Test Plan for Demonstration of Rapid Transuranic Monitoring Laboratory

**C. V. McIsaac
C. W. Sill
R. J. Gehrke
E. W. Killian
K. D. Watts**

Published June 1993

**Idaho National Engineering Laboratory
EG&G Idaho, Inc.
Idaho Falls, Idaho 83415**

**Prepared for the
U.S. Department of Energy
Office of Environmental Restoration and Waste Management
Under DOE Idaho Operations Office
Contract DE-AC07-76ID01570**

MASTER

Test Plan for Demonstration of Rapid Transuranic Monitoring Laboratory

EGG-WTD-10758

Prepared by

C. V. McIsaac, Senior Scientist
Radiation Measurements and Development

5-27-93

Date

Reviewed by

P. D. Randolph, Technical Leader,
Radiation Measurements and Development

5-27-93

Date

Approved by

K. M. Kostelnik, Coordinator
Buried Waste Integrated Demonstration

6/10/93

Date

K. L. Martin, Manager
Radiation Measurements and Development

5-27-93

Date

ABSTRACT

This plan describes tests to demonstrate the capability of the Rapid Transuranic Monitoring Laboratory (RTML) to monitor airborne alpha-emitting radionuclides and analyze soil, smear, and filter samples for alpha- and gamma-emitting radionuclides under field conditions. The RTML will be tested during June 1993 at a site adjacent to the Cold Test Pit at the Radioactive Waste Management Complex at the Idaho National Engineering Laboratory. Measurement systems installed in the RTML that will be demonstrated include two large-area ionization chamber alpha spectrometers, an x-ray/gamma-ray spectrometer, and four alpha continuous air monitors. Test objectives, requirements for data quality, experimental apparatus and procedures, and safety and logistics issues are described.

SUMMARY

This test plan describes objectives, instrumentation, and experimental procedures to field test measurement systems installed in the Rapid Transuranic Monitoring Laboratory (RTML), a mobile laboratory developed at the Idaho National Engineering Laboratory (INEL) for use in characterizing radiological source terms at buried radioactive waste remediation sites. The demonstration of the RTML at the INEL is sponsored by the Buried Waste Integrated Demonstration (BWID) Program under Technical Task Plan ID121210, "BWID Contamination Control." The BWID Program develops and demonstrates new technologies for remediating buried waste at facilities throughout the U.S. Department of Energy (DOE) complex. Analytical technologies that will be tested in the field include two large-area ionization chamber alpha spectrometers, an x-ray/gamma-ray spectrometer, and four alpha continuous air monitors (CAMs).

The DOE has a recognized need for analytical technologies that can be used to assess radiological source terms both prior to and during remediation of buried radioactive waste. Since the RTML is housed in a tractor trailer, it can be moved to the remediation site, eliminating the need for shipping samples to distant analytical laboratories. Because the RTML is equipped with only direct spectrometric analysis technologies that require minimal sample preparation, samples can be prepared for analysis and analyzed in less than 1 hour. The mobile laboratory provides a rapid, cost-effective means of characterizing and monitoring radioactive waste remediation sites for alpha- and gamma-emitting contaminants.

During fiscal year 1990 to 1992, a number of promising measurement methods and systems were evaluated for possible use in monitoring transuranic (TRU) concentrations in ambient air and in soil, smear, and dust samples collected during remediation of buried radioactive waste. These evaluations demonstrated that the alpha CAM used at Argonne National Laboratory-West (ANL-W) had a sensitivity for airborne ^{239}Pu 10 to 20 times better than the sensitivities of four commercial alpha CAMs that were also tested. Six of the ANL-W alpha CAMs were fabricated during FY-92, and four of them are now installed in the alpha CAM sampling stations that are operated remotely from the RTML. Direct alpha spectrometry with a large-area ionization chamber and x-ray/gamma-ray spectrometry with a high-resolution thin-window germanium spectrometer were shown to be rapid, sensitive methods for the measurement of TRU isotopes on smears and in soil and dust samples. The two analysis techniques do not require elaborate sample preparation or time-consuming chemical separations; soil samples are only dried and sieved before analysis. Smears and filters are analyzed directly without any sample preparation.

The RTML consists of two separate trailers that are powered from a 50 kW diesel generator. One trailer, 2.4 m wide $\times$ 7.3 m long (8 $\times$ 24 ft), houses the sample preparation laboratory that is equipped with a Class A hood, oven, sieving equipment, analytical balance, and sample containers. Samples will be received and prepared for analysis in this trailer. The second trailer, 2.4 m wide $\times$ 14.6 m long (8 $\times$ 48 ft), houses the computer terminal used to control the operation of the four alpha CAMs that will be installed in the Cold Test Pit: two Ordela large-area ionization chamber alpha spectrometers, a thin-window x-ray/gamma-ray spectrometer installed in an automatic sample changer, a VAX 4000 model 100 computer, and computer terminals and printers used to display and generate reports of analysis results. The RTML will be moved to a location adjacent to the RWMC Cold Test Pit at the INEL and will be demonstrated during June 1993 in conjunction with a remote retrieval demonstration.

Although the field tests are being conducted at the Cold Test Pit and the bulk of the samples will be taken from the vicinity of the Cold Test Pit, some spiked samples of known activity will be introduced into the sample processing stream. The operators in the counting trailer will be unaware of which samples contain known activity concentrations. Plutonium, americium, and gamma-ray emitting radionuclides will be introduced to the process stream. The results of these analyses will be compared to the known values and will be the basis of evaluating the analysis accuracy and precision of counting unknown samples.

Samples that are "free" of radioactivity and those that have been spiked will be counted to demonstrate the capability of the RTML to operate on generator power, acquire quality data, and obtain accurate results (measured activity for the spiked samples and no statistically significant activity for the "cold" samples for all types of samples, i.e., radioactive and nonradioactive and smear, and air filter samples). Since the purpose of this field test does not involve sampling techniques, the source of "nonradioactive" soil samples will not be discussed in this test plan except to acknowledge that soil from the vicinity of the Cold Test Pit will be used to prepare "nonradioactive" samples.

During the demonstration, ambient air over the test pit and samples of soil and dust from the pit will be analyzed for alpha- and gamma-emitting radionuclides using the RTML. The primary objective of the field test of the RTML is to demonstrate and evaluate the capability of the RTML to analyze soil, smear, and filter samples for ^{239}Pu , ^{241}Am , and gamma-emitting radionuclides such as ^{60}Co and ^{137}Cs .

Data quality objectives of the field demonstration of the RTML focus primarily on determining the sensitivity of each measurement system for detection of TRU and/or fission product radionuclides using counting times of 30 minutes or less. Technical performance goals include producing field-deployable measurement systems that have the following sensitivities: (a) the alpha CAM system will have a sensitivity for airborne TRU isotopes that is superior to the sensitivity of 8 derived air concentration (DAC) hours required by DOE Order 5480.11, "Radiation Protection for Occupational Workers," (b) the large-area ionization chamber alpha spectrometer will be capable of analyzing soil samples in 30 minutes or less with a lower limit of detection (LLD) of 50 pCi/g for TRU isotopes, and (c) the x-ray/gamma-ray spectrometer will be capable of analyzing soil samples in 30 minutes or less with LLDs of 50 pCi/g for plutonium, 1 pCi/g for ^{241}Am , and 4 pCi/g for gamma-emitting radionuclides such as ^{60}Co and ^{137}Cs . The software codes used to control the measurement systems and analyze spectral data will reside on a VAX 4000 model 100 computer installed in the RTML analysis trailer.

The definition of lower limit of detection given by Currie^a will be used to calculate the sensitivities of the analytical instrumentation. His equation for paired observations of sample and background with a probability of 0.05 that activity will be detected when none is present or that activity will not be detected when in fact it is present is

a. L. A. Currie, "Limits for Qualitative Detection and Quantitative Determination," *Anal Chem* 40:586-593, 1968.

$$LLD = 2.71 + 4.66 B^{1/2}$$

In terms of concentration in soil, expressed as pCi/g, the equation becomes

$$LLD = \frac{2.71 + 4.66 B^{1/2}}{2.22 \cdot T \cdot \epsilon \cdot Y \cdot M}$$

where

2.22 = number of disintegrations per minute per pCi

B = total number of background counts in the region of interest (counts)

T = counting time (minutes)

ϵ = absolute counting efficiency (counts per disintegration)

Y = fractional recovery in the region of interest

M = mass of sample (g).

CONTENTS

ABSTRACT	iii
SUMMARY	v
ACRONYMS	xiii
1. INTRODUCTION	1
2. PROJECT DESCRIPTION	2
2.1 Background Information	2
2.2 Objectives	3
2.3 Data Quality Objectives	3
3. PROJECT ORGANIZATION	6
4. APPARATUS AND EQUIPMENT	8
4.1 RTML Configuration	8
4.2 Apparatus	8
4.3 Analytical Instruments	10
4.3.1 Large-Area Ionization Chamber Alpha Spectrometer	10
4.3.2 X-ray/Gamma-ray Spectrometer	12
4.3.3 Alpha Continuous Air Monitor	14
5. TEST PROCEDURES	18
5.1 Preparation	18
5.2 Precautions	18
5.3 Test Operations	19
5.3.1 Sample Collection	19
5.3.2 Sample Pretreatment	20
5.3.3 Sample Preparation for Counting	20
5.3.4 Sample Analysis	21
5.4 Data Analysis	24

6. SAMPLE CONTROL AND DOCUMENT MANAGEMENT	26
7. EQUIPMENT	27
7.1 Sample Collection	27
7.2 Sample Preparation	27
7.3 Analytical Instruments	27
7.3.1 Large-area Ionization Chamber Spectrometer	27
7.3.2 X-ray/Gamma-ray Spectrometer	28
7.3.3 Alpha Continuous Air Monitor	28
7.4 Maintenance and Operation	29
7.5 Calibration	29
7.5.1 Large-area Ionization Chamber Alpha Spectrometer	29
7.5.2 X-ray/Gamma-ray Spectrometer	29
7.5.3 Alpha Continuous Air Monitor	30
8. ANALYTICAL PROCEDURES	31
8.1 Instrumentation	31
8.1.1 Large-area Ionization Chamber Alpha Spectrometer	31
8.1.2 X-ray/Gamma-ray Spectrometer	31
8.1.3 Alpha Continuous Air Monitor	31
8.2 Calculation Procedures	31
9. DATA MANAGEMENT AND ANALYSIS	35
9.1 Reduction, Validation, and Recording	35
9.2 Data Calculations	35
10. QUALITY ASSURANCE	36
10.1 Quantitative Quality Assurance Objectives	36
10.2 Replicate Blanks	36
10.3 Standards	36
10.4 Cross Contamination	37

11. SAFETY, TRAINING, AND LOGISTICS	38
11.1 Radiological Safety and Training	38
11.2 Laboratory Safety and Training	39
11.3 Waste Management Practices and Training	41
12. REFERENCES	42

FIGURES

1. Organizational structure.	7
2. Block diagram of RTML electronics.	9
3. Cut-away view of Ordela large-area ionization chamber.	11
4. Block diagram of x-ray/gamma-ray spectrometer electronics.	13
5. Diagram of alpha CAM sampling cabinet.	15
6. Block diagram of the alpha CAM electronics cabinet.	16
7. Ionization chamber gas and vacuum connections.	22

TABLE

1. LLD concentrations for x-ray/gamma-ray spectrometer	4
--	---

ACRONYMS

ADC	Analog-to-digital converter
AIM	Acquisition interface module
ANL-W	Argonne National Laboratory-West
BWID	Buried Waste Intergrated Demonstration
CAM	Continuous air monitor
DAC	Derived air concentration
DOE	U.S. Department of Energy
FY	Fiscal year
HP	Health physics
INEL	Idaho National Engineering Laboratory
LLD	Lower limit of detection
MCA	Multichannel analyzer
NIM	Nuclear instrument module
N&RP	Nuclear and Radiological Physics
OSHA	Occupational Safety and Health Administration
RADCON	Radiological control
RCT	Radiological controls technician
RM&D	Radiation Measurements and Development
RML	Radiation Measurements Laboratory
ROI	Region of interest
RTML	Rapid Transuranic Monitoring Laboratory

RWMC Radioactive Waste Management Complex

TRU Transuranic

Test Plan for Demonstration of Rapid Transuranic Monitoring Laboratory

1. INTRODUCTION

This test plan describes objectives, instrumentation, and experimental procedures to field test measurement systems installed in the Rapid Transuranic Monitoring Laboratory (RTML), a mobile laboratory developed at the Idaho National Engineering Laboratory (INEL) for use in characterizing radiological source terms at buried radioactive waste remediation sites. Analytical technologies that will be tested in the field include two large-area ionization chamber alpha spectrometers, an x-ray/gamma-ray spectrometer, and four alpha continuous air monitors (CAMs).

The first technology involves direct alpha spectrometry in a 35 cm diameter ionization chamber. Soil samples are dried, sieved, finely ground and, with each sample, 100 mg of the ground soil is suspended in an ethanol/water solution that is sprayed onto a 25 cm diameter stainless steel pan. The pan is positioned in a recess in the bottom of the ionization chamber for counting. Air dusts are collected with a high-volume air sampler on 20 × 25 cm (8 × 10 in.) membrane filters with 1.0 μm pores. In the case of air filters, no sample preparation is required before counting in the ionization chamber. A milled cutout in the sample plate is used to position the air filter reproducibly inside the ionization chamber for counting.

The second technology involves the simultaneous measurement of L-shell x-rays and gamma rays using a high-resolution germanium spectrometer equipped with a thin entrance window. Soil samples are dried, sieved to remove any particles larger than 300 μm, and with each sample, 10 g of sieved soil is loaded into a thin plastic sample container having a diameter slightly larger than the diameter of the germanium crystal and counted with the sample container positioned near the face of the end-cap of the germanium detector. Smears and air filters are centered in the same plastic sample container used to analyze soil samples and are analyzed directly using a reproducible counting geometry.

The third technology involves the direct measurement of alpha activity in air by pulse-height analysis of the alpha activity removed from air that is sampled. The measurement of airborne alpha-emitting radionuclides is performed by pulling a continuous sample of ambient air through a two-stage virtual impactor and then through a filter having a pore size small enough to trap aerosolized particles. The virtual impactor removes a large fraction of the naturally occurring radon daughter isotopes from the airstream before it passes through the sample filter. The alpha-emitting radionuclides are attached to the aerosol particles that are trapped by the filter. Alpha activity on the filter is measured by a solid-state silicon detector positioned about 3 mm from the influent face of the filter.

The practical application of these technologies in determining radiological source terms at buried radioactive waste remediation sites will be tested experimentally during June 1993 in conjunction with a remote retrieval demonstration conducted at the Radioactive Waste Management Complex (RWMC) at the INEL.

2. PROJECT DESCRIPTION

The demonstration of the RTML at the INEL is sponsored by the Buried Waste Integrated Demonstration (BWID) Program under Technical Task Plan ID121210, "BWID Contamination Control." The BWID Program develops and demonstrates new technologies for application to remediation of buried waste at facilities throughout the U.S. Department of Energy (DOE) complex.

The DOE has a recognized need for analytical technologies that can be used in the field to assess radiological source terms both before and during remediation of buried radioactive waste. Existing field-deployable technologies do not have sufficient sensitivities for alpha- and gamma-emitting radionuclides. Consequently, samples collected from a remediation site must be shipped to a laboratory for wet-chemical analysis. Analytical techniques currently used at such laboratories to determine transuranic (TRU) isotopes and radiostrontium require rigorous sample pretreatment and time consuming radiochemical separation of the radionuclides of interest before analysis. Because the RTML is housed in a trailer, it can be moved to the remediation site, eliminating the need for shipping samples to analytical laboratories. Using the RTML, high-priority samples can be analyzed promptly at the remediation site. Because the RTML is equipped with only direct spectrometric analysis technologies that require minimal or no sample preparation, samples can be prepared for analysis and analyzed in less than 1 hour. The mobile laboratory provides a rapid, cost-effective means of characterizing and monitoring radioactive waste remediation sites for alpha- and gamma-emitting contaminants.

2.1 Background Information

During fiscal year (FY)-90 to -92 a number of promising measurement methods and systems were evaluated for possible use in monitoring TRU concentrations in ambient air and in soil, smear, and dust samples collected during remediation of buried radioactive waste.^{1,2,3} The sensitivities of five different alpha CAMs for airborne ²³⁹Pu were determined experimentally.^{4,5,6,7,8} The following six methods of making quantitative measurements of TRU contamination on smears and in soil and dust samples were also evaluated experimentally: (a) alpha liquid scintillation spectrometry, (b) rapid radiochemical separation followed by high-resolution alpha spectrometry, (c) gamma-ray spectrometry, (d) x-ray/gamma-ray spectrometry, (e) direct alpha spectrometry using a large-area ionization chamber, and (f) gross alpha counting using a scintillation detector.^{6,9,10,11,12}

These evaluations demonstrated that the alpha CAM used at Argonne National Laboratory-West (ANL-W) had a sensitivity for airborne ²³⁹Pu 10 to 20 times better than the four commercial alpha CAMs that were also tested. Six of the ANL-W alpha CAMs were fabricated during FY-92, and four of them are now installed in the alpha CAM sampling stations that are operated remotely from the RTML. Direct alpha spectrometry with a large-area ionization chamber and x-ray/gamma-ray spectrometry with a high-resolution, thin-window germanium spectrometer were shown to be rapid (count times ≤ 30 minutes) sensitive methods for the measurement of TRU isotopes on smears and in soil and dust samples. The two analysis techniques do not require elaborate and time-consuming chemical separations; soil samples are only dried and sieved before analysis, and smears and filters are analyzed directly without any sample preparation.

Two large-area ionization chamber alpha spectrometers, a thin-window germanium spectrometer installed in an automatic sample changer, four alpha CAMs, and a VAX 4000 model 100 computer were installed in the RTML during March and April 1993. The RTML will be moved to a location adjacent to the Cold Test Pit at the INEL and will be demonstrated during June 1993 in conjunction with a remote retrieval demonstration.

2.2 Objectives

During the demonstration, ambient air over the test pit and samples of soil and dust from the pit will be analyzed for alpha- and gamma-emitting radionuclides using the RTML. The primary objective of the field test of the RTML is to demonstrate and evaluate the capability of the RTML to analyze soil, smear, and filter samples for ^{239}Pu , ^{241}Am , and gamma-emitting radionuclides such as ^{60}Co and ^{137}Cs . Spiked samples will be introduced into the sample stream before being delivered to the counting trailer. The spiked samples will contain both quantifiable amounts of activity and amounts that are near the claimed detection limit. To achieve this objective, the following specific tasks will be performed.

- Test the performance and reliability of instrumentation installed in the RTML under field conditions when power is supplied from a diesel generator.
- For each measurement system, determine the lower limit of detection concentration/activity for each of the alpha- and gamma-emitting radionuclides of interest and the precision and accuracy achieved with minimal counting times.
- Determine the sample throughput rate for each measurement system and the total number of samples that can be analyzed per day.
- Determine the cost of operating the RTML.

2.3 Data Quality Objectives

The intermediate-range technical performance objective of this project is to produce a field-deployable, mobile laboratory that will provide Environmental Restoration and Waste Management Programs throughout the DOE complex with the capability to monitor airborne TRU isotopes and analyze soil, smear, and air filter samples for alpha- and gamma-emitting radionuclides using direct spectrometric methods that are rapid, sensitive, and cost-effective.

Data quality objectives of the field demonstration of the RTML focus primarily on determining the sensitivity of each measurement system for detection of TRU and/or fission product radionuclides using counting times of 30 minutes or less. Technical performance goals include producing field-deployable measurement systems that will have the following sensitivities: (a) alpha CAM system will have a sensitivity for airborne TRU isotopes that is superior to the sensitivity of 8 derived air concentration (DAC) hours required by DOE Order 5480.11, "Radiation Protection for Occupational Workers," (b) large-area ionization chamber alpha spectrometer will be capable of analyzing soil samples in 30 minutes or less with a lower limit of detection (LLD) of 50 pCi/g for TRU isotopes and air samples with an LLD of 0.1 DAC-h, and (c) x-ray/gamma-ray spectrometer will

be capable of analyzing soil samples in 30 minutes or less with LLDs of 50 pCi/g for plutonium, 1 pCi/g for ^{241}Am , 4 pCi/g for ^{60}Co , and 3 pCi/g for ^{137}Cs . LLD goals for the x-ray/gamma-ray spectrometer for other gamma-emitting radionuclides are given in Table 1. The software codes used to control the measurement systems and analyze spectral data will reside on a VAX 4000 model 100 computer installed in the RTML analysis trailer.

Table 1. LLD concentrations for x-ray/gamma-ray spectrometer.

Radionuclide	Dose equivalent of 100 mrem/year (pCi/g) ^a	LLD (pCi/g) ^b
^{54}Mn	10	4*
^{57}Co	200	2*
^{58}Co	30	4*
^{60}Co	4	4
^{106}Ru	60	30*
^{125}Sb	20	10*
^{134}Cs	6	3*
^{137}Cs	10	3
^{144}Ce	300	20*
^{152}Eu	10	10*
^{154}Eu	7	12*
^{155}Eu	400	8*
^{227}Ac	7	20*
^{232}U	3	6*
^{239}Pu	300	50
^{241}Am	80	1

a. Concentration in soil corresponding to an effective dose equivalent of 100 mrem in first year after release offsite. Taken from Table 14 of Reference 13.

b. Lower limit of detection concentration in soil for a 12 g sample counted for 30 minutes and calculated at the 95% confidence level. Values with an asterisk are estimated LLDs. All other values are measured LLDs.

DOE Order 5400.5, "Radiation Protection of the Public and the Environment," specifies that the basic public dose limits for exposure to residual radioactive material are 100 mrem effective dose equivalent in a year. Soil concentration guides derived from the 100 mrem/year criterion for the farming scenario are reported in Reference 13. The concentration in soil corresponding to an effective dose equivalent of 100 mrem in the first year after release of the remediation site is 4 pCi/g ^{60}Co , 50 pCi/g ^{90}Sr , 10 pCi/g ^{137}Cs , 300 pCi/g ^{239}Pu , and 80 pCi/g ^{241}Am ¹³. The measurable performance goals of the demonstration include achieving LLDs for soil samples that are at or below these 100 mrem/year concentrations.

In the case of the x-ray/gamma-ray spectrometer, the total uncertainty reported with measured activities will include components with the following minimum uncertainties: calibration ($\pm 5\%$), sample mass or volume ($\pm 1\%$), cascade coincidence gamma-ray summing ($\pm 10\%$), and reproducibility of counting geometry ($\pm 3\%$). Since the LXRAY code does not currently propagate all uncertainty components in determining the uncertainty due to counting statistics, a value of $\pm 10\%$ will be used for activity concentrations that are a factor of three above the quoted LLD, and a value of $\pm 3\%$ will be used for activity concentrations that are a factor of 20 above the quoted LLD. These values were determined empirically from the analysis of a number of samples containing known plutonium activities.

3. PROJECT ORGANIZATION

The organizational affiliations and responsibilities of the key personnel involved in the demonstration of the RTML are shown in Figure 1. Personnel from the Radiation Measurements and Development (RM&D) Unit of EG&G Idaho, Inc. will collect and deliver samples to the RTML and will also prepare samples for analysis. During the demonstration, personnel from the RM&D and the Nuclear and Radiological Physics (N&RP) Units of EG&G Idaho, Inc. will operate all RTML analytical instruments. Principal investigators will be responsible for their individual analysis methodologies. Overall arrangements of the Cold Test Pit demonstration are the responsibility of C. V. McIsaac.

The radiological controls technician (RCT) will be responsible for radiological monitoring of the area during the demonstration. The RCT will assist in monitoring samples delivered to the RTML and will verify that all personnel are wearing proper dosimetry.

Y93 0170

Figure 1. Organizational structure.

4. APPARATUS AND EQUIPMENT

This section describes the main structural components of the RTML, equipment used to prepare samples for analysis, laboratory instruments used to analyze samples, and type of data that will be collected during the demonstration.

4.1 RTML Configuration

The RTML consists of two separate trailers and mobile 5, 12.5, and 20 kW diesel generators. One trailer, 2.4 m wide $\times$ 7.3 m long (8 $\times$ 24 ft), houses the sample preparation laboratory that is equipped with a Class A hood, oven, sieving equipment, analytical balance, and sample containers. Samples will be received and prepared for analysis in this trailer. The second trailer, 2.4 m wide $\times$ 14.6 m long (8 $\times$ 48 ft), houses the analytical instruments and computer used to analyze soil, smear, and filter samples and also houses the computer terminal used to control the operation of the four alpha CAMs that will be installed inside the Cold Test Pit enclosure. During the demonstration, both trailers and diesel generator will be parked adjacent the Cold Test Pit.

4.2 Apparatus

To facilitate analyzing samples as quickly as possible following their collection, the RTML is equipped with four alpha CAMs, two large-area ionization chamber alpha spectrometers, a thin-window germanium x-ray/gamma-ray spectrometer installed in an automatic sample changer, a VAX 4000 model 100 computer, and associated computer terminals, monitors, and printers. A block diagram of the instrumentation and associated electronics that will be installed in the RTML analysis trailer and inside the tent enclosing the pit is shown in Figure 2.

As shown in Figure 2, four alpha CAMs will be installed inside the tent covering the pit to monitor concentrations of airborne TRU isotopes. The four CAMs will be controlled from and spectral data transmitted to the VAX computer installed in the RTML using an Ethernet coaxial cable link between the VAX computer and two acquisition interface modules (AIMs) installed in two CAM electronics cabinets. Each electronics cabinet will house the electronics needed to operate two alpha CAMs. The current spectrum and status of each of the four CAMs will be displayed on an X-terminal monitor in the RTML. For each CAM, a plot of the concentrations of airborne TRU isotopes during the previous 24 hours will be displayed on the same monitor used to display CAM status. Hard copies of tabulations and plots of the concentrations of airborne TRU isotopes over any given time period will be prepared using a laser printer.

The two large-area ionization chamber alpha spectrometers and thin-window, germanium x-ray/gamma-ray spectrometer will be controlled from and spectral data transmitted to the VAX computer using an Ethernet link between the VAX computer and two AIMs. The x-ray/gamma-ray spectrometer sample changer will be controlled by the VAX computer through the terminal server.

The high-volume air sampler used to obtain samples of airborne dust for analysis with the large-area ionization chamber alpha spectrometer is a model HV-1SH manufactured by F & J Specialty Products, Inc., Miami Springs, Florida. The sampler is equipped with a flowmeter calibrated for a range from 0.3 to 1.6 m³/minute (10 to 55 cfm) of air and can be used with filter holders for

Y93 0171

Figure 2. Block diagram of RTML electronics.

both 10-cm circular filters with an effective area 9 cm in diameter and 20 × 25-cm rectangular filters with an effective area of 18 × 23 cm. The no-load sampling rate is about 2.7 m³/minute (97 cfm) while the rate with 20 × 25-cm membrane filters is about 1.1 m³/minute (38 cfm) with 1.2 μm pore-size Versapor 1200 filters and about 0.5 m³/minute (16 cfm) with 0.8-μm pore-size Versapor 800 and 0.45-μm pore-size Versapor 450T filters. The sampler is equipped with a cooling fan in the motor housing so that the motor does not depend solely on the air being drawn through the filter for cooling.

To disperse prepared soil samples onto the large counting planchet used with the gridded ionization counter, a 10-mL all-glass, air-operated hand sprayer (Kontes, Vineland, New Jersey, No. 422550) is used to spray a suspension of the finely ground soil uniformly over the 25 cm diameter stainless-steel sample pan.

4.3 Analytical Instruments

4.3.1 Large-Area Ionization Chamber Alpha Spectrometer

Two custom-built gridded ionization chambers (Ordela, Inc., Oak Ridge, Tennessee, models 8200A and 8210A) similar to those built by others^{14,15,16,17} are used with a low-noise preamplifier, linear amplifier, power supply, and VAX computer for data accumulation and analysis.

A cutaway view of the ionization chamber is shown in Figure 3. The ionization chamber is constructed of nickel-plated mild steel of suitable thickness to permit pressurization of the chamber to 100 kPa above atmospheric pressure, which is the pressure used during counting. Both chambers have an inside diameter of 34 cm and internal height of 21.7 cm. Thus, the counting gas volumes of the two chambers are both about 20 L. A pressure-relief valve and vacuum/pressure gauge are installed in the top of each chamber. The access port of each chamber is sealed by a drawer-seal assembly that slides in and out of the port. The sample holder has milled cutouts to hold both 25.4-cm circular pans and 20 × 25-cm rectangular filters. The circular sample pans are made from 0.86 mm thick mirror-finish stainless steel. Each pan is 3 mm deep and has a surface area of 507 cm². The 20 × 25-cm filters are held flat by a steel hold-down plate of the same outside dimensions with an opening 18 × 23 cm to match the effective filtration area of the high-volume sampler. Additional details of construction can be obtained from the manufacturer's instruction manual. Membrane filters with a diameter of 10.0 cm can also be counted by centering them on the steel planchet.

The counting gas is P-10 (90% Ar and 10% CH₄) used without further purification from a commercial cylinder through a two-stage regulator. From the regulator mounted on the gas cylinder, the P-10 gas flows through a small gas shut-off valve that controls flow rate and then through a 0.45-μm Acro-50 filter disk to filter any large-size particulate material present in the gas stream that would gradually increase the contamination and background of the chamber.

Soil samples are initially dried and sieved to remove root mat, rocks, and other debris. A 100 mg sample of the soil is then ground using a pestle and mortar with 30% ethanol, and following grinding, the liquid containing the finely-divided suspended material is removed from the mortar and placed in the glass tube of a small sprayer. The process is repeated twice to recover as much of the material as possible. The suspension is then shaken and sprayed with occasional additional shaking

Figure 3. Cut-away view of Ordela large-area ionization chamber.

onto the 25-cm stainless steel sample pan as uniformly as possible. The sample pan is then dried under an infrared lamp.

High-volume air sampler membrane filters and large-area smear filters require no sample preparation. The filter is simply placed in the filter holder in the ionization chamber and is secured by the appropriate hold-down plate.

4.3.2 X-ray/Gamma-ray Spectrometer

The x-ray/gamma-ray spectrometer determines the presence of plutonium in soils, smears, and filters by counting uranium L x-rays produced by the alpha decay of plutonium radioisotopes. These x-rays, which are lower in energy (longer wavelength) than the K x-rays, are often emitted following alpha decay. It is possible to measure the presence and quantity of many radionuclides such as plutonium and americium radionuclides by measuring the number of emitted L x-rays per unit time. This is the principle upon which the L x-ray spectrometer operates. The technique involves accumulating both x-rays and gamma rays in one spectrum in one count so that the spectrometer can simultaneously measure concentrations of alpha and gamma-ray emitting radioisotopes.

The spectrometer consists of a 60 mm diameter, thin-window, n-type germanium detector capable of efficiently measuring x-rays and gamma rays. The gain is chosen to cover energies from about 10 keV to about 1,350 keV with an 8,192-channel analyzer. Under these conditions, the germanium spectrometer can measure simultaneously the presence of plutonium, ^{241}Am , and radioisotopes that emit gamma rays having energies below 1,350 keV (e.g., ^{60}Co and ^{137}Cs). The detector is equipped with a 30-L liquid nitrogen dewar, dual energy pulser,¹⁸ and automatic sample changer capable of processing 100 samples without the need for operator intervention. The pulser injects pulses into the test input of the resistive feedback preamplifier as shown in Figure 4. These pulses are processed through the preamplifier, linear amplifier, pile-up rejector, and analog to digital converter (ADC) the same way that photon pulses are processed except that, before storage, the pulser pulses are digitally offset after being converted to digital format so that in the multichannel analyzer (MCA) spectrum pulser pulses are separated from photon events. The pulser data are stored with each spectrum and subsequently used during spectrum analysis to determine the energy calibration of the spectrum, coefficients of the peak-width function, and dead time during spectrum accumulation.

Soil samples are prepared in the same way that bulk soil samples are prepared for analysis using the large-area ionization chambers. The bulk soil sample is dried and then sieved to reduce the maximum particle size to less than 50 mesh ($<300\ \mu\text{m}$). A thin plastic container having a 65 mm inside diameter and 3 mm depth is placed on the weighing pan of an analytical balance, and its tare weight is determined. Ten grams of the minus 50 mesh soil is loaded into the sample container while the container is on the weighing pan of the balance. When the sample container contains 10 g of soil, the container lid is pressed onto the lower half of the container and the container is shaken from side to side to distribute the soil evenly.

The x-ray portion of each spectrum is analyzed by a linear least-squares spectral fitting technique originally developed for the analysis of spectra from NaI(Tl) detectors. The x-ray spectrum analysis

Y93 0172

Figure 4. Block diagram of x-ray/gamma-ray spectrometer electronics.

code, LXRAY,^b was developed and tailored for use with the x-ray/gamma-ray spectrometer. The gamma-ray portion of each spectrum is analyzed using VAXGAP,^{19,20} the gamma-ray spectrum analysis code that has been in routine use at the INEL for a number of years. The integration of LXRAY as a subroutine of VAXGAP has been completed.

4.3.3 Alpha Continuous Air Monitor

The alpha CAM selected for use, the ANL-W alpha CAM,^{c,21,22} is equipped with a two-stage virtual impactor to eliminate radon progeny and concentrate airborne plutonium-bearing particulates. Air, which is sampled at the relatively high rate of 280 L/min (10 ft³/min), is drawn through a circular array of inlet jets and directed towards a complementary array of receiving tubes. A major portion of the air leaving the jets (200 L/min) is drawn off laterally before the airstream enters the receiving tubes. Small particles entering the inlet jets follow the lateral flow of air and do not enter the receiving tubes. Large particles not able to follow the lateral airflow enter the receiving tubes. This arrangement is repeated in a second stage in the impactor, with the lateral flow in this stage being 58 L/min. In principle, the minor (20 L/min) airflow exiting the second set of receiving tubes contains most of the plutonium-bearing particulates that entered the impactor. This minor airflow is directed to a sample collection filter spaced about 3 mm from a 900-mm² solid-state silicon detector.

Each alpha CAM consists of an ANL-W type air sampling head, 900-mm² solid-state silicon detector, detector bias supply, preamplifier, linear amplifier, ADC, multiplexer, and AIM. As shown in Figures 5 and 6, the CAM sampling head, silicon detector, preamplifier, air sampling pumps, and mass flow sensors are housed in one cabinet, and the detector bias supply, linear amplifier, ADC, multiplexer, and AIM are housed in a separate electronics cabinet. As shown in Figure 6, the electronics for two CAMs are housed in one electronics cabinet. Thus, for the case of four CAMs, four CAM sampling cabinets and two electronics cabinets will be installed inside the pit enclosure tent. For each CAM, the major and minor air flows through the CAM are continuously monitored using two mass flow sensors. The voltage output of each mass flow sensor is input into a multiplexer so that the flow data can be stored along with the spectral data being stored in the AIM. Both flow rates are displayed and routinely updated in the corresponding spectrum display window on the alpha CAM X-terminal monitor located inside the RTML analysis trailer. Because the preamplifiers are temperature sensitive and temperatures inside the pit enclosure tent are expected to vary diurnally, each CAM cabinet is equipped with a heater/air conditioner. Each electronics cabinet is equipped with an air conditioner to compensate for the heat generated by the ADCs and AIMS.

Of the spectrum analysis methods that have been developed, the most commonly used are those that determine net counts in the ²³⁹Pu peak by subtracting from the total counts in the ²³⁹Pu peak some fraction of the counts in adjacent regions of the spectrum. In each case, the expression for net ²³⁹Pu counts is a linear function of counts in the ²³⁹Pu and background regions of the spectrum. Three such spectrum analysis algorithms, which are currently installed in the majority of commercial alpha CAMs, were evaluated during FY-91 and FY-92. The results showed that the coefficients used

b. M. H. Putnam, *An Operator's Guide to LXRAY*, unpublished, March 1993.

c. J. M. Larson and D. L. Hall, *ZPPR Plutonium Monitor*, ZPR-I Memo No. 389, February 1976.

Figure 5. Diagram of alpha CAM sampling cabinet.

Figure 6. Block diagram of the alpha CAM electronics cabinet.

for background subtraction are strongly dependent on the concentration of dust in the air being sampled and also vary as a function of sampling time.

Methods were identified that might allow calculating background coefficients during air sampling using parameters of the background alpha peaks. However, these methods have not yet been adequately tested. Alpha spectrum peak fitting codes have recently become available (e.g., the GENE code developed at Westinghouse Hanford and the ALPHAMAT code now available from EG&G ORTEC) that might circumvent these problems. Both codes fit a Gaussian function to the high-energy portion of the alpha peak and a quadratic tail to the low-energy portion of the peak. The fitting is an iterative stripping technique, starting at the highest energy peak and moving downward, peak by peak. The GENE alpha spectrum analysis code was recently installed on the VAX computer located in the Radiation Measurements Laboratory (RML) at the Test Reactor Area and was evaluated experimentally for use in analyzing spectra collected using the ANL-W alpha CAMs. To date, the results obtained using the GENE code have not been satisfactory.

A two-window spectrum analysis algorithm that was previously tested with the ANL-W CAM will be used to analyze alpha CAM spectra collected during the demonstration. The algorithm determines net counts in the ^{239}Pu peak by subtracting from the total counts in the ^{239}Pu peak some fraction of the counts in an adjacent background region of the spectrum. These regions, or energy windows, are commonly referred to as regions of interest (ROIs). The algorithm employs one ROI that encompasses the energy region of the ^{239}Pu peak and one background ROI. The background ROI used in the two-window algorithm is located just above the ^{239}Pu ROI and encompasses the low-energy tail of the 6.00 MeV $^{218}\text{Po}/^{212}\text{Bi}$ alpha peak. The number of net counts in the ^{239}Pu peak is expressed as a simple linear equation involving a single coefficient whose value must be determined experimentally. The value of the coefficient determines what fraction of the counts in the background ROI must be subtracted from the counts in the ^{239}Pu ROI to yield zero net counts in the absence of ^{239}Pu . The equation for net counts in the ^{239}Pu ROI is

$$C_{\text{Pu}} = C_1 - kC_2 \quad (1)$$

where

C_1 = total number of counts in the ^{239}Pu ROI (counts)

C_2 = total number of counts in the background ROI (counts)

k = a proportionality constant whose value must be determined experimentally.

5. TEST PROCEDURES

The test procedures discussed in this section include how the samples will be collected, prepared, counted, and analyzed during the remote excavation demonstration at the INEL's RWMC Cold Test Pit. During actual retrieval in a TRU pit or trench, samples collection would be more closely related to the actual retrieval operation. For example during the Cold Test Pit retrieval, samples will be collected near the actual excavation but not in the actual pit because of safety reasons (interference with the remotely controlled excavation). During hot operation, many samples would be remotely collected directly from the pit.

5.1 Preparation

The RTML sample preparation trailer, analysis trailer, and 6.5, 30, and 50 kW diesel generators will be moved to a location adjacent to the Cold Test Pit. The two trailers will be parked next to one another, preferably with the personnel doors facing one another to minimize the distance between them. The HEPA filter, exhaust fan, and duct-work for the Class A hood in the sample preparation trailer, which are mounted on a platform, will be installed on the roof of the sample preparation trailer after arrival at the Cold Test Pit. A small crane will be used to hoist the platform to the roof of the sample preparation trailer. Four support poles, one for each corner of the platform, will be installed to help support the weight of the platform.

The four alpha CAM sampling stations and two associated CAM electronics cabinets will be installed inside the Cold Test Pit enclosure tent. Power to the six alpha CAM cabinets will be supplied from the 6.5 kW diesel generator. A thin-wire Ethernet cable will connect the two CAM electronics cabinets to the VAX computer installed in the RTML analysis trailer. To make the connection to the VAX computer, the Ethernet cable will pass through a penetration in the wall of the RTML analysis trailer.

The x-ray/gamma-ray spectrometer and its shield will be installed in the sample changer cabinet after the RTML analysis laboratory is moved to the Cold Test Pit. The x-ray/gamma-ray spectrometer's dewar will be filled with liquid nitrogen before the start of measurements and filled with liquid nitrogen at least once per week during the demonstration. The dewar will be filled either by connecting a portable liquid nitrogen tank to the fill line mounted on the outside wall of the analysis trailer or by pouring liquid nitrogen into the fill-port on the neck of the dewar using a small hand-held liquid nitrogen container.

5.2 Precautions

Procedures governing the handling of radioactive materials, liquid nitrogen, and performance of laboratory activities are provided in the EG&G Idaho *Company Procedures Manual* and in the procedures manual being developed for this test and will be adhered to during the field test demonstration. The sample preparation, counting, and analysis activities will be covered by the Radiation Work Permit.

5.3 Test Operations

Procedures for collecting, preparing, and analyzing samples are described in this section.

5.3.1 Sample Collection

Nonradioactive soil samples will be selected from the vicinity of the Cold Test Pit. Representative sampling of an area is not applicable for this test because none of the sampled soil is radioactive. However, the following protocol is recommended to reduce inclusion of extraneous material in the soil brought to the sample preparation trailer for drying and sieving. Before starting the retrieval demonstration, several hundred soil, filter, and smear samples will be collected from the area under the tent enclosure. These samples will be catalogued in the logbook by the location and time obtained and assigned a number. The samples will be prepared for the counting laboratory so when the actual retrieval demonstration starts, the counting laboratory can process the samples. During the demonstration, up to 20 samples of soil filters and smears will be obtained from each catalogued box of pit debris that comes out of the tent enclosure. Additionally, up to 20 samples will be obtained from within the tent enclosure during each box (dumpster) changeout.

5.3.1.1 Soil. The soil selected for sampling will be as free as possible from root mat, stones, twigs, or other debris by using a small garden hand trowel to ensure that the finest material is collected. At least five separate scoops will be taken from each area to obtain a representative sample and placed in a 1-quart heavy-duty polyethylene bag with a friction closure and write-on area. Each bag will be filled about half full without interfering with closure of the bag or unduly contaminating the outside of the bag, and the bag will be marked with a two-digit identification number and the date and time of collection. This descriptive information, which is necessary to identify the location where the sample was collected, will be recorded in a sample logbook for permanent reference. The sample will then be surveyed with alpha and gamma survey instruments to ensure that the outside of the sample bag is not contaminated before sending it to the RTML sample preparation laboratory.

5.3.1.2 Airborne Dust. Samples of airborne dust to be analyzed by large-area ionization chamber alpha spectrometry will be collected on 20 × 24-cm fluoropore membrane filters with 1.0 μm pores for 10 minutes at about 420 L/min, or longer if desirable, using a high-volume sampler. Each filter will be placed in a suitable polyethylene bag, and the bag marked with a two-digit identification number, date and time sampling commenced, date and time sampling was terminated, and average flow rate during sampling. This descriptive information, which is necessary to identify the location where the sample was collected, will be recorded in a sample logbook for permanent reference.

Samples of airborne dust for analysis by x-ray/gamma-ray spectrometry will be collected on 47 mm diameter filters installed in the four alpha CAMs. The particular filters installed in the alpha CAMs will be Millipore 47 mm diameter fluoropore filters having 1.0 μm pore size. In each case, the filter will be placed in a filter holder that will be secured to the CAM virtual impactor before the start of sampling. For each CAM, the initial sampling flow rate will be 280 L/min. Following its removal from the filter holder, each CAM filter will be placed in a suitable polyethylene bag and the bag marked with an identification number, date and time sampling was initiated, and date and time

sampling was terminated. This descriptive information, which is necessary to identify the location where the sample was collected, will be recorded in a sample logbook for permanent reference.

5.3.1.3 Smears. Up to 10 smears of surface contamination taken for analysis by x-ray/gamma-ray spectrometry will be collected on 47 mm diameter filters. Each smear will be placed directly in a plastic sample container normally used with the x-ray/gamma-ray spectrometer to minimize handling of the smear, and the container will be marked with a two-digit identification number and date and time of sampling. Samples of surface dust collected for analysis with the large-area ionization chamber alpha spectrometers will be collected with a 20 × 25-cm fluoropore membrane filter wrapped around a paint roller or with a vacuum hose attached to a high-volume sampler equipped with a 20 × 25-cm fluoropore membrane filter. In each case, the sample identification number and descriptive information needed to identify the surface smeared will be recorded in a sample logbook for permanent reference.

5.3.2 Sample Pretreatment

The soil sample in the 1-quart polyethylene bag is emptied into a disposable aluminum pan, approximately 6-1/2 × 9 × 1-1/4 in. (Reynolds Metals Co., Richmond, Virginia, No. RC 576). Any root mat, twigs, stones, or other obvious debris is removed and discarded into a waste container. The pan is placed in a drying oven at 190°C until the sample is dry, then removed and allowed to cool in air to room temperature. Any large clods are broken up with a porcelain pestle, taking care not to penetrate the bottom of the pan. The entire sample is poured into a 50-mesh, 300 µm stainless-steel standard testing sieve, 8-in. outside diameter × 2 in. deep (Fisher Scientific, Pittsburgh, Pennsylvania, No. 04-881-10T), and shaken rapidly horizontally until at least 50 g of minus 50 mesh material has been collected in the bottom pan. Enough of this material is then transferred with a long, thin scoop to a 2-oz polypropylene wide-mouth bottle (Fisher Scientific, Pittsburgh, Pennsylvania, No. 02-893 BB) to fill the bottle about half full, about 50 g. The bottle is shaken vigorously for a few seconds to homogenize the contents, is marked with the same identification number that was on the original polyethylene bag, and is placed in order in a 20-bottle carrier tray.

5.3.3 Sample Preparation for Counting

5.3.3.1 Large-area Ionization Chamber Alpha Spectrometry. A 100-mg sample of the prepared soil is ground thoroughly with four consecutive 2-mL portions of 30% ethanol in a diamonite mortar for about 1 minute each. After each grinding, the mortar is tipped slightly to allow the ethanol containing material finely divided enough to remain suspended to run off the heavier material. The liquid suspension is then lifted out immediately with a suction-type pipet and placed in the reservoir of the glass sprayer. The residue remaining after the third grinding is suspended in the last portion as well as possible and added to the sprayer. The suspension is then shaken and sprayed with frequent additional shaking onto the 25 cm diameter stainless-steel sample pan as uniformly as possible. The pan is heated with an infrared lamp during spraying and then thoroughly dried after spraying is completed. The dried sample is inserted into the ionization chamber and counted.

No special preparation is necessary for counting smears or filters used to collect dust samples. The membrane filter is placed in the ionization chamber, held flat by the hold-down frame, and counted.

5.3.3.2 X-ray/Gamma-ray Spectrometry. The bottom portion of the thin plastic sample container used with the x-ray/gamma-ray spectrometer is placed on the analytical balance, and its tare weight is measured. Ten grams of minus 50 mesh prepared soil is poured into the sample container. The mass of soil added to the sample container is recorded to the nearest milligram. The container is then removed from the balance and placed on a solid surface and the container lid is pressed onto the lower half of the container. The container is shaken from side to side to distribute the soil evenly. The sealed sample container is placed in the sample magazine of the x-ray/gamma-ray spectrometer sample changer for counting.

5.3.4 Sample Analysis

The large-area ionization chamber will operate on an 8-hour/day basis, and determinations of throughput will be extrapolated to longer shifts by using the two available ionization chambers. The U-L-Shell x-ray system will process samples in a semibatch mode during the 8-hour shift. For the following two shifts, 100 samples will be loaded for a 16-hour count. This long, overnight count will be repeated at least twice during the demonstration. The alpha CAMS will be run on a variety of shifts, including four 8-hour shifts and overnight depending on dust loadings.

5.3.4.1 Large-area Ionization Chamber Alpha Spectrometry. The gas and vacuum connections to the ionization chamber are shown in Figure 7. V-1, V-2, and V-3 are three-way gas valves with a closed position in the center. V-2 permits direct venting of the gases to the atmosphere during flushing to avoid gradual contamination of the chamber that would occur if the excess gas was allowed to exit through the access port.

At least once a day immediately before first use, V-2 is opened to the atmosphere and P-10 gas is allowed to come through V-3 just long enough to refill the lines and filter back to the cylinder with fresh, oxygen-free P-10. The sample holder containing a sample pan or filter is placed into the ionization chamber, the door of the access port is closed and dogged tightly against the rubber "O"-ring. V-1 is then opened to the vacuum pump for at least 1 minute after no further movement of the gauge needle is detected. The Leybold-Heraeus pump requires about 1-1/2 minutes to evacuate the chamber to the limit of the vacuum gauge, so a total of at least 3 minutes is allowed to ensure adequate removal of air. V-3 is then opened to the P-10 line, V-2 to V-1, and V-1 to the ionization chamber. The lower stage of the P-10 regulator is set at about 110 kPa so that the gas can be admitted rapidly to minimize loss of time but not so rapidly as to blow the sample around or to permit significant overpressure to occur accidentally. When the gauge indicates a gas pressure of 100 kPa above atmospheric pressure, V-1 is closed. A voltage of 2,750 V is applied to the ionization chamber anode, and spectrum accumulation is initiated.

Following completion of the 10 minute count, the high voltage is reduced to zero to avoid possible damage to the grid when the chamber is next evacuated. V-1 is opened to the vacuum pump until the pressure gauge indicates zero at which time V-1 is closed to stop evacuation, the sliding access drawer is opened, and the sample is removed. The entire alpha spectrum is stored in the VAX 4000 memory and analyzed using a ROI spectrum analysis algorithm. Analysis results are printed using a laser printer. When not in use, the ionization chambers are kept in an atmosphere of P-10 gas to prevent absorption of oxygen and nitrogen by the internal surfaces of the chamber.

Figure 7. Ionization chamber gas and vacuum connections.

5.3.4.2 X-ray/Gamma-ray Spectrometry. Prepared samples are loaded into the magazines of the spectrometer's sample changer. As currently configured, the sample changer can accommodate 100 samples. Before counting is initiated, a count que is constructed using the XGAP program. For each of the samples to be counted, a sample identification number, sample description, and count time are entered into a counting que. Automatic analysis of a set of samples is started by entering the command "GOQ" on the x-ray/gamma-ray spectrometer's X-terminal. Samples are analyzed sequentially beginning with the sample located in the position having the lowest position number. After each sample is counted, the XGAP program automatically analyzes the x-ray/gamma-ray spectrum and prints the results using the laser printer. Gamma-ray results are printed first, followed by the x-ray results.

5.3.4.3 Alpha Continuous Air Monitor. The continuous air monitoring system consists of four air sampling cabinets and two electronics cabinets. As shown in Figure 2, each electronics cabinet serves two air sampling cabinets. Each of the air sampling cabinets is equipped with an ANL-W two-stage virtual impactor, 900-mm² solid-state silicon detector, filter holder, pumps for pulling air through the impactor and sampling filter, and flow sensors for monitoring air flow during sampling.

Before operation, a Millipore 47 mm diameter Fluoropore filter having a pore size of 1.0 μm must be installed in the CAM filter holder. Access to the filter holder is achieved through the rear door of the air sampling cabinet. A switch mounted to an internal shelf automatically shuts off the detector bias voltage when the rear cabinet door is opened. The filter holder, which is located at the bottom of the virtual impactor, is removed from the impactor body by turning it one-half turn counter-clockwise. Using tweezers, the 47 mm diameter filter is placed over the fine-mesh screen in the filter holder and centered. Once the filter is centered, the air sampling pumps are turned on to hold the filter in place during the installation of the filter holder. The air sampling pumps are turned on with the power switch located in the upper right hand corner of the rear frame of the cabinet. The filter holder is secured to the impactor body by inserting it into the filter holder receptacle at the bottom of the impactor body and turning it clockwise one-half turn.

Using the alpha CAM X-terminal located in the RTML analysis trailer, spectrum accumulation is initiated using the ACAM program by entering the command "START," desired counting time in seconds, and depressing the "RETURN" key. When spectrum accumulation is started, the spectrum for the CAM will be displayed in the spectrum display window on the CAM X-terminal monitor corresponding to the CAM. The impactor exhaust and filter effluent air flow rates for each CAM will be displayed below the corresponding alpha spectrum.

Following the completion of a sampling cycle, the filter is removed from the filter holder using the following procedure. The filter holder is removed from the impactor body by turning it one-half turn counter-clockwise. This operation is performed with the air sampling pumps running so that the filter remains with the filter holder during removal. When the filter holder is disengaged, the air sampling pumps are turned off. The filter is transferred from the filter holder to a 2 in. diameter petri dish, and a new filter is installed using the procedure previously described.

5.4 Data Analysis

The measurement of the activities of radionuclides in soils requires that the mass of sample analyzed be known. In the case of soil samples prepared for analysis using the x-ray/gamma-ray spectrometer, the bottom portion of the thin plastic sample container will be placed on the balance pan and the tare weight measured before soil is added to the container. Ten grams of minus 50-mesh soil will then be added to each container. The mass of soil will be recorded to the nearest milligram. In the case of samples prepared for the large-area ionization chamber alpha spectrometer, the mass of sample removed for grinding using the pestle and mortar will remain fixed at 100 mg. The quantity of soil removed for grinding will be recorded to the nearest tenth of a milligram. Some losses are expected during the suspension and spraying of the soil onto the sample pan used for counting. Additional experimental work is required to quantify the losses accurately.

Alpha and x-ray/gamma-ray spectra will be analyzed using software programs installed on the VAX 4000 model 100 computer. Photon spectra collected using the x-ray/gamma-ray spectrometer will be analyzed automatically using the program XGAP, which is a combination of the LXRAY and VAXGAP programs that analyze, respectively, the x-ray and gamma-ray regions of each spectrum. Alpha spectra collected using the four alpha CAMs and two ionization chambers will be analyzed automatically using the ACAM and ION programs, respectively. Both programs analyze alpha spectra by means of ROI-type analysis algorithms.

The methodology of Currie²³ is being used to calculate the sensitivities of the analytical measurements. His equation for paired observations of sample and background with a probability of 0.05 that activity will be detected when none is present or that activity will not be detected when in fact it is present is

$$LLD = 2.71 + 4.66 B^{1/2} \quad (2)$$

In terms of concentration in soil, expressed as pCi/g, the equation becomes

$$LLD = \frac{2.71 + 4.66 B^{1/2}}{2.22 \cdot T \cdot \epsilon \cdot Y \cdot M} \quad (3)$$

where

B = total number of background counts in the region of interest (counts)

2.22 = number of disintegrations per minute per pCi

T = counting time (minutes)

ϵ = absolute counting efficiency (counts per disintegration)

Y = fractional recovery in the region of interest

M = mass of sample (g).

6. SAMPLE CONTROL AND DOCUMENT MANAGEMENT

During the demonstration, laboratory notebooks will be maintained for the large-area ionization chamber alpha spectrometer, x-ray/gamma-ray spectrometer, alpha CAMs, and VAX 4000 model 100 computer. Instrument calibration results for each measurement system will be documented in the project file or instrument logbook. Any changes made to settings or maintenance performed will be noted in the laboratory notebook assigned to the system.

Sample identification numbers and descriptions will be entered in a sample logbook. The sample logbook will be taken into the field by personnel collecting samples. Following the collection of each sample, personnel collecting the sample will enter into the sample logbook: (a) two-digit sample identification number, (b) date and time of sampling, (c) sampling location, and (d) description of the sample. The date and time of sampling and sample identification number will also be written on the sample container.

7. EQUIPMENT

Samples collected from the Cold Test Pit will be prepared for analysis in the RTML sample preparation trailer and analyzed in the RTML analysis trailer. Electrical power for both trailers will be supplied from a 50 kW diesel generator. A VAX 4000 model 100 computer installed in the RTML analysis trailer will analyze all spectral data. Two laser printers installed in the analysis trailer will be used to print hard copies of analysis results.

7.1 Sample Collection

• 1-quart polyethylene bag with friction closure	500 ea
• Indelible marking pen	6 ea
• Garden hand trowel	2 ea
• High-volume air sampler model HV-1SH	1 ea
• 20 × 25-cm fluoropore membrane filter	5 ea
• 21 × 26-cm polyethylene bag with friction closure	20 ea
• 47 mm diameter, Millipore cellulose acetate filter	50 ea
• 47 mm diameter, Millipore fluoropore 1.0 µm filter	50 ea
• 2 in. diameter petri dish	100 ea

7.2 Sample Preparation

• Sample preparation trailer with Class A hood	1 ea
• Aluminum sample pan	50 ea
• Gravity-flow drying oven	1 ea
• Sieve assembly, 50 mesh	1 ea
• 2-oz polypropylene bottle	500 ea
• Metler AE200 direct reading balance	1 ea
• Pestle and mortar	1 ea
• Ethanol	1 qt
• Water	1 gal
• 10-mL, all-glass air-operated sprayer	1 ea
• 25 cm diameter stainless-steel pan	50 ea
• Infrared lamp	1 ea
• 65 mm diameter × 3 mm deep plastic sample container	500 ea
• 4-gal polyethylene bucket	1 ea

7.3 Analytical Instruments

7.3.1 Large-area Ionization Chamber Spectrometer

• Ionization chamber with preamplifier (Ordela, Inc. models 8200A and 8210A)	2 ea
• Cylinder P-10 gas and two-stage regulator	1 ea
• Cylinder compressed air and two-stage regulator	1 ea
• Vacuum pump	1 ea
• High voltage bias supply	2 ea

• Amplifier	2 ea
• ADC	2 ea
• Acquisition interface module	1 ea
• Nuclear instrument module (NIM) bin	2 ea
• X-terminal	1 ea

7.3.2 X-ray/Gamma-ray Spectrometer

• PGT NIGC 2830 thin-window, germanium detector with preamplifier and dual-energy pulser	1 ea
• EG&G ORTEC 30-L liquid nitrogen dewar	1 ea
• Liquid nitrogen	30 L
• Gamma Products, Inc. G3200 gamma counting system	1 ea
• Gamma Products, Inc. G6000 driver controller	1 ea
• High voltage bias supply	1 ea
• Amplifier	1 ea
• ADC	1 ea
• AIM	1 ea
• X-terminal	1 ea
• NIM bin	1 ea

7.3.3 Alpha Continuous Air Monitor

• Alpha CAM air sampling cabinet	4 ea
- Solid-state heater/air conditioner	1 ea
- ANL-W two-stage virtual impactor	1 ea
- Solid-state silicon detector	1 ea
- Preamplifier	1 ea
- 10 cfm air pump	1 ea
- 1 cfm air pump	1 ea
- High-volume flow sensor	1 ea
- Low-volume flow sensor	1 ea
- Fluoropore 47-mm filter	50 ea
- 120 Vac extension cord	50 ft
• Alpha CAM electronics cabinet	2 ea
- NIM bin	1 ea
- High voltage bias supply	2 ea
- Amplifier	2 ea
- ADC	2 ea
- Multiplexer	2 ea
- AIM	1 ea
- X-terminal	1 ea
- 120 Vac extension cord	100 ft

7.4 Maintenance and Operation

RTML equipment will be maintained and operated according to RTML operating procedures. No preventive maintenance will be performed on RTML instruments during the demonstration. The dewar of the germanium spectrometer will be filled with liquid nitrogen twice a week while the RTML is at the RWMC.

7.5 Calibration

7.5.1 Large-area Ionization Chamber Alpha Spectrometer

Calibration standards containing three radionuclides having principal alpha energies as widely separated as possible are used to adjust the energy calibration and determine the linearity of response. The radionuclides used are ^{230}Th , ^{239}Pu , and ^{244}Cm that emit alpha particles having average alpha energies of 4,684, 5,155, and 5,805 keV, respectively. The activity of each radionuclide is sufficient to give excellent counting statistics in a 10-minute count. The gain and digital offset will be maintained to keep the three alpha peaks in their desired channel positions. The analyzer has been set to cover 512 channels at 10 keV per channel beginning at 2,800 keV so that the alpha peak of the principal radon daughter ^{214}Po at 7,687 keV is displayed at the higher energy end of the spectrum while providing as much space as possible at the lower energy end to accommodate degraded pulses.

7.5.2 X-ray/Gamma-ray Spectrometer

The following calibrations will be performed before the demonstration using the following procedures and programs. If instabilities or other unanticipated problems arise, the spectrometer will be recalibrated in the field.

The energy scale for the entire energy region is determined from the pulser, which is calibrated against a special radioactive source comprised of ^{57}Co , ^{137}Cs , and ^{60}Co using the XCALIB program. Since two different analysis programs are used to analyze the L x-ray and gamma-ray energy regions of each spectrum, the spectrometer requires two separate and distinct radionuclide activity calibrations.

The spectrometer is calibrated for plutonium and ^{241}Am by counting standards containing known quantities of these radionuclides. The COMPONENT program subtracts the background from each component of the spectrum, corrects for the sample mass or volume, and scales each component to a fixed disintegration rate. When an unknown sample is analyzed, the L x-ray portion of the spectrum is fit to the components of the calibration spectra using linear least squares methods. A standardization coefficient relates the relative magnitudes of the standard component with that of the unknown sample. The standardization coefficient is then scaled by the mass or volume of the sample and the count time to yield the sample activity or activity concentration.

With one exception, the gamma-ray energy region from about 59 keV to 1,350 keV is calibrated following the same procedure as is normally used for germanium gamma-ray detectors. A special mixed radionuclide calibration source is used that contains ^{54}Mn and ^{65}Zn in addition to ^{241}Am , ^{109}Cd ,

^{57}Co , ^{139}Ce , ^{203}Hg , ^{113}Sn , ^{137}Cs , ^{88}Y , and ^{60}Co . This mix was chosen because most of the gamma rays emitted by this mix are relatively free of cascade coincidence summing. Since the gamma rays from ^{88}Y and ^{60}Co are in cascade, these gamma rays are not included in the efficiency measurements but can be used to estimate the amount of coincidence summing occurring with other radionuclides having gamma rays with energies above 800 keV. An efficiency curve from 50 to 1,400 keV is developed using an efficiency measurement program EFF developed for use by the RML at the INEL.

7.5.3 Alpha Continuous Air Monitor

The energy calibration of each solid-state silicon detector is determined and intermittently checked by measuring a filter standard that contains ^{230}Th , ^{239}Pu , and ^{244}Cm . The three isotopes emit alpha particles having average energies of 4.684, 5.155, and 5.805 Mev, respectively. The absolute counting efficiency of each alpha detector is determined and checked by measuring either the same filter standard used to perform energy calibrations or filter standards that contain known quantities of ^{239}Pu . The latter standards were prepared using membrane filters having a pore size of 0.1 μm . The ^{239}Pu activity was transferred to the filters using a neodymium fluoride carrier in dilute perchloric acid. The method²⁴ has been shown to provide an even distribution of activity over a filter surface with minimal loss of alpha particle energy. The quantity of material deposited on the filter standards is about 30 $\mu\text{g}/\text{cm}^2$.

8. ANALYTICAL PROCEDURES

8.1 Instrumentation

8.1.1 Large-area Ionization Chamber Alpha Spectrometer

Soil and filter samples are prepared for analysis following the procedures described in Sections 5.3.2 and 5.3.3.1 and analyzed according to the procedure described in Section 5.3.4.1. Spectral data are analyzed automatically using the ION alpha spectrum analysis program.

8.1.2 X-ray/Gamma-ray Spectrometer

Soil samples are prepared for analysis using the procedures described in Sections 5.3.2 and 5.3.3.2 and then loaded into one of the sample changer magazines. The sample identification number is entered into the XGAP program. After all of the samples are loaded into magazines of the sample changer and their identification numbers entered into XGAP, the count que is started. All samples entered into the XGAP program count que are analyzed automatically.

The chi-square and amount of gain or zero shift necessary to obtain an optimum fit for the L x-ray components are included in the printout of the plutonium and ^{241}Am activities or activity concentrations and are measures of the quality of the fit of the components to the spectral data. The uncertainties in the activities or activity concentrations determined using the LXRAY program are currently underestimated.

The activities of gamma-ray emitting radionuclides are determined by analyzing specific gamma-ray energy regions of the spectrum using the VAXGAP program. The results of these fitted regions are reported with their uncertainties whether positive or negative.

8.1.3 Alpha Continuous Air Monitor

Each alpha CAM sample filter is installed in the CAM filter holder and spectrum accumulation initiated following the procedures described in Section 5.3.4.3. For each of the four alpha CAMs, the cumulative alpha spectrum is analyzed at regular intervals during sampling, and the average airborne TRU concentration calculated for each counting interval is plotted on the alpha CAM monitor immediately following completion of the spectrum analysis. Spectrum analysis is performed using the ACAM program and the spectrum analysis algorithm described in Section 4.3.3.

8.2 Calculation Procedures

In the case of the large-area ionization chamber and germanium spectrometers, activities in each sample are calculated automatically using the spectrum analysis programs ION and XGAP, respectively. In the case of the alpha CAMs, airborne TRU concentrations are automatically calculated and plotted using the ACAM program. The three spectrum analysis programs are installed on the VAX computer in the RTML analysis trailer.

For the large-area ionization chamber alpha spectrometers, the net area of each TRU alpha peak is determined by subtracting from the gross counts in the peak the counts contributed by instrument background and the low-energy tails of the $^{218}\text{Po}/^{212}\text{Bi}$ and ^{214}Po peaks. In the case of soil samples, activity concentration, C_i , expressed as pCi/g is calculated as

$$C_i = \frac{C_G - B_i - (k_1 \cdot R_1) - (k_2 \cdot R_2)}{2.22 \cdot \epsilon \cdot T \cdot M} \quad (4)$$

where

- C_G = total counts in TRU alpha peak (counts)
- B_i = instrument background in TRU alpha peak (counts)
- k_1 = fraction of counts in background ROI, R_1 , that fall in energy region of TRU alpha peak
- R_1 = ROI encompassing $^{218}\text{Po}/^{212}\text{Bi}$ peak
- k_2 = fraction of counts in background ROI, R_2 , that fall in energy region of TRU alpha peak
- R_2 = ROI encompassing ^{214}Po peak
- 2.22 = number of disintegrations per minute per pCi
- ϵ = absolute counting efficiency (counts per disintegration)
- T = count time (minutes)
- M = sample mass (g).

In the case of the x-ray/gamma-ray spectrometer, the net number of counts in each L x-ray peak is determined using the LXRAY subroutine of the XGAP program, and the net number of counts in each gamma-ray peak is determined using the VAXGAP subroutine of the XGAP program. For a soil sample, activity concentration, C_x , expressed as pCi/g, is calculated as

$$C_x = \frac{C_N}{2.22 \cdot P \cdot \epsilon \cdot T \cdot M} \quad (5)$$

where

C_N = net counts in x-ray or gamma-ray peak

P = emission probability of x-ray or gamma ray.

Other terms are defined in Equation 4.

For the gridded ionization counter and the L x-ray/gamma-ray spectrometer, the value of ϵ is determined by calibration with a sample whose activity is known. However, in the case of the measurement of plutonium and americium by the measurement of their emitted L x-rays, a standardization coefficient is determined instead of an efficiency. The standardization coefficient is the ratio of the radionuclide component from the sample to that from the standard correcting for differences in counting time, mass of a soil sample, or volume of air through a filter.

In the case of the alpha CAMs, the airborne TRU concentration during the first counting interval, X_1 , expressed as pCi/L, is calculated as

$$X_1 = \frac{C_1}{2.22 \cdot \epsilon \cdot Y \cdot F_1 \cdot \frac{T_1^2}{2}} \quad (6)$$

where

C_1 = net counts in TRU alpha peak during first counting interval (counts)

Y = activity collection efficiency of CAM

F_1 = average flow rate during first counting interval (L/min)

T_1 = length of first counting interval (min).

The airborne TRU concentration during the second counting interval is calculated as

$$X_2 = \frac{C_2 - C_1 - 2C_1(T_2/T_1)}{2.22 \cdot \epsilon \cdot Y \cdot F_2 \cdot \frac{T_2^2}{2}} \quad (7)$$

where

C_2 = net counts in TRU alpha peak at end of second counting interval (counts)

F_2 = average flow rate during second counting interval (L/min)

T_2 = length of second counting interval (min).

The airborne TRU concentration during each subsequent counting interval, X_n , expressed as pCi/L, is calculated as

$$X_n = \frac{(C_n - C_{n-1}) - (C_{n-1} - C_{n-2})(T_n/T_{n-1})}{2.22 \cdot \epsilon \cdot Y \cdot F_n \cdot \frac{T_n^2}{2}} - X_{n-1} \cdot (T_{n-1}/T_n)(F_{n-1}/F_n) \quad (8)$$

where

C_n = net counts in TRU alpha peak at end of counting interval n (counts)

F_n = average flow rate during counting interval n (L/min)

T_n = length of counting interval n (min).

9. DATA MANAGEMENT AND ANALYSIS

Aspects of data management and analysis discussed in this section include data reduction, validation, recording, and calculations. Copies of all raw and validated data will be stored on the RMTL VAX 4000. Backup copies of all data will be downloaded to the BWID Data Management System.

9.1 Reduction, Validation, and Recording

Data collected during the demonstration will be evaluated and documented in an EG&G Idaho informal report. Data included in the report will be qualified by the principal investigators and will be subjected to peer review. Data obtained during the demonstration that will be presented in the report include

- LLD activities or concentrations of ^{239}Pu and ^{241}Am in soils, smears, and airborne dust determined for the large-area ionization chamber alpha spectrometers
- LLD activities or concentrations of plutonium, ^{241}Am , ^{60}Co , and ^{137}Cs in soils, smears, and airborne dust determined for the x-ray/gamma-ray spectrometer
- LLD concentration of airborne ^{239}Pu determined for each of the four alpha CAMs
- Sample throughput rates for the large-area ionization chamber spectrometers and the x-ray/gamma-ray spectrometer.

In addition, the report will present qualitative assessments of

- RTML hardware and software performance
- Procedures used to collect, prepare, and analyze soil, smear, and airborne dust samples.

9.2 Data Calculations

Radionuclide concentrations in soil and air will be calculated using the methods described in Section 8.2. LLD activities and concentrations will be calculated at the 95% confidence level using Currie's methodology²³ described in Section 5.8.

10. QUALITY ASSURANCE

10.1 Quantitative Quality Assurance Objectives

Quality assurance goals include producing field-deployable measurement systems that will satisfy project measurement sensitivity requirements. As described in Section 5.4, the methodology of Currie²³ was used to calculate, at the 95% confidence level, the LLDs of the measurement systems. Estimated LLDs of the x-ray/gamma-ray spectrometer for plutonium, americium, and gamma-ray emitting radionuclides are given in Table 1 for the case of 12-g soil samples counted for 30 minutes. LLDs of the large-area ionization chamber alpha spectrometers for alpha-emitting TRU isotopes in soil and air are estimated to be 50 pCi/g and 0.1 DAC-h, respectively, using counting times of 10 minutes. LLDs of the alpha CAMs for airborne ²³⁹Pu and ²⁴¹Am are estimated to be less than 8 DAC-h using a spectrum analysis interval of 30 minutes.

The precision of measurements made using the large-area ionization chamber alpha spectrometers, x-ray/gamma-ray spectrometer, and alpha CAMs is dictated primarily by Poisson statistics and the uncertainty of background estimates. Therefore, instrument precision is dependent on factors such as quantity of activity present in the sample and the counting time used to analyze the sample. Instrument precision will be determined in the field by analyzing given standards multiple times.

The accuracy of measurements made using the RMTL measurement systems is dependent on counting statistics, uncertainty of estimated backgrounds, quantity of activity present in the sample, counting time, and uncertainties of parameters used to calculate isotope concentration such as sample preparation chemical yield, sample mass or volume, and absolute counting efficiency. Instrument accuracy will be determined in the field by analyzing traceable standards of the National Institute of Standards and Technology with sample geometries that are the same as the unknown samples.

10.2 Replicate Blanks

Most of the test samples will not contain alpha activity detectable in the specified counting times. Failure to detect activity in nonradioactive samples will demonstrate the capability of the analysis technique to not report false positive activities. Capability to accurately identify the radionuclides present in spiked samples and measure their activities, when above LLDs, demonstrates the capability of the analysis technique to perform correct assays even on samples near the detection limit. Lower limits of detection will be calculated for the spectra obtained using the Currie methodology.²³

10.3 Standards

Standard soils^{25,26} containing known quantities of ²³⁹Pu will be spotted randomly throughout the samples, some at levels only slightly higher than the expected lower limit of detection to validate its value, and some several times higher to evaluate the accuracy obtainable when better statistical precision can be achieved. Samples of air containing known quantities of alpha activity are more difficult and expensive to prepare. The detection limit can be calculated from Currie's equation, but

some alternative will have to be devised to demonstrate the accuracy with which higher activities can be measured.

10.4 Cross Contamination

Cross contamination among samples is always a problem when large numbers of samples must be dried, sieved, and ground in proximity to one another using the same pans, sieves, pestle, and other nondisposable items. This is particularly true in a mobile laboratory without running water and sewage facilities. For this reason, the drying oven used is of the gravity-flow type, without forced air circulation, and as much spacing between sample pans will be maintained as is consistent with the work load to be accommodated. Disposable aluminum sample pans will be used. However, the pans will be reused to minimize waste disposal when the previous analysis indicates absence of significant activity in the sample contained in that pan. Nondisposable items such as sieves, pestle, etc. will be wiped with laboratory tissue until visually clean. It should be emphasized that mere presence of residual sample does not necessarily result in detectable contamination of the following sample. If 1,000 g samples are prepared for analysis and even 0.1 g of residual sample is allowed to remain on the pan, sieve, pestle, etc., the contamination of the subsequent sample will be diluted 10,000 fold following homogenization. If the detection limit were 50 pCi/g, the previous sample would have to have contained 0.5 μ Ci/g of activity to be detectable. It will not be difficult to avoid residuals as large as 0.1 g.

11. SAFETY, TRAINING, AND LOGISTICS

Safety is of utmost concern with all experiments. It is the policy of EG&G Idaho to take every practical precaution to guard the health and safety of its employees and general public. Three types of safety and training concerns are emphasized: radiological, laboratory, and waste management. No one participating in the field test of the RTML will be allowed inside the fence of the Cold Test Pit area. Only radiation workers trained in the operation of radiation measuring instrumentation will be used to operate the counting instrumentation. The sample preparation will be conducted by experienced radiochemists or performed under their direct supervision. Occupational Safety and Health Administration (OSHA)/HAZWORKER training will be obtained by all personnel who will collect samples from the Cold Test Pit. The work will be conducted under a Radiation Worker Permit. Accidents will be reported to the Cold Test Pit Facility Manager and the unit managers of the personnel conducting this field test.

11.1 Radiological Safety and Training

Radiological safety and conformance to the EG&G Idaho *Radiological Controls Manual* (RADCON) will be ensured by consulting on-duty RADCON personnel before beginning work involving radioactive materials. Subsections of the manual that apply to this document include

- Chapter 3, "Airborne Radioactivity Limits, HEPA Filter Requirements, Monitoring for Airborne Radioactivity"
- Chapter 4, "Enclosure for Containing Contamination and Severe Measurements"
- Chapter 8, "Constant Air Monitors, Instrument Calibration and Control, Selection and Installation of Continuous Air Monitors"
- Chapter 11, "Continuous Availability of RADCON Personnel, Inspecting Radiological Work, Planning Radiological Controls, Radiation Worker Training, Radiological Controls, Review and Approval."

The radioactive samples being assayed consist of small quantities of soil (<25 g), smears (47 mm diameter filter paper), and CAM filters that are contained within a 70 mm diameter × 8 mm thick plastic container with a snap on lid. Typical activities or activity concentrations are <1100 pCi/g and <5,000 pCi, respectively. Therefore, the radiological hazard is small and consists in potential contamination by very low-activity radioactive material and the possibility of a high-activity sample not being identified by the up-stream screening performed with portable health physics (HP) instrumentation.

1. Hazards
 - a. Exposure by a high-activity sample not identified by up-stream monitoring with HP instrumentation.

- b. Spread of low-activity radioactive material due to leakage or by unsealed or broken containers.

2. Precautions

- a. All samples will be monitored by HP instrumentation before released from the sample preparation trailer to be counted in the mobile laboratory trailer.
- b. Samples will be sealed with sealant (e.g., super glue, RTV, or model airplane glue) or with transparent tape before handling. Prepared samples will be promptly loaded into the sample holder for most handling operations and will not be removed until all counting is complete.
- c. The sample magazine shall always be held at an upward angle to prevent individual samples from sliding out of the sample holder magazine.

11.2 Laboratory Safety and Training

Laboratory safe practices are described in the EG&G Idaho *Science and Technology Standard Practices Manual*, Section 2. Subsections that apply to this document in Section 2.1, "General Laboratory Practices," include

- 2.1.5, "Eye Protection and Protective Clothing"
- 2.1.6, "Laboratory Safety"
- 2.1.8, "Laboratory Hood Use."

Subsections that apply to Section 2.2, "Site Laboratory Practices," include

- 2.2.4, "Radioactive Materials Handling"
- 2.2.7, "Laboratory Radiological Training."

The photon analysis spectrometer uses a liquid nitrogen (LN_2) cooled germanium spectrometer. The dewar that maintains the detector at LN_2 temperatures is filled weekly, or when the operation requires, twice a week.

1. Hazards

- a. Asphyxiation, if LN_2 evaporates rapidly in a confined space. This is a very remote possibility even when dewars develop a vacuum leak.
- b. Burns, if LN_2 gets on tight fitting clothing, trapped in shoes or pockets, or gets on bare skin.
- c. Explosion, if LN_2 is confined without pressure relief.

2. Precautions

- a. Protective equipment should be worn including leather or cryogenic gloves and goggles.
- b. Piping and transfer lines should be constructed to avoid trapping of LN₂ in the lines.
- c. LN₂ should be vented to a well ventilated room or to the outside whenever possible, or an oxygen depletion monitor should be used when ventilation is not adequate.

No electric circuits are exposed to the operator and most of the instrumentation operates on +12, -12, +24, and -24 V circuits. Therefore, use of frayed cords or careless operations such as operation with wet hands or unauthorized repairs, could result in electrical shock.

1. Hazards

- a. Frayed power cords.
- b. Operation with wet hands.

2. Precautions

- a. Before every campaign or on the first day of each month that the photon analysis spectrometer is used, power cords should be checked for fraying especially near the electrical plug.
- b. System should be operated with dry hands only.

Although no heavy lifting or extended reaching is involved in preparing or counting samples, various common physical hazards potentially exist. Most of these hazards are encountered in the sample preparation trailer and include cuts from sharp objects and burns from touching objects that were heated in the drying oven; however, eye strain, repetitive motions (carpal tunnel syndrome), and tripping hazards are also subtle concerns. Personnel involved in the in-field test demonstration should also be cautious of these hazards and take measures to avoid them. Personnel should exercise good office safety.

1. Hazards

- a. High temperature
- b. Sharp edges
- c. Eye strain and repetitive motions.

2. Precautions

- a. Handle heated objects with tongs or gloves.
- b. Beware of any sharp objects that could inflict a wound.
- c. Take 10 minute breaks from the computer console every hour.

11.3 Waste Management Practices and Training

Waste management practices are prescribed in the EG&G Idaho *Safety Manual*, Section 15, and the *Science and Technology Standard Practices Manual*, Section 2. Pertinent sections include

- 2.1.1, "Nonradioactive Chemical Waste Disposal"
- 2.1.3, "Chemical Lab Inventory"
- 2.1.4, "Chemical Spill Control"

and

- 2, "Incinerable Low-level Radioactive Waste"
- 3, "Compactible Low-level Radioactive Waste"
- 9, "Transuranic Waste"
- 15.1, "Routine Solid Waste Collection and Handling."

12. REFERENCES

1. P. G. Shaw et al., *Test Plan for Contamination Control Related Experiments*, EGG-WM-9059, March 1990.
2. G. G. Loomis and C. V. McIsaac, "Rapid Monitoring for Transuranic Contamination During Buried Waste Retrieval," *Waste Processing, Transportation, Storage and Disposal, Technical Programs and Public Education, Volume I, Proceedings, Symposium on Waste Management at Tucson, Arizona, February 24-28, 1991*:723-728, 1991.
3. G. G. Loomis, *Technology Status Report Transuranic Contamination Control at INEL*, EGG-WTD-9899, September 1991.
4. C. V. McIsaac and C. R. Amaro, *Real-time Transuranic Monitoring with a Victoreen Model 758 Alpha Continuous Air Monitor*, EGG-WM-8774, September 1989.
5. C. V. McIsaac et al., "Sensitivities of Alpha Continuous Air Monitors for Detection of Airborne Pu-239," *Waste Processing, Transportation, Storage and Disposal, Technical Programs and Public Education, Volume II, Proceedings, Symposium on Waste Management at Tucson, Arizona, February 24-28, 1991*:695-705, 1991.
6. C. V. McIsaac et al., *Rapid Monitoring for Transuranic Contaminants During Buried Waste Retrieval*, EGG-WTD-9412, March 1991.
7. M. R. Winberg, *Test Plan for FY-91 Alpha CAM Evaluation*, EGG-WTD-9571, March 1991.
8. C. V. McIsaac and C. R. Amaro, *Sensitivities of Five Alpha Continuous Air Monitors for Detection of Airborne Pu-239*, EGG-WTD-10314, July 1992.
9. C. W. Sill and R. J. Gehrke, *Test Plan for Rapid Transuranic Monitoring of Soil and Air Dusts by Direct Large-Area Alpha Spectrometry and L-Shell X-Ray Spectrometry*, EGG-WTD-9735, May 1991.
10. C. W. Sill, *Rapid Monitoring of Soil, Smears, and Air Dusts by Direct Large-Area Alpha Spectrometry*, EGG-WTD-10080, January 1992.
11. R. J. Gehrke et al., "Rapid Assay of Plutonium in Soils by Passive L X-Ray Counting," *Trans of the Amer Nucl Soc*, 64:94, 1991.
12. C. W. Sill, "Rapid Monitoring of Soil, Water, and Air Dusts by Direct Large-Area Alpha Spectrometry," submitted to *Health Physics*, September 1992.
13. P. G. Voilleque et al., *Development of Criteria for Release of Idaho National Engineering Laboratory Sites Following Decontamination and Decommissioning*, EGG-2400, August 1986.

14. R. V. Osborne and C. R. Hill, "High Resolution Alpha Particle Spectroscopy at Very Low Specific Activities," *Nucl Inst & Methods* 29:101-106, 1964.
15. H. Hotzl and R. Winkler, "Large Area Gridded Ionization Chamber and Electrostatic Precipitator. Application to Low-level Alpha Spectrometry of Environmental Air Samples," *Nucl Inst & Methods* 150:177-181, 1978.
16. H. Hotzl and R. Winkler, "Experiences with Large Area Frish Grid Chambers in Low-level Alpha Spectrometry," *Nucl Inst & Methods in Phys Res* 223:290-294, 1984.
17. L. Johansson, B. Roos, and C. Samuelsson, "Alpha-particle Spectrometry of Large-area Samples Using an Open-flow Pulse Ionization Chamber," *Appl Radiat Isot* 43:119-125, 1992.
18. J. K. Hartwell and S. G. Goodwin, "Pulser Injection with Subsequent Removal- Implementation and Applications," *IEEE Trans on Nucl Sci*, NS-36, No.1, 1989.
19. E. W. Killian and J. K. Hartwell, *VAXGAP: A Code for the Routine Analysis of Gamma-Ray Pulse Height Spectra on a VAX Computer*, EGG-2533, May 1988.
20. E. W. Killian and D. A. Femec, *An Operator's Guide to VAXGAP*, Rev. 2.0, ST-CS-031-90, November 1990.
21. G. K. Rusch, W. P. McDowell, and W. G. Knapp, "The ZPR-9 Airborne Plutonium Monitoring System," *IEEE Tran on Nucl Sci*, Vol.NS-23, No.1:690-692, 1976.
22. T. J. Yule, "An On-Line Monitor for Alpha-Emitting Aerosols," *IEEE Tran on Nucl Sci*, Vol.NS-25, No.1:762-766, 1978.
23. L. A. Currie, "Limits for Qualitative Detection and Quantitative Determination," *Anal Chem* 40:586-593, 1968.
24. C. W. Sill, "Precipitation of Actinides as Fluorides or Hydroxides for High-Resolution Alpha Spectrometry," *Nucl and Chem Waste Management*, Vol.7:201, 1987.
25. C. W. Sill and F. D. Hindman, "Preparation and Testing of Standard Soils Containing Known Quantities of Radionuclides," *Anal Chem* 46:113-118, 1974.
26. C. W. Sill, "Long-term Stability of Solid Standards for Radiochemical Analysis," *Anal Chem* 61:2255-2258, 1989.

END

**DATE
FILMED**

9/2/93