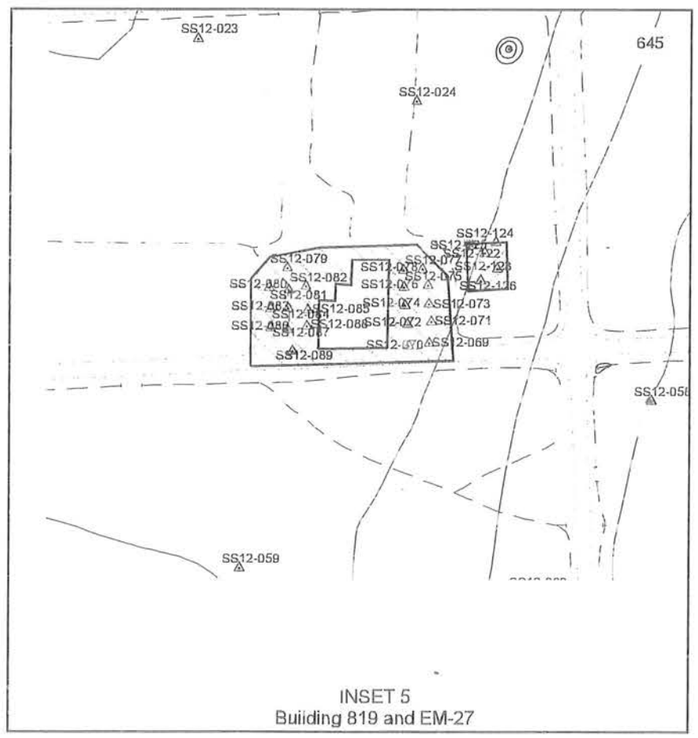
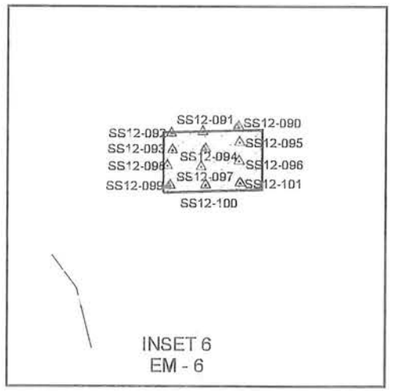


INSET 4
Former Sead- 12B



INSET 5
Buiiding 819 and EM-27



INSET 6
EM - 6

N

▲ SS12-15
SOIL SAMPLE LOCATION
WITH LABEL

.1 J
▲ SS12-15
SURFACE SOIL SAMPLE
TO BE RECOLLECTED

RADIATION
CLASSIFICATION AREAS

- CLASS 1
- CLASS 2
- CLASS 3

150 0 150 300 Feet

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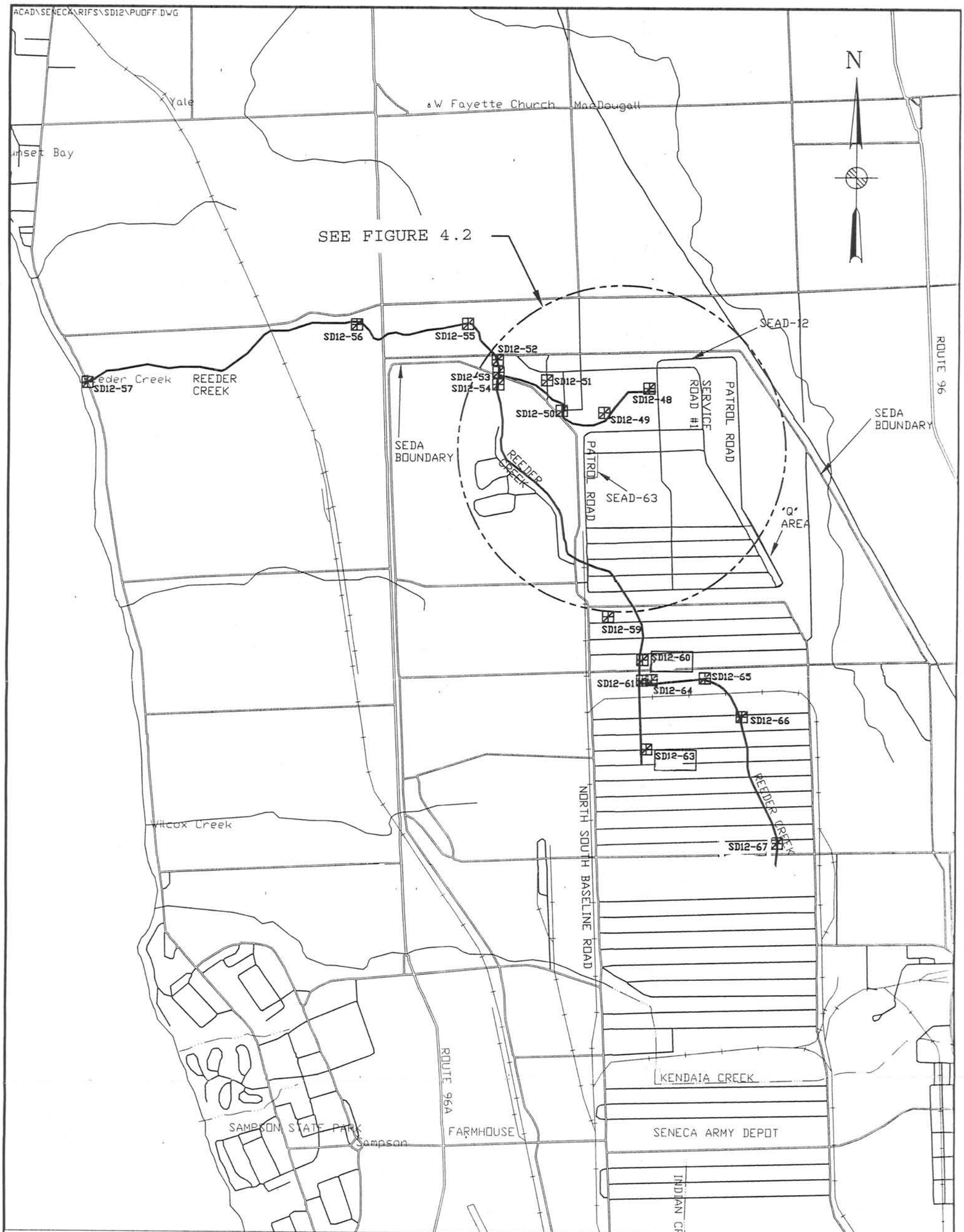
SEAD-12

FIGURE 3.3
LOCATION OF SURFACE SOIL
SAMPLES TO BE RECOLLECTED

NOV. 1999 | SHEET 1 OF 1

C:\AV_GIS30\SENECA\SEAD12\SAMPLELO\SS_LOCS



LEGEND

CREEK OR TRIBUTARY

SD12-65 SEDIMENT LOCATION

SD12-60 RE-SAMPLE LOCATION

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CLIENT/PROJECT TITLE

SENECA ARMY DEPOT

DEPT. ENVIRONMENTAL ENGINEERING

Dwg No

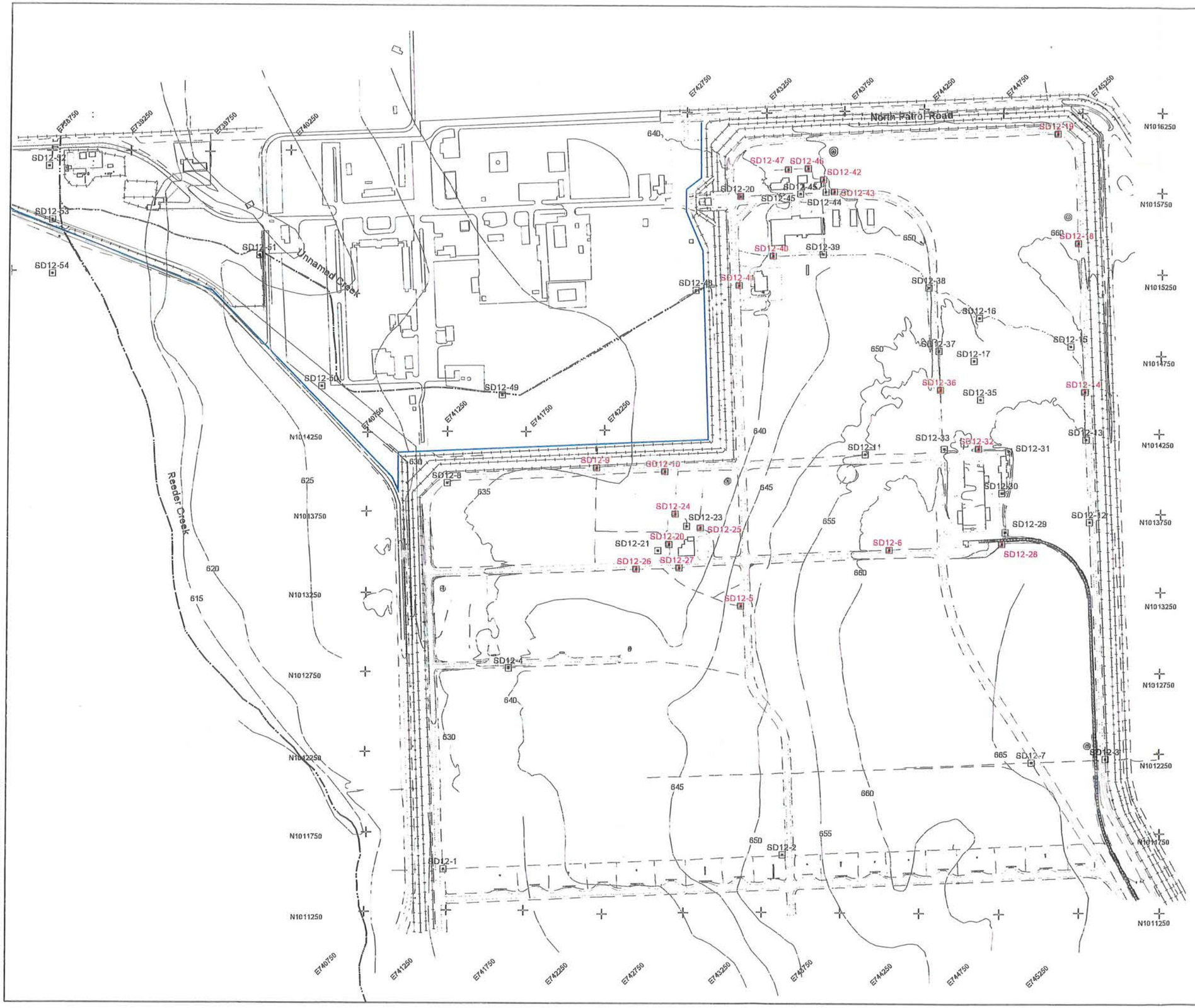
FIGURE 4.1

OFF-SITE SEDIMENT LOCATIONS

SCALE NA

DATE DECEMBER 1999

REV NA



PARSONS

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SENECA ARMY DEPOT ACTIVITY

RI / FS

SEAD-12

FIGURE 4.2

LOCATION OF SEDIMENT SAMPLES TO BE RECOLLECTED

NOV, 1999 | SHEET 1 OF 1

O:\AV_GIS30\SENECA\SEAD12\SAMPLELO\LOCATION

ATTACHMENT A
LABORATORY STANDARD OPERATING PROCEDURE
FOR THE ISOTOPIC DETERMINATION
OF AMERICIUM, CURIUM, PLUTONIUM, AND URANIUM

UNCONTROLLED DOCUMENT

STANDARD OPERATING PROCEDURE

FOR

THE ISOTOPIC DETERMINATION

OF

AMERICIUM, CURIUM, PLUTONIUM AND URANIUM

(GL-EPI-A-011-Revision5)

HARD COPY ORIGINAL REPOSITORY:

NOTE: This Standard Operating Procedure has been prepared for the sole use of the General Engineering Laboratories and may not be specifically applicable to the activities of other organizations.

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TABLE OF CONTENTS

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1.0 Title: Standard Operating Procedure for the Isotopic Determination of Americium, Curium, Plutonium, and Uranium.

2.0 Method Objective, Purpose, Code, and Summary

2.1 This standard operating procedure provides the necessary instructions to conduct the analysis for isotopic americium, curium, plutonium and uranium in a variety of matrices. This method also gives specific guidance on determining U-232 and Am-243, which are typically used as isotopic tracers.

2.2 A sample is digested if necessary and aliquoted. Transuranic elements are scavenged by coprecipitation with iron hydroxide. The precipitate is dissolved and separation of elements is accomplished through extraction chromatography and ion exchange resins. The elements are then prepared for the measurement of radioactive isotopes by coprecipitation with cerium fluoride. The cerium fluoride precipitate is trapped on a filter, mounted on a stainless steel disk and placed in a partially evacuated chamber for measurement of isotopic alpha emission.

2.3 General Engineering Laboratories (GEL) utilizes methods that are derived from established sources. This method is based on the source method from DOE EML Methods Manual HASL 300 E-U-04 and uses similar principles of radiochemical separation and counting. This method is also very similar in concept to the source method from the DOE Methods Manual for Evaluating Environmental and Waste Management Samples, 1997 Edition, RP800: "Sequential Separation of Americium and Plutonium by Extraction Chromatography."

2.4 This revision combines several related procedures. The method for the determination of isotopic Plutonium originally in SOP "The Isotopic Determination of Thorium, Plutonium and Neptunium" (GL-EPI-E-A012) has been added to this procedure. The following standards operating procedures are canceled without replacement having served their purpose:

2.4.1 SOP GL-EPI-E-A012b, "The Determination of Isotopic Neptunium and Plutonium in Soil and Vegetation."

2.4.3 SOP GL-EPI-E-A012d, "The Determination of Isotopic Thorium, Plutonium and Neptunium and Plutonium in Air Filters."

3.0 Method Applicability

3.1 Method Detection Limit: Typical minimum detectable activity (MDA) for samples analyzed for U/Am/Cm/Pu is 1 pCi/L or 0.1 pCi/g for all isotopes. For this procedure, MDAs as low as 0.01 pCi/L or 0.002 pCi/g for all isotopes can be obtained by appropriately increasing the sample size and/or count time.

3.2 Method Precision: Typical relative percent difference (RPD) is 20%.

3.3 Method Bias (Accuracy): Acceptable criteria for method accuracy, measured by running with each batch a laboratory control sample, is ±25% of true value.

3.4 Analysts are trained and certified to run this analysis after the analyst has completed a batch with acceptable duplicate and laboratory control sample, as well as completed an unknown sample within ±25% of true value. Analyst training records are kept on hand in the human resource department.

4.0 Definitions

4.1 National Institute of Standards and Technology (NIST). For the purpose of this method, the national scientific body responsible for the standardization and acceptability of analyte solutions.

4.2 Type II water: Deionized water.

4.3 LIMS: Laboratory Information Management System. The database system used to store and report data.

5.0 Method Variations

5.1 Some variations may be necessary due to special matrices encountered in the lab. These variations may be used with approval from a Group Leader or Senior Technical Specialist. Variations to a method will be documented with the analytical raw data.

6.0 Safety Precautions and Warnings

6.1 Wear eye protection with side shields while in the laboratory.

6.2 All chemicals and samples should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest level possible. GEL maintains a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals

in the laboratory as well as a reference file of Material Safety Data Sheets (MSDS). These documents are maintained in the library and in the laboratory, respectively. Individual sample MSDS forms provided by the clients are kept in Login."

6.3 Gloves are required when handling the chemicals in this procedure. The gloves approved for this procedure are nitrile gloves for concentrated acids and bases, and potassium ferricyanide in neat form. Work under a hood when using concentrated acids and bases.

6.4 The handling of radioactive samples is outlined in SOP "Handling of Radioactive Samples" (GL-EPI-E-S004). General guidelines include:

6.4.1 Prior to handling radioactive samples, analysts must have had radiation safety training and understand their full responsibilities in radioactive sample handling. Some general guidelines follow:

6.4.2 A plastic apron may be worn over the lab coat for added protection from contamination when working with radioactive samples.

6.4.3 Protect counter tops with counter paper or work from radioactive sample handling trays.

6.4.4 Prohibit admittance to immediate work area.

6.4.5 Post signs indicating radioactive samples are in the area.

6.4.6 Take swipes of the counter tops upon completion of work. Deliver those swipes to the swipe count box in the radiochemistry laboratory.

6.4.7 Segregate radioactive wastes. Radioactive waste containers are obtained from Waste Management."

6.5 Refer to SOP "Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011) for instructions on how materials are disposed.

6.6 If there is any question regarding the safety of any laboratory practice, **stop immediately**, and consult the Group Leader prior to carrying out the rest of the procedure.

6.7 When handling biological samples protect the hands and forearms by wearing gloves and a laboratory coat to avoid contact of the biological material with the skin. Protect the eyes by wearing safety glasses and if desired a splash shield.

6.8 If cutting of meats or other tissue with cutlery is required, the analyst will wear a cutting glove of mesh steel over disposable gloves to avoid cuts, which could infect the analyst with pathogens.

6.9 Any procedure, which volatilizes biological substances such as drying or ashing, must be conducted in a hood or other suitable containment device.

6.10 Decontamination of work surfaces exposed to biological samples is performed by wiping the work area with a diluted (1:10) bleach solution and water as soon as possible following analytical operations.

6.11 Exterior protective clothing shall be removed prior to exiting the bioassay sample preparation area in order to prevent the inadvertent spread of biohazards to the rest of the laboratory.

6.12 Hands will be washed with an antibacterial soap directly after handling biological samples.

7.0 Interferences

7.1 Internal tracer standards may have ingrown daughters that may interfere with the analysis. For example Th-228 will be present in aged U-232 standard, Fr-221 will be present in Th-229 which will interfere with the curium analysis, and U-232 will be present in Pu-236. These problems are overcome by running separate aliquots of sample for thorium analysis.

7.2 Short lived radioactive progeny may ingrow on prepared filters. For example, the Ra-224 alpha peak will be present if the Th-228 parent is present. These interferences are minimized by counting samples as soon as possible after separation chemistry.

8.0 Apparatus, Materials, Reagents, Equipment, and Instrumentation

8.1 Ancillary Equipment

8.1.1 Silicon surface barrier detectors with associated electronics, vacuum chambers, and data reduction capabilities

8.1.2 Ion exchange columns. Eichrom TRU-Spec™ prepackaged column with 25 mL reservoir

8.1.3 Vacuum pump and filtration apparatus (25 mm)

8.1.4 Gelman metricel 25 mm filters with 0.1 µm pore size

8.1.5 Gelman polypropylene 25 mm support filter

8.1.6 Stainless steel disks. 29 mm diameter

8.1.7 Stainless steel tweezers

8.1.8 Polypropylene centrifuge tube (50 mL)

8.1.9 Sample drying and ashing apparatus

8.1.10 Sample homogenization apparatus

8.1.11 AG1X8 anion exchange resin 100-200 mesh

8.2 Reagents, Chemicals and Standards

8.2.1 Ammonium hydroxide concentrated (14 N)

8.2.2 Ascorbic acid (0.8M). Dissolve 14.1 g of ascorbic acid in 100 mL of DI water. This solution should be prepared weekly to maintain its effectiveness in reducing iron.

8.2.3 Cerium(III)nitrate hexahydrate. Dissolve 0.155g in 100 mL DI water. (500 µg Ce/mL).

NOTE: This is normally purchased in the correct concentration from an approved vendor such as High Purity Standards.

8.2.4 Ethyl alcohol (80%). Dilute 400 mL ethanol to 500 mL with DI water.

8.2.5 Hydrochloric acid 0.1N. Dilute 8.3 mL of concentrated HCl to 1 liter with DI water.

8.2.6 Hydrofluoric acid concentrated (48%)

8.2.7 Ion exchange resin. Bio-Rad AG 1X8, chloride form, 100-200 mesh

8.2.8 Iron Carrier. 10 mg/mL

8.2.9 KSCN indicator (0.1M). Dissolve 0.972 g of potassium thiocyanate in 100 mL of DI water.

8.2.10 NIST traceable standards: U-232, Am-241, Am-243, Cm-244, U-238, Pu-242, Pu-239, Pu-238, Pu-236

8.2.11 Nitric acid concentrated 16M

8.2.12 2M Nitric acid/0.5M aluminum nitrate. Dissolve 93.8g of $Al(NO_3)_3 * 9H_2O$ in 0.5 liter of 2M HNO_3.

8.2.13 Nitric acid (2M) Dilute 12.5 mL of concentrated nitric acid to 100 mL of deionized water.

8.2.14 Titanous chloride. 20% reagent

9.0 Sample Handling and Preservation

9.1 Samples should be preserved to approximately pH 2 with nitric acid and collected in a plastic bottle.

9.2 Before beginning an analysis the analyst should check the sample pH with a pH strip. If necessary, adjust the pH with nitric acid to a pH=1-2. If the sample was pH adjusted let the sample sit overnight before continuing the batch.

9.3 If the sample has exceeded the hold time the analyst should contact the Group Leader before continuing with the batch.

9.4 Soil samples require no preservation and may be shipped in any suitable container.

10.0 Sample Preparation

Soil Sample Preparation

10.1 If not already done, homogenize the sample by performing SOP "Preparation of Soils for the Determination of Radionuclides" (GL-EPI-E-A021).

10.2 It is recommended that the samples be ashed in a muffle furnace as specified in SOP "Soil Sample Ashing for the Determination of Radionuclides" (GL-EPI-E-A021b).

10.3 For uranium analysis, take an appropriate aliquot and digest as specified in SOP "Digestion for Soils and Sand" (GL-EPI-E-A015).

10.4 A separate Am/Cm/Pu aliquot is analyzed with an aggressive acid leach as described in the following steps. Uranium should not be run by this leaching technique.

10.4.1 Place the sample in a beaker and add 10 mL 10 M HCl per gram of sample, with a minimum of 10 mL. Add the appropriate tracers as described in section 10.6.

10.4.2 Heat the samples and cover with a watch glass, allow to leach for a minimum of 2 hours. Agitate the sample periodically to enhance the leaching process.

10.4.3 Allow the sample to partially cool and transfer to a centrifuge tube. Centrifuge to separate the solid and leached portions of the sample. Decant the leachate to a clean labeled beaker and rinse the solid phase with DI water. Centrifuge the sample, combine the leachates and dilute with DI water. Proceed to section 10.7.

Aqueous Sample Preparation

10.5 Add an appropriate aliquot of sample to a labeled beaker. Add a certified dpm of the following tracers to each of the samples:

- For isotopic uranium, U-232 is normally used
- For isotopic americium and curium, Am-243 is used
- For isotopic plutonium, Pu-242 is normally used

10.5.1 If the analysis of the sample calls for quantification of U-232 or Am-243, the following steps shall be taken:

10.5.1.1 The sample will be run normally with the tracer indicated in section 10.6.

10.5.1.2 Review of the data shall be undertaken to determine if there are any peaks in the spectrum with which a ratio can be setup with the tracer isotope.

10.5.1.3 If there is a peak with which a ratio can be setup, then a second run of the sample shall completed with no tracer addition, and the ratios of peaks used to make corrections as described in the equations in section 15.4.

10.5.1.4 If no suitable peaks are available to ratio, a second run of the sample shall be made with a different tracer isotope such as U-236 or Am-241. The quantification of the isotope that was normally the tracer can then be made. If there is any quantifiable activity a correction can be made to the initial run by calculating a correction ratio for the tracer recovery of the first run from the second run results and following the equations outlined in 15.5.

NOTE: Other sample matrices, such as vegetation, air filters, tissue etc. are run as outlined in SOP "Preparation of Special Matrices for the Determination of Radionuclides" (GL-EPI-E-A026). The analyst must ensure that the appropriate tracer(s) are added to these other matrices as discussed in section 10.6.1.1-10.6.1.4.

10.6 Add 1 mL of iron carrier (10 mG/mL).

NOTE: For soil samples iron carrier may not be needed.

10.7 Bring to a slight boil and add concentrated NH_4OH until turbidity persists, or pH>9. Heat to near boiling for 10 minutes and then allow to settle and cool.

10.8 Decant excess supernate and discard. Collect the remaining precipitate by centrifugation in a 50 mL centrifuge tube and discard the supernate.

NOTE: Exercise care in this step because finely divided material which contains the actinides may also be present in addition to the large iron hydroxide flocks.

10.9 Dissolve the precipitate in 10-15 mL of 9M HCl/H_2O_2 solution.

10.10 Slurry AG 1x8 anion resin (Cl form 100-200 Mesh) in a squirt bottle with DI water. Transfer the resin to a small column to obtain a settled resin bed of 2.5 mL.

10.11 Condition the column with 10 mL of 9 M HCl.

10.12 Pass the sample solution from step 10.9 through the column and catch the effluent in a labeled, disposable 50 mL centrifuge tube for americium/curium analysis.

10.13 Rinse the column with 5 mL of 9 M HCl and catch with the Am/Cm fraction. Proceed to step 10.17 for Am/Cm analysis.

10.14 Rinse the column with an additional 10 mL of 9 M HCl and catch in a drip pan for disposal.
Elute plutonium by adding 20mL of 9M HCl/0.05M ammonium iodide solution. Proceed to step 10.25 for plutonium microprecipitation source preparation

10.15 Elute ~~plutonium~~ neptunium by adding 15 mL of 6M HCl/0.52 M HF solution, catching the ~~Pu~~ Np elute in a ~~labeled, disposable 50 mL centrifuge tube.~~ ~~Proceed to step 10.25 for plutonium microprecipitation source preparation for alpha spectroscopy.~~ drip pan for disposal.

10.16 Elute uranium from the column by adding 15 mL of 0.1 M HCl, catching the U elute in a labeled, disposable 50 mL centrifuge tube. Proceed to step 10.26 for uranium microprecipitation source preparation for alpha spectroscopy.

10.17 To the column elute from step 10.13, add 25 mL of DI water and 0.5 mL of iron carrier (10 mG/mL). Add concentrated ammonium hydroxide solution to pH > 9 to coprecipitate iron and actinides

10.18 Centrifuge the samples and decant the supernate into a proper waste container for disposal.

10.19 Rinse the precipitate with 10 mL of DI water that has been adjusted to pH 10 with concentrated ammonium hydroxide. Centrifuge and decant the supernate into a proper waste container for disposal.

10.20 Dissolve the iron precipitate in 10 mL of 2M HNO3/0.5M aluminum nitrate solution. Add 1 drop of KSCN indicator solution and swirl to mix. The solution color should become a deep red color, indicating the presence of Fe^{+3}.

10.21 Add 0.8 M ascorbic acid dropwise to the sample to reduce the iron valence to +2.

10.22 Precondition a 2 mL TRU spec column with 10 mL of 2 M HNO3, catching the rinse in a drip pan for disposal.

10.23 Pass the sample solution from step 10.21 through the column, catching the load solution in a drip pan for disposal. Rinse the column twice with 5 mL of 2 M HNO3 and catch the rinses in a drip pan for disposal.

10.24 Place a labeled, disposable 50 mL centrifuge tube under each column. Elute Am and Cm from the column using 2 mL of 9 M HCl, followed by 10 mL of 4 M HCl. Proceed to step 10.27 for americium microprecipitation source preparation for alpha spectroscopy.

10.25 To the plutonium solution from step 10.15, add 10 mL of DI water and 0.1 mL of cerium carrier solution (250 uL/mL). Add 3 drops of 25% dihydrazine dihydrochloride solution and swirl to mix. Let the solution sit for 10 minutes, then add 1.5 mL of concentrated hydrofluoric acid. Swirl to mix. Allow the solution to sit for 30 minutes, then proceed to step 10.28 for source preparation..

10.26 To the uranium solution from step 10.16, add 1 mL of titanium trichloride solution and allow the sample to sit for 30 seconds. Add 0.1 mL of cerium carrier solution (500 uG/mL) and swirl to mix. Add 1.5 mL of concentrated hydrofluoric acid to precipitate fluorides. Allow the solution to sit for 30 minutes, then proceed to step 10.28 for source preparation.

10.27 To the americium/curium solution from step 10.24, add 0.1 mL of cerium carrier solution (500 uG/mL) and swirl to mix. Add 1.5 mL of concentrated hydrofluoric acid and swirl to precipitate fluorides. Allow the solution to sit for 30 minutes, then proceed to step 10.28 for source preparation.

10.28 Place a 0.1 µm metricel filter on the filter funnel base.

10.29 Rinse the filter and funnel under vacuum with 5 mL of 80% ethanol. With minimum delay, add the sample to the filtering apparatus and rinse the beaker several times into the funnel with type II water. Complete the filtering by adding 5 mL of 80% ethanol.

10.30 Dry the filter under a heat lamp in a labeled petri dish. Label a 29 mm flat planchet with the applicable laboratory number and desired radionuclide. Care should be taken to center the filter and make it as flat as possible on the planchet.

NOTE: Care should be taken not to touch the active area of the filter with forceps.

10.31 Count under vacuum on the alpha spectrometer long enough to reach requested MDA. Consult the operating manual for instruction on operating the alpha spectrometer.

11.0 Preparation of Standard Solutions and Quality Control Standards

11.1 Refer to SOP "Preparation of Radioactive Standards" (GL-EPI-E-M001).

11.2 All standard solutions are NIST traceable. Certificates are given to the Quality Group Leader who logs the appropriate information. Source preparation data and certificate inventory is described in SOP "Preparation of Radioactive Standards" (GL-EPI-E-M001), Section 19.0.

11.3 Primary standards are kept in the laboratory in a secured cabinet. Secondary, working, standards are kept at the bench area in an enclosed plastic cabinet.

12.0 Instrument Calibration and Performance

12.1 For direction on calibration and instrument performance see SOP "Micro-VAX 4000 Alpha Spectroscopy System" (GL-EPI-E-I009).

13.0 Analysis and Instrument Operation

13.1 For analysis and instrument operation see SOP "Micro-VAX 4000 Alpha Spectroscopy System" (GL-EPI-E-I009).

14.0 Equipment and Instrument Maintenance

14.1 For maintenance of system see "Counting Room Maintenance and Performance Checks" (GL-EPI-E-I010).

15.0 Data Recording, Calculation, and Reduction Methods

15.1 The instrument will report sample pCi/unit according to the following equation:

$$\text{pCi / unit} = \frac{S_{cpm} - B_{cpm}}{2.22 * E * V * A * \text{decay} * R}$$

15.2 Counting uncertainty is propagated according to the following equation:

$$\text{pCi / unit} = Ac * 1.96\sqrt{\left(\frac{ef\_er}{E}\right)^2 + \left(\frac{pk\_er}{pk}\right)^2 + \left(\frac{ab\_er}{A}\right)^2 + \left(\frac{sy}{100}\right)^2 + (dk)^2}$$

15.3 The minimum detectable activity (MDA) is calculated according to the following equation:

$$MDA(pCi / unit) = \frac{2.71 + 4.65 * \sqrt{B_{cpm} * T_c}}{(2.22 * E * V * R * A * decay * T_c)}$$

where:

$$decay = \frac{1}{e^{\left(\frac{-\ln(2)T_d}{T_{1/2}}\right)}}$$

$$R = \frac{T_{cpm} - B_{cpm}}{T_{dpm} * E}$$

$$dk = \frac{T_{1/2}\,err}{T_{1/2}} * \left(\frac{\lambda Tr}{1 - e^{-\lambda Tr}} - \lambda\left(T_c + T_r\right) - 1\right)$$

And where:

Scpm	=	Sample counts per minute
Bcpm	=	Background counts per minute
E	=	Counting efficiency (decimal form)
V	=	Volume in liters,g,cfm, etc.
A	=	Isotopic abundance (decimal form)
ef_er	=	1 sigma efficiency error (decimal form)
pk_er	=	1 sigma peak error
ab_er	=	1 sigma isotopic abundance error (decimal form)

sy	=	1 sigma systematic error
pk	=	peak area
Tc	=	Sample count time in minutes
Td	=	Time interval for radioactive decay
Tr	=	Elapsed real time in minutes
Tcpm	=	Tracer counts per minute
Tdpm	=	Tracer known disintegrations per minute
Ac	=	Sample calculated activity
T1/2	=	Isotopic half life
T1/2err	=	Isotopic half life error
•	=	Isotopic decay constant
e	=	exponential function
ln	=	natural log function

15.4 This section describes the calculation of U-232 by a ratio method. This process uses two analyses of the same sample, one run traced with U-232 and one run untraced. A limitation of this method is that sufficient activity of a non tracer isotope (such as U-238) must be present to ratio the two peaks. Because of this limitation, the preferred method is to use a two tracer approach as described in 15.5.

15.4.1 Ratio determination: To set up a ratio between the peaks of the untraced sample run, use the following equation.

$$\text{Ratio} = \frac{U_{232u}}{U_{238u}}$$

15.4.2 The corrected yield of the U-232 traced sample is calculated as follows:

$$U_{232s} = U_{238i} * \text{Ratio}$$

$$U_{232t} = U_{232i} - U_{232s}$$

$$Yield_{corrected} = \frac{U_{232t}}{E * T_{dpm} * T_c}$$

where:

U232s = U-232 counts in the traced sample run due to sample activity.

U238i = total U-238 counts in the traced sample run

U232i = total U-232 counts in the traced sample run

U232t = U-232 counts in the traced sample run due to the tracer addition

U232u = U-232 counts in the untraced sample run

U238u = U-238 counts in the untraced sample run

E = Efficiency for the detector used in analysis

Tdpm = Known dpm of the U-232 tracer added

Tc = Time interval of the sample count in minutes

15.4.3 The final results are then corrected by substituting the corrected yield into the equations listed in sections 15.1 through 15.3.

15.5 This section describes the calculation of U-232 by an alternate tracer method. This process uses two analyses of the same sample, one run traced with U-232 and one run traced with U-236 (or another suitable standard).

NOTE: No corrections are necessary, if there is no U-232 activity in the U-236 traced run. If U-232 activity is present in the U-236 traced run, then the yield of the U-232 must be corrected as follows:

$$U_{232s} = U_{232f} * \left(\frac{eff_i}{eff_f}\right) * \left(\frac{counttime_i}{counttime_f}\right) * \left(\frac{squant_i}{squant_f}\right)$$

$$U_{232t} = U_{232obs} - U_{232}$$

$$Yield_{corr} = \frac{U_{232t}}{(Eff_i * Counttime_i * U_{232tadded})}$$

Where:

U232s	=	U-232 counts in the traced sample run due to sample activity.
U232t	=	U-232 counts in U-232 traced analysis due to tracer addition
U232f	=	U-232 counts in the U-236 traced analysis
U232added	=	U-232 dpm added as the tracer
U-232obs	=	Total U-232 counts observed in the U-232 traced analysis
Effi	=	Efficiency of detector used in U-232 traced analysis
Eff_f	=	Efficiency of detector used in U-236 traced analysis
$Counttime_i$	=	Count time of U-232 traced analysis
$Counttime_f$	=	Count time of U-236 traced analysis
$Squant_i$	=	Sample aliquot of U-232 traced analysis
$Squant_f$	=	Sample aliquot of U-236 traced analysis
$Yield_{corr}$	=	Corrected Yield of U-232 traced analysis

15.5.4 The final results are then corrected by substituting the corrected yield into the equations listed in sections 15.1 through 15.3:

15.6 Record the following information on the alpha que sheet: preparation date, analysts initials, spike isotope, spike code, spike volume, LCS isotope, LCS code, LCS volume, nominal concentration LCS, and nominal concentration MS. For each sample record the detector number, sample mass, sample date, and sample time.

16.0 Quality Control Requirements

16.1 Analyst and Method Verification

16.1.1 Refer to SOP "Analyst and Analytical Methods Validation Procedures" GL-EPI-E-D002) for instructions concerning the validation of analysts and analytical methods.

16.2 Method Specific Quality Control Requirements

16.2.1 A method blank will accompany each batch of 20 or less samples. The reported value should be less than or equal to the CRDL for all target isotopes.

16.2.2 A matrix spike (MS) should be run with every batch of 20 samples. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\%\text{Rec} = \frac{\text{spike(pCi/ unit)} - \text{sample(pCi/ unit)}}{\text{spiked amount(pCi/ unit)}} * 100$$

16.2.3 A sample duplicate should be run with every batch of 20 or less samples. The relative percent difference (RPD) between the sample and the duplicate should be less than or equal to 20%. The RPD is calculated as follows.

$$\text{RPD} = \frac{\text{high sample(pCi / unit)} - \text{low sample(pCi / unit)}}{\text{Average (pCi / unit)}} * 100$$

16.2.4 A laboratory control spike (LCS) should be run with every batch of 20 samples or less. The recovery of the spike should fall between 75 and 125%. The recovery is calculated as follows:

$$\text{LCS} = \frac{\text{observed\_pCi / unit}}{\text{known\_pCi / unit}} * 100$$

16.3 Actions Required if the Quality Control Requirements Are Not Met

16.3.1 If any of the above criteria cannot be satisfied, the analyst should inform the Group Leader and initiate a non-conformance report as outlined GEL SOP "Documentation of Nonconformance Reporting and Dispositioning, and Control of Nonconforming Items" (GL-QS-E-004).

17.0 Data Review, Approval, and Transmittal

17.1 Refer to EPI SOP "Data Review and Validation Procedures" (D-003) for instructions concerning the data review process, approval, and transmittal.

18.0 Records Management

18.1 Each analysis that is performed on the instrument is documented in the run log according to GEL SOP "Run Logs" (GL-LB-E-009).

18.2 All raw data printouts, calculation spreadsheets and batch checklists are filed with the sample data for archival and review.

19.0 Laboratory Waste Handling And Waste Disposal

19.1 Radioactive samples and material shall be handled and disposed of as outlined in SOP "Laboratory Waste Disposal and Emergency Instructions" (GL-EPI-E-S011).

20.0 References

20.1 EPA Environmental Monitoring and Support Laboratory. Las Vegas. Radiochemical Analytical Procedures for Analysis of Environmental Samples. March 1979.

20.2 EML Procedures Manual HASL-300, 1982.

20.3 Analytical Chemistry. Rapid Determination of Th-230 in Mill Tailings by alpha spectroscopy. UNC Geotech, Grand Junction Projects Office. Steve Donivan, Mark Hollenbach, and Mary Costello. Vol. 59, No. 21, 1987.

20.4 Los Alamos Health and Environmental Chemistry: Analytical Techniques. LA-10300-M Vol. 1, September 1987.

20.5 Special thanks to Dr. Bill Burnett and his associates for assistance in developing this method at Florida State University.