Science Applications International Corporation

FUSRAP St Louis Laboratory Quality Assurance Plan and Laboratory Procedures Manual

800 Oak Ridge Turnpike • P.O. Box 2502 • Oak Ridge, TN 37831

SCIENCE APPLICATIONS INTERNATIONAL CORPORATION
FUSRAP ST LOUIS LABORATORY QUALITY ASSURANCE PLAN and LABORATORY PROCEDURES MANUAL

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LABORATORY QUALITY ASSURANCE PLAN

FOR THE

FUSRAP ST. LOUIS RADIOLOGICAL LABORATORY

ST. LOUIS MISSOURI

JANUARY 1999

Revision 0

Prepared for
U.S. Army Corps of Engineers (USACE)
St. Louis District

Prepared by
Science Applications International Corporation

FUSRAP ST. LOUIS

RADIOLOGICAL LABORATORY

LABORATORY QUALITY ASSURANCE PLAN APPROVALS

Revision 0

C. Glen Cowart, SAIC QA/QC Officer — Date: 01/08/99

James R. Moos, St. Louis EC&HS Officer — Date: 01/19/99

Brian Sparks, Laboratory Supervisor — Date: 01/19/99

FOREWORD

This Laboratory Quality Assurance Plan (LQAP) has been written to assure that all of the systems, services, processes, and deliverables of the Formerly Utilized Sites Remedial Action Program (FUSRAP) St. Louis Radiological Laboratory are of a quality that meets or exceeds client requirements, and to foster a culture in which there is a commitment to a continuously improving standard of quality.

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ATTACHMENTS

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ACRONYMS AND INITIALISMS

ASTM	American Society for Testing and Materials
COC	chain of custody
DQO	data quality objective
EPA	U.S. Environmental Protection Agency
ES&H	Environmental Safety and Health
FUSRAP	Formerly Utilized Sites Remedial Action Program
LCS	laboratory control sample
LQAP	Laboratory Quality Assurance Plan
MDC	Minimum Detectable Concentration
MSDS	Material Safety Data Sheet
NIST	National Institute of Standards and Technology
OSHA	Occupational Safety and Health Administration
PM	preventive maintenance
QA	quality assurance
QAP	Quality Assurance Program
QAPP	Quality Assurance Project Plan
QC	quality control
RPD	relative percent difference
SAIC	Science Applications International Corporation

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1.0 INTRODUCTION

This Laboratory Quality Assurance Plan (LQAP) has been developed to provide laboratory-specific quality assurance requirements and protocols which are to be adhered to when performing laboratory operations including, but not limited to, sample handling, preparation, analysis, and reporting. Specific requirements are delineated throughout the remainder of this LQAP.

This LQAP must be used in conjunction with the Science Applications International Corporation (SAIC) Quality Assurance Program (QAP) and applicable administrative and technical procedures. The SAIC QAP applies to all Formerly Utilized Sites Remedial Action Project (FUSRAP) activities including those performed by subcontractors, and will be referred to in this LQAP, where appropriate. The SAIC QAP describes the management controls, objectives, and scope of the quality program, outlines the general policies to be followed, and identifies associated responsibilities.

2.0 ORGANIZATION

The subsections below summarize the responsibilities of functional positions associated with management and operation of the FUSRAP St. Louis Radiological Laboratory. In general, all personnel have the responsibility and authority to stop work when there is a clear danger to the health or safety of personnel, and to report to management any situations or practices which could be unsafe or adverse to the quality of work.

2.1 SAIC ST. LOUIS PROJECT MANAGER

The Project Manager has overall responsibility for operation of the FUSRAP St. Louis Radiological Laboratory and for implementation of this LQAP.

2.2 SAIC RADIOLOGICAL LABORATORY TASK MANAGER

The Task Manager has direct responsibility for implementation of this LQAP and all applicable procedures by the Radiological Laboratory staff. The Task Manager is also specifically responsible for:

- Training laboratory personnel to assure that suitable proficiency is achieved and maintained.
- Periodic review and revision of laboratory procedures.

- Coordination with the Project Manager and USACE to resolve problems related to operation of the laboratory that could prevent completion of required analysis.
- Instruction in laboratory safety (including radiation safety), hazard communications, and hazardous waste management.

2.3 RADIOLOGICAL LABORATORY SUPERVISOR

The Supervisor is responsible for implementation of the requirements specified in this LQAP and applicable procedures for all laboratory activities. The Supervisor assures that laboratory personnel receive and document training appropriate to their duties, and that they perform their duties in accordance with specified plans, procedures and protocols. The Supervisor is also specifically responsible for:

- Operation, maintenance, and calibration of the Gamma Spectroscopy system, the Alpha Spectroscopy system, and the Gas Proportional Counting system.
- Determination of isotopic thorium and uranium, and radium-226, each via alpha spectroscopy through chemical separation, and also determination of gamma emitting isotopes in bulk samples via gamma spectroscopy
- Control and retention of laboratory quality assurance records.
- Determination of gross alpha and beta on air filters and swipes via Gas Proportional Counting.

2.4 RADIOLOGICAL LABORATORY STAFF

Staff members are responsible for the quality of their work and for implementing the requirements of this LQAP and applicable procedures on a daily basis.

2.5 SAIC QA/QC OFFICER

The QA/QC Officer is responsible for oversight of laboratory activities and specifically for:

- Performance and documentation of periodic surveillances and/or audits in accordance with SAIC procedures.
- Initiation of corrective action, as necessary and performance of any follow-up, as required.

3.0 QA OBJECTIVES FOR MEASUREMENT DATA

Quality Assurance objectives for measurement data will be in accordance with the objectives for precision, accuracy, representativeness, completeness and comparability (PARCC) described in the overall Quality Assurance Project Plan (QAPP) found in the Sampling and Analysis Program Plan (SAPP) for the St. Louis FUSRAP Sites. Any additional or different objectives described in activity-specific QAPPs and/or survey plans for the various St. Louis Sites scopes of work will also be addressed.

4.0 MATERIAL PROCUREMENT AND CONTROL

The methods for procurement, receipt, storage, identification, and use of quality-related, purchased items are documented in the laboratory operating procedures.

The quality level of each purchased item will be determined by its eventual use. For commercial items, catalog numbers referencing the item description in the supplier's catalog provide sufficient documentation. Other items which provide evidence of quality, such as reference standards, or certificates of analysis and traceability will be submitted with the standards. These certificates will be maintained by the laboratory as quality records.

5.0 SAMPLE CUSTODY

5.1 GENERAL

Identification and documentation of the history of sample possession from collection through analysis and ultimate disposition (i.e., chain of custody) will be maintained to ensure that the validity of the sample has not been compromised. The overall objective of sample custody is to ensure the traceability of a given sample from the time it is collected until final disposition (i.e., reporting or disposal). Chain of custody will be maintained in accordance with the protocols specified in the QAPP.

5.2 SAMPLE IDENTIFICATION

Each sample submitted for analysis will be uniquely identified to ensure proper identification for prompt, correct, and complete analysis for all parameters requested. Sample identification numbers will be assigned for each identified task in accordance with the protocols specified in the QAPP. The field sampling team will be responsible for using these numbers for proper identification of samples. Samples improperly identified may be refused.

5.3 CHAIN-OF-CUSTODY PROCEDURES

Chain-of-custody procedures are used for all samples collected during field activities. These procedures are in accordance with the *NEIC Policies and Procedures* (EPA 1985). These procedures provide for sample labeling and tracking reports that contain the following types of information:

- Unique sample identification
- Documentation of specific reagents or supplies that become an integral part of the sample (absorbing reagents, filters, etc.)
- Sample preservation methods
- Sample custody logs

Upon receipt, the laboratory will control chain-of-custody in accordance with laboratory procedure ML-013.

6.0 CALIBRATION PRACTICES

All work performed in the onsite laboratory will be in accordance with the approved Laboratory Procedures Manual, latest revisions. Refer to the subsections below for specifics with regard to calibration.

6.1 GAMMA SPECTROSCOPY INSTRUMENTATION

Gamma spectroscopy instrumentation will be calibrated as described in laboratory procedure # ML-003.

6.2 ALPHA SPECTROSCOPY INSTRUMENTATION

Alpha spectroscopy instrumentation will be calibrated as described in laboratory procedure # ML-004.

7.0 PREVENTIVE MAINTENANCE

Preventive maintenance (PM) activities are those routine scheduled activities which are performed to maintain proper instrument and equipment performance, minimize deterioration, and prevent instruments and equipment from failing during use.

Routine PM activities are normally performed prior to instrument use to ensure that instruments which have not passed required inspections are not inadvertently used. Vendor-supplied PM/service contracts will be utilized for all major instrumentation. Additionally, all PM activities will be documented by the person performing the maintenance in a logbook assigned to the appropriate instrument. In some instances the PM activities may be logged on check sheets that are maintained on file in the laboratory. Required PM activities are detailed in the following laboratory procedures:

ML-003, Operation of the Gamma Spectroscopy System
ML-007, Balance Calibration Check
ML-011, Use and Maintenance of Mechanical Pipette
ML-012, D. I. Water Back-up Check.

8.0 INTERNAL QUALITY CONTROL CHECKS

8.1 METHOD BLANKS

The purpose of method blank analysis is to determine the existence and magnitude of potential analytical process contamination problems. If problems with the blank samples exist, all of the associated samples must be carefully evaluated. Method blank results shall not be greater than 2X their respective Minimum Detectable Concentration (MDC) for the radionuclide of interest for that sample. However, it is possible that method blank contamination can occur requiring corrective action. If it is determined that the cause is related to sample preparation, all affected samples prepared with the affected method blank will be prepared again and recounted. In instances where the samples did not require reanalysis, a detailed description will be included in the laboratory case narrative.

8.2 LABORATORY CONTROL SAMPLES (LCS)

A laboratory control sample (LCS) will be processed with each batch of samples. The analyst will process samples in accordance with the laboratory procedures manual. The evaluation of the LCS analysis will be based on an examination of observed value and an accepted reference value. A statistical analysis (normalized absolute difference) of the data may be performed when cursory evaluation indicates a problem with the results. If the two results agree within 2 standard deviations, more detailed evaluation will generally not be necessary. Results of the LCS will be included in the data deliverable. If it is determined that a problem occurred that is related to sample preparation, all samples prepared with the affected LCS will be prepared again and recounted. In instances where the samples did not require reanalysis, a detailed description will be included in the laboratory case narrative.

8.3 LABORATORY DUPLICATE

Duplicate aliquots of randomly selected samples will be processed with each batch of samples. The analyst will process samples in accordance with the laboratory procedures manual. Evaluation of the duplicate analysis will be based on examination of the duplicates. A statistical analysis (normalized absolute difference) of the data will be performed when cursory evaluation indicates a problem with the results. If the two results agree within 2 standard deviations, more detailed evaluation will generally not be necessary. Results of the duplicate analysis will be included in the data deliverable. If it is determined that a problem occurred that is related to sample preparation, all samples prepared with the affected duplicate will be prepared again and recounted. In instances where the samples did not require reanalysis, a detailed description will be included in the laboratory case narrative.

8.4 TRACER USE FOR ALPHA SPECTROSCOPY

A National Institute of Standards and Technology (NIST) traceable radioactive tracer will be added in a chemical and physical form appropriate to the analytical procedure to help assure uniform reproduction of the path followed by radionuclides present in the sample. Acceptable criteria for tracer recovery is project specific and is defined in the sampling process design, also termed sampling and analysis plan. Otherwise, tracer recoveries must be greater than 35% or less than 120%. Samples that fail to meet the specified criteria will be prepared again and recounted. In instances where the samples did not require reanalysis, a detailed description will be included in the laboratory case narrative.

8.5 PERFORMANCE EVALUATION

Additional overall analytical quality will be evaluated based on participation in external quality control programs as required by regulatory agencies. Such programs may include the Environmental Protection Agency (EPA), Research and Development Quality Assurance program, and the Department of Energy (DOE) mixed analyte performance evaluation program. The FUSRAP St. Louis Radiological Laboratory will participate in such a program. Results of performance evaluation samples will be received by the laboratory. A copy will be maintained in the laboratory and a copy will be sent to the SAIC Control Records Facility in accordance with the SAIC QAP.

9.0 ANALYTICAL PROCEDURES

Analytical procedures will be performed in accordance with the FUSRAP St. Louis Laboratory Procedures Manual. Specific references are given in the applicable procedure to be performed. Methods are compiled from national standards including, but not limited to EPA standard methods, American Society for Testing and Materials (ASTM) standards, and others.

10.0 DATA VERIFICATION

As part of the analytical quality control, the Radiological Laboratory will determine precision and accuracy by calculating relative percent difference (RPD), normalized absolute difference, and percent recovery (%R), where applicable. The formulas for calculating RPD and %R are given in the appropriate procedures in the Laboratory Procedures Manual, or are calculated automatically by certified software using industry accepted time-proven algorithms. The following formulas are used for calculating normalized absolute difference.

$$\frac{LCS_{meas.} - LCS_{exp.}}{\sqrt{(CU_{LCS\,meas.})^2 + (CU_{LCS\,exp.})^2}}$$

$LCS_{meas.}$ = Measured LCS result
$LCS_{exp.}$ = Expected result of LCS
$CU_{LCS\,meas.}$ = 2σ Counting Uncertainty of LCS measured result
$CU_{LCS\,exp.}$ = 2σ Counting Uncertainty of LCS expected result

If the normalized difference is either greater than 2.58 or less than -2.58, appropriate corrective action will be initiated in accordance with Section 8.2.

$$\frac{|S - D|}{\sqrt{(CU_S)^2 + (CU_D)^2}}$$

S = Sample result
D = Laboratory duplicate result
CU_S = 2σ Counting Uncertainty of the sample
CU_D = 2σ Counting Uncertainty of the duplicate

If a normalized absolute difference is greater than 2.58, corrective action will be initiated in accordance with Section 8.3.

Analytical review of the data generated for submittal will be performed in accordance with Laboratory Procedure #ML-014.

Project data review will be performed in accordance with the QAPP.

11.0 DATA REPORTS

Data reports generated for submittal will be prepared in accordance with laboratory procedure #ML-014. Data will be reported in accordance with the requirements specified in the QAPP.

12.0 RECORDS MANAGEMENT

12.1 DOCUMENT CONTROL

Approved Radiological Laboratory documents such as this LQAP and the laboratory procedures will be controlled and maintained in accordance with the SAIC QAP requirements for document control. Controlled documents are managed to assure that only current and approved documents are in use and to preclude the use of obsolete or superseded documents.

12.2 DOCUMENT REVISIONS

Changes or revisions to documents and data are reviewed by the same organizations that performed the original review and approval. Revisions are managed in accordance with the requirements of the SAIC QAP.

12.3 LABORATORY-SPECIFIC RECORDS

The following records and documents are required to be maintained by the laboratory:

- Logbooks (ML-001, ML-007, ML-009, ML-013, ML-014)
- Daily quality control (QC) reports (ML-003)
- Daily background checks (ML-003)
- Weekly chamber background checks (ML-003)
- Weekly energy and efficiency calibration (ML-004)
- Daily pulser checks (ML-004)
- Calibration checks for pipettes (ML-011)
- Documentation of instrument tag-out (ML-010)
- Deionized water conductivity checks (ML-012)
- All sample documentation (e.g., COC, sample results) (ML-014)
- Corrective action documents (ML-003, ML-004)
- Analytical standard certificates (ML-017)
- Standard preparation worksheets (ML-017)
- Sample preparation logs (ML-001)
- Analysis run logs (ML-014)

13.0 NONCONFORMANCE AND CORRECTIVE ACTION

Conditions adverse to the quality or integrity of laboratory data will be identified and corrected as soon as practical. Where necessary, follow-up action will be taken by laboratory personnel and the QA/QC Officer to verify implementation of the corrective action and

determine the need for any additional action. Nonconformance and corrective action will be documented and managed in accordance with the requirements of the SAIC QAP.

14.0 AUDITS

Periodic assessments of laboratory operations will be performed in accordance with the requirements of the SAIC QAP and associated administrative procedures. Types of assessments include internal, independent assessments (audit or surveillance) performed by either SAIC QA or Environmental Safety and Health (ES&H) personnel, and management reviews performed by line or project management personnel which are not considered to be independent.

Audits or surveillances will be performed annually, at a minimum, or as determined by the QA/QC Officer.

Any discrepancies discovered during the course of an audit or surveillance will be documented and handled in accordance with the corrective action process described in the SAIC QAP and associated administrative procedures

15.0 QUALITY REPORTS TO MANAGEMENT

Performance evaluation reports are compiled by the Laboratory Manager at least annually and submitted to the SAIC Data Validation organization. Quarterly QC reports are submitted for review by the Laboratory Manager, and are submitted as quality records in accordance with the SAIC QAP and associated administrative procedures. Additional copies are maintained at the laboratory.

16.0 PERSONNEL QUALIFICATION

Training requirements, tracking, and review frequency are managed in accordance with the SAIC QAP and associated administrative procedures.

16.1 GENERAL REQUIREMENTS

The following general requirements apply to the laboratory:

- Job descriptions – developed by laboratory management. These descriptions identify training requirements associated with specific job titles.

- Tracking – project-specific training requirements are entered into a training database by project administrative personnel. Individual training reports are issued quarterly to all staff through laboratory management.
- Review frequency – the laboratory Task Manager or designee reviews position descriptions and training requirements at least annually.

16.2 SPECIFIC REQUIREMENTS

Training requirements are defined in the SAIC Health Physics Manual, Section 6.3. Laboratory employees are required to have the following training as a minimum: Rad Worker II, OSHA 40 Hour HAZWOPER, Site Specific, and training required by Section 6.3 of the Health Physics Manual.

Additional training for laboratory employees includes the Mobile Laboratory Procedures (Attachment I), and Material Safety Data Sheets (MSDS) applicable to laboratory operation (Attachment II).

17.0 REFERENCES

Handbook for Analytical Quality Control in Radioanalytical Laboratories, EPA-600/7-7-088, August 1977.

Standard Test Method for Radionuclides of Radium in Water, Method D2460-70, 1991 Annual Book of ASTM Standards, Section 11, Water and Environmental Technology, Vol. 11.02.

"Upgrading Environmental Radiation Data", J.E. Watson, EPA 520/1-80-012, August 1980. Canberra NDSP Sample Counting Software.

1990 Annual Book of ASTM Standards, Volume 12.02, E181.

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Claude W. Sill, Kenneth Puphal, and Forest D. Hindman, Anal. Chem.46, 1725 (1974).

Claude W. Sill and Roger L. Williams, Anal Chem 53, 412 (1981).

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Roger P. Bernabee, Donald R. Percival, and Forest D. Hindman, Anal. Chem. 52, 2351 (1980).

Annual Book of ASTM, Standards Vol. 11.02, pp. 300-303, pp. 380-381, pp. 407-411.

Claude W. Sill, Nuclear and Chemical Waste Management, Vol. 7, pp. 201-215, (1987).

ORISE/ESSAP, Correspondence with Jack Beck, Mark Laudeman, and Sally Shipley.

Claude W. Sill, David Sill, Waste Management, Vol. 9, pp. 219-229 (1989).

Claude W. Sill, "Determination of Radium-226 in Ores, Nuclear Wastes and Environmental Samples by High-Resolution Alpha Spectrometry" Nuclear and Chemical Waste Management, Vol.7, pp.239-256, (1987).

David W. Sill and Claude W. Sill, "Simultaneous Determination of the Actinides in Small Environmental Samples", Radioactivity & Radiochemistry, Vol. 5, No.2,pp.8-19 (1994).

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ATTACHMENT I

FUSRAP Mobile Laboratory Procedures Training Record

Employee:_____
Job Title:_____
Supervisor:_____

Procedure	Date Certified	Method	Instructor
ML-001 Sample Preparation I			
ML-002 Sample Preparation II			
ML-003 Operation of the Gamma Spectroscopy System			
ML-004 Operation of the Alpha Spectroscopy System			
ML-005 Isotopic Determination of Thorium (Fusion)			
ML-006 Determination of Radium-226 (Fusion)			
ML-007 Balance Calibration Check			
ML-008 Glassware Washing and Storing			
ML-009 Waste Disposal			
ML-010 Segregation of Out-of-Tolerance Instruments			
ML-011 Use and Maintenance of Mechanical Pipettes			
ML-012 D.I. Water Back-Up Check			
ML-013 Sample Management			
ML-014 Data Package Assembly, Transmittal, and Generation of Electronic Deliverable			
ML-015 Isotopic Determination of Uranium (Fusion)			
ML-016 Determination of Self-Absorption Factors			
ML-017 Radioactive Reference Standard Solutions and Records			

ATTACHMENT II

MSDS Training Verification

Employee_____

My signature below indicates that I have read all the MSDS's applicable to the operations for which I have been trained and understand the physical and health hazards and all of the safety and handling requirements.

Employee Signature _____ Date _____

Supervisor Signature _____ Date _____

FUSRAP
LABORATORY PROCEDURES MANUAL

ML-001
Rev. 0

SAMPLE PREPARATION

Approved By:

Project Manager: James R. Moos 1/24/99
James R. Moos

SAIC Analytical Lab Coordinator: Nile Leudtke 1/22/99
Nile Leudtke

Laboratory Supervisor: Brian J. Sparks 1/26/99
Brian J. Sparks

QA/QC Officer: Glen Cowart 1/25/99
Glen Cowart

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1.0 PURPOSE AND SCOPE

1.1 This procedure provides instructions for the initial preparation of environmental samples for laboratory analysis.

1.2 This procedure applies to all samples requiring drying, grinding, chopping, or other preparation techniques prior to specific analysis.

2.0 SUMMARY OF METHOD

2.1 This procedure provides for the initial preparation of all samples other than urine and fecal samples. The samples are either prepared wet or dried and homogenized as necessary to provide a uniform matrix.

3.0 DEFINITIONS

3.1 MSDS – Material Safety Data Sheets

4.0 SAFETY

4.1 Lab coat, gloves, and safety glasses will be worn at all times when in the laboratory.

4.2 When using or preparing reagents that consist of 1000 milliliters (ml) or more of strong caustic or acidic materials, the analyst will also be required to wear an apron, and face shield.

4.3 Acids will be used in the appropriate designated hoods.

4.4 MSDS – Material Safety Data Sheets – will be available for other specific safety instructions on chemicals and reagents.

4.5 Appropriate precautions will be adhered to when handling radioactive materials, as defined in Radwork II Training. See the SAIC Health Physics Manual, Section 6.3.

5.0 INTERFERENCES

None

6.0 EQUIPMENT/MATERIALS

6.1 Balance (0-3600.00 grams capacity)

6.2 Drying oven

6.3 Mortar and pestle

6.4 Muffle furnace

6.5 Pulverizer

6.6 Grinder

6.7 Paint can shaker

6.8 Assorted laboratory glassware

6.9 Assorted stainless steel or aluminum pans

6.10 Assorted Marinelli beakers

6.11 Assorted Petri dishes

6.12 Assorted tools and routine laboratory equipment

7.0 REAGENTS

None

8.0 PROCEDURE

8.1 Soil Samples, All analysis

8.1.1 Soil samples are prepared in one of two geometries (Marinelli or Petri dish) depending on the sample volume.

8.1.1.1 For the Marinelli geometry, approximately 500 grams (g) or 500 ml of sample is required for the analysis.

8.1.1.2 For Petri dish geometry (3.50 inches diameter) approximately 68 g of sample is required for the analysis.

8.1.2 Weigh and record the tare weight of a drying pan, and place sample in pan. Weigh and record the weight as the gross wet weight in grams on the appropriate laboratory worksheet.

8.1.3 Place the sample in drying oven until dry.

8.1.4 Remove the sample from the oven and allow it to cool to room temperature. If visible moisture is observed, place sample back in the drying oven and heat again until dry.

8.1.5 Weigh and record the weight as the gross dry weight in grams on the appropriate laboratory worksheet.

8.1.6 Homogenize the sample using the Bico pulverizer, Grinder, Paint can shaker, or mortar and pestle. Clean grinding equipment between samples as described in Section 8.2. Transfer the sample to an appropriate holding container and label with the laboratory sample number.

8.1.7 Weigh an empty container with lid, (i.e., tare weight), fill with the required amount of sample and close the sample container securely. Determine net weight of sample and record. Seal with vinyl tape.

8.1.8 Write the appropriate sample information (ID number, ect.) on the container, on the appropriate laboratory worksheet.

8.1.9 Submit for counting (Gamma analysis).

8.2 After grinding each sample, clean the grinder as follows:

8.2.1 After removing the sample, vacuum out the grinding chamber using the rough prep vacuum system.

8.2.2 Release the grinding chamber and grind approximately one cup of ≈2" gravel.

8.2.3 Place the used gravel in the Dry Active Waste container and vacuum out the grinding chamber. If there is any sample material remaining stuck to the grinding plates or walls of the grinding chamber, use a scrub brush to remove the material.

8.2.4 If a mortar and pestle is used for grinding the sample, rinse the mortar and pestle with deionized water between uses and allow both parts to dry before using again.

8.3 If specific chemical analysis is requested, place approximately 5 g of material in a labeled centrifuge tube for use in gross alpha/beta and isotopic analysis.

8.4 Vegetation Samples, Gamma Isotopic

8.4.1 Vegetation samples are prepared in 500 ml Marinelli geometry. A sufficient quantity of sample to fill the beaker is needed for this geometry.

8.4.2 Weigh empty container with lid (i.e., tare weight) and record in the appropriate laboratory worksheet and Sample Prep logbook.

8.4.3 Use the appropriate tool to cut the vegetation sample into small pieces such that the sample can be placed into the 500 ml Marinelli beaker. If the results are to be reported in dry weight proceed with 8.1.2 through 8.1.5.

8.4.4 Cover the container, determine net weight, and secure the lid with vinyl tape.

8.4.5 Write appropriate sample information (ID number, etc.) on the container and on the appropriate laboratory worksheet.

8.4.6 Submit for counting (Gamma analysis)

8.4.7 If the Bico pulverizer or mortar and pestle was used, clean as described in Section 8.2.

8.5 Air Filters, Gamma Isotopic

8.5.1 Air filter samples are counted in the appropriate air filter geometry (i.e. single filter or composite filters).

8.5.2 Load the air filter(s) into the Petri dish. Cover dish and secure lid with vinyl tape.

8.5.3 Write the sample information (ID number, etc.) on the lid of the dish and on the appropriate laboratory worksheet.

8.6 Core Samples, All analysis

8.6.1 Cut Shelby tube with a rigid pipe cutter and slice sample at 2 inch intervals.

8.6.2 After cutting each slice off of the sample tube clean the pipe cutting blade with a piece of clean paper towel. If necessary, use a wet paper towel.

8.6.3 If more than 500 g of sample is available, carefully remove sample from every other sliced section of the Shelby tube, dry and grind as in Sections 8.1.1–8.1.8, and place in a tared 500 ml Marinelli beaker (tare weight

includes empty container and lid). If less than 500 g of sample is available, all of the sample may be utilized as directed by the laboratory supervisor or designee.

8.6.4 Clean grinding equipment between samples as described in Section 8.2.

8.6.5 Place approximately 5 g of material in a labeled centrifuge tube for use in gross alpha/beta and isotopic analysis.

8.6.6 Close the sample container securely, determine the net weight, seal with vinyl tape, and store unused portions.

8.6.7 Write sample information (ID number, ect.) on the container, on the appropriate laboratory worksheet.

8.6.8 Submit for counting (Gamma analysis).

8.7 Water, Gamma Isotopic

8.7.1 All liquid samples other than bioassay samples are made up in 500 ml polyethylene Marinelli beakers. The volume of the sample depends on the activity level and the amount of sample supplied by the customer. It may be required to concentrate the sample by drying on a hot plate as directed by the Lab Manager.

8.7.2 Using a clean graduated cylinder, measure the required sample volume (500 ml) and pour the liquid into the appropriate Marinelli beaker.

8.7.3 Place lid securely on the Marinelli beaker and seal with electrical tape. Inspect for any leakage before sending the sample for counting.

8.7.4 Write sample information (ID number, etc.) on the beaker on the appropriate laboratory worksheet.

8.8 Air Filter Samples – Isotopic Analysis – Cellulose Filters

8.8.1 The air filter sample is placed in a suitable sized glass beaker and the beaker placed on the hot plate.

8.8.2 The sample is heated in concentrated Sulfuric acid until it is dissolved. Remove sample from the hotplate, allow to cool. Bring to desired volume with Deionized water.

8.9 Air Filter Samples – Isotopic Analysis – Fiberglass Filters

8.9.1 Place the air filter in a centrifuge tube. Add one to two milliliters of concentrated hydrofluoric acid, HF, to the sample.

8.9.2 Gently heat in the water bath until the filter dissolves. Bring to desired volume with Deionized water.

8.10 Vegetation Samples – Gross Alpha/Beta and Isotopic Analysis

8.10.1 Place the sample in a tared container. Weigh and record the weight as the wet weight in grams, if not accomplished in step 8.3.1 above. Record the data on appropriate laboratory worksheet and in Sample Prep logbook.

8.10.2 Place the sample in a drying oven until dry.

8.10.3 Remove sample from the oven and allow to cool to room temperature. If the sample is not dry, place in drying oven and heat until dry.

8.10.4 Weigh and record the weight as the dry weight in grams.

8.10.5 Homogenize the sample using a mortar and pestle.

8.10.6 Rinse the mortar and pestle with DI water between uses and allow to dry.

8.10.7 Place the sample in a labeled centrifuge tube for transfer to analysis.

9.0 QUALITY CONTROL

9.1 The technician performing this procedure shall be responsible for inspection of samples after ashing, blending, drying, or grinding to determine if processes are complete. The technician shall be responsible for inspecting labels for correctness and for inspecting quantities of samples.

9.2 The technician shall also maintain a high level of cleanliness of the sample work area and equipment to minimize the possibility of cross contamination.

10.0 REFERENCES

10.1 Standard Methods, 17th Edition, 1989, Method 2550.

10.2 FUSRAP Laboratory Quality Assurance Manual.

10.3 SAIC Health Physics Manual.

FUSRAP
LABORATORY PROCEDURES MANUAL

ML-003

Rev. 0

OPERATION OF THE GAMMA SPECTROSCOPY SYSTEM

Approved By:

Project Manager:	James R. Moos	1/24/99
SAIC Analytical Lab Coordinator:	Nile Leudtke	1/22/99
Laboratory Supervisor:	Brian J. Sparks	1/26/99
QA/QC Officer:	Glen Cowart	1/25/99

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1.0 PURPOSE AND SCOPE

1.1 This procedure establishes the method for the operation, calibration, and maintenance of the gamma spectroscopy systems.

1.2 This procedure applies to the operation of the high energy (5 - 3000 KeV) gamma spectrometers.

2.0 SUMMARY OF METHOD

A typical gamma-ray spectrometry system consists of a germanium detector (with its cryostat, liquid nitrogen dewar, and preamplifier) in conjunction with a detector bias supply, linear amplifier, ADC (Analog to Digital Converter), AIM (Acquisition Interface Module), and a multichannel analyzer.

Germanium detectors used for the detection of gamma-rays are actually semiconductor diodes having P-I-N (Positive-Intrinsic-Negative) structure in which the intrinsic (I) region is sensitive to ionizing radiation, particularly x-rays and gamma-rays. Under the reverse bias applied, an electric field extends across the intrinsic or depleted region. When gamma-rays interact with the material within the depleted region of the detector, charge carriers (holes and electrons) are produced and are swept by the electric field to the P (positive) and N (negative) electrodes. This charge, which is proportional to the energy deposited in the detector by the incident gamma-ray, is converted into a voltage pulse by an integral charge sensitive preamplifier.

The signal is then amplified and sent to an ADC where the signal is converted from an analog signal to a digital signal, which can be interpreted by a central VAX (or equivalent) computer. The digital signal is then sent to the AIM where the pulse is entered into a spectral channel corresponding to a particular gamma-ray energy. The data is also sent to a central VAX computer, where the spectral data is stored and visually displayed. When acquisition is completed, the VAX computer quantifies the events recorded in each channel of the sample spectrum. The peaks of the spectrum, characteristic of both a particular radionuclide(s) and the quantity of the radionuclide present in the sample, are identified by comparison to a nuclide library stored on disk.

Prior to counting samples, the detector and associated electronics must be energy, resolution, and efficiency calibrated for each counting geometry. Energy calibration is performed by counting a radioactive source containing known gamma-ray emitting radionuclides, at a fixed amplifier gain. An energy calibration factor is then generated by determining the channel numbers corresponding to full energy peak centroids from gamma-rays emitted over the full energy range of interest from multipeaked and/or multinuclide radioactivity sources. Efficiency calibration is accomplished by counting a calibrated source of a particular geometry at a reproducible source-to-detector orientation. The measured emission rate of the calibration standard is then compared to the actual disintegration rate to determine the

detector counting efficiency. The values for energy and efficiency calibration are maintained in configuration files which are referenced when analyzing samples.

3.0 DEFINITIONS

3.1 Efficiency – the percent of decay events from a standard radioactive source, in a specific reproducible geometry, that are seen and measured by a detector.

3.2 Traceable Calibration Standard – a calibrated radioactive source prepared as or from a standard reference material traceable to National Institute of Standards & Technology (NIST), New Brunswick Laboratory (NBL), Amersham, or other acceptable supplier.

3.3 Check Source – a radioactive source used to confirm the satisfactory operation of the instrument.

3.4 ADC – analog to digital converter.

3.5 FWHM (Full Width Half Maximum) – the full width of a gamma-ray peak distribution measured at half the maximum peak height, measured above the continuum (background).

3.6 Dead Time – the time while the amplifier is processing a pulse and is unable to process another pulse.

3.7 Geometry – a standard sample or source counting configuration (i.e. 20 ml vial, Marinelli beaker, petri dish, jar, and others) and its relationship to the detector.

4.0 SAFETY

4.1 Individuals performing this procedure should be aware of the precautions necessary for the proper handling of radioactive materials, as defined in Radiation Worker II. See the SAIC Health Physics Manual, Section 6.3.

4.2 Follow the manufacturer's instructions for set up, intercomponent connections, and preliminary testing of the equipment. Observe all of the manufacturer's limitations and cautions.

4.3 For HPGe (High Purity Germanium) detectors, the crystal may be allowed to warm to room temperature and subsequently recooled. These cycles should be minimized to extend the life of the crystal. If warmed, the crystal must be warmed all the way to room temperature before recooling.

4.4 Ensure the detector coldfinger is submersed in liquid nitrogen at least 12 hours before applying a bias to the crystal.

4.5 Never exceed the manufacturer's recommended operating voltage for the detector.

4.6 Ensure the high voltage power supply is set to zero before energizing the unit. Turn on the high voltage power supply and begin to slowly raise the voltage (approx. 100 V/sec) until the correct operating voltage is reached.

4.7 When handling LN_2 (liquid nitrogen), stay clear of confined spaces, wear non-absorbent, insulated gloves, and observe all compressed gas safety precautions found on the Material Safety Data Sheets.

5.0 INTERFERENCES

None.

6.0 EQUIPMENT

6.1 High Purity Germanium detector with built in preamplifier.

6.2 Graded lead shield.

6.3 Dewar flask (30 Liter).

6.4 Linear amplifier.

6.5 Analog to Digital Converter (ADC).

6.6 Liquid nitrogen level monitor (optional).

6.7 Acquisition Interface Module (AIM).

6.8 Ethernet transceiver with cable.

6.9 Multichannel Analyzer.

6.10 VAX Workstation, or equivalent, with gamma analysis software.

7.0 REAGENTS AND STANDARDS

7.1 Traceable Calibration Standard - the actual standard is dependent upon the sample geometry being calibrated.

7.2 Liquid nitrogen (LN_2)

8.0 PROCEDURE

8.1 The Laboratory Supervisor or his designee has overall responsibility for implementation of this procedure: for determination of the applicability of the procedure to any particular sample type, for the review of data generated by the procedure, for approval of final results, and for certifications of laboratory personnel performing the procedure if applicable.

8.2 The Laboratory Supervisor is responsible for investigation of any quality failures that arise in the application of this procedure and approves any procedural modification that might be required.

8.3 This procedure is performed only after certification by the Laboratory Supervisor. This certification requires that the analyst receive training by senior laboratory staff and includes a laboratory demonstration of competence. Training is documented in accordance with SAIC QAPP 2.1.

8.4 Energy and FWHM Calibration

This section should be performed either during initial instrument setup, annually, after system maintenance that may affect the calibration, prior to efficiency calibrating, or whenever a problem is suspected.

8.4.1 Position an appropriate calibration standard on the detector and close the shield.

8.4.2 Following section 8.9, count the standard until a minimum of 1000 counts have been acquired in the target peaks.

8.4.3 From the **Gamma Counting Main Menu** select **6) Calibrate a Detector**, to access the **Detector Calibration Menu**.

8.4.4 Select **1) Energy/Shape Initial Calibration**.

8.4.5 Answer the prompts:

8.4.5.1 Choose detector from **Detector's Menu**.

8.4.5.2 Enter **Yes** to use the spectrum acquired in 8.4.2 above.

8.4.5.3 Enter the geometry name used for calibration.

8.4.5.4 Complete the on screen choices as required.

8.4.5.5 If the Energy/FWHM calibration being performed is an initial calibration, position the cursor over the appropriate peaks when prompted, and press return. If the calibration is an update, the system will automatically select appropriate peaks and display a graph.

The Energy and FWHM calibration parameters will now be calculated and a graph displayed for each. To clear the graphs, place the cursor arrow over the **DISMISS** key on the graph and press the left mouse button and then press **Return** to continue.

8.4.6 Review the calibration results displayed for energy and FWHM. The energy calibration slope should be approximately 0.5 KeV/channel. The FWHM calibration should calculate a FWHM at 1332.5 KeV to within 0.10 of the rated FWHM of the detector. If the values attained are not within these tolerances, contact the Lab Supervisor.

8.4.7 Submit the calibration report to the Lab Supervisor for review.

8.5 Efficiency Calibrations

This section should be performed either during initial instrument setup, after system maintenance that may have affected the calibration, or annually.

8.5.1 Position an appropriate calibration standard on the detector and close the shield.

8.5.2 Following section 8.9, count the standard until a minimum of 1000 counts have been acquired in the target peaks.

8.5.3 From the **Gamma Counting Main Menu** select **Calibrate a Detector**, to access the **Detector Calibration Menu**.

8.5.4 Update Calibration. Select Energy/Shape/Efficiency.

8.5.5 Answer the Prompt:

8.5.5.1 Choose detector from **Detector's Menu**.

8.5.5.2 Enter **Yes** to use the spectrum acquired in 8.5.2 above.

8.5.5.3 From the **Calibration Types** menu choose 3) **Efficiency Calibration** which accesses the **Certificate Files** menu.

8.5.5.4 From the **Certificate Files** menu choose the appropriate certificate which accesses the **Geometry Files** menu.

8.5.5.5 From the **Geometry Files** menu choose the appropriate geometry.

8.5.5.6 The calibration is then updated and the **Update Calibration Functions** menu is shown. From this menu, the choices are 1) **Results,** 2) **Plot Results,** and 3) **Change Energy Tolerance.** Choose any or choose PF3) Exit, which updates the Calibration Coefficients. Then choose **Yes** to generate a calibration report which accesses the **Detector Calibration** menu.

8.5.5.7 From the **Detector Calibration** menu choose 3) **Approve Efficiency Results** which displays a detector and geometry to choose. Approve as required.

8.5.5.8 Submit the calibration report to the Lab Supervisor for review.

8.6 Daily QC Checks

This performance check should be performed daily prior to counting samples, following instrument maintenance which may affect the system calibration, or whenever a problem is suspected.

8.6.1 Position the appropriate calibration/check source on the detector in the proper position.

8.6.2 From the **Gamma Counting Main Menu** select **Start a QC Count**, to access the **Quality Control Menu**.

8.6.3 From the **Quality Control Menu**, select **Daily Calibration Check**, to access the **Detectors Menu**.

8.6.4 From the **Detectors Menu**, choose the appropriate detector to access **Geometry Files Menu**.

8.6.5 From the **Geometry Files Menu**, choose the appropriate geometry which is being counted which accesses the **Certificate Files Menu**.

8.6.6 From the **Certificate Files Menu**, choose the certificate which matches the geometry file.

8.6.7 At the prompt, "**Enter A RETURN to Continue...**", Enter **Return**.

8.6.8 The standard then begins counting for a preselected count time.

8.6.9 After completion of data acquisition, a daily QC report is automatically generated. The predetermined parameters (i.e., CS-137 activity, CS-137 peak centroid, Cs-137 efficiency, etc.) are checked to verify that the results are within acceptable limits. A check source should have gamma peaks that cover the range (i.e., low, mid, high) of detection, generally 59 KeV to 2000 KeV. If a parameter is outside acceptable limits, a flag (i.e., investigate, 2-sigma; action, 3-sigma) will be printed on the Daily QC Report. If any of the parameters indicate an Action flag, contact the Laboratory Supervisor, investigate the cause, and recount the standard. If any parameters indicate an Action flag a second time, contact the Laboratory Supervisor and proceed under his/her direction.

8.7 Daily Background Checks

This procedure should be performed daily for a predetermined count time.

8.7.1 Ensure that the detector chamber is empty and close the lid.

8.7.2 From the **Gamma Counting Main Menu** choose 5) **Start a QC Count**.

8.7.3 From the **Quality Control Menu** choose 3) **Daily Background Check**, which accesses the **Detectors Menu**.

8.7.4 From the **Detectors Menu** select the appropriate detector.

8.7.5 At the Prompt **Enter a Return to start acquisition**.... Enter **Return**.

8.7.6 After completion of data acquisition, a daily background report is automatically generated. The predetermined parameters (i.e., background counts, background count rate) are checked to verify that the results are within acceptable limits. If a parameter is outside acceptable limits, a flag (i.e., investigate, 2-sigma; action, 3-sigma) will be printed on the Daily Background Report. If any of the parameters indicate an Action flag, contact the Laboratory Supervisor, investigate the cause, and recount the empty chamber. If any parameters indicate an Action flag a second time, contact the Laboratory Supervisor and proceed under his/her direction.

8.8 Weekly Chamber Background Checks

This procedure should be performed weekly for a predetermined count time.

8.8.1 Ensure that the detector chamber is empty and close the lid.

8.8.2 From the **Gamma Counting Main Menu** choose 2) **Start a Background Count** which accesses the **Detectors Menu**.

8.8.3 From the **Detectors Menu** choose the appropriate detector.

8.8.4 From prompt, "**Enter Return to Continue...**" enter **Return**, which accesses **Analysis Sequence File Menu**.

8.8.5 From **Analysis Sequence Files Menu** choose 1) **Chamber Background**, which accesses the **Geometry Files Menu**.

8.8.6 From **Geometry Files Menu** choose **No Specific Geometry**.

8.8.7 From the prompt "**Place Sample on detector in geometry**", enter a **Return** to continue (or Ctrl Z to abort).... Enter a **Return**.

8.8.8 From **Sample Parameters Menu** enter the following:

Preset Live Time: (i.e., 16:00:00.00)
Sample Quantity: (i.e., 1)
Sample ID: (i.e., Background)
Sample Units: (i.e., Chamber)
Filter Sample? (Y/N): (i.e., NO)
Sample Date/Filter End: (i.e., Today's Date)

Then enter **PF1** (Accept) which returns user to the **Gamma Counting Main Menu**.

8.9 Sample Counting

8.9.1 Ensure the Daily QC Check has been performed and all parameters are in bounds, per Section 8.6.9.

8.9.2 Ensure the sample is in a standard calibrated geometry and is in a new plastic bag.

8.9.3 From the **Sample Counting Main Menu** select **1) Gamma Counting** to access the **Gamma Counting Main Menu**.

8.9.4 Select **1) Start a Sample Count**, to access the **Detectors Menu**.

8.9.5 Select appropriate detector to access the **Analysis Sequence Files Menu**.

8.9.6 Select appropriate analysis name (i.e., List One) to access **Geometry Files Menu**.

8.9.7 Select the geometry used (i.e. 500 mar soil) to access **Sample Parameters Menu**.

8.9.8 Choose to place sample on appropriate detector with correct geometry, then enter RETURN to continue, or abort at this time.

8.9.9 Then enter the appropriate data that you are prompted for:

- Present live time (i.e., 1:00:00.00)
- Sample-Quantity
- Sample Type (matrix)
- Sample ID
- Sample Units (i.e., gram, liter)
- Client ID
- Filter Sample? (Y/N): Enter: **NO**.
- Filter Start Date: No entry, unless it is a filter.
- Sample Date/Filter End: Use the sample collection date.
- Choose accept and return to **Gamma Counting Main Menu**

8.10 Terminating a Count

8.10.1 From the **Gamma Counting Main Menu** select **Terminate a Count**.

8.10.2 Enter the Detector ID for the count to be terminated.

8.10.3 Press **Return** to exit to the main menu.

8.11 Filling Detectors with Liquid Nitrogen

CAUTION: Compressed gas and temperature hazard (see Sec. 4.7).

The high purity germanium detectors used for measuring gamma-ray emissions are designed to operate at very low (cryogenic) temperatures. These temperatures are attained by immersing the cold finger or dip stick of the detector in a dewar flask containing a cryogenic fluid (liquid nitrogen). The following method describes the proper procedure for filling the dewar flasks with liquid nitrogen. This procedure

will be performed on a weekly basis, with intervals between fillings not to exceed eight (8) days.

8.11.1 Insure all detector fill vent and the vent line drain valve are open.

8.11.2 Verify the liquid nitrogen supply line is connected to the fill tube the detector dewar. If it is not, connect it.

8.11.3 Open the supply valve on the supply dewar.

8.11.4 When liquid begins to flow from the overflow line shut the fill valve and record the date and time the detector was filled in the appropriate log book.

9.0 CALCULATIONS

The data reduction algorithms can be found in the Canberra Spectroscopy Algorithms and Software Verification and Validation Manuals, document numbers 07-0368, 07-0464, 07-0479, 07-D196.09, maintained on file in the laboratory.

10.0 QUALITY CONTROL

10.1 Efficiency calibration will be performed with a traceable calibration standard which as closely approximates the sample counting geometry as possible.

10.2 All data generated by this procedure will be reviewed by the Laboratory Supervisor.

10.3 The calibration portions of this procedure will be performed at initial installation, following system maintenance which may affect the electronics, when a problem is suspected, and at the following frequencies.

10.3.1 Efficiency Calibration - verified or reperformed annually or as needed.

10.3.2 Energy and FWHM Calibration - annually or as needed.

10.4 The operator performing this procedure will ensure that all daily instrument checks have been performed and meet performance criteria before counting any samples.

10.5 Detector backgrounds will be monitored at least once each calendar week.

11.0 REFERENCES

11.1 Canberra Genie Sample Counting Software.

11.2 Material Safety Data Sheets

11.3 SAIC Health Physics Manual

FUSRAP
LABORATORY PROCEDURES MANUAL

ML-004

Rev. 0

OPERATION OF THE ALPHA SPECTROSCOPY SYSTEM

Approved By:

Project Manager:	James R. Moos	1/24/99
SAIC Analytical Lab Coordinator:	Nile Leudtke	1/22/99
Laboratory Supervisor:	Brian J. Sparks	1/26/99
QA/QC Officer:	Glen Cowart	1/25/99

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1.0 PURPOSE AND SCOPE

This procedure establishes the methods for general use and calibration of the Canberra VAX Station Multichannel Analyzer System used to obtain and analyze alpha spectra for samples containing single or multiple alpha-emitting radionuclides. The method also describes how specific radionuclide analyses are determined from the alpha spectral data.

2.0 SUMMARY OF METHOD

Alpha particles with discrete energies are emitted during the decay of some radionuclides. The number of alpha particles emitted per unit time is directly related to the disintegration rates of the radionuclides, and the energies of the alpha particles are characteristic of the radionuclides in a sample. By obtaining an alpha spectrum one can identify the radionuclides in a mixture and establish the relative amounts of each.

Alpha spectrometry is normally performed with silicon surface barrier or ion implant detectors. This type detector produces a current pulse whenever an alpha particle dissipates its energy in the sensitive volume of the detector. The amplitude of the current pulse is directly proportional to the energy of the alpha particle, and the number of alpha particles entering the sensitive volume of the detector per unit time is directly related to the disintegration rate of the sample. Current pulses are amplified to give a voltage output that is proportional to the incident alpha particle energy. These voltage pulses are processed and stored by a multichannel analyzer for subsequent display and analysis.

Energy and efficiency calibrations will be performed after initial installation, weekly, following system maintenance, and/or whenever any problem is suspected. A National Institute of Standards and Technology (NIST) traceable standard approximating the sample geometry is counted in a reproducible shelf position, a spectrum collected, and subsequent analysis performed. The energy calibration is generated from the newly acquired data by plotting peak centroids versus actual energies of the standard. The efficiency calibration is attained by comparing the observed count rate of the calibration standard to the known decay corrected emission rate of the standard. This efficiency value is then used for the energy range of 3-7 MeV.

3.0 DEFINITIONS

3.1 Background – those counts that can be observed and thereby allowed for by measuring a source that is identical to the unknown source in all respects except for the absence of radioactivity. These counts can be attributed to environmental radioactivity, recoil contamination of the detector, sample containers, cosmic rays, electronic noise pulses, etc.

3.2 Traceable Calibration Standard – a calibrated radioactive source, prepared as or from a standard reference material traceable to National Institute of Standards & Technology (NIST), New Brunswick Laboratory (NBL), or other acceptable supplier.

3.3 Check Source – a radioactive source used to confirm the satisfactory operation of the instrument.

3.4 Efficiency – a percent of decay events from a standard radioactive source in a specific reproducible geometry that are seen and measured by a detector.

3.5 FWHM (Full Width Half Maximum) – the full width of an alpha peak distribution measured at half the maximum peak height, measured above the continum (background).

4.0 SAFETY

4.1 Individuals performing this procedure shall be aware of the precautions necessary for the proper handling of radioactive materials, as defined in Radioation Worker II. See the SAIC Health Physics Manual, Section 6.3.

4.2 The detector bias supply must remain off until the detector chamber reaches a minimum of 150 microns of vacuum to prevent damage to the surface barrier detectors.

4.3 Turning off, or loss of power to, the vacuum pumps could lead to oil contamination of the alpha detectors, therefore all detectors must be brought to atmospheric pressure prior to turning the vacuum system off or immediately after a loss of power.

4.4 Follow the manufacturer's instructions for set up, inter component connections, and preliminary testing of the equipment. Observe all the manufacturer's limitations and precautions.

4.5 Never exceed the manufacturer's recommended operating voltage for the detector. This may lead to detector damage.

5.0 INTERFERENCE

None.

6.0 EQUIPMENT

6.1 Canberra Alpha Spectrometer Model 7401

6.2 Surface Barrier Detector (or equivalent)

6.3 Multichannel Analyzer (VAX workstation or equivalent)

6.4 Vacuum Pump

6.5 Remote Parallel Interface (RPI)

6.6 AMX analog multiplexer module

6.7 Acquisition Interface Module

6.8 ADC (analog to digital converter)

7.0 REAGENTS AND STANDARDS

7.1 Traceable Calibration Standard - the actual standard is dependent upon the sample geometry being calibrated.

8.0 PROCEDURE

8.1 Standardization

8.1.1 Energy and Efficiency Calibration (Weekly Checks)

Alpha calibration standards are counted once each calendar week while in use to update the detector energy and efficiency calibrations.

8.1.1.1 Load a standard into each counter. Close the chamber doors and start evacuation of the chambers.

8.1.1.2 After the vacuum has fallen below 150 microns turn on the detector bias voltage supplies.

8.1.1.3 From the **VMS $** prompt type **Count** to access the **Sample Counting Main Menu**.

8.1.1.4 From the **Main Menu** select **Counting** which accesses the **Alpha Counting Menu**.

8.1.1.5 From the **Alpha Counting Menu** select **Primary Calibration Check**.

8.1.1.6 At the prompt "**Doing Prime Calibration Checks in Detectors, ALPHA 1. Check BIAS is on...and Return to continue...**" select **Return** after making sure detector biases are on.

8.1.1.7 The sequence file is set up, counting begins, and the **ALPHA Counting Menu** is automatically accessed.

8.1.2 Processing the Weekly Calibrations

8.1.2.1 From the **Main Menu** select **Create Names from Headers**, then **Process Configurations in NAMES.DAT**.

8.1.2.2 The Process begins and the results are shown on the screen.

8.1.2.3 Review the Results at the question "**Should the results be saved (YES/NO)?**" Select the appropriate answer (i.e., YES).

8.1.2.4 These results are displayed for each detector and the appropriate answers should be chosen until all detectors calibrated are reviewed.

8.1.2.5 The spectra for the appropriate results are then printed and the **ALPHA Counting Menu** is displayed. A supervisor's action report will be printed. Review the printout for any out of control conditions. If any of the data is out of control, contact the Laboratory Supervisor immediately. The control limits are based upon 3 sigma.

8.1.3 Daily Pulser Checks

This pulser check is performed daily prior to counting samples to verify the proper operation of the detectors. Peak centroid, Peak Energy Pulser count rate, peak FWHM, and control charts are monitored and stored in quality assurance files within the VAX workstation.

8.1.3.1 Ensure that all detectors are empty. Close the chamber doors and start evacuation of the chambers.

8.1.3.2 After the vacuum has fallen below 150 microns turn on the detector bias voltage supplies.

8.1.3.3 From the **VMS $** prompt type **Count** to access the **Main Menu**.

8.1.3.4 Select **1) Counting** to access the **Alpha Counting Menu**.

8.1.3.5 Select **2) Pulser Check**.

8.1.3.6 At the **Do you want to use ALL detector banks?** Select the appropriate detector bank(s).

8.1.3.7 From the **Detector Usage Selection Menu** type the appropriate numbers.

(i.e.) Start Detector Number: (1)
Number of Detectors to Use: (16)

Then Choose Accept.

8.1.3.8 At the prompt **Check if Bias Voltage is on**, enter **Return**.

8.1.3.9 Configuration parameters will then be set up, pulsers started and the display will automatically return to the ALPHA COUNTING MENU.

8.1.4 Processing the Daily Pulser Checks

8.1.4.1 From the **Main Menu** select **Create Names from Headers**, then **Process Configurations In Names.Dat**.

8.1.4.2 Then the display will automatically return to the Alpha Counting Menu. The program will proceed to automatically process the pulser data one detector at a time. After the data is processed (GRAPHS FOR FWHM, PEAK ENERGY, CTS/SEC., AND PEAK CENTROID), a Supervisor's Action Report will be printed. Review the printout for any out of control conditions. If any of the data is out of control, contact the Laboratory Supervisor immediately. The criteria for control limits is based upon three sigma for the particular parameters listed above. In all cases file the supervisors action report in the appropriate file folder.

8.1.5 Weekly Background Checks

8.1.5.1 Ensure that all detectors are empty. Close the chamber doors and start evacuation of the chambers.

8.1.5.2 After the vacuum has fallen below 150 microns turn on the detector bias supplies.

8.1.5.3 From the **Main Menu** select 1) **Counting** which accesses the **Alpha Counting Menu**.

8.1.5.4 From the **Alpha Counting Menu** select 5) **Backgrounds** which accesses the prompt, "**Do you want to use all detector banks?**" Select **YES**.

8.1.5.5 The **Detector Usage Selection Menu** is accessed and the system displays the prompt -

Start Detector Number: (i.e., 1)
Number of Detectors to Use: (i.e., 16)
Select the appropriate numbers and then PF1 (Accept).

8.1.5.6 This accesses the prompt -

Doing Backgrounds in Detectors Alpha 1
Check **Bias** is on....and **Return** to continue...
Then select **Return** and counting begins.

8.1.5.7 Configuration parameters will then be set up, backgrounds started and the display will automatically return to the **Alpha Counting Menu**.

The system will then start data acquisition on all alpha detectors and count the backgrounds for 700 minutes.

8.1.6 Processing Weekly Backgrounds

8.1.6.1 From the **Main Menu** select **Create Names from Headers**, then **Process Configurations in NAMES.DAT**.

8.1.6.2 The program will proceed to automatically process the background data one detector at a time. After the data is processed a Supervisor's Action Report will be printed. Review the printout for any out of control conditions. If any of the data is out of control contact the Laboratory Supervisor immediately. Typically, a detector with greater than 100 total counts for a 12 hr. background count will be out of control and require review by the Laboratory Supervisor. Results of the weekly background are stored in Quality Assurance files and control charts on the VAX workstation.

8.2 Operation

8.2.1 Sample Counting

8.2.1.1 Ensure the bias supply to the detector is off, then vent the alpha spectrometer sample chamber to atmosphere by placing the "pump/vent" switch on the spectrometer to the "vent position".

8.2.1.2 Open the door of the sample chamber. Carefully remove any sample that is in the chamber and place it in a petri dish.

8.2.1.3 Carefully position the next sample that is to be counted in the sample holder then center the sample beneath the detector face and ensure the sample shelf is in the proper location.

8.2.1.4 Close the door on each sample chamber and position the vacuum manifold selector switch that corresponds to that sample chamber, to the pump position and begin to evacuate the chamber. Continue to pump the chamber until a pressure of 150 microns or less is reached as indicated by the vacuum gauge.

8.2.1.5 Turn on the detector bias voltage supply as required.

8.2.1.6 From the **VMS $** prompt type **Count** to access the **Main Menu**.

8.2.1.7 Select **1) Counting** to access the **Alpha Counting Menu**.

8.2.1.8 Select **1) Samples**.

8.2.1.9 At the prompt, **Do You Want To Use All Detector Banks?**, select **Yes** or **No**.

8.2.1.10 At the prompt, **Which Detector Bank Are You Using? (1-3):** Choose detector bank being used.

8.2.1.11 The Sample Detector Usage Selection Menu will be displayed. Then enter the applicable information:

Batch #
Sample Date and Time
Units (i.e., grams, liters)
Start Detector Number
Number of Detectors to Use
Preset Count Time for Samples (i.e., 03:00:00)
Activity Units (e.g., pCi)
Tracer Certificate ID
Tracer Lambda (250)
MDA Confidence Level: 4.65
MDA LLD Constant: 2.71

Then press F1, which will set up chamber configuration parameters.

8.2.1.12 After accessing the Chamber Parameters enter the applicable information for each sample:

Chamber Contents Code: (e.g., S,R,C)

Sample ID
Analysis Library Code: (i.e., Th, UU, Ra, etc.)
Custody/Batch ID
Sample Title: (Client ID)
Sample Date/Time: (Date Sample Taken)
Sample Quantity: (Aliquot Size)
Units: (i.e., gram, liter, etc.)
Tracer Lambda: (Tracer weight in μL)
Tracer ID: (Tracer Certificate ID)
Activity Units: (i.e., pCi)
Tracerless Recovery: (i.e., .75=75%): Barium yields for Ra-226; otherwise 1.
MDA Confidence Level: 4.65
MDA LLD Constant: 2.71

8.2.1.13 Press PF1 to exit the Chamber Parameter Editor. Continue with the next sample if applicable. When all the sample data is entered into the system, choose PF1 and the system will ask, **Do you want to edit them again? [No]**. Choose **Yes** to edit the entries again or **No** and the system will ask if the bias is on, hit **Return** and then the system will start counting and check configuration parameters submit acquisition procedure and exit to Alpha Counting Menu.

8.2.2 Processing Sample Data

8.2.2.1 Visually review the data collected. Each spectrum should contain an individual sample spectra corresponding to the sample loaded in that particular chamber. If any of the spectrum are missing or appear to be wrong, contact the Laboratory Supervisor.

8.2.2.2 From the **Main Menu** select **Create Names from Headers**, then **Process Configurations in NAMES.DAT**.

8.2.2.3 After each spectrum is displayed answer the question "**Do the regions look ok?** (Y/N). If an N is entered then the regions may be adjusted to correct for a gain shift or expanded for a sample with elevated activity. Then after making this correction type a Y (or YES) when prompted again.

8.2.2.4 After this has been accomplished answer the question. Select the number (1 thru 5). Select the appropriate # (e.g., 1) Report.

8.2.2.5 After the sample processing is completed the system will return to the **Alpha Counting Menu**.

9.0 CALCULATIONS

The data reduction algorithms can be found in the Canberra Spectroscopy Algorithms and Software Verification and Validation Manuals, document numbers 07-0368, 07-0464, 07-0479, and 07-D196-09, maintained on file in the laboratory.

10.0 QUALITY CONTROL

10.1 Pulser checks shall be performed daily prior to counting samples.

10.2 Calibrations shall be performed once each calendar week to verify/update energy and efficiency calibration.

10.3 Detector backgrounds shall be performed once each calendar week.

11.0 REFERENCES

11.1 1990 Annual Book of ASTM Standards, Volume 12.02, E181.

11.2 Canberra NDSP Sample Counting Software.

11.3 Canberra Model 7104 Operating Manual.

11.4 SAIC Health Physics Manual

FUSRAP
LABORATORY PROCEDURES MANUAL

ML-005

Rev. 0

ISOTOPIC DETERMINATION OF THORIUM (FUSION)

Approved By:

Project Manager:	James R. Moos	1/24/99
SAIC Analytical Lab Coordinator:	Nile Leudtke	1/22/99
Laboratory Supervisor:	Brian J. Sparks	1/26/99
QA/QC Officer:	Glen Cowart	1/25/99

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1.0 PURPOSE AND SCOPE

1.1 The purpose of this procedure is to describe an analytical method for the determination of alpha emitting Thorium isotopes in soil, water, air filters, and biotic materials.

2.0 SUMMARY OF METHOD

2.1 Soil samples are dissolved completely in Potassium fluoride fusion. The pyrosulfate cake is then dissolved in hot dilute HCl solution. Barium chloride is added to precipitate Thorium, Plutonium, and Americium. The supernate is reduced with Titanous chloride and the Uranium is then precipitated with Barium chloride. The Thorium precipitation is dissolved in KEDTA and is reprecipitated as Titanous hydroxide. This hydroxide precipitate is dissolved in HCl and mounted with Neodymium for Alpha spectroscopy on 0.1 μm filters.

- Samples of water are acidified and evaporated to dryness. The residue is then treated as soil.

- Samples of air filters are dissolved in acid and evaporated to dryness. The residue is then treated as soil.

- Samples of biotic material are dried and homogenized. The residue is then treated as soil.

3.0 DEFINITIONS

3.1 NIST – National Institute of Standards and Technology

3.2 NBL – New Brunswick Laboratory

4.0 SAFETY

4.1 Lab coat, gloves, and safety glasses will be worn at all times when in the laboratory.

4.2 When using or preparing reagents that consist of 1,000ml or more of strong caustic or acidic materials, the analyst will also be required to wear an apron, gloves, and face shield.

4.3 Acids will be used in the appropriate designated hoods.

4.4 MSDS – Material Safety Data Sheets – will be available for other specific safety instructions on chemicals and reagents.

4.5 Individuals performing this procedure should be aware of the precautions necessary for the proper handling of radioactive materials, as defined in Radiation Worker II. See the SAIC Health Physics Manual, Section 6.3.

5.0 INTERFERENCES

5.1 The chemical composition of samples may cause interferences.

6.0 EQUIPMENT/MATERIALS

6.1 Balance (0-1,000.00 grams capacity)

6.2 Drying oven

6.3 Mortar and pestle

6.4 Muffle furnace

6.5 Pulverizer

6.6 Grinder

6.7 Assorted laboratory glassware

6.8 Assorted stainless steel or aluminum pans

6.9 50 ml Platinum crucibles

6.10 Air/gas blast burner

6.11 Ring stand

6.12 Wire triangle

6.13 Compressed air supply or air pump

6.14 0.1 μm Polypropylene 25 mm filters

6.15 Petri dishes

6.16 Routine laboratory tools and equipment

6.17 Appropriate pipettes

6.18 Centrifuge

6.19 Hot plate

6.20 Transfer pipettes

6.21 50 ml disposable centrifuge tubes

6.22 0.45 µm PVDF 25mm twist-lock syringe filters

6.23 Vortex

6.24 Vacuum pump

6.25 Filtering apparatus

6.26 47mm membrane filters

7.0 REAGENTS

7.1 Barium chloride, 0.45%: Dissolve 4.5 g $BaCl_2$ $2H_2O$ in 900 ml of reagent water. Dilute to 1 liter.

7.2 Barium seeding suspension:

7.2.1 Take .450g of $BaCl_2$ $2H_2O$ and place in a 250 ml Erlenmeyer flask. Add 12 ml concentrated H_2SO_4 and 20g of Na_2SO_4.

7.2.2 Heat with swirling over the blast burner until mixture is dissolved and H_2SO_4 begins to fume. Do not achieve pyrosulfate fusion.

7.2.3 Cool mixture to room temperature or below (ice water bath).

7.2.4 Add approximately 160 ml of deionized (DI) water with rapid swirling.

7.2.5 Warm until NaHS cake is dissolved. Should get white turbidity ($BaSO_4$ ppt). Cool in water bath as soon as dissolved. Dilute to 200 ml.

7.2.6 This solution is replaced every two (2) weeks.

7.3 Carbon suspension: Dissolve nine 47 mm membrane filters in 5 ml of concentrated H_2SO_4. Bring to a final volume of 50 ml with deionized water, adding the acid to the water.

7.4 Carbon substrate preconditioned filter: To 800 ml of deionized water, add 40 ml of concentrated HCl, 20 ml of concentrated HF, 20 mg of Nd carrier. Adjust volume to 1 Liter with deionized water and add 10 ml of Carbon suspension.

7.5 Isopropyl alcohol, 70%: Reagent grade, concentrated.

7.6 Ferrous Ammonium sulfate: 20% solution, prepared fresh daily.

7.7 Fluoride fusion flux (KF, KHF_2, and KNO_3): 300 grams of KF added to 210 grams of KHF_2 and 6 grams of KNO_3. Blend thoroughly using jar mill, mortar and pestle or similar method to insure that all chunks of material are broken up and the reagents are thoroughly mixed.

7.8 Hydrochloric acid (HCl); 12 M, reagent grade, concentrated.

7.9 Hydrochloric acid (HCl); 3 M, to 750 ml DI water, add 250 ml concentrated HCl.

7.10 Hydrofluoric acid (HF); 48%, reagent grade, concentrated.

7.11 Neodymium carrier, 1 mg/ml, commercial Neodymium carrier, spectroscopy grade.

7.12 Nitric acid, (HNO_3); 16 M, reagent grade, concentrated.

7.13 Potassium EDTA, 0.1 M (Using acid EDTA): Dissolve 29.2 g EDTA in DI water, dilute to 1 Liter. Note: May require low heat to dissolve solution. Using digital pH meter, adjust pH to 10.6 using 10 M KOH.

7.14 Potassium hydroxide, 10 M: Slowly dissolve 280.5 g KOH in 400 ml DI water in a cold water bath. Dilute to 500ml with DI water.

7.15 Potassium metabisulfite ($K_2S_2O_5$)

7.16 Sodium hydroxide (NaOH); 0.25 M; Dissolve 5 g of NaOH in 400 ml DI water. Dilute to 500ml.

7.17 Sodium sulfate, anhydrous (Na_2SO_4); reagent grade.

7.18 Sulfuric acid (H_2SO_4); 36 N, reagent grade, concentrated.

7.19 Titanous chloride ($TiCl_3$); 20%, reagent grade

8.0 PROCEDURE

8.1 Precisely weigh out an appropriate aliquot of sample in a 50 ml Platinum dish and record the weight. If sample matrix interferences are suspected, reduce the sample size.

8.2 Add an appropriate amount of Th-229 tracer to each sample. To the laboratory control add the appropriate concentrations of Th-230, Th-228, and Th-232.

8.3 Add approximately 2ml of concentrated Nitric acid. Carefully evaporate the solution on the hotplate. Heat on low heat to avoid splashing.

8.4 Add 4.5 grams of fluoride fusion flux to the dish and mix thoroughly. Depending upon the amount of metals in the sample as much as 5 grams may be needed.

8.5 Place the Platinum dish in a wire triangle over a blast burner and heat with hottest possible flame until a clear fusion is obtained and no more bubbles form when removed from heat.

8.6 Remove the dish from the flame and allow to cool to room temperature. Do not let this cake absorb moisture before performing the pyrosulfate fusion.

8.7 Add 5 ml of concentrated Sulfuric acid to the dish in two portions, heating carefully on a hot plate until reaction slows between additions.

8.8 Heat the solution slowly over the blast burner until the fusion cake is completely dissolved. The sample may be clear, or thick and milky. Continue heating the solution to drive off the water present in the Sulfuric acid. When Sulfuric fumes are generated, remove heat and add 2 g of anhydrous Sodium sulfate.

8.9 Continue to heat the sample until a clear yellow to red colored pyrosulfate fusion melt is obtained. Add 1 to 2 ml of concentrated Sulfuric acid if the solution is too thick.

8.10 Remove the dish from the heat. Swirl the mixture on the sides of the Platinum dish as the fusion melt cools. Deposit the melt on the sides of the dish to obtain a thin deposit.

8.11 Heat 35 ml of DI water and 5 ml of concentrated HCl in a 150 ml Erlenmeyer flask on a hot plate to boiling.

8.12 Once the Platinum dish has cooled to room temperature, gently flex the sides and rap the bottom of the dish while covering the top of the dish to prevent spilling any pieces.

8.13 Carefully transfer the contents of the dish into the hot dilute HCl solution and heat with high heat on the hot plate, swirling the flask to dissolve the contents as fast as possible. Wash the dish with the hot HCl solution. It is important that the cake dissolve very quickly to avoid formation of Calcium sulfate.

8.14 Add 1 ml of 20% Ferrous Ammonium sulfate.

8.15 Boil the solution for 15 minutes to hydrolyze condensed phosphates and ensure reduction of Plutonium if present.

8.16 Transfer the solution to a centrifuge tube and add enough hot DI water to bring the total volume to 35 ml.

8.17 Add 1 ml of seeding suspension.

8.18 Add 8 ml of 0.45%Barium chloride in four 2ml portions.

8.19 Centrifuge for five minutes while the solution is still hot to prevent Calcium sulfate from precipitating. Decant and discard the supernate from step 8.18.

8.20 Wash the precipitate from step 8.20 with approximately 10 ml of DI water, directed in a forceful stream from a wash bottle.

8.21 Vortex and Centrifuge for 5 minutes. Decant and discard the wash.

8.22 Dissolve the Barium sulfate precipitate in 20 ml of 0.1 M KEDTA. Vortex mixture. Heat in a water bath until the Barium sulfate has dissolved completely (approximately 5 minutes).

8.23 While swirling, add 4 drops of $TiCl_3$ followed by 2 ml of 10 M Potassium hydroxide (KOH).

8.24 Heat in a hot water bath for 10 minutes to flocculate the Titanous hydroxide precipitate.

8.25 Centrifuge for 5 minutes, decant and discard the supernate.

8.26 Wash the precipitate with 5 ml of 0.25 M Sodium hydroxide directed in a forceful stream from a wash bottle.

8.27 Vortex and Centrifuge for 5 minutes. Decant and discard the wash.

8.28 Add 3 ml of concentrated HCl and vortex. Place centrifuge tube in a hot water bath for 5-10 minutes.

8.29 Filter with a 0.45 µm PVDF 25mm twist-lock syringe filter (or equivalent). Rinse the syringe filter with 3 ml of DI water.

8.30 Add 0.1 ml of Neodymium carrier. Swirl mixture. Add 0.3 ml of 20% Titanous chloride.

8.31 Add 1 ml of Hydrofluoric acid and swirl. Place tube in a bath of cold water and let stand for 15 minutes to maximize the yield of Thorium.

8.32 Filtering Stage

8.32.1 Place a 0.1 µm Polypropylene 25mm filter in filter apparatus and seal the filter with 70% Isopropyl Alcohol. Vacuum suction the Isopropyl Alcohol through the filter.

8.32.2 Add 5 ml of the Carbon substrate to the filter and slowly vacuum suction the substrate through the filter.

8.32.3 Add the sample to the filter, rinse centrifuge tube with 5-10 ml of deionized water and add to filter. Slowly vacuum suction the solution through the filter.

8.32.4 Rinse the filter with deionized water, and vacuum suction the water through the filter.

8.32.5 Remove the filters from the filtering apparatus and place in a labeled petri dish. After allowing the filters a sufficient amount of time to dry submit them for Alpha spectroscopy.

9.0 QUALITY CONTROL

9.1 A minimum of one or a client specific number of sample duplicates shall be analyzed with every 20 samples. If there are less than 20 sample per analysis batch, then a minimum of one or a sufficient number of duplicates to meet client criteria shall be analyzed per batch.

9.2 One analysis blank shall be run with every 20 samples. If there are less than 20 samples per analysis batch, then one blank per batch shall be analyzed.

9.3 One spike standard (NBL or NIST when possible) shall be analyzed with every 20 samples. If there are less than 20 samples per analysis batch, then one spike per batch shall be analyzed.

9.4 If requested, a matrix spike consisting of a sample spiked with a standard (NBL or NIST when possible) shall be run with each batch.

9.5 If any of the above control samples do not fall within acceptable quality control criteria as defined in the Quality Assurance Manual, or as modified by specific contract requirements, the analytical portion may be repeated for the entire batch.

10.0 REFERENCES

10.1 Claude W. Sill, Kenneth Puphal, and Forest D. Hindman, Anal. Chem. 46, 1725 (1974)

10.2 Claude W. Sill and Roger L. Williams, Anal. Chem. 53, 412 (1981).

10.3 Claude W. Sill, Anal. Chem. 46, 1426 (1974).

10.4 Claude W. Sill, Anal. Chem. 36, 675 (1964).

10.5 Roger P. Bernabee, Donald R. Percival, and Forest D. Hindman, Anal. Chem. 52, 2351 (1980).

10.6 Annual Book of ASTM, Standards Vol. 11.02, pp. 300-303, pp. 380-381, pp.407 – 411.

10.7 Claude W. Sill, Nuclear and Chemical Waste Management, Vol. 7, pp. 201-215, (1987).

10.8 ORISE/ESSAP, Correspondence with Jack Beck, Mark Laudeman, and Sally Shipley.

10.9 Claude W. Sill, David Sill, Waste Management, Vol. 9, pp 219-229 (1989).

10.10 SAIC Health Physics Manual.

FUSRAP
LABORATORY PROCEDURES MANUAL

ML-009

Rev. 0

WASTE DISPOSAL

Approved By:

Project Manager: James R. Moos 1/24/99
James R. Moos

SAIC Analytical Lab Coordinator: Nile A Leudtke 1/22/99
Nile Leudtke

Laboratory Supervisor: Brian J Sparks 1/26/99
Brian J. Sparks

QA/QC Officer: Glen Cowart 1/25/99
Glen Cowart

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1.0 PURPOSE AND SCOPE

1.1 The purpose of this procedure is to provide instructions for the disposal of laboratory non-hazardous waste and corrosive wastes only; any other waste produced by the laboratory, hazardous or otherwise, is out of the scope of this instruction and will be handled on a case by case basis.

1.2 The procedure does not apply to any hazardous waste generated by the laboratory, with the exception of acids in that they are hazardous solely because they exhibit the characteristic of corrositivity.

2.0 SUMMARY OF METHOD

2.1 Waste material is segregated into various waste streams and disposed of according to type.

3.0 DEFINITIONS

3.1 DOT: Department of Transportation

3.2 DAW: Dry Active Wastes

4.0 SAFETY

4.1 Combining of incompatible laboratory waste products is strictly prohibited. Mixtures can be extremely dangerous and are costly to dispose of. If questions arise concerning compatibility of laboratory waste products, contact Laboratory Supervisor.

4.2 Lab coat, gloves and safety glasses will be worn at all times when in the sample prep section of the laboratory.

4.3 When working with wastes that consist of strong caustic or acidic materials, the analyst will be required to wear an apron, gloves, and face shield.

NOTE: Face shields, aprons, and gloves are also required for solutions producing excessive heat.

4.4 Acids will be used in the appropriate designated hoods or in a well ventilated area.

4.5 MSDS – Material Safety Data Sheets – are available for other specific safety instructions on chemicals and reagents.

4.6 Appropriate precautions will be adhered to when handling radioactive material. As defined in Radiation Worker II Training, see the SAIC Health Physics Manual, Section 6.3.

4.7 Hazardous materials shall be dealt with according to the Chemical Hygiene Plan.

4.8 When handling caustic materials, an eyewash/safety shower must be available in the immediate vicinity.

5.0 EQUIPMENT AND MATERIALS

5.1 Carboy - 6.5 gallon (25 Liter) polyethylene carboy with shoulder handles

5.2 Polyethylene funnel

5.3 Radioactive trash cans

5.4 6 Mil Polyethylene bags

5.5 Appropriate warning labels and signs

5.6 Appropriate radiation survey instruments

5.7 Logbook or worksheets

5.8 NaOH Pellets

6.0 PROCEDURE

6.1 General

6.1.1 It is expected that the laboratory will not generate any hazardous waste with the exception of acidic solutions. These solutions will be neutralized, and thus rendered non-hazardous, prior to disposal. If any other hazardous or potentially hazardous waste is generated by the laboratory, it will be handled on a case by case basis.

6.2 Sanitary Sewer Disposal

6.2.1 It is forbidden to dispose of heavy metals, solvents, pyrophoric materials, oxidizers, or laboratory radionuclides into the sanitary sewer system. General wash water may be discarded into the sanitary sewer. Water used for washing glassware may be discarded to the sewer if a preliminary rinse has been made in a solution which is retained for disposal through an

approved disposal method. Water used for washing hands (not to remove contamination) may be discarded to the sanitary sewer. All materials disposed of in the sanitary sewer system are permitted through the Metropolitan Saint Louis Sewer District.

6.3 Liquid Waste

Carboys in the laboratory are to be used for the disposal of potentially low-level radioactive solutions, acidic or caustic waste, potentially radioactively contaminated wash water, or small amounts of water soluble reagents and initial glassware rinse water. No organic solvent waste may be released to the carboys.

6.3.1 Reagent waste is neutralized prior to transfer to the carboy.

6.3.2 When a carboy is filled to approximately 5.5 gallons, start a Waste Batch Record Form using the date on which neutralization is verified.

6.3.3 Adjust the waste to a pH between 5 and 10, if necessary.

6.3.4 If the pH is within 5 to 10, the value is recorded on the Waste Batch Record and signed by the analyst performing the test, and the Lab Manager or Representative. If the pH is outside of the acceptable range, further neutralization is carried out. If the solution is too basic (pH>10), a strong acid can be slowly added to the carboy while mixing.

6.3.5 Sign the Waste Batch Record and transfer the waste to an appropriate waste receptical. Note the condition of the waste receptical on the Waste Batch Record.

6.3.6 Waste is held until an approved disposal method has been determined and approved by the Environmental, Health, and Safety manager.

6.4 Dry Non-Radioactive, Non-hazardous Waste Disposal

6.4.1 No dry chemicals, used labware, or lab wipes may be disposed of with site sanitary waste.

6.4.2 Dry waste such as cardboard, paper, packaging, and office waste may also be disposed of with site sanitary waste.

6.4.3 Materials used in the lab which have been surveyed or which, through administrative controls or process knowledge, are known to be free of radioactive contamination may be disposed of in this manner if they are empty (as defined in 49CFR171.8, 40CFR261.7) and have been triple

rinsed with water or a suitable solvent (as defined for the compound in the Handbook of Chemistry and Physics) and have dried thoroughly.

6.5 Dry Active Waste (DAW)

6.5.1 Any dry materials used in the lab which are contaminated or suspected of being contaminated are placed in a dry "Radioactive Waste" container. The materials in these containers are transferred to an appropriate staging area for the site DAW stream.

6.5.2 DAW shall be relinquished to Site Waste Management for final disposition.

6.6 Other Hazardous Wastes

6.6.1 In the event that any hazardous wastes, other than corrosive wastes, are generated (i.e., out of date laboratory reagents) all laboratory work will stop and will not proceed without specific direction from the Environmental Health, and Safety manager.

6.7 Other Hazardous Wastes

6.7.1 At no time will the laboratory generate more than 90 Kilograms of corrosive waste (or any other hazardous waste) in a calendar month. Under no conditions will the laboratory have more than 5.5 gallons of corrosive waste on hand at any given time. If it appears that the laboratory will exceed either of these limits all laboratory activities will immediately cease and will not proceed without specific direction from the Environmental, Health, and Safety manager.

Note: Conforming with these requirements will prevent the laboratory from requiring a RCRA permit. Failure to comply with this section can result in severe civil and criminal penalties for the operators of the laboratory.

7.0 References

7.1 Handbook of chemistry and physics.

7.2 FUSRAP Laboratory Quality Assurance Manual.

7.3 FUSRAP Laboratory Chemical Hygiene Plan.

7.4 SAIC Health Physics Manual.

8.0 Attachments

8.1 FUSRAP Laboratory Waste Batch Record

FUSRAP Laboratory Waste Batch Record

ML-009

Batch Date:_____ (Neutralization Started)

Preliminary Sample Collected_____ _____
Signature Date

Preliminary pH Check_____ Meter Number_____

_____ _____
Analyst Date

_____ _____
Laboratory Manager or Representative Date

Approximate Volume Transferred: _____

Waste Receptical Condition: _____ (i.e., leaking)

FUSRAP
LABORATORY PROCEDURES MANUAL

ML-015

Rev. 0

ISOTOPIC DETERMINATION OF URANIUM (FUSION)

Approved By:

Project Manager:	James R. Moos	1/24/99
SAIC Analytical Lab Coordinator:	Nile Leudtke	1/22/99
Laboratory Supervisor:	Brian J. Sparks	1/26/99
QA/QC Officer:	Glen Cowart	1/25/99

TABLE OF CONTENTS

1.0 PURPOSE AND SCOPE

1.1 The purpose of this procedure is to describe an analytical method for the determination of alpha emitting Uranium isotopes in soil, water, air filters, and biotic materials.

2.0 SUMMARY OF METHOD

2.1 Soil samples are dissolved completely in potassium fluoride fusion flux. The pyrosulfate cake is then dissolved in hot dilute Hydrochloric (HCL) acid solution. Barium chloride is added to precipitate Thorium, Plutonium, and Americium. The supernate is reduced with titanous chloride and the Uranium is then precipitated with the Barium chloride. The Uranium precipitation is dissolved in KEDTA and is reprecipitated as Titanous Hydroxide. This hydroxide precipitate is dissolved in HCL and mounted with Neodymium for alpha spectroscopy on 0.1 μm filters.

- Samples of water are acidified and evaporated to dryness. The residue is then treated as soil.

- Samples of air filters are dissolved in acid and evaporated to dryness. The residue is then treated as soil.

- Samples of biotic material are dried and homogenized. The residue is then treated as soil.

3.0 DEFINITIONS

3.1 NIST – National Institute of Standards and Technology

3.2 NBL – New Brunswick Laboratory

4.0 SAFETY

4.1 Lab coat, gloves, and safety glasses will be worn at all times when in the laboratory.

4.2 When using or preparing reagents that consist of 1,000 milliliters (ml) or more of strong caustic or acidic materials, the analyst will also be required to wear an apron and face shield.

4.3 Acids will be used in the appropriate designated hoods.

4.4 MSDS – Material Safety Data Sheets – will be available for other specific safety instructions on chemicals and reagents.

4.5 Individuals performing this procedure should be aware of the precautions necessary for the proper handling of radioactive materials, as defined in RadiationWorker II. See the SAIC Health Physics Manual, Section 6.3.

5.0 INTERFERENCES

5.1 Chemical composition of samples may cause interferences.

6.0 EQUIPMENT/MATERIALS

6.1 Balance (0-1,000.00 grams capacity)

6.2 Drying oven

6.3 Mortar and pestle

6.4 Muffle furnace

6.5 Pulverizer

6.6 Grinder

6.7 Assorted laboratory glassware

6.8 Assorted stainless steel or aluminum pans

6.9 50 ml Platinum crucibles

6.10 Air/gas blast burner

6.11 Ring stand

6.12 Wire triangle

6.13 Compressed air supply or air pump

6.14 0.1 μm Polypropylene 25 mm filters

6.15 Petri dishes

6.16 Routine laboratory tools and equipment

6.17 Appropriate pipettes

6.18 Centrifuge

6.19 Hot plate

6.20 Transfer pipettes

6.21 50 ml disposable centrifuge tubes

6.22 0.45 µm PVDF 25mm twist-lock syringe filters

6.23 Vortex

6.24 Vacuum pump

6.25 0.45 µm Supor 47mm filter

6.26 Filtering apparatus

6.27 47mm membrane filters

7.0 REAGENTS

7.1 Barium chloride, 0.45%: Dissolve 4.5 g $BaCl_2$ $2H_20$ in 900 ml of reagent water. Dilute to 1 liters.

7.2 Carbon suspension: Dissolve nine 47 mm membrane filters in 5 ml of concentrated H_2SO_4. Bring to a final volume of 50 ml with deionized (DI) water, adding the acid to the water.

7.3 Carbon substrate preconditioned filter: To 800 ml of deionized water, add 40 ml of concentrated HCl, 20 ml of concentrated Hydrofluoric (HF) acid, 0.02 g of Neodymium (Nd) carrier. Adjust volume to 1 Liter with DI water and add 10 ml of Carbon suspension.

7.4 Chromous chloride, 3 M:

7.4.1 In a 30 ml beaker dissolve 4 g $HgCl_2$ in 15 ml 6 M HCl.

7.4.2 In a 250 ml beaker mix 20 g of 20 mesh Zinc shot and 50 ml DI H_2O. Put the beaker in a cold water bath. (A large 1000 ml beaker works well for bath.)

7.4.3 Add 5 ml 12 M HCl to the zinc shot mixture, and stir with glass rod.

7.4.4 Place the Zinc shot mixture in a cool water bath. Slowly add the $HgCl_2$ solution (1-2 minutes) while stirring. Stir occasionally for 5 minutes.

7.4.5 Decant and discard all the liquid from the Zinc shot mixture. Transfer the amalgamated Zinc to a 250 ml squirt bottle.

7.4.6 In a 400 ml beaker, dissolve 80 g $CrCl_3$ $6H_2O$ mixture.

7.4.7 Pour $CrCl_3$ $6H_2O$ mixture into 250 ml squirt bottle containing the Zinc shot.

7.4.8 Tighten cap and shake. Loosen cap every minute to release pressure.

7.4.9 Continue shaking until mixture changes to a blue color – about 15 minutes.

7.4.10 Pour ¼ inch mineral oil into the bottle to serve as a protective barrier. Label the bottle with a note not to tighten the lid completely.

7.5 Isopropyl alcohol, 70%: reagent grade, concentrated.

7.6 Ferrous Ammonium sulfate: 25% solution, prepared fresh daily.

7.7 Fluoride fusion flux (KF, KHF_2, and KNO_3): 300 grams of KF added to 210 grams of KHF_2 and 6 grams of KNO_3. Blend thoroughly using jar mill, mortar and pestle or similar method to insure that all chunks of material are broken up and the reagents are thoroughly mixed.

7.8 Hydrochloric acid (HCl); 12 M, reagent grade, concentrated.

7.9 Hydrochloric acid (HCl); 3 M, to 750 ml DI water, add 250 ml concentrated HCl.

7.10 Hydrofluoric acid (HF); 48%, reagent grade, concentrated.

7.11 Neodymium carrier, 1 mg/ml, commercial Neodymium carrier, spectroscopy grade.

7.12 Nitric acid, (HNO_3); 16 M, reagent grade, concentrated.

7.13 Potassium EDTA, 0.1 M (Using acid EDTA): Dissolve 29.2 g EDTA in DI water, dilute to 1 Liter. Note: May require low heat to dissolve solution. Using digital pH meter, adjust pH to 10.6 using 10 M KOH.

7.14 Potassium hydroxide, 10 M: Slowly dissolve 280.5 g KOH in 200 ml DI water in a cold water bath. Dilute to 500ml with DI water.

7.15 Potassium metabisulfite ($K_2S_2O_5$)

7.16 Safranin – O (red); 1.0%

7.17 Safranin – O (red); 0.1%

7.18 Sodium hydroxide (NaOH); 0.25 M; Dissolve 5 g of NaOH in 400 ml DI water. Dilute to 500ml.

7.19 Sodium sulfate, anhydrous (Na_2SO_4); reagent grade.

7.20 Sulfuric acid (H_2SO_4); 36 N, reagent grade, concentrated.

7.21 Titanous chloride ($TiCl_3$); 20%, reagent grade

7.22 Barium seeding suspension

7.22.1 Take .450g of $BaCl_2$, $2H_2O$ and place in a 250 ml Erlenmeyer flask. Add 12 ml concentrated H_2SO_4 and 20g of Na_2SO_4.

7.22.2 Heat with swirling over the blast burner until mixture is dissolved and H_2SO_4 begins to fume. Do not achieve pyrosulfate fusion.

7.22.3 Cool mixture to room temperature or below (ice water bath).

7.22.4 Add approximately 160 ml of DI water with rapid swirling.

7.22.5 Warm until NaHS cake is dissolved. Should get white turbidity ($BaSO_4$ ppt). Cool in water bath as soon as dissolved. Dilute to 200 ml.

7.22.6 This solution is replaced every two (2) weeks.

8.0 PROCEDURE

8.1 Precisely weigh out an appropriate aliquot of sample in a 50 ml Platinum dish and record the weight. If sample matrix interferences are suspected, reduce the sample size.

8.2 Add an appropriate amount of Uranium-232 tracer to each sample. To the laboratory control add the appropriate concentration of natural Uranium.

8.3 Add approximately 2ml of concentrated Nitric acid. Carefully evaporate the solution on the hotplate. Heat on low heat to avoid splashing.

8.4 Add 4.5 grams of fluoride fusion flux to the dish and mix thoroughly. Depending upon the amount of metals in the sample as much as 5 grams may be needed.

8.5 Place the Platinum dish in a wire triangle over a blast burner and heat with hottest possible flame until a clear fusion is obtained and no more bubbles form when removed from heat.

8.6 Remove the dish from the flame and allow to cool to room temperature. Do not let this cake absorb moisture before performing the pyrosulfate fusion.

8.7 Add 5 ml of concentrated Sulfuric acid to the dish in two portions, heating carefully on a hot plate until reaction slows between additions.

8.8 Heat the solution slowly over the blast burner until the fusion cake is completely dissolved. The sample may be clear, or thick and milky. Continue heating the solution to drive off the water present in the Sulfuric acid. When Sulfuric fumes are generated, remove heat and add 2 g of anhydrous Sodium sulfate.

8.9 Continue to heat the sample until a clear yellow to red colored pyrosulfate fusion melt is obtained. Add 1 to 2 ml of concentrated Sulfuric acid if the solution is too thick.

8.10 Remove the dish from the heat. Swirl the mixture on the sides of the Platinum dish as the fusion melt cools. Deposit the melt on the sides of the dish to obtain a thin deposit.

8.11 Heat 35ml of DI water and 5 ml of concentrated HCl in a 150ml Erlenmeyer flask on a hot plate to boiling.

8.12 Once the Platinum dish has cooled to room temperature, gently flex the sides and rap the bottom of the dish while covering the top of the dish to prevent spilling any pieces.

8.13 Carefully transfer the contents of the dish into the hot dilute HCl solution and heat with high heat on the hot plate, dissolving the contents as fast as possible. Wash the dish with the hot HCl solution. It is important that the cake dissolve very quickly to avoid formation of Calcium sulfate.

8.14 Add 1 ml of 20% Ferrous Ammonium sulfate and boil the solution for 15 minutes.

8.15 Transfer the solution to a centrifuge tube and adjust the volume to 35 ml with deionized water.

8.16 Add 1 ml of Barium seeding suspension.

8.17 Add 8 ml of 0.45%BaCl in four 2 ml portions

8.18 Add 42 ml of deionized water and 12 ml of conc. HCL to a 250 ml beaker for each sample and place on the hot plate. Centrifuge and pour the liquid from 8.17 into the 250 ml beaker before it cools.

8.19 Add 1.25 g of $K_2S_2O_5$.

8.20 Boil for 20 minutes to volatilize the SO_2.

8.21 Add 1 drops of 1% aqueous safranin-O. This should produce a reddish color.

8.22 Add 20% $TiCl_3$ dropwise until the reddish color disappears.

8.23 Add 4 drops of 3M $CrCl_2$.

8.24 Add 2.5 g of anhydrous Na_2SO_4 and wait for the Na_2SO_4 to dissolve.

8.25 Add 6 ml of barium as 0.45% $BaCl_2 \bullet 2H_2O$ in two 3 ml portions.

8.26 Remove the samples from heat and place in an ice water bath for 2 hours or overnight at room temperature, allowing for the $BaSO_4$ precipitate to settle.

8.27 Decant as much liquid as possible without disturbing the precipitate. Discard the supernate into the acid waste. Transfer the precipitate to a centrifuge tube, with DI water.

8.28 Centrifuge for 10 minutes, decant and discard the wash.

8.29 Dissolve the precipitate in 20 ml of 0.1 M KEDTA. Vortex mixture. Heat in a water bath until the Barium sulfate has dissolved completely (approximately 5 minutes).

8.30 Add 4 drops of $TiCl_3$ followed by 2 ml of 10 M Potassium hydroxide (KOH).

8.31 Heat in a hot water bath for 10 minutes to flocculate the Titanous hydroxide precipitate.

8.32 Centrifuge for 10 minutes, decant and discard the supernate.

8.33 Add 10 ml of 3M HCl and vortex. Place centrifuge tube in a hot water bath for 5-10 minutes. Swirl mixture frequently during dissolution.

8.34 Filter with a 0.45 µm PVDF 25mm twist-lock syringe filter (or equivalent). Rinse the syringe filter with 3 ml of DI water.

8.35 Add 1 drop of 0.1% safranin-O and mix. Add 4 drops of $TiCl_3$. Add 0.05 ml of Neodymium carrier.

8.36 Add 1 ml of Hydrofluoric acid and swirl. A definite green color should be noticeable; if not, add another drop of $TiCl_3$. Let stand for 15 minutes.

8.37 Filtering Stage

8.37.1 Place a 0.1 µm Polypropylene 25mm filter in filter apparatus and seal the filter with 70% Isopropyl Alcohol. Vacuum suction the Ethanol through the filter.

8.37.2 Add 5 ml of the Carbon substrate to the filter and slowly vacuum suction the substrate through the filter.

8.37.3 Add the sample to the filter, rinse centrifuge tube with 5-10 ml of DI water and add to filter. Slowly vacuum suction the solution through the filter.

8.37.4 Rinse the filter with deionized water, and vacuum suction the water through the filter.

8.37.5 Remove the filters from the filtering apparatus and place in a labeled petri dish. After allowing the filters a sufficient amount of time to dry submit them for Alpha spectroscopy.

9.0 QUALITY CONTROL

9.1 A minimum of one or a client specific number of sample duplicates shall be analyzed with every 20 samples. If there are less than 20 sample per analysis batch, then a minimum of one or a sufficient number of duplicates to meet client criteria shall be analyzed per batch.

9.2 One analysis blank shall be run with every 20 samples. If there are less than 20 samples per analysis batch, then one blank per batch shall be analyzed.

9.3 One spike standard (NBL or NIST when possible) shall be analyzed with every 20 samples. If there are less than 20 samples per analysis batch, then one spike per batch shall be analyzed.

9.4 If requested, a matrix spike consisting of a sample spiked with a standard (NBL or NIST when possible) shall be run with each batch.

9.5 If any of the above control samples do not fall within acceptable quality control criteria as defined in the Quality Assurance Manual, or as modified by specific contract requirements, the analytical portion may be repeated for the entire batch.

10.0 REFERENCES

10.1 Claude W. Sill, Kenneth Puphal, and Forest D. Hindman, Anal. Chem. 46, 1725 (1974)

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10.6 Annual Book of ASTM, Standards Vol. 11.02, pp. 300-303, pp. 380-381, pp.407 – 411.

10.7 SAIC Health Physics Manual.