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CONTAMINATED SOILS ADJACENT TO THE SEWAGE TREATMENT PLANT INCINERATOR REMOVAL ACTION NUMBER 14 WORK PLAN JULY 1992

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DOE/EPA
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WORK PLAN

CONTAMINATED SOILS ADJACENT TO THE SEWAGE TREATMENT PLANT INCINERATOR

REMOVAL ACTION NUMBER 14 WORK PLAN

FERNALD ENVIRONMENTAL MANAGEMENT PROJECT

JULY 1992

U.S. DEPARTMENT OF ENERGY

CONTAMINATED SOILS ADJACENT TO THE SEWAGE TREATMENT PLANT INCINERATOR

REMOVAL ACTION #14 WORK PLAN

JULY 1992

U. S. Department of Energy
P. O. Box 398705
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CONTAMINATED SOILS ADJACENT TO THE SEWAGE TREATMENT PLANT INCINERATOR

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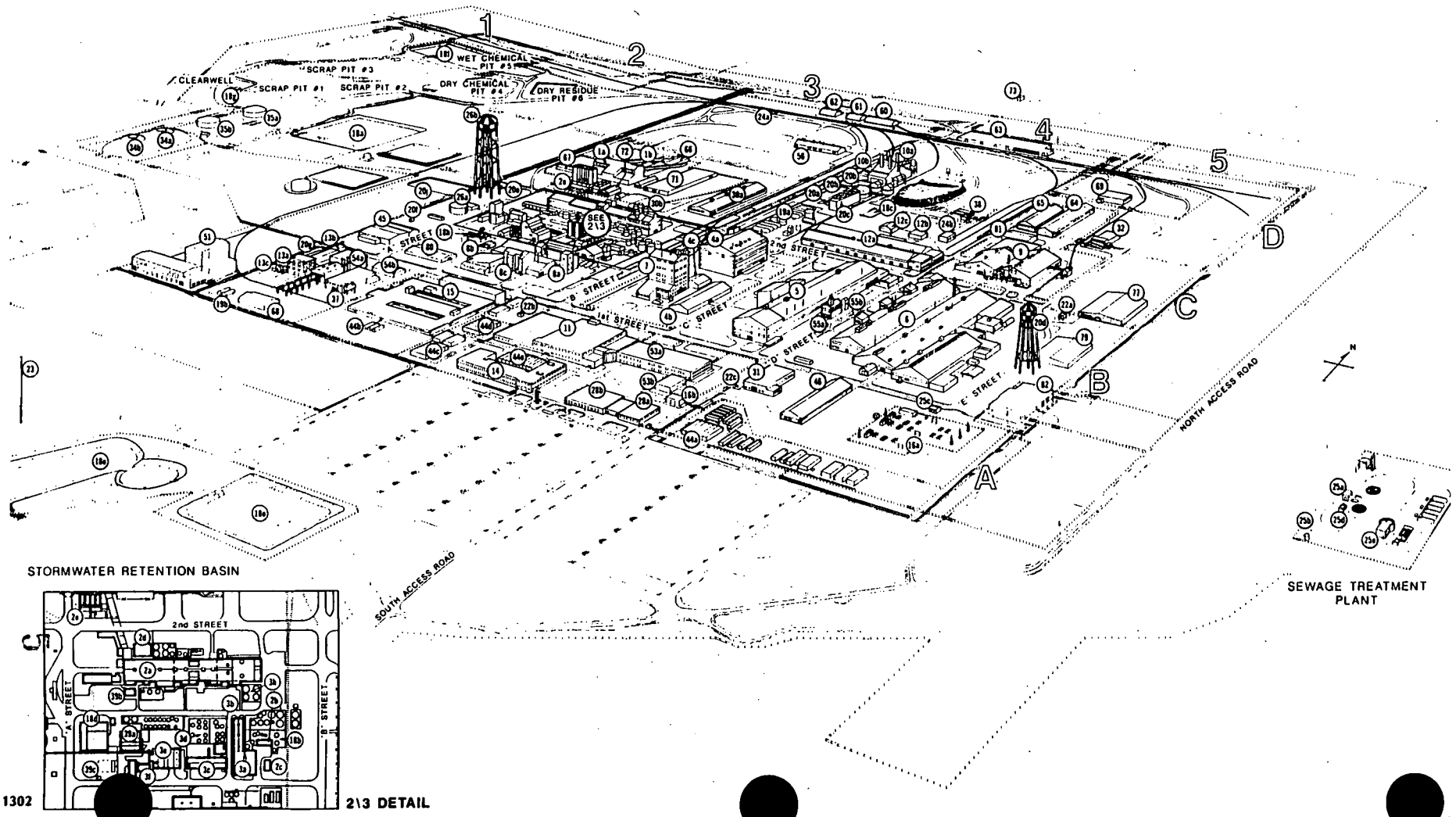
1.0 INTRODUCTION

On September 20, 1991, the U.S. Department of Energy (DOE) and the U.S. Environmental Protection Agency (U.S. EPA) jointly signed an Amended Consent Agreement establishing milestones for the implementation of Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) response actions at the Fernald Environmental Management Project (FEMP). One such milestone provided that the DOE submit a work plan to U.S. EPA by January 23, 1992 addressing Removal Action No. 14, Contaminated Soils Adjacent To The Sewage Treatment Plant Incinerator. After the submittal and U.S. EPA's review, comments were received from USEPA on February 28, 1992 which corresponded with a milestone for re-submittal by March 30, 1992. On May 20, 1992 the DOE was given a conditional approval pending incorporation of EPA's comments. This document provides the revised work plan for Removal Action No. 14. The objective of the removal action is to mitigate any incremental threat to human health and the environment, until final remediation of this area can be accomplished. The DOE conducted a Removal Site Evaluation (RSE), Appendix I, to determine if conditions present in the soil in the Sewage Treatment Plant area warranted a removal action under CERCLA, consistent with Section 300.410 of the National Oil and Hazardous Substance Pollution Contingency Plan (NCP). Based upon the information in the RSE, the DOE issued an Action Memorandum stating that a Removal Action is warranted under authorities delegated to the DOE under Section 104 of CERCLA, through Executive Order 12580. The proposed removal action is protective of human health and the environment and will be conducted in accordance with all CERCLA requirements.

As shown in Figure 1, the Sewage Treatment Plant is located on the eastern edge of the FEMP property. The FEMP Sewage Treatment Plant has operated from 1952 to the present providing physical and biological treatment of FEMP wastewater. Also located at the Sewage Treatment Plant is an abandoned-in-place solid waste incinerator (see Figure 2). The incinerator operated from 1954 until 1979 burning combustible wastes generated from FEMP administrative and process areas. Process area wastes burned at the incinerator contained low levels of radioactive materials and potentially other hazardous substances.

As summarized in the attached RSE and the attached radiological walkover survey (see Appendix II), which employed a 2"x2" NaI Detector, characterization activities completed as part of the FEMP Environmental Monitoring Program and the Remedial Investigation/ Feasibility Study (RI/FS) identified elevated concentrations of radionuclides in soils in the vicinity of the Sewage Treatment Plant. To date, no analysis has been completed for the possible presence of non-radiological hazardous substances in the soils in the study area. As evidenced by the findings of these characterization efforts, the highest activity concentrations of radiological constituents were found on-site in the surface soils adjacent to the abandoned incinerator and adjacent to some of the operational facilities associated with sewage treatment. There is one exception, a localized area adjacent to the FEMP property-line fence (to the southeast of the Sewage Treatment Plant compound) in a vacant field.

FIGURE 3: FMPC Site Perspective



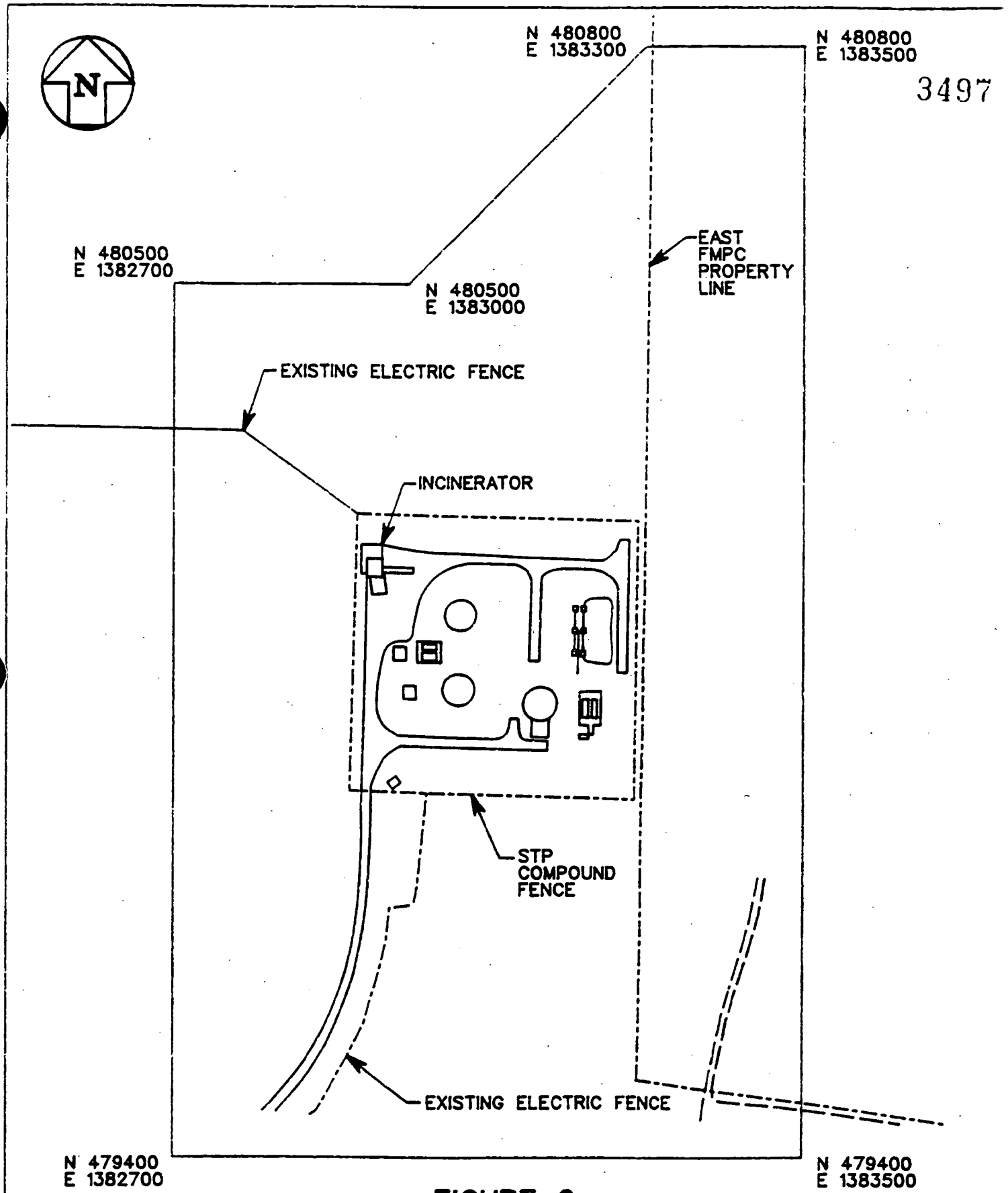


FIGURE 2

SCALE: 1" = 165'

Executive Resource Associates, Inc.
A Technical Services Company

SUSPECT AREAS

DRAWN BY: JIM KING
DATED: 3-25-92

047-1-0007
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This work plan utilizes a three phase approach to execute the removal action. Phase I: layout the study area boundary; perform instrument correlation; perform off-property surface soil sampling along the established sampling grid; perform a radiological walkover survey covering the entire study area (both on and off property) to highlight localized areas exceeding 100 pCi/g total uranium in soil; sample the center of each off-property "hot spot"; and excavate and containerize the on-property soil with a total uranium concentration exceeding 100 pCi/g. Phase II: sample the containerized soil; perform post-excavation verification sampling within the excavated areas; perform on-property post-excavation surface soil sampling activities along the established grid; and complete and issue interim report outlining excavation, sampling activities and reported analytical results from Phases I and II. Phase III: revise the existing RSE based on Phase I and II sampling results; and issue a final report outlining any further actions warranted in the study area.

In the absence of final remedial action goals, an interim field action level of 100 pCi/g has been adopted for purposes of directing on-property excavation activities to areas of highest radionuclide activity which can be readily identified by hand-held radiological instrumentation and immediately excavated. 100 pCi/g will also be used for the walkover "action level" off-property due to correlation limitations of the hand-held radiological instrumentation used. The analytical results from the off-property samples, which will be expedited, will provide accurate data upon which the need for further action may be examined. While evaluating the off-property data, an action level of 35 pCi/g will be used in examining the need for further action. The off-property action level of 35 pCi/g is being utilized based on previous clean-up activities at Manhole (MH) 180.

All project activities will be completed in accordance with the requirements defined in applicable Westinghouse Environmental Management Company of Ohio (WEMCO) procedures, in the NCP, CERCLA, pertinent DOE Orders, and the FEMP Quality Assurance Plan. Final remedial actions in the vicinity of the Sewage Treatment Plant will be conducted as part of Operable Units 3 and 5 (OU3 & OU5).

1.1 FERNALD SITE BACKGROUND

The FEMP is owned by the DOE and was operated from 1952 until 1989 for the processing of high purity uranium metal. In 1989 facility production operations were placed on stand-by to focus on environmental compliance related issues. The facility was formally shutdown in 1991 after appropriate congressional notifications. Today, remaining workforces at the facility are focused solely on the implementation of environmental restoration related initiatives.

The facility is a 1,050-acre parcel located in southwestern Ohio. In November, 1989, the FEMP was placed on the CERCLA National Priorities List (NPL) as a result of concerns related to past and potential releases of hazardous substances to the environment. Consistent with Section 120 of CERCLA, the DOE and U.S. EPA jointly

signed a Consent Agreement in March, 1990 establishing a schedule for the implementation of a sitewide RI/FS and a series of removal actions at the FEMP. This agreement was amended in September, 1991. This removal action work plan has been completed consistent with the terms of this Amended Consent Agreement.

Since October 1, 1990, responsibility for the FEMP has been administered through the Environmental Restoration and Waste Management Division of the DOE in order to better manage activities on the site.

1.2 BACKGROUND OF THE SEWAGE TREATMENT PLANT AREA

As previously discussed, the Sewage Treatment Plant area is located on the eastern edge of the FEMP property. The Sewage Treatment Plant, associated facilities and the abandoned incinerator are contained within a six foot chain link fenced area on FEMP property where access is restricted by security officers. The Sewage Treatment Plant became operational in 1952 for the treatment of FEMP sanitary wastewater. The system was later transitioned to receive both sanitary and process related wastewaters. The practice of employing the Sewage Treatment Plant to treat process related wastewater flows was discontinued recently with the installation and start-up of the biodenitrification effluent treatment system. Surface radiological measurements and limited soil samples collected in the vicinity of these facilities indicate the presence of localized elevated concentrations of radionuclides.

The solid waste incinerator is located in the northwest corner of the Sewage Treatment Area. This incinerator was operated from November 1954 through December 1979 at which time a new solid waste incinerator at Building 39 was put into service. The incinerator at the Sewage Treatment Plant was used to burn contaminated and uncontaminated combustible trash during its period of operation. Soil sampling results from the RI/FS indicate that radiological concentrations in the soils adjacent to the solid waste incinerator exceed those observed in prior routine environmental sampling conducted in 1984 and 1985 as part of the Environmental Monitoring Program. The concentrations of uranium-238 ranged from 1.8 to 25,670 pCi/g, in surface soil sampling results (see RI/FS data utilized in the RSE, Appendix I).

The abandoned solid waste incinerator is located within the fence around the Sewage Treatment Plant area, but the majority of the radiologically contaminated soil, as evidenced by the available data, is located outside the Sewage Treatment Plant's fenced boundary on FEMP property, adjacent to the incinerator. The area outside the fence has primarily been used for grazing cattle (under a lease agreement with the DOE). As a result of the RSE and the Action Memorandum, administrative control of some of the surrounding areas was established in December, 1990 with the transfer of all cattle grazing in the pasture areas directly north of the Sewage

Treatment Plant area. In order to allow grazing in areas further north in the spring, when they would normally be returned to this area, livestock fencing was installed in April of 1991 to preclude access to areas adjacent to the incinerator. Based on RI/FS data, the new fence was installed approximately 665 feet north of the incinerator at the Sewage Treatment Plant.

The solid waste incinerator at the Sewage Treatment Plant has been identified as a suspect facility to be addressed under the RI/FS for OU3. The RI/FS for OU3, aimed at investigating the remedial alternatives in the Production Area and associated facilities, is presently underway. The soils in the vicinity of the structures at the Sewage Treatment Plant are within the scope of the RI/FS for Operable Unit 5.

1.3 SUMMARY OF EXISTING DATA

Both the routine Environmental Monitoring Program (EMP) and the on-going RI/FS have shown evidence of localized radiological contamination in the vicinity of the Sewage Treatment Plant area. Once again, it is important to note that no analyses have been completed for the possible presence of non-radiological hazardous substances in the soils in the study area. These analyses are part of Phases I and II of this removal action.

The EMP contains data from surface soil sampling locations 3 and 11 (EMP-SS3 & EMP-SS11) and Air Monitoring Station No. 3 (AMS 3), all shown on Figure 1 of the RSE. The RSE includes data from this sampling program for the years 1984, 1985, and 1989. Historical air sampling data for 1989 from AMS 3, approximately 350 feet downwind (northeast) of the incinerator, shows average radiological concentrations which lead to an inhalation dose estimate of less than one millirem (mrem) per year (see the RSE for further details).

The on-going RI/FS surface soil samples and sub-surface core samples collected in the vicinity of the solid waste incinerator at the Sewage Treatment Plant showed considerably higher radiological concentrations than previously observed under the EMP. The two highest surface soil radiological concentrations, closest to the incinerator, showed 25,670 pCi/g and 2,376 pCi/g of uranium-238. Figure 1 of the RSE shows sampling locations for the RI/FS data utilized for the RSE. The data from these sample points are listed in Table B.1 of the RSE (RI/FS Soil Sample Results). Table B.2 of the RSE includes additional RI/FS data obtained since the Action Memorandum was issued. None of this additional data exceeds any of the data utilized for the RSE.

In addition to surface soil samples, there were a limited number of core samples taken in this area as part of the RI/FS. These borings extended to a depth of 20 feet. The results from these samples are listed in Table 4 of the RSE and show only one sample exceeding the 100 pCi/g field action level at a depth of 1.5 - 3.0 feet. All of

these sample points are within the Sewage Treatment Plant compound. There has been some sub-grade disturbance within the compound due to sewage treatment plant upgrades and the placing of fill to improve drainage. Since there have been little to no known disturbance of the soils outside the fenced area at the Sewage Treatment Plant, contamination is likely to be limited to surface soils resultant from air deposition from incinerator operation.

Radiological walkover surveys were performed as part of the RI/FS using 2"x2" NaI detectors (Eberline SPA-3). Appendix II is a map showing isopleths developed from this data. The normalized SPA-3 walkover data presented in Appendix II provides the relative indications of "hot spots" exhibiting concentrations of gamma emitting radionuclides. All of these areas of higher concentrations are on FEMP property with the exception of a localized area adjacent to the FEMP property-line fence in a vacant field. The study area was established based on radiological walkover survey data (see Appendix III). As a result, the study area, and consequently the sampling grid, were developed to encompass all areas of "higher concentration".

1.4 NEED FOR A REMOVAL ACTION

Utilizing available data, three potential exposure pathways of radiological contamination to man were examined in the RSE: external exposure, inhalation, and milk ingestion. Other pathways were discounted in the RSE due to the relatively short durations of potential exposure until final remedial actions are implemented, and due to the existing access controls in place in the area.

Eight factors were considered in the assessment of the need for a removal action. These eight factors are listed in 40 CFR 300.415 (b)(2). The following factors apply specifically to the above background concentrations of contaminants occurring in the soils adjacent to the Sewage Treatment Plant area.

<u>40 CFR 300.415 (b)(2)(i)</u>

Actual or potential exposure to nearby human populations, animals, or the food chain from hazardous substances or pollutants or contaminants.

Appropriate due to nearby resident farmer and nearby grazing cattle.

<u>40 CFR 300.415 (b)(2)(iv)</u>

High levels of hazardous substances or pollutants or contaminants in soils largely at or near the surface, that may migrate.

Appropriate based on radiological concentrations found in surface soil samples taken adjacent to the solid waste incinerator at the Sewage Treatment Plant.

40 CFR 300.415 (b)(2)(v)

Weather conditions that may cause hazardous substances or pollutants or contaminants to migrate or be released.

Appropriate based on radiological concentrations found in surface soil samples taken adjacent to the solid waste incinerator at the Sewage Treatment Plant and the possibility of significant weather events carrying the contaminants out of the study area in surface runoff.

1.5 OBJECTIVES OF THE REMOVAL ACTION

The objectives of the removal action are to reduce the potential for contaminant migration to previously uncontaminated areas, and minimize the potential for unacceptable exposures to human or environmental receptors until implementation of final remedial actions. Consistent with the NCP, the removal action shall contribute to the efficient performance of projected final remedial actions. The removal action shall be performed to minimize the potential for releases of hazardous substances incidental to removal field operations and in a cost efficient and safe manner consistent with site Standard Operating Procedures and worker health and safety requirements.

2.0 REMOVAL ACTION

This removal action is composed of three phases. Phase I: layout the study area boundary; perform instrument correlation; perform off-property surface soil sampling along the established sampling grid; perform a radiological walkover survey covering the entire study area (both on and off property) to highlight localized areas exceeding 100 pCi/g total uranium in soil; sample the center of each off-property "hot spot"; and excavate and containerize the soil on-property with a total uranium concentration exceeding 100 pCi/g. Phase II: sample the containerized soil; perform post-excavation verification sampling within the excavated areas; perform on-property post-excavation surface soil sampling activities along the established grid; and complete and issue interim report outlining excavation, sampling activities and reported analytical results from Phases I and II. Phase III: revise the existing RSE based on Phase I and II sampling results; and issue a final report outlining any further actions warranted in the study area.

Once the study area boundary is established, the instrument correlation samples will be taken on-property, so that the analysis will be expedited and off-property surface soil sampling will be initiated along the grid. DOE will ensure the expeditious collection and analysis of these samples due to the potential for off-property contamination. For uranium an off-property action level of 35 pCi/g will be utilized while evaluating this data. It is not anticipated that thorium or radium will be detected, however if they are, while examining the need for further action, appropriate action levels will be evaluated.

After the off-property surface soil sampling is complete, a surface radiological survey will be performed across the study area (see Appendix III), which encompasses areas both on and off FEMP property to identify localized areas exceeding 100 pCi/g total uranium in soil. The radiological survey will be performed across the study area utilizing a shielded 2"x2" NaI detector. The Sampling and Analysis Plan outlines the method for correlating the NaI detector to 100 pCi/g total uranium in soil. Areas exceeding 100 pCi/g will be temporarily marked for excavation on-property and marked and sampled at the center off-property. The off-property "hot spots" will remain marked and undisturbed pending analytical results for total U, total Th, and Ra-226 from the on-site lab.

While it is recognized that detailed chemical data are not available, the DOE considers it prudent to proceed with the excavation of "hot spots" based on available radiological data. It is the intent of DOE to excavate and containerize soils from these localized "hot-spot" areas to allow progressive cleanup activities to proceed while awaiting detailed radiological and chemical analytical data from the study area. In the event that the work plan for the Improved Storage of Soil and Debris (Removal Action (RA) #17) is approved by EPA prior to excavation, all excavated soil will be managed in accordance with provisions outlined in the RA #17 Work Plan.

Excavations will proceed only on FEMP property. Additional discussion on the excavation process can be found in Section 2.1. In the event that elevated concentrations are identified off-property, the area will be marked and the property owner will be notified and negotiations for a prudent course of action will be initiated with the owner. This course of action could range from simple notification or access controls to excavation activities. The U.S. EPA and Ohio EPA will be notified in this event and consulted on the appropriate course of action prior to implementation.

Following excavation: each container will be sampled; verification samples will be collected from the center of each excavation and sent to the on-site lab for total uranium analysis; and surface soil samples will be collected along the established grid to determine the concentrations of radiological and chemical constituents present in the surface soils remaining in the study area (see Table 1). Soil samples will be collected consistent with the Sampling and Analysis Plan (Section 3.0) and RI/FS Quality Assurance Project Plan (QAPP) protocols. Soil sampling is further discussed in Section 3.0. Once analytical results are received, an interim report will be completed and provided to EPA describing Phase I and II activities. On the basis of the analytical results obtained from Phases I and II, the RSE will be revised to examine the need for further action. Based on the findings of the revised RSE, a final report will be submitted proposing the need, if any, for additional field actions.

2.1 PHASE I

The first field activity of the removal action is to layout the study area boundary. Once the boundary has been established, the correlation samples will be obtained from the study area, on-property. While the correlation samples are being collected, the off-property surface soil samples will also be collected along the established grid, so that the analyses can be expedited. All grid locations (both on and off property) were chosen based upon existing walkover and soil sample data (Appendix II and RSE). The grid will be laid out in accordance with the map provided in Appendix III. Final sample locations will be surveyed and tied into the state planar coordinate system.

As previously discussed, a radiological survey will be performed within the study area employing a shielded 2"x2" NaI detector. A correlation will be established to permit the real-time approximation of soils exhibiting greater than 100 pCi/g of total uranium (see Sampling and Analysis Plan for correlation method).

As the walkover survey progresses, any area found to exceed 100 pCi/g will be marked for excavation, on-property, and marked and sampled at the centers, off-property. Duplicate samples will be taken from the centers of the off-property "hot spots" with one set going to the on-site lab for quick turn-around. After the areas on-property have been marked, excavation can begin and will continue until in-situ soil concentrations are below 100 pCi/g total U as determined by direct radiological measurement using the NaI detector. Based on existing soil sample data, it is estimated that an average of 6"-12" will need to be excavated as part of Phase I from the localized "hot spot" areas. Because of these shallow excavations, this activity can be performed by hand. If any of the excavations pose a safety threat, temporary access controls, such as fencing or roping, may be installed. In the event that excavations threaten structural integrity of nearby facilities, DOE will evaluate the need for engineered reinforcement. During excavation activities, logs including maps will be developed to record excavated areas which will be included in the interim report to be completed at the end of Phase II.

As previously stated, this action level will approximate 100 pCi/g of total uranium in soil assuming a natural isotopic activity distribution. A hand-held organic vapor analyzer will also be used during excavation; any detection will direct the need for excavation.

Excavated soil will be containerized with the containers being placed in an interim storage area within the study area boundary for sampling. Pending analytical results, a hazardous waste determination will be completed. In the event the soil is determined to be hazardous pursuant to 40 CFR 262.11, the containers will be transferred to on-site hazardous waste storage facilities. Upon

approval of the work plan for Removal Action No.17 - Improved Storage of Soil and Debris by U.S. EPA, management of the excavated soil will be transitioned to be consistent with the requirements of this approved plan.

Excavations will not be backfilled or re-graded. If it is determined that any of the excavations pose a safety threat, access controls will be put in place.

2.2 PHASE II

After excavation activities are complete: all containers will be representatively sampled in accordance with the methodology presented in Appendix IV (see Section 3.0 for further details); post-excavation validation samples will be taken from the excavated areas and sent to the on-site laboratory for total uranium analysis only to verify the 100 pCi/g in-situ concentration; and post-excavation surface soil samples will be taken at the 32 on-property locations shown in Appendix III (see Section 3.0 for further details). Once all analytical results become available, an interim report will be issued containing: correlation sampling results; off-property sampling results; walkover survey results (including a map showing "hot spot" locations); an outline of excavation activities; verification sampling results; and post-excavation surface soil sampling results.

2.3 PHASE III

Once results from the containerized and surface soil sampling activities are available, the RSE will be revised to incorporate all sampling results, including non-radiological parameters. The RSE will consider multiple contaminant exposure scenarios. The RSE will consider the recommendations contained in the 1981 Nuclear Regulatory Commission (NRC) Branch Technical Position (BTP). Based on the results of the revised RSE, a final report will be submitted defining the need, if any, for additional actions to address the soil in the Sewage Treatment Plant Area.

3.0 SAMPLING AND ANALYSIS PLAN

As previously mentioned, sampling activities take place during both Phase I and Phase II. During Phase I, correlation samples will be collected, off-property surface soil samples will be collected and a walkover survey (both on and off FEMP property) will be performed to highlight areas exceeding 100 pCi/g total U in soil, and the "hot spots" off-property will be sampled at their centers. Once the on-property "hot spots" have been excavated, the containers will be sampled, verification samples will be taken from the excavations, and the on-property post-excavation surface soil samples will be taken along the established grid. Table 1 provides a list of sampling and analysis activities.

TABLE 1

Samples to be Taken

Phase	Sample Description	Types of Analyses	on-site/ off-site lab
I	1) Correlation	20 for constituents in Table 1B	off-site
I	2) 8 off-property surface soil samples	6 for radiological constituents in Table 1A 2 for radiological and HSL constituents in Table 1A	off-site off-site
I	3) Off-property "hot-spots" sampled @ centers (duplicate samples)	1 set for total U, total Th & Ra-226 1 set for radiological constituents in Table 1	on-site off-site
II	4) Containers	All constituents in Table 1A & TCLP metals	off-site
II	5) Post-excavation verification samples, from centers of excavations	Total U analysis	on-site
II	6) Post-excavation samples along established grid	20 for radiological constituents in Table 1A 8 for radiological and HSL constituents in Table 1A 4 for radiological, HSL and dioxin/furan	off-site off-site off-site

In order to initiate the walkover survey to highlight the areas exceeding the 100 pCi/g action level, the shielded 2"x2" NaI detector response must first be correlated to known soil activities. The NaI scintillation detector will effectively survey gamma-ray emitters. This would include thorium, radium-226, and uranium which has a natural isotopic activity distribution of uranium-238 and uranium-235. The normal uranium is effectively detected because of its uranium-235, uranium-238 and daughters of uranium-238 (protactinium-234 and thorium-234). The process of correlating the 2"x2" NaI detector to 100 pCi/g of total uranium will be accomplished through the collection of a series (approximately 20) of surface soil samples from locations within the study area (on-property) representing a range of instrument measurements. Samples will be collected in a manner consistent with the criteria called out in Section 3.1 from a depth of 0" - 8" at a location directly below the detector. Collected samples shall be submitted to the RI/FS laboratory for analysis of the radionuclides listed in Table 1B. A plot will be developed depicting NaI scintillation response in counts per minute (cpm) versus total uranium activity concentrations in pCi/g (dry weight). Samples exhibiting significant activity concentrations of other gamma emitting radionuclides will be excluded from consideration. A curve will be fit against the plotted data. This curve will be established as the basis for correlation of the 2"x2" NaI detector and rate-meter scaler measurements to 100 pCi/g total uranium. DOE considers this method to provide an adequate basis for Phase I excavation activities (i.e. limited soil removal from hot-spot locations).

Concurrent with the collection of the correlation samples, the off-property surface soil samples will be collected. See Table 1 for sampling and analyses required.

Once the instrument correlation is complete the walkover survey will begin (see Table 2 for Data Quality Objectives). The field measurement procedure is made up of the following steps:

- Check proper operation of the detector and its associated ratemeter/scaler according to the manufacturer's instructions.

- Perform daily source checks with a known source and record the response.

- Perform daily background checks at the field office and record the response.

- Survey the study area with an unshielded detector by walking at a steady rate across the study area in a serpentine manner. When a "hot spot" is detected the area will be outlined and the 1/2" lead shielding will be put on the detector to take detailed measurements to more clearly define the "hot spot" boundary. The defined boundary will be marked with stakes for subsequent on-property excavation and off-property sampling.

TABLE 1 A

RADIONUCLIDES

1 Uranium 234
2 Uranium 235
3 Uranium 238
4 Radium 226
5 Radium 228
6 Thorium 228
7 Thorium 230
8 Thorium 232

INORGANICS

1 Aluminum
2 Antimony
3 Arsenic
4 Barium
5 Beryllium
6 Cadmium
7 Calcium
8 Chromium
9 Cobalt
10 Copper
11 Cyanide
12 Iron
13 Lead
14 Magnesium
15 Manganese
16 Mercury
17 Molybdenum
18 Nickel
19 Potassium
20 Selenium
21 Silicon
22 Silver
23 Sodium
24 Thallium
25 Vanadium
26 Zinc

VOLATILE ORGANICS

1 1,1-Dichloroethane
2 1,1-Dichloroethene
3 1,1,1-Trichloroethane
4 1,1,2-Trichloroethane
5 1,1,2,2-Tetracholorethane
6 1,2 Dichloroethene (total)
7 1,2-Dichloroethane
8 1,2-Dichloroethylene
9 1,2-Dichloropropane
10 2-Butanone
11 2-Hexanone
12 4-Methyl-2-pentanone
13 Acetone
14 Benzene
15 Bromodichloromethane
16 Bromoform
17 Bromomethane
18 Carbon disulfide
19 Carbon tetrachloride
20 Chlorobenzene
21 Chloroethane
22 Chloroform
23 Chloromethane
24 cis-1,3-Dichloropropene
25 Dibromochloromethane
26 Ethylbenzene
27 Methylene chloride
28 Styrene
29 Tetrachloroethene
30 Toluene
31 Total xylenes
32 trans-1,3-Dichloropropene
33 Trichloroethene
34 Vinyl acetate
35 Vinyl chloride

SEMIVOLATILE ORGANICS

1 1,2-Dichlorobenzene
2 1,2,4-Trichlorobenzene
3 1,3-Dichlorobenzene
4 1,4-Dichlorobenzene
5 2-Chloronaphthalene
6 2-Chlorophenol
7 2-Mehylnaphthalene
8 2-Methylphenol
9 2-Nitroaniline
10 2-Nitrophenol
11 2,4-Dichlorophenol
12 2,4-Dimethylphenol
13 2,4-Dinitrophenol
14 2,4-Dinitrotoluene
15 2,4,5-Trichlorophenol
16 2,4,6-Trichlorophenol
17 2,6-Dinitrotoluene
18 3-Nitroaniline
19 3,3'-Dichlorobenzidine
20 4-Bromophenyl phenylether
21 4-Chloro-3-methylphenol
22 4-Chloroaniline
23 4-Chlorophenyl-phenyl ether
24 4-Methylphenol
25 4-Nitroaniline
26 4-Nitrophenol
27 4,6-Dinitro-2-methylphenol
28 Acenaphthene
29 Acenaphthylene
30 Anthracene
31 Benzoic acid
32 Benzo(a)anthracene
33 Benzo(a)pyrene
34 Benzo(b)fluoranthene
35 Benzo(g,h,i)perylene
36 Benzo(k)fluoranthene
37 Benzyl alcohol
38 bis(2-Chloroethoxy)methane
39 bis(2-Chloroethyl)ether
40 bis(2-Chloroisopropyl)ether
41 bis(2-Ethylhexyl)phthalate
42 Butylbenzylphthalate
43 Carbazole
44 Chrysene
45 Dibenzofuran
46 Dibenzo(a,h)anthracene
47 Diethylphthalate
48 Dimethylphthalate
49 Di-n-butyl phthalate
50 Di-n-octyl phthalate
51 Fluoranthene
52 Fluorene
53 Hexachlorobenzene
54 Hexachlorobutadiene
55 Hexachlorocyclopentadiene
56 Hexachloroethane
57 Indeno(1,2,3-cd)pyrene
58 Isophorone
59 Naphthalene
60 Nitrobenzene
61 N-Nitroso-di-n-propylamine
62 N-Nitrosodiphenylamine
63 Pentachlorophenol
64 Phenanthrene
65 Phenol
66 Pyrene

DIOXINS

1 2,3,7,8-Tetrachlorodibenzo-p-dioxin

TABLE 1B

RADIOLOGICAL PARAMETERS FOR CORRELATION SAMPLES

1	Actinium 227
2	Cesium 137
3	Protactinium 231
4	Radium 224
5	Radium 226
6	Radium 228
7	Ruthenium 106
8	Thorium 230
9	Thorium 234
10	Total Uranium
11	Uranium 235
12	Uranium 238

TABLE 2

DATA QUALITY OBJECTIVES FOR FIELD SURVEYS AND OFF-PROPERTY "HOT SPOT" SAMPLING ASSOCIATED WITH THE CONTAMINATED SOILS ADJACENT TO THE SEWAGE TREATMENT PLANT INCINERATOR REMOVAL ACTION

Activity	Conduct a radiological walkover survey within the study boundary to highlight areas which exceed 100 pCi/g total uranium in soil. Sample off-property "hot spots".
Objectives	Define in-situ localized areas which exceed the 100 pCi/g total uranium action level to direct on-property excavations. This field survey will also direct the sampling of off-property "hot spots" (greater than 100 pCi/g total uranium). Duplicate samples will be taken from the "hot spots" with one Set #1 sent to the on-site lab, and Set #2 sent off-site.
Prioritized Data Use(s)	The priority data use is to support excavation and sampling activities within the scope of this removal action.
Appropriate Analytical Level	Walkover survey: Level II "Hot spot" surface soil samples - Radionuclides: Level IV
Constituents of Concern	Walkover survey: Total Uranium Set #1 - Surface Soil Samples: Total U, Total Th, Ra-226 Set #2 - Surface Soil Samples: See Radionuclides in Table 1A
Required Detection Limits	See RI/FS Work Plan, Volume V - Quality Assurance Project Plan, Revision 3, Table 4-1, 4-2, and 4-3.
Critical Samples	No individual sample is considered critical; however, representative sampling is considered critical to determine in-situ concentrations. The study requires that a sufficient number of the samples pass the specified data validation to meet the criteria of Section 4.0 of the approved RI/FS QAPP for the sampling interval.

The areas marked off-property will be sampled at their centers with duplicate samples taken. One set will be sent to the on-site lab for quick analyses of total U, total Th and Ra-226 and the other set will be sent off-site and analyzed for the radionuclides listed in Table 1A. (See Table 2 for Data Quality Objectives).

Once on-property excavations are complete, Phase II sampling will begin: the containers will be representatively sampled (see Table 2A for Data Quality Objectives). At the same time the excavations will be sampled to verify that the in-situ total U concentration is less than 100 pCi/g (see Table 2B for Data Quality Objectives). Once the verification samples have been taken the surface soil sampling along the established grid will begin (see Table 2B for Data Quality Objectives). Soil samples will be collected from a depth of zero to six inches. See Table 1 for clarification of samples to be taken and types of analyses to be performed. See Table 3 for the Analytical Support Levels (ASLs) referenced in the Data Quality Objectives. It is important to note that for this document ASLs I - V were referenced and included in Table 3, however the draft Sitewide CERCLA Quality Assurance Project Plan (SCQ) utilizes ASLs A - E which are consistent with ASLs I - V. In the event that the SCQ is approved prior to initiation of these sampling activities, the SCQ will be utilized.

The following procedures will be used for all surface soil sampling:

- Samples will be surface soil samples from 0 to 6 inches depth. The samples will be collected using a stainless steel sampling tool such as a trowel. The VOC portion of the samples (where required) is to be collected first. The sample portion for non-VOC parameters will be composited by hand in a stainless steel bowl.

- The trowel and other sampling equipment such as bowls, gloves, etc. will be decontaminated prior to sample collection by cleaning with a non-phosphate detergent and brush, rinsing with tap water, methanol (acetone may be substituted if volatile organic compounds are not being determined), and two separate deionized water rinses.

- The final deionized water rinse following equipment decontamination will be sampled and analyzed at a frequency of one per every set of twenty locations or fraction thereof to check for potential cross-contamination between surface soil sample locations.

- Appropriate rinseates are to be collected at a frequency of one per twenty samples.

- Samples will be transferred directly from the hand trowel to the sample containers.

TABLE 2A

DATA QUALITY OBJECTIVES FOR EXCAVATED AND CONTAINERIZED SOIL SAMPLING ASSOCIATED WITH THE CONTAMINATED SOILS ADJACENT TO THE SEWAGE TREATMENT PLANT INCINERATOR REMOVAL ACTION

Activity	Conduct representative sampling of excavated and containerized soil. Samples will be analyzed for the constituents listed in Table 1.
Objectives	Define the concentration of radionuclides and HSL constituents including inorganic metals, volatile organics, and semi-volatile organics and dioxins.
Prioritized Data Use(s)	The priority data use is to support future removal action identification. Soil characterization data will be used for hazardous waste determination and integrated into the OU5 RI/FS.
Appropriate Analytical Level	Radionuclides: Level V Chemical: Level IV
Constituents of Concern	See Table 1
Required Detection Limits	See RI/FS Work Plan, Volume V - Quality Assurance Project Plan, Revision 3, Table 4-1, 4-2, and 4-3.
Critical Samples	No individual sample is considered critical; however, representative sampling is considered critical to characterize the excavated soil. The study requires that a sufficient number of the samples pass the specified data validation to meet the criteria of Section 4.0 of the approved RI/FS QAPP for the sampling interval. The containerized soil will be sampled in accordance with SMS-EXM-91-018 (see Appendix IV).

TABLE 2B

DATA QUALITY OBJECTIVES FOR IN-SITU SOIL SAMPLING ASSOCIATED WITH THE CONTAMINATED SOILS ADJACENT TO THE SEWAGE TREATMENT PLANT INCINERATOR REMOVAL ACTION

Activity	Conduct hand soil sampling with trowels and hand augers at 40 locations. Samples will be collected at 0-6 inch depth. 14 of the samples will be analyzed for the constituents listed in Table 1; the remainder will be analyzed only for the radionuclides listed in Table 1 (see Appendix III for sample locations). Four of the 14 locations will also be analyzed for dioxins. In addition to the 40 locations, verification samples will be taken from all excavated areas to ensure that in-situ soil concentrations are less than 100 pCi/g of total uranium.
Objectives	Define the concentration of radionuclides and HSL constituents including inorganic metals, volatile organics, and semi-volatile organics and dioxins.
Prioritized Data Use(s)	The priority data use is to support future removal action identification. Soil characterization data will be used for RSE revision and integrated into the OU5 RI/FS.
Appropriate Analytical Level	Radionuclides: Level V Chemical: Level IV Verification Samples (Total U): Level IV
Constituents of Concern	See Table 1
Required Detection Limits	See RI/FS Work Plan, Volume V - Quality Assurance Project Plan, Revision 3, Table 4-1, 4-2, and 4-3.
Critical Samples	No individual sample is considered critical; however, an aggregate of samples is considered critical to characterize the area and the excavated soil. The study requires that a sufficient number of the samples pass the specified data validation to meet the criteria of Section 4.0 of the approved RI/FS Work Plan for the sampling interval. The verification samples will be analyzed for total uranium utilizing the procedures attached as Appendix V.

TABLE 3
SUMMARY OF ANALYTICAL LEVELS APPROPRIATE TO DATA USES

DATA USES	ANALYTICAL LEVEL	TYPE OF ANALYSIS	LIMITATIONS	DATA QUALITY
SITE CHARACTER-IZATION MONITORING DURING IMPLEMENTATION	LEVEL I	- TOTAL ORGANIC/INORGANIC VAPOR DETECTION USING PORTABLE INSTRUMENTS - FIELD TEST KITS	- INSTRUMENTS RESPOND TO NATURALLY-OCCURRING COMPOUNDS	- IF INSTRUMENTS CALIBRATED AND DATA INTERPRETED CORRECTLY, CAN PROVIDE INDICATION OF CONTAMINATION
SITE CHARACTERIZATION EVALUATION OF ALTERNATIVES ENGINEERING DESIGN MONITORING DURING IMPLEMENTATION	LEVEL II	- VARIETY OF ORGANICS BY GC; INORGANICS BY AA; XRF - TENTATIVE ID; ANALYTE-SPECIFIC - DETECTION LIMITS VARY FROM LOW ppm TO LOW ppb	- TENTATIVE ID - TECHNIQUES/ INSTRUMENTS LIMITED MOSTLY TO VOLATILES, METALS	- DEPENDENT ON QA/QC STEPS EMPLOYED - DATA TYPICALLY REPORTED IN CONCENTRATION RANGES
RISK ASSESSMENT PRP DETERMINATION SITE CHARACTERIZATION EVALUATION OF ALTERNATIVE ENGINEERING DESIGN MONITORING DURING IMPLEMENTATION	LEVEL III	- ORGANICS/INORGANICS USING EPA PROCEDURES OTHER THAN CLP CAN BE ANALYTE-SPECIFIC - RCRA CHARACTERISTIC TESTS	- TENTATIVE ID IN SOME CASES - CAN PROVIDE DATA OF SAME QUALITY AS LEVELS IV, NS	- SIMILAR DETECTION LIMITS TO CLP - LESS RIGOROUS QA/QC
RISK ASSESSMENT PRP DETERMINATION EVALUATION OF ALTERNATIVES ENGINEERING DESIGN	LEVEL IV	- HSL ORGANICS/ INORGANICS BY GC/MS; AA; ICP - LOW ppb DETECTION LIMIT	- TENTATIVE IDENTIFICATION OF NON-HSL PARAMETERS - SOME TIME MAY BE REQUIRED FOR VALIDATION OF PACKAGES	- GOAL IS DATA OF KNOWN QUALITY - RIGOROUS QA/QC
RISK ASSESSMENT PRP DETERMINATION	LEVEL V	- NON-CONVENTIONAL PARAMETERS - METHOD-SPECIFIC DETECTION LIMITS - MODIFICATION OF EXISTING METHODS - APPENDIX 8 [illegible]METERS	- MAY REQUIRE METHOD DEVELOPMENT/ MODIFICATION - MECHANISM TO OBTAIN SERVICES REQUIRES SPECIAL LEAD TIME	- METHOD-SPECIFIC

- If soil samples are being collected for volatile organics analysis (VOA), the sample must be transferred to standard VOA vials. Vials should be completely filled to minimize any head space in the containers.

- Soil samples collected for other organic or inorganic analyses should be transferred to glass containers with screw cap closures.

- A field/trip blank for each sample will accompany the samples back to the laboratory. The field/trip blank will consist of organic-free deionized water used in the field to decontaminate the sampling equipment.

- Matrix Spike/Matrix Spike Duplicates: at least one spiked sample analysis must be performed on each group of samples of a similar matrix type and concentration for each set of samples or for each 20 samples received, whichever is more frequent. Samples identified as field blanks cannot be used for spiked sample analysis.

- Collected samples will be stored in the field in an ice chest filled with blue ice and maintained at approximately 4 degrees Celsius.

- Samples will be properly labeled and chain-of-custody records, field collection reports, and laboratory requests for analysis forms will be properly completed.

All samples will be collected, documented, packaged, shipped and analyzed in accordance with the RI/FS QAPP. All samples will be analyzed at a laboratory approved for use by the RI/FS and all data collected will be made available to on-going RI/FS activities. Final sample locations will be surveyed and tied into the state planar coordinate system.

In order to ensure the quality of the analytical data resulting from the sampling activities performed under this removal action, several criteria are listed in Table 4 with reference to the appropriate section in the RI/FS Volume V - QAPP, Rev. 3.

TABLE 4

REFERENCES FOR QUALITY ASSURANCE CRITERIA CONTAINED IN THE RI/FS WORK PLAN, VOLUME V - QAPP, REVISION 3

SECTION 4.0 - QUALITY ASSURANCE PROGRAM
SECTION 5.0 - FIELD PROCEDURES
SECTION 6.0 - SAMPLE COLLECTION PROCEDURES
SECTION 7.0 - CHAIN-OF-CUSTODY PROCEDURES
SECTION 8.0 - EQUIPMENT CALIBRATION/MAINTENANCE
SECTION 9.0 - LABORATORY ANALYTICAL PROCEDURES
SECTION 10.0 - DATA REDUCTION, VALIDATION, AND REPORTING
SECTION 11.0 - INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY
SECTION 12.0 - QUALITY ASSURANCE AUDITS
SECTION 13.0 - PREVENTIVE MAINTENANCE
SECTION 14.0 - SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION, ACCURACY, AND COMPLETENESS
SECTION 16.0 - NONCONFORMANCE/CORRECTIVE ACTION AND VARIANCES
SECTION 17.0 - QUALITY ASSURANCE REPORTS TO MANAGEMENT

4.0 RELATED ACTIVITIES

In order to help meet the clean-up goals of the DOE's nuclear waste sites, such as the FEMP, the Director of Environmental Restoration and Waste Management initiated the Office of Technology Development (OTD). One program initiated by OTD was the Integrated Demonstration (ID). The ID program focuses on improving/validating technologies by demonstrating effectiveness, cost savings, risk reduction potential, site applicability, and regulatory and public acceptance. The FEMP was selected to host an ID for uranium contaminated soils. The ID will address the issues from "cradle to grave" surrounding the remediation of uranium contaminated soils, specifically soils of a high clay/silt content. As such, one area of focus of the ID is demonstrating and evaluating technologies and approaches which can be applied to improve the current site characterization methods in terms of cost, risk, safety, and duration.

One of the major challenges facing any remedial action program is the need to quickly and accurately delineate (i.e. identification of those soils exceeding a regulatory standard) the extent of contamination at the site. Because such studies generally rely on the collection of soil samples that are subsequently shipped off-site for analysis, the cost and time required to perform these studies is significant. One means of improving the efficiency of site characterization is through the infusion of new screening technologies into such programs. The benefits represented by employing screening technologies include:

- reduction in sampling requirements
- reduction in sampling phases
- improved spatial resolution

For these reasons, testing and evaluation of four screening technologies

for delineating the distribution of uranium contamination in surface and sub-surface soils will be tested and evaluated in late August and September of 1992 under the ID. The technologies to be tested include:

- High-resolution gamma spectroscopy
- a wide-area beta scintillation counter
- inductively coupled plasma -- atomic emission spectroscopy (ICP-AES)
- a long range alpha detector (LRAD) system

The ID has proposed integrating this field demonstration with this removal action. After the contaminated soil above the 100 pCi/g action level have been excavated under the removal action (Phase I), and the post-excavation sampling is complete, the four screening techniques will be utilized to evaluate the completeness of this activity. The results of the screening techniques will be compared with the data obtained from Phase I and II sampling activities. The integration of the testing of the screening techniques with this removal action will provide a higher quantity of data for utilization in remedial design. The integration will also provide a cost savings to the ID through the utilization of the removal action data.

5.0 PROGRAM MANAGEMENT

The following activities will be undertaken to provide planning and management for the removal action.

5.1 RESPONSIBILITIES

The DOE is the lead agency for this removal action and will coordinate the execution of this removal action. As stated in the Amended Consent Agreement under CERCLA 120 and 106(a), if the DOE determines under Section 104 that any activities or work being implemented under this Amended Consent Agreement may create an imminent threat to human health or the environment from the release or threat of release of hazardous substance, pollutant, contaminant, or hazardous constituent, it may stop any work or activities for such period of time as needed to respond and take whatever action is necessary to abate the danger. Reporting to the U.S. EPA will be in accordance with Section XXIII of the Amended Consent Agreement.

U.S. EPA shall review, comment and approve the work plan and follow progress through the interim report, meetings/site visits, the final report and the Amended Consent Agreement progress reports.

WEMCO, the Maintenance and Operations Contractor at the FEMP, will coordinate, manage, implement, monitor activities and prepare all reports associated with the removal action in a manner consistent with the DOE and regulatory requirements and guidance.

This removal action shall be managed by the WEMCO/DOE OU3 team to ensure compatibility with the final remedial action(s) selected for OU3 and OU5. Data and results from this removal action will be used

to evaluate the final remedial options for OU3 and OU5.

Ohio EPA, while not a signature to the Amended Consent Agreement, maintains a significant role in the successful implementation of removal actions at FEMP. Ohio EPA shall review and comment on the work plan and follow progress through the interim report, meetings/site visits, the final report and the Amended Consent Agreement progress reports.

All personnel directly involved in the planning and implementation of this removal action will be trained in accordance with 29 CFR 1910.120, the standard operating procedures for the work involved, and with the requirements of the approved work plan.

5.2 SCHEDULES

A proposed schedule has been developed and key milestones of this schedule are given in Table 5-1.

Table 5-1 - Key Milestones of Proposed Project Schedule

	Duration (months)	Accumulated Duration* (months)
Complete Phase I - Excavation - Off-Property Sampling	3	3
Complete Phase II - Post-Excavation Sampling - Submit Interim Report	8	11
Complete Phase III - Revise RSE - Submit Final Report	3	14

*From Approval of Work Plan By U.S. EPA

6.0 QUALITY ASSURANCE PLAN

This removal action will be conducted in accordance with the overall quality assurance program at the FEMP as described in the site Quality Assurance Plan. The Quality Assurance Plan is based on the criteria specified in ASME NQA-1, Federal EPA Guideline QAMS-005/80 and DOE Orders 5700.6 and 5400.1. Detailed requirements are implemented by the WEMCO Site Policies and Procedures Manual, FMPC-2054, by WEMCO Departmental procedures, and Topical Manuals. Sampling and analysis activities will be conducted consistent with the RI/FS QAPP. The U.S. EPA is in the process of reviewing a draft Sitewide Quality Assurance Project Plan (QAPjP) covering all sitewide sampling and analysis activities. Upon approval,

remaining sampling and analysis activities will be conducted consistent with the Sitewide QAPP.

7.0 HEALTH AND SAFETY PLAN

The removal action will be conducted in accordance with the provisions of the FEMP site-wide health and safety program (WMCO June 1990). Consistent with this program and 29 CFR 1910.120, a task specific health and safety plan will be prepared addressing the proposed work activities. The task specific Health and Safety Plan is currently in draft and will be revised to incorporate any changes resulting from the final approval of the work plan. A copy of the Health and Safety Plan will be finalized prior to field mobilization and will be made available to U.S. EPA upon request at that time. The Health and Safety Plan identifies, evaluates, and controls all safety and health hazards associated with this removal action. In addition, it provides for emergency response for hazardous operations.

APPENDIX I

REMOVAL SITE EVALUATION

Contaminated Soils Adjacent to Solid Waste Incinerator

at the Sewage Treatment Plant

Feed Materials Production Center
U. S. Department of Energy

October 1990

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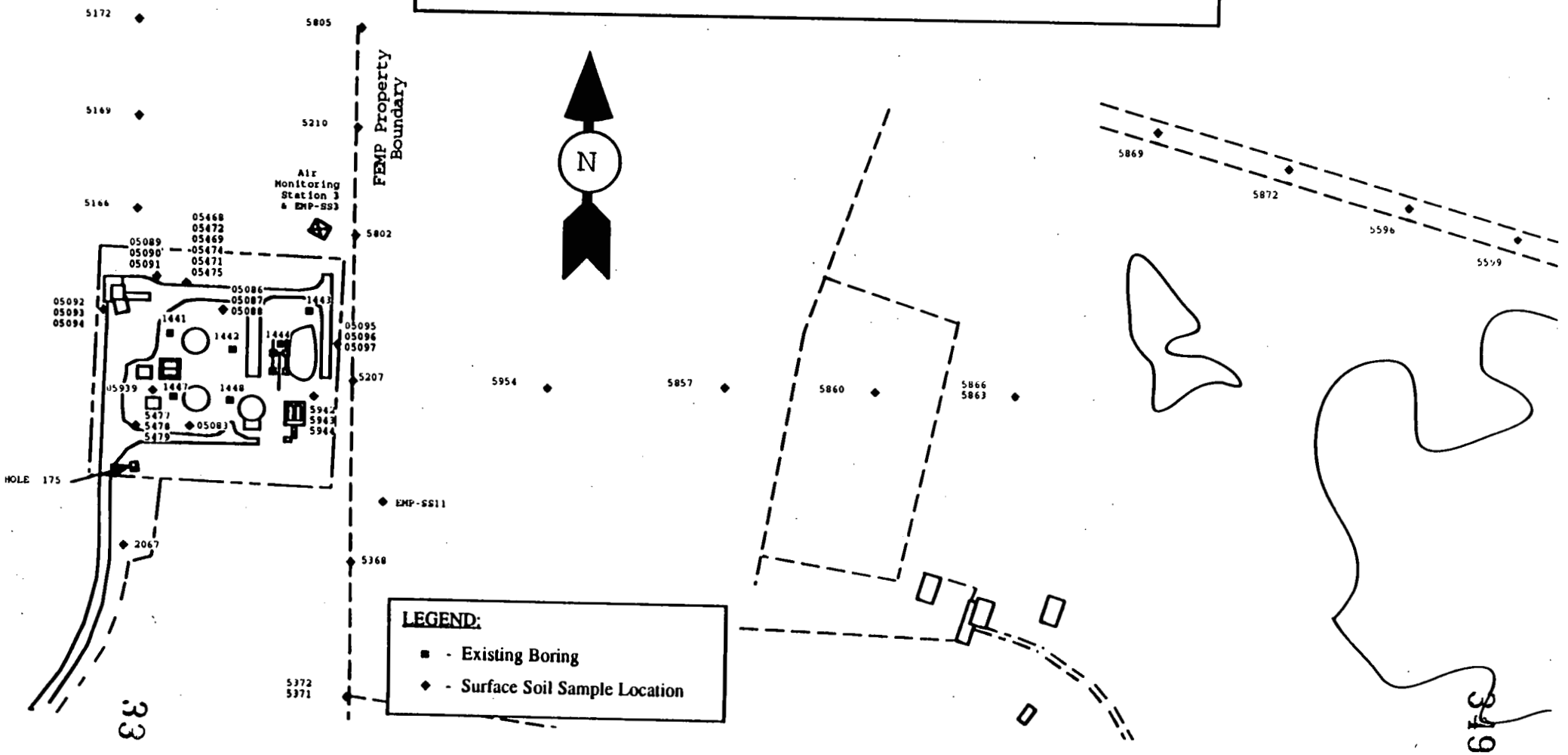
FIGURE 1: SOIL SAMPLE LOCATIONS



LEGEND:

- ■ - Existing Boring
- ◆ - Surface Soil Sample Location

1.0 INTRODUCTION

The solid waste incinerator is located in the northwest corner of the sewage treatment plant area at the Fernald Environmental Management Project (FEMP). This incinerator was operated from November of 1954 through December of 1979 at which time a new solid waste incinerator at Building 39 was placed into service. The incinerator at the sewage treatment plant was used to burn contaminated and uncontaminated burnable trash during its period of operation. Soil sampling results from the Remedial Investigation and Feasibility Study (RI/FS) indicate that concentrations of radionuclides in the soils adjacent to the solid waste incinerator are above background levels and exceed those observed in prior routine environmental sampling. The solid waste incinerator is located within the fenced area of the sewage treatment plant but the majority of the area with contaminated soils is located outside the sewage treatment plant's fenced boundary. The area outside the fence is primarily used for grazing dairy cattle (under a lease agreement with the DOE) owned by a neighboring farmer. Access to the sewage treatment plant is controlled by WEMCO personnel, however access for the grazing dairy cattle to the areas adjacent to the incinerator is uncontrolled. The solid waste incinerator at the sewage treatment plant has been identified as a "suspect area" to be addressed under the RI/FS Operable Unit 3. The RI/FS for Operable Unit 3, aimed at investigating the remedial alternatives in the Production Area and other identified suspect areas outside the Production Area, is presently underway.

This Removal Site Evaluation (RSE) has been completed by the DOE under authorities delegated by Executive Order 12580 under Section 104 of CERCLA and is consistent with Section 300.410 of the National Oil and Hazardous Substance Pollution Contingency Plan (NCP). This RSE addresses contaminated soils adjacent to the solid waste incinerator at the sewage treatment plant and has been completed to support the decision as to whether the present conditions warrant a removal action.

2.0 SOURCE TERM

Both the routine Environmental Monitoring Program (EMP) and the on-going Remedial Investigation/Feasibility Study (RI/FS) have shown evidence of contamination in the vicinity of the incinerator. It is possible that there was some contribution from other facilities at FMPC, but it is likely that most, if not all, of the activity is due to incinerator effluent.

2.1 Environmental Soil Sampling Data

There have been two environmental soil sampling locations in the area that are routinely monitored through the EMP. Sampling Point No. 3 is adjacent to the incinerator and on-site. Sampling Point No. 11 is nearby but off-site (see Figure 1). The U-238 soil concentrations from the 1984[1] and 1985[2] and 1989[3] environmental monitoring were:

Table 1. Historical Uranium-238 (pCi/g) Soil Concentrations
Incinerator Area

	No. 3	No. 11
1984 (Aug.)	68.5 ± 3.5 (2σ)	13.8 ± 0.7 (2σ)
(Dec.)	39.9 ± 1.7	19.3 ± 0.8
1984 (Resample)	2.8 ± 0.1	10.8 ± 0.5
1985	35.9 ± 14.5	14.2 ± 0.7
1989 0-5 cm	79 ± 13	Discontinued
5-10 cm	58 ± 9	Discontinued

These analyses were for elemental uranium, so the amounts of U-235 and U-234 present were not known. Subsequent RI/FS analyses showed the average activity ratios for typical soils with this range of uranium concentration to be

U-238	U-235	U-234
1.00	0.07	0.48

These ratios indicate a mixture of depleted and normal uranium. Very low concentrations of U-234 daughters indicate that this is not natural uranium. By 1984, the incinerator had not been used for five years.

2.2 Environmental Air Sampling Data

The nearest EMP environmental air sampling location (BS 3 and later AMS 3) east-northeast of the incinerator (see Figure 1) showed the highest concentrations among the FEMP-wide air sampling network. The source is likely to be a combination of entrained contaminated soil and effluent from other FEMP facilities. Airborne uranium concentrations for that air sampling location are summarized below

Table 2. Annual Average Airborne Uranium at BS 3 (AMS 3) (pCi/m^3)

	Average of Weekly Samples	Composite Analysis			
	U-238	U-234	U-235	U-238	Th-230
1984	1.36E-02				2.61E-04
1985	5.57E-03				7.64E-04
1988	3.59E-03				----
1989	7.1 E-04	2.0E-04	1.3E-05	3.6E-04	<1.1E-05

The committed effective dose equivalent from these concentrations is considerably less than the 10 mRem/yr NESHAPS criteria even with 100 percent occupancy. There are no residents at this location.

Annual composite sample analyses, for a number of radionuclides, showed either very low airborne concentrations or very low inhalation dose commitments relative to the uranium concentrations. Radionuclides that were identified included

Sr-90	U-236
Tc-99	Np-237
Ra-228	Pu-238
Th-228	Pu-239,40
Th-230	Pu-241
Th-232	Pu-242

No measurable radium-226 was noted at this air sampling location. A more complete summary of air sampling data is in Appendix A.

Exposure rates measured by environmental dosimeters, at the air sampling location, are not statistically different from the ambient background.

2.3 RI/FS Soil and Core Sampling Data

During the on-going RI/FS, soil samples and sub-surface core samples collected in this vicinity showed considerably higher concentrations than previously observed. Twelve of 24 samples were above background and six of these 12 exceeded guidance for unrestricted use[4]. The two highest samples, closest to the incinerator, showed 25,670 pCi/g and 2376 pCi/g of uranium-238. Figure 1 shows the sampling locations and Appendix B summarizes the radionuclide concentrations.

From inspection of the data, there are two distinct concentration distributions. The lower group remained below 10 pCi/g. This group included samples:

5368	5857
5371	5860
5372	5863
5596	5866
5599	5869
5854	5872

The U-238 concentration among these samples averaged:

$$5.1 \pm 2.7 \text{ pCi/g } (1\sigma)$$

On that basis, one can be 99.7 percent confident that concentrations exceeding 13.2 pCi/g (average plus 3σ) are above background. The 12 remaining samples exceeded that concentration.

Inspection of data for the 12 background samples showed that all other radionuclide analyses, in addition to uranium, yielded results at expected ambient background concentrations or near the analytical sensitivity. These 12 background soils were all at the greatest distances and/or not downwind compared to average meteorological conditions.

Highest soil concentrations were in the immediate vicinity of the incinerator and the apparent plume extended toward the northeast which is the most probable wind direction.

In order to assess the potential impacts, an average concentration of the various radionuclides was established for the 12 samples exceeding background.

Table 3. Average Soil Concentrations for 12 Samples Exceeding Background

Isotope	Detected in % of Samples	Average Concentration (pCi/g)	Range (pCi/g)
U-238	12/12	2391 ± 7361	13.6 - 25,670
U-235	11/11	172 ± 518	0.8 - 1730
U-234	11/11	1151 ± 3272	12.8 - 10,977
Ra-226	11/11	7.7 ± 17.2	0.9 - 57.4
Th-230	11/11	17.4 ± 33.5	1.8 - 102
Ra-228	10/11	2.9 ± 3.5	< 1.8 - 12.2
Th-228	11/11	2.7 ± 2.8	1.0 - 10.2
Th-232	11/11	2.9 ± 3.3	0.7 - 11.3
Tc-99	4/11	4.6 ± 6.6	< 0.9 - 14.4
Pu-239,40	1/11	1.1 (single value)	< 0.6 - 1.1
Sr-90	6/11	1.3 ± 0.7	< 0.5 - 2.3

The selected 12 samples were based upon elevated uranium concentrations. Many of the other radionuclide concentrations in those samples appeared to be at background levels but they were, none-the-less, included to create the average values above. A number of samples showed unique features. Sample 5095 had the highest radium-226 concentration (57.4 ± 1.2 pCi/g) which was also high when compared to uranium concentrations in that sample. In any case, no allowance or subtraction was made for background in the average concentrations given in Table 3. Note that there is a relatively large standard deviation associated with the averages. For uranium, the standard deviation gives roughly plus or minus 300 percent.

Data from a limited number of core samples from this area, down to twenty feet, suggest that contaminant particle sizes are small enough (or soluble enough) to penetrate into soil (e.g. gravel and sand). This will affect any decision to physically move the contaminated soil zones.

As part of the RI/FS, a limited set of data are available from core samples collected in this area. Sample locations 1441, 1442, 1447, and 1448 are in the central area within the fenced compound which includes the incinerator and the sewage treatment plant. They are approximately 100-300 ft from the incinerator. Location 1444 is farther east (approximately 350 ft from the incinerator) and 1443 is approximately 400 ft due east of the incinerator (see Figure 1). The profiles are shown in Table 4.

Table 4. Uranium-238 in Soil Core Samples (pCi/g)

Depth (ft)	1441	1442	1443	1444	1447	1448
(0 - 1.5)	58.3	42.1	45.6	12.9	19.9	41.9
(1.5-3.0)	6.67	6.14	11.9	224.4		3.9
(3.0-5.5)	15.3	4.60	33.0	69.6	4.9	
(5.5-10)					1.7	4.7
(10-15)	13.8		35.4	4.7 (2.3)		5.7 (2.0)
(15-20)	2.53		25.3			

Note: Parenthetical values are for second samples at same location

Relative to potential excavation, these data suggest that no removal is required in some areas. Other areas may require that a one foot layer be removed, but some removal beyond a three foot depth may be necessary.

Additional RI/FS sample results were obtained after calculations for this RSE were performed. These results are included in Appendix B in Table B.2. It is important to remember this data is included for informational purposes and wasn't used in the calculations of this RSE. These sample points are also shown on Figure 1.

2.4 Pathway Assessment

Because the contributing effluent is believed to be incinerator ash particles (and condensate nuclei) it is probable that the particle matrix containing the radionuclides is relatively insoluble. Leaching to subsurface water, and root uptake by vegetation, can be expected to be relatively low and slow. Inhaled particulate and deposition in bovine nasal turbinates should result in minimal transfer to the bloodstream and to other organs and milk. The fraction deposited in the lower respiratory system will depend upon the airborne particle size distribution. That fraction in the deep lung will be slowly cleared with longer term cumulative radiation dose to the lung and to the tracheobronchial lymph nodes.

Although RESRAD[6] transfer parameters are used later for dose estimates, cow soil ingestion should result in very little G.I. absorption. Similarly low vegetation (forage) root uptake is expected but RESRAD values are used.

3.0 EVALUATION OF THE MAGNITUDE OF THE POTENTIAL THREAT

The available data permits only a conservative assessment, that appears adequate to justify consideration of removal action(s). Several comparisons can be made by focusing on the RI/FS soil sample data. The net average concentration has been developed for the radionuclides in 12 soil samples. This is conservative since 50 percent of those samples were below cleanup guidance concentrations[4]. Other RI/FS samples from within the incinerator compound, and also those from the FMPC EMP, show considerably lower concentrations. More data will be required to characterize the magnitude and extent of the contamination.

One comparison is afforded by comparison to concentrations recommended for unrestricted use in the NRC Branch Technical Position[4]. These comparisons are summarized below.

Table 5. Average Soil Concentration Compared to NRC Guidance

Table 3 Averages		Guidance Concentration	
Th-232	2.9 pCi/g	10 pCi/g	Th-232 + daughters
Th-228	2.7		
Ra-228	2.9		
U-238	2391 pCi/g	35 pCi/g	Depleted U
U-238	2391 pCi/g	30 pCi/g	Enriched U
U-235	172		
U-234	1151		

As previously described, uranium isotopes most commonly exceed the cleanup criteria. Some locations have unique radionuclide mixtures. For example sample locations 5092 and 5095 have radium-226 concentrations which exceed the 5 pCi/g limit provided in 40 CFR 192[5] for uranium mill tailings sites. The FEMP is not a mill tailings site and current data do not permit averaging over 100m^2 for better comparison to 40CFR192.

Another comparison can be made through analyses for dose estimates and the associated risk. Appendix C shows the derivation of these estimates. The preponderance of the radiation doses accrue through:

External exposure	3.8 mRem/yr
Inhalation	4.7 mRem/yr
Milk ingestion	7.6 mRem/yr

These are modeled to human (not bovine) exposure (see Appendix C for model assumptions). The farmer visits the area for approximately one hour per week. A fraction of the dairy herd typically occupies the area about 10 hours per week, although calculations for the milk pathway assumed 100 percent occupancy.

No water pathways were analyzed since the scope of this evaluation is for the short term and also because geological characteristics are unknown.

The milk pathway is also analyzed in Appendix C however the potential contribution by that path was relatively low (7.6 mRem/yr) and a number of conservative assumptions were made. A fraction of the dairy herd ingests only a fraction of their total forage from the contaminated area. Through the routine EMP, monthly samples of milk production from Knollman's Dairy (adjacent to the FEMP) have only rarely shown concentrations in excess of the detection limit of 0.7 pCi/L. One outstanding analysis in 1989 showed 12.8 pCi/L[3]; one milk sample showed 1.35 pCi/L in 1983 and another showed 1.0 pci/l in 1988. These concentrations can be compared to the conservative model which yielded 198 pCi/L of U-238 and 95.7 pCi/L of U-234. Recent conversation with the farmer indicates

that most of the cows grazing this area are not milkers.

By adding effective dose equivalents and committed effective dose equivalents (to simplify this assessment), the total estimated annual dose equivalent is 16.1 mRem. Using the EPA risk estimate of 2×10^{-4} per person - Rem effective dose, the associated risk for a fatal cancer is

$$0.016 \text{ Rem} \times (2 \times 10^{-4}) = 3.2 \times 10^{-6}/\text{yr}$$

The risk for a fatal cancer for a 70 year lifetime estimate is less than 2.2×10^{-4} because weathering will reduce the available quantities of the radionuclides.

The dose estimate of 4.7 mRem/yr due to inhalation can be compared to the EPA NESHAPS limit of 10 mRem/yr (40 CFR 61.92). The milk ingestion estimated dose of 7.6 mRem/yr could be compared to the EPA limit of 4 mRem/yr (40 CFR 141.15) through drinking water. The total of 16 mRem/yr does not exceed DOE guidance of 100 mRem/yr in DOE Order 5400.5.

4.0 ASSESSMENT OF THE NEED FOR REMOVAL ACTION

Consistent with Section 40 CFR 300.410 of the National Contingency Plan, the Department of Energy (DOE) shall determine the appropriateness of a removal action. Eight factors to be considered in this determination are listed in 40 CFR 300.415 (b)(2). The following apply specifically to the above background concentrations of contaminants occurring in the soils adjacent to the solid waste incinerator at the sewage treatment plant.

40 CFR 300.415 (b)(2)(i)

Actual or potential exposure to nearby human populations, animals, or the food chain from hazardous substances or pollutants or contaminants.

40 CFR 300.415 (b)(2)(iv)

High levels of hazardous substances or pollutants or contaminants in soils largely at or near the surface, that may pose a threat of release.

40 CFR 300.415 (b)(2)(v)

Weather conditions that may cause hazardous substances or pollutants or contaminants to migrate or be released.

These factors are considered appropriate as a result of the concentrations of contaminants in the soils adjacent to the solid waste incinerator at the sewage treatment plant. Livestock grazing or significant storm events have a potential to cause these concentrations to migrate or be carried to areas which are uncontaminated.

5.0 APPROPRIATENESS OF A RESPONSE

If it is determined that a response action is appropriate due to both the level of contamination found in the soils adjacent to the solid waste incinerator at the sewage treatment plant and the potential of contaminant migration, a removal action may be required to address the existing situation.

If a planning period of less than six months exists prior to initiation of a response action, DOE will issue an Action Memorandum. The Action Memorandum will describe the selected response and provide supporting documentation for the decision.

If it is determined that there is a planning period greater than six months before a response is initiated, DOE will issue an Engineering Evaluation/Cost Analysis (EE/CA) Approval Memorandum. This memorandum is to be used to document the threat of public health and the environment and to evaluate viable alternative response actions. It will also serve as a decision document to be included in the Administrative Record.

Table A.1
Annual Average Airborne Radionuclide Concentrations at BS 3 (AMS 3)[1]
Composite Sample Analyses (pCi/m^3)

Radionuclide	1984	1985	1989
Sr-90	[3]	2.61 ± 0.65E-04	
Tc-99		6.38 ± 0.80E-03	1.60 ± 1.10E-04
Ra-226		<5.2E-05	<5.3E-07
Ra-228		3.91 ± 3.91E-05	<1.1E-05
Th-228	5.06 ± 0.24E-05	2.61 ± 0.52E-05	<1.1E-05
Th-230	7.46 ± 0.12E-04 (2.61E-04)[2]	2.61 ± 0.52E-04 (7.64E-04)[2]	<1.1E-05
Th-232	2.77 ± 0.24E-05	1.44 ± 0.26E-05	<1.1E-05
Np-237	1.07 ± 0.06E-05	<1.3E-06	
Pu-238	2.65 ± 0.7E-06	2.0 ± 0.8E-06	3.0 ± 2.1E-06
Pu-239,240	5.54 ± 0.6E-05	1.25 ± 0.2E-05	4.9 ± 0.03E-07
Pu-240			1.3 ± 0.01E-07
Pu-241		6.06 ± 0.16E-05	2.0 ± 0.06E-06
Pu-242			7.1 ± 0.2E-05
U-234			2.0 ± 0.7E-04
U-235			1.3 ± 0.04E-05
U-236			8.4 ± 1.3E-06
U-238	1.36 ± 1.30E-02	5.57E-03	3.60 ± 0.01E-04 (7.1E-04)[2]
Gross Beta	6.38 ± 1.28E-02	2.64 ± 1.13E-02	2.65E-02

(1) Designation of BS 3 was changed to AMS 3 at reorganization of the Air Sampling Network.

(2) Parenthetical concentrations represent averages of individual analyses during that year.

(3) Blank fields indicated that the specific analysis was not performed.

APPENDIX B

Table B.1
RI/FS Soil Sample Results (pCi/g)

Sample Number	U-238	U-235	U-234	Ra-226	Th-230	Ra-228	Th-228	Th-232
5083	13.6±2.7			*				
5086	259±18	16.4±2.4	341±27	1.8±0.3	3.9±0.8	2.5±0.5	1.8±0.5	2.3±0.6
5089	2376±114	123±8	1010±60	5.52±0.51	31.4±4.8	5.13±0.85	3.6±0.8	3.8±0.8
5092	25670±1281	1730±87	10977±549	19.4±	58.9±8.7	12.2±2.6	10.2±1.7	11.3±1.9
5095	187±13	13.4±2.3	165±12	57.4±1.2	102±15	<1.8	5.1±1.0	6.6±1.3
5166	51.0±8.1	4.5±1.0	50.4±8.0	1.3±0.2	2.8±0.7	1.3±0.3	1.6±0.4	1.5±0.4
5169	16.7±2.9	0.9±0.4	16.7±2.9	1.1±0.1	3.0±0.7	1.3±0.3	1.4±0.4	1.2±0.4
5172	16.5±2.7	0.8±0.3	15.5±2.5	1.2±0.2	2.2±0.6	1.3±0.3	1.4±0.4	1.3±0.4
5207	13.5±2.1	0.8±0.3	12.8±2.1	2.5±0.4	6.5±1.2	1.2±0.3	1.0±0.3	1.1±0.3
5210	46.3±6.6	3.6±0.8	34.8±5.0	0.9±0.1	1.8±0.6	1.6±0.3	1.0±0.4	0.7±0.3
5368	4.4±0.6	0.7±0.2	4.4±0.6	1.1±0.2	2.6±0.4	0.8±0.2	1.6±0.3	1.3±0.3
5371	4.2±0.6	<0.6	3.8±0.5	0.9±0.1	1.9±0.3	0.8±0.2	1.1±0.2	1.2±0.2
5372	9.6±1.4	<0.6	8.4±1.2	0.9±0.1	2.2±0.4	<0.7	1.4±0.3	1.2±0.2
5596	2.2±0.4	<0.6	2.2±0.4		*			
5599	2.3±0.4	<0.6	2.0±0.4					
5802	21.6±2.7	1.0±0.2	19.0±2.4	1.2±0.4	3.0±0.4	1.1±0.2	1.2±0.2	1.1±0.2
5805	23.5±2.9	1.4±0.3	22.9±2.8	0.9±0.1	3.0±1.5	1.1±0.2	1.8±1.1	1.2±0.9

* Blank fields indicate data was not available.

Table B.1 (Cont.)
RI/FS Soil Sample Results (pCi/g)

Sample Number	U-238	U-235	U-234
5854	5.0±0.7	<0.6	4.6±0.6
5857	7.2±1.0		*
5860	4.0±0.7	<0.6	3.5±0.6
5863	9.2±1.6	1.3±0.4	8.9±1.5
5866	7.8±1.2	0.6±0.2	7.3±1.2
5869	3.2±0.6	<0.6	3.0±0.6
5872	1.8±0.3	<0.6	1.6±0.3

* Blank fields indicates that data was not available.

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Table B.1 (Cont.)
RI/FS Soil Sample Results (pCi/g)

Sample Number	Tc-99	Pu-239,40	Sr-90
5083			
5086	<9	<0.6	<0.5
5089	<9	<0.6	0.8±0.2
5092	14.4±1.8	1.1±0.4	1.9±0.3
5095	<9	<0.6	<0.5
5166	<2.8	<0.6	<0.5
5169	<2.5	<0.6	0.8±0.2
5172	<2.8	<0.6	0.6±0.2
5207	2.1±1.0	<0.6	<0.5
5210	<9	<0.6	1.6±0.3
5368	<0.9	<0.6	<0.5
5371	<0.9	<0.6	<0.5
5372	<0.9	<0.6	<0.5
5596		*	
5599			
5802	1.0±0.4	<0.6	<0.5
5805	0.9±0.4	<0.6	2.3±0.4

* Blank fields indicate that data was not available.

Table B.2
RI/FS Surface Soil Sample Results (pCi/g)

Data presented by sample number and depth in inches. NA indicates not analyzed; a number preceded by a "less than" (<) symbol indicates that the compound was not present above the detection limit of the analytical instrument.

Radiological Parameters	05087 2 - 4	05088 4 - 7	05090 2 - 4	05091 4 - 6	05093 2 - 4	05094 4 - 6	05096 2 - 4	05097 4 - 6
Cs-137	1.1	0.4	0.7	0.3	0.6	<0.4	<0.6	<0.4
Pu-238	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6
Pu-239/240	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6
Ra-226	1.6	1.4	6.4	1.9	<1.3	2.1	188.0	67.0
Ra-228	2.2	1.7	3.9	1.9	5.7	6.9	<2.6	<1.7
Sr-90	1.8	0.8	0.5	1.0	1.2	0.7	0.6	<0.5
Tc-99	1.0	<0.9	4.3	2.0	12.8	6.1	<0.9	<0.9
Th-228	2.0	1.4	3.3	1.5	10.2	3.4	1.3	1.3
Th-230	7.7	4.9	28.7	7.0	50.5	8.3	325.0	87.0
Th-232	2.2	1.4	3.9	1.4	14.0	4.8	2.2	1.5
U-234	228.0	116.0	2913.0	543.0	7591.0	2160.0	312.0	98.0
U-235/236	15.4	6.9	98.0	26.4	372.0	82.0	14.9	3.5
U-238	186.0	98.0	3100.0	560.0	7771.0	2006.0	328.0	101.0

Table B.2 (cont.)
RI/FS Surface Soil Sample Results (pCi/g)

Data presented by sample number and depth in inches. NA indicates not analyzed; a number preceded by a "less than" (<) symbol indicates that the compound was not present above the detection limit of the analytical instrument.

Radiological Parameters	05468 0 - 6	05469 (dup.)	05471 6 - 12	05472 (dup)	05474 12 -18	05475 (dup)	05477 0 - 6	05478 6 -12
Cs-137	0.3	0.3	0.3	0.2	0.3	<0.2	0.7	<0.2
Pu-238	1.4	1.5	2.4	<0.6	<0.6	1.7	<0.6	<0.6
Pu-239/240	<0.6	<0.6	<0.6	<0.6	<0.6	<0.6	1.5	<0.6
Ra-226	4.9	4.3	3.0	2.7	1.4	3.4	2.4	2.1
Ra-228	18.0	16.9	8.5	7.2	1.2	4.3	1.0	0.8
Sr-90	1.8	1.4	0.7	<0.5	0.7	1.2	0.6	<0.5
Tc-99	13.0	9.1	7.4	4.3	1.2	4.4	228.0	56.9
Th-228	17.6	24.8	8.2	5.7	2.0	6.4	1.1	1.3
Th-230	755.0	806.0	460.0	359.0	11.3	385.0	8.2	5.2
Th-232	30.8	61.9	12.0	6.1	1.8	7.8	2.9	0.9
U-234	1644.0	1110.0	960.0	602.0	345.0	660.0	1627.0	808.0
U-235/236	134.0	95.3	54.9	37.8	17.6	38.6	143.0	41.5
U-238	1941.0	1445.0	1124.0	771.0	389.0	720.0	1477.0	790.0

Table B.2 (cont.)
RI/FS Surface Soil Sample Results (pCi/g)

Data presented by sample number and depth in inches. NA indicates not analyzed; a number preceded by a "less than" (<) symbol indicates that the compound was not present above the detection limit of the analytical instrument.

Radiological Parameters	05479 12-18	05939 0 - 6	05942 0 - 6	05943 6 - 12	05944 12 -18
Cs-137	<0.2	<0.2	0.6	<0.2	<0.2
Pu-238	<0.6	<0.6	<0.6	<0.6	<0.6
Pu-239/240	<0.6	<0.6	<0.6	<0.6	<0.6
Ra-226	2.2	1.2	2.3	0.9	1.0
Ra-228	1.1	0.6	1.1	0.8	1.3
Sr-90	<0.5	1.1	1.0	0.8	3.9
Tc-99	44.2	1.5	3.5	<0.9	<0.9
Th-228	1.2	0.7	1.1	0.8	1.1
Th-230	4.4	1.8	5.5	1.5	2.2
Th-232	1.0	0.6	0.8	0.9	1.2
U-234	516.0	3.7	44.8	NA	NA
U-235/236	23.3	<0.6	1.9	NA	NA
U-238	500.0	4.1	42.5	NA	NA

APPENDIX C

C-1

Dose Estimate and Risk Assessment

The following represent a limited assessment of three potential radiation dose paths using the method given in RESRAD[6]. Those paths are external ground radiation, inhalation through resuspension of contaminated soil, and the indirect path to milk.

External Ground Radiation

The assumptions made for this path are:

1. Soil bulk density is 1.8 g/cm^3
2. The potential receptor occupancy and shielding factor is 0.006 (one hour per week)
3. The lateral extent and shape of the surface contamination is extensive and uniform relative to uniform contamination
4. Contamination is uniform in depth to several inches for gamma ray absorption
5. There is no cover attenuating gamma rays
6. The effective dose equivalent conversion factors given in Table B.1 of Reference 6 are used

U-238 (Incl. Th-234, Pa-234m)

$2391\ pCi/g \times 1.8\ g/cm^3 = 4303.8\ pCi/cm^3$

$4303.8\ pCi/cm^3 \times 6.97 \times 10^{-2}\ mRem/yr/pCi/cm^3 \times 0.006$

$= 1.8\ mRem/yr$

U-235 (Incl. Th-231)

$172\ pCi/g \times 1.8\ g/cm^3 \times 4.90 \times 10^{-1}\ mRem/yr/pCi/cm^3 \times 0.006$

$= 0.9\ mRem/yr$

U-234

$1151\ pCi/g \times 1.8\ g/cm^3 \times 6.97 \times 10^{-4}\ mRem/yr/pCi/cm^3 \times 0.006$

$= 0.009\ mRem/yr$

Ra-226

7.7 pCi/g x 1.8 g/cm^3 x 8.56 mRem/yr/pCi/cm^3 x 0.006

= 0.71 mRem/yr

(Th-230 Contribution is negligible)

Ra-228

2.9 pCi/g x 1.8 g/cm^3 x 4.51 mRem/yr/pCi/cm^3 x 0.006

= 0.14 mRem/yr

Th-228 (Incl. 7 daughters)

2.7 pCi/g x 1.8 g/cm^3 x 7.36 mRem/yr/pCi/cm^3 x 0.006

= .22 mRem/yr

(Th-232, Tc-99, Pu-239,40 and Sr-90 contributions are negligible)

Total External Dose Rate

3.8 mRem/yr

Inhalation Pathway

The assumptions made for this path are:

1. Average airborne mass loading is 2 x 10^{-4} g/m^3
2. The potential receptor occupancy factor is 0.006
3. There is no cover over the contaminated soil
4. The distribution of contaminant in the soil extends over a large area and is at depth relative to the surface entrained.
5. Annual air intake is 8400 m^3/yr
6. The committed effective dose equivalent conversion factors given in Reference 6 Table C.1 are used.

U-238 (Class Y)

2391 pCi/g x 2 x 10^{-4} g/m^3 x 0.006 x 8400 m^3/yr x 1.2 x 10^{-1} mRem/pCi

= 2.9 mRem/yr

U-235 (Class Y)

172 pCi/g x 2 x 10^{-4} g/m^3 x 0.006 x 8400 m^3/yr x 1.2 x 10^{-1} mRem/pCi

= 0.21 mRem/yr

U-234 (Class Y)

1151 pCi/g x 1.0 x 10^{-2} g/yr x 1.3 x 10^{-1} mRem/pCi

= 1.5 mRem/yr

(Ra-226 contribution is negligible)

Th-230 (Class Y)

17.4 pCi/g x 1.0 x 10^{-2} g/yr x 0.26 mRem/pCi

= 0.05 mRem/yr

(Ra-228 contribution is negligible)

Th-228 (Class Y)

2.7 pCi/g x 1.0 x 10^{-2} g/yr x 0.31 mRem/pCi

= 0.008 mRem/yr

(Tc-99, Pu-239,40 and Sr-90 contributions are negligible)

Total Inhalation Dose

4.7 mRem/yr

Milk Pathway

The assumptions made for this path are

1. Vegetative to soil transfer factors are used from Table D.3 of RESRAD[6]
2. Cow forage consumption is 55 kg/day[6]
3. Milk transfer factors are from Table D.4 of RESRAD[6]
4. Human milk consumption is 92 L/yr
5. Ingestion dose factors are from Table D.1 of RESRAD[6]
6. A milking cow ingests all forage for one year from the contaminated area.

Isotope	Soil Concentration	Transfer Factor	Forage Concentration
U-238	2391 pCi/g	2.5×10^{-3}	6.0 pCi/g
U-235	172	2.5×10^{-3}	0.4
U-234	1151	2.5×10^{-3}	2.9
Ra-226	7.7	1.4×10^{-3}	0.01
Th-230	17.4	4.2×10^{-3}	0.07
Ra-228	2.9	1.4×10^{-3}	0.004
Th-228	2.7	4.2×10^{-3}	0.01
Th-232	2.9	4.2×10^{-3}	0.01
Tc-99	4.6	2.5×10^{-1}	1.2
Pu-239,40	1.1	2.5×10^{-4}	3×10^{-4}
Sr-90	1.3	2.0×10^{-1}	0.26

Milk concentrations are estimated given the assumptions in reference 6 including daily cow forage consumption of 55 kg and the milk transfer factors in Table D.4.

Isotope	Forage Concentration		Forage Consumption		Milk Transfer		Milk Concentration
U-238	6 pCi/g	x	5.5×10^4 g/day	x	6×10^{-4} day/L	=	198 pCi/L
U-235	0.4		5.5×10^4		6×10^{-4}		13.2
U-234	2.9		5.5×10^4		6×10^{-4}		95.7
Ra-226	0.01		5.5×10^4		2×10^{-4}		0.1
Th-230	0.07		5.5×10^4		2.5×10^{-6}		0.01
Ra-228	4×10^{-3}		5.5×10^4		2.0×10^{-4}		0.04
Th-228	0.01		5.5×10^4		2.5×10^{-6}		1×10^{-3}
Th-232	0.01		5.5×10^4		2.5×10^{-6}		1×10^{-3}
Tc-99	1.2		5.5×10^4		1.2×10^{-2}		792
Po-239,40	3×10^{-4}		5.5×10^4		2.5×10^{-8}		4×10^{-7}
Sr-90	0.26		5.5×10^4		1.5×10^{-3}		21.5

An annual dose estimate is made based upon ingestion of 92 L of milk per year and using the ingestion dose factors given in Table D.1 of reference 6.

Isotope	Milk Concentration		Milk Consumption		Dose Conversion		Annual Dose
U-238	198 pCi/L	x	92 L/yr	x	2.5×10^{-4} mRem/pCi	=	4.6 mRem/yr
U-235	13.2		92		2.5×10^{-4}		0.3
U-234	95.7		92		2.6×10^{-4}		2.3
Ra-226	0.1		92		1.1×10^{-3}		0.01
Th-230	0.01		92		5.3×10^{-4}		5×10^{-4}
Ra-228	0.04		92		1.2×10^{-3}		4×10^{-3}
Th-228	1×10^{-3}		92		7.5×10^{-4}		7×10^{-5}
Th-232	0.01		92		2.8×10^{-3}		3×10^{-3}
Tc-99	792		92		1.3×10^{-6}		0.09
Pu-239,40	4×10^{-7}		92		4.3×10^{-3}		2×10^{-7}
Sr-90	21.5		92		1.4×10^{-4}		0.3
					Total Milk Dose		7.6 mRem/yr

References

1. Facemire, C.F., Jones, D.L. and Keys, R.W., Feed Materials Production Center Environmental Monitoring Annual Report for 1984, NLO-2028, July 15, 1985, NLO, Inc., Cincinnati, Ohio. (p. 20, Fig. 7) (p. 58, Table 6)

2. Aas, C.A., Jones, D.L., and Keys, R.W., Feed Materials Production Center Environmental Monitoring Report for 1985, FMPC-2047, May 30, 1986, Westinghouse Materials Co. of Ohio, Cincinnati, Ohio.

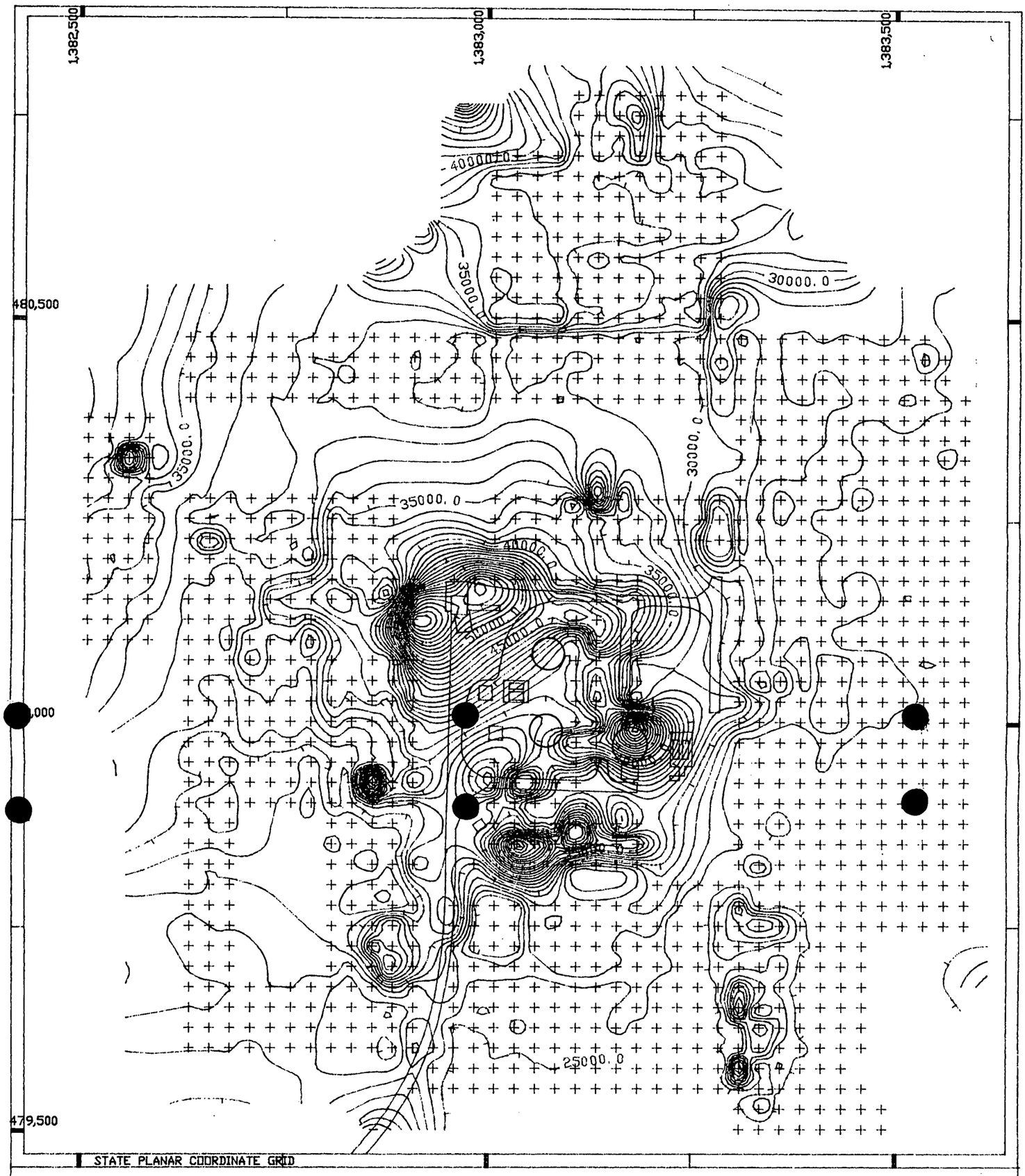
3. Feed Materials Production Center Site Environmental Report for Calendar Year 1989, FMPC-2200, June 25, 1990, Westinghouse Materials Co. of Ohio, Cincinnati, Ohio.

4. U.S. Nuclear Regulatory Commission Branch Technical Position "Disposal or On-Site Storage of Thorium and Uranium Wastes from Past Operations" Fed. Reg. Vol. 46, No. 205, Friday, October 23, 1981, p. 52061 f.

5. 40 CFR 192 Uranium and Thorium Mill Tailings, Subpart B. Standards for Cleanup of Land and Buildings Contaminated with Residual Radioactive Material from Inactive Uranium Processing Sites.

6. Yu, C., et. al. A Manual for Implementing Residual Radioactive Materials Guidelines, DOE/CH/8901, Argonne National Laboratory, June, 1989.

APPENDIX II



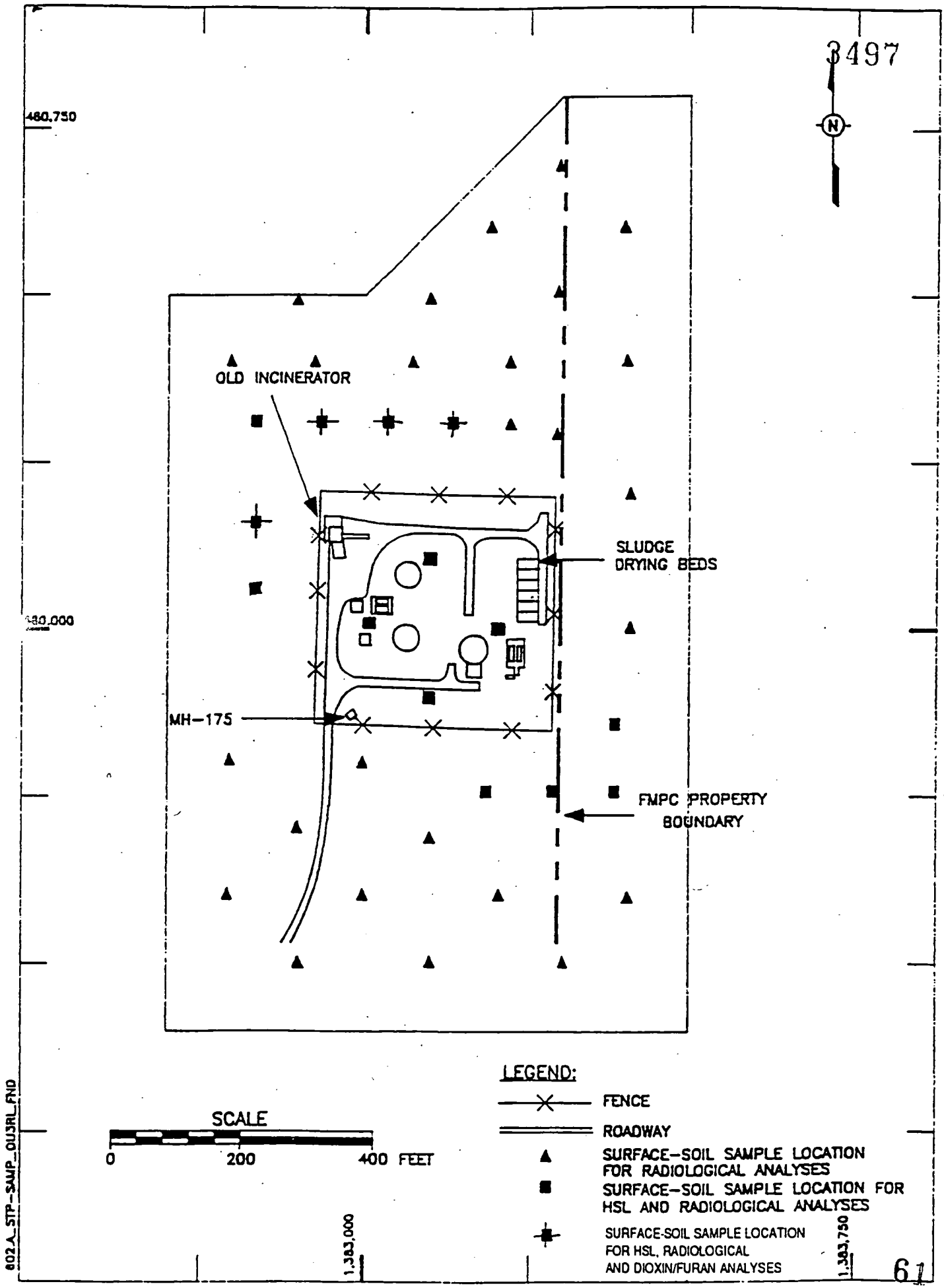
NORMALIZED SPA 3 WALKOVERS
SEWAGE TREATMENT PLANT
REPORTING UNITS = 2 MINUTE COUNTS
CONTOURS INTERVAL = 1000 cpm

SCALE (feet)

0 130 260

SPA_STP.PLT FMPCRAD.DWG 1-14-92

APPENDIX III



SEWAGE TREATMENT PLANT – SURFACE SAMPLING LOCATIONS

APPENDIX IV

SMS-EXM-91-018
REV - 01

SITE MEDIA SAMPLING EXTRACTION METHODOLOGY

FOR OBTAINING SOIL SAMPLES FROM METAL OVERPACK CONTAINERS

PREPARED BY: Lawrence Love Jr. (TECHNOLOGIST) DATE: 10/16/91

REVIEWED BY: Bus Henderson (TECHNOLOGIST) DATE: 10/16/91

APPROVED BY: Thomas E Bullage (MANAGER) DATE: 10-16-91

1.0 PURPOSE

To establish a methodology for extracting representative soil samples from metal overpack containers. A minimum of two composite samples per container will be required per this methodology for characterization purposes (RCRA constituents & radiological analysis).

2.0 DEFINITIONS

2.1 SMS - Site Media Sampling

2.2 Sample Location - An area designated as a location to obtain a representative sample of soil. For this methodology, a sample area shall be approximately 2'(length) x 1'(width) x 4'(depth), and will be further divided into individual aliquots as follows 0" to 12", 12" to 24", 24" to 36", and 36" to 48".

2.3 Composite Sample - The combining of two or more aliquots of a sample location.

3.0 REQUIRED EQUIPMENT

Stainless Steel Auger	Blue Ice
Auger Extension	Cooler
T - Handle	Deionized Water
Stainless Steel Scoop	Decon Sprayer
Stainless Steel Buckets	Decon Cleaning Solution
Stainless Steel Pans	Bailer Brushes

3.0 REQUIRED EQUIPMENT

Secondary Containment Systems
Nitrile Gloves
Measuring Tape
Glass Sample Containers
Field Logbook
Polyethylene sheeting
Thermometer
Custody Tape
Sample Labels

4.0 PERSONAL PROTECTIVE EQUIPMENT (PPE)

NOTE - The following list of PPE is a minimal requirement for entry into a drum or container where the possibility of unknown hazardous constituents exist.

PPE

- Company Issued Clothing
  - Coveralls
  - Safety Shoes
- Safety Glasses
- Saranex or Tyvek Anti - Contamination Clothing
- Full Face Respirator (air purifying dual purpose cartridge)
- Nitrile Gloves

SMS prior to the sampling of metal overpack containers will contact FEMP Radiological Safety, and FEMP Industrial Hygiene so that preliminary surveys concerning worker safety and health can be performed. SMS sampling activities will not be instituted until (1) an FEMP Safety Work Permit and (2) an FEMP Radiation Safety Work Permit, have been issued, and signed by the SMS lead technician, and all SMS sample team personnel.

This list will be upgraded or downgraded according to instructions relative to the required permits. All permits will be obtained by the SMS lead technician or his designate, and will be present at all times during the sample extraction.

3.0 METHODOLOGY

3.1 Contact the cognizant facility owner for the approximate storage location of the white metal overpack container.

3.2 Arrange with facility owner for a time and a place that sampling of the contents (soil) of the white metal overpack container can be initiated.

3.3 Arrange with facility owner to provide a fork truck driver for the moving of container to a designated sampling area.

3.4 Arrange with facility owner to provide a chemical operator to open white metal overpack containers.

3.5 Contact FEMP Radiological Safety and Industrial Hygiene to conduct a real time field survey of exposed contents.

3.6 The SMS sampling team shall prepare a decontamination area as per EM-CS-001 Environmental Monitoring On-Site Media Sampling, and a sample preparation area as follows.

SAMPLE PREPARATION AREA - All samples will be prepared on clean polyethylene sheeting. Place 4 stainless steel pans on the polyethylene sheeting approximately 1" contiguous to each other (for composite sampling). Obtain the appropriate sample containers, proper closures, sample labels, cooler, blue ice, thermometer, an approved custody tape, and a clean stainless steel scoop.

3.7 Obtain the appropriate PPE as prescribed in the completed permits.

3.8 The SMS sampling team, wearing the appropriate PPE shall obtain a measuring tape, and divide the white overpack container into 16 grid sections. **(Each individual grid shall be approximately 2'(length) x 1'(width) x 4' (depth).**

3.9 The individual grids shall be numbered 1 thru 16.

3.10 Randomly select any of the 16 grid locations as a starting point. The second grid shall be selected using a random number generator table as in attachment a **(Instructions for use of the random number table generator is also provided).**

3.11 After all grids locations have been selected, note grid selections into the field logbook.

3.12 The SMS technician wearing the appropriate PPE shall obtain a stainless steel auger, with T handle, and an appropriate extension.

3.13 Assemble the auger, appropriate extension, and T handle.

3.14 Locate the center most point of the selected grid and advance the auger into the grid location.

3.15 Deposit all extracted material into a stainless steel bucket.

3.16 Continue advancing the auger assembly into that grid location until all material (soil) has been extracted 0" to 48".

3.17 Deposit all extracted material into a clean stainless steel bucket.

3.18 When the extraction of all material (soil) has been completed, the SMS technician shall transport the stainless steel bucket to the sample preparation area.

3.19 Using a stainless steel scoop, equally distribute the extracted material between the four stainless steel pans placed side by side in the sample preparation area.

3.20 After all material has been equally distributed, prepare the composite sample.

3.21 Using a clean stainless steel scoop, thoroughly mix all material in each individual pan.

3.22 After the mixing of material in each pan is completed, transfer the material in pan #1 to pan #2, and the material in pan #3 to pan #4.

3.23 Using the stainless steel scoop, thoroughly mix the material now located in pan #2 and pan #4.

3.24 After the mixing of material in pan #2 and pan #4 is completed, transfer the material in pan #2 to pan #4.

3.25 Using the stainless steel scoop, again, thoroughly mix the material now located in pan #4.

3.26 After the mixing of the material now located in pan #4 is completed, transfer the composited material to the proper sample containers.

3.27 Properly close all containers using the appropriate closure and seal with an approved custody tape.

3.28 Properly label all containers, and log the appropriate sample identification into the field logbook.

3.29 Place all sample containers requiring cooling to 4°C into a cooler utilizing blue ice as the cooling media.

3.30 Log all samples onto the appropriate SMS chain of custody/analytical request form.

3.31 Decontaminate all sampling equipment as per procedure EM-CS-001 Environmental Monitoring, On-Site Media Sampling.

3.32 Obtain a decontaminated auger system and move to the next selected grid.

3.33 Repeat 3.12 through 3.31.

3.34 Decontaminate all sampling equipment as per procedure EM-CS-001 Environmental Monitoring, On-Site Media Sampling.

3.35 Transport and relinquish all samples under chain of custody to the FEMP analytical Laboratory.

ATTACHMENT A

RANDOM NUMBER TABLE

03	47	43	73	86	36	96	47	36	61	46	98	63	71	62
97	74	24	67	62	42	81	14	57	20	42	53	32	37	32
16	76	62	27	66	56	50	26	71	07	32	90	79	78	53
12	56	85	99	26	96	96	68	27	31	05	03	72	93	15
55	59	56	35	64	38	54	82	46	22	31	62	43	09	90
16	22	77	94	39	49	54	43	54	82	17	37	93	23	78
84	42	17	53	31	57	24	55	06	88	77	04	74	47	67
63	01	63	78	59	16	95	55	67	19	98	10	50	71	75
33	21	12	34	29	78	64	56	07	82	52	42	07	44	38
57	60	86	32	44	09	47	27	96	54	49	17	46	09	62
18	18	07	92	46	44	17	16	58	09	79	83	86	19	62
26	62	38	97	75	84	16	07	44	99	83	11	46	32	24
23	42	40	64	74	82	97	77	77	81	07	45	32	14	08
52	36	28	19	95	50	92	26	11	97	00	56	76	31	38
37	85	94	35	12	83	39	50	08	30	42	34	07	96	88
70	29	17	12	13	40	33	20	38	26	13	89	51	03	74
56	62	18	37	35	96	83	50	87	75	97	12	25	93	47
99	49	57	22	77	88	42	95	45	72	16	64	36	16	00
16	08	15	04	72	33	27	14	34	09	45	59	34	68	49
31	16	93	32	43	50	27	89	87	19	20	15	37	00	49

INSTRUCTIONS

1. Starting at any corner, the SMS technician shall measure the length of the metal overpack container. Mark the length of the container in 2' increments, and the width of the container in 1' increments. When completed this will total 16 grids approximately 2' length by 1' width by 4' depth.

2. Starting from any corner number each grid one through 16.

3. Select any grid as a starting point.

4. When using the random number table generator and this methodology, only use and/or select grid numbers 1 through 16 for sample point locations.

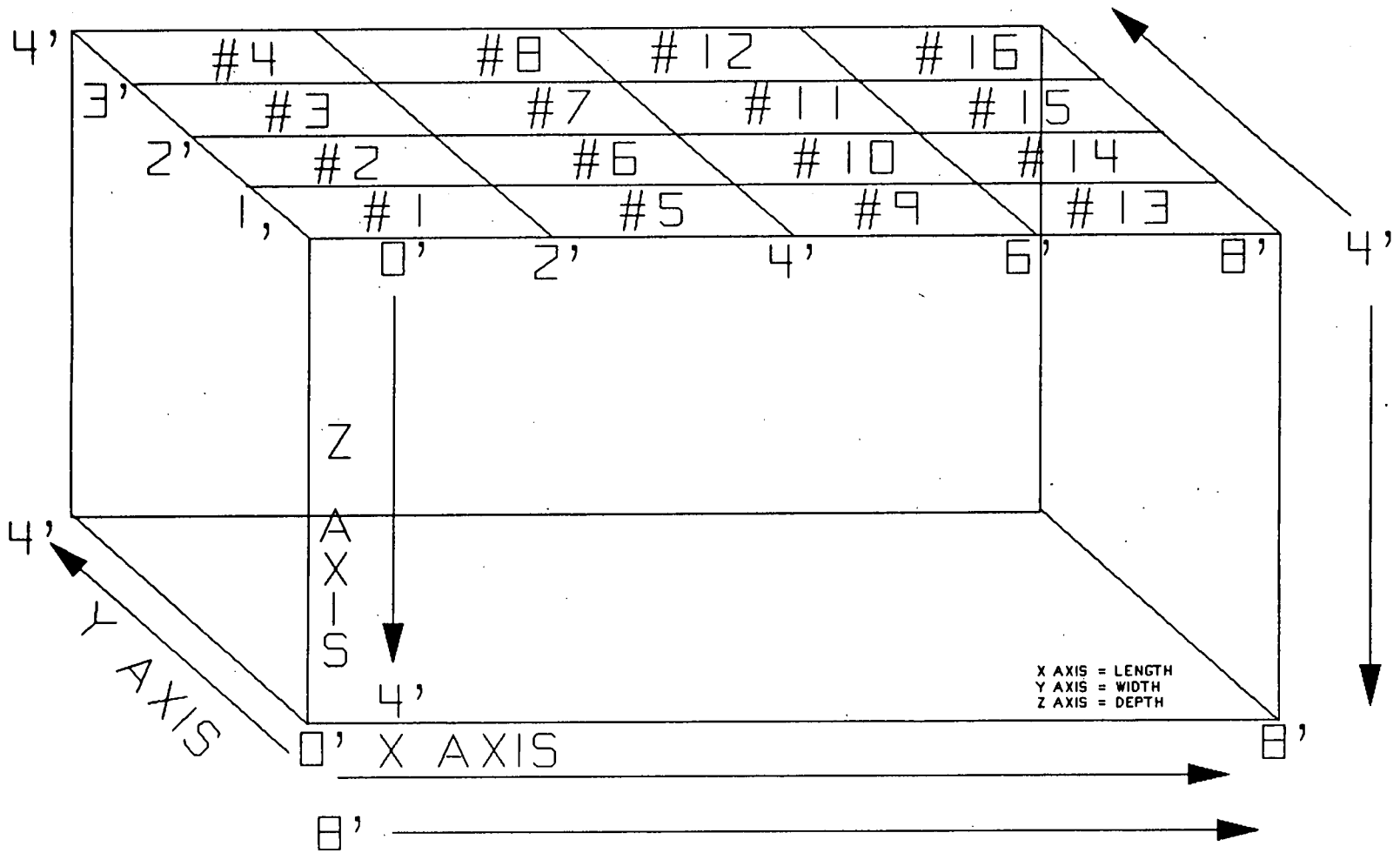
EXAMPLE

I have selected as a starting point grid #12. Using the random number table on page 6 of 7, the #12 is located in the first column.

> **NOTE: The sample grids selected for the sampling of metal overpack containers and this methodology contains a total of 16 grids. The numbers to be utilized when randomly selecting the sample locations must fall between 1 and 16. Never select the same number twice.**

Moving down this column of numbers, the next logical sample point is grid number 16. The locations selected for this sample extraction then are grids #12 and #16. If I wanted another location I would proceed down this column and find that the next available number in this column is again 16. This number can not be selected again since it has been used previously. No other numbers are available that fall between 1 and 16 in this column. Moving to the right of the first column to the second column and straight down I find that the next available number is 1. This would be the next available sample location if desired. If more sample locations were needed the user would continue in this method until all sample location were selected. **(See diagram of overpack container pg. 8 of 8)**

Metal Overpack Container



ENVIRONEMTAL CHARACTERIZATION & SURVEILLANCE

TECHNICIAN REVIEW AND SIGN OFF SHEET

EM-EXM-91-Ø18 Soil Sampling - Metal Overpack Containers

1. Andrew M. Russell
2. Pat Brennan
3. [illegible]
4. William A Meyer
5. [illegible]
6. [illegible]
7. Linda Mad
8. John R. Delt
9. Don Brown
10.
11.
12.
13.
14.
15.
16.

APPENDIX V

1.0 SCOPE

This procedure establishes a method for quantitatively determining the concentrations of Uranium (U) and Thorium (Th) in soil by energy dispersive X-ray fluorescence (EDXRF) spectroscopy. The method was developed and the system calibrated using ground soils from the plant site. Any soil samples that differ from this general composition (i.e. fertilizer or a sample of mostly rock) would have to be calibrated separately for X-ray analysis or analyzed by some other method. The procedure uses the Kevex Instruments Least Squares Method of quantitation, that incorporates a least squares linear calibration curve. The results are on a dry sample basis (initial drying and grinding of soils is done by the Sample Receiving Lab).

2.0 PRINCIPLE

2.1 The purpose of the procedure is to determine the concentration of Uranium (U) and Thorium (Th) in soil samples by Energy Dispersive X-Ray Fluorescence (EDXRF) spectroscopy.

2.2 Energy dispersive X-ray fluorescence (EDXRF) spectroscopy uses high energy X-rays to excite electrons out of the inner atomic orbitals of the atoms which make up the sample. As an atom restabilizes, outer electrons drop into the inner orbitals and emit an X-ray whose energy is characteristic of the atom and that particular orbital transition. The emitted X-rays are detected via a solid state detector [Si(Li)] and counted in discrete energy channels on a multi-channel analyzer (MCA) to form an energy spectrum. The spectrum is then processed in a number of ways: 1) to remove escape peaks; 2) to remove the background and X-ray source spectra; 3) to identify the peaks present; and 4) to measure the peak intensities by fitting a Gaussian distribution to the peaks and deconvoluting any overlapping peaks. For this procedure, a program is written to acquire a spectrum using a Molybdenum (Mo) secondary target to excite the sample, collect spectra for a multiple number of samples between 1 and 24, and simultaneously process the data without operator attendance. The program runs for approximately 40 minutes per sample. The soil samples are ground into a fine powder using a tungsten carbide mixing vial and then pressed into a pellet in an aluminum sample holding cap.

3.0 SPECIAL APPARATUS

3.1 KEVEX ANALYST 8000 instrument, that is a computer-based multichannel analyzer (MCA) with a keyboard, display terminal, and printer.

3.2 KEVEX 770 SPECTROMETER, that houses the X-ray tube, secondary target turret, sample chamber and carousel, high voltage power supply, and detector.

3.0 SPECIAL APPARATUS (cont.)

3.3 SPEX MODEL 8000 mixer/mill, or equivalent.

3.4 Tungsten carbide grinding/mixing vial set, that includes two tungsten carbide mixing balls for use with the model 8000 mixer/mill.

3.5 31 mm pre-flared aluminum sample holding caps.

3.6 31 mm die press set with two tungsten carbide pellets.

3.7 Manual hydraulic press capable of 25 ton load.

3.8 KEVEX 316 stainless steel (ss) standard.

3.9 KEVEX 2036 Cu-Al standard sample.

3.10 Electronic pot adjusting screwdriver.

3.11 Vacuum desiccator.

3.12 400 grit sandpaper.

4.0 REAGENTS

Not applicable.

5.0 SAFETY PRECAUTIONS

5.1 Safety glasses shall be worn in the laboratories at all times.

5.2 Do not have the X-rays on without having the front and side panels of the 770 spectrometer in place. The X-rays will not turn on unless the sample chamber lid is closed.

5.3 The X-ray tube port and the detector window are covered with a thin fragile foil of beryllium under vacuum. Beryllium metal is highly toxic if inhaled and is easily airborne if broken. If the beryllium is broken, special safety precautions shall be followed to remove the fragments. In such a case, consult the manager of Industrial Hygiene and Safety prior to removing the beryllium fragments. A respirator shall be worn.

5.4 The model 8000 mixer/mill shall be operated in a fume hood to prevent the fine particulate dust from being dispersed into the air. Sample loss could occur if the vial is not sealed or secured properly in the mixer/mill.

D - MATERIAL REVISED, ADDED, OR DELETED.

5.0 SAFETY PRECAUTIONS (cont.)

5.5 When cleaning out sample mixing vials with coarse ground glass, the resulting finely powdered glass shall be removed while working in an exhaust hood. Finely powdered glass is a health hazard if inhaled; it is known to cause silicosis if exposed to on a regular basis.

5.6 All personnel shall receive operator certification from their manager before they can operate the X-ray instrument. Certification shall include that the operator and the immediate supervisor have read the instruction manuals and Section 2 of the Radiation Control Manual.

5.7 When operating the manual hydraulic press, always bend at the knees rather than risk back injury.

5.8 When using compressed air inside the hood, latex gloves shall be worn to prevent air bubbles from entering the blood stream through the skin.

6.0 SAMPLE PREPARATION

PROCEDURE	COMMENTS
6.1 Grinding the Sample	Items necessary for sample grinding for 6.1 are shown in Figure 1.
6.1.1 Measure approximately 15 milliliters (mL) of a dried soil sample using a spatula and a 15 mL porcelain crucible.	Polyethylene scoops, which are available, or any other type of measuring device can also be used to measure out about 15 mL.
6.1.2 Pour the soil into a clean tungsten carbide mixing vial with two 7/16 in. tungsten carbide balls and screw on the top end of the vial.	If the vial is not clean, refer to Step 6.5 on how to clean the vial. Each end of the vial must have a corprene gasket. Try to keep the gaskets aligned in the vial end caps when screwing them together.
6.1.2.1 Write the sample lab number and the mixing vial number onto the Sample Preparation Log Sheet (See Figure 2).	

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.1.3 Place the vial inside the mixer/mill (Special Apparatus 3.3) and tighten down the holding clamp to secure it.	The Mixer/Mill device is shown in Figure 3.
6.1.4 Close the lid of the mixer/mill, set the timer for 25 minutes, turn on the fume hood, then press the start button.	
6.1.5 After 25 minutes has elapsed and the mixer/mill has stopped, open the mixer/mill lid and remove the vial.	
6.1.6 Remove one end cap of the vial. Use a pair of clean tweezers to remove the mixing balls from the powdered sample. Place the balls in the removed end cap.	
6.2 Assembling the Die Press	The die press set, shown in Figure 4, consists of a cylindrical die body, base, plunger, two tungsten carbide pellets (polished on one face), and a knock-out cup.
6.2.1 To assemble and/or prepare the 31 mm die set for making a pressed sample, wipe off with KEMWIPES, or equivalent, any residue from the following die pieces: the die base; inside of the die body; the plunger; and the pellets.	

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.2.1.1 If the inside of the die body, the sides of the pellets, or the plunger have material deposits on them that will not wipe off, use a piece of 400 grit sandpaper to remove the material and then wipe them off.	Deposits of aluminum from the sample cup and sample material often build up on the areas noted.
6.2.2 Place the die body on the base and gently slide a tungsten carbide pellet into the body with the polished side facing up.	Do not force the pellet into the body. It could damage the pellet.
6.2.3 Place an aluminum sample holding cap into the die and press it down flush with the pellet.	The holding caps are preflared to fit flush with the sides of the die. The cap can be pressed down flush easily using the round face of a piece of 1 inch diameter bar stock metal.
6.2.4 Using a spatula, transfer approximately one-half of the powdered sample into the die from the mixing vial.	Do not attempt to scrape off portions of the sample from the vial wall. Doing so could contaminate the sample with tungsten from the vial and possibly with the spatula material.
6.2.4.1 Level the material off in the die with the spatula.	Do not tamp soil down by tapping the die body onto the table more than a couple of times. This may cause some particle size segregation.
6.2.4.2 Write the die press number onto the Sample Preparation Log Sheet (See Figure 1).	
6.2.4.3 Refer to Steps 6.4 and 6.5 for storage of the remaining sample and cleaning of the mixing vial.	

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.2.5 Gently slide the other pellet into the die body with the polished side facing down.	Do not force the pellet into the body.
6.2.6 Place the plunger on the top of the pellet (with the beveled end facing up) and allow its weight to push the pellet down or gently push it down so as not to force the air out quickly and cause dust to emerge around the edges.	
6.3 Pressing the Sample	The sample is pressed into a solid pellet using the manual hydraulic press. The manual hydraulic press is shown in Figure 5.
6.3.1 If the platen of the hydraulic press is not low enough for the die to fit between it and the top of the press, release the pressure by turning the pressure release knob counter clockwise and pressing down on the platen until there is room for the die.	The platen is the flat plate piece of the press which moves upward when pressure is applied. The pressure is most easily released when the knob is turned approximately 3/4 of a turn.
6.3.1.1 Close the pressure release by turning the pressure release knob clockwise.	
6.3.2 Center the die on the platen of the press.	
6.3.3 Pump the lever until the pressure gauge reaches 25 tons (full scale).	**WARNING: A PIPE MAY BE USED AS AN EXTENSION OF THE LEVER HANDLE TO GET BETTER LEVERAGE, BUT MAY CAUSE THE PRESS TO TIP.**

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.3.4 Allow the die to remain at full pressure (25 tons) for a minimum of 1 minute.	
6.4.5 Gradually turn the pressure release knob counter clockwise to release the pressure.	The platen will slowly lower. **CAUTION: AVOID RELEASING THE PRESSURE QUICKLY AS IS NOTED BY A HISSING SOUND. THIS MAY CAUSE THE SAMPLE PELLET TO FLAKE OR FRACTURE.**
6.3.6 Remove the die press and gently invert the die, while holding onto the plunger, and set it onto a table, resting on the plunger.	
6.3.7 Close the pressure release knob on the press by turning it clockwise so the platen doesn't continue to drop.	
6.3.8 Twist off the base of the die and place the knock-out cup in its place.	
6.3.9 Remove the die press from the table while holding onto the plunger and place it back on the hydraulic press in its inverted position.	
6.3.10 If the platen of the press is not low enough for the die to fit, release the pressure by turning the pressure release knob counter clockwise and pressing down on the platen.	

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.3.10.1 Close the pressure release by turning the knob clockwise.	
6.3.11 Center the die on the platen of the press.	
6.3.12 Hold the die body so that it does not drop when the pellet is released from the die body and pump the lever of the press until the pellet and sample is loose.	
6.3.13 Gently remove the die from the press while holding the plunger from the bottom so the plunger does not fall and slide the die set onto the table.	
6.3.14 Lift the knock-out cup and remove the pressed soil pellet from between the two tungsten carbide pellets.	
6.3.14.1 Lay the pressed sample on the table with the exposed soil side up.	**CAUTION: HANDLE THE SAMPLE WITH CARE. THE SAMPLE IS FRAGILE.**
6.3.15 Using a black marker, write the lab number on the aluminum back side of the pellet.	
6.3.16 If the center area of the pellet soil surface has a crack, then a new pellet should be pressed out for that sample. If so, return to Step 6.2, and use the remaining half of the powdered soil in the mixing vial.	

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.3.17 Write the analyst initials and the date and time the sample was prepared on the Sample Preparation Log form (See Figure 1).	
6.3.18 If the sample is not going to be analyzed within the next 24 hours, place the pellet in a vacuum desiccator.	A vacuum desiccator is shown in Figure 6.
6.4 Storing the Sample Powder	
6.4.1 Use a spatula to remove the remaining ground portion of the sample from the vial and place it in a 13 mL plastic vial.	
6.4.1.1 Label the vial using a black marker with the lab number and "fine grd".	
6.4.1.2 After filling the vial, discard any excess soil in an appropriate mixed contaminated waste container.	The waste container shall be labeled "Finely Ground Glass and Soil Residue from X-Ray Lab Vial Cleaning Process".
6.4.1.3 Store the vial for a period of 90.	**NOTE**: The vial is stored for a recheck of the sample analysis if required.
6.4.2 Use a spatula to transfer a portion of the original soil sample into another 13 mL plastic vial.	
6.4.2.1 Label the vial using a black marker with the lab number and "course grd".	

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.4.2.2 Store the vial for a period of 90 days.	**NOTE**: The vial is stored for a recheck of the sample analysis if required.
6.5 Cleaning the Vial	The tungsten carbide grinding vial is cleaned with a water rinse and then by grinding coarse glass. The grinding vial consists of a cylindrical body, two end caps, two corprene gaskets and two tungsten carbide balls.
6.5.1 Remove both end caps of the vial.	
6.5.2 Using tap water, rinse off all pieces except the corprene gaskets.	Dry the pieces immediately, as directe below. Pieces rust very easily after rinsing.
6.5.3 Blow off loose material from the gaskets using compressed air inside an operating exhaust hood.	**CAUTION**: **WHEN USING COMPRESSED AIR TO BLOW OFF LOOSE MATERIAL, ENSURE THE HOOD SASH IS PULLED DOWN TO THE POSITION OF YOUR ELBOWS.**
6.5.4 Wipe dry and then blow dry the rinsed metal pieces using a compressed air line inside an operating exhaust hood.	
6.5.5 If the corprene gaskets appear worn, replace with new ones.	As a general rule, the gaskets can be replaced every ten samples.
6.5.6 Assemble the vial and place the two balls inside and fill it half full with coarse ground glass.	Keep the gaskets aligned in the end caps when screwing them together to prevent wear.

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.5.6.1 To obtain coarse ground glass, crush 4 oz. glass sample jars in a stainless steel beaker.	
6.5.6.2 Perform the crushing using a hammer or similar tool with a piece of plexiglass over the remaining part of the beaker.	**WARNING: THIS OPERATION IS TO BE DONE IN A HOOD WITH THE SASH DOWN AS MUCH AS POSSIBLE AND THE ANALYST SHALL WEAR HEAVY RUBBER GLOVES, RUBBER APRON AND A SAFETY SHIELD.**
6.5.7 Place the vial inside the mixer/mill (Special Apparatus 3.4) and tighten down the holding clamp.	
6.5.8 Close the lid of the mixer/mill, set the timer for 5 minutes, turn on the fume hood, and then press the start button.	
6.5.9 After 5 minutes has elapsed and the mixer/mill has stopped, open the lid and remove the vial.	
6.5.10 In an operating exhaust hood, remove one end cap of the vial and use a pair of clean tweezers to remove the mixing balls from the powdered glass.	Two separate pairs of tweezers should be designated for removing the balls, one from the powdered glass and one from the powdered soil.
6.5.10.1 Place removed mixing balls into removed end cap.	**WARNING: DO NOT INHALE FINELY POWDERED GLASS. HANDLING OF FINELY POWDERED GLASS SHALL BE IN AN EXHAUSTED HOOD. REFER TO ITEM 5.5.**

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.5.11 Discard the powdered glass into a waste container and disassemble the vial again.	The waste container should be labelled "Finely Ground Glass and Soil Residue from X-Ray Lab Vial Cleaning Process".
6.5.11.1 The waste containers shall be collected in 5 gallon metal pails labeled "Fine Ground Glass". The pails shall then be placed in 50 gallon drums used for laboratory glass waste removal.	
6.5.12 Wipe the pieces with a paper wipe.	Do not use sandpaper on the inside of the vial. It could cause the next sample to be contaminated with tungsten.
6.5.13 Blow off loose material from all pieces using compressed air inside an operating exhaust hood.	
6.5.14 Assemble the vial for reuse or storage.	
6.6 Rinse clean the crucible, spatulas, and tweezers, and dry them for reuse.	
6.7 Proceed to Step 6.2 if other soil samples need to be prepared.	

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7.0 PROCEDURE

PROCEDURE	COMMENTS
7.1 Booting the Software	The procedure for loading software into computer memory is known as "booting up" or simply "booting". If at any time in the procedure the keyboard appears to be "locked up", that is, not responding, try pressing the CTRL key and the A simultaneously to return to an * prompt. If that does not work,perform the "Warm Start" procedure listed in Figure 7.
7.1.1 If the system is booted with the most recent copy of the "TOOLBOX MASTER COPY" in disk drive 0, hold the CTRL key down and type C twice until a dot prompt appears on the CRT display. Then go to Step 7.1.2.4.	The software is already booted up if the disk is in the drive, the green light is on, and an * prompt or dot prompt is on the screen.
7.1.2 If the system is not booted, proceed with the following steps:	
7.1.2.1 Hold the labeled end of the program disk labeled "TOOLBOX MASTER COPY" with the side label toward the keyboard and insert it into disk drive 0.	Refer to Figure 8. Use the most recent copy of the disk. Copies are stored as the disks are filled with data.
7.1.2.2 Turn the load lever control clockwise. The green disk drive indicator light should flash for a period and then remain on.	**CAUTION: DO NOT REMOVE A DISK BY TURNING THE LOAD LEVER COUNTER CLOCKWISE AND PULLING ON THE DISK. THIS COULD DAMAGE THE DISK AND THE DISK DRIVE. TO REMOVE THE DISK, FOLLOW STEPS 7.13 THROUGH 7.15.**

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.1.2.3 To turn on the display terminal or cathode ray-tube (CRT), push the button in the front on the lower right corner of the CRT.	The red light emitting diode (LED) to the left of the button should light up when the CRT comes on.
7.1.2.4 Press the PROGRAM key on the keyboard.	Figure 9 shows the keyboard. The program key is one of the yellow "control" keys. A list of the program items will be displayed on the display terminal.
7.1.2.5 Select "System Setup" from the displayed menu by pressing the keys 5 and RETURN on the keyboard.	A list of the system setup items will be displayed on the CRT.
7.1.2.6 Select "Boot DEC Computer" from the display menu by pressing the keys 5 and RETURN on the keyboard.	A booting message will appear on the CRT: "BOOT>".
7.1.2.7 Press the RETURN key on the keyboard.	A title page and then the RT-11 system menu will appear on the CRT.
7.1.2.8 To run the toolbox language, select item X (Run XRF 8000 Toolbox) by pressing the RETURN key on the keyboard, since item X is the default selection.	This runs a program called "set up". The computer recalls a system file 0:KEV770.3.0 and a library file 0:MATRIX.1.0, and then displays the toolbox prompt * on the display terminal.
7.1.3 To recall the library file that is currently being used for soil sample data storage, type: LIB/ASS 0=SOILF.KVX as an example and press the RETURN key on the keyboard.	A new library file is created whenever one is filled with data; therefore, the name to be recalled will change. The format of the file names used is SOIL_.KVX with a letter. The files are created in alphabetical succession.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.1.3.1 To determine if there is enough space available in the library, type LIB/FREE and press the RETURN key on the keyboard.	A message similar to the following will appear: Unit #0 XX files XXX free blocks Room for XXX spectra
7.1.3.2 If 2 times the total number of samples to be run is greater than the number of spectra that there is room for, then a new library file needs to be created.	
7.1.4 If a new library file needs to be created, proceed as follows; otherwise, go to Step 7.2.	
7.1.4.1 Press the CTRL and C keys on the keyboard simultaneously to return to the RT-11 system menu.	
7.1.4.2 Type U and press the RETURN key on the keyboard to enter into the utility software.	The utilities menu will appear on the screen.
7.1.4.3 Type 10 and press the RETURN key to select menu item 10, "Create a large Kevex Library".	The computer will respond with an information message and ask the question, "... Do you wish to continue?"
7.1.4.4 Type Y and press the RETURN key on the keyboard	The computer will respond with "... Size of library in blocks, (default=1600).

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7.0 PROCEDURE (cont.)

PROCEDURE		COMMENTS
7.1.4.5	Press the RETURN key to select the default size of 1600 blocks.	The following message appears, "A library name may be up to 6 letters long". The default extension of .KVX will be added.
7.1.4.6	Enter the next successive file name, for example SOILG and press the RETURN key on the keyboard.	The computer will pause while creating the library and then return to the utilities menu. If the disk is full the following message will appear: "?PIP-F-Device full DL:SOILG.KVX WARNING! Some error has occurred in creating the new library. There may not be enough space for the request without squeezing the disk. NO NEW LIBRARY HAS BEEN CREATED. (Returning to main menu...please wait.)"
7.1.4.6.1	If the computer indicates that the disk is full, see Figure 10 for the procedure on how to: format and initialize a new disk; copy the working disk over to the new disk; and delete old library files from the working disk.	
7.1.4.7	Press the RETURN key on the keyboard to exit the utilities menu.	The computer will return to the RT-11 system menu.
7.1.4.8	Press the RETURN key again to select menu item X, (Run XRF 8000 Toolbox).	The computer will recall the files as in Step 7.1.2.8 and display the * prompt.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.1.4.9 Assign the new library to virtual memory group zero by typing LIB/ASS 0= followed by the new file name with the .KVX suffix and then press the RETURN key on the keyboard.	
7.1.4.10 Assign the old library file to virtual memo group one by typing LIB/ASS 1= followed by the old file name with the .KVX suffix and then press the RETURN key on the keyboard.	
7.1.4.11 To copy the necessary files from the old library file to the new library file, enter in the following commands which cause the computer to query the files of each filetype. Respond by entering Y and pressing the RETURN key on the keyboard for those files listed in the comments and respond by pressing the RETURN key (which selects the default=N) for those files not listed.	Press the CTRL and A keys on the keyboard simultaneously at any time during the query process to return to the * prompt.
7.1.4.11.1 Type LIB/WIND/COPY *..*=1:*..* and press the RETURN key on the keyboard.	filetype: files: window BA-NORM-M01 BA-MOD-M01 DEC-MO
7.1.4.11.2 Type LIB/SPEC/COPY *..*=1:*MO..* and press the RETURN key on the keyboard.	spectrum BLK-MO

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7.0 PROCEDURE (cont.)

PROCEDURE		COMMENTS	
7.1.4.11.3	Type LIB/MAT/COPY *..*=1:*..* and press the RETURN key on the keyboard.	matrix	SOIL-MO
7.1.4.11.4	Type LIB/PRO/COPY *..*=1:*..* and press the RETURN key on the keyboard.	procedures	NAME SOIL-MO MO DEC-PRO PB ID UH UL
7.1.4.11.5	Type LIB/LS/COPY *..*=1:*..* and press the RETURN key on the keyboard.	least squares	SOIL-U-LOW SOIL-U-HIGH THLSRECAL
7.1.4.11.6	Type LIB/SYS/DEL *..* and press the RETURN key on the keyboard.	system	KEV770 KEV700 KEVNONE
7.1.4.11.7	Type LIB/SYS/COPY *..*=1:*..* and press the RETURN key on the keyboard.	system	KEV770
7.1.4.11.8	Type LIB/MAT/DEL *..* and press the RETURN key on the keyboard.	matrix	MATRIX

7.2 Data Acquisition

7.2.1 Turn off the X-rays and raise the lid on the sample chamber.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.2.1.1 To turn off the X-rays first reduce the voltage and current to at/or below 10 kV and 0.2 mA by following the steps below and then press the X-RAY OFF button on the KEVEX 770 Spectrometer.	The X-RAY OFF button is shown in Figure 11.
7.2.1.1.1 Press the kV key on the keyboard.	The KV key is one of the "user XRF" keys shown in Figure 9. If the voltage level is greater than 20 kilovolts (kV) from the desired setting of 10 kV, then it must be stepped down in increments not greater than 20 kV. Wait 30 seconds after each level is reached.
7.2.1.1.2 Enter the desired voltage level (10 kV) and then press the RETURN key on the keyboard.	
7.2.1.1.3 Press the KV key on the keyboard again to exit the displayed menu.	
7.2.1.1.4 Press the MA key on the keyboard.	The MA key is one of the "user XRF" keys shown in Figure 9. If the current level is greater than 0.5 milliampheres (mA) from the desired setting of 0.2 mA, then it must be stepped down in increments not greater than 0.5 mA. Wait 30 seconds after each level is reached.
7.2.1.1.5 Enter the desired current level (0.2 mA) and then press the RETURN key on the keyboard.	

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.2.1.1.6 Press the MA key on the keyboard again to exit the displayed menu.	
7.2.1.2 If the lid to the sample chamber does not open, the vacuum may have been left on. If so, perform the following steps to release the vacuum:	
7.2.1.2.1 Press the ATMOS key on the keyboard.	The atmos key is one of the "user XRF" keys shown in Figure 9. This displays the atmosphere selection table.
7.2.1.2.2 Type 1 to select item 1 (air) and press the RETURN key on the keyboard.	This selects an air atmosphere for the chamber.
7.2.1.2.3 Wait two minutes before proceeding.	
7.2.1.2.4 Type 5 and press the RETURN key on the keyboard.	This turns the vacuum pump off.
7.2.1.2.5 Press the ATMOS key on the keyboard to exit.	
7.2.2 If the energy calibration has not been reference checked within two weeks (check logbook), place the 316 stainless steel and 2036 Cu-Al standards on the wheel and proceed to Step 8.0.	

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.2.3 A molybdenum (Mo) secondary target is required in the secondary target position number 3, which is the standard position for the silver (Ag) secondary target. If the Mo secondary target is not installed, perform the following steps:	The secondary target turret, with its plate cover in place, is shown in Figure 12.
7.2.3.1 Remove the sample wheel by turning the center knurled knob until loose then lift.	
7.2.3.2 Remove the plate cover to the secondary target turret by unscrewing the two hex bolts which hold it in place.	
7.2.3.3 Remove the aluminum top of the turret by unscrewing the four phillips screws and turning until the top can be lifted off with the beveled edge below the X-ray tube collimator.	
7.2.3.4 Remove the bottom portion of the turret and push out from the underside the secondary target occupying position number 3.	
7.2.3.5 Place the Mo secondary target in position number 3.	
7.2.3.6 Replace the top and bottom portions of the turret and plate cover in the manner in which they were removed.	The screw holes in the turret are offset so that the top and bottom portions align in only one way with the holes in the base plate.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.2.3.7 Replace the 24-position sample wheel and turn the center knob a few turns to tighten.	Do not over tighten the center knob.
7.2.4 There are two detector collimators used on the instrument, one made from Ag and one from tantalum (Ta). The Ag detector collimator is needed for this analysis. If it is not installed, follow the instructions below for changing the detector collimator.	Figure 12 shows the detector collimator area with the sample wheel removed.
7.2.4.1 Remove the sample wheel by turning the center knurled knob until it is loose and lifting the wheel up.	
7.2.4.2 Press the FILTER TARGET key on the keyboard.	The filter/target key is one of the "user XRF" keys shown in Figure 9. This displays the "Filter/target Positions" table.
7.2.4.3 Press the SPACEBAR on the keyboard to move from the column labeled filter to the column labeled target.	
7.2.4.4 Select "Beam Dump" from the displayed menu by typing: 0 and then pressing the RETURN key on the keyboard.	This places the secondary target wheel in position so that the collimator can be removed.
7.2.4.5 Slide the detector collimator off of the detector snout slowly so as not to damage the beryllium window.	**CAUTION: THE BERYLLIUM WINDOW MATERIAL IS FRAGILE AND TOXIC.** See Safety Precaution 5.3.

7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.2.4.6 Carefully slide the other detector collimator over the detector snout.	
7.2.4.7 Place the unused collimator behind the X-ray tube (as shown in Figure 12).	The spare collimator can then be seen through the chamber window so as to quickly identify which collimator is in place.
7.2.4.8 Press the FILTER TARGET key on the keyboard to exit the displayed table.	
7.2.4.9 Replace the sample wheel and turn the center knob a few turns to tighten.	Do not over tighten the center knob.
7.2.5 If the 16-position sample wheel is in place in the chamber, remove it by turning the center knurled knob until it is loose and replace it with the 24 position sample wheel.	
7.2.6 Place the samples on the sample wheel with ID markings facing outward so the mark-ings can be read through the window.	One blank soil sample and one reference standard are to be run with every ten samples. One recycle control is to be run with every batch.
7.2.6.1 If there are any liquid samples in the chamber, remove them.	
7.2.6.2 Record sample position on the wheel on the Sample Position Form (See Figure 13).	Maintain the sample position form on file for reference.
7.2.7 Release the sample chamber lid lock near the hinge, close the lid, and turn on the X-rays by pressing the X-RAY ON button on the KEVEX 770 spectrometer.	The interlock light on the spectrometer should go off when the lid is closed. The X-ray on button and the interlock light are shown in Figure 11.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.2.8 Adjust the vertical count scale to 5000 counts per second (cps) by pressing the blue (up) and (down) arrow keys.	The vertical scale is given on the left hand side on the fourth line of the display.
7.2.9 Set the pulse rate as follows:	
7.2.9.1 Remove the KEVEX 8000 instrument panel under the keyboard by pulling outward.	This exposes the model 4460 pulse processor.
7.2.9.2 On the model 4460 pulse processor, push the RATE pushbutton to select a pulse rate of 2.	See Figure 14.
7.2.9.3 Replace the instrument panel.	
7.2.10 To set the voltage up, press the KV key on the keyboard.	The KV key is one of the "User XRF" keys shown in Figure 9.
7.2.10.1 Type 30 and then press the RETURN key on the keyboard.	This sets the voltage at 30 kV.
7.2.10.2 Press the KV key on the keyboard again to exit the displayed menu.	
7.2.11 To set the current, press the MA key on the keyboard.	The MA key is one of the "user XRF" keys shown in Figure 9.
7.2.11.1 Type 1.5 and then press the RETURN key on the keyboard.	
7.2.11.2 Press the MA key on the keyboard again to exit the displayed menu.	

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.2.12 Run the program which acquires and processes spectra for each sample as follows:	Figure 15 shows the program named SOIL-MO to be run. Figures 16, 17, and 18 show the subroutine programs called NAMES, MO, and DEC-PRO.
7.2.12.1 Type RUN SOIL-MO and press the RETURN key on the keyboard.	The comment statements in the first 15 lines of the program shown in Figure 15 will be displayed momentarily, and then the following statement will be displayed: ENTER FIRST SAMPLE POSITION.
7.2.12.2 Type the sample wheel position of the first sample to be analyzed and press the RETURN key on the keyboard.	The following statement will then be displayed: ENTER LAST SAMPLE POSITION.
7.2.12.3 Type the sample wheel position of the last sample to be analyzed and press the RETURN key on the keyboard.	The statement ENTER SAMPLE LABELS WHEN PROMPTED will be displayed, followed by SAMPLE NO. (first sample position) NAME.
7.2.12.4 Type the lab number of the sample in the sample wheel position stated by the computer and press the RETURN key on the keyboard.	

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.2.12.5 Repeat Step 7.2.12.4 until all the sample lab numbers have been entered.	The instrument will now operate unattended until the sample analyses are complete. The instrument will run for approximately 40 minutes per sample. Each sample will have a spectrum acquired upon it. The spectrum will be saved, printed, and then processed. A peak identification list will be printed. The background will be removed and the net integral peak intensities of U and Th will be determined, saved in results file, and printed out. When the program is completed, the word FINISHED will be displayed and the asterisk prompt will appear below it.
7.2.12.6 Confirm that the printer "on line" indicator is lit; if not, push the button.	
7.2.12.7 Check that the printer has enough paper for the results to be printed out. Three to four sheets are printed per sample. If there is not enough, put more paper in the printer.	
7.3 Quantifying the Data	The U and Th concentrations are calculated separately using the intensity data stored in the results file.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.3.1 For each sample, examine the printout which gives the net integral peak intensities (it is entitled GAUSSIAN DECONVOLUTION). If the chi-squared value is greater than 100, then the results are invalid. If so, examine the spectrum and ID list to determine why the peak fit was so poor.	The chi-squared value is a measure of the degree of peak fit to a Gaussian distribution curve. The chi-squared is measured throughout the deconvolution window region; from 7.780 kiloelectron-volts (KeV) to 15.340 KeV. The following elements are integrated in the deconvolution process: W; Pb; Th; Rb; U; Sr; and Y. Notify the lab supervisor to investigate the cause of the high chi-squared value.
7.3.1.1 The following instrumental errors are possible causes of high chi-squared values, and in these cases, the problem should be corrected and the samples rerun.	
7.3.1.1.1 Improperly set up excitation conditions such as the wrong secondary target.	
7.3.1.1.2 A window file missing, i.e.: not copied in Step 7.1.4.11.	

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.3.1.2 The following errors are possible causes of high chi-squared values, and in these cases, the sample cannot be run properly by this method and will have to be submitted for analysis by some other method.	
7.3.1.2.1 One of the interfering elements integrated is higher in concentration than normal and interferes in the integration of U and Th.	Soils with high Pb levels can interfere with the integration.
7.3.1.2.2 An element that is not integrated and not normally found in the spectrum interferes with the integration of U and Th.	
7.3.2 Refer to the printout that gives the U and Th peak intensities for each sample that was determined to be valid. Based on the following criteria, run the appropriate program to quantify the U and Th.	There are two calibration curves established for U, one at a low concentration range and one at a high concentration range. Upon initiating the proper program, the following message will appear: "Enter Sample Name []:".
7.3.2.1 If the U peak intensity is less than or equal to 49.76 counts/second then type RUN UL and press the RETURN key on the keyboard.	

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.3.2.2 If the U peak intensity is greater than 49.76 counts/second then type RUN UH and press the RETURN key on the keyboard.	
7.3.3 Enter the sample lab number and press the RETURN key on the keyboard.	The computer will generate two pages. The first page quantifying U and the second page quantifying Th. The following messages that the computer may generate should be ignored: "Warning [276] (U or Th) is "OUT" or absent in LS file": "Warning [194] Extrapolated value for (U or Th)".
7.3.4 Repeat Steps 7.3.2 to 7.3.3 to quantify each sample in which the chi-squared value was found to be acceptable.	
7.3.5 Assemble the data for each sample and staple together.	There are 5 data sheets for each sample.
7.3.6 To delete a second results file in the computer which contains a duplication of the results data, enter the following command which will cause the computer to query the results files with a version 2 suffix name. Respond to each file name query by entering Y and pressing the RETURN key on the keyboard to delete that file.	This is performed to conserve memory space in the library.
7.3.6.1 Type LIB/RESU/DEL*.2.0 and press the RETURN key on the keyboard.	

7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.4 Verifying the Reliability of the Data	Before the results can be reported, the results from the blanks and the reference standards must be checked.
7.4.1 Examine the results from the blank sample(s). If the results are above the minimum detection limits (MDL) for U, and/or Th, proceed as follows:	The reported minimum detection limits (MDL) are 11 ppm for U and 18 ppm for Th. The logic flow in verifying the reliability of the blank sample data is presented in the flowchart form in Figure 19.
7.4.1.1 Prepare and rerun the blank sample from the original container.	
7.4.1.1.1 If the results of the second trial are below the detection limits, prepare and rerun the blank a third time to determine which result is valid.	
7.4.1.1.2 If the results of the second trial are above the detection limits (similar in magnitude to the original result) then this indicates that cross contamination occurred in the preparation process performed by the Sample Receiving Lab (SRL). Notify the SRL to have them determine which unknown samples were prepared at the same time as the blank.	If decided that cross contamination may have occurred between samples of significant (measurable) magnitude, then those samples should be reprepared by Sample Receiving Lab and resubmitted for analysis.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.4.1.2 If the blank is found after the third trial to be below the detection limits, then rerun the original pressed pellet for that blank sample.	If the blank is verified by the third trial to be below the detection limit, then the initial results which were above the detection limits may be due to cross contamination in the X-Ray Lab sample preparation or to sample mix-up during the data acquisition.
7.4.1.2.1 If the results of the second trial on the original pellet are the same as the original result, this indicates either mismarking of the original blank sample with another sample or contamination occurred in the X-Ray Lab preparation. Inform the lab supervisor and make a note of the problem in the logbook. Also examine the sample preparation log sheet and the analysis results to see if a sample-to-blank mismarking may have occurred (See Step 7.4.1.3) or if cross contamination may have occurred from perhaps a high level sample to the blank (See Step 7.4.1.4).	

R - MATERIAL REVISED, ADDED, OR DELETED.

7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.4.1.2.2 If the results of the second trial are below the detection limit, this indicates a mix-up in the initial sample positioning or entering of sample names. Examine the other results and the sample position form to see where a mix-up may have occurred. If it is obvious where the mix-up occurred then rerun the necessary samples to confirm this, correct the data, and proceed to Step 7.4.2.	
7.4.1.3 If it is apparent from the data where a mismarking has occurred then prepare and rerun the necessary sample(s) to confirm this.	
7.4.1.3.1 If a mismarking is confirmed from the repeat data then report the correct data and mark the original data as a sample mix-up.	
7.4.1.3.2 If a mismarking is not confirmed from the repeat data then cross contamination or outside contamination must have occurred.	

7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.4.1.4 If it is not apparent from the data that mismarking has occurred then either cross contamination or outside contamination must have occurred. Examine the sample preparation log sheet and analysis results to see if sample carry over or cross contamin-ation may have occurred from perhaps a high level sample to the blank, i.e., from mixing vial or die set.	**NOTE**: High contamination level samples should have been screened with a Geiger-Mueller counter and marked as high by the Sample Receiving Lab. Precautions then should have been taken in their preparation to avoid sample carry over.
7.4.1.4.1 If there is evidence of possible sample carry over from a high level sample to other sample(s), rerun the samples which were possibly affected (including the blank). Mark the original results as invalid due to cross contamination and report the repeat results.	
7.4.1.4.2 If there is no evidence of sample carry over then the contamination is from an outside source which may not be limited to a few samples. The entire sample run is then invalid and should be reprepared and rerun.	

7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.4.2 Examine the results from the standard sample(s). Compare the results to the tabulated results of that standard from previous analyses and any analyses by Process Control Lab.	The logic flow in verifying the reliability of the reference standard data is presented in flowchart form in Figure 24. The tabulated reference standard data for the standards currently in use are kept in the room with the instrument.
7.4.2.1 If the results are within ± two times the standard deviation, then the data is considered acceptable and so proceed to Step 7.4.3.	
7.4.2.2 If the results are not in agreement within ± two times the standard deviation, from the mean then proceed as follows.	
7.4.2.3 Prepare and rerun the reference sample from the original container.	
7.4.2.3.1 If the second trial is not the same as the initial results, then run a third time to determine which result is valid.	If the results differ by more than one times the standard deviation of the mean then they are said to disagree with each other.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.4.2.3.2 If the second trial is the same as the initial results, this indicates that the calibration curve is inaccurate. This is due to a change in the X-ray energy flux. The unknown sample results are invalid as they are quantified now. A factor called the Reference Monitor Ratio, which adjusts for changes in the X-ray energy flux, needs to be calculated. See Figure 20 to calculate and enter into the computer files the Reference Monitor Ratio.	NOTE: If more than one reference standard was run in a batch then the results from both of them should be proportionally off from their established mean values if the inaccuracy is due to a change in the X-ray energy flux. A change in the X-ray energy flux can result from a changing or realigning of the X-ray tube or changing of the high voltage power supply. Also the X-ray energy flux can change gradually as would be indicated by a gradual but steady change in the tabulated reference standard results. Once the Reference Monitor Ratio Value is entered into the proper files, the present run of samples can be requantitated.
7.4.2.4 If the second and third trials of the reference standard agree with each other and they fall within ± two times the standard deviation from the mean value of the tabulated results, then reanalyze the original pressed pellet for that standard.	The error in the initial results may be due to cross contamination in the X-ray lab sample preparation or to sample mix-up in the data acquisition.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.4.2.4.1 If the results of the second trial on the original pellet are the same as the original result, this indicates either a mismarking of the original reference sample with another sample or that contamination occurred in the X-Ray Lab preparation. Inform the lab supervisor and make a note of the problem in the log book. Also examine the sample preparation log sheet and the analysis results to see if a sample-to-reference mismarking may have occurred (See Step 7.4.2.5) or if cross contamination may have occurred from perhaps a high level sample to the reference (See Step 7.4.2.6).	
7.4.2.4.2 If the results of the second trial agree with the tabulated results, this indicates a sample mix-up in the initial sample positioning or entering of sample names. Examine the other results and the sample position form to see where a mix-up occurred. Then rerun the necessary samples to confirm this. Correct the data, and proceed to Step 7.4.3.	

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.4.2.5 If it is apparent from the data where a mismarking has occurred then prepare and rerun the necessary sample(s) to confirm this.	
7.4.2.5.1 If a mismarking is confirmed from the repeat data then report the correct data and mark the original data as a sample mix-up.	
7.4.2.5.2 If a mismarking is not confirmed from the repeat data then cross contamination or outside contamination must have occurred.	
7.4.2.6 If either cross contamination or outside contamination is suspected, then examine the sample preparation log sheet and analysis results to see where sample carry over or cross contamination may have occurred from perhaps a high level sample to the blank, ie from the mixing vial or die set.	NOTE: High contamination level samples should have been screened with a Geiger-Mueller counter and marked as high by the Sample Receiving Lab. Precautions then should have been taken in their preparation to avoid sample carry over.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.4.2.6.1 If there is evidence of possible sample carry over from a high level sample to other sample(s), rerun the samples which were possibly affected (including the blank). Mark the original results as invalid due to cross contamination and report the repeat results.	
7.4.2.6.2 If there is no evidence of sample carry over, it is assumed that the contamination is from an outside source, which may not be limited to a few samples. The entire sample run is then invalid and should be prepared and rerun.	
7.4.2.7 If the three results on the reference standard all disagree with each other, this indicates an instability in the X-ray energy flux. The unknown sample results are invalid. A stability test needs to be run on the instrument to verify that it is unstable. (Notify the lab supervisor who will run a stability test using an instrument computer program.)	An instability in the X-ray energy flux can be an indication that the X-ray tubing is rapidly degrading. Another possibility is that the power source is not working properly.
7.4.3 If the results from both the blanks and reference standards check out, then the results can be reported.	

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.4.3.1 Enter the reference standard results into the table of U and Th values for that reference standard.	
7.4.3.2 The analyst checking the transcription and quality of the data signs and dates the top of the front data sheet.	
7.5 If there are more samples to be analyzed, return to Step 7.1.3.1.	
7.6 Press the ATMOS key on the keyboard.	
7.6.1 Type 1 to select item 1 (Air) and then press the RETURN key on the keyboard.	The chamber is now at atmospheric pressure.
7.6.1.1 Wait two minutes before proceeding.	
7.6.2 Type 5 to select item 5 (Pump off) and press the RETURN key on the keyboard.	
7.6.3 Press the ATMOS key on the keyboard to exit the displayed menu.	
7.7 Turn off the X-rays by pressing the X-RAY OFF button on the 770 spectrometer.	The X-RAY OFF button is shown in Figure 11.
7.8 Raise the sample chamber lid and remove the samples.	
7.9 Complete Steps 7.2.3.1.to 7.2.3.7 to replace the Ag secondary target for the Mo secondary target.	

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.10 Release the sample chamber lid lock near the hinge, close the lid, and turn on the X-rays by pressing the X-RAY ON button on the 770 spectrometer.	
7.11 Type CONT/kV=40 and press the RETURN key on the keyboard.	
7.12 Type CONT/mA=0.9 and press the RETURN key on the keyboard.	The spectrometer is now in its idle conditions with the voltage at 40 kV and the current at 0.9 mA.
7.13 Hold the CTRL key down and press the C twice until a dot prompt appears on the CRT.	
7.14 Press the unload or release button next to the indicator light on disk drive 0 (See Figure 8).	This spins down the disk drive. The green light will flash for a few seconds.
7.14.1 Wait until the light goes out before proceeding.	
7.15 Turn the lever holding the data cartridge disk in place on disk drive 0 counter clockwise and remove the cartridge.	See Figure 8.

8.0 STANDARDIZATION

PROCEDURE	COMMENTS
8.1 Energy Calibration Check	The energy calibration is to be checked with a reference sample of 316 stainless steel within a two week period before samples are analyzed. Check the log book for the date of the last check (See Figure 21).

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8.0 STANDARDIZATION (cont.)

PROCEDURE	COMMENTS
8.1.1 Follow Steps 7.1.1 through 7.1.2.8 if the system is not "booted up".	
8.1.2 Complete Steps 7.2.1 and 7.2.6 if the 316 stainless steel and 2036 Cu-Al standards are not on the wheel or if there are liquid samples in the chamber which need to be removed.	
8.1.2.1 Place the Kevex 316SS and 2036 Cu-Al standards on the sample wheel.	
8.1.2.2 Remove liquid samples from the chamber.	
8.1.3 Release the sample chamber lid lock near the hinge, close the lid, and turn on the X-rays by pressing the X-RAY ON button on the KEVEX 770 spectrometer.	The interlock light on the spectrometer should go off when the lid is closed. The X-ray on button and the interlock light are shown in Figure 11.
8.1.4 To position the 316SS sample for the check of the energy calibration press the SAMPLE key on the keyboard.	The sample key is one of the "user XRF" keys shown in Figure 9. This displays the sample position table. As an alternative you can just type: CONT/POS "position number", then press the return key on the keyboard.
8.1.4.1 Type the sample wheel position number in which the 316SS standard is located and press the RETURN key on the keyboard.	

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8.0 STANDARDIZATION (cont.)

PROCEDURE	COMMENTS
8.1.4.2 Press the SAMPLE key on the keyboard to exit the displayed menu.	
8.1.5 Type RE/MAT CALIB and press the RETURN key on the keyboard.	This recalls the matrix file for running the standard samples.
8.1.6 To automatically set up the excitation conditions for the 316SS sample type : CONT/COND 1 and press the RETURN key on the keyboard.	The following conditions should be set up: Ag Target #3, 40 kV 0.5 mA (for pulse rate 1) Air, 20 eV/ch 60 sec. livetime.
8.1.7 Proceed as follows to change the pulse rate to 2 or to verify that it is set at 2.	
8.1.7.1 Remove the KEVEX 8000 instrument panel under the keyboard by pulling it toward yourself.	
8.1.7.2 On the model 4460 pulse processor, push the appropriate RATE pushbutton to select the pulse rate to be used.	See Figure 14.
8.1.8 Setting of appropriate deadtime.	The preliminary conditions just set up need to be adjusted to achieve a 50%±5% deadtime count rate for the sample.
8.1.8.1 Press the mA key on the keyboard.	The mA key is one of the "User XRF" keys shown in Figure 9.

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8.0 STANDARDIZATION (cont.)

PROCEDURE	COMMENTS
8.1.8.2 Type the proper current shown in the table for pulse rate 2 and press the RETURN key on the keyboard.	To achieve nominally 50% deadtime on the 316SS standard sample, use the currents below:

Pulse Rate	Current (mA)
1	0.5
2	0.8
3	1.2
4	1.7
5	2.5

8.1.8.3 Press the mA key on the keyboard to exit the displayed menu.	
8.1.9 To acquire a spectrum, press the ACQUIRE key on the keyboard.	The computer will acquire data for the preset livetime of 60 seconds.
8.1.10 After the spectrum is acquired, remove the escape peaks by typing PRO/ESC and press the RETURN key on the keyboard.	The computer will draw the escape peaks, subtract them and add them to the primary peak.
8.1.11 Type ID/AUTO/PR and press the RETURN key on the keyboard.	The computer will printout a list of peak energies and possible identifications. One peak will be Fe K-alpha and another will be the Mo K-alpha.
8.1.11.1 Confirm that the printer "on line" indicator is lit; if not press the button.	
8.1.12 Type: PRO/WIND/Cl and press the RETURN key on the keyboard.	
8.1.13 Type: ACQ/CL and press the RETURN key on the keyboard.	This clears the spectrum.

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8.0 STANDARDIZATION (cont.)

PROCEDURE	COMMENTS
8.1.14 Check the Fe K-alpha and Mo K-alpha peak energies to see if they are within the following ranges: Fe K-alpha.... 6.40±0.01 keV; Mo K-alpha.... 17.44±0.02 keV.	
8.1.14.1 If the energies are within specification for the pulse rate setting of 2 to be used in subsequent analyses, then the instrument is considered to be properly calibrated and you can proceed to Step 8.3.	
8.1.14.2 If the energies are outside specification, proceed to Step 8.2.	
8.2 Autocalibration Procedure	
8.2.1 If not in position, position the standard to be used for calibration by following Steps 8.1.4 through 8.1.4.2.	If only heavy elements are to be analyzed, use the 316SS standard; otherwise, use the 2036 Cu-Al standard. Heavy elements would be considered to be elements above Fe in the periodic table.
8.2.2 Setting up exitation conditions for the specific standard used.	
8.2.2.1 If the 316SS standard is being used, condition code 1 is already set from Step 8.1.5 and 8.1.6, so proceed to Step 8.2.3.	

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8.0 STANDARDIZATION (cont.)

PROCEDURE	COMMENTS
8.2.2.2 If the 2036 Cu-Al standard is to be used type CONT/COND 2 and press the RETURN key on the keyboard to change the excitation conditions.	**WARNING: NO LIQUID SAMPLES CAN BE IN THE CHAMBER FOR THIS VACUUM CONDITION.** Ensure the lever to the helium inlet line is perpendicular to the line. The following conditions should be set up: Direct Target No. 8, 13 kV 0.01 mA (for pulse rate 1) Vacuum, 10eV/ch 60 sec. livetime.
8.2.3 On the pulse processor press the appropriate RATE pushbutton to select the pulse rate 2 that will be used in subsequent analyses.	See Figure 14.
8.2.4 Press the mA key on the keyboard.	The current is set according to the pulse rate which was just set to achieve nominally 50% deadtime as follows:

Pulse Std.	Current Rate	(mA)
2036	1	0.04
2036	2	0.06
2036	3	0.09
2036	4	0.13
2036	5	0.22
316SS	1	0.50
316SS	2	0.80
316SS	3	1.20
316SS	4	1.70
316SS	5	2.50

8.2.4.1 Type in the proper current for pulse rate 2 and press the RETURN key on the keyboard.	See comment in Step 8.2.4 to determine the proper mA setting.

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8.0 STANDARDIZATION (cont.)

PROCEDURE	COMMENTS
8.2.4.2 Press the mA key on the keyboard to exit the displayed menu.	
8.2.5 Press the PROGRAM key on the keyboard.	The program key is one of the "control keys" shown in Figure 9.
8.2.5.1 Type 4 to select item 4 (SELF TEST) and then press the RETURN key on the keyboard.	
8.2.5.2 Type 4 to select item 4 (EDC Calibration) and then press the RETURN key on the keyboard.	In the menu displayed on the terminal, item 5 should be 1.487 for the 2036 standard or 6.400 for the 316SS standard. Item 6 should be 8.041 for 2036 standard or 17.443 for 316SS standard. EDC is an abbreviation for energy-digital converter.
8.2.5.3 If item 5 or 6 ("Desired low peak") and ("Desired high peak") are correct, proceed to Step 8.2.5.4; otherwise, proceed as follows.	Refer to the comment from Step 8.2.5.2 for the appropriate value.
8.2.5.3.1 Type the item number to be corrected and press the RETURN key on the keyboard.	
8.2.5.3.2 Type the correct value as given in comment 8.2.5.2 and press the RETURN key on the keyboard.	

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8.0 STANDARDIZATION (cont.)

PROCEDURE	COMMENTS
8.2.5.3.3 Repeat the above step (b) until values for item 5 and 6 are correct, then proceed to the next step.	
8.2.5.4 Type 1 and press the RETURN key on the keyboard.	This will execute the EDC calibration routine. It will acquire spectra, measure the peak positions, and adjust the gain and offset iteratively until the desired accuracy is reached or 20 iterations have occurred. The desired accuracy as set by item 2 in the menu is 0.003 keV. This is the instrument default value and it should not be changed. At the end it will either display "Autocalibration successful" or a failure message.
8.2.5.5 If the autocalibration is not successful, repeat Step 8.2.5.4 once.	See Figure 22 for the manual calibration procedure.
8.2.5.5.1 If the autocalibration is still not successful, a manual calibration will have to be performed. Follow the procedure in Figure 22.	
8.2.5.5.2 If the autocalibration was successful, proceed to Step 8.3.5.6.	
8.2.5.6 Push the PROGRAM key on the keyboard to exit the displayed menu.	

8.0 STANDARDIZATION (cont.)

PROCEDURE	COMMENTS
8.3 Post Calibration Procedure	This pertains to recording the calibration information, releasing the vacuum on the chamber, and clearing the screen.
8.3.1 Enter into the instrument log book the energy calibration check data, if a calibration was performed and at what pulse rates and whether manual adjustments were made.	
8.3.2 If the sample chamber is under a vacuum, release as follows:	
8.3.2.1 Press the ATMOS key on the keyboard.	
8.3.2.2 Type 1 to select air and then press the RETURN key on the keyboard.	The chamber is now at atmospheric pressure.
8.3.2.2.1 Wait 2 minutes before proceeding.	
8.3.2.3 Type 5 to turn pump off and then press the RETURN key on the keyboard.	
8.3.2.4 Press the ATMOS key on the keyboard to exit displayed menu.	
8.3.3 To erase MLD markers on the screen:	
8.3.3.1 Press the PROGRAM key, type 1, press the RETURN key, and then press the CLEAR key on the keyboard.	

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8.0 STANDARDIZATION (cont.)

PROCEDURE	COMMENTS
8.3.3.2 Press the PROGRAM key on the keyboard to exit the displayed menu.	
8.3.4 If samples are to be analyzed, return to Step 7.2.	

9.0 CALCULATIONS

Not Applicable.

10.0 PRECISION AND ACCURACY

10.1 The accuracy of the energy discrimination established by the autocalibration is 0.003 keV.

10.2 The system is recalibrated when the accuracy falls below the deviations given in Step 8.1.11. The peak positions are known to an accuracy of ± 0.001 keV.

10.3 The automatic identification matching criterion is normally set at a deviation of ± 2 channels.

10.4 The automatic identification sensitivity criterion is set for a minimum peak to background ratio of 0.50.

11.0 QUALITY ASSURANCE

One blank soil sample and one reference standard sample are run for every 10 unknown samples and one recycle sample is run for every set of samples.

12.0 REFERENCES

Kevex Analyst 8000 User's Manual.

13.0 FIGURES

13.1 Figure 1 - Sample Grinding Items

13.2 Figure 2 - X-Ray Lab Log of Sample Preparation

13.3 Figure 3 - Mixer/Mill Device

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13.0 FIGURES (cont.)

13.4 Figure 4 - 31mm Die Press Set

13.5 Figure 5 - Manual Hydraulic Press

13.6 Figure 6 - Vacuum Desiccator

13.7 Figure 7 - Warm Start Procedure

13.8 Figure 8 - Disk Drive 0 Illustration

13.9 Figure 9 - Program Keyboard

13.10 Figure 10 - Procedure to Format, Initialize and Copy a Disk

13.11 Figure 11 - X-RAY OFF Button

13.12 Figure 12 - Secondary Target Turret

13.13 Figure 13 - Sample Position Form

13.14 Figure 14 - Model 4460 Pulse Processor

13.15 Figure 15 - Program Procedure File SOIL-MO

13.16 Figure 16 - Program Procedure File NAMES

13.17 Figure 17 - Program Procedure File MO

13.18 Figure 18 - Program Procedure File DEC-PRO

13.19 Figure 19 - Verifying the Reliability of the Blank Sample Data

13.20 Figure 20 - Procedure for Calculating the Reference Monitor Ratio

13.21 Figure 21 - General Information on Standardization of KEVEX 770 EDXRF System

13.22 Figure 22 - Manual Calibration Procedure

13.23 Figure 23 - Verifying the Reliability of the Reference Standard Data

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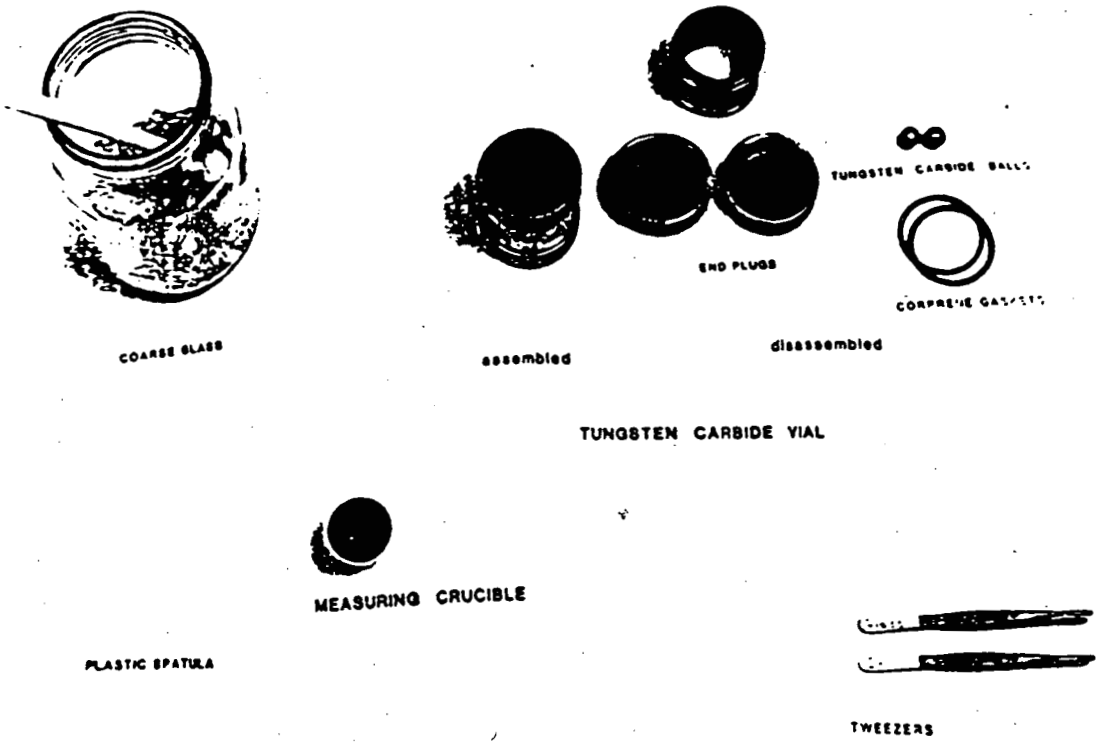

SAMPLE GRINDING ITEMS
Figure 1

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X-RAY LAB LOG OF SAMPLE PREPARATION OF SOIL SAMPLES (MIXED AND PRESSED)

Lab No.	Mixing Vial No.	Die Press No.	Time (hr:min)	Date (Mo/Day/Yr)	Analyst Initials

X-RAY LAB LOG OF SAMPLE PREPARATION
Figure 2

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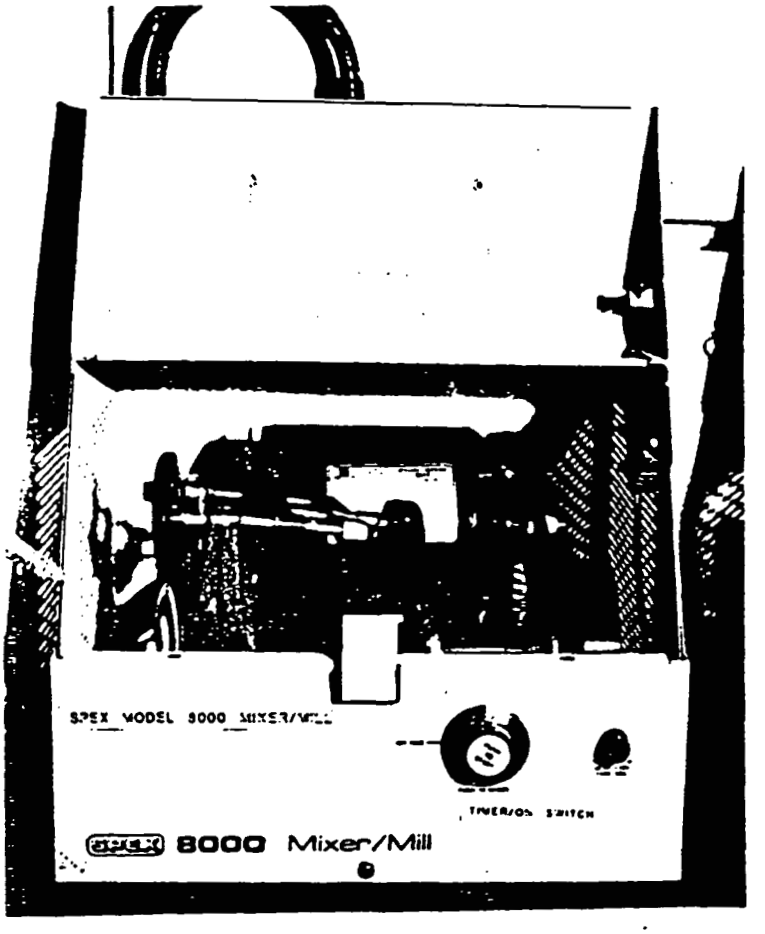
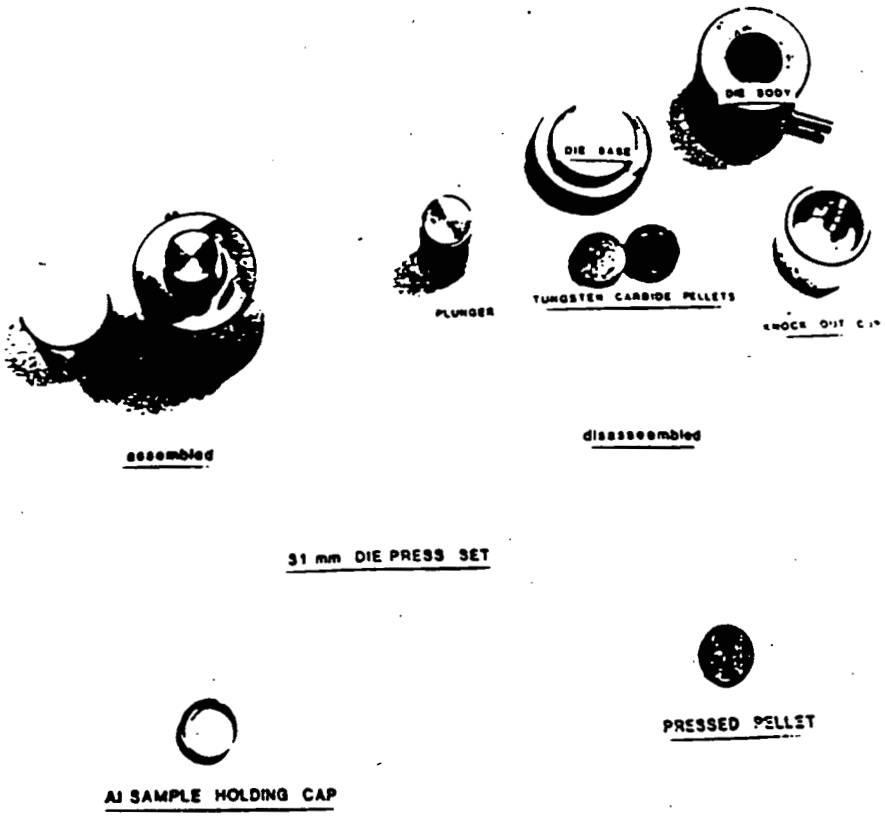


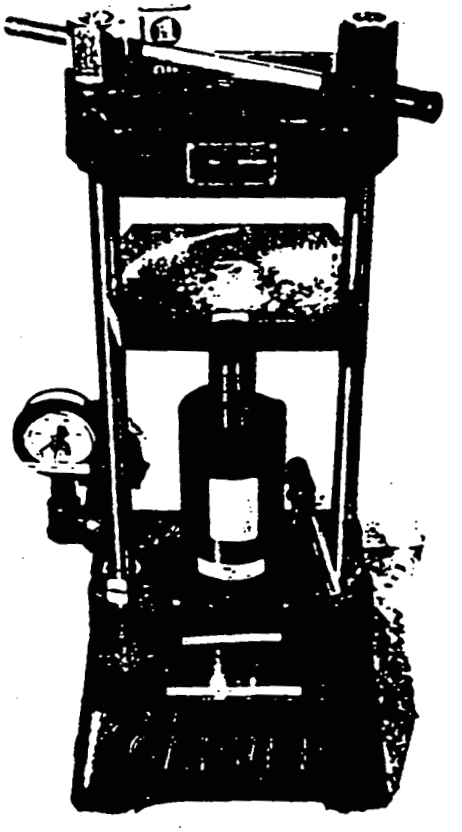
MIXER/MILL DEVICE
Figure 3

R - MATERIAL REVISED, ADDED, OR DELETED.



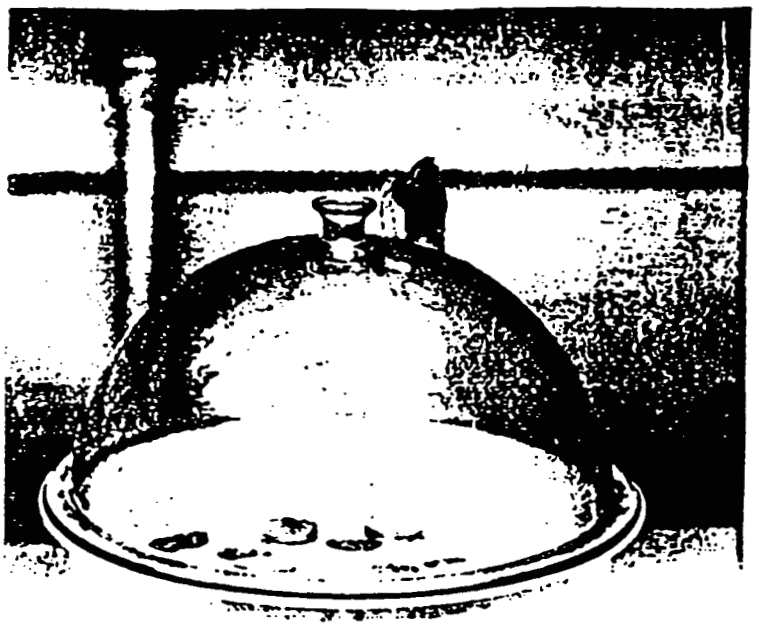
31mm DIE PRESS SET
Figure 4

R - MATERIAL REVISED, ADDED, OR DELETED.



MANUAL HYDRAULIC PRESS
Figure 5

R - MATERIAL REVISED, ADDED, OR DELETED.



DESICCATOR

VACUUM DESICCATOR
Figure 6

R - MATERIAL REVISED, ADDED, OR DELETED.

1.0 Press the CLEAR and the HELP keys simultaneously on the keyboard.

The keyboard is shown in Figure 9. The clear key is one of the "control" keys and the help key is one of the "user XRF" keys.

1.1 The Kevex Corporation logo will appear for a few seconds and then the display will read "Kevex Analyst R06-24 warm start".

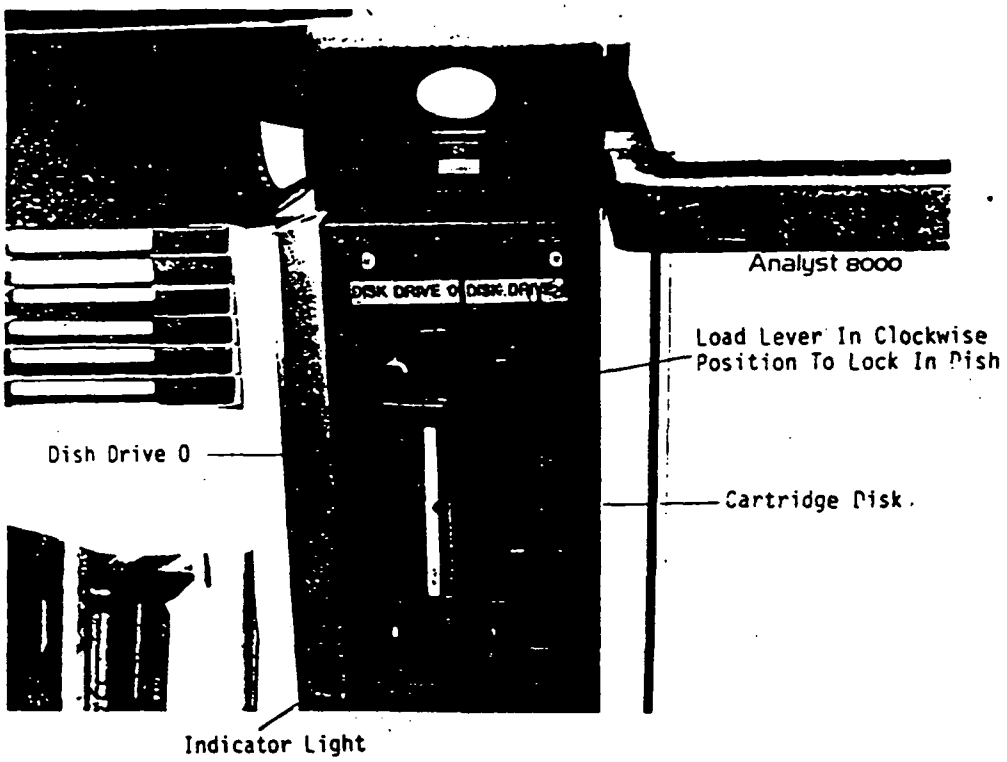
2.0 Press the RETURN key on the keyboard.

A question mark and then an @ symbol will appear on the display. The "@" is the machine language prompt.

3.0 Repeat Steps 7.1.2.4 through 7.1.2.9 of the procedure to reboot the system.

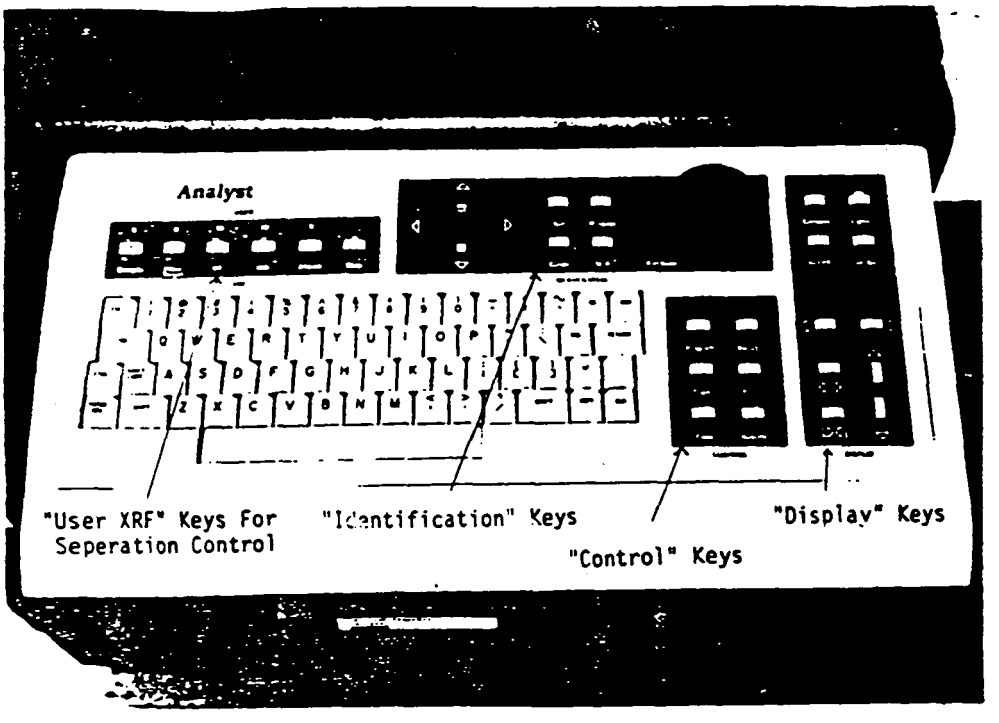
WARM START PROCEDURE
Figure 7

R - MATERIAL REVISED, ADDED, OR DELETED.



DISK DRIVE 0 ILLUSTRATION
Figure 8

R - MATERIAL REVISED, ADDED, OR DELETED.



PROGRAM KEYBOARD
Figure 9

R - MATERIAL REVISED, ADDED, OR DELETED.

1. Follow Steps 7.1.1 through 7.1.2.2 if the system is not "booted up" with the working disk copy in disk drive 0 and a dot prompt present.	
2. Hold the labelled end of a new disk with the side label toward the keyboard and insert it into disk drive 1.	Refer to Figure 8.
3. Turn the Load Lever Control clockwise.	The green disk drive indicator light should flash for a period and then remain on. **<u>CAUTION</u>: NEVER REMOVE A DISK BY SIMPLY TURNING THE LOAD LEVER COUNTERCLOCKWISE AND PULLING ON THE DISK. THIS COULD DAMAGE THE DISK AND THE DISK DRIVE. TO REMOVE A DISK, FOLLOW STEPS 7.14 THROUGH 7.15.**
4. Type: RUN FORMIO and press the RETURN key on the keyboard.	The computer will begin to format the new disk in drive 1. The red disk drive indicator light will stay light while the disk is being formatted. A dot prompt will be displayed on the screen when the formatting is complete (in about 3 minutes).
5. Type: INI/VOL/BAD DL1: and press the RETURN key on the keyboard.	The following message will be displayed on the CRT: DL1:Initialize; Are you sure?
6. Type in Y and press the RETURN key on the keyboard.	The computer will begin to initialize the new disk in drive 1. The following message will be displayed on the CRT when the initializing is complete (in about 2 minutes). ?DUP-I-No Bad blocks detected DL1: Volume ID?

PROCEDURE TO FORMAT, INITIALIZE AND COPY A DISK
Figure 10 (Sheet 1 of 3)

R - MATERIAL REVISED, ADDED, OR DELETED.

7.	Type in 1 and press the RETURN key on the keyboard.	The following message will appear: Owner?
8.	Type in your initials and press the RETURN key on the keyboard.	The dot prompt will then be displayed and the initializing is complete.
9.	To copy all of the files from the working disk in drive 0 to the new disk in drive 1 type: COPY/SYS DL0:*.* DL1: and press the RETURN key on the keyboard.	The computer will list on the CRT display each file as it is copied. The red indicator lights will alternate being lit between the two drives as information is transferred. A dot prompt will be displayed on the screen when the copying is complete.
10.	To enable the new disk to be "booted up" type the following: COPY/BOOT DL1:RT11XM.SYS DL1: and press the RETURN key on the keyboard.	
11.	Press the unload or release button next to the indicator light on disk drive 1.	See Figure 8. This spins down the disk drive. The green light will flash for a few seconds.
12.	Wait until the light goes out before proceeding.	
13.	Turn the lever holding the data cartridge disk in place on disk drive 1 counter-clockwise and remove the cartridge.	See Figure 8.
14.	Apply the adhesive labels which go on the edge of the cartridge disk, provided with a new disk, and write on it the following: FULL, DO NOT ERASE.	
15.	Apply the large adhesive label to the side of the new disk and write on it "COPIED" and the date.	

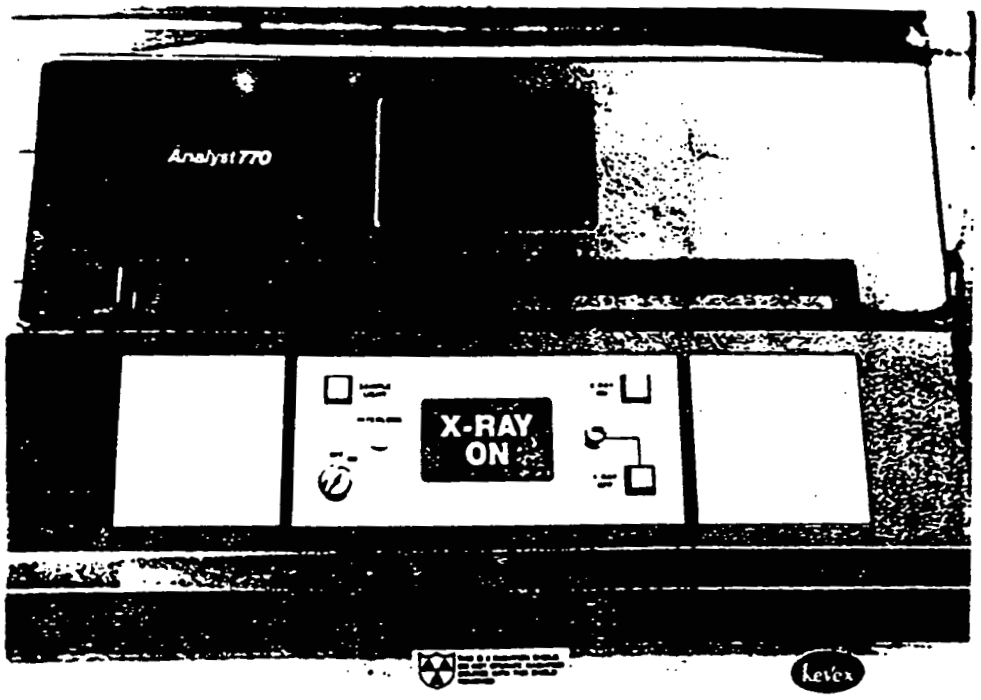
PROCEDURE TO FORMAT, INITIALIZE AND COPY A DISK
Figure 10 (Sheet 2 of 3)

R - MATERIAL REVISED, ADDED, OR DELETED.

16.	Type onto the computer DIR/PRI and press the RETURN key on the keyboard.	The computer will print out a list of the files on the working disk.
17.	Press the SELECT button on the printer.	This takes the printer off line.
18.	Press the FORM FEED button on the printer.	This advances the printer paper to the next page.
19.	Press the SELECT button again on the printer.	This puts the printer back on line.
20.	Type in: DIR/PRI*.KVX and press the RETURN key on the keyboard.	The computer will print out a list of the Kevex library files on the working disk.
21.	Press the SELECT button on the printer.	This takes the printer off line.
22.	Press the FORM FEED button on the printer.	This advances the printer paper to the next page.
23.	Press the SELECT button again on the printer.	This puts the printer back on line.

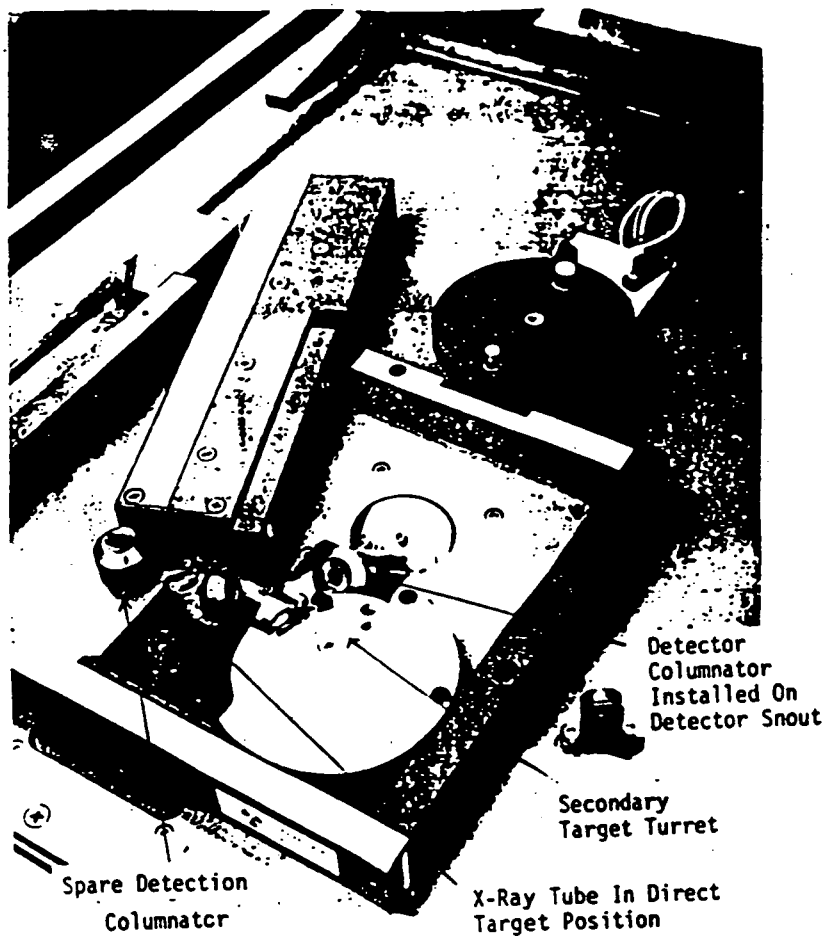
PROCEDURE TO FORMAT, INITIALIZE AND COPY A DISK
Figure 10 (Sheet 3 of 3)

R - MATERIAL REVISED, ADDED, OR DELETED.



X-RAY OFF BUTTON
Figure 11

R - MATERIAL REVISED, ADDED, OR DELETED.



SECONDARY TARGET TURRET
Figure 12

R - MATERIAL REVISED, ADDED, OR DELETED.

SAMPLE POSITION FORM
FOR QUANTITATIVE ANALYSIS OF U AND Th IN SOIL SAMPLES BY ENERGY DISPERSIVE
X-RAY FLUORESCENCE SPECTROSCOPY METHOD No. 7003

DATE:_____

SAMPLE POSITION	SAMPLE LAB NUMBER	
1		(REFERENCE STANDARD)
2		(BLANK)
3		(RECYCLE)
4		
5		
6		
7		
8		
9		
10		
11		
12		

SAMPLE POSITION	SAMPLE LAB NUMBER	
13		
14		
15		
16		
17		
18		
19		
20		
21		
22		
23		(BLANK)
24		(REFERENCE STANDARD)

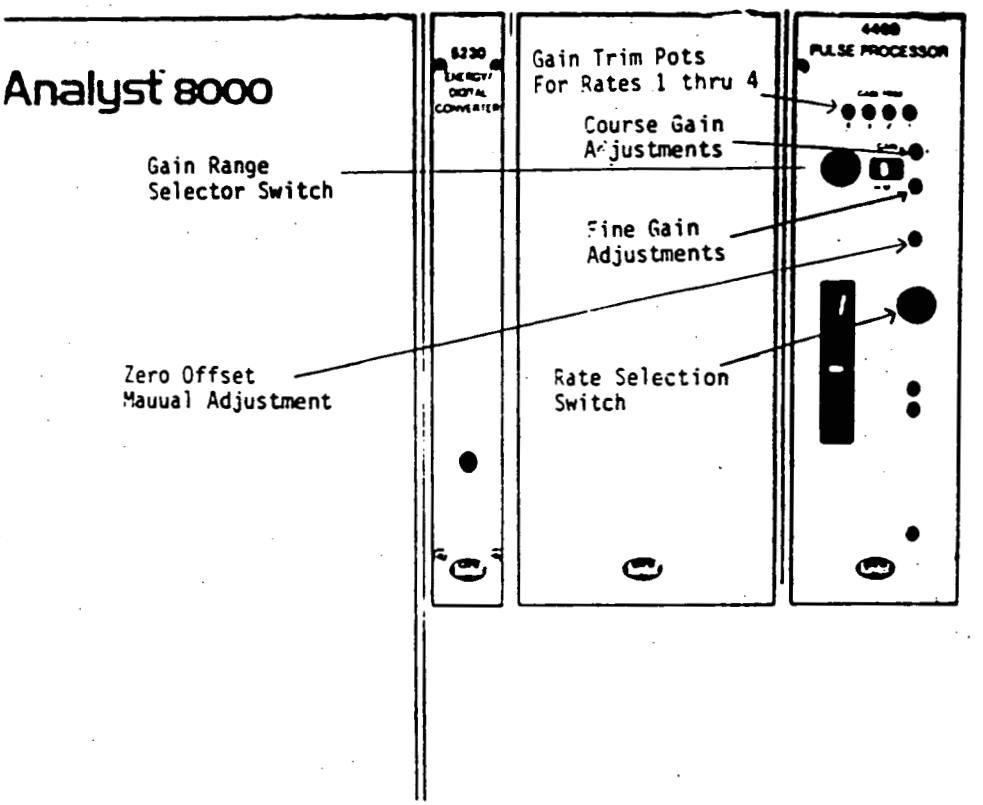
DATE:_____

SAMPLE POSITION	SAMPLE LAB NUMBER	
1		(REFERENCE STANDARD)
2		(BLANK)
3		(RECYCLE)
4		
5		
6		
7		
8		
9		
10		
11		
12		

SAMPLE POSITION	SAMPLE LAB NUMBER	
13		
14		
15		
16		
17		
18		
19		
20		
21		
22		
23		(BLANK)
24		(REFERENCE STANDARD)

SAMPLE POSITION FORM
Figure 13

R - MATERIAL REVISED, ADDED, OR DELETED.



MODEL 4460 PULSE PROCESSOR
Figure 14

R - MATERIAL REVISED, ADDED, OR DELETED.

PROCEDURE FILE 0:SOIL-MO: 1:0 1-NOV-88

Number of lines: 36

```
**************************************************************
TYPE ' THIS PROCEDURE WILL SIMULTANEOUSLY ACQUIRE AND PROCESS'
TYPE ' SPECTRA FROM SOIL SAMPLES AT CONDITION CODE 1 (Mo) '
TYPE ' FOR A MULTIPLE NUMBER  OF SAMPLES '
TYPE '~M~6SET THE VERTICAL SCALE TO 5000 CPS FOR PRINTING. '
TYPE '    '
TYPE '~M~5USE A AG DETECTOR COLUMNATOR' ; TYPE '  '
TYPE '~M~6SET THE PULSE RATE TO 2.'
TYPE '~A~Q'
!           FILES NEEDED:
!    SYSTEM:  KEV770.1.0
!    WINDOWS:  BA-NORM-MO1; BA-MOD-MO1; DEC-MO
!    SPECTRUM:  BLK-MO;
!    MATRIX:  SOIL-MO
!    PROCEDURE:  SOIL-MO; NAME; MO; DEC-PRO
!    LEAST SQUARES:  SOIL-U-LOW; SOIL-U-HIGH; SOIL-TH; SOIL-THU
RUN NAME
RE/SYS KEV770.1.0
SET/XRF A1=1 ; SET/XRF P2=3 ; SET/XRF P1=2
RE/MAT SOIL-MO
CONT/COND 1 ; CONT/POS #IST
ACQ/CL/STA
LOOP #POS=#IST,#LAST
ACQ/WAIT ; COPY P1=A1 ; CONT/POS +1 ; ACQ/CL/STA
PRO/ESC ; SAV/SPEC ;;
^Z
RUN MO
ID/CL ; ID/AUTO/PRI ; ID/LI/PR ; ID/CL
ID/ADD SI,S,K,CA,TI,BA,MN,FE,CU,ZN,W,PB,U,TH,RB,BR,SR,Y,ZR,MO
ID/LI/PR
PRO/WIND/CL ; ^L
RUN DEC-PRO
LOOP/END
SET/ACQ VAL=5 ; ACQ/CL/STA/WAIT
CONT/KV 30
CONT/MA 0.9
TYPE 'FINISHED'
```


PROGRAM PROCEDURE FILE SOIL-MO
Figure 15

R - MATERIAL REVISED, ADDED, OR DELETED.

PROCEDURE FILE 0:NAME: 1:0 30-AUG-88

Number of lines: 19

```
 1
 2 ^k
 3 ^t
 4 #IST=1
 5 #LAST=24
 6 ASK/INT '~4ENTER FIRST SAMPLE POSITION' #IST
 7 ASK/INT '~3ENTER LAST SAMPLE POSITION' #LAST
 8 ^Z
 9 !
10 !~B ENTER SAMPLE LABELS WHEN PROMPTED
11 #NAME=' '
12 LOOP #POS=#IST,#LAST
13 ASK/FILE '~7SAMPLE NO. #POS NAME' #NAME
14 SET/NAME #POS=#NAME
15 ^Z
16 #NAME=' '
17 LOOP/END
18 ^T
19
```


PROGRAM PROCEDURE FILE NAMES
Figure 16

R - MATERIAL REVISED, ADDED, OR DELETED.

```
PROCEDURE  FILE 0:MO:  1:0  1-NOV-88


Number of lines:      11
          ************************************
        1 TYPE 'Soil Sample'
        2 TYPE 'Pressed Pellet'
        3 TYPE '32.0 kV, 2.8mA'
        4 TYPE 'Mo Target'
        5 TYPE 'vacuum'
        6 TYPE '1000 sec. live time'
        7 TYPE '20 eV/ch'
        8 TYPE 'pulse rate 2'
        9 TYPE 'Ag Detector Columnator'
       10 TYPE 'Ag Target filter'
       11 ^P
```


PROGRAM PROCEDURE FILE MO
Figure 17

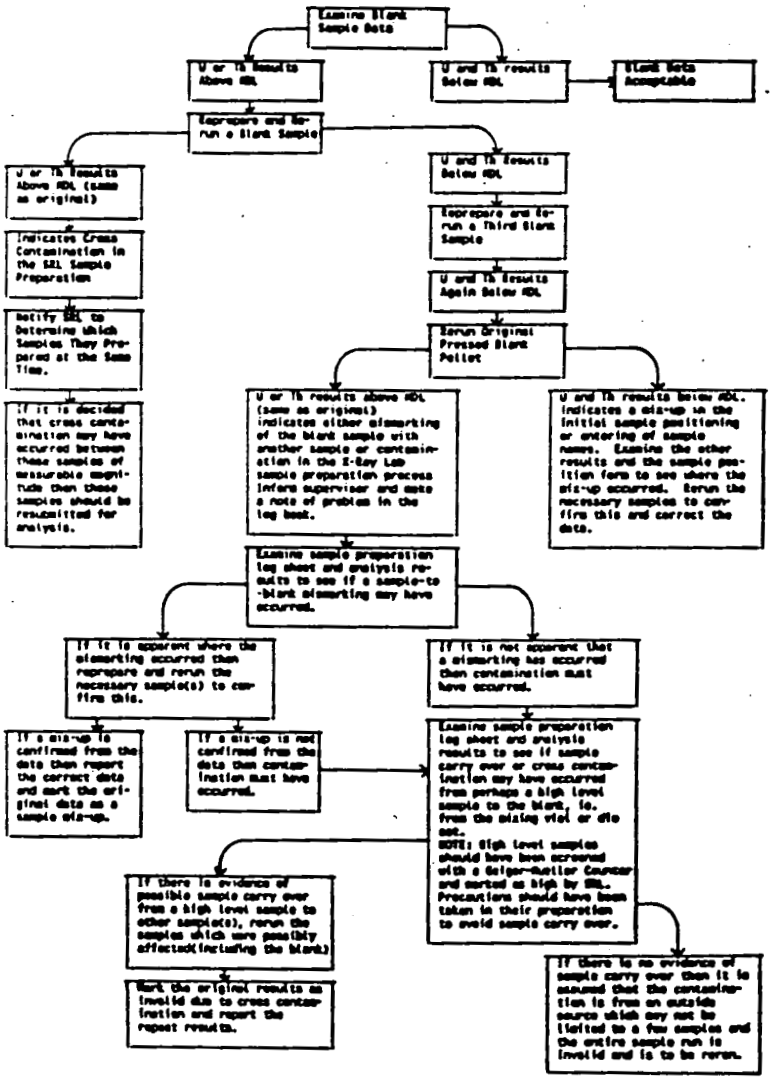
R - MATERIAL REVISED, ADDED, OR DELETED.

```
PROCEDURE  FILE 0:DEC-PRO:  1:0  1-NOV-88


Number of lines:      6
        ***********************************************
      1 ID/CL
      2 RE/COM BLK-MO ; BA/NORM BA-NORM-MO1
      3 BA/MOD BA-MOD-MO1 ; BA/SUB
      4 ID/ADD U,RB,PB,TH,SR,W,Y
      5 RE/WIND DEC-MO ; DEC/GAUS -RB,-PB,-SR,-W,-Y
      6 ^L ; PRO/WIND/CL ; BA/CL
```

PROGRAM PROCEDURE FILE DEC-PRO
Figure 18

R - MATERIAL REVISED, ADDED, OR DELETED.



VERIFYING THE RELIABILITY OF THE BLANK SAMPLE DATA
Figure 19

R - MATERIAL REVISED, ADDED, OR DELETED.

1. Select a reference standard for which the mean value(s) has been determined from six or more trials and the mean value for U is within the range from 70 to 300 ppm.	This may be the standard which was run and found to be out of bounds of its tabulated results provided it falls within the range. In that case the data already collected on that sample can be used in the following calculations.
2. Prepare and run the standard sample, following Steps 6 and 7 to acquire at least four concentration results for U.	These results can be from a minimum of two prepared pellets, in which case if less than four separate pellets are used some may be ran twice.
3. Calculate an average of the U concentrations.	
4. Calculate an adjustment factor by dividing the average value by the tabulated mean value for the particular reference standard.	
5. Type ED/MAT SOIL-MO and press the RETURN key on the keyboard.	This command is to edit a matrix file called SOIL-MO.
6. Use the black right arrow key to move the cursor over to the 13th column titled "Monitor Ratio".	The presently existing reference monitor ratio should be displayed in the first row which is for excitation condition No. 1.
7. Multiply the adjustment factor times the existing reference monitor ratio to get the new reference monitor ratio.	
8. Enter the new ratio in place of the old one by typing it over the old one.	

PROCEDURE FOR CALCULATING THE REFERENCE MONITOR RATIO
Figure 20 (Sheet 1 of 3)

9. Press the CTRL key and the E simultaneously on the keyboard to exit the matrix file.

10. In order to requantify the analysis results of a series of samples which were initially decided to be invalid because the reference standard did not agree with the tabulated results, do the following.

11. Type ED/RESU followed by one of the sample lab numbers and press the RETURN key on the keyboard.

12. Press the CTRL key and the V simultaneously on the keyboard twice to advance the third page in the file.

13. Press the RETURN key on the keyboard to move the cursor to the first row.

14. Use the black right arrow key to move the cursor over to the 11th column titled "Monitor Ratio".

15. In the first row enter the new ratio in place of the old one by typing over the old one.

16. Press the black down arrow key to move the cursor to the second row.

17. Enter the new ratio in place of the old one here as well.

PROCEDURE FOR CALCULATING THE REFERENCE MONITOR RATIO
Figure 20 (Sheet 2 of 3)

R - MATERIAL REVISED, ADDED, OR DELETED.

18. Press the CTRL key and the E simultaneously on the keyboard to exit the results file.

19. Repeat Steps 10 through 18 for each sample in the series.

20. Return to Step 7.3.2 in the procedure to requantify U and Th concentrations for each sample.

PROCEDURE FOR CALCULATING THE REFERENCE MONITOR RATIO (cont.)
Figure 20 (Sheet 3 of 3)

R - MATERIAL REVISED, ADDED, OR DELETED.

The procedure described in section 8.0 checks the present energy calibration, of the energy-to-digital converter (EDC) and gives subsequent instructions based on the check.

Important points related to standardization to remember:

* The pulse processor zero and gain are adjusted to calibrate the EDC.
* The KEVEX 316 stainless steel is used to check the calibration of the EDC.
* The KEVEX 316 stainless steel or 2036 copper-aluminum standard is used to calibrate the EDC.
* This standardization check procedure should be performed: 1) once every two weeks or 2) whenever the pulse rate is changed or 3) whenever a "cold start" has been performed.
* The EDC is recalibrated whenever the energy deviation on a check exceeds the proper variance.
* Normally, the autocalibration routine is performed to adjust the calibration; however, if the adjustments are beyond the limits of the routine, a manual calibration procedure (described in Figure 22) will have to be used.
* The system must be calibrated for each pulse rate used.
* The system must be booted up to begin this standardization procedure.

GENERAL INFORMATION ON STANDARDIZATION OF KEVEX 770 EDXRF SYSTEM
Figure 21

R - MATERIAL REVISED, ADDED, OR DELETED.

1. On the model 4460 pulse processor select pulse rate 5 by pressing the pushbutton RATE switch.	The rate switch is shown in Figure 9.
2. In the "EDC Calibration Items" select item 7 and press RETURN.	This assumes that Step 8.2.5 of this procedure has been completed. This will "Reset EDC gain and zero DAC's for manual calibration".
3. Press the RETURN key twice to return to the "Program Items" menu.	
4. Enter 1 to select item 1 (MULTIPLE-ELEMENT MLK) and press the RETURN key on the keyboard.	This selects the multiple-element MLK routine. This is used to display lines from more than one element at a time.
5. If the 2036 std. is being used for calibration, enter Cu, RETURN, Al, RETURN, and if the 316SS std. is used enter FE, RETURN, Mo, RETURN.	
6. Press the PROGRAM key again to exit.	
7. Follow Steps 8.2.3 through 8.2.4.2 of this analytical procedure to set the pulse rate to 5 and the current accordingly.	

MANUAL CALIBRATION PROCEDURE
Figure 22 (Sheet 1 of 3)

R - MATERIAL REVISED, ADDED, OR DELETED.

8.	Make sure the gain is in the medium range on the pulse processor and if not press the GAIN pushbutton to select M (medium).	See the gain range selection switch shown in Figure 14.
9.	To adjust the zero setting expand the horizontal scale by using the (<>) key and center the Al or Fe peak on the screen by turning the KEVSCAN wheel.	The changing of the zero setting produces linear changes of energy.
10.	Press the ACQUIRE key to acquire a spectrum.	
11.	If the centroid is more than 1 to 2 channels off, adjust the zero pot with a pot screwdriver.	The zero pot is shown in Figure 14.
12.	Press the ACQUIRE key to stop acquiring the spectrum.	
13.	Press the CLEAR key and then the SHIFT and CLEAR simultaneously.	This erases the spectrum.
14.	Repeat Steps 10 to 13 until no further adjustment is needed.	
15.	To adjust the gain setting next center the expanded screen on the Cu or Mo peak by turning the KEVSCAN wheel.	The changing of the gain setting procedures exponentially proportional changes of the energy.
16.	Press the ACQUIRE key to acquire a spectrum.	
17.	If the centroid is more than 1 to 2 channels off, adjust the coarse gain pot with the pot screwdriver.	The coarse gain pot is shown in Figure 14.
18.	Press the ACQUIRE key to stop acquiring the spectrum.	

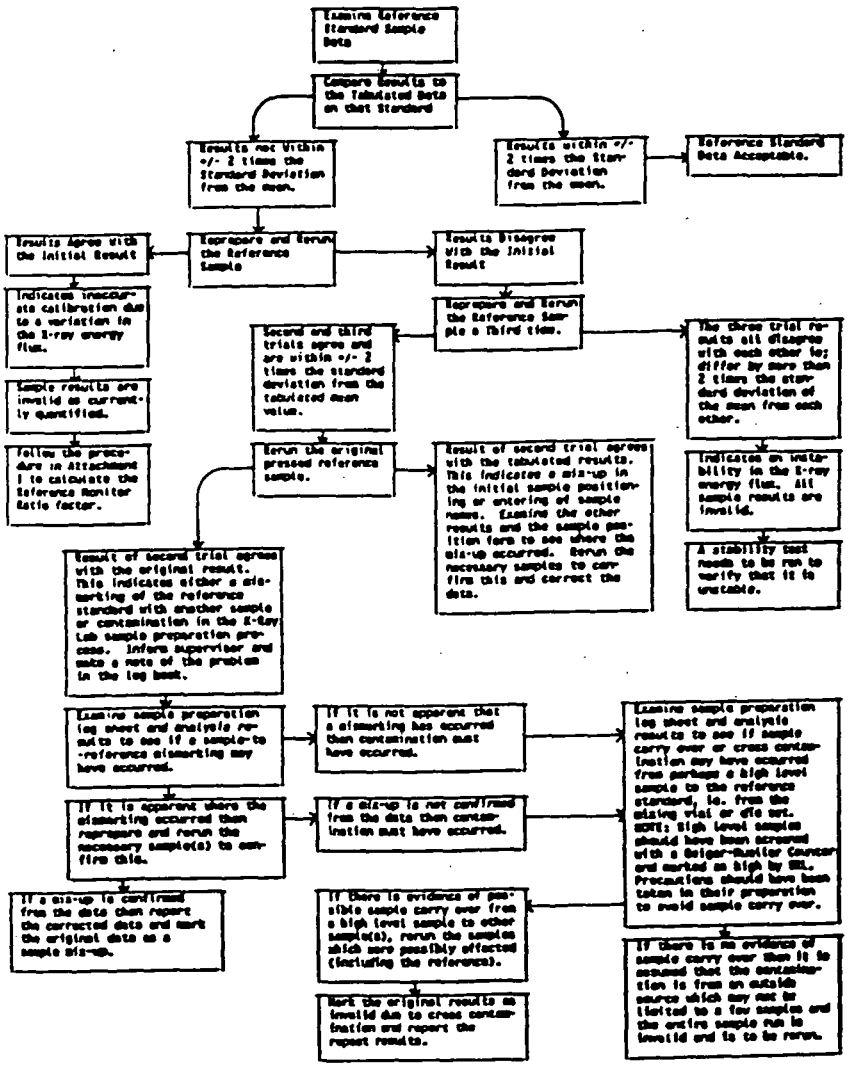
MANUAL CALIBRATION PROCEDURE
Figure 22 (Sheet 2 of 3)

R - MATERIAL REVISED, ADDED, OR DELETED.

19. Press the CLEAR key and then the SHIFT and CLEAR simultaneously.	This erases the spectrum.
20. Repeat Steps 16 to 19 until no further adjustment is needed.	
21. Select another pulse rate to be used for analyses by pressing the pushbutton RATE switch.	Calibrate at whichever of the remaining four rates are to be used in the following order 2-3-4-1.
22. Follow Steps 8.2.3 to 8.2.4.2 of this analytical procedure to set the pulse rate and the current accordingly.	
23. Press the ACQUIRE key to acquire a spectrum.	
24. If the centroid of the Cu or Mo peak is more than 1 or 2 channels off, adjust the gain trim pot for the particular rate setting.	There are four gain trim pots (label 1 through 4) for each of the pulse ra settings shown in Figure 14. Normally, they do not require any adjustments.
25. Press the ACQUIRE key to stop acquiring the spectrum.	
26. Press the CLEAR key and then the SHIFT and CLEAR simultaneously.	
27. Repeat Steps 23 to 26 until no further adjustment is needed.	
28. If another pulse rate is to be used for analysis return to Step 21.	
29. Return to Step 8.2.3 of the analytical procedure to repeat the autocalibration for each pulse rate.	

MANUAL CALIBRATION PROCEDURE
Figure 22 (Sheet 3 of 3)

R - MATERIAL REVISED, ADDED, OR DELETED.



VERIFYING THE RELIABILITY OF THE REFERENCE STANDARD DATA
Figure 23

R - MATERIAL REVISED, ADDED, OR DELETED.

RECORD OF ISSUE/REVISIONS

DATE	REV. NO.	DESCRIPTION AND AUTHORITY
04-17-91	0	Procedure for Quantitative Analysis of Uranium and Thorium in Soil Samples by Energy Dispersive X-ray Fluorescence (EDXRF) Spectroscopy per Request No. P90-425 (CIO C90-035).

R - MATERIAL REVISED, ADDED, OR DELETED.

1.0 SCOPE

This procedure is applicable to the determination of low concentrations of uranium. It is used as an alternate to the fluorometric method. The normally applied concentration ranges are: 0-10 g/L^{-1}; 0-10%; and 0-10,000 ppm. By applying the appropriate modification most types of samples may be analyzed employing this procedure.

This procedure includes sample preparation instructions for the following:

* Aqueous, Step 6.1
* AX/TBP in Kerosene, Step 6.2
* Thorium containing, Step 6.3
* Organic, unknown, Step 6.4
* Soil, Step 6.5
* Magnesium fluoride, residues containing F, Step 6.6
* Residues, low F^-, aqua regia soluble, Step 6.7
* Aqua regia insoluble, Step 6.8
* Raffinate cake, Step 6.9

The procedure for analysis is divided into three sections:

* Low interference type samples, Step 7.1
* Samples containing more than 10 g/L^{-1} thorium, Step 7.2
* Samples containing more than 10 g/L^{-1} zirconium and lime slurry samples, Step 7.3

R - MATERIAL REVISED, ADDED, OR DELETED.

2.0 PRINCIPLE

Uranium is estimated by measuring the absorbance of the uranium 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol (BrPADAP) complex, which absorbs strongly at 580 nm. See Attachment J, for a diagram of the structure of this molecule. Uranium is separated from major interferences by extraction into trioctylphosphine oxide (TOPO) in cyclohexane. The extracting solution contains F^- and PO_4^{-3} ions to inhibit the extraction of Th, Zr, and Fe, and HNO_3 to establish the proper acidity for uranium extraction. Ascorbic acid reduces V^{+5} to V^{+4}, Ce^{+4} to Ce^{+3}, Cr^{+6} to Cr^{+3}, and Fe^{+3} to Fe^{+2}. The interferences of these cations are much less severe at the lower valence states.

A complexing solution is added during color development to prevent interference from traces of interfering elements which extract. Sulfosalicylic acid masks the interferences of Be and Al. NaF reduces Th and Zr interferences and 1,2-diaminocyclohexanetetraacetic acid (Cy DTA) complexes many cations but not UO_2^{+2}.

An AutoAnalyzer continuous flow system is used to analyze the samples via this colorimetric method. An HP-85 personal computer has been interfaced to this analyzer using an HP-BCD parallel interface, which is connected to an HP model 3470 Digital Voltmeter. The DVM receives signals directly from the colorimeter via a connection to the telemetry output junction of the colorimeter. See Attachment H, for a printout of the computer program used in this procedure.

3.0 SPECIAL APPARATUS

3.1 An AutoAnalyzer system consisting of a sampler, proportioning pump, flowcell colorimeter, and strip chart recorder. The colorimeter is equipped with a 15 mm flowcell and 580 nm filters. See Attachment G, for a flow diagram of the system.

3.2 Hewlett-Packard (HP) 85 or 85B Personal Computer.

3.3 Hewlett-Packard Model 3470 Digital Voltmeter (DVM).

3.4 ROM Drawer, HP 82936A.

3.5 BCD Interface, HP 82941A.

3.6 I/O ROM module, HP 85-15003.

3.7 Attenuator, interfaces DVM to colorimeter.

3.0 SPECIAL APPARATUS (cont.)

3.8 Retsch model "BB-0" Laboratory Jaw Crusher.

3.9 T-Handle core sampler.

3.10 Pyrex culture tube, 19.5 x 145 mm with screw caps, Corning Glass Works, WMCO Stores No. 1045.

3.11 Pump Tubes, Acidflex, black/black, 0.030" I.D., Bran & Luebbe Part No. 116-0651P07.

3.12 Pump Tubes, Lolvaflex blue/blue, 0.065" I.D., Bran & Luebbe Part No. 116-0533P13.

3.13 Pump Tubes, Tygon orange/yellow, 0.16 mL min^{-1}, Bran & Luebbe Part No. 116-0549P05.

3.14 Pump Tubes, Tygon, yellow/yellow, 1.20 mL min^{-1}, Bran & Luebbe Part No. 116-0549P12.

3.15 Pump Tubing, Esco Rubber, Bran & Luebbe Part No. 116-0189-01.

3.16 Sample Pick-up Tubing, Polythylene, 0.015" x 0.045", Bran Luebbe Part No. 562-2002-01.

NOTE: The pump tube and connecting tubing for the sample must always be kept the same length and diameter for the computer program to function correctly. When changing any of the sample tubing make sure it is identical in length and diameter to the old tubing. (Use 20.25 inches of 0.015" x 0.045" polyethylene tubing for the sample pick-up tube and 6.5 inches of ESCO rubber, Special Apparatus 3.15, for the Br PADAP pump tubes.)

4.0 REAGENTS

NOTE: Unless otherwise specified, water used in preparing reagents is distilled water.

4.1 Extracting Solution. Dissolve 100 g of $Al(NO_3)_3 \cdot 9H_2O$ in 1000 mL of water. Dissolve 200 g of $KF \cdot 2H_2O$ in 1000 mL of water. Add 1100 mL of 70% HNO_3 to 1000 mL of water, mix, and add the $Al(NO_3)_3$ solution and the KF solution. Add 25 mL of concentrated H_3PO_4, dilute to 5000 mL, and mix thoroughly.

4.2 Ascorbic Acid, 200 g/L^{-1}. Dissolve 20 g of ascorbic acid in 100 mL of water. Make fresh daily.

4.0 REAGENTS (cont.)

4.3 TOPO in Cyclohexane, 25 g/L^{-1}. Dissolve 25 g of trioctylphosphine oxide in 1000 mL of cyclohexane (practical grade).

4.4 Complexing Solution, Stock. Suspend 12.5 g of 1,2-diaminocyclohexane tetraacetic acid (Cy DTA), 2.5 g of NaF, and 32.5 g of sulfosalicylic acid in 800 mL of water. Adjust the pH to 8.2 to 8.3 with 45% NaOH solution. Use a diluted solution of about 10% NaOH to adjust the complexing solution to 8.35 ± 0.02 pH. If the pH limits are exceeded use dilute nitric acid to return to pH 8.35. Dilute the pH adjusted solution to 1000 mL with water.

4.5 Complexing Solution, Diluted. Dilute a volume of stock complexing solution with an equal volume of water.

4.6 Buffer Solution, pH 8.35. Dissolve 149 g of triethanolamine in 800 mL of water. Adjust the pH to 8.35 ± 0.02 with perchloric acid. If necessary, use diluted NaOH solution to bring the pH within tolerances.

4.7 Methanol, reagent grade.

4.8 Br PADAP, Stock Solution, 0.5 g/L^{-1}. Dissolve 0.5 g of Br PADAP in 1000 mL of methanol (reagent grade).

4.9 Br PADAP, Working Solution, 0.050 g/L^{-1}. Add 400 mL of 0.5 g/L^{-1} Br PADAP solution to 3400 mL of methanol (reagent grade). Then add 200 mL of water.

4.10 Mixed Acid, 2 M HNO_3; 1 M HF. Add 130 mL of 70% HNO_3 (reagent grade) to 800 mL of water and mix. Then add 30 mL of 50% HF (reagent grade) and dilute to 1000 mL with water. Store in a polyethylene container.

NOTE: Wear "Solvex" rubber gloves when handling HF.

4.11 Uranium Standard Solution, 0.025 g/L^{-1}. Ignite a sample of standard U_3O_8, NBS 950, in a muffle furnace for 1 hour at the temperature designated in the NBS Certificate of Analysis (950b should be ignited at 800°C). Cool and store in a desiccator until used. Weigh 0.1180 g of U_3O_8 standard into a 150 mL beaker. Dissolve the U_3O_8 in 20 mL of concentrated HNO_3. Quantitatively transfer the solution to a 4000 mL volumetric flask, add 80 mL concentrated HNO_3, and dilute to the mark with distilled water.

4.12 Uranium Calibration Standard for High Thorium Samples, Th = 380 g/L^{-1}.

4.0 REAGENTS (cont.)

4.12.1 U = 0.0125 g/L^{-1}. Dissolve 905 g of reagent grade thorium nitrate crystals in 200 mL of water. Add 65 mL of 70% HNO_3. Transfer the solution to a 1000 mL volumetric flask. Add 500 mL of the 0.025 g/L^{-1} uranium standard (Reagent 4.11). Dilute to the mark with water.

4.12.2 Blank. Prepare a second solution as a "blank" for the calibration standard (i.e. - delete the 0.025 g/L^{-1} uranium standard).

4.13 Sodium Nitrite, 227 g/L^{-1}. Dissolve 454 g (one pound) $NaNO_2$ in 2000 mL of water.

4.14 Nitric Acid, 3M. Dilute 189 mL of 70% HNO_3 to 100 mL with water.

4.15 Hydrofluoric Acid (HF), 2%. Dilute 10 mL of 50% HF (reagent grade) to 250 mL and store in a polyethylene container.

NOTE: Wear "Solvex" rubber gloves when handling HF.

4.16 Nitric Acid, 35%. Dilute 50 mL of 70% HNO_3 to 100 mL with water.

4.17 Wash Solution. Add about 300 mL of 25 g/L^{-1} TOPO in cyclohexane (Reagent 4.3) to a 1000 mL separatory funnel. Add an equal volume of 3M nitric acid (Reagent 4.14), stopper the funnel, and shake the funnel vigorously for 1 minute. Allow the aqueous and organic phases to separate and remove the stopper. Drain and discard the aqueous (bottom) layer. The retained organic layer is the wash solution.

4.18 Uranium Reference Standard Solution, 0.0300 g/L^{-1}. Ignite a sample of standard U_3O_8, NBS 950, in a muffle furnace for 1 hour at the temperature designated in the NBS Certificate of Analysis (950b should be ignited at 800°C). Cool and store in a desiccator until used. Weigh 0.1416 g of U_3O_8 standard into a 150 mL beaker. Dissolve the U_3O_8 in 20 mL of concentrated HNO_3. Quantitatively transfer the solution to a 4000 mL volumetric flask, add 80 mL concentrated HNO_3, and dilute to the mark with distilled water.

5.0 SAFETY PRECAUTIONS

5.1 Safety glasses shall be worn in the laboratory at all times.

5.2 Wear "Solvex" rubber gloves when handling HF.

5.3 Perform all dissolution steps of this procedure, using heat, in an activated fume hood.

R - MATERIAL REVISED, ADDED, OR DELETED.

5.0 SAFETY PRECAUTIONS (cont.)

R 5.3.1 All operations involving the heating of materials on a hotplate (i.e., acid digestions) shall be performed in an appropriate fume hood which has been determined by IS&H to maintian an acceptable face velocity of 150 fpm or greater.

R 5.3.2 The type of fume hood utilized is determined by the fumes which will be generated. For example, if perchloric acid fumes are generated, then the only appropriate fume hood is one specifically designated for perchloric acid fuming. Contact supervisor, if unclear, as to what type of hood is appropriate for specific fuming operation.

5.4 Disposal of RCRA Hazardous Wastes:

5.4.1 Methanol-Cyclohexane Waste

5.4.1.1 Collect all waste coming from the Technicon AutoAnalyzer in a 4 liter plastic bottle during each shift.

5.4.1.2 Transfer the waste material to the appropriately labelled 5-gallon safety can in the RCRA Accumulation Storage Area as per SOP ANL-01-0053.

5.4.2 TOPO-Cyclohexane Waste

5.4.2.1 Collect all TOPO-Cyclohexane from the individual culture tubes into a glass separatory funnel during each shift.

5.4.2.2 At the end of each shift, the aqueous (lower) phase may be discarded down the sink.

5.4.2.3 Transfer the organic phase as in step 5.4.1.2.

5.4.3 Hazardous Solid Waste

5.4.3.1 Discard all hazardous solid waste in the bag contained in the metal safety can in the RCRA Accumulation Storage Area as per SOP ANL-01-0053.

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION

PROCEDURE	COMMENTS
6.1 For Aqueous Samples	
6.1.1 Place a magnetic stirring bar in the sample jar, and place the jar on a magnetic stirrer.	If a large amount of solids is present, shake the sample first to disperse the solids.
6.1.2 While the solution is being stirred, draw a sample into a clean dry pipet.	The volume of sample normally used is given in Table I (Attachment E). However, changes in plant operations may require a change in volume.
6.1.3 Adjust the meniscus to the line.	
6.1.4 Drain the pipet into a clean 125 mL Erlenmeyer flask.	
6.1.5 Add dissolution reagents to the flask.	Consult Table I (Attachment E) for the volumes and types of dissolution reagents required.
6.1.6 Place a Pyrex buret funnel in the neck of the flask.	
6.1.7 Place the flask on a hotplate and evaporate the contents to dryness.	For Lime Slurry and ZIRNLO samples, DO NOT TAKE TO DRYNESS.
6.1.8 Remove the flask from the hotplate and cool.	
6.1.9 Rinse any residue from the funnel and the inner walls of the flask into the flask with 3M HNO_3. Add several drops of $NaNO_2$ solution to the flask.	For lime Slurry and ZIRNLO samples, do not add $NaNO_2$. Instead, transfer the cooled sample to a 100 mL volumetric flask dilute to volume with 3M HNO_3, then proceed to Step 7.3.

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.1.10 Place the flask, without the funnel, back on the hotplate until the evolution of orange/yellow gas stops.	
6.1.11 Remove the flask from the hotplate and cool.	
6.1.12 Analyze the sample according to Section 7.1.	The sample may require further dilution before proceeding with the procedure for analysis. Consult Table I (Attachment E), and also review the latest trends in Production Plant processes.

6.2 For AX/TBP in Kerosene Samples - Soluble U

6.2.1 Pipet 2 mL of organic sample (AX or CR) into a 60 mL polyethylene separatory funnel.	Organic samples are TBP in kerosene.
6.2.2 Add 5 mL of 2% HF to the separatory funnel.	Wear "Solvex" rubber gloves when handling HF.
6.2.3 Stopper and shake the funnel for 1 minute.	
6.2.4 Permit the phases to separate and drain the aqueous phase into a 100 mL platinum beaker.	
6.2.5 Repeat Steps 6.2.2 through 6.2.4.	
6.2.6 Add 5 mL of distilled water to the funnel and shake it for 1 minute.	

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.2.7 When the phases separate, drain the aqueous phase into the platinum beaker containing the 2% HF extract.	
6.2.8 Add 5 mL of 70% $HClO_4$ to the beaker, place the beaker on a hotplate, and evaporate the solution to dryness.	
6.2.9 Remove the beaker from the hotplate and cool.	
6.2.10 Rinse any residue from the inner walls of the platinum beaker into the beaker with 3M HNO_3.	
6.2.11 Add several drops of $NaNO_2$ solution to the beaker and place it on the hotplate.	
6.2.12 When the evolution of orange/yellow gas stops, remove the beaker from the hotplate and cool.	
6.2.13 Analyze the sample according to Section 7.1.	
6.3 For Thorium-containing Samples	
6.3.1 Most thorium-containing samples will be soluble, requiring no dissolution procedure.	Ask the supervisor whether the thorium-containing sample is soluble.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.3.2 For thorium metal samples, dissolve 2.0 ± 0.1 grams of sample in 15 mL of 70% HNO_3 plus 12 drops of 2% HF in a 125 mL Erlenmeyer flask.	Perform this dissolution in an activated fume hood. Wear "Solvex" rubber gloves when handling HF. Heat is required for this dissolution.
6.3.3 For ThO_2 samples, dissolve 2.0 ± 0.1 grams of sample in 30 mL of 35% HNO_3 plus 12 drops of 2% HF in a 125 mL Erlenmeyer flask.	Perform this dissolution in an activated fume hood. Wear "Solvex" rubber gloves when handling HF. Heat is required for this dissolution.
6.3.4 Analyze the dissolved sample according to Section 7.2.	

6.4 For Organic Samples of Unknown Matrix - Total U

6.4.1 While stirring, pipet 2 mL of sample into a 150 platinum beaker.	
6.4.2 Add 15 mL of 70% HNO_3 to the beaker.	
6.4.3 Cover the beaker with a platinum lid.	
6.4.4 Place the beaker on a hotplate and evaporate the contents to dryness.	Make sure the contents don't boil out of the beaker during this step.
6.4.5 Remove the beaker from the hotplate and cool.	

R - MATERIAL REVISED, ADDED, OR DELETED.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.4.6 Transfer the beaker (with lid) to a muffle furnace and heat at 1000°C for 2 hours.	
6.4.7 Remove the beaker from the muffle furnace and cool.	
6.4.8 Add 5 mL of 70% HNO_3 and 5 mL of 70% $HClO_4$ to the beaker.	
6.4.9 Place the beaker (with lid) on a hotplate and evaporate the contents to dryness.	
6.4.10 Remove the beaker from the hotplate and cool.	
6.4.11 Rinse any residue from the lid and the inner walls of the beaker into the beaker with 3M HNO_3 and add several drops of $NaNO_2$ solution to the beaker.	
6.4.12 Place the beaker, without the lid, on the hotplate and heat until the evolution of orange/yellow gas stops.	
6.4.13 Remove the beaker from the hotplate and cool.	
6.4.14 Transfer the contents to a 100 mL volumetric flask, and add 5 mL of 70% HNO_3, and dilute to volume with distilled water.	

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6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.4.15 Pipet a suitable aliquot from the 100 mL flask (usually 2 or 5 mL and analyze the sample according to Section 7.1.	

6.5 For Soil Samples

NOTE: Aliquot and dilution volumes given below and in Table II (Attachment F) are tentative and may change as the sample composition varies.

6.5.1 Weigh about 1.5 grams of sample and transfer it to a 125 mL Erlenmeyer flask. Record this sample weight on the data card (w), to the nearest tenth of a milligram.	The soil sample should have been dried, ground and blended by the Sample Receiving Laboratory as per SOP ANL-40-0051.
6.5.2 Add 15 mL of 70% HNO_3 and 15 mL of 70% $HClO_4$ to the flask.	
6.5.3 Place a Pyrex buret funnel in the neck of the flask.	
6.5.4 Place the flask on a hotplate and evaporate the contents to dryness.	
6.5.5 Remove the flask from the hotplate and cool.	
6.5.6 Rinse any residue from the funnel and the inner walls of the flask into the flask with 3M HNO_3, and add several drops of $NaNO_2$ solution to the flask.	

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6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.5.7 Place the flask, without the funnel, on the hotplate and heat until the evolution of orange/yellow gas stops.	
6.5.8 Remove the flask from the hotplate and cool.	
6.5.9 Analyze the sample according to Section 7.3.	The sample may need to be centrifuged to separate the aqueous and organic phases. Consult the supervisor.

6.6 For Magnesium Fluoride (MgF_2) Samples and all Residue Samples Thought to Contain Fluoride

NOTE: Aliquot and dilution volumes given below, and in Table II (Attachment F), are tentative and may change as the sample composition changes from day to day.

6.6.1 MgF_2 samples should be ground and sieved (35 mesh) in the Sample Receiving Laboratory before being submitted for determination of uranium.

6.6.1.1 For samples marked "-35", weigh about 0.5 gram of sample, and transfer to a 100 mL platinum beaker. Record the weight to the nearest milligram.

6.6.1.2 For samples marked "+35", weight the entire sample and transfer to a 100 mL platinum beaker. Record the weight to the nearest milligram.

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.6.2 Add 10 mL of 70% HNO_3 and 35 mL of 70% $HClO_4$ to the beaker.	
6.6.3 Cover the beaker with its corresponding platinum lid, place the beaker on a hotplate, and evaporate the contents to dryness.	
6.6.4 Remove the beaker from the hotplate and cool.	
6.6.5 Rinse any residue which may have formed on the inner walls of the beaker and lid into the beaker with 3M HNO_3. Add several drops of $NaNO_2$ solution to the beaker.	
6.6.6 Place the beaker back on the hotplate and heat until the evolution of orange/yellow gas stops.	
6.6.7 Remove the beaker form the hotplate and cool.	
6.6.8 Transfer the sample to a 100 mL volumetric flask, add 5 mL of 70% HNO_3, and dilute to volume with distilled water.	
6.6.9 Pipet a suitable aliquot (usually 2 or 5 mL) from the 100 mL flask and analyze according to Section 7.1.	

6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.7 For All Residue Samples (Except Aqua Regia Insoluble Samples) Known to Contain Negligible Amounts of Fluoride.	
NOTE: Analyze fluoride free samples following Section 6.6, but use a 125 mL Erlenmeyer flask with a funnel in Step 6.6.1 instead of platinum beaker.	
6.8 For Aqua Regia Insoluble (A.R.I.) Samples	
NOTE: Aliquot and dilution volumes given below and in Table II (Attachment F), are tentative and may change as the sample composition varies.	
6.8.1 Weigh about 0.5 gram of sample and record the weight to the nearest milligram.	
6.8.2 Transfer the sample to a 125 mL Erlenmeyer flask.	
6.8.3 Add 10 mL of 70% HNO_3 and 10 mL of 70% $HClO_4$ to the flask. Place a pyrex buret funnel in the neck of the flask.	
6.8.4 Place the flask on a hot plate and evaporate the contents to dryness.	
6.8.5 Remove the flask from the hotplate and cool.	
6.8.6 Rinse any residue from the funnel and the inner walls of the flask into the flask with 3M HNO_3. Add several drops of $NaNO_2$ solution to the flask.	

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6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.8.7 Place the flask, without the funnel, back on the hotplate until the evolution of orange/yellow gas stops.	
6.8.9 Transfer the sample to a 25 mL volumetric flask and dilute to volume with distilled water.	
6.8.10 Pipet a suitable aliquot (usually 2 or 5 mL) from the 25 mL flask and analyze according to Section 7.1.	

6.9 For Raffinate Cake Samples

NOTE: Aliquot and dilution volumes given below and in Table II (Attachment F), are tentative and may change as the sample composition varies.

6.9.1 Using the T-handle core sampler, remove two representative portions and add to a clean, tared weighing dish. Weigh to the nearest 0.1 gram. Record the net weight on a data sheet. Transfer the sample quantitatively to a newly cleaned 600 mL beaker.	The combined weight of the two portions should be 20-40 grams.
6.9.2 Add 100 mL of 70% HNO_3 and 100 mL of 38% HCl to the sample in the beaker. Place a watch glass on top of the beaker.	

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6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.9.3 Place the beaker on the hotplate and evaporate the contents to about 100 mL. (This usually takes about an hour if a "Speedy-vap" watch glass is used.)	
6.9.4 Remove the beaker from the hotplate and let the beaker and solution cool to room temperature.	
6.9.5 Rinse any residue or condensate which may have formed on the watch glass into the beaker with distilled water.	
6.9.6 Transfer the solution from the beaker to a 250 mL volumetric flask and dilute to volume with distilled water.	
6.9.7 Pipet an aliquot from the 250 mL volumetric flask (usually 2 or 5 mL) into a 125 mL Erlenmeyer flask.	
6.9.8 Add 5 mL of 70% HNO_3 and 5 mL of 70% $HClO_4$ to the sample in the flask.	

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6.0 SAMPLE PREPARATION (cont.)

PROCEDURE	COMMENTS
6.9.9 Place a Pyrex buret funnel in the neck of the flask, place the flask on a hotplate and evaporate the solution to dryness.	
6.9.10 Remove the flask from the hotplate and cool.	
6.9.11 Rinse any residue from the funnel and the inner walls of the flask into the flask with 3M HNO_3. Add several drops of $NaNO_2$ solution to the flask.	
6.9.12 Place the flask, without the funnel, on the hotplate and heat until the evolution of orange/yellow gas stops.	
6.9.13 Remove the flask from the hotplate and cool.	
6.9.14 Analyze the sample according to Section 7.1.	

7.0 PROCEDURE

NOTE: Before analyzing any standards or samples, see Attachment A, General Information Notes/Start-up Procedure. Change all pump tubes weekly.

PROCEDURE	COMMENTS
7.1 For Low Interference Samples	
7.1.1 Quantitatively transfer the dissolved sample to a culture tube with 3M HNO_3.	Refer to Section 6.0 for dissolution instructions.
7.1.1.1 Write the dilution used on each tube.	

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.1.1.2 Analyze a blank in the same manner as the samples are analyzed, substituting 3M HNO_3 for the sample.	
7.1.2 Add 10 mL of extracting solution (Reagent 4.1), using a repipet reagent dispenser.	
7.1.3 Add 1 mL of ascorbic acid solution, using a repipet reagent dispenser.	
7.1.4 Add 5 mL of TOPO-cyclohexane solution, using a repipet reagent dispenser.	Extracting volumes of TOPO-cyclohexane should be 5 mL only. The concentration of uranium extracted into the TOPO-cyclohexane should not exceed 30.0 ug mL^{-1}.
7.1.5 Cap the tube and shake it for 1 minute on the Burrell Shaker.	
7.1.6 When the phases separate, place the tube in position on the sampler.	
7.1.6.1 Set the timers for 2.0 minutes "sample" and 4.5 minutes "total".	
7.1.6.2 Adjust the solution level with 3M HNO_3 so that the sample is taken from the organic layer.	
7.1.6.3 Turn on the pump 5 minutes prior to starting the sampler.	

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.1.7 The prompt message on the CRT should read ENTER 0 TO RUN CALIBRATION FACTORS, ENTER 1 TO RUN UNKNOWNS. Press 1 then ENDLINE.	Standardization, Section 8.0 of this procedure, must be completed before attempting to analyze samples. The standardization procedure is to be done at the beginning of each shift.
7.1.8 Te prompt message on the CRT now reads ENTER NUMBER OF SAMPLES TO BE RUN. Press the numeric key corresponding to the number of samples to be run and press the ENDLINE key.	Do not analyze more than 6 samples per run.
7.1.9 The prompt message on the CRT now reads ENTER LAB NUMBERS OF SAMPLES IN ORDER THAT THEY WILL BE ANALYZED. Press the numeric keys corresponding to the sample lab number and press ENDLINE for each sample.	Example: for lab number 10.402, press 10.0402 and ENDLINE.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.1.10 The prompt message on the CRT now reads ENTER NUMBER (MAX. OF 4) OF DILUTIONS FOR SAMPLE ____. (The lab number will be displayed in the blank.) Press 1,2,3, or 4 then press ENDLINE.	Examples: If 2 mL of sample is extracted into 5 mL of TOPO-cyclohexane (see Step 7.1.4), the dilution is 2-5, or 1 dilution. For sample diluted 10-100-2-5 there are 2 dilutions. If a sample has been weighed and dissolved for analysis, consider the first aliquot to be 1 mL. For example, if 0.2500 g of sample is dissolved and then diluted to 100 mL, from which 2 mL are taken and extracted into 5 mL of TOPO-cyclohexane for analysis, then the dilution is 1-100-2-5, or 2 dilutions. The sample weight will be accounted for in Step 7.1.12.
7.1.11 For each sample, after the number of dilutions is entered, the CRT will display one of the following messages (7.1.11.1 - 7.1.11.4), depending on the entry made in Step 7.1.10 above. Press the numeric keys corresponding to the proper aliquot and dilution volumes and press ENDLINE, in response to the applicable CRT message.	
7.1.11.1 If the number of dilutions entered is 1, the message will read ENTER ALIQUOT (1), VOLUME (1).	Example: If 2 of a sample is diluted to 5 mL, the number of dilutions = 1, Aliquot (1) = 2 mL, and Volume (1) = 5 mL. Press 2,5 ENDLINE. The printout will display 2-5.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.1.11.2 If the number of dilutions entered is 2, the message will read ENTER ALIQUOT (1), VOLUME (1), A(2), V(2).	
7.1.11.3 If the number of dilutions entered is 3, the message will read ENTER ALIQUOT (1), VOLUME (1), A(2), V(2), A(3), V(3).	
7.1.11.4 If the number of dilutions entered is 4, the message will read ENTER ALIQUOT (1),VOLUME (1), A(2), V(2), A(3), V(3), A(4), V(4).	Example: If the number of dilutions = 4, Aliquot (1) = 10 mL, Volume (1) = 100 mL, A(2) = 5 mL, V(2) = 100 mL, A(3) = 5 mL, V(3) - 50 mL, A(4) = 5 mL, V(4) = 5 mL, press 10,100,5,100,5,50,5,5 ENDLINE. The printout will display 10-100-5-100-5-50-5-5.
7.1.12 After all dilutions are entered, the CRT prompt message will read TYPE CONCENTRATION UNITS; ENTER 1 FO U gL^{-1}, 2 FOR U #/GAL., 3 FOR U PPM, 4 FOR % U, AND 5 FOR TOTAL U mg. Each lab number entered at Step 7.1.9 will be brought up one at a time on the CRT. Press the numeric key (1,2,3,4, or 5) corresponding to the desired concentration units, then press ENDLINE.	The entries #1 and 2, for U g/L^{-1}, and U#/GAL, will also print U mg/L^{-1}. Entry #5 for Total U mg is to be used for Stack Filters.

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.1.12.1 If key 3 (U ppm) or key 4 (%U) is pressed, a further input is required for the sample. The prompt message on the CRT will read ENTER WEIGHT N GRAMS OF SAMPLE. Simply type in the weight and press ENDLINE.	
7.1.13 After the concentration units for the last sample have been selected, the CRT will display the following message: TURN PUMP AND RECORDER ON AND ALLOW TO COME TO EQUILIBRIUM. Do as instructed.	It should take no more than 5 minutes for the system to come to equilibrium (indicated by a steady baseline). If equilibrium is not obtained in 5 minutes, consult the supervisor.
7.1.14 The CRT will now read ENTER 1.0 AS SOON AS SAMPLER IS INITIATED. If the system has come to equilibrium, press the START button on the sampler and immediately press 1.0 and then ENDLINE.	
7.1.15 During the sample run, the computer will present the following information for each sample:	Example: Attachment I, Sample Data Sheet.
7.1.15.1 LAB NUMBER.	
7.1.15.2 DILUTIONS (aliquots and volumes).	
7.1.15.3 DILUTION FACTOR.	

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7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.1.15.4 BASE LINE.	
7.1.15.5 PEAK HEIGHT.	
7.1.15.6 CORRECTED % ABS (which is peak height less base line ± Blank). If corrected % ABS is greater than 230, the sample must be diluted or a smaller aliquot taken. If corrected % ABS is less than 10 and dilution factor is 10 or more, (for example 5-50-5-5), a larger aliquot must be taken.	All % ABS readings appearing in this method mean percent of full scale absorbance. Compare the strip-chart recording of the sample to the computer printout. If they don't agree within 2.0% ABS units, then the concentration must be calculated manually (see section 9.0), and the strip-chart recording must be attached to the data sheet before the data are submitted for approval.
7.1.15.7 WEIGHT OF SAMPLE (if specified).	
7.1.15.8 FACTOR (calibration factor).	
7.1.15.9 CONCENTRATION IN... (units specified in Step 7.1.12).	
7.1.16 After all samples have been analyzed in this run, the CRT will display the same message as stated in Step 7.1.7: ENTER 0 TO RUN CALIBRATION FACTORS, ENTER 1 TO RUN UNKNOWNS.	Refer to Step 5.4 for disposal of Hazardous Material.

7.2 For Samples Containing More Than 10 Grams per Liter Thorium

7.2.1 Transfer a dissolved sample that contains 2.0 ± 0.1 grams of thorium to a 100 mL volumetric flask.	Perform Section 8.2 before analyzing any samples. See Section 6.3 for dissolution instructions.

7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.2.2 Add 50 mL of HF-HNO_3 mixed acid solution (Reagent 4.10) to the flask.	This procedure is designed for 2 grams of thorium. If less thorium is used, the volume of mixed acid must be adjusted proportionately.
7.2.3 Add 5 mL of TOPO-cyclohexane solution, using a repipet reagent dispenser.	
7.2.4 Add 2 mL of ascorbic acid solution, using a repipet reagent dispenser.	
7.2.5 Stopper the flask and shake it for 2 minutes.	
7.2.6 Add 3 M HNO_3 to the flask until the organic layer is brought into the neck of the flask.	
7.2.7 Transfer the organic to a culture tube.	Use a pipet to transfer the organic.
7.2.7.1 Write the dilution used on the tube.	
7.2.8 Add 10 mL of extracting solution to the tube, using a repipet reagent dispenser.	
7.2.9 Proceed with Steps 7.1.5 through 7.1.16 of this method.	

7.3 For Samples Containing More than 10 Grams per Liter Zirconium (ZIRNLO) and for Lime Slurry Samples.

7.3.1 Transfer a dissolved sample containing no more than 0.05 gram of zirconium into a 60 mL separatory funnel.	See Section 6.See Section 6.1 for dissolution instructions.

R - MATERIAL REVISED, ADDED, OR DELETED.

7.0 PROCEDURE (cont.)

PROCEDURE	COMMENTS
7.3.2 Add 10 mL of extracting solution, using a repipet reagent dispenser.	
7.3.3 Add 1 mL of ascorbic acid solution, using a repipet reagent dispenser.	
7.3.4 Add 5 mL of TOPO-cyclohexane solution, using a repipet reagent dispenser.	
7.3.5 Stopper the funnel and shake it for 2 minutes.	
7.3.6 Discard the aqueous layer and repeat Steps 7.3.2, 7.3.3, and 7.3.5. Then proceed with Step 7.3.7.	Omit Step 7.3.4.
7.3.7 Transfer the organic to a culture tube.	Use a pipet to transfer the organic.
7.3.7.1 Write the dilution used on the tube.	
7.3.8 Add 10 mL of extracting solution to the tube, using a repipet reagent dispenser.	
7.3.9 Add 1 mL of ascorbic acid solution, using a repipet reagent dispenser.	
7.3.10 Cap the tube and shake it for 2 minutes on the Burrell Shaker.	
7.3.11 Proceed with Steps 7.1.6 through 7.1.16.	

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8.0 STANDARDIZATION

NOTE: Standardization must be performed at the beginning of each shift. See Attachment A, for Start-Up Procedure.

PROCEDURE	COMMENTS
8.1 For Sections 7.1 and 7.3	
8.1.1 Pipet appropriate aliquots of the 0.025 g/L^{-1} calibration standard into culture tubes.	Use 0, 1, 2, 3, and 5 mL aliquots (the aliquot of 10 mL is for a blank). The computer program assumes these aliquots are used.
8.1.2 Add 5 mL of TOPO-cyclohexane to each tube, using a repipet reagent dispenser.	
8.1.3 Add 10 mL of extracting solution to each tube, using a repipet reagent dispenser.	
8.1.4 Add 1 mL of ascorbic acid solution to each tube, using a repipet reagent dispenser.	
8.1.5 Cap the tubes and shake for 1 minute on the Burrell Shaker.	
8.1.6 When the phases separate, place the tube in position on the sampler.	
8.1.6.1 Set the timers for 2.0 minutes "sample" and 4.5 minutes "total".	

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8.0 STANDARDIZATION (cont.)

PROCEDURE	COMMENTS
8.1.6.2 Adjust the solution level with 3M HNO_3 so that the sample is taken from the organic layer.	
8.1.6.3 Turn on the pump 5 minutes prior to starting the sampler.	
8.1.7 Turn on the power to the HP-85 and then insert the tape cassette that has a program entitled "LOW U" into the HP-85.	
8.1.8 Press the LOAD key and type "LOW U" (include quotation marks) then press ENDLINE.	This operation erases the previous program from the computer's memory and places the program entitled "LOW U" into the current memory. The indicator light under the tape drive will go out when the program is loaded into the computer's memory.
8.1.9 Press the RUN key.	
8.1.10 The following prompt message will appear on the CRT: ENTER YOUR LAST NAME, FIRST INITIAL. Type in your last name, first initial, then press ENDLINE.	EXAMPLE: Name: John Doe Entry: Doe, J ENDLINE The comma must be typed in.
8.1.11 The prompt message on the CRT will now read ENTER TODAY'S DATE (in form of July 26, 1983). Type in today's date as demonstrated and then press ENDLINE.	The name and date entered in Steps 8.1.10 and 8.1.11 will be printed with each result from the computer until standardization is performed again.

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8.0 STANDARDIZATION (cont.)

PROCEDURE	COMMENTS
8.1.12 The prompt message on the CRT will now read ENTER 0 TO RUN CALIBRATION FACTORS, ENTER 1 TO RUN UNKNOWNS. Press 0, then ENDLINE.	
8.1.13 The prompt message will now read ENTER CONCENTRATION OF STANDARD USED FOR FACTORS (U g/L^{-1}). Type in the concentration in g/L^{-1} units, then press ENDLINE.	Example: When concentration of std. is 0.0250 g/L^{-1} U, press 0.0250 ENDLINE.
8.1.14 The prompt message on the CRT will now read PLACE BLANK IN POSITION #1 OF SAMPLER FOLLOWED BY FACTORS. Do as instructed, arranging factors on sampler from least concentrated to most concentrated.	
8.1.15 Turn the pump and recorder on and allow the system to come to equilibrium.	It should take no more than 5 minutes for the system to come to equilibrium (indicated by a steady baseline). If equilibrium is not obtained in 5 minutes, consult the supervisor.
8.1.16 The CRT prompt will now read START SAMPLER AND ENTER 1.0 TO START PROGRAM RUNNING. Press the sampler START button and immediately press 1.0 and then ENDLINE.	Refer to Step 5.4 for disposal of Hazardous Materials.

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8.0 STANDARDIZATION (cont.)

PROCEDURE	COMMENTS
8.1.17 The computer will then determine and print the reagent blank, 4 factors, and 3 break points.	See Section 9.0 for details of the determinations of blank, factors, and break points. See Attachment B, for instructions on how to cross-check factors.
8.1.18 When standardization is complete, the computer will return to the prompt message as stated in Step 8.1.12, ready to analyze unknowns or rerun factors.	See Attachment C, for instructions on how to enter factors manually. See Attachment D, for instructions on how to reinitialize program while retaining old factors.
8.2 Standardization for Section 7.2	
8.2.1 Pipet 5 mL of the thorium calibration factor standard (Reagent 4.12.1) into a 100 mL volumetric flask.	
8.2.2 Pipet 5 mL of the thorium blank solution (Reagent 4.12.2) into a second 100 mL flask.	
8.2.3 Proceed with Steps 7.2.2 through 7.2.9, for both the blank and the standard.	Refer to Step 5.4 for disposal of Hazardous Material.

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9.0 CALCULATIONS

NOTE: When the HP-85 is interfaced with the AutoAnalyzer, only the corrected reading from the computer and the dilution factor need be cross-checked. See Attachment B.

9.1 For Sections 7.1 and 7.3

Let:

R = Uncorrected reading for sample solution (%ABS)
D = Uncorrected reading for reagent blank (%ABS)
L = Base line reading (%ABS)
B = Corrected reading for reagent blank in (%ABS)
S = Total correction for base line and blank (%ABS)
A = Reading of sample solution corrected for S (%ABS)
C = Uranium concentration of standard (mg/mL^{-1})
F = Factor for converting reading to U mg/mL^{-1} ($Umg\%ABS^{-1}\ mL^{-1}$)
T = Break point (%ABS)
Z = Dilution factor for pipetted aliquot
Y = Dilution factor for weighed aliquot
v = Initial volume of sample (mL)
v_1 = First diluted volume (mL)
a_1 = First aliquot (mL)
v_2 = Second volume (mL)
w = Weight of aliquot (grams)

Then:

9.1.1 B = D - L

Examples:

L = 1.0
D = 0.0
B = 0.0 - 1.0 = -1.0

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9.0 CALCULATIONS (cont.)

9.1.2 $S = L + B$

Examples:

(a) $L = 1.5$
$B = 1.0$
$S = 1.5 + 1.0 = 2.5$

(b) $L = 1.5$
$B = -1.0$
$S = 1.5 - 1.0 = 0.5$

9.1.3 $A = R - S$

Examples:

$R = 25.5$
$S = 0.5$
$A = 25.5 - 0.5 = 25.0$

9.1.4 $F = \frac{C}{A}$

Examples:

$C = 0.005\ mgmL^{-1}$
$A = 38.4$

$$F = \frac{0.0050}{38.4} = 0.000130$$

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9.0 CALCULATIONS (cont.)

9.1.5 $T(n) = \frac{A(n) + A(n+1)}{2}$

Examples:

A(1) = 38.4 = corrected reading for first standard

A(2) = 75.4 = corrected reading for second standard

T(1) = first break point = $\frac{38.4 + 75.4}{2}$ = 56.9

NOTE: Break points are used to compensate for the slight nonlinearity of colorimeter response vs uranium concentration (i.e. - the four factors won't be identical). In reference to the example above, the factor (F) calculated with A(1) would be used for any samples with $A \leq 56.9$, and the factor (F) calculated with A(2) would be used for any samples with $A > 56.9$ and $\leq T(2)$.

9.1.6 $Z = \frac{v_1 \times v_2}{v \times a_1}$

Examples:

v = 2.00 mL
v_1 = 5.00 mL

$Z = \frac{5}{2} = 2.5$

9.1.7 $Y = \frac{v_1 \times v_2}{w \times a_1}$

Examples:

w = 1.00 g
v_1 = 100 mL
a_1 = 5.00 mL
v_2 = 5.00 mL

$Y = \frac{100 \times 5}{1.00 \times 5} = \frac{500}{5} = 100\ mL/g^{-1}$

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9.0 CALCULATIONS (cont.)

9.1.8 U g/L^{-1} = FAZ

Examples:

F = 0.000132
A = 20.0
Z = 2.5

U g/L^{-1} = 0.000132 x 20.0 x 2.5 = 0.007

9.1.9 U ug/mL^{-1} = 1000 FAZ

Examples:

F = 0.000132
A = 20.0
Z = 2.5

U ug/mL^{-1} = 1000 x 0.000132 x 20.0 x 2.5 = 6.6

9.1.10 $\% U = \frac{FAY}{10}$

Examples:

F = 0.000132
A = 40.0
Y = 100 mL/g^{-1}

$\% U = \frac{0.000132 \times 40.0 \times 100}{10} = 0.053$

9.1.11 U ppm = 1000 FAY

Examples:

F = 0.000132
A = 40.0
Y = 100 mL/g^{-1}

U ppm = 1000 x 0.000132 x 40.0 x 100 = 528

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9.0 CALCULATIONS (cont.)

9.2 For Section 7.2

9.2.1 U ppm (Th basis) $= \frac{1{,}000{,}000\ FAZ}{Th}$

Where: Th = Thorium g/L^{-1}

Examples:

F = 0.000132
A = 15.0
Z = 1.0
Th = 250 g/L^{-1}

U ppm (Th basis) $= \frac{1{,}000{,}000 \times 0.000132 \times 15.0 \times 1.0}{250} = 7.9$

9.2.2 U ppm (Th basis) $= 100{,}000\ \frac{FAY}{Th}$

Where: Th = % Th

Example:

F = 0.000132
A = 10.0
Y = 5
Th = 87.5%

U ppm (Th basis) $= \frac{100{,}000 \times 0.000132 \times 10.0 \times 5}{87.5} = 7.5$

10.0 PRECISION AND ACCURACY

10.1 The limits of detection for this method are 0.0001 in g/L^{-1}, 0.000001 in #/gal, 0.001 in %, and 0.1 in ppm.

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10.0 PRECISION AND ACCURACY (cont.)

10.2 The table below lists the precision which may be expected for a production sample analysis, based on the analysis of "blind" duplicates over an extended time period.

PRECISION TABLE

SAMPLE TYPE	DETERMINATION	NO. OF SAMPLES	± LEX 95% C.L.	AVERAGE CONC. LEVEL
Sump "E"	U Col (<0.11 g/L)	138	0.004	0.024
Sump "E"	U Col (0.11-0.50 g/L)	166	0.032	0.27
Sump "E"	U Col (0.51- 1.00 g/L)	148	0.061	0.71
Sump "E"	U Col (1.01- 5.00 g/L)	205	0.19	2.03
Residues	U Col Br PADP (%)	48	0.70	2.90

11.0 QUALITY ASSURANCE

11.1 Analyze a 0.0300 g/L^{-1} Uranium Reference Standard at the beginning, middle, and end of each shift, and additionally as the analyst deems necessary. Acceptable values for this standard fall in the range 0.0284 to 0.0316 g/L^{-1} uranium.

11.2 Analyze at least one SAQA control sample during each shift. Analyze additional SAQA unknown samples according to the variety of plant samples received and the availability of corresponding SAQA controls.

11.3 Using a set of weights, check the calibration of the balance each day. Record the performance check in a bound notebook.

11.4 Ensure that service, calibration, and maintenance of the balance are professionally performed at six month intervals.

11.5 Ensure that all calculations are cross-checked for accuracy before reporting results.

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12.0 REFERENCES

12.1 T.M. Florence, AAEC/Tm 552, Proceedings of a Symposium on the Analytical Chemistry of Uranium and Thorium, 1970.

12.2 J.R. Prall, NLCO-1091, The Semi-Automatic Determination of Traces of Uranium, 1972.

12.3 C.A. Horton and J.C. White, Separation of Uranium by Solvent Extraction with Tri n-octylphosphine Oxide, Analytical Chemistry, Vol. 30, 1958.

12.4 Hewlett Packard, HP-85 Owner's Manual and Programming Guide, 1979.

12.5 Hewlett Packard, Operations and Service Manual for Measurement System 3470 (DVM).

12.6 V.R. Gill, The Interfacing of Technicon AAII Unit to HP-85 Personal Computer to Determine Uranium concentrations by Br PADAP Colorimetric Method, March 1984.

12.7 Hewlett Packard, HP-82941A BCD Interface Owner's Manual, Series 80, 1082.

13.0 ATTACHMENTS

13.1 Attachment A - General Information Notes/Start-Up Procedure

13.2 Attachment B - Cross-Checking Factors

13.3 Attachment C - Manual Entry of Factors

13.4 Attachment D - Reloading Program

13.5 Attachment E - Table I (AQUEOUS SAMPLE PREPARATION)

13.6 Attachment F - Table II (SOLID SAMPLE PREPARATION)

13.7 Attachment G - Table III (PUMP TUBES AND INSTRUMENT SET-UP)

13.8 Attachment H - List of All Steps in Program Entitled "Low U"

13.9 Attachment I - Sample Data Worksheets

13.10 Attachment J - Structure of Br PADAP

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1.0 RECORDER AND DVM ADJUSTMENTS

PROCEDURE	COMMENTS
1.1 Turn the recorder, colorimeter, pump, DVM, and HP-85 ON at least 15 minutes prior to making adjustments.	
1.2 Rotate the 5 position colorimeter switch to ZERO. Turn the recorder chart drive ON.	
1.3 If necessary, adjust the recorder to zero with the ZERO ADJUST potentiometer on the recorder. (If the DVM does not read 0 ± 0.5, contact the Electronics Laboratory.)	
1.4 Rotate the 5 position switch on the colorimeter to FULL SCALE.	
1.5 If necessary, adjust the FULL SCALE potentiometer until the recorder pen reaches 100%. Adjust DVM to read 100.0 by turning the adjustment screw on the ATTENUATOR BOX that connects the DVM to the colorimeter.	Recheck zero and full scale after making adjustments.
1.6 Rotate the 5 position switch on the colorimeter to NORMAL.	If signals are excessively noisy on normal setting, use the DAMP I setting.
1.7 If necessary, adjust the BASE-LINE potentiometer to about 5% of full scale.	
1.8 Adjust the recorder to read about 95% of full scale when a 15.0 ug/mL^{-1} standard has reached its peak.	Use the STD CAL potentiometer of the colorimeter to make the adjustment.

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1.0 Compare corrected % ABS. (A) from the Computer to the corresponding strip chart recording. If they agree within ± 2.0, then the computer printout reading is acceptable. If they don't agree within ± 2.0, then check to make sure the timer is set at 2.0 minutes for sampling and 2.5 minutes for washing (4.5 minutes total). If the timer is set properly, consult the supervisor.

1.0 If the analyst chooses to enter a blank, 4 factors, and 3 break points manually, without actually analyzing them again, the following procedure must be followed. (This might be necessary following a power outage, when the analyst makes an incorrect entry, or when a blank or factor is found to be different than the value calculated by the computer.)

1.1 Press STEP/PAUSE.

1.2 Press RUN.

1.3 Type in your name as requested by the prompt message.

1.4 Press ENDLINE.

1.5 Type in today's date as requested by the prompt message.

1.6 Press ENDLINE.

1.7 The prompt message will now read ENTER 0 TO RUN CALIBRATION FACTORS, ENTER 1 TO RUN UNKNOWN. Press STEP/PAUSE.

1.8 Enter the following set of data, filling in the blanks with the values that were being used before the need for manual factor entry arose, or with the manually calculated values.

B = ____ ENDLINE
F(1) = ____ ENDLINE
F(2) = ____ ENDLINE
F(3) = ____ ENDLINE
F(4) = ____ ENDLINE
T(1) = ____ ENDLINE
T(2) = ____ ENDLINE
T(3) = ____ ENDLINE

NOTE: Calculations to determine the blank, factors, and break points are in Section 9.0.

1.9 Press SCRATCH/CONT.

MANUAL ENTRY OF FACTORS
Attachment C

1.10 Press SHIFT and then COPY. A Copy of the data entered in Step 1.8 will be printed. Have this copy cross-checked and attach it to the appropriate Calibration/Maintenance log book.

1.11 Now you are ready to respond to the prompt message ENTER 0 TO RUN CALIBRATION FACTORS, ENTER 1 TO RUN UNKNOWN. Press 1 then ENDLINE.

1.0 In some cases following erroneous data entry, the analyst may want to go back to the start of the program, without having to restandardize or manually enter the factors (as outlined in Attachment C). To restart the program while retaining the factors that were in use, perform the following steps.

1.1 Press STEP/PAUSE.

1.2 Type CHAIN "LOW U" and press ENDLINE.

This action will reload the program entitled LOW U into memory and start the program running while at the same time retaining the blank, factors, and break points.

1.3 Now your are ready to respond to the prompt message, ENTER 0 TO RUN CALIBRATION FACTORS, ENTER 1 TO RUN UNKNOWNS.

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Plant No.	Sample Type	Determination	Filter	Sample mL	70% HNO_3 mL	70% $HClO_4$ mL
1	Sump Liquor	Total U	no	5	5	5
2	AR & Raffinate	Extractable U	no	2	**none	**none
2	AR & Raffinate	Total U	no	2	5	5
2	Filtrate	Total U	no	5	5	5
2	Slop	Total U	no	2	5	5
2	Sump Liquor	Total U	no	5	5	5
2	Cake Slurry	Soluble	yes	1-2-5-100-2	5	**none
2	Cake Slurry	Total U	no	1-2-5-100-2	5	5
2	ZIRNLO	Total U	no	2-100-5	10	**none
4	Filtrate	Total U	no	1	5	**none
4	HF*	Total U	no	1*	5	5
5	Sump Liquor	Total U	no	2	5	5
6	Sump Liquor	Total U	no	5	5	5
8	Floor Water	Total U	no	2	5	5
8	Filtrate	Total U	no	5	5	5
9	Sump Liquor	Total U	no	5	5	5
9	Lime Slurry**	Total U	no	2-100-5	10	**none
10	Tank Sump Liquor	Total U	no	5	5	5
10	Heel	Total U	no	2-100-5	5	5
10	Pit Pumping	Soluble U	yes	5	5	**none
10	Pit Pumping	Total U	no	5	5	5
[illegible]	River Pumping	Total U	no	10	5	5
[illegible]	[illegible]contamination Sump Liquor	Total U	no	2	5	5
10	Tracer (Plt. 2)	Total U	no	5	5	5
10	Tracer (Lab.)	Total U	no	5	5	5
10	Tracer (Plt. 6)	Total U	no	5	5	5
10	Tracer (Decontam.)	Total U	no	2	5	5
10	Tracer (Pilot Plt.)	Total U	no	5	5	5
10	Tracer (Plt. 5)	Total U	no	5	5	5
10	Tracer (Tank Farm)	Total U	no	5	5	5
13	AR	U g/L	no	5	5	5

*Use polyethylene pipet and fume in a platinum dish.
**It has been determined that acid is not needed or that acid might interfere with the analysis.

TABLE
(AQUEOUS SAMPLE PREPARATION)
Attachment E

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	SOIL	Mg F_2 and Residue With Fluorine	Residue Without Fluorine	AQUA REGIA INSOLUBLE	Raffinate Cake
Sample Size (grams)	10	0.5 for -35 fraction, entire sample for +35 fraction.	0.5 for -35 entire sample for +35	0.5	20 to 40
Dissolution Reagents	50 mL HNO_3 50 mL $HClO_4$	10 mL HNO_3 35 mL $HClO_4$	10 mL HNO_3 35 mL $HClO_4$	10 mL HNO_3 10 mL $HCLO_4$	100 mL HNO_3 100 mL HCl
Evaporate to _____ mL	25	to dryness	to dryness	to dryness	100 mL
Filtering Required?	No	No	No	No	No
Dilute to _____ mL	100	100	100	25	250
Normal Aliquot Volume (mL)	5	2 or 5	2 or 5	2 or 5	2 or 5
Fume Aliquot in $HNO_3/HClO_4$	Yes	No	No	No	Yes
NOTES:	Use glassware set aside for soil samples only.	Use 100 mL platinum beaker for dissolution.	Use 125 mL Erlenmeyer flask with funnel for dissolution.		Use T-handle core sampler.

TABLE II
SOLID SAMPLE PREPARATION
Attachment F

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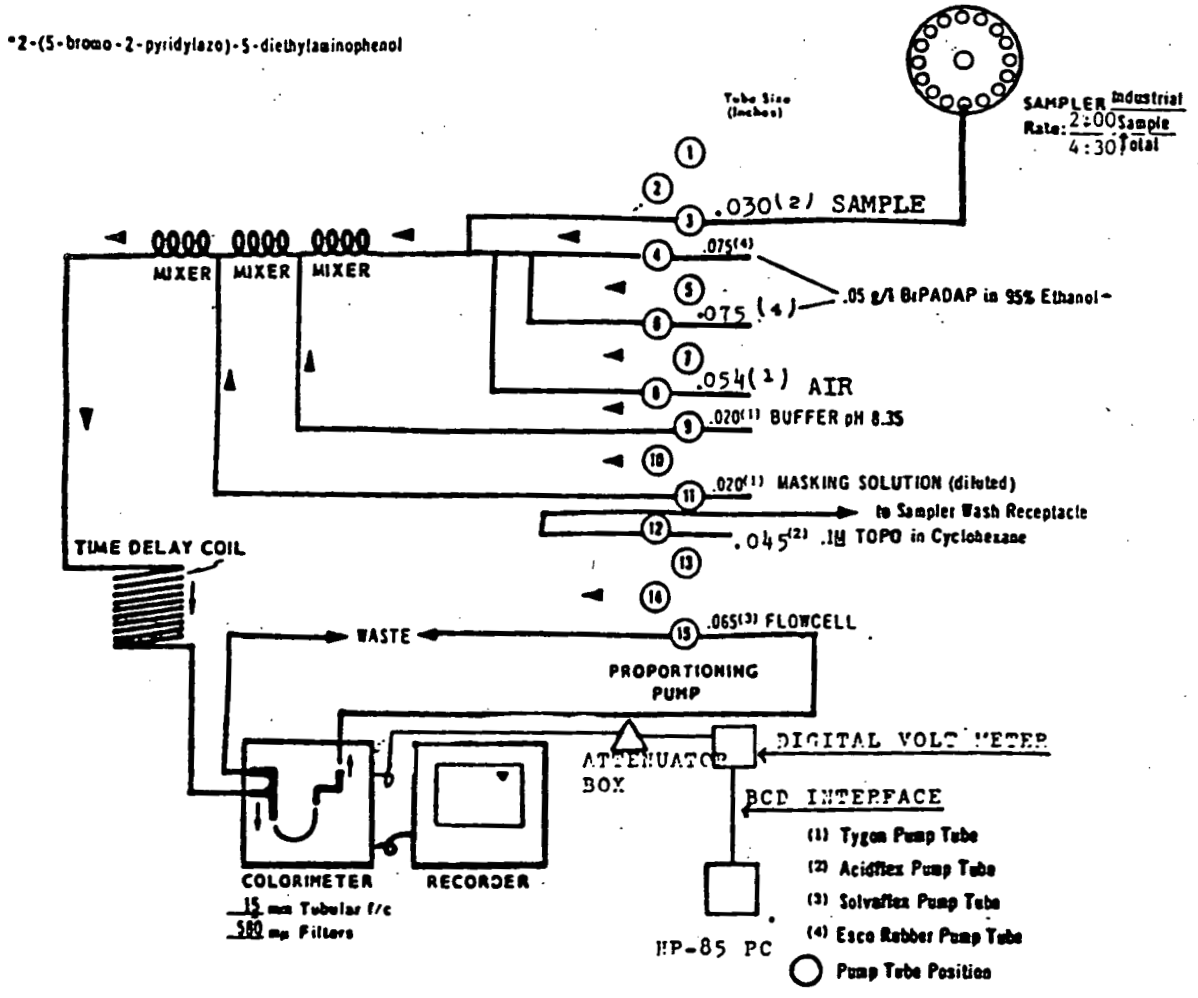

TABLE III
(PUMP TUBES AND INSTRUMENT SET-UP)
Attachment G

R - MATERIAL REVISED, ADDED, OR DELETED.

LIST OF ALL STEPS IN PROGRAM ENTITLED "LOW U"

```
 10 DISP "ENTER YOUR LAST NAME,
                    FIRST INIT
    IAL"
 20 INPUT A$,C$
 30 DISP "ENTER TODAY'S DATE(IN
    FORM OF     JULY 25,1983)"
 40 INPUT B$,A
 50 REM PROGRAM FOR LOW ASSAY CO
    LORIMETRIC URANIUM ANALYSIS
 60 DISP "ENTER 0 TO RUN CALIBRA
    TION FACTORS,ENTER 1 TO RUN
    UNKNOWNS."
 70 INPUT K@ IF K=0 THEN GOTO 26
    0
 80 IF K=1 THEN GOTO 830
 90 REM Y=BASE LINE,X=PEAK HEIGH
    T
100 REM Z=NO. OF FACTORS @ REM R
    =CONCENTRATION OF STD. USED(
    U G/L)
110 REM I&U ARE COUNTERS
120 REM P=ALIQUOT
130 REM H=VOLUME
140 REM E=NO. OF DILUTIONS
150 REM L=START PROGRAM RUNNING
    INICATOR.
160 REM S=X-Y-B @ REM F=FACTOR(M
    G/ML-% TRANS.)
170 REM T=RANGE OF TRANSMITTANCE
     FOR EACH FACTOR
180 REM V=NO. OF SAMPLES TO BE R
    UN
190 REM C()=LAB NO.
200 REM D()=DILUTION FACTOR
210 REM Z()=TYPE OF CONC. UNITS
220 REM W()=WEIGHT OF SAMPLE
230 REM G()=CIRRECTED % ABS.   I
    NCLUDING BLANK
240 REM C=CONC. OF UNKNOWN @ B=B
    LANK@J=COMPARISON NUMBER
250 REM M=G/L*.00835=@/GAL@ REM
    K=BRANCHING DEVICE
260 DISP "TURN ON PUMP AND RECOR
    DER OF TECHNICON UNIT."
270 CONTROL 3,6 ; 4@ CONTROL 3,2
     ; 0@ Z=4
280 PRINT "NO. OF FACTORS=";Z
290 DISP "ENTER CONCENTRATION OF
     STANDARD USED FOR FACTORS(U
     G/L)"
300 COM F(4)
310 DIM A(4)
320 DIM Y(125)
330 DIM X(150)
340 COM B
350 INPUT R@ DISP "CONC. OF STD.
     (U G/L)=";R @ PRINT "CONC.
    OF STD. (U G/L)=";R @ PRINT
360 A(1)=5 @ A(2)=2.5 @ A(3)=1.6
    7 @ A(4)=1
370 DISP "PLACE BLANK IN POSITIO
    N #1 OF SAMPLER FOLLOWED BY
    FACTORS."
380 DISP "START SAMPLER AND ENTE
    R 1.0 TO START PROGRAM RUNNI
    NG." @ INPUT L
390 DISP "BLANK DETERMINATION" @
     PRINT "BLANK DETERMINATION"
400 GOSUB 2600
410 DISP "BASE LINE=";-Y
420 PRINT USING 1700 ; -Y
430 GOSUB 2690
440 DISP "PEAK HEIGHT=";-X @ DIS
    P
450 PRINT USING 620 ; -X
460 LET B=ABS(X-Y)
470 IF X<Y THEN GOTO 490
480 LET B=B*-1
490 PRINT USING 500 ; B @ PRINT
    @ PRINT
500 IMAGE "BLANK=",DD.D
510 LET I=0
520 I=I+1
530 PRINT USING 540 ; I @ CONTRO
    L 3,6 ; 4
540 IMAGE "FACTOR#",DD.D
550 DISP "FACTOR#",I
560 GOSUB 2790
570 DISP "BASE LINE=";-Y
580 PRINT "BASE LINE=";-Y
590 GOSUB 2950
600 DISP "PEAK HEIGHT=";-X @ DIS
    P
610 PRINT USING 620 ; -X
620 IMAGE "PEAK HEIGHT=",XX,DDD.
    DD
630 LET S(I)=ABS(X-Y)-B
640 PRINT "DILUTION FACTOR=";A(I
    )
650 PRINT "CONC. OF STD. (U G/L)
    =";R
660 PRINT USING 670 ; ABS(S(I))
670 IMAGE "CORRECTED % ABS.   =",
    XX,DDD.DD
680 LET F(I)=R/A(I)/ABS(S(I))
690 PRINT USING 700 ; I,F(I) @ P
    RINT @ PRINT
700 IMAGE "FACTOR #",D,X,D.DDDDD
    D,X,"MG/ML-% ABS.   "
710 IF Z=1 THEN GOTO 60
720 IF I=1 THEN GOTO 820
730 COM T(3)
740 LET T(I-1)=(ABS(S(I))-ABS(S(
    I-1)))/2+ABS(S(I-1))
750 PRINT USING 760 ; I-1,T(I-1)
760 IMAGE "MAX. READING TO USE F
    ACTOR#",X,D,X,"IS",XX,DDD.D,
    "% ABS.   "
770 PRINT @ PRINT
780 IF I=Z THEN GOTO 800
```


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```
790 GOTO 520
800 PRINT "LAST NAME,FIRST INITI
    AL"
810 PRINT USING 820 ; A$,C$
820 IMAGE 10A,1A
830 PRINT "DATE"
840 PRINT USING 850 ; B$,A
850 IMAGE 12A,DDDD
860 PRINT @ PRINT @ PRINT
870 GOTO 60
880 DIM C(10),D(10)
890 DISP "ENTER NUMBER SAMPLES T
    O BE RUN"
900 INPUT V@ DISP "NUMBER SAMPLE
    S=";V @ DISP
910 PRINT "NUMBER SAMPLES=";V @
    PRINT @ PRINT @ LET U=0
920 DISP "ENTER LAB NUMBERS OF S
    AMPLES IN ORDER THAT THEY WI
    LL BE ANALYZED"
930 U=U+1
940 INPUT C(U)
950 IF U=V THEN GOTO 970
960 GOTO 930
970 LET U=0
980 U=U+1
990 DISP "ENTER NUMBER(MAX. OF 4
    )OF DILUTIONS MADE FOR SAMPL
    E";C(U)
1000 INPUT E(U)
1010 IF E(U)=1 THEN GOTO 1100
1020 IF E(U)=2 THEN GOTO 1160
1030 IF E(U)=3 THEN GOTO 1220
1040 DISP "ENTER ALIQUOT(1),VOLU
     ME(1),A(2),V(2),A(3),V(3),A
     (4),V(4)"
1050 INPUT P1(U),H1(U),P2(U),H2(
     U),P3(U),H3(U),P4(U),H4(U)
1060 PRINT "DILUTIONS=";P1(U);"-
     ";H1(U);"-";P2(U);"-";H2(U)
     ;"-";P3(U);"-";H3(U);"-";P4
     (U);"-";H4(U)
1070 D(U)=H1(U)*H2(U)*H3(U)*H4(U
     )/(P1(U)*P2(U)*P3(U)*P4(U))
1080 IF U=V THEN GOTO 1280
1090 GOTO 980
1100 DISP "ENTER ALIQUOT(1),VOLU
     ME(1)"
1110 INPUT P1(U),H1(U)@ PRINT C(
     U)
1120 PRINT "DILUTIONS=";P1(U);"-
     ";H1(U)
1130 D(U)=H1(U)/P1(U)
1140 IF U=V THEN GOTO 1280
1150 GOTO 980
1160 DISP "ENTER ALIQUOT(1),VOLU
     ME(1),A(2),V(2)."
1170 INPUT P1(U),H1(U),P2(U),H2(
     U)@ PRINT C(U)
1180 PRINT "DILUTIONS=";P1(U);"-
     ";H1(U);"-";P2(U);"-";H2(U)
1190 D(U)=H1(U)*H2(U)/(P1(U)*P2(
     U))
1200 IF U=V THEN GOTO 1280
1210 GOTO 980
1220 DISP "ENTER ALIQUOT(1),VOLU
     ME(1),A(2),V(2),A(3),V(3)"
1230 INPUT P1(U),H1(U),P2(U),H2(
     U),P3(U),H3(U)@ PRINT C(U)
1240 PRINT "DILUTIONS=";P1(U);"-
     ";H1(U);"-";P2(U);"-";H2(U)
     ;"-";P3(U);"-";H3(U)
1250 D(U)=H1(U)*H2(U)*H3(U)/(P1(
     U)*P2(U)*P3(U))
1260 IF U=V THEN GOTO 1280
1270 GOTO 980
1280 LET U=0
1290 U=U+1
1300 DISP "TYPE CONCENTRATIION U
     NITS;ENTER 1=UG/L,2=U #/GAL
     ,3=U PPM,4=%U, & 5=TOTAL U
     MG"
1310 DISP "SAMPLE#",C(U)
1320 INPUT Q(U)
1330 IF Q(U)=1 THEN GOTO 1380
1340 IF Q(U)=2 THEN GOTO 1380
1350 IF Q(U)=3 THEN GOTO 1400
1360 IF Q(U)=4 THEN GOTO 1400
1370 IF Q(U)=5 THEN GOTO 1380
1380 IF U=V THEN GOTO 1430
1390 GOTO 1290
1400 DISP "ENTER WEIGHT IN GRAMS
      OF SAMPLE"
1410 INPUT W(U)@ IF U=V THEN GOT
     O 1430
1420 GOTO 1290
1430 DISP "TURN PUMP & RECORDER
     ON & ALLOW TO COME TO EQUIL
     IBRIUM."
1440 DISP "ENTER 1.0 AS SOON AS
     SAMPLER IS INIATED."
1450 INPUT L
1460 BEEP
1470 BEEP
1480 BEEP
1490 LET U=0
1500 U=U+1
1510 PRINT
1520 PRINT @ CONTROL 3,6 ; 4
1530 DISP "SAMPLE #",C(U)
1540 GOSUB 2790
1550 DISP "BASE LINE=";-Y
1560 GOSUB 2950
1570 DISP "PEAK HEIGHT=";-X @ DI
     SP
1580 PRINT USING 1590 ; C(U)
1590 IMAGE "LAB NUMBER=",X,DDDDD,
     DDD.DDDD
1600 IF E(U)=4 THEN GOTO 1660
1610 IF E(U)=3 THEN GOTO 1640
1620 IF E(U)=2 THEN GOTO 1650
```


LIST OF ALL STEPS IN PROGRAM ENTITLED "LOW U"
Attachment H (cont.)

R - MATERIAL REVISED, ADDED, OR DELETED.

```
1630 PRINT "DILUTIONS=";P1(U);"-
     ";M1(U) @ GOTO 1670
1640 PRINT "DILUTIONS=";P1(U);"-
     ";M1(U);"-";P2(U);"-";M2(U)
     ;"-";P3(U);"-";M3(U) @ GOTO
      1670
1650 PRINT "DILUTIONS=";P1(U);"-
     ";M1(U);"-";P2(U);"-";M2(U)
      @ GOTO 1670
1660 PRINT "DILUTIONS=";P1(U);"-
     ";M1(U);"-";P2(U);"-";M2(U)
     ;"-";P3(U);"-";M3(U);"-";P4
     (U);"-";M4(U)
1670 PRINT USING 1680 ; D(U)
1680 IMAGE "DILUTION FACTOR=",X,
     DDDDD.DD
1690 PRINT USING 1700 ; -Y
1700 IMAGE "BASE LINE=",X,DD.DD
1710 PRINT USING 1720 ; -X
1720 IMAGE "PEAK HEIGHT=",X,DDD.
     DD
1730 LET G(U)=ABS(X-Y)-B
1740 PRINT USING 1750 ; G(U)
1750 IMAGE "CORRECTED % ABS. .="
     ,X,DDD.DD
1760 IF Q(U)=5 THEN GOTO 2430
1770 IF Q(U)=1 THEN GOTO 2190
1780 IF Q(U)=2 THEN GOTO 2190
1790 IF Q(U)=3 THEN GOTO 1810
1800 IF Q(U)=4 THEN GOTO 2000
1810 PRINT USING 1820 ; W(U)
1820 IMAGE "WT. OF SAMPLE=",X,DD
     D.DDDD,X,"GRAMS"
1830 IF G(U)<T(1) THEN GOTO 1870
1840 IF G(U)<T(2) THEN GOTO 1880
1850 IF G(U)<T(3) THEN GOTO 1890
1860 LET C=F(4)*G(U)*D(U)*1000/W
     (U) @ PRINT "FACTOR=";F(4)
     @ GOTO 1900
1870 LET C=F(1)*G(U)*D(U)*1000/W
     (U) @ PRINT "FACTOR=";F(1)
     @ GOTO 1900
1880 LET C=F(2)*G(U)*D(U)*1000/W
     (U) @ PRINT "FACTOR=";F(2)
     @ GOTO 1900
1890 LET C=F(3)*G(U)*D(U)*1000/W
     (U) @ PRINT "FACTOR=";F(3)
     @ GOTO 1900
1900 PRINT USING 1910 ; C
1910 IMAGE "CONCENTRATION OF U P
     PM=",X,DDDD.D
1920 PRINT @ PRINT @ PRINT "LAST
      NAME,FIRST INITIAL"
1930 PRINT USING 1940 ; A$,C$
1940 IMAGE 10A,1A
1950 PRINT "DATE"
1960 PRINT USING 1970 ; B$,A
1970 IMAGE 12A,DDDD
1980 IF U=V THEN GOTO 60
1990 GOTO 1500
2000 PRINT USING 2010 ; W(U)
2010 IMAGE "WT. OF SAMPLE=",X,DD
     D.DDDD,X,"GRAMS"
2020 IF G(U)<T(1) THEN GOTO 2060
2030 IF G(U)<T(2) THEN GOTO 2070
2040 IF G(U)<T(3) THEN GOTO 2080
2050 LET C=F(4)*G(U)*D(U)/(10*W(
     U)) @ PRINT "FACTOR=";F(4)
     @ GOTO 2090
2060 LET C=F(1)*G(U)*D(U)/(10*W(
     U)) @ PRINT "FACTOR=";F(1)
     @ GOTO 2090
2070 LET C=F(2)*G(U)*D/U)/(10*W(
     U)) @ PRINT "FACTOR=";F(2)
     @ GOTO 2090
2080 LET C=F(3)*G(U)*D(U)/(10*W(
     U)) @ PRINT "FACTOR=";F(3)
     @ GOTO 2090
2090 PRINT USING 2100 ; C
2100 IMAGE "CONCENTRATION IN % U
     ",X,DD.DDD
2110 PRINT @ PRINT @ PRINT "LAST
      NAME, FIRST INITIAL"
2120 PRINT USING 2130 ; A$,C$
2130 IMAGE 10A,1A
2140 PRINT "DATE"
2150 PRINT USING 2160 ; B$,A
2160 IMAGE 12A,DDDD
2170 IF U=V THEN GOTO 60
2180 GOTO 1500
2190 IF G(U)<T(1) THEN GOTO 2230
2200 IF G(U)<T(2) THEN GOTO 2240
2210 IF G(U)<T(3) THEN GOTO 2250
2220 C=F(4)*G(U)*D(U) @ PRINT "F
     ACTOR=";F(4) @ GOTO 2260
2230 LET C=F(1)*G(U)*D(U) @ PRIN
     T "FACTOR=";F(1) @ GOTO 226
     0
2240 LET C=F(2)*G(U)*D(U) @ PRIN
     T "FACTOR=";F(2) @ GOTO 226
     0
2250 LET C=F(3)*G(U)*D(U) @ PRIN
     T "FACTOR=";F(3) @ GOTO 226
     0
2260 IF C>=.0001 THEN GOTO 2300
2270 PRINT "CONC. IN U G/L=<0.00
     01"
2280 PRINT "CONC. IN U #/GAL=<0.
     000001"
2290 PRINT "CONC. IN U MG/L=<0.1
     "
2300 PRINT USING 2310 ; C
2310 IMAGE "CONC. IN U G/L=",X,D
     D.DDDDD
2320 LET M=C*.00835
2330 PRINT USING 2340 ; M
2340 IMAGE "CONC.IN U #/GAL.=",X
     ,D.DDDDDDD
2342 LET N=C*1000
2344 PRINT USING 2345 ; N
```

LIST OF ALL STEPS IN PROGRAM ENTITLED "LOW U"
Attachment H (cont.)

R - MATERIAL REVISED, ADDED, OR DELETED.

```
2345 IMAGE "CONC. IN U MG/L= ",D
     DD.DD
2350 PRINT @ PRINT @ PRINT "LAST
      NAME,FIRST NAME"
2360 PRINT USING 2370 ; A$,C$
2370 IMAGE 10A,1A
2380 PRINT "DATE"
2390 PRINT USING 2400 ; B$,A
2400 IMAGE 12A,DDDD
2410 IF U=V THEN GOTO 60
2420 GOTO 1500
2430 IF G(U)<T(1) THEN GOTO 2470
2440 IF G(U)<T(2) THEN GOTO 2480
2450 IF G(U)<T(3) THEN GOTO 2490
2460 C=F(4)*G(U)*D(U)*100 @ PRIN
     T "FACTOR=";F(4) @ GOTO 250
     0
2470 C=F(1)*G(U)*D(U)*100 @ PRIN
     T "FACTOR=";F(1) @ GOTO 250
     0
2480 C=F(2)*G(U)*D(U)*100 @ PRIN
     T "FACTOR=";F(2) @ GOTO 250
     0
2490 C=F(3)*G(U)*D(U)*100 @ PRIN
     T "FACTOR=";F(3) @ GOTO 250
     0
2500 PRINT USING 2510 ; C
2510 IMAGE "CONC. IN TOTAL U MG=
      ",DD.DDD
2520 PRINT @ PRINT @ PRINT "LAST
      NAME,FIRST INITIAL"
2530 PRINT USING 2540 ; A$,C$
2540 IMAGE 10A,1A
2550 PRINT "DATE"
2560 PRINT USING 2570 ; B$,A
2570 IMAGE 12A,DDDD
2580 IF U=V THEN GOTO 60
2590 GOTO 1500
2600 ON TIMER# 1,133000 GOTO 266
     0
2610 Y=0
2620 I=0
2630 I=I+1
2640 WAIT 1043 @ ENTER 3 ; Y(I)
2650 Y=Y+Y(I) @ GOTO 2630
2660 OFF TIMER# 1
2670 Y=Y/I
2680 GOTO 410
2690 ON TIMER# 1,154056 GOTO 276
     0
2700 X=0
2710 I=0
2720 I=I+1
2730 WAIT 1043 @ ENTER 3 ; X(I)
2740 X=X(I)+X
2750 GOTO 2720
2760 OFF TIMER# 1
2770 X=X/I
2780 GOTO 440
2790 ON TIMER# 1,123079 GOTO 292
     0
2800 WAIT 1043 @ ENTER 3 ; Y(1)@
      ENTER 3 ; Y(2)
2810 IF ABS(Y(2))<=ABS(Y(1)) THE
     N GOTO 2840
2820 Y=Y(1)
2830 GOTO 2850
2840 Y=Y(2)
2850 LET J=0
2860 J=J+1
2870 WAIT 1043 @ ENTER 3 ; Y(J)
2880 IF ABS(Y(J))<=ABS(Y) THEN G
     OTO 2900
2890 GOTO 2870
2900 Y=Y(J)
2910 GOTO 2870
2920 OFF TIMER# 1
2930 IF K=0 THEN GOTO 570
2940 IF K=1 THEN GOTO 1550
2950 ON TIMER# 1,154056 GOTO 302
     0
2960 WAIT 1043 @ ENTER 3 ; X(1)@
      ENTER 3 ; X(2)
2970 IF ABS(X(2))>=ABS(X(1)) THE
     N GOTO 3000
2980 X=X(1)
2990 GOTO 3010
3000 X=X(2)
3010 LET J=0
3020 J=J+1
3030 WAIT 1043 @ ENTER 3 ; X(J)
3040 IF ABS(X(J))>=ABS(X) THEN G
     OTO 3060
3050 GOTO 3030
3060 X=X(J)
3070 GOTO 3030
3080 OFF TIMER# 1
3090 IF K=0 THEN GOTO 600
3100 IF K=1 THEN GOTO 1570
3110 END
```

LIST OF ALL STEPS IN PROGRAM ENTITLED "LOW U"
Attachment H (cont.)

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SAMPLE DATA SHEETS

ANALYTICAL DATA SHEET

SAMPLE NO.	DIL.	BLANK OR CORR.	FACTOR OR NORM.	O.D. READ OR VOL. TITRANT	% U
#4					
5-1687	0.5031 grams			127.40	4.36

ANALYZED BY: MR
CHECKED BY: RWZ
DATE: 3-4-87

FMPC-T-2274 (REV. 4/15/88)

```
LAB NUMBER= 15.1687
DILUTIONS= 1 - 100 - 5 - 50 - 5
- 5
DILUTION FACTOR=  1000.00
BASE LINE=  4.70
PEAK HEIGHT= 132.10
CORRECTED % ABS.  .= 127.40
WT. OF SAMPLE=    .5031 GRAMS
FACTOR= .000172
CONCENTRATION IN % U  4.356

LAST NAME, FIRST INITIAL
RAMIREZ   M
DATE
MARCH 4      1987
```


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LAB NUMBER= .0300
DILUTIONS= 2 - 5
DILUTION FACTOR= 2.50
BASE LINE= 5.10
PEAK HEIGHT= 68.40
CORRECTED % ABS. .= 61.64
FACTOR= 1.89964932306E-4
CONC. IN U G/L= .02927
CONC.IN U #/GAL.= .0002444
CONC. IN U MG/L= 29.27

LAST NAME,FIRST NAME
PS/CW
DATE
JAN 12 1989

LAB NUMBER= 13.4252
DILUTIONS= 1 - 5
DILUTION FACTOR= 5.00
BASE LINE= 1.10
PEAK HEIGHT= 3.80
CORRECTED % ABS. .= 2.76
WT. OF SAMPLE= 24.9557 GRAMS
FACTOR= 1.52140582616E-4
CONCENTRATION OF U PPM= .1

LAST NAME,FIRST INITIAL
WEHRFRITZ C/MJH
DATE
MAY 23 1989

LAB NUMBER= 89050.2020
DILUTIONS= 10 - 5
DILUTION FACTOR= .50
BASE LINE= 5.00
PEAK HEIGHT= 7.00
CORRECTED % ABS. .= 1.96
FACTOR= 1.37132001834E-4
CONC. IN TOTAL U MG= .0134

LAST NAME,FIRST INITIAL
RAMIREZ M.
DATE
MAY 2 1989

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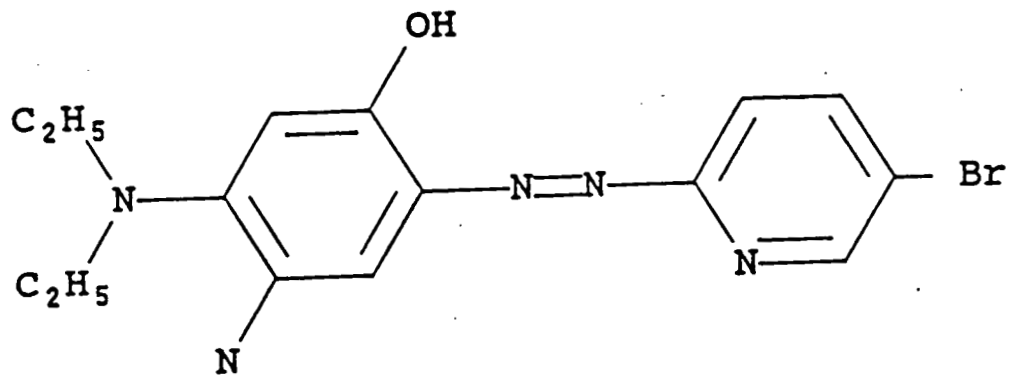
STRUCTURE OF Br PADAP



2-(5-bromo-2-pyridylazo)-5-diethylaminophenol

STRUCTURE OF Br PADAP
Attachment J

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RECORD OF ISSUE/REVISIONS

DATE	REV. NO.	DESCRIPTION AND AUTHORITY
05-84	N/A	V. R. Gill, Original Procedure
05-84	N/A	V. R. Gill
09-86	N/A	V. R. Gill/R. W. Lehrter
10-24-86	N/A	R. W. Lehrter
12-08-88	N/A	R. W. Lehrter
08-22-89	N/A	All pages updated to reflect new format. RCRA procedure included, initiated by D. L. Herman.
06-18-91	1	Original Issue Date is unknown. Revision 1 incorporates CIO C90-077 per Request No. P91-007 and change in document format from "Page Revision Date" to "Revision No." to meet current Site Services Document Program requirements. Authorization individual name for original issue changed to reflect current organization.

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