

FINAL

Sampling and Analysis Plan Volume 2 - Quality Assurance Project Plan

*Former Guterl Specialty Steel Corporation
Lockport, New York*

Prepared for

**US Army Corps of Engineers
Buffalo District**
Contract W912P4-05-D-0001
Delivery Order 0001



Prepared by

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**January 2007
Revised June 14, 2007**

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FUSRAP Site
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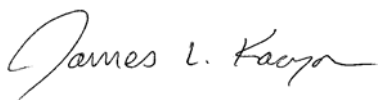
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LIST OF ATTACHMENTS

Attachment A – Standard Forms to Be Used

- Condition upon Receipt [STL-St. Louis]
- Chain of Custody [STL-St. Louis]
- Laboratory Notification Checklist [from USACE 200-1-3, Fig 3-4]
- A-E Daily Quality Control Summary Report (DQCSR)

Attachment B – STL-St. Louis Laboratory SOPs

- IP-0002 (Acid Digestion of Soils, SW-846 Method 3050B for ICP, ICP/MS)
- MT-0001 (Analysis of Metals by Inductively Coupled Plasma/Mass Spectroscopy)
- RC-0004 (Preparation of Soil, Sludge, Filter, Biota, and Oil and Grease Samples for Radiochemical Analysis)
- RC-0020 (Determination of Gross Alpha/Beta Activity)
- RC-0025 (Preparation of Samples for Gamma Spectroscopy)
- RC-0040 (Total Alpha Emitting Isotopes of Radium)
- RC-0041 (Radium 228 in Water)
- RC-0240 (Isotopic Americium, Curium, Plutonium, Thorium, and Uranium in Various Matrices by EICRoM Separation Resins)
- RD-0101 (Daily Operation, Calibration, and Maintenance of a Germanium Spectroscopy System)
- RD-0210 (Daily Operations of an Alpha Spectroscopy System)
- PM-0002 (Sample Receipt and Chain of Custody)

Attachment C – Other Documents and Forms

- STL St Louis Self-Certification Form
- Analytics - Certificate of Calibration (Standard Radionuclide Source)

LIST OF ACRONYMS

AEC	Atomic Energy Commission
ARS	American Radiation Services, Inc.
ARAR	Applicable or Relevant and Appropriate Requirement
ASTM	ASTM International (formerly American Society for Testing and Materials)
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CLP	Contract Laboratory Program
COC	Chain-of-Custody
COPC	Constituent of Potential Concern
DGAR	Data Gap Analysis Report
DoD	Department of Defense
DQI	Data Quality Indicator
DQO	Data Quality Objective
DQCSR	Daily Quality Control Summary Report
EDD	Electronic Data Deliverable
EML	Environmental Measurements Laboratory
FS	Feasibility Study
FSP	Field Sampling Plan
FUSRAP	Formerly Utilized Sites Remedial Action Program
GIS	Geographic Information System
HASL	Health and Safety Laboratory
HHRA	Human Health Risk Assessment
IA	Investigative Area
ICP-MS	Inductively Coupled Plasma – Mass Spectroscopy
IDW	Investigation Derived Waste
LCS	Laboratory Control Sample
LCSD	Laboratory Control Sample Duplicate
LIMS	Laboratory Information Management System
LQMP	Laboratory Quality Management Plan
MARLAP	Multi-Agency Radiological Laboratory Analytical Protocols
MARSSIM	Multi-Agency Radiation Survey and Site Investigation Manual
MCAWW	Methods for the Chemical Analysis of Water and Wastes
MD	Matrix Duplicate
MDA	Minimum Detectable Activity
MDC	Minimum Detectable Concentration
MDL	Method Detection Limit
MED	Manhattan Engineer District
µg/kg	Micrograms per Kilogram
µg/L	Micrograms per Liter
mg/kg	Milligrams per Kilogram
mg/L	Milligrams per Liter
MQO	Measurement Quality Objective
MS	Matrix Spike

MSD	Matrix Spike Duplicate
NELAC	National Environmental Laboratory Accreditation Conference
NIST	National Institute of Standards and Technology
NCP	National Oil and Hazardous Substances Pollution Contingency Plan
PARCC	Precision, Accuracy, Representativeness, Comparability, and Completeness
pCi/g	PicoCuries per Gram
pCi/L	PicoCuries per Liter
PRG	Preliminary Remediation Goal (USEPA Region 9)
QAPP	Quality Assurance Project Plan
QA	Quality Assurance
QC	Quality Control
QCSR	Quality Control Summary Report
QSM	Quality Systems Manual
RA	Remedial Action
RD	Remedial Design
RI	Remedial Investigation
RL	Reporting Limit
RPD	Relative Percent Difference
SAP	Sampling and Analysis Plan
SARSG	San Antonio Radiation Safety Group
SLERA	Screening Level Ecological Risk Assessment
SOP	Standard Operating Procedure
SOW	Scope of Work
STL	Severn Trent Laboratories
TCLP	Toxicity Characteristic Leaching Procedure
TSS	Total Suspended Solids
TPP	Technical Project Planning
USACE	United States Army Corps of Engineers
USDOE	United States Department of Energy
USEPA	United States Environmental Protection Agency
VTSR	Verified (or Validated) Time of Sample Receipt

1. PROJECT LABORATORY ORGANIZATION AND RESPONSIBILITIES

In accordance with United States Army Corps of Engineers (USACE), Buffalo District contract number W912P4-05-D-0001, delivery order number 0001, Earth Tech has prepared this *Quality Assurance Project Plan* (QAPP) for the former Guterl Specialty Steel Corporation site (Guterl Steel site), as part of the Formerly Utilized Sites Remedial Action Program (FUSRAP), in accordance with Task 5 of the March 2005 delivery order Scope of Work (SOW) (USACE, 2005a).

This QAPP is part of the Remedial Investigation (RI) *Sampling and Analysis Plan* (SAP). The overall SAP consists of this QAPP and the companion *Field Sampling Plan* (FSP). The SAP contains the overall RI approach, rationale, procedures, and quality assurance/quality control (QA/QC) program for the various field activities planned during the Site RI. The SAP has been developed using available background information, and relevant guidance documents such as the USACE Requirements for the Preparation of Sampling and Analysis Plans Engineer Manual (EM 200-1-3 (USACE, 2001)), the United States Environmental Protection Agency (USEPA), US Department of Energy (USDOE), and US Department of Defense (DoD), Multi-Agency Radiation Survey and Site Investigation Manual (MARSSIM), 2000, Multi-Agency Radiological Laboratory Analytical Protocols Manual (MARLAP, 2004), and the DoD Quality Systems Manual for Environmental Laboratories (QSM, Final Version 3; DoD, 2006).

The current and future uses of the data may include performing a RI, feasibility study (FS); risk assessments; remedial design (RD), and remedial action (RA). Within these broad programs, data may be used to establish the nature and extent of contamination; fate and transport; human health risk assessments; screening level ecological risk assessment; estimation of quantities and classification (e.g., hazardous or non-hazardous; low level radioactive waste; etc.) of contaminated material of various matrices (soil; groundwater; surface water; building materials); and achievement of cleanup goals (release criteria).

Laboratory analytical work conducted for this project will be of three principal types.

- Radiological (Radionuclide) analyses, which will be the major portion of the work and is the data set which will be used for assessing the nature and extent of the Manhattan Engineer District (MED)/Atomic Energy Commission (AEC)-related materials on site.
- Chemical and conventional parameter analyses will be performed on a limited number of samples to aid in assessing the impact of non-MED/AEC materials on contaminant fate, risks, and remediation.
- Geotechnical analyses will be performed on some samples to aid in assessing migration potential and remedial options.

It is anticipated that a single laboratory will be used for radiological and conventional parameters, and that a different laboratory will be utilized for geotechnical analyses.

At this point, not all the specific laboratories have been identified or selected. The generic laboratory organization requirements are those identified in the Department of Defense Quality

Systems Manual (DoD QSM; 2006) and reproduced below (in paraphrased form) in Section 1.1. Laboratory-specific organization and requirements will be specified in Section 1.2.

In addition to the off-site laboratory (qualifications described below in Sections 1.1 and 1.2), Earth Tech will also utilize an on-site counting laboratory for radiological analysis. The organization and personnel qualifications for the on-site laboratory are described in Section 1.3.

1.1 Laboratory Organization Requirements - General

Laboratory qualifications and organization will be consistent with the DoD QSM; these requirements are consistent with the requirements of the National Environmental Laboratory Accreditation Conference (NELAC). In accordance with these requirements, the laboratory shall:

- Have managerial staff with the authority and resources needed to discharge their duties.
- Have processes to free its personnel from any commercial, financial and other undue pressures which adversely affect the quality of their work.
- Be organized in such a way that confidence in its independence of judgment and integrity is maintained at all times.
- Specify and document the responsibility, authority, and interrelationship of all personnel who manage, perform or verify work affecting the quality of calibrations and tests.
- Such documentation shall include:
 - A clear description of the lines of responsibility in the laboratory and shall be proportioned such that adequate supervision is ensured, and
 - Job descriptions for all positions.
- Provide supervision by persons familiar with the calibration or test methods and procedures, the objective of the calibration or test, and the assessment of the results.
- The ratio of supervisory to non-supervisory personnel shall be such as to provide adequate supervision to maintain adherence to laboratory procedures and accepted techniques.
- Have a technical director(s) (however named) who has (have) overall responsibility for the technical operation of the environmental testing laboratory.
- The laboratory shall have a quality assurance officer (however named) who has responsibility for the quality system and its implementation.
- Nominate deputies in case of absence of the technical director(s) and/or quality assurance officer.
- Have documented policy and procedures to ensure the protection of clients' confidential information and proprietary rights (this may not apply to in-house laboratories).
- For purposes of qualifying for and maintaining accreditation, each laboratory shall participate in a proficiency test program as outlined in Chapter 2 of NELAC.

1.1.1 Technical Director(s)

The technical director(s) shall certify that personnel with appropriate educational and/or technical background perform all tests for which the laboratory is accredited. Such certification shall be documented. The technical director(s) shall meet the requirements specified in the Accreditation Process (see NELAC, Section 4.1.1.1). Technical directors are responsible for following through with proficiency testing programs and for verifying that corrective actions are implemented after testing and evaluating the effectiveness of the corrective actions.

1.1.2 Quality Assurance Officer

The laboratory quality assurance officer shall have direct access to the highest level of management at which decisions are taken on laboratory policy or resources, and to the technical director. Where staffing is limited, the quality assurance officer may also be the technical director or deputy technical director. The quality assurance officer (and/or his/her designees) shall:

- Serve as the focal point for QA/QC and be responsible for the oversight and/or review of QC data.
- Have functions independent from laboratory operations for which they have quality assurance oversight.
- Be able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence.
- Have documented training and/or experience in QA/QC procedures and be knowledgeable in the quality system, as defined under NELAC.
- Have a general knowledge of the analytical test methods for which data review is performed.
- Arrange for or conduct internal audits annually.
- Notify laboratory management of deficiencies in the quality system and monitor corrective action.
- The quality assurance officer shall be responsible for ensuring continuous improvement at the laboratory through the use of control charts and other method performance indicators (for example, proficiency testing samples and internal and external audits).

1.2 Laboratory-Specific Organization

Different laboratories may be used for radiological, chemical/conventional, and geotechnical analyses, as described below.

1.2.1 Radiological Laboratory

The proposed laboratory for radiological analyses is Severn Trent Laboratories (STL), St. Louis. STL-St. Louis is a 31,000-sf laboratory in Earth City, MO and has been part of the STL network for approximately five years. STL-St. Louis is NELAC-certified by the State of Florida for radiochemistry, solid waste, and wastewater. Florida has NELAC reciprocity with many state certifying agencies, including New York (NY ID 11616). STL is compliant with the DoD QSM (2006), and their self-certification form is included in Appendix C of this QAPP. (STL-St. Louis has previously provided the necessary backup documentation to USACE, and this information is not reproduced herein.)

Radiological analysis types will include isotope-specific uranium and thorium; radium-226 and radium-228; and gross alpha and beta radiation.

For the Guterl Steel project, the STL Project Manager, Mr. Terry Romanko, will serve as the principal point of contact for technical and administrative issues between STL and Earth Tech staff (principally the Earth Tech radiological analysis coordinator, Mr. Tim Snider, and the Earth Tech QA manager, Mr. Allen Burton). The following technical staff at STL-St. Louis will also have important roles in this project:

- Bill Deckelmann, Laboratory Director, has overall responsibility for all analyses and data reported by STL-St. Louis. Mr. Deckelmann has a B.S. in biology and has 24 years of laboratory experience.
- Terry Romanko will serve as the STL Project Manager. He will be the point of contact for technical administrative issues and will see that the appropriate technical staff responds to any questions or problems which may arise during the execution of this project. He has a degree in chemistry and 17 years of experience.
- Elaine Wild is the STL-St Louis QA Manager. She has a B.S. in chemistry and 17 years experience.
- Joel Kempema is the STL-St Louis Radiochemistry Technical Director. He has a B.A. in chemistry and 17 years experience.
- Rhonda Rupprecht is the Radiochemistry Count Room Team Leader. She has a B.S. in biology and five years experience.
- Jason Dillard is the radiochemistry Separations Team Leader. Mr. Dillard has three years experience.
- Kim Young is the Radiochemistry Actinide Preparation Team Leader and has four years experience.
- Jeff Gross is the Metals Team Leader. He has a B.A. in chemistry and eight years experience.
- Connie Dedner is the Data Reporting Supervisor. She has a B.A. in marketing and 11 years experience.
- Jill Clarke is the Sample Control Supervisor and has eight years experience.

1.2.2 Chemical and Conventional Parameters Analyses Laboratory

It is currently planned to use STL-St. Louis for chemical and conventional parameter analyses. The analyses currently planned include total (non-isotopic) uranium, total organic carbon, and investigation-derived waste (IDW) characterization analyses (soil samples) and total suspended solids (TSS) (groundwater samples). STL-St. Louis' organization and personnel are described above (Section 1.2.1).

1.2.3 Geotechnical Laboratory

The geotechnical laboratory has not yet been selected. Experienced laboratories will be solicited and will be subject to USACE approval. Analysis types will include Atterberg limits, grain size distribution, and hydraulic conductivity. Analyses will be performed using ASTM International (ASTM; formerly American Society for Testing and Materials) methods as specified in Section 5.1.3 of this QAPP.

1.3 On-Site Laboratory

Earth Tech will subcontract an on-site laboratory (provided by American Radiation Services, Inc. (ARS), Baton Rouge, LA) for radiological analyses of samples during the course of the RI. The equipment and layout of the laboratory will be established in greater detail in the laboratory quality management plan (LQMP) and associated standard operating procedures (SOPs) to be developed later (see QAPP Section 3.6).

The on-site laboratory will be subject to review and approval by USACE. As a component of this approval, a LQMP and SOPs for various laboratory activities and procedures will be developed by Earth Tech and submitted to USACE for review. The elements to be addressed by these SOPs are discussed in Section 3.6 of this QAPP.

The on-site laboratory will be staffed with experienced and qualified personnel, including key personnel listed below.

Laboratory Director: The Laboratory Director will be solely responsible for all laboratory operations. The Laboratory Director will review and verify all results by affixing his/her signature to all documentation, results and reports. The Laboratory Director will oversee and maintain the Radiological Quality Assurance program to maintain compliance with applicable regulations and data quality objectives of site operations. At a minimum, the Laboratory Director will be a senior Radiochemist/Health Physicist with a minimum of a Bachelor's degree in a related field, with over 10 years of radiological laboratory experience.

Data Manager: The Data Manager will oversee, maintain and review all data deliverables and associated Laboratory Information Management System (LIMS). The Data Manager will be responsible for geographic information system (GIS) mapping of field results, as well as correlating analytical results.

Additional qualified laboratory technicians will be provided to meet the sample counting requirements, which are identified in the FSP.

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2. DATA ASSESSMENT ORGANIZATION AND RESPONSIBILITIES

Data generated for the Guterl Steel site Remedial Investigation/Feasibility Study (RI/FS) will be of three general types: radiological; conventional/chemical; and geophysical. In addition, within each data type, the intended use of the data will vary (e.g., screening to identify general areas needing further sampling; or definitive isotope-specific analysis for risk assessment or accurate contaminant delineation). Therefore, the organizations and personnel performing the data quality review (data assessment) will vary according to the type of data being reviewed; and the level of the review will be driven by the intended use of the data.

2.1 Assessment of Radiological Data

Radiological data will be generated by both the on-site laboratory (gamma spectroscopy only), and by the off-site laboratory. The assessment of the radiological data is discussed below.

2.1.1 Assessment of On-Site Laboratory Radiological Data

The on-site laboratory will analyze soil samples by gamma spectroscopy for isotopic uranium and thorium. The eight radiological constituents of potential concern (COPCs) will be reported by the on-site laboratory, although some of the COPCs [e.g., Th-228, Th-230, and Ra-228] are inferred from the presence of other radionuclides under the assumption of secular equilibrium.

Quality control for the on-site laboratory will be established in the LQMP and SOPs, to be submitted (subject to USACE review and approval) as part of RI Task 6. An example table of contents, illustrating the subject areas to be covered by the LQMP along with identification of key SOPs, is provided as Figure 3-1. In addition (and as noted in the FSP), 5 percent of the samples analyzed (minimum of 100) on site will also be analyzed by the off-site laboratory (this fraction will include all the samples with concentrations measured at least 50 percent of the screening level). The on-site and off-site laboratory data will be qualitatively and quantitatively assessed (for precision and accuracy [bias]) and this assessment will be presented in the QCSR. This comparison will also be used as a feedback loop to develop an algorithm relating the on-site data to the off-site results; see further discussion in Section 3.6.2.

The on-site laboratory is subject to review and approval by USACE. The SOPs for the laboratory operations (to be developed during mobilization) will specify the necessary level of data review.

2.1.2 Assessment of Off-Site Laboratory Radiological Data

Radiological data to be generated by the off-site laboratory will include total uranium, isotopic uranium and thorium by alpha and gamma spectroscopy, radium 226 and 228, and gross alpha and beta radiation.

The first step in the assessment of radiological data is internal review by the laboratory generating the data (see Sections 1.1 and 6.1 of this QAPP). The data will then be subject to formal independent review and validation in accordance with the criteria specified in Section 8.2, including compliance with the measurement quality objectives for radiological data discussed in Section 3.3.

2.2 Assessment of Chemical and Conventional Parameters Data

Chemical and conventional parameter data to be generated include chemical-specific data (isotopic uranium and organic carbon, by SW-846 methods) and conventional parameters (e.g., TSS in water samples; typically using EPA Methods for the Chemical Analysis of Water and Wastes (MCAWW) Methods). The waste disposal subcontractor is responsible for analyses necessary for proper characterization and disposal of investigation-derived waste (IDW).

As noted for radiological data in Section 2.1, the first step in the assessment of the chemical and conventional parameters data is internal review by the laboratory generating the data (see Sections 1.2 and 6.1.1 of this QAPP). The data will then be subject to formal independent review and validation in accordance with the criteria specified in Section 8.2, including compliance with the measurement quality objectives for chemical and conventional parameters data discussed in Section 3.4. It is not anticipated that IDW characterization data generated by the waste disposal subcontractor will be subject to the same level of formal review as chemical data utilized for defining the nature and extent of contamination and risk assessment.

2.3 Assessment of Geotechnical Data

Geotechnical data to be generated include grain size distribution, Atterberg limits, and hydraulic conductivity data, as discussed in Section 5.1.3.

The first step in the assessment of geotechnical data is internal review by the laboratory generating the data. After receipt of the laboratory data, the data will be reviewed by an experienced Earth Tech geologist, but will not be formally validated. The level of review, and a summary of the review, will be presented in the final Quality Control Summary Report (QCSR).

2.4 Overall Data Assessment

At the completion of the project, and concurrently with the preparation of the RI report, a QCSR will be prepared, as described in Section 8.6.

3. DATA QUALITY OBJECTIVES

3.1 Data Use Background

The overall project objective is to collect the necessary data for decisions concerning the cleanup of radiological material at the Guterl Steel site, as well as to make remedial decisions based upon the nature and extent of radiological material contamination. The MED/AEC-related COPCs are limited to radiological material (U-234, U-235, U-238; Th-228, Th-230, Th-232; Ra-226 and Ra-228).

Additional data will be collected to evaluate the presence of enriched and recycled uranium. Presence of U-236 indicates recycled uranium; enhanced abundances of U-234 and U-235 indicate enriched uranium. A limited number of samples (anticipated to be 12) will be selected from the offsite laboratory alpha spectroscopic analyses that have significantly elevated uranium activities to also undergo inductively coupled plasma – mass spectroscopy (ICP-MS) analysis for uranium isotopes to determine the relative abundances of U-234, U-235, U-236, and U-238 to be used to evaluate the presence of recycled or enriched uranium. A similar number of background samples will also be analyzed by ICP-MS.

Gross alpha and gross beta radiation analyses will also be performed on the above samples for comparison to values from alpha spectroscopy and/or mass spectroscopy for COPC total alpha and total beta emissions.

The selection criteria in the Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) and the National Oil and Hazardous Substances Pollution Contingency Plan (NCP) will be used for site evaluation and remedy. Therefore, the data collected must be of sufficient quantity and quality to support this determination.

3.2 Project Data Quality Objectives

Project Data Quality Objectives (DQOs) are qualitative and quantitative statements that specify the quality of data required to support the RI/FS at the Guterl Steel site, while considering the intended use of the data. The project DQOs for field and laboratory activities were established based upon available site history and previous investigations (summarized in the *Data Gap Analysis Report*, USACE, 2006) and potential remedial criteria and Applicable, or Relevant and Appropriate Requirements (ARARs) for the Guterl Steel site. Development of the project DQOs was initiated at the Technical Project Planning (TPP) meeting conducted by the USACE in August 2005, which was attended by the stakeholders associated with the site. The intent of the project DQOs is to comply with applicable regulations related to the handling and assessment of radiological contaminants present at the site, and evaluate potential remedial alternatives to address the radiological waste and impacted site media. The full list of DQOs developed for the project (including DQOs which were completed prior to, or will be completed subsequent to, this RI is provided in the Data Gap Analysis Report (DGAR) (USACE, 2006)).

A further elaboration on the intended data use and the associated data need requirements for each project DQO to be achieved in the RI is presented in Table 3-1. The appropriate sampling and analysis methods are presented in the Summary of Data Quality Objectives included as Table 3-

2, and a summary of the QA Objectives to be achieved in this RI is presented in Table 3-3. An assessment of project (RI) completeness is presented in Section 8.5 of this QAPP.

3.3 Measurement Quality Objectives for Radiological Data

Measurement quality objectives (MQOs; also referred to as data quality indicators, or DQIs) establish specific criteria for the generation of data of known and acceptable quality, which allows for eventual data usability review. Systematic QC checks are incorporated into the sampling and analyses to show that procedures and test results remain reproducible and that the analytical method is actually measuring the quality of target analytes without unacceptable bias. Systematic QC checks include the analyses of field/laboratory duplicates, calibration check standards, tracers, matrix spike samples (for non-tracer/carrier analyses), laboratory control samples, and method blanks. MQOs (acceptance criteria or ranges) for these QC checks are established to verify DQIs support data usability, and contract compliance. The program of systematic QC checks may be reviewed from two aspects, batch QC and matrix-specific QC, as presented in Section 5.5.

In order to generate defensible data of the necessary quality, criteria will be established and measured for the following DQIs:

- precision
- accuracy
- representativeness
- comparability
- completeness
- sensitivity

These DQIs apply to all definitive data produced by off-site (laboratory) analysis. Calculation of DQIs is presented in Section 6.0.

3.3.1 Radiological Data Precision

Precision refers to the distribution of a set of reported values about the mean, or the closeness of agreement between individual test results obtained under prescribed conditions. Precision reflects the random error, may be affected by systematic error, and also characterizes the natural variation of the matrix and how the contamination exists or varies within that matrix (USACE 2001). Precision is evaluated using analyses of an analytical sample and its corresponding matrix duplicate (MD), laboratory matrix spike duplicate (MSD; non-tracer analyses only), and/or laboratory control sample duplicate (LCSD) which not only assess sampling precision, but indicate analytical precision through the reproducibility of the analytical results.

For the radiological analyses, field duplicates (blind to the laboratory) will be submitted to the off-site laboratory at a frequency of one per 20 environmental samples (excluding swipe samples); field duplicate data provide an indication of the overall precision of the sampling and analytical process. In addition, STL-St. Louis will analyze a matrix duplicate (MD) with each analytical batch of 20 or fewer samples (applicable to uranium, thorium, and radium analyses). Relative percent difference (RPD) is a qualitative performance indicator used to evaluate precision. RPD criteria must meet the method requirements summarized in Table 3-3.

3.3.2 Radiological Data Accuracy

Accuracy is the measure of the closeness of an observed value to the “true” value (e.g., theoretical or reference value, or population mean). Accuracy includes a combination of random error and systematic error (bias) components that result from sampling and analytical operations (USACE, 2001). Sources of error are the sampling process, field contamination, preservation, handling, sample matrix, sample preparation, and analysis techniques. The laboratory objective for accuracy is to equal or exceed the accuracy demonstrated for the analytical methods on samples of the same matrix. The percent recovery criterion is used to estimate accuracy based on recovery in the matrix spike (MS) and MSD and laboratory control sample/laboratory control sample duplicate (LCS/LCSD) samples. The MS and MSD, which will give an indication of matrix effects that may be affecting target compounds, are also a good gauge of method efficiency.

The alpha spectroscopy methods utilized for isotopic uranium and thorium utilize a ‘tracer’ radionuclide, which is used to calculate the efficiency (recovery) of the analysis. The tracers (also referred to as ‘yield monitors’ by STL-St. Louis) used are Th-229 for the isotopic thorium analysis and U-232 for isotopic uranium. As the tracer compounds are utilized to measure the recovery of radionuclides in each sample, MS/MSD analyses are not required. However, a LCS and LCSD will be analyzed for each batch. In addition, accuracy of isotopic identification is achieved through use of alpha spectroscopy methods on a subset of the off-site analyses, allowing definitive confirmation of the isotopic identification from the gamma spectroscopy methods.

Analysis for Ra-226 and Ra-228 will utilize STL-St. Louis SOPs, which are based on USEPA methods 903 (for Ra-226) and 904 (for Ra-228). Accurate measurement of these isotopes requires allowing sufficient time for ingrowth of short-lived daughters, as specified in STL-St. Louis’ SOPs (RC-0040 for Ra-226 and RC-0041 for Ra-228). Each batch (20 or fewer samples) includes analysis of an LCS for radium isotopes. A MS analysis is not performed.

Accuracy is also measured through the analyses and evaluation of method and field QC blanks, which aids in assessing the potential concentration contribution from various outside sources. COPC concentrations should not exceed one-half the project specific minimum detectable concentrations (MDC). Acceptable ranges of recovery are reported in the referenced methods and summarized in Table 3-3.

3.3.3 Radiological Data Representativeness

Representativeness expresses the degree to which the sample data are indicative of the characteristics of a population of samples, parameter variations at a sampling point, or environmental conditions. Representativeness is a qualitative parameter which is most concerned with the proper design of the sampling program or subsampling of a given sample (USACE, 2001). Objectives for representativeness are defined for sampling and analysis tasks and are a function of the investigative objectives. The sampling procedures, as described in the FSP, have been selected with the goal of obtaining representative samples for the media of concern. Representativeness of the samples and analytical processes can be assessed qualitatively by the use of field and laboratory duplicate samples. Analytical representativeness is also enhanced through the use of gamma spectroscopy methods (in both the on-site and off-site laboratory)

which utilize relatively large sample volumes, facilitating obtaining a representative aliquot for analysis.

3.3.4 Radiological Data Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another (USACE, 2001). Comparability within this RI is achieved using standard techniques to collect and analyze representative samples and report analytical results in appropriate units; and also by using gamma spectroscopy methods in both the on-site and off-site laboratory. Complete field documentation using standardized data collection forms will support the assessment of comparability. Comparability is limited by the other parameters, because only when precision and accuracy are known, can data sets be compared with confidence. In order to generate comparable (internally consistent) data sets it is imperative that contract-required methods and procedures be explicitly followed.

Comparability to previous generated data is enhanced by utilizing methodologies (sampling and analytical) similar to those used for previous investigations. Where utilizing previous methods is not practical or appropriate (e.g., due to advances in analytical methodology), comparability between data sets can be estimated by collecting samples at the same or nearby locations and comparing the results.

3.3.5 Radiological Data Completeness

Overall completeness is defined as the percentage of measurements that are judged to be usable (i.e., those which meet project-specific requirements) compared to the total number of measurements planned. Completeness is a function of both field and laboratory activities. Field sampling completeness is assessed through comparison of the number of samples collected and submitted to the number of planned samples (as specified in the FSP). Field sampling completeness may be less than 100 percent for various reasons, including field conditions (e.g., boring refusal at shallower depths than expected) or field error (sampling team fails to collect a planned sample). Laboratory completeness is assessed by calculating the usable data points generated relative to the total data expected (based on the number of samples submitted). It is important that appropriate QA procedures be maintained to verify that valid data are obtained in order to meet project needs. For the data generated, the goals required for completeness (or usability) of the analytical data are presented on Table 3-3. If these goals are not met, then USACE and Earth Tech project personnel will determine whether the deviations might necessitate corrective actions, such as collection and analysis of additional samples.

3.3.6 Radiological Data Sensitivity

The term sensitivity is used to describe contract method detection limits (MDLs), quantitation limits, and reporting limits (RL) established to meet project DQOs (USACE, 2001). The sensitivity terminology used for radiological analyses is the MDC or minimum detectable activity (MDA). The MDC limits that are required for each analysis are those described in Section 5.0 and summarized on Table 5-1 and are consistent with applicable method requirements and Guterl Steel site project DQOs. MDCs are sample-specific and represent the lowest activity levels that are achievable above instrument background. Method sensitivities published in USDOE and USEPA methods are based on a reagent water matrix, and do not

incorporate sample matrix interferences, dilutions, or dry-weight basis reporting (for non-aqueous samples) and the resulting effect on limits; therefore, the published limits may not be achievable for environmental samples.

Equations for calculating the MDA for field equipment are provided in the FSP, Attachment A, San Antonio Radiation Safety Group (SARSG) SOP 002, Section 5.2.5.

3.4 Measurement Quality Objectives for Chemical and Conventional Parameters Data

The purpose and use of the indicators for chemical data is the same as for radiological data, as discussed above in Section 3.3. That is, MQOs (also referred to as DQIs) establish specific criteria for the generation of data of known and acceptable quality, which allows for eventual data usability review.

In order to generate defensible chemical data of the necessary quality, criteria will be established and measured for the same DQIs used for assessment of radiological data:

- precision
- accuracy
- representativeness
- comparability
- completeness
- sensitivity

These DQIs apply to all definitive data produced by off-site (laboratory) chemical analysis. Calculation of data quality indicators is presented in Section 6.0.

3.4.1 Precision

Precision refers to the distribution of a set of reported values about the mean, or the closeness of agreement between individual test results obtained under prescribed conditions. Precision reflects the random error, may be affected by systematic error, and also characterizes the natural variation of the matrix and how the contamination exists or varies within that matrix (USACE 2001). Precision is evaluated using analyses of an analytical sample and its corresponding MD, laboratory MS/MSD, and/or LCS/LCSD which not only exhibit sampling precision, but indicate analytical precision through the reproducibility of the analytical results. As with radiological data, field duplicates will be generated at a frequency of one for each 20 environmental samples submitted for each parameter. RPD is a qualitative performance indicator used to evaluate precision. RPD criteria must meet the method requirements summarized in Table 3-3.

3.4.2 Accuracy

Accuracy is the measure of the closeness of an observed value to the “true” value (e.g., theoretical or reference value, or population mean). Accuracy includes a combination of random error and systematic error (bias) components that result from sampling and analytical operations (USACE, 2001). Sources of error are the sampling process, field contamination, preservation, handling, sample matrix, sample preparation, and analysis techniques. The laboratory objective for accuracy is to equal or exceed the accuracy demonstrated for the applied analytical methods

on samples of the same matrix. The percent recovery criterion is used to estimate accuracy based on recovery in the MS/MSD and LCS/LCSD samples. The MS and MSD, which will give an indication of matrix effects that may be affecting target compounds, are also a good gauge of method efficiency. Accuracy is also measured through the analyses and evaluation of method and field QC blanks, which aids in assessing the potential concentration contribution from various outside sources. Target analyte concentrations in blanks should not exceed one-half the project-specific RLs. Acceptable ranges of recovery and RL are reported in the referenced methods and summarized in Table 3-3.

3.4.3 Representativeness

Representativeness expresses the degree to which the sample data are indicative of the characteristics of a population of samples, parameter variations at a sampling point, or environmental conditions. Representativeness is a qualitative parameter which is most concerned with the proper design of the sampling program or subsampling of a given sample (USACE, 2001). Objectives for representativeness are defined for sampling and analysis tasks and are a function of the investigative objectives. The sampling procedures, as described in the FSP, have been selected with the goal of obtaining representative samples for the media of concern. Representativeness can be assessed qualitatively by the use of field and laboratory duplicate samples.

3.4.4 Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another (USACE, 2001). Comparability within this RI is achieved using standard techniques to collect and analyze representative samples and report analytical results in appropriate units. Complete field documentation using standardized data collection forms will support the assessment of comparability. Comparability is limited by the other parameters, because only when precision and accuracy are known, can data sets be compared with confidence. In order to generate comparable (internally consistent) data sets it is imperative that contract-required methods and procedures be explicitly followed.

Comparability to previously generated data is enhanced by utilizing methodologies (sampling and analytical) similar to those used for previous investigations. Where utilizing previous methods is not practical or appropriate (e.g., due to advances in analytical methodology), comparability between data sets can be estimated by collecting samples at the same or nearby locations and comparing the results.

3.4.5 Completeness

Overall completeness is defined as the percentage of measurements that are judged to be usable (i.e., those which meet project-specific requirements) compared to the total number of measurements planned. Completeness is a function of both field and laboratory activities. Field sampling completeness is assessed through comparison of the number of samples collected and submitted to the number of planned samples (as specified in the FSP). Field sampling completeness may be less than 100 percent for various reasons, including field conditions (e.g., boring refusal at shallower depths than expected) or field error (sampling team fails to collect a planned sample). Laboratory completeness is assessed calculating the usable data points

generated relative to the total data expected (based on the number of samples submitted). It is important that appropriate QA procedures be maintained to verify that valid data are obtained in order to meet project needs. For the data generated, the goals required for completeness (or usability) of the analytical data are presented on Table 3-3. If these goals are not met, then USACE and Earth Tech project personnel will determine whether the deviations might necessitate corrective actions, such as collection and analysis of additional samples.

3.4.6 Sensitivity

The term sensitivity is used to describe MDLs, quantitation limits, and RLs established to meet project DQOs (USACE, 2001). The RLs that are required for each analysis are those described in Section 5.0 and are consistent with applicable method requirements. The RL is the lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions. The RL is generally 5 to 10 times the MDL (USEPA, 1997). Instrument detection limits, MDLs, and RLs published in USDOE and USEPA methods are based on a reagent water matrix, and do not incorporate sample matrix interferences, dilutions, or dry-weight basis reporting (for non-aqueous samples) and the resulting effect on limits; therefore, the published limits may not be achievable for environmental samples.

3.5 Assessment of Geotechnical Data

There are no quantitative DQIs for the geotechnical data. Review of geotechnical data will be limited to review to determine if the data are complete, analyses were performed appropriately (specified method was used, appropriate sample size) and that the results are reasonable (e.g., particle size distribution data are consistent with field soil classification).

3.6 Measurement Quality Objectives for On-Site Laboratory Radiological Data

MQOs (DQIs) establish specific criteria for the generation of data of known and acceptable quality, which allows for eventual data usability review. Systematic QC checks are incorporated into the sampling and analyses to show that procedures and test results remain reproducible and that the analytical method is actually measuring the quality of target analytes without unacceptable bias. Systematic QC checks include the analyses of field/laboratory duplicates, calibration check standards, tracers, matrix spike samples (for non-tracer/carrier analyses), laboratory control samples, and method blanks. Measurement quality objectives (acceptance criteria or ranges) for these QC checks are established to verify DQIs support data usability, and contract compliance. The program of systematic QC checks may be reviewed from two aspects, batch QC and matrix-specific QC, as presented in Section 5.5.

In order to generate defensible data of the necessary quality, SOPs governing on-site laboratory operations will be prepared for USACE review and approval. Criteria will be established and measured for the following DQIs:

- precision
- accuracy
- representativeness
- comparability

- completeness
- sensitivity

These DQIs apply to the data produced by the on-site radiological laboratory. Calculation of data quality indicators is presented in Section 6.0.

3.6.1 On-Site Laboratory Radiological Data Precision

Precision of the on-site laboratory data is evaluated using analyses of an analytical sample and its corresponding MD, which will not only exhibit sampling precision, but indicates analytical precision through the reproducibility of the analytical results.

For the radiological analyses, precision will be evaluated by analysis of matrix duplicates. Field duplicates (blind to the laboratory) will be submitted to the on-site laboratory at a frequency of one per 20 environmental samples (excluding swipe samples); field duplicate data provide an indication of the overall precision of the sampling and analytical process. In addition, the on-site laboratory will analyze a sample duplicate (MD) with each analytical batch of 20 or fewer samples. RPD is a qualitative performance indicator used to evaluate precision. Preliminary RPD criteria must meet the method requirements summarized in Table 3-3; the final criteria will be established in the on-site LQMP. Precision is also assessed by comparison of the on-site laboratory data to the results generated by the off-site laboratory.

3.6.2 On-Site Radiological Data Accuracy

The on-site laboratory objective for accuracy is to equal or exceed the accuracy demonstrated for the analytical methods on samples of the same matrix. However, the on-site laboratory associated procedures include limited sample preparation (drying and removal of non-representative matter such as twigs or rocks) and therefore the introduction of spikes (known quantities) will not be introduced into the sample matrix.

In order to determine accuracy of the measurements made in the on-site laboratory, a correlation algorithm between STL-St. Louis results and the on-site laboratory results will be developed. Earth Tech anticipates that isotopic uranium data from the off-site laboratory will be available within four or five business days of receipt. As soon as the off-site laboratory data are received, a correlation algorithm (most likely by a linear regression) will be developed, and the correlation assessed. This will provide a direct comparison between samples analyzed in the fixed and on-site laboratories for accuracy comparison. The correlation will be evaluated and updated on an on-going basis as additional data are received and the database of paired results increases.

Accuracy is also measured through the analyses and evaluation of method blanks (analyzed daily), which aids in assessing the potential concentration contribution from various outside sources. COPC concentrations in blanks should not exceed one-half the project specific MDCs.

3.6.3 On-Site Radiological Data Representativeness

Representativeness expresses the degree to which the sample data are indicative of the characteristics of a population of samples, parameter variations at a sampling point, or environmental conditions. Consistent with the discussion in Section 3.3.4, the RI sampling procedures, as described in the FSP, have been selected with the goal of obtaining representative

samples for the media of concern. Representativeness of the samples and analytical processes can be assessed qualitatively by the use of field and laboratory duplicate samples. The large sample volume utilized for the on-site gamma spectroscopy method also enhances representativeness of the sample analyzed.

3.6.4 On-Site Radiological Data Comparability

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared with another (USACE, 2001). For the field (on-site) laboratory, comparability is the extent to which the same parameter is being measured (relative to the data reported by the off-site laboratory [STL-St. Louis]), as well as by the comparability of the results (assessed through the precision and accuracy criteria within the on-site laboratory, and the agreement of the on-site results with the data on the same sample generated by the off-site laboratory for both the off-site laboratory gamma spectroscopy and alpha spectroscopy results).

Comparability within this RI is achieved using standard techniques to collect and analyze representative samples and report analytical results in appropriate units. Complete field documentation using standardized data collection forms will support the assessment of comparability. Comparability is limited by the other parameters, because only when precision and accuracy are known, can data sets be compared with confidence. In order to generate comparable (internally consistent) data sets it is imperative that contract-required methods and procedures be explicitly followed.

Comparability to previously generated data is enhanced by utilizing methodologies (sampling and analytical) similar to those used for previous investigations. Where utilizing previous methods is not practical or appropriate (e.g., due to advances in analytical methodology), comparability between data sets can be estimated by collecting samples at the same or nearby locations and comparing the results.

3.6.5 On-Site Radiological Data Completeness

On-site laboratory completeness is assessed calculating the usable data points generated relative to the total data expected (based on the number of samples submitted). It is important that appropriate QA procedures be maintained to verify that useable data are obtained. For the on-site laboratory data generated, the goals required for completeness (or usability) of the analytical data are presented on Table 3-3. If these goals are not met, then USACE and Earth Tech project personnel will determine whether the deviations might necessitate corrective actions, such as collection and analysis of additional samples.

3.6.6 On-Site Radiological Data Sensitivity

The term sensitivity is used to describe contract MDLs, quantitation limits, and RLs established to meet project DQOs (USACE, 2001). The sensitivity terminology used for radiochemistry analyses is the MDC. The MDC limits for each analysis are described in Section 5.0 and summarized on Table 5-1 and are consistent with applicable method requirements and Guterl Steel site project DQOs. MDCs are sample-specific and represent the lowest activity levels that are achievable above instrument background. It is estimated that the count time will be on the order of 10 to 20 minutes, which will be adjusted as necessary to achieve the MDCs and laboratory throughput.

The sensitivity of the on-site laboratory is a function of many factors including counting time, sample matrix, size and geometry (among others); however, the laboratory sensitivity will be adequate to detect COPCs at concentrations of 1 picoCurie per gram (pCi/g) above background. Th-228, Th-230, and Ra-228 are inferred from the presence of other isotopes in the decay chain and the assumption of secular equilibrium.

4. SAMPLE RECEIPT, HANDLING, CUSTODY, AND HOLDING TIME REQUIREMENTS

Laboratory procedures for sample receipt and handling are critical to providing data that is of usable quality and legally defensible. Laboratories assigned to the Guterl Steel site must have written procedures for the acceptance and receipt of samples, sample handling and integrity, maintenance of the internal chain-of-custody (COC), and storage of samples upon completion of the required analytical procedures. The laboratory policies are described in the laboratories' Quality Management Plan(s). Table 4-1 contains analytical methods and container types, preservation, and holding time requirements for the Guterl Steel site RI.

4.1 Verification/Documentation of Cooler Receipt Information

Samples submitted by Earth Tech to the off-site laboratory will be received in a central sample receiving area by the laboratory sample custodian, who acknowledges receipt of the samples by signing the COC and recording the date and time that custody was transferred from the field to the laboratory. The date, time, cooler temperature, and person receiving the samples are also recorded on a Cooler Receipt Form (see USACE, 2001, Figure 3-3), or functional equivalent (e.g., Condition Upon Receipt form utilized by STL-St. Louis; example copy in Attachment A). The laboratory sample custodian is responsible for noting the condition of the samples upon receipt. STL-St. Louis' sample receipt and custody procedures are documented in SOP STL-PM-0002.

Similar procedures are utilized in the on-site laboratory, as described below (Section 4.3.).

4.2 Corrective Action for Incoming Samples

If the sample custodian discovers any problems with the documentation or the condition of the samples, the laboratory PM is notified immediately. Problems noted during sample receipt will be documented on a Cooler Receipt Form. The Earth Tech QA Manager will be contacted immediately for problem resolution. All corrective actions will be documented thoroughly in writing (e.g., copies of emails or faxes; written telephone conversation logs) and incorporated into the laboratory record and deliverable.

Documentation problems (e.g., inconsistencies between information on sample containers and the COC form; requested analyses not correct) can normally be corrected by communication between Earth Tech and the laboratory and do not adversely affect data quality. Other problems (broken or leaking containers; samples not properly preserved; insufficient sample volume) may have the potential to affect sample data quality and may require corrective action, up to and including re-sampling (if practical). The Earth Tech QA Manager will have the responsibility for specifying the appropriate corrective action. If possible (within the time constraints to make a decision and the availability of appropriate staff), USACE concurrence will be requested prior to implementation of the recommended action.

4.3 Receipt of Samples at the On-Site Laboratory

Soil samples collected for on-site laboratory analysis will be collected and placed into a standard 12-inch by 12-inch Ziploc bag, labeled with a unique sample number (as described in FSP Section 6.4.1) and submitted to the on-site laboratory by field personnel.

Custody documentation will be initiated at the sample site by the field team, who will transfer custody to the on-site laboratory personnel who receive the sample. The on-site laboratory will verify the completeness and accuracy of the sample information and log the samples in to the laboratory. The on-site laboratory will then process the sample (drying, sieving, and homogenizing) and perform the analyses. The on-site laboratory will serve as the central collection point for all samples being sent to an off-site laboratory, including maintaining custody documentation and transferring the field samples to the appropriate containers designated by the off-site laboratory (typically, 4-ounce or 8-ounce glass jars).

In the case of swipe samples, each swipe sample cover will be marked with the appropriate sample number. Multiple swipes will then be placed in a 12-inch by 12-inch Ziploc bag and delivered to the on-site laboratory with the appropriate COC. Upon receipt, each swipe sample and the COC will be checked for completeness and accuracy.

5. ANALYTICAL PROCEDURES

The laboratory procedures to be performed include methodologies from the USDOE Environmental Measurements Laboratory (EML) Health and Safety Laboratory (HASL) and USEPA (drinking water methods [USEPA, 1980] and SW-846 [USEPA, 1997]), as presented in Table 4-1. To the extent applicable to radiological analyses, samples will be analyzed following the guidance the DoD QSM (DoD, 2006). Table 5-1 indicates the MDCs for radionuclides in soil and water, the soil preliminary screening levels, and estimated background concentrations.

5.1 Identification of Analytical Procedures to Achieve DQOs

The analytical methods selected for the Guterl Steel site RI/FS are shown on Table 4-1. Details on how many of which analyses will be performed on samples from various media are provided in the FSP. This section briefly discusses the rationale for the selected methods.

5.1.1 Radiological Methods (Off-Site Laboratory)

The DGAR (USACE, 2006) identified a lack of isotope-specific radiological data as one of the principal data gaps for the Guterl Steel site. Site COPCs were initially identified as isotopic thorium and isotopic uranium. Thorium has been identified as a site COPC, and radium-228 is an important daughter product of Th-232 decay. Therefore, Ra-228 data is needed for risk assessment, as well as for IDW characterization; and therefore analysis for isotopic radium will also be performed. A limited number of analyses for uranium isotopes by ICP-MS will also be conducted to evaluate the possible presence of enriched or recycled U. The broad categories of radiological analyses performed by the off-site laboratory include:

- Isotopic uranium and isotopic thorium by gamma spectroscopy (soil samples only)
- Isotopic uranium and thorium by alpha spectroscopy (aqueous and non-aqueous samples)
- Radium-226 and Ra-228 by modified USEPA methods 903.0/904 (aqueous and non-aqueous samples)
- Gross alpha and beta radiation (groundwater and soil samples)
- Isotopic uranium by SW-846 method 6020 (ICP-MS)

As noted previously, 5 percent of the samples (minimum of 100) analyzed in the on-site laboratory will be sent to the off-site laboratory (STL-St. Louis) for isotopic analysis for isotopic uranium and thorium by gamma spectroscopy, comparable to the on-site laboratory (for comparability), and which utilizes a relatively large sample volume, improving the representativeness of the data. The STL-St. Louis gamma spectroscopy method is based on DOE-GA-01-R Mod; and the STL SOPs (RC-0025 for sample preparation and RD-0101 for instrumental analysis) are included in Attachment B. As noted on Table 5-1, not all the COPCs are amenable to direct measurement by the off-site laboratory by gamma spectroscopy.

About 50 percent of the samples submitted to STL-St. Louis for isotopic U and Th by gamma spectroscopy will also be analyzed for Ra-226 and Ra-228. STL-St. Louis' quantitative methods for these isotopes are based on USEPA methods 903.0 and 904; although STL-St. Louis' SOP incorporates some of the sample preparation steps of USEPA method 904 into their SOP for Ra-

226. The radium analysis is sequential, with an aliquot of the sample prepared for Ra-226 analysis then being utilized for the Ra-228 analysis. STL-St. Louis' SOPs (RC-0040 for Ra-226 and RC-0041 for Ra-228) are included in Attachment B.

Between 12 and 30 surface soil samples and between 12 and 30 subsurface soil samples from each investigative area (IA) or sub-area (see FSP Tables 5-8, 5-9, 5-11, and 5-12) will be analyzed for uranium and thorium isotopes by alpha spectroscopy and for Ra-226 and Ra-228. (Note that the samples submitted for alpha spectroscopy analysis are independent of the samples submitted for gamma spectroscopy analysis; some, though by no means all, of the analyses may overlap on any given sample. See FSP Section 5.4.3.1.3) Alpha spectroscopy utilizes a small sample mass (typically on the order of one gram), making obtaining a representative sample more difficult, but providing more definitive identification of the isotopes present in the sample. The STL-St. Louis method (SOP-RD-0210), based on the DOE HASL-300 alpha spectroscopy (DOE A-01-R), will be used for site COPCs (Th-232 and U-234, U-235, and U-238); copies of these SOPs are also included in Attachment B. The radiological methods selected have the necessary specificity and also are sensitive enough to achieve the preliminary radiological screening criteria identified in the DGAR (Section 2.6). In addition, low-concentration samples will be analyzed with sufficient sensitivity (i.e., using STL-St. Louis' 'long count' method) to determine the presence or absence of radionuclides at background levels.

Analysis for radium isotopes will be performed utilizing STL-St. Louis SOPs RC-0040 and RC-0041, based on USEPA (1980) drinking water methods 903.0 (for Ra-226) and 904 (Ra-228). Due to the 14 to 21 days needed for ingrowth (i.e., to allow for the buildup of short-lived daughter products), accelerated turnaround time is not possible for isotopic Ra analyses. Soil samples will be prepared for isotopic radium analysis by STL SOP RC-0004.

In addition to isotope-specific analyses, groundwater samples will be analyzed for gross alpha and beta radioactivity using STL SOP RC-0020, which is based on EPA method 900.0 and SW-846 method 9310. These analyses will be conducted to provide general information on the presence or absence of radionuclides in groundwater, and also to confirm previous data from landfill monitoring wells indicating the presence of radionuclides at levels exceeding New York water quality standards. The STL-St. Louis method has the specificity to report the analytes as noted in the water quality criteria and is also sufficiently sensitive to measure the analytes at concentrations below the standard.

Total uranium (non-isotopic) concentrations for risk assessment purposes (to assess the chemical toxicity of uranium) and also to assess compliance with the groundwater criteria for total uranium will be derived (calculated) from the alpha spectroscopy results for isotopic uranium. Preliminary aqueous screening levels for isotopic radium, thorium, and uranium have been developed and are shown on Table 5-1.

5.1.2 Chemical and Conventional Parameters Analyses

Only a very limited amount (in terms of parameters and sample quantities) of chemical and conventional parameters analyses are planned. (The only MED/AEC-related contaminants identified at the Guterl Steel site are the radionuclides discussed above.) STL-St. Louis will perform isotopic uranium analysis by their SOP MT-0001, which is based on SW-846 Method 6020 (metals by ICP-MS); soil samples will be prepared for analysis by STL SOP IP-0002 (SW-

846 3050B). Waste characterization analyses (e.g., hazardous characteristics including TCLP metals analyses) will be performed by the waste disposal subcontractor as necessary to develop data for the classification and disposal of the Guterl RI IDW.

In addition to isotopic uranium by ICP-MS, a few samples from across the site will be analyzed for conventional parameters such as total organic carbon to assist in evaluating contaminant fate and transport. Groundwater samples will also be analyzed for TSS to assess the likelihood that inorganic contaminants detected are bound to the sediment entrained in the sample, as opposed to being in the dissolved phase.

5.1.3 Geotechnical Analyses

Analysis or estimation of various geotechnical parameters is necessary for site characterization (including contaminant transport) and input to the RESRAD model. Relevant parameters include:

- Hydraulic conductivity, to assess groundwater movement and contaminant transport
- Grain size distribution
- Atterberg limits

Other parameters (bulk density, porosity) needed for input to the RESRAD model will be estimated from literature values which may also be confirmed through site specific data derived from recovered soil cores (e.g., the weighing of core segments of known dimensions at the field laboratory).

5.1.4 On-Site Laboratory Radiological Methods

Field samples will be analyzed in the on-site laboratory for radiological constituents utilizing gamma spectroscopy. The specifics of the on-site laboratory operations will be provided in the LQMP and SOPs to be submitted as part of Task 6 of this task order.

5.2 Preventive Maintenance

Preventive maintenance will be provided both for the off-site laboratory (Section 5.2.1) and on-site laboratory (Section 5.2.2), as described below.

5.2.1 Off-Site Laboratory Preventive Maintenance

The laboratory is responsible for the maintenance of its analytical equipment. The instrument manufacturer, model number, accessories, etc., required for analysis are detailed in the laboratory Quality Management Plan (QMP). Preventive maintenance is provided on a regular basis to minimize downtime and the potential interruption of analytical work. Instruments are maintained in accordance with the manufacturers' recommendations. If instruments require maintenance, only trained laboratory personnel or manufacturer-authorized service specialists are permitted to do the work. Maintenance activities will be documented in permanent logs. These logs will be available for inspection by auditing personnel. STL-St. Louis' preventive maintenance policy and procedures are documented in their SOP QA-0024.

5.2.2 On-Site Laboratory Preventive Maintenance

The on-site laboratory is responsible for the maintenance of its analytical equipment. The details of the instrument manufacturer, model number, accessories, etc., required for analysis will be detailed in the laboratory SOPs (to be provided as a Task 6 deliverable). Preventive maintenance is provided on a regular basis to minimize downtime and the potential interruption of analytical work. Instruments are maintained in accordance with the manufacturers' recommendations. If instruments require maintenance, only trained personnel or manufacturer-authorized service specialists are permitted to do the work. In addition, ARS has service contracts in place for the analytical equipment utilized in the on-site laboratory. Maintenance activities will be documented in permanent logs. These logs will be available for inspection by auditing personnel.

5.3 Analytical Support Areas (Off-Site Laboratory)

Prior to generating quality data, several analytical support areas must be considered:

Standard/Reagent Preparation. Primary reference standards and secondary standard solutions will be obtained from NIST, or other reliable commercial sources, to verify the composition of the material. The preparation and maintenance of standards and reagents will be accomplished per the methods referenced in Table 5-1. The source, preparation, and composition of standards and standard solutions are to be formally documented (i.e., in a bound logbook) and should identify the supplier, lot number, purity/concentration, receipt/preparation date, preparer's name, method of preparation, expiration date, and any other pertinent information. Standard solutions will be validated prior to use. Care will be exercised in the proper storage and handling of standard solutions (e.g., separating volatile standards from nonvolatile standards). The laboratory will monitor the quality of the standards and reagents at a frequencies and procedures identified in the laboratory QMP and in the STL-St. Louis facility SOP QA-002.

Balances. The analytical balances will be calibrated and maintained in accordance with manufacturers' specifications. Calibration is conducted with two ASTM Class 1 weights that bracket the expected balance use range. The laboratory will check the accuracy of the balances daily or before use and document the check in a bound logbook. Annual calibration by a certified technician is also required. STL SOP QA-005 meets the NELAC requirement for balance calibration.

Refrigerators/Freezers. The temperature of the refrigerators and freezers within the laboratory will be monitored and recorded daily (or continuously). This will verify that the quality of the standards and reagents is not compromised and the integrity of the analytical samples is upheld. The applicable acceptance ranges (2° to 6° C for refrigerators; normally -10° to -20° C for freezers) will be clearly posted on each unit in service.

Water Supply System. The laboratory must maintain a sufficient water supply for all project needs. Water used for analytical work must be analyte-free (e.g., ASTM Type II [ASTM D 1193-99 or current version] or better) to prevent false positives in the data. Ultraviolet or carbon absorption treatment is recommended for organic analyses and ion-exchange treatment is recommended for inorganic tests. Appropriate documentation of the quality of the water supply system(s) will be performed on a regular basis. STL SOP QA-0028 meets the NELAC require for water system maintenance and monitoring.

The same procedures and standards will be met by the on-site laboratory with regard to standards and balances. The on-site laboratory does not require an analyte free water supply and none will be maintained at the field laboratory. Although refrigeration will not be required for those samples analyzed for radionuclides, samples collected for chemical and conventional analyses require being maintained at 4° C while stored on site and in shipment. A lockable refrigerator may be installed in the field laboratory trailer for convenience but is not required for the maintenance of the on-site samples.

5.4 Calibration Procedures and Frequency

In order to obtain the necessary level of precision and accuracy during sample processing, laboratory instruments must be calibrated properly. Several analytical support areas must be considered so the integrity of standards and reagents is upheld prior to instrument calibration. The following sections describe these analytical support areas and associated laboratory instrument calibration procedures.

Instruments calibration is required to verify that the analytical system is operating properly and at the sensitivity necessary to meet established quantitation limits. Each instrument for organic and inorganic analyses will be calibrated with standards appropriate to the type of instrument and linear range established within the analytical method. Calibration of laboratory instruments will be performed according to the requirements of the specified methods, the DoD QSM, and the LQMP.

In addition to the requirements stated in the analytical methods, the off-site laboratory will be required to analyze an additional low-level standard at or near the detection limits reported in Section 6.0. In general, standards will be used that bracket the expected concentration of the samples. This will require the use of different concentration levels, which will be used to demonstrate the instrument's linear range of calibration.

Calibration of an instrument must be performed prior to the analysis of any samples and then at periodic intervals (i.e., continuing calibration) during the sample analysis to verify that the instrument is still properly calibrated. If the laboratory cannot meet the method-required calibration requirements, corrective action will be taken as discussed in Section 5.7. All corrective action procedures taken by the laboratory will be documented, summarized within the case narrative, and submitted with the analytical results.

5.5 Laboratory QC Procedures

5.5.1 Analytical Sequence QC

The QC elements required for each analytical sequence will be performed in accordance with requirements stated within the analytical methods.

5.5.2 Batch/Matrix-Specific/Performance-Based QC

Internal QC checks are used to determine if analytical operations at the laboratory are being performed in accordance with specified quality control procedures, as well as to determine the effect the sample matrix may have on data being generated. Three types of internal checks are performed: batch QC; matrix-specific QC; and performance-based procedures. The type and

frequency of specific QC samples performed by the laboratory will be selected according to the specified analytical method and project-specific requirements. Acceptable criteria or target ranges for these QC samples are presented in the analytical methods referenced in Table 5-1 as detailed in the laboratory SOPs.

If QC results are outside acceptable ranges, appropriate corrective measures will be implemented; the impact these corrective measures may have on the established data quality objectives will be assessed; and the data may be qualified (either by indicating the exceedance on the laboratory data report [“Q” qualifier in accordance with DoD QSM]) or during data quality review. Quality control samples, including any project-specific QC samples (i.e., split samples) that will be analyzed, are discussed below.

5.5.2.1 Batch QA

Method Blanks. A method blank is defined as an interference-free blank matrix similar to the sample matrix to which all reagents are added in the same volumes or proportions as used in sample preparation and carried through the complete sample preparation, cleanup, and determinative procedures. For soil analyses, a purified solid matrix (e.g., clean sand is used for the method blank for isotopic radionuclide analysis) will be used. The method blank is used to determine the level of laboratory background contamination. Method blanks are analyzed at a frequency of one per analytical batch.

Laboratory Control Samples (LCS). A LCS is an aliquot of standard control matrices spiked (fortified) with all the elements being analyzed for calculation of precision and accuracy to verify that the analysis that is being performed is in control. A laboratory control sample will be analyzed (with every extraction / analytical batch) for each matrix and parameter.

Sample Duplicate. STL-St. Louis’ SOPs for radionuclides in aqueous and non-aqueous samples requires that a sample duplicate (a duplicate generated by the laboratory) be analyzed with each batch of 20 samples or fewer. Note that this duplicate is in addition to any blind field duplicates submitted by the field sampling team.

5.5.2.2 Matrix-Specific and Field QC

Matrix Spike Samples. A matrix spike sample is an aliquot of a matrix spiked with known concentrations of all target analytes being analyzed (the DoD QSM requires all target analytes to be included as spiked compounds) as stipulated by the selected methodology. The MS/MSDs are subjected to the entire analytical procedure in order to assess both precision and accuracy of the method for the matrix by measuring the percent recovery and the relative percent difference of the two spiked samples. The samples are used to assess matrix interference effects on the method, as well as to evaluate instrument performance. MS/MSDs are analyzed at a frequency of one each per 20 samples per matrix, or one for each analytical batch, whichever is more frequent. The DoD QSM requires that the matrix QC be performed on a site sample (i.e., on one of the samples submitted from the Guterl Steel site, not on a sample analyzed in the batch from a different site). MS/MSDs will be performed for the parameters as shown on Table 4-2. Note that a matrix spike is not required for radiological analyses (including isotopic uranium, thorium, and radium) for which a tracer is added to monitor yield. However, for analyses for which MS/MSD is not performed, a LCSD will be analyzed (if a sample duplicate is not analyzed).

Field Duplicates. The field duplicate (submitted blind to the laboratory) is the second of two representative aliquots of the same sample, which are prepared and analyzed identically. Collection of duplicate samples provides for the evaluation of precision both in the field and at the laboratory by comparing the analytical results of two samples taken from the same location. Obtaining duplicate samples from a solid matrix (i.e., soil) requires homogenization of the sample aliquot prior to filling sample containers, in order to best achieve representative samples. Due to interferences, lack of homogeneity, and the nature of the solid samples, the analytical precision goals for non-aqueous samples are less stringent than those for aqueous samples, and may not always be achievable. Field duplicate samples are to be included at a maximum of one per 20 environmental samples per analysis type and matrix.

Equipment Rinsate Blanks. Rinsate blanks are collected to assess the potential for cross-contamination of samples during collection. Rinsate blanks will be collected for each sampling equipment type (e.g., split spoon sampler, macro-core, etc.) and analyzed at a rate of one per decontamination event, with a minimum of one per week (to verify that the equipment has not become contaminated from ongoing storage). Disposable, dedicated equipment purchased pre-cleaned from a vendor and from the same lot is considered a single decontamination event for that equipment type. Rinsate blanks consist of distilled water or analyte-free water obtained from the laboratory collected from the final rinse of aqueous sampling equipment after the decontamination procedures described in the FSP.

5.5.2.3 Performance-Based QC

QA samples (or split samples) are used for performance audits or inter-laboratory comparability of data. A QA sample is defined as a homogenized replicate of a field sample. QA samples will be taken at a five percent frequency (relative to field samples) and sent to a QA laboratory designated by USACE. The QA laboratory will be notified approximately two weeks prior to any QA samples being shipped.

QA samples will be collected in the field. A USACE-provided sample ID number will be applied to the labels, chain-of-custody records, and all correspondence for all QA samples shipped to the QA laboratory throughout the project.

QA split samples will be collected at a 5 percent frequency and submitted to a laboratory designated by USACE for analysis of radiological COPCs. Evaluation of the split sample results is the responsibility of the USACE. If available, Earth Tech will incorporate the USACE split sample evaluation into the final QCSR.

5.5.3 On-Site Laboratory QC

On-site laboratory QC will be specified in the LQMP, which will be developed and approved by USACE prior to implementation. Areas to be addressed by the on-site LQMP are shown in the example table of contents, provided as Figure 3-1 of this QAPP. Typical items may include system calibration (three times daily; at the beginning, middle and end of the day); method blanks (analyzed at the beginning of the day) and duplicates (analyzed once for every 20 environmental samples). The on-site laboratory calibration is prepared by an outside vendor (Analytics of Atlanta, GA) and is NIST-traceable. A typical calibration standard is provided in Attachment C and spans a range of gamma-ray energies from low to high.

5.6 Performance and System Audits

Audits will include a careful evaluation of both field and laboratory QC procedures and will be performed before or shortly after systems are operational. The audits will be conducted by an individual who is technically knowledgeable about the operation(s) under review. Performance audits will be conducted by introducing control samples into the data production process. These control samples may include performance evaluation samples, field samples spiked with known amounts of analyte, and split field samples that will be analyzed by two or more analysts within or outside the organization.

System audits are on-site qualitative inspections and reviews of the QC system used by some part of or the entire measurement system. They provide a quantitative measure of the quality of the data produced by one section of or the entire measurement process. The audits are performed against a set of requirements, which may be a QA project plan or work plan, a standard method, or a project statement of work. The primary objective of the system audits is to verify that the QA/QC procedures are being followed.

The laboratory must conduct internal technical audits and systems audits, as specified in the DoD QSM (Section 5.3.1). Audits will be conducted by persons independent of the activity being audited. The laboratory shall schedule audits so that all elements and areas of laboratory operations are reviewed over the course of one year. If the audit indicates problems, the laboratory shall take corrective action as quickly as possible and notify Earth Tech if any results or reports submitted may be affected.

5.6.1 Performance and External Audits

In addition to conducting internal reviews and audits, as part of its established QA program, the laboratory is required to take part in regularly-scheduled performance evaluations and laboratory audits performed by state and federal agencies (e.g., USACE), as well as by independent agencies (i.e., NELAC). They are conducted as part of the certification process and to monitor the laboratory performance. The audits also provide an external QA check of the laboratory and provide reviews and information on the management systems, personnel, standard operating procedures, and analytical measurement systems. Acceptable performance on evaluation samples and audits is required for certification and accreditation. The laboratory will use the information provided from these audits to monitor and assess the quality of its performance. Problems detected in these audits will be reviewed by the QA Officer and Laboratory Manager, and corrective action will be instituted as necessary.

Earth Tech is not contracted to perform laboratory audits. However, Earth Tech will assign qualified personnel to conduct laboratory audits if requested by USACE. These project-specific laboratory performance review audits would be conducted only at the direction of and in conjunction with the USACE, when requested. The scope and format of any such audits will be determined jointly between USACE and Earth Tech QA staff.

5.6.2 Systems and Internal Audits

As part of its QA Program, the Laboratory QA Manager will conduct periodic checks and audits of the analytical systems, as directed by the laboratory QMP. The purpose of these is to verify that the analytical systems are working properly, and that personnel are adhering to established

procedures and documenting the required information. These checks and audits also assist in determining or detecting where problems are occurring, and include examination of laboratory documentation of sample receipt, sample log-in, sample storage, COC procedures, sample preparation and analysis, instrument operating records, etc.

Periodically, the Laboratory QA Manager will submit single-blind performance evaluation samples that are prepared along with project samples to the laboratory for analysis. These samples will serve to check the entire analytical method, the efficiency of the preparation method, and the analytical instrument performance. The results of the internal performance evaluation sampling will be reviewed by the Laboratory QA Manager who will report the results to the analyst and the Laboratory Director. When a problem is indicated, the Laboratory QA Manager will assist the analyst and laboratory management in determining the reason and in developing solutions. The Laboratory QA Manager also will recheck the systems as required.

5.6.3 On-Site Laboratory Audits

On-site laboratory audits will be conducted in exactly the same manner as identified above, and will be conducted by the Laboratory QA Manager. At least one audit of the on-site laboratory will be conducted. Audits will be conducted every six months in the event that on-site laboratory analysis is conducted for more than a six-month period. If field work (including on-site laboratory analysis) shuts down for an extended period (e.g., for the winter) and then resumes, another audit will be conducted.

5.7 Non-Conformance/Corrective Actions

The laboratory shall have established, documented procedures to identify and control work and identify results that do not conform to the specified requirement. The laboratory shall also have a policy and procedures for actions to be taken in the event of a non-conformance and to prevent a recurrence. Corrective actions will be implemented to resolve problems and restore proper functioning to the analytical system when errors, deficiencies, or out-of-control situations exist at the laboratory. Full documentation of the corrective action procedure needed to resolve the problem will be filed in the project records, and the information will be summarized in the case narrative. STL-St. Louis' policy and procedure for dealing with nonconformance issues is addressed in SOP QA-0036. Discussion of the corrective actions to be taken is presented in the following sections.

5.7.1 Incoming Samples

Problems noted during sample receipt at the off-site laboratory will be documented on a Cooler Receipt Form (see Attachment A), as discussed in Section 4.2 of this QAPP. (STL-St. Louis uses its own form, a "Condition Upon Receipt" form, for this purpose.) The Earth Tech QA manager will be contacted as soon as practical for problem resolution. Corrective actions will be documented thoroughly.

Problems associated with sample receipt at the on-site laboratory will be addressed in real time by field personnel. If necessary, re-sampling will be conducted to resolve such problems.

5.7.2 Sample Holding Times

If sample extractions or analyses exceed method holding time requirements, the Earth Tech QA manager will be notified promptly for problem resolution. Corrective actions will be documented thoroughly. Due to the extended holding times allowed for the principal COPCs for the Guterl Steel site (isotopic uranium, isotopic thorium, and Ra-226 and Ra-228), holding time exceedances are not expected. Samples analyzed by the on-site laboratory will normally be analyzed within hours of collection and holding time exceedances will not be a problem. It should be noted that the holding times shown on Table 4-1 for radionuclides in non-aqueous matrices are based on STL's SOPs (RC-004 and RC-005) and internal policies. There is no technical holding time limit for these analyses, and there is no adverse affect on data generated from samples held for longer periods.

5.7.3 Instrument Calibration

Sample analysis will not be allowed until all initial calibrations meet the appropriate requirements. Laboratory instrumentation must be calibrated in accordance with method requirements. If any initial/continuing calibration standards exceed method QC limits, recalibration must be performed and, if necessary, reanalysis conducted of all affected samples analyzed since the previous acceptable calibration check.

5.7.4 Minimum Detectable Concentrations and Reporting Limits

The laboratory must meet the project-required MDCs and RLs presented in Table 5-1. If difficulties arise in achieving these limits due to a particular sample matrix, the laboratory must notify the Earth Tech QA manager (or designee) for problem resolution. In order to achieve those project-required MDCs and RLs, the laboratory must utilize the necessary and appropriate cleanup procedures. When a sample requires a secondary dilution due to high levels of target analytes (applicable only to chemical and conventional parameters analyses), the laboratory must document the initial analysis and secondary dilution results; the results of both analyses must be reported. Secondary dilution will be permitted only to bring target analytes within the linear range of calibration.

5.7.5 Method QC

QC samples, including blanks, matrix duplicates, matrix spikes, laboratory control samples, and other method-specified QC samples, will meet the requirements of them methods referenced in Table 4-1 and laboratory SOPs. Failure to achieve the method-required QC criteria will result in the review and possible qualification of all affected or associated data. If the laboratory cannot find any errors, the affected sample(s) will be reanalyzed or re-extracted/redigested, then reanalyzed within method-required holding times (if possible) to verify the presence or absence of matrix effects. If matrix effect is confirmed, the corresponding data will be flagged accordingly (as specified by the method and defined by the data validation guidelines identified in Section 8.2). If matrix effect is not confirmed, then the entire batch of samples may have to be reanalyzed or re-extracted/redigested. The Earth Tech QA manager will be notified as soon as possible to discuss possible corrective actions should unusually difficult sample matrices be encountered.

5.7.6 Calculation Errors

The analytical results must be reviewed systematically for accuracy prior to submittal. If, upon data review, calculation and/or reporting errors are discovered to exist, the laboratory will be required to reissue the analytical data report with the corrective actions appropriately documented in the case narrative.

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6. DATA REDUCTION/CALCULATION OF DATA QUALITY INDICATORS

Data quality and quantity are measured by comparison of resulting data with established acceptable limits for data precision, accuracy, representativeness, comparability and completeness (PARCC) and sensitivity, collectively referred to as data quality indicators (DQIs). Data outside PARCC/sensitivity QA objectives will be evaluated according to Section 8 and the criteria contained in the specified analytical methods, to determine what, if any, aspects of the data can be defensibly used to meet the project objectives.

The analytical data generated by the laboratory will be reviewed prior to generating the RI Report to assess and document the usability/validity of the reported results. This internal data review process will consist of data generation, reduction, a minimum of three levels of documented review, and reporting. As discussed in Section 3.3 and Section 3.4, DQIs will be measured during off-site chemical analysis. Calculations of these DQIs are presented below.

6.1 Laboratory Data Reduction and Review

Laboratory analytical data are first generated in raw form at the instrument. These data may be in either graphic or tabular form. Specific data reductions, generation procedures, and calculations are found in each of the methods referenced in Table 4-1, as well as within the laboratory QMP and individual analytical SOPs. Analytical results must be reported consistently.

- Data for soils and other non-aqueous matrices will be reported in concentrations of pCi/g for radiological parameters, and reported in micrograms per kilogram ($\mu\text{g/kg}$) or milligrams per kilogram (mg/kg) for other parameters (including isotopic uranium by ICP-MS). Soil samples will be reported by the on-site laboratory and the off-site laboratory by dry weight.
- Data for water samples will be reported in concentrations of picoCuries per liter (pCi/L) for radiological parameters, and reported in micrograms per liter ($\mu\text{g/L}$) or milligrams per liter (mg/L) for other parameters.

Data reduction will be performed by individuals experienced with a particular analysis, and knowledgeable of project QA/QC requirements.

The technician/analyst who generates the analytical data is responsible for its correctness and completeness. The data review process involves evaluating both the results of the QC data and the professional judgment of the person(s) conducting the review. Applying technical knowledge and experience to the evaluation of data is essential in verifying that data generated are of a quality adequate for the intended use.

The laboratory has documented procedures that are to be followed and must be accessible to all laboratory personnel. The data review is generally conducted in a three-step process at the laboratory prior to submittal:

- Level 1 - Technical Data Review - The analysts review the quality of their work based on an established set of guidelines. The review will verify, at a minimum, that appropriate preparation, analysis, and standard operating procedures have been followed; analytical

results are correct and complete; QC samples are within established control limits; and that documentation is complete (e.g., any anomalies have been documented).

- Level 2 - Technical Review - This level of review will be performed by a supervisor or data review specialist whose function is to provide an independent review of the data package. This review will also be conducted according to an established set of guidelines (i.e., method requirements and laboratory standard operating procedures). The Level 2 review includes a review of qualitative and quantitative data, and a review of documented anomalies.
- Level 3 - Administrative Data Review - The final review of the data, prior to submittal, is performed by a QA/QC officer or program administrator at the laboratory. This level provides a total overview of the data package to verify its consistency and compliance with project requirements.

A detailed description of the laboratory's data review process is described in the QMP of the laboratories proposed for the analyses to be conducted for this project. STL-St. Louis' internal review, verification, and reporting policy and procedures are documented in SOP PM-0004.

6.2 Precision

According to NELAC (as cited in DoD, 2006), precision is “the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator.” Precision reflects the random error and may be affected by systematic error. Precision also characterizes the natural variation of the matrix and how the contamination exists or varies within that matrix. Precision is evaluated using analyses of an analytical sample and its corresponding matrix duplicate, MS/MSD, or LCS/LCSD, which not only exhibit sampling precision, but indicate analytical precision through the reproducibility of the analytical results. Field duplicate and laboratory duplicate samples will be used to measure precision for project samples. Both sampling and analysis will be as consistent as possible. For a pair of measurements, RPD (or absolute difference; see Section B5.1) will be used, as presented below:

$$RPD(\%) = \frac{|D_1 - D_2|}{\left[\frac{(D_1 + D_2)}{2} \right]} \times 100$$

Where:

D_1 and D_2 = the two replicate values.

The upper limit for precision in non-aqueous matrix field duplicates is 100 percent RPD (in accordance with USEPA Region 2 data validation criteria for inorganics) for analytes present at five times the sample quantitation limit, and 50 percent RPD for aqueous duplicates. Duplicate data will also be assessed using the DoD QSM default precision goal of 30 percent RPD (DoD, 2006). However, it should be noted that the DoD QSM criteria were developed for laboratory duplicates; field duplicates would be expected to have lesser precision (i.e., higher RPDs). In

addition, the DoD QSM criteria were developed for conventional chemical analyses (i.e., metals and organics) and may not be fully applicable to radiological analytical methods.

The laboratory's in-house acceptance criteria will also be used in the assessment of laboratory duplicates (i.e., MD, MSD, and LCSD).

Precision will also be assessed for sample pairs analyzed both in the on-site laboratory and the off-site laboratory, using the same equation as above.

6.3 Accuracy and Bias

Accuracy is defined as the degree of agreement between an observed value and an acceptance value. Accuracy includes a combination of random error and systematic error (bias) (DoD, 2006). Analytical bias (accuracy) may be assessed through the use of known and unknown QC samples and spiked samples. Bias is presented as percent recovery. Bias will be determined from matrix spike and laboratory control samples, as well as from tracer compounds added to alpha spectrometry fractions, and is calculated using the equation below:

$$\%R = \frac{SSR - SR}{SA} \times 100$$

Where:

%R = % recovery

SSR = spike sample result

SR = sample result

SA = amount of spike added to sample

Radiochemistry accuracy is also assessed through method and/or field QC blank evaluation.

Accuracy and bias of the on-site laboratory data will also be assessed by comparison with the off-site laboratory data. In addition to the calculation of precision (relative to the off-site laboratory data; discussed above in Section 6.2), the on-site laboratory data will be evaluated for bias relative to the off-site laboratory data.

6.4 Minimum Detectable Activities and Method Detection Limits

The procedure for determining the minimum detectable concentrations or activities for radionuclides is discussed in Section 6.4.1, below. The protocol for establishing the detection limit for chemical analyses is discussed in Section 6.4.2.

6.4.1 Minimum Detectable Concentrations / Activities

MDCs, or MDAs, will be determined for each radionuclide using procedures outlined in MARSSIM and MARLAP. The MDC is defined as a level of activity concentration which is practically achievable by an overall measurement method, and considers not only the instrument characteristics (e.g., background and efficiency), but all other factors and conditions which influence the measurement (including sample size, counting time, self-absorption and decay factors, and chemical yield).

6.4.2 Method Detection Limits

MDLs will be determined for non-radiological (chemical) analyses (including uranium isotopes determined by ICP-MS by SW-846 method 6020) using procedures outlined in 40 CFR Part 136, Appendix B. The MDL normally is calculated using data generated from reagent water. The basic procedure involves analysis of at least seven replicates of a standard prepared at a concentration near (within a factor of five) the estimated detection limit; calculating the standard deviation of the results; and multiplying the standard deviation by the appropriate Student's *t* value (3.143 for seven replicates [six degrees of freedom] at the 99 percent confidence level).

6.5 Completeness

Completeness is defined as the percentage of data that is judged to be valid to achieve the objectives of the investigation compared to the total amount of data planned. Analytical completeness is the percentage of the usable data relative to the amount of data generated. Deficiencies in the data may be due to sampling techniques, poor accuracy, precision, or laboratory error. While the deficiencies may affect certain aspects of the data, usable data may still be extracted from applicable samples. An evaluation of completeness necessarily involves an evaluation of the impact of missing data on the ability of the project to achieve its goals. The goal for off-site laboratory analytical completeness is 95 percent. The equation used for analytical completeness is presented below:

$$C(\%) = \frac{D \times 100}{P \times n}$$

Where:

- C = Completeness
- D = Number of usable data points (includes both detections and non-detected results). Usable data are those with no qualifier; or with the U, J, or UJ qualifiers. (The 'D' flag, indicating a result from a dilution, is not considered a data quality qualifier; D-flagged data are also fully usable.)
- P = Number of analytical parameters per sample requested for analysis (e.g., three for isotopic uranium by HASL-300 Method A-01-R [as there are three discrete uranium isotopes which are reported by that method])
- n = Number of samples requested for analysis

Sampling completeness is calculated in the same manner, except that the numerator is the number of samples collected, and the denominator is the number of planned samples. The goal for sampling completeness is 95 percent.

Overall completeness, which is a function of both sampling completeness and analytical completeness, is calculated in a similar manner; except that the denominator is the number of data points planned. The goal for overall completeness (analytical and sampling combined) is 90 percent.

7. LABORATORY OPERATIONS DOCUMENTATION

7.1 Sample Management Records

Procedures addressing sample management documentation in the laboratory (i.e., sample chain-of-custody, sample receipt verification and/or handling requirements, and any intra-laboratory custody requirements) are presented in the laboratory quality management plans of the laboratories proposed for this project. STL-St. Louis' document control procedures and policy are documented in SOP QA-0023.

7.2 Data Reporting Procedures

7.2.1 Data Package Format and Content

The laboratory analytical reports will meet the requirements of Appendix DoD-A in the DoD QSM (DoD, 2006). At a minimum, the following will be included for the radiological data packages. Note that for the Guterl RI, the “optional items” will also be submitted (to the extent relevant to the specific data being reported). The list below is a summary; the reader is referred to the QSM for the comprehensive list of required items.

DoD QSM App A Item	Summary Description of Item Contents
1. Cover Sheet	Identifies data package, lab, site, contract, etc.
2. Table of Contents	Data package should be paginated
3. Case Narrative	Full case narrative, including descriptions of any deviations which may affect the data, and a summary of any issues which need to be highlighted for the user to assess the usability of the data. The narrative will also include a list of all samples in the package and a definition of all data qualifiers used by the laboratory.
4. Analytical results	Report results and qualifiers, field and laboratory sample ID, matrix, dates (preparation, analysis, etc) including reporting limits, dilutions, re-analyses, etc. The “optional information” under this item is also to be submitted, with special attention to the reporting of the qualitative estimate of uncertainty for radiological analyses.
5. Sample Management Records	Chain-of-Custody form, shipping documents, cooler receipt form, telephone logs, etc.
6. QA/QC information	MS and MSD results; LCS and LCSD results; tracer recoveries; method blanks results; QC acceptance criteria; spike concentrations/spike added values; and batch numbers (preparation, analysis, other).
7. Information for Third-Party Review	Initial and continuing calibration data; performance standards; raw data (to include percent solids determination logs); QA/QC information not previously provided to fulfill item 6; and other supporting documentation.

A Cooler Receipt Form (or functional equivalent; see Section 5.7.1), one form per cooler, will also be required with each deliverable data package for the purposes of noting problems in sample packaging, documentation, preservation, and condition on receipt. The laboratory will also be requested to provide the data package in an Adobe portable document format (pdf).

7.2.2 Electronic Deliverables

In addition to the required deliverables summarized above, the laboratory will also provide an electronic data deliverable (EDD) in a Microsoft-compatible format (Access or Excel) for transferring the data readily into spreadsheets and database applications.

7.3 Data Management Procedures

7.3.1 Laboratory Turnaround Time

The contract laboratory will be required to submit the analytical data packages, in accordance with Section 7.2.1, four weeks from verified (or validated) time of sample receipt (VTSR) at the laboratory.

The contract laboratory will be required to fax a copy of the Cooler Receipt Form (see Attachment A) to Earth Tech upon receipt of the samples. Alternatively, the laboratory may scan the form and email the form as a pdf file.

7.3.2 Data Archival/Retention Requirements

The laboratory is responsible for generating, controlling, and archiving laboratory records for the Guterl Steel RI. This information should be maintained with a system that is effective for retrieval of any documentation that affects the reported results. All reported data packages must be retained by the laboratory for a minimum of seven years, or longer, as dictated by project requirements. In the event of laboratory closure, all applicable documents must be transferred to the USACE Buffalo District office.

7.4 On-Site Laboratory Documentation

The onsite laboratory will maintain hard copy and electronic records as part of the project files. Reporting will be conducted through a Microsoft Access database and customized for automatic input into a GIS system for mapping. In addition, quality control records will be reported with the sample results. Data will be recorded on bench sheets, as shown on a gamma spectroscopy work card. These sheets will be filed in the on-site laboratory and scanned routinely (typically on a daily basis) and converted to electronic files (e.g., in pdf) and saved to disc or emailed to an off-site location for backup and review. Details will be established in the LQMP and associated SOPs.

7.5 Real-Time Data Management

In order to utilize the data in near real-time for feedback into the sampling design (i.e., Triad approach), a system will be implemented for uploading the data in graphical (spatial) form to be available to the field team and other data users, as discussed in FSP Section 6.1.

8. DATA ASSESSMENT AND MANAGEMENT PROCEDURES

8.1 Data QC Review

Data QC review is a systematic procedure for reviewing a body of data against a set of established criteria to provide a specified level of assurance of validity prior to its intended use. The data assessment discussed in this chapter is distinct from, and subsequent to, the laboratory's in-house review of the data prior to its release to the client (see QAPP Section 6.1).

8.2 Data Verification/ Validation

Data verification is a process of evaluating the completeness, correctness, consistency, and compliance of a data package against a standard or contract. The off-site laboratory analytical reports will be evaluated against the Comprehensive Data Package requirements, as defined in EM 200-1-3, Appendix I (USACE, 2001). The EDDs will be verified for accuracy against the laboratory data packages.

The validation of the radiological data will be performed following the general guidelines in MARLAP (2001). Validation of the chemical and conventional parameters data (including TCLP data) will be performed following the USEPA Region 2 Data Validation SOPs, augmented as necessary by the general guidelines in the DoD QSM, and the USEPA Contract Laboratory Program (CLP) National Functional Guidelines for Organic Data Review, EPA 540/R-99/008, October 1999 and USEPA CLP National Functional Guidelines for Inorganic Data Review, EPA 540-R-04-004, October 2004. The sample data (from both the on-site and off-site laboratory) will be reviewed independently (i.e., by personnel not involved in the generation of the data) for evaluation of the following:

- QC data provided in the laboratory deliverables are scientifically sound, appropriate to the method, and completely documented
- QC samples are within established guidelines
- Data were appropriately flagged by the laboratory
- Documentation of all anomalies in sample preparation and analysis is complete and correct
- Corrective action forms, if required, are complete
- Holding times and preservation are documented
- Data are ready for incorporation into the final report
- Data package (documentation and backup) is complete and ready for data archive

It is anticipated that a higher level of review (full validation) will be performed on approximately 10 percent of the radiological and definitive chemical data generated during this investigation. This higher level of review includes verification of instrument calibration, assessment of laboratory precision and accuracy based upon duplicates and spike results (including LCS, LCSD, MS, MSD, MD, and field duplicates), verification of adherence to method specifications, assessment of matrix interference, and review of raw data (e.g., instrument printouts, calibration, etc.). The independent review of data will be performed by environmental chemists to verify compliance with specified analytical methods and project-specific method quality objectives. The organization responsible for this independent review has not yet been established.

The same procedures, to the extent applicable, will be used by the on-site laboratory. Note that the near-real time use of on-site data for implementation of the Triad approach limits the degree of review that is possible prior to uploading the data for use by the project team.

8.3 Project Data Quality Objective Reconciliation

The quality of data collected during the RI must be sufficient to achieve the project DQOs listed in Table 3-1. The analytical results will first be compared to off-site background data (where applicable) in order to determine if the contamination is naturally occurring or if it is intrinsic to past MED/AEC-related activities. The analytical results will then be compared to the site-specific DQOs, ARARs, maximum contaminant levels, and TCLP criteria as part of the RI/FS.

A more detailed discussion of how the Guterl Steel RI site data will be assessed with regard to achieving site-specific DQOs is presented in Section 8.5, below.

8.4 Data Management

This section presents the data management procedures for the Guterl Steel site RI. The characterization activities planned for the RI will produce a large amount of information. The information collected is critical for several reasons. The information collected will provide the foundation for determining the nature and extent of contamination at the site, for assessing the risks at the site, and for evaluating potential remedial actions.

Project activities will generate data, including sample location, measurements of field parameters, and results of sample analysis and data reviews. Data from other sources (i.e., non direct measurements) is addressed in Section 4 of the FSP. Important records regarding the collection and analysis of the samples and data will also be generated. The data management process requires the proper flow of data from field collection and processing by the analytical laboratory to those involved in the project evaluation and decision making.

Data acquisition and management activities associated with the Guterl Steel site fall into the following broad categories:

- Field data
- On-Site laboratory data
- Off-Site laboratory data
- Mapping data (survey data from surveying subcontractor)
- Document management and retention

8.4.1 Field Data

Prior to beginning field sampling, field personnel will be trained in the project-specific field data recording requirements so that standard procedures are followed in sample collection field logbooks, chain-of-custody forms, labels, and custody seals. The primary purpose of these documents is to record each day's field activities, personnel on each sampling team, and any administrative occurrences, conditions, or activities that may have affected the field work or data quality of any environmental samples for any given day.

Each field sampling team will have a field logbook, in which it will record data collection in the field. To the extent possible, pre-printed field logbook sheets will be generated from the data

management system. If pre-printed logbook sheets are not used for a given sample, required information will be recorded manually. As samples are collected in the field, sampling team members will complete the logbooks with sample collection data and required field measurements as specified in the FSP. Standardized reporting formats will be used to document this information. The field logbooks will be signed and dated by the data recorder and will specify whether field methods and procedures were followed. Sample collection and measurements information from the logbooks and data forms will be manually entered into the electronic spreadsheets or data base and checked for accuracy. As necessary, the actual forms will be modified to include the appropriate information codes to facilitate data entry. Completed logbooks and appropriate field forms will be submitted to the project file upon completion of the project.

Sample containers will be tracked from the field collection activities to the analytical laboratory following proper COC protocols and using standardized COC forms.

Electronic data will be downloaded from field computers or system instruments frequently (e.g., at least weekly) to provide data backup in the event of computer loss or instrument failure. Hand-written data may be data entered into electronic format as needed during or after the completion of field activities. Field notes and logbooks will be managed appropriately and will be stored in the field office when not in use.

Discrete samples will be collected from soil, groundwater, surface water, sediment, and other building matrices as part of the planned RI activities. Field sampling data to be stored includes sample ID; sample station information (including location and depth or elevation, as appropriate); sample descriptions (including designations of QC samples such as field duplicates); and field screening results associated with samples.

Location information for sampling stations will be from the surveyed grid established by the surveying subcontractor prior to initiation of field sampling activities. Sampling station location data will be mapped and visually inspected for gross errors.

Field survey data also includes many types of data that are generated during the course of completing soil borings, temporary well points, and monitoring wells. It can include stratigraphic information, soil classification data, water level data, and notes recorded by staff during field activities and typically are hand-entered in field notebooks.

Building layouts and dimensions will be established to the accuracy necessary to complete the feasibility study. These measurements may be conducted by the surveying subcontractor, by field personnel (using tape measures or similar devices), or a combination of methods.

Radiological data including field gamma spectroscopy, radiation swipe count data, and field screening radiation monitoring data will be recorded in appropriate field logbooks and survey sheets. The logbooks and survey sheets will be maintained in a controlled location (field office) and will be organized in a filing system for ease of use and retrieval.

8.4.2 On-Site Laboratory Data Management

Details of the on-site laboratory procedures will be established in the LQMP and SOPs, including laboratory data reduction and review procedures and laboratory operations documentation (including archiving and retention requirements). Laboratory protocols for

verification and documentation of sample receipt are addressed in Section 4.1. Data management procedures for use in the Triad approach are summarized in QAPP Section 7.5 and detailed in FSP Section 6.1.

8.4.3 Off-Site Laboratory Data Management

The interface with the analytical laboratory is crucial for achieving the goal of generating technically sound data. Laboratory analytical methods, validation criteria, and deliverable formats are described in the laboratory QMP and in STL-St. Louis SOPs QA-0023 and PM-0004. Laboratory data reduction and review procedures are presented in Section 6.1 and laboratory operations documentation (including archiving and retention requirements) are documented in Section 7. Laboratory protocols for verification and documentation of sample receipt are addressed in Section 4.1.

8.4.4 Mapping (Survey) Data

Mapping data will consist of surveying sample points collected during the course of the RI. This data will identify discrete locations for sampling stations/monitoring wells produced as part of this characterization effort. The primary issue associated with mapping data is that of insuring the various data sets that include spatial location information are consistent relative to each other. The subcontractor or Earth Tech field representative responsible for the survey work will provide the project with electronic and hard copy reports of the civil survey data, as appropriate.

The base coordinate system for the characterization work is the New York State Plane Coordinate System (West Zone). All data produced by this characterization effort will be delivered in State Plane feet. Topographical data (i.e., mean sea level readings, depth to samples, depth to water table measurements, etc.) will be delivered in feet.

8.4.5 Data and Document Management and Tracking

To meet the regulatory requirements for the acquisition of technically sound and legally defensible data, an audit trail will be established from the development of the project SAP through the archiving of information and data. Each step or variation of the sampling and analytical process will be documented.

8.4.5.1 Data Compilation and Storage

Once the data for a given sample or group of samples are complete and entered into the appropriate electronic media, the data coordinator will check that logbooks, other field records, and all analytical data are complete and properly stored, including both the electronic form and associated data packages. Each piece of information will be documented as to its source, and hard copy information will be appropriately indexed and filed.

Any changes or corrections made to the completed data set will be documented on standardized forms which will be placed into the project file.

8.4.5.2 Data Summarization and Reporting

Project data will be screened for potential data errors, compared to site-specific background values and applicable regulatory limits, and summarized in both tabular and graphical form to

facilitate data interpretation. Data reduction and summation will be accomplished using quality-controlled and documented reporting programs. Data summaries will be generally produced using predefined report formats and approved by the USACE Project Manager.

8.4.5.3 Records Management and Document Control

Hard copies of original site and field logbooks, COC forms, data packages with analytical results and associated QA/QC information, data validation forms, and other project-related information will be indexed, catalogued into appropriate file groups and series, and archived. The project Data Manager will archive the project data to the appropriate electronic media. A data archive information package will be prepared that describes the data system, file format, and method of archive. Sufficient documentation will accompany the archived data to fully describe the source, contents, and structure of the data to provide future usability. Non-standard computer programs used to manipulate or report the archived data will also be included in the data archive information package to further enhance the future usability of the data.

8.5 Project Completeness Assessment

Project (RI) completeness will be assessed by determining if the DQOs summarized on QAPP Table Section 3.1 have been satisfactorily addressed. (Note that the list below does not include all 21 project DQOs identified in the DGAR, as some of the DQOs have been already achieved, and some will be achieved in tasks conducted subsequent to the completion of the RI.) The attainment of the RI DQOs will be assessed on an individual basis, as presented below.

DQO No. 1:

Determine the nature and extent of MED/AEC related constituents present at the site (i.e., uranium and thorium, and the media and locations in which they are present).

This DQO will be considered complete if the overall project completeness goals are met, and if no significant data gaps are noted in the RI report. Isotopic uranium data from ICP-MS analyses will be used to determine if recycled or enriched uranium may be present.

DQO No. 2:

Acquire information to define the fate and transport of contaminants from the site.

This DQO will be considered complete if the overall project completeness goals are met, especially for IA02, 03, 04, 05, 07, and 08.

DQO No. 4:

Provide sufficient characterization data to allow completion of subsequent Feasibility Study, Remedial Design, and Remedial Action.

This DQO will be considered complete if the overall project completeness goals are met, and if no significant data gaps are noted in the RI report. (See also DQO No. 1.)

DQO No. 6:

Identify the underground utility system within the site, including if possible, utilities in place at the time of AEC contracted efforts and utilities installed after the AEC contracted efforts. Includes both between building and within building utilities.

This DQO is addressed by acquisition of ‘as-built’ drawings (received December, 2005) and reviewing them; designing the field sampling program with location of these systems /utilities in mind (e.g., remote sensing); and physical inspections/observations during the field program. This DQO will be considered complete if the investigative tasks associated with this investigation (IA08) are completed. However, the completion of this task will be assessed qualitatively in that successful completion of the investigative tasks does not guarantee that all buried or hidden utilities will have been located.

DQO No. 9:

Define nature and extent of isotopic uranium and thorium in surface soils, subsurface soils, and buildings to support risk assessment (using Nuclear Regulatory Commission screening levels for human health and Department of Energy [DOE, 2002] for ecological) and development and evaluation of FS alternatives (volume determination).

This DQO will be considered complete if the overall project completeness goals are met (including isotopic uranium, thorium, and radium 226 and 228), and if no significant data gaps are noted in the RI report. (See also DQOs No. 1 and 4.)

DQO No. 10:

Determine whether groundwater has been impacted by isotopic uranium and thorium above screening levels; and if so, determine nature and extent to support risk assessment, and development and evaluation of FS alternatives.

This DQO will be considered complete if the IA07 investigation is completed as planned without significant data gaps.

DQO No. 11:

Determine whether surface water and sediments have been impacted by isotopic uranium and thorium above screening levels (screening levels for these media will need to be researched and developed during RI/FS tasks).

This DQO will be considered complete if the Sediment and Surface Water investigation (FSP Section 5.4.5.3) is completed as planned without significant data gaps.

DQO No. 13:

Determine if isotopic uranium and thorium has contaminated underground utilities.

This DQO will be considered complete if the IA08 investigation is completed as planned without significant data gaps, and the post-implementation review indicates that the techniques utilized were appropriate and achieved their goals.

DQO No. 14:

Determine the magnitude of any chemical contamination to support establishing transportation and disposal requirements (e.g., waste classification) and associated costs to be included in various Feasibility Study alternatives.

Review of historical data indicates that non-radiological constituents are unlikely to impact transportation or disposal requirements (i.e., unlikely that much, if any, of media contaminated with MED/AEC wastes will be subject to classification as hazardous waste). The pre-investigation assumption will be confirmed by successful TCLP metals analysis of a limited number of samples in process areas considered most likely to be contaminated with non-MED/AEC materials.

DQO No. 15:

Conduct an inventory of building content/structures to support FS alternatives and evaluations.

This DQO will be considered complete if sufficient information (e.g., measurements of dimensions, survey of locations, etc.) is recorded during the RI to enable locations, areas, and volumes to be established to the appropriate degree of accuracy, and if discrepancies in building dimensions noted in the DGAR are resolved.

DQO No. 19:

Gather sufficient data to complete a Baseline Human Health Risk Assessment (HHRA) for human health and an Screening Level Ecological Risk Assessment (SLERA).

Achieving this DQO will be determined subsequent to completion of the RI; and will be addressed by completing the HHRA and SLERA. For the purpose of this RI, achieving DQO No. 19 will be addressed in the planning stage (e.g., incorporating risk assessor input into the development of the SAP) and successful implantation of the project plans.

8.6 Quality Control Summary Report

At the completion of the RI and data review and validation, Earth Tech will prepare a QCSR for submission to USACE. The QCSR will address the QA/QC-related components of the following items.

- Data Collection. Deviations from the procedures identified in the QAPP in sampling procedures, sampling handling, or custody will be discussed, in addition to the potential affect on the usability of the resultant data.
- Data Analysis and Validation. The analytical method reference (including the underlying agency method for analyses performed in accordance with a laboratory-specific SOP) will be indicated, along with significant alterations or modifications to the method. Data validation approach and criteria will be discussed, as will QC results that are outside of the applicable criteria or limits. The QCSR will identify QC result deviations that are greater than typically encountered, as well as recommendations for the usability of the results generated. Copies of the data validation or data review reports or memoranda will be included as an attachment to the QCSR, as well as a narrative summary and overall assessment of the data.

- **Data Comparison.** A comparison between RI data and data generated by the USACE QA laboratory will be included, if the QA data are available in a timely manner and USACE requests this comparison to be included in the QCSR.
- **Data Summaries.** The QCSR will include a summary of qualified data and positive detections in tabular format. Data generated by both the on-site and off-site laboratories will be included in the summaries.
- **System Audits.** Any inspections, deficiencies noted, and corrective actions implemented will be summarized. USACE audit reports (if any) will be included as an attachment to the QCSR.

The QCSR will fulfill the ‘Reconciliation with User Requirements’ (as specified in USEPA QA/R-5) and ‘Project Objectives Reconciliation’ (USACE EM-200-1-3) criteria.

9. REFERENCES

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TABLES

Table 3-1
Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site
Project Data Quality Objectives and Data Needs to be Achieved in RI/FS

Project Data Quality Objective	Data Needed
1. Determine the nature and extent of MED/AEC related constituents present at the site (i.e., uranium, thorium, radium and the media and locations in which they are present).	Isotope-specific data for the COPCs in each Investigative Area. Preliminary Gamma Walkover Survey to target areas for intrusive investigation. Subsurface sampling in IAs 01, 02, 03, 04, 05, 08, and 10. Also need to establish local background conditions for COPCs.
2. Acquire information to define the fate and transport of contaminants from the site.	Same as DQO 1; also geotechnical data (soil properties – porosity, conductivity, pH, bulk density). Also requires groundwater sampling (IA 07) and surface water/sediment sampling (IA 09).
4. Provide sufficient characterization data to allow completion of subsequent Feasibility Study (FS), Remedial Design (RD), and Remedial Action (RA).	Same as DQO 2. Additional data relevant to the FS, RD, and RA to be obtained from subcontractor-generated IDW characterization data and from the ongoing NYSDEC RI/FS.
6. Identify the underground utility system within the site, including if possible, utilities in place at the time of AEC contracted efforts and utilities installed after the AEC contracted efforts. Includes both between-building and within-building utilities.	Acquire as-built utility drawings (completed; quality is low). Evaluate other geophysical and/or remote sensing methods (see FSP).
9. Define nature and extent of isotopic uranium and thorium in surface soils, subsurface soils, and buildings to support risk assessment (using Nuclear Regulatory Commission screening levels for human health and Department of Energy [DOE, 2002] for ecological) and development and evaluation of FS alternatives (volume determination).	See DQO 1 and 2, above. Review of DOE 2002 suggests that ecological risk unlikely to be a driver at Guterl. Discuss with USACE using RESRAD models (including RESRAD-BUILD) for human health risk assessment. (See also DQO 4).
10. Determine whether groundwater has been impacted by isotopic uranium, thorium, or radium above screening levels; and if so, determine nature and extent to support risk assessment, and	Additional monitoring wells to be installed; groundwater to be sampled for radiological constituents (radiological

Table 3-1
Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site
Project Data Quality Objectives and Data Needs to be Achieved in RI/FS

Project Data Quality Objective	Data Needed
development and evaluation of FS alternatives.	COPCs and gross alpha/beta radiation).
11. Determine whether surface water and sediments (IA09 and elsewhere) have been impacted by isotopic uranium, thorium, or radium above screening levels (screening levels for these media will need to be researched and developed during RI/FS tasks).	Determine, if possible, location(s) of historical outfalls to barge canal (see DQO 6). Limited sediment sampling upstream, at discharge location, and downstream for COPCs. Surface water sampling (IA09) to be conducted, but unlikely to be useful.
13. Determine if isotopic uranium, thorium, and radium has contaminated underground utilities (IA08).	Sample solids from sewers, drains, trenches (in conjunction with DQO 6). Contingency for water sampling if present.
14. Determine the magnitude of any chemical contamination to support establishing transportation and disposal requirements (e.g., waste classification) and associated costs to be included in various FS alternatives.	See DQO 4.
15. Conduct an inventory of building content/structures to support FS alternatives and evaluations.	Compile observations from structural survey and field sampling activities in IA 01 and IA 02.
19. Gather sufficient data to complete a Baseline Human Health Risk Assessment (HHRA) for human health and a screening level ecological risk assessment.	See DQOs 9 and 10 (for use in future DQOs 17 and 18).

Note: DQO numbering, as presented in the Data Gap Analysis Report (USACE, 2006), has been retained. DQOs 5, 7, 8, 12, and 16 have already been addressed. DQOs 3, 17, 18, 20, and 21 are to be addressed in tasks subsequent to the completion of the RI/FS.

Table 3-2
Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site
RI Data Collected to Achieve Project Data Quality Objectives

Data to be Collected	Intended Data Use(s)	Data Need Requirements	Sample/Analysis Methods
Surface Soils (IA01, 02, 03, 04, 05, 10) COPCs: U-234, U-235, U-238, Th-228; Th-230; Th-232; Ra-226, Ra-228	DQO 1; DQO 2; DQO 4; DQO 9; DQO 14; DQO 19.	<i>Data User Perspective</i> Site Investigation Risk Assessment FS/ Remedy selection. <i>Contaminants of Interest:</i> COPCs (U-234, U-235, U-238, Th-228, Th-230, Th-232, Ra-226, Ra-228) <i>Media of Interest:</i> Surface Soil (0-6 inches bgs) <i>Areas/Locations:</i> IA01, IA02, IA03, IA04, IA05, IA10; also background location(s) in Rollin T. Grant Wilderness Area. See FSP for details.	Discrete – Biased/Unbiased Sample – Trowel Analysis: HASL-300 for U and Th; EPA 903/904 for Ra (STL SOPs) QA/QC: Duplicates (1:20)
Subsurface Soils (IA01, 02, 03, 04, 05, 10) COPCs: U-234, U-235, U-238, Th-228; Th-230; Th-232; Ra-226, Ra-228 IDW characterization	DQO 1; DQO 2; DQO 4; DQO 9; DQO 14; DQO 19.	<i>Data User Perspective</i> Site Investigation Risk Assessment FS/ Remedy selection. <i>Contaminants of Interest:</i> COPCs (U-234, U-235, U-238, Th-228, Th-230, Th-232, Ra-226, Ra-228); Waste characteristics <i>Media of Interest:</i> Subsurface Soil (0.5 to 6 ft bgs typical) <i>Areas/Locations:</i> IA01, IA02, IA03, IA04, IA05, IA10; also background locations. See FSP for details.	Discrete – Biased/Unbiased Sample – Geoprobe Analysis: HASL-300 for U and Th; EPA 903/904 for Ra (STL SOPs) QA/QC: Duplicates (1:20) Equipment blanks (weekly) Waste characterization (e.g., EPA SW-846 methods)

Table 3-2
Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site
RI Data Collected to Achieve Project Data Quality Objectives

Data to be Collected	Intended Data Use(s)	Data Need Requirements	Sample/Analysis Methods
Building Surfaces and Floors (IA01, 02, 04) COPCs: U-234, U-235, U-238, Th-228; Th-230; Th-232; Ra-226, Ra-228 Building dimensions (length, width, height)	DQO 1; DQO 2; DQO 4; DQO 6; DQO 9; DQO 14; DQO 15; DQO 19.	<i>Data User Perspective</i> Site Investigation Risk Assessment FS/ Remedy selection. <i>Contaminants of Interest:</i> COPCs (U-234, U-235, U-238, Th-228, Th-230, Th-232, Ra-226, Ra-228) <i>Media of Interest:</i> Building walls (structural components – cinder block, brick, etc.) <i>Areas/Locations:</i> IA01, IA02, IA04. See FSP for details.	Discrete – Biased/Unbiased Sample – Hand Coring; Swipes; Geoprobe for sub-floor sampling. Analysis: U and Th by HASL-300; Ra 226/228 by EPA 903/904 (STL SOPs) Physical measurements (tape measure, etc) for dimensions. QA/QC: Duplicates (1:20)
Groundwater (IA07) COPCs: U-234, U-235, U-238, Th-228; Th-230; Th-232; Ra-226, Ra-228 Conventional parameters Geotechnical parameters on boring soils from new well installations	DQO 1; DQO 2; DQO 4; DQO 10; DQO 19.	<i>Data User Perspective</i> Site Investigation Risk Assessment FS/ Remedy selection. <i>Contaminants of Interest:</i> COPCs (U-234, U-235, U-238, Th-228, Th-230, Th-232, Ra-226, Ra-228) Gross alpha and beta radioactivity Boring soils: grain size; hydraulic conductivity; porosity; bulk density; TOC <i>Media of Interest:</i> On-site groundwater	Discrete – Biased Well installation: Hollow-stem auger (overburden); water rotary (bedrock) Development: Bailer (overburden); submersible pump (bedrock) Purging: Peristaltic pump Groundwater sampling: Low flow; Bailer (overburden); submersible pump (bedrock); filtered and unfiltered samples Analysis: Isotopic U and Th: HASL-300; Ra 226/228 by EPA 903/904 (STL SOPs).

Table 3-2
Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site
RI Data Collected to Achieve Project Data Quality Objectives

Data to be Collected	Intended Data Use(s)	Data Need Requirements	Sample/Analysis Methods
		<i>Areas/Locations:</i> Existing and new (to-be-installed) monitoring wells. See FSP for details.	Alpha and beta: EPA 900 TOC: EPA 9060 Geotech: ASTM QA/QC: Duplicates (1:20) Equipment blanks
Surface Water and Sediment (IA03, 08, 09) COPCs: U-234, U-235, U-238, Th-228; Th-230; Th-232; Ra-226, Ra-228	DQO 1; DQO 2; DQO 4; DQO 10; DQO 11; DQO 13; DQO 19.	<i>Data User Perspective</i> Site Investigation Risk Assessment FS/ Remedy selection. <i>Contaminants of Interest:</i> COPCs (U-234, U-235, U-238, Th-228, Th-230, Th-232, Ra-226, Ra-228) <i>Media of Interest:</i> Surface Water and Sediment in Landfill Area, Erie Canal. Water and solids entrained in on-site sewers, drains, and trenches. <i>Areas/Locations:</i> Landfill Area (IA 03). Barge canal transects (upstream, near outfall, downstream) – off-site (IA 09) Drains/trenches within buildings and other locations as found (IA 08). See FSP for details.	Discrete – Biased Location: Remote sensing Sampling: Hand equipment; other as needed (e.g., boat and Ponar for IA09) Analysis: U and Th: HASL-300; Ra 226/228 by EPA 903/904 (STL SOPs) QA/QC: Duplicates (1:20) Equipment blanks

Table 3-3
Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site
Field Sample and QA/QC Sample Quantity Summary and Criteria

Investigative Area	Data Use	Matrix	Sample Type	Analytical Method	Field Sample Quantity ¹²	QC Sample Qty (Duplicates)	QA Split Qty	Data Type	Precision		Lab Accuracy ¹	Sensitivity	Sampling Completeness
									Field Dup	Lab Dup			
Background	Determine Background concentrations of COPCs. Samples to be collected Rollin T. Grant Wilderness Park	Unbiased background surface and subsurface soil	Discrete	Alpha Spec ⁽⁷⁾ for U and Th isotopes; EPA 903/904 for Ra ⁽⁸⁾ Isotopic U by ICP-MS ¹³	Surface: 12 Subsurface: 12	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	RPD <40; DoD QSM generic goal is RPD ≤30.	73 – 131 % LCS Recovery; 75 – 150 % MS Recovery	1.0 pCi/g (isotope-specific) ² < 1 ug/kg (isotopic U by ICP-MS); see Table 5-1	95%
IA01	Determine nature and extent of COPCs above background in structures and soils. See FSP for building-specific details.	Unbiased and biased building materials ⁽⁹⁾ , surface soil, subsurface soil	Discrete	Gamma Spec ⁽⁶⁾ for U, Th, Ra isotopes	Surface: 17 Subsurface: 17	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	RPD <40 DoD QSM generic goal is RPD ≤30.	73 – 131 % LCS Rec; 75 – 150 % MS Rec	1.0 pCi/g (isotope-specific) ²	95%
				Alpha Spec ⁽⁷⁾ for U and Th isotopes; EPA 903/904 for Ra ⁽⁸⁾	Surface: 49 Subsurface: 49	Surface: 5% Subsurface: 5%							
				SW-846 6020B for Isotopic U by ICP-MS ¹³	Surface and Subsurface: TBD ¹³	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	MD or MSD <30 RPD <50	MS 75–125; LCS 80–120% or better	< 1 ug/kg (soil); see Table 5-1	95%
				SW-846 9060 for TOC	Surface: 2 Subsurface: 6	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	SD ⁴	ICS ⁴	100 mg/kg	95%
IA02	Determine nature and extent of COPCs in Excised Area structures and soils	Unbiased and biased, building materials ⁽⁹⁾ , surface soil, subsurface soil ⁽¹⁰⁾	Discrete	Gamma Spec ⁽⁶⁾ for U, Th, Ra isotopes	Surface: 3 Subsurface: 3	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	RPD <40	73 – 131 % LCS Rec; 75 – 150 % MS Rec	1.0 pCi/g (isotope-specific) ²	95%
				Alpha Spec ⁽⁷⁾ for U and Th isotopes; EPA 903/904 for Ra ⁽⁸⁾	Surface: 30 Subsurface: 30	Surface: 5% Subsurface: 5%							
				SW-846 6020B for Isotopic U by ICP-MS ¹³	Surface and Subsurface: TBD ¹³	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	MD or MSD <30 RPD <50	MS 75–125; LCS 80–120% or better ⁵	< 1 ug/kg (soil); see Table 5-1	95%
				SW-846 9060 for TOC	Surface: 1 Subsurface: 2	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	SD ⁴	ICS ⁴	100 mg/kg	95%

Table 3-3

**Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site**

Field Sample and QA/QC Sample Quantity Summary and Criteria

Investigative Area	Data Use	Matrix	Sample Type	Analytical Method	Field Sample Quantity ¹²	QC Sample Qty (Duplicates)	QA Split Qty	Data Type	Precision		Lab Accuracy ¹	Sensitivity	Sampling Completeness
									Field Dup	Lab Dup			
IA03	Determine presence or absence of COPCs in Landfill Area soils	Biased and unbiased, surface and subsurface soil, surface water, and sediment ⁽¹⁰⁾	Discrete	Gamma Spec ⁽⁶⁾ for U, Th, Ra isotopes	Surface: 3 Subsurface: 3 Sediment: 6	Surface: 5% Subsurface: 5% Sediment: 5%	TBD ³	Definitive	RPD <50	RPD <40 DoD QSM generic goal is RPD ≤30.	73 – 131 % LCS Rec; 75 – 150 % MS Rec	1.0 pCi/g (isotope-specific) ²	95%
				Alpha Spec ⁽⁷⁾ for U and Th isotopes; EPA 903/904 for Ra ⁽⁸⁾	Surface: 30 Subsurface: 30 Sediment: 6 Surface Water: 6	Surface: 5% Subsurface: 5% Sediment: 5% Surface Water: 5%							
				SW-846 6020B for Isotopic U by ICP-MS ¹³	Surface, Subsurface, Sediment: TBD ¹³	Surface: 5% Subsurface: 5% Sediment: 5%	TBD ³	Definitive	RPD <50	MD or MSD <30 RPD <50	MS 75–125; LCS 80–120% or better	< 1 ug/kg; see Table 5-1	95%
				SW-846 9060 for TOC	Surface: 1 Subsurface: 2 Sediment: 3	Surface: 5% Subsurface: 5% Sediment: 5%	TBD ³	Definitive	RPD <50	SD ⁴	ICS ⁴	100 mg/kg	95%
IA04	Determine nature and extent of COPCs in NCIDA soils	Biased and unbiased, building floors, surface and subsurface soil ⁽¹⁰⁾	Discrete	Gamma Spec ⁽⁶⁾ for U, Th, Ra isotopes	Surface: 12 Subsurface: 13	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	RPD <40	73 – 131 % LCS Rec; 75 – 150 % MS Rec	1.0 pCi/g (isotope-specific) ²	95%
				Alpha Spec ⁽⁷⁾ for U and Th isotopes; EPA 903/904 for Ra ⁽⁸⁾	Surface: 84 Subsurface: 84	Surface: 5% Subsurface: 5%							
				SW-846 6020B for Isotopic U by ICP-MS ¹³	Surface and Subsurface: TBD ¹³	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	MD or MSD <30 RPD <50	MS 75–125; LCS 80–120% or better	< 1 ug/kg (soil); see Table 5-1	95%
				SW-846 9060 for TOC	Surface: 1 Subsurface: 5	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	SD ⁴	ICS ⁴	100 mg/kg	95%

Table 3-3
Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site
Field Sample and QA/QC Sample Quantity Summary and Criteria

Investigative Area	Data Use	Matrix	Sample Type	Analytical Method	Field Sample Quantity ¹²	QC Sample Qty (Duplicates)	QA Split Qty	Data Type	Precision		Lab Accuracy ¹	Sensitivity	Sampling Completeness
									Field Dup	Lab Dup			
IA05	Determine presence, absence, nature, extent of COPCs in RR ROW north of Site	Biased and unbiased, surface and subsurface soil ⁽¹⁰⁾	Discrete	Gamma Spec ⁽⁶⁾ for U, Th, Ra isotopes	Surface: 4 Subsurface: 3	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	RPD <40	73 – 131 % LCS Rec; 75 – 150 % MS Rec	1.0 pCi/g (isotope-specific) ²	95%
				Alpha Spec ⁽⁷⁾ for U and Th isotopes; EPA 903/904 for Ra ⁽⁸⁾	Surface: 42 Subsurface: 42	Surface: 5% Subsurface: 5%							
				SW-846 6020B for Isotopic U by ICP-MS ¹³	Surface and Subsurface: TBD ¹³	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	MD or MSD <30 RPD	MS 75–125; LCS 80–120% or better	< 1 ug/kg (soil); see Table 5-1	95%
				SW-846 9060 for TOC	Surface: 1 Subsurface: 2	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	SD ⁴	ICS ⁴	100 mg/kg	95%
IA07	Determine presence, absence, nature, extent of COPCs in Site groundwater	Biased groundwater; one sample from each new and existing monitoring well; two rounds of 30 wells	Discrete	Alpha Spec ⁽⁶⁾ for U, Th, Ra isotopes	Unfiltered Groundwater: 60 Filtered Groundwater: 60	Groundwater: 5% (1 blank)	TBD ³	Definitive	RPD <50	RPD <40 DoD QSM generic goal is RPD ≤30.	75 – 131% LCS Recovery; 59 – 150% MS rec	1.0 pCi/g (isotope-specific) ²	95%
				EPA 9310 for gross alpha / beta	Unfiltered Groundwater: 60 Filtered Groundwater: 60	Groundwater: 5%	TBD ³	Definitive/screening	RPD <25	RPD <25; DoD QSM generic goal is RPD ≤30.	NA	5 pCi/L	95%
				EPA 160.2 for TSS	Unfiltered Groundwater: 60	Groundwater: 5%	TBD ³	Definitive	RPD <25	RPD <20	± 0.4 mg (absolute)	4 mg/L	95%

Table 3-3

**Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site**

Field Sample and QA/QC Sample Quantity Summary and Criteria

Investigative Area	Data Use	Matrix	Sample Type	Analytical Method	Field Sample Quantity ¹²	QC Sample Qty (Duplicates)	QA Split Qty	Data Type	Precision		Lab Accuracy ¹	Sensitivity	Sampling Completeness
									Field Dup	Lab Dup			
IA08	Determine presence, absence, nature, extent of COPCs in solids in site sewers, drains, trenches	Biased utility-based non-aqueous; one sample from each location where found	Discrete	Gamma Spec ⁽⁶⁾ for U, Th, Ra isotopes	Non-aqueous: 32 (Qty depends on locating utilities and presence of solids)	Non-aqueous: 5%	TBD ³	Definitive	RPD <50	RPD <40 DoD QSM generic goal is RPD ≤30.	73 – 131 % LCS Rec; 75 – 150 % MS Rec	1.0 pCi/g (isotope-specific) ²	95%
				Alpha Spec ⁽⁷⁾ for U and Th isotopes; EPA 903/904 for Ra ⁽¹¹⁾	Non-aqueous: 32 (Qty depends on locating utilities and presence of solids)								
				SW-846 6020B for Isotopic U by ICP-MS ¹³	Non-aqueous: TBD ¹³	Non-aqueous: 5%	TBD ³	Definitive	RPD <50	MD or MSD <30 RPD <50	MS 75–125; LCS 80–120% or better	< 1 ug/kg; see Table 5-1	95%
	Determine presence, absence, nature, extent of COPCs in liquids in site sewers, drains, trenches	Biased utility-based aqueous; one sample from each location where found	Discrete	Alpha Spec ⁽⁷⁾ for U and Th isotopes; EPA 903/904 for Ra ⁽⁸⁾	Aqueous: 32 (Qty depends on locating utilities and presence of liquids)	Aqueous: 5%	TBD ³	Definitive	RPD <50	RPD <40	73 – 131 % LCS Rec; 75 – 150 % MS Rec	1.0 pCi/g (isotope-specific) ²	95%
IA09	Determine presence, absence, nature, extent of COPCs in surface water and sediment in Erie Barge Canal	Unbiased surface water and sediment	Discrete	Gamma Spec ⁽⁶⁾ for U, Th, Ra isotopes	Sediment: 12	Sediment: 5%	TBD ³	Definitive	RPD <50	RPD <40; DoD QSM generic goal is RPD ≤30.	73 – 131 % LCS Rec; 75 – 150 % MS Rec	1.0 pCi/g (isotope-specific) ²	95%
				Alpha Spec ⁽⁷⁾ for U and Th isotopes; EPA 903/904 for Ra ⁽⁸⁾	Sediment: 12 Surface Water: 12	Sediment: 5% Surface Water: 5%							
				SW-846 6020B for Isotopic U by ICP-MS ¹³	Sediment: TBD ¹³	Sediment: 5%	TBD ³	Definitive	RPD <50	MD or MSD <30 RPD <50	MS 75–125; LCS 80–120% or better	< 1 ug/kg ; see Table 5-1	95%
				SW-846 9060 for TOC	Sediment: 6	Sediment: 5%	TBD ³	Definitive	RPD <50	SD ⁴	ICS ⁴	100 mg/kg	95%

Table 3-3
Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site
Field Sample and QA/QC Sample Quantity Summary and Criteria

Investigative Area	Data Use	Matrix	Sample Type	Analytical Method	Field Sample Quantity ¹²	QC Sample Qty (Duplicates)	QA Split Qty	Data Type	Precision		Lab Accuracy ¹	Sensitivity	Sampling Completeness
									Field Dup	Lab Dup			
IA10	Determine nature and extent of COPCs in Lot 4.1 ("Lombardi Property") soils	Biased and unbiased surface and subsurface soil	Discrete	Gamma Spec ⁽⁶⁾ for U, Th, Ra isotopes	Surface: 1 Subsurface: 1	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	RPD <40	73 – 131 % LCS Rec; 75 – 150 % MS Rec	1.0 pCi/g (isotope-specific) ²	95%
				Alpha Spec ⁽⁷⁾ for U and Th isotopes; EPA 903/904 for Ra ⁽⁸⁾	Surface: 12 Subsurface: 12	Surface: 5% Subsurface: 5%							
				SW-846 6020B for Isotopic U by ICP-MS ¹³	Surface and Subsurface: TBD ¹³	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	MD or MSD <30 RPD	MS 75–125; LCS 80–120% or better	< 1 ug/kg (soil); see Table 5-1	95%
				SW-846 9060 for TOC	Surface: 1 Subsurface: 1	Surface: 5% Subsurface: 5%	TBD ³	Definitive	RPD <50	SD ⁴	ICS ⁴	100 mg/kg	95%

Notes:

1. Laboratory (STL) limits are based on ongoing in-house statistics. Values shown on this table are those current as of 3/1/06. Criteria at time of analysis may vary from those shown here.
2. STL reporting limits for soil are 1.0 pCi/g for each of the target isotopes (U-234, U235, U2-238 and Th-228, 230, and 232) by alpha spectroscopy and are similar by gamma spectroscopy, and approximately 0.5 pCi/g for Ra-226 and Ra-228 (see Section 5 and Table 5-1) and 1.0 pCi/L in water for target isotopes. Reporting limits are based on short count. Improved sensitivity (by a factor of about 10) will be obtained through longer count times on a limited number of non-aqueous samples.
3. USACE has indicated that QA split samples will be submitted at 5 percent frequency, and parameters will match the analysis of the primary sample.
4. Method 9060 specifies a spike duplicate (SD) every 10 samples and independent check standard (ICS) every 15. Control limits are lab-specific but should be at 80-120% recovery and less than 20% RPD (or better).
5. DoD QSM does not have recommendation of LCS recovery for metals by ICP-MS; EPA CLP limits are 80-120% for aqueous samples but limits are not established for non-aqueous samples.
6. Gamma spec - Gamma spectroscopy by DOE HASL-300 (STL Method GA-01-R); certain COPC isotopes inferred.
7. Alpha spec - Alpha spectroscopy by DOE HASL-300 (STL Method A-01-R).
8. 100% of samples U and Th; 50% of samples Ra.
9. See FSP Table 5-8 for an estimate of building material sample quantities in IA01 and IA02. Building materials to be analyzed for isotopic U, Th, Ra by gamma and alpha spectroscopy methods.
10. Gamma walkover survey, surface scan, and swipe sample data results to be incorporated into location selection.
11. 100% of samples U and Th; 100% of samples Ra.
12. Field sample quantity is number of samples, excluding QA/QC, to be sent to the off-site laboratory for analysis.
13. Isotopic U (to include U-236) by ICP-MS performed on all background samples plus 12 samples with highest alpha spec U activity. QA/QC limits shown in "Background" row.

Table 4-1

**Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site**

Sample Bottle, Volume, Preservation, and Holding Time Summary

MATRIX/ANALYSIS	Sample Prep Method ¹	Analytical Method ¹	Sample Bottles (2)				Minimum Vol Rqd	Preservation (3)	Holding Time (3, 4)		Comment
			Mat'l	Size	Qty	Source			Extraction	Analysis	
Non-Aqueous⁵											
Uranium 234, 235, 238 (γ-spec)	STL RC-0025	STL RD-0101	P or G ⁷	8 oz ⁸	1	STL	500 g	None	NA	180 days ⁹	
Thorium-228,230,232 (γ-spec)	STL RC-0025	STL RD-0101	P or G ⁷	"	"	STL	500 g	None	NA	180 days ⁹	
Radium 226, 228 (γ-spec)	STL RC-0025	STL RD-0101	P or G ⁷	"	"	STL	500 g	None	NA	180 days ⁹	
Uranium 234, 235, 238 (α-spec)	STL RC-004 and RC-240	STL RD-0210	P or G ⁷	4 oz	1	STL	50 g	None	NA	180 days ⁹	Prep based on Eichrom Tech SOPs; sample size based on long count
Thorium-228, 230, 232 (α-spec)	STL RC-004 and RC-240	STL RD-0210	P or G ⁷	"	"	STL	50 g	None	NA	180 days ⁹	A single sample volume to be used for isotopic U and Th analysis.
Radium 226, 228 (GFP)	STL RC-004 and RC-240	STL RC-0040/0041	P or G ⁷	"	"	STL	50 g	None	NA	180 days ⁹	EPA 903/904 (modified)
Gross alpha and beta	NA	STL-RC-0020	P or G ⁷	"	"	STL	50 g	None	NA	180 days ⁹	
Uranium (isotopic)	STL IP-0002	STL MT-001 (ICP/MS-6020B)	P or G ⁷	4 oz	1	STL	30 g	None	NA	180 days	EPA SW-846 method 6020 (ICP-MS)
Total Organic Carbon	NA	SW 846 9060	P or G ⁷	4 oz	1	STL	5 g	None	NA	28 days	
Aqueous Samples (IA03,07,08,09)											
Uranium 234, 235, 238 (α-spec)	NA	STL RD-0210	P or G	1 L	1	STL	1 L	HNO ₃ to pH < 2	NA	180 days	Alpha spectroscopy
Thorium-232 (α-spec)	NA	STL RD-0210	P or G	"	"	STL	"	HNO ₃ to pH < 2	NA	180 days	Alpha spectroscopy
Radium 226, 228 (GFP)	NA	STL RC-0040/0041	P or G	1 L	1	STL	1 L	HNO ₃ to pH < 2	NA	180 days	EPA 903/904 (modified)
Gross alpha and beta	NA	STL-RC-0020	P or G	1 L	1	STL	250 mL	Acid to pH < 2	NA	180 days	Based on SW-846 9310 and EPA 900.0
Total Suspended Solids	NA	EPA 160.2	P or G	250 mL	1	STL	100 mL	NA	NA	7 days	
Geotechnical Analyses											
Grain Size (sieve/hydrometer)	NA	ASTM D 421/422	P or G	1 kg ⁽⁶⁾	1	TBD	1000 g	None	NA	NA	
Atterberg Limits	NA	ASTM D 4318	P or G	100 g	1	TBD	100 g	None	NA	NA	
Hydraulic Conductivity	NA	ASTM D 5084-03 or 5856-95	P or G	1 kg ⁽⁶⁾	1	TBD	1000 g	None	NA	NA	Remolded

(1) STL SOPs included in QAPP Attachment B

(2) Bottles as planned and provided by STL. Other materials or sizes may be acceptable.

(3) All samples for chemical analysis should be held at 4 degrees C in addition to any chemical preservation required.

(4) Holding time for calculated from day of collection, unless noted as being from time of extraction.

(5) Non-aqueous matrices include surface & subsurface soil (IA02, 03, 04, 05, 10), buildings (IA01; walls and floors), solids in sewers/drains/trenches (IA08), and sediment (IA09); non-aqueous matrices do not include swipe samples.

(6) Some geotech methods (e.g., grain size) require large sample size for accurate determination, depending on soil type. Multiple bottles may be submitted.

(7) Either plastic or glass are acceptable; STL tentatively plans to provide 4-oz or 8-oz (as noted) glass jars for this project.

(8) A single 8-oz sample is sufficient for all radiological analyses (gamma spec, alpha spec, and GFP)

(9) There is no technical holding time applicable to radionuclides by alpha or gamma spectroscopy in non-aqueous samples; the holding times shown are from STL SOPs.

G = Glass

P = plastic

EPA = Methods for the Chemical Analysis of Water and Wastes, EPA-600/4-79-020.

SW-846: Test Methods for Evaluating Solid Waste, Physical/Chemical Methods. USEPA SW-846. Complete through Update IIIB, January 2005.

α-spec = analysis by alpha spectroscopy

γ-spec = analysis by gamma spectroscopy

GFP = Gas Flow Proportional

Table 4-2
Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site
Field Sample and QA/QC Sample Off-Site Laboratory Quantity Summary

MATRIX/ANALYSIS	Analytical Method	Laboratory ¹		Reporting Limit Goal (units as specified)	Estimated Field Sample Quantity	Matrix Spike (MS) or LCS	MS Duplicate or Matrix Duplicate ⁽²⁾	Field Duplicate	Equipment Blank ⁷	USACE QA Split Samples ⁸
Non-Aqueous ³										
Uranium 234, 235, 238 (γ-spec)	STL RD-0101 (DOE GA-01-R)	STL-St L	STL-RD-0101 (DOE GA-010-R)	1.0 pCi/g (each isotope) ⁴	175	1/batch ⁵	1/batch ⁵	5%	1/week	5%
Thorium-232 (γ-spec)	STL RD-0101 (DOE GA-01-R)	STL-St L	STL RD-0101 (DOE GA-01-R)	1.0 pCi/g (each isotope) ⁴	175	1/batch ⁵	1/batch ⁵	5%	1/week	5%
Radium 226, 228 (γ-spec)	STL RC-0040, 0041 (EPA 903/904)	STL-St L	STL RC-0040, 0041 (EPA 903/904)	1.0 pCi/g (each isotope) ⁴	175	1/batch ⁵	1/batch ⁵	5%	1/week	5%
Uranium 234, 235, 238 (α-spec)	STL RD-0210 (DOE A-01-R)	STL-St L	STL RD-0210 (DOE A-01-R)	1.0 pCi/g (each isotope) ⁴	578	1/batch ⁵	1/batch ⁵	5%	1/week	5%
Thorium-228, 230, 232 (α-spec)	STL RD-0210 (DOE A-01-R)	STL-St L	STL RD-0210 (DOE A-01-R)	1.0 pCi/g (each isotope) ⁴	578	1/batch ⁵	1/batch ⁵	5%	1/week	5%
Radium 226, 228 (GFP)	STL RC-0040, 0041 (EPA 903/904)	STL-St L	STL RC-0040, 0041 (EPA 903/904)	1.0 pCi/g (each isotope) ⁴	325	1/batch ⁵	1/batch ⁵	5%	1/week	5%
Uranium (isotopic)	STL MT-001 (ICP/MS-6020B)	STL-St L	STL MT-001 (ICP/MS-6020B)	0.00002 to 0.00013 mg/kg ⁹	36	1/batch	1/batch	5%	1/week	5%
Gross alpha and beta	STL-RC-0020 (900.0/9310)	STL-St L	STL-RC-0020 (900.0/9310)	10 pCi/g (each)	36	1/batch	1/batch	5%	1/week	5%
Total Organic Carbon	SW 846 9060	STL-St L	SW 846 9060	50 mg/kg	78	1/batch	1/batch	5%	1/week	NA
Aqueous Samples (IA03,07,08,09) ¹⁰										
Uranium 234, 235, 238 (α-spec)	STL RD-0210 (DOE A-01-R)	STL-St L	STL RD-0210 (DOE A-01-R)	1 pCi/L (each isotope) ⁴	180	1/batch ⁵	1/batch ⁵	5%	1/week	5%
Thorium-228, 230, 232 (α-spec)	STL RD-0210 (DOE A-01-R)	STL-St L	STL RD-0210 (DOE A-01-R)	1 pCi/L (each isotope) ⁴	180	1/batch ⁵	1/batch ⁵	5%	1/week	5%
Radium 226, 228 (GFP)	STL-RC-0040, 0041 (EPA 903/904)	STL-St L	STL-RC-0040, 0041 (EPA 903/904)	0.5 pCi/L (each isotope) ⁴	180	1/batch ⁵	1/batch ⁵	5%	1/week	5%
Uranium (total)	STL MT-001 (ICP/MS-6020B)	STL-St L	STL MT-001 (ICP/MS-6020B)	10 ug/L	180	1/batch	1/batch	5%	1/week	5%
Gross alpha and beta	STL-RC-0020 (900.0/9310)	STL-St L	STL-RC-0020 (900.0/9310)	5 pCi/L (each)	180	1/batch ⁶	1/batch ⁶	5%	1/week	5%
Total Suspended Solids	EPA 160.2	STL-St L	EPA 160.2	5 mg/L	60	1/batch	1/batch	5%	1/week	NA
Geotechnical Analyses										
Grain Size (sieve/hydrometer)	ASTM D 421/422	TBD	ASTM D 421/422	1 percent of total	15	NA	NA	NA	NA	NA
Atterberg Limits (LL/PL/PI)	ASTM D 4318	TBD	ASTM D 4318	NA	15	NA	NA	NA	NA	NA
Hydraulic Conductivity	ASTM D 5084-03 or 5856-95	TBD	ASTM D 5084-03 or 5856-95	10 ⁻⁷ cm/sec	15	NA	NA	NA	NA	NA

(1) Laboratory information as of February, 2006.

(2) For analyses using tracer (i.e., isotopic Uranium and Thorium [except by ICP-MS]), a laboratory control sample (LCS) and duplicate (LCSD) may be substituted for MS/MSD analyses.

(3) Non-aqueous matrices include surface and subsurface soil (IA02, 03, 04, 05, 10, and background), buildings (IA01; walls and floors), solids in sewers/drains/trenches (IA08), and sediment (IA 03 and IA09); non-aqueous matrices do not include swipe samples.

(4) Alpha spectroscopy reporting limits shown are STL's limits as of February 2006 and are based on default (short count) analyses. U-235 result also includes any U-236 present in sample.

(5) Normal QC is one LCS and one laboratory duplicate for each analytical batch of 20 field samples or fewer.

(6) Normal QC for gross alpha/beta is one MS and one duplicate for each analytical batch of 20 samples or fewer.

(7) Equipment (field) blanks collected one per week.

(8) USACE has indicated that QA split samples will be analyzed at 5 percent frequency for same radiological parameters as field samples.

(9) MDLs are isotope-specific; see QAPP Table 5-1.

(10) Aqueous sample quantities include both groundwater (IA07; two rounds; filtered and unfiltered for COPCs; see FSP Table 5-11) and surface water (IA03, IA08, and IA09; see FST Table 5-12).

α-spec = analysis by alpha spectroscopy

γ-spec = analysis by gamma spectroscopy

GFP = Gas Flow Proportional

Table 5-1
Quality Assurance Project Plan
Former Guterl Specialty Steel FUSRAP Site
Minimum Detectable Concentrations for COPCs

Radionuclide/ Analyte	Method	Solid MDC ⁽¹⁾ (pCi/g)	Background Concentration (pCi/g) ⁽³⁾	Preliminary Soil Screening Level (pCi/g)	Aqueous MDC ⁽¹⁾⁽⁴⁾ (pCi/L)	Preliminary Aqueous Screening Level (pCi/L)
Gross Alpha	EPA 900/SW-846 9310	10 pCi/g ⁽¹⁴⁾	NA	NC	5	NC
Gross Beta	EPA 900/SW-846 9310	10 pCi/g ⁽¹⁴⁾	NA	NC	5	NC
Radium-226	Gas Flow Proportional	1 pCi/g	NA	0.7 ⁽¹⁰⁾	0.5 pCi/L	2.5 ⁽¹¹⁾
Radium-228	Gas Flow Proportional	1 pCi/g	NA	NA	0.5 pCi/L	2.5 ⁽¹¹⁾
Thorium-228	Alpha spec - short count ⁽⁴⁾	1 pCi/g	NA	4.7 ⁽¹⁰⁾	1 pCi/L	5.9 ⁽¹²⁾
Thorium-230	Alpha spec - short count ⁽⁴⁾	1 pCi/g	NA	1.8 ⁽¹⁰⁾	1 pCi/L	8.8 ⁽¹²⁾
Thorium-232	Alpha spec - short count ⁽⁴⁾	1 pCi/g	1.05	1.1 ⁽²⁾	1 pCi/L	1.8 ⁽¹²⁾
Uranium-234	Alpha spec - short count ⁽⁴⁾	1 pCi/g	1.75	13 ⁽²⁾	1 pCi/L	16.4 ⁽¹³⁾
Uranium-235/236	Alpha spec - short count ⁽⁴⁾	1 pCi/g	0.08	8.0 ⁽²⁾	1 pCi/L	0.475 ⁽¹³⁾
Uranium-238	Alpha spec - short count ⁽⁴⁾	1 pCi/g	1.75	14 ⁽²⁾	1 pCi/L	10.1 ⁽¹³⁾
Thorium-228	Alpha spec - long count ⁽⁵⁾	0.1 pCi/g	NA	4.7 ⁽¹⁰⁾	0.1 pCi/L	5.9 ⁽¹²⁾
Thorium-230	Alpha spec - long count ⁽⁵⁾	0.1 pCi/g	NA	1.8 ⁽¹⁰⁾	0.1 pCi/L	8.8 ⁽¹²⁾
Thorium-232	Alpha spec - long count ⁽⁵⁾	0.1 pCi/g	1.05	1.1 ⁽²⁾	0.1 pCi/L	1.8 ⁽¹²⁾
Uranium-234	Alpha spec - long count ⁽⁵⁾	0.1 pCi/g	1.75	13 ⁽²⁾	0.1 pCi/L	16.4 ⁽¹³⁾
Uranium-235/236	Alpha spec - long count ⁽⁵⁾	0.1 pCi/g	0.08	8.0 ⁽²⁾	0.1 pCi/L	0.475 ⁽¹³⁾
Uranium-238	Alpha spec - long count ⁽⁵⁾	0.1 pCi/g	1.75	14 ⁽²⁾	0.1 pCi/L	10.1 ⁽¹³⁾
Radium-226	Gamma spectroscopy	0.5 pCi/g ⁽⁶⁾	NA	0.7 ⁽¹⁰⁾	NA	2.5 ⁽¹¹⁾
Radium-228	Gamma spectroscopy	0.5 pCi/g ⁽⁷⁾	NA	NA	NA	2.5 ⁽¹¹⁾
Thorium-228	Gamma spectroscopy	Inferred ⁽⁸⁾	NA	4.7 ⁽¹⁰⁾	NA	5.9 ⁽¹²⁾
Thorium-230	Gamma spectroscopy	Inferred ⁽⁸⁾	NA	1.8 ⁽¹⁰⁾	NA	8.8 ⁽¹²⁾
Thorium-232	Gamma spectroscopy	0.5 pCi/g ⁽⁹⁾	1.05	1.1 ⁽²⁾	NA	1.8 ⁽¹²⁾
Uranium-234	Gamma spectroscopy	Inferred ⁽⁸⁾	1.75	13 ⁽²⁾	NA	16.4 ⁽¹³⁾
Uranium-235/236	Gamma spectroscopy	1 pCi/g ⁽⁹⁾	0.08	8.0 ⁽²⁾	NA	0.475 ⁽¹³⁾
Uranium-238	Gamma spectroscopy	1.5 pCi/g ⁽⁹⁾	1.75	14 ⁽²⁾	NA	10.1 ⁽¹³⁾
Uranium - 233	ICP-MS (SW-846 6020)	0.00005 mg/kg	NA	NA	NA	NA
Uranium - 234	ICP-MS (SW-846 6020)	0.00003 mg/kg	1.75	13 ⁽²⁾	NA	16.4 ⁽¹³⁾
Uranium - 235	ICP-MS (SW-846 6020)	0.00013 mg/kg	0.08	8 ⁽²⁾	NA	0.475 ⁽¹³⁾
Uranium - 236	ICP-MS (SW-846 6020)	0.00002 mg/kg	NA	NA	NA	NA
Uranium - 238	ICP-MS (SW-846 6020)	0.00011 mg/kg	1.75	14 ⁽²⁾	NA	10.1 ⁽¹³⁾

1. Minimum Detectable Concentrations (MDCs) are highly matrix-dependent and may not always be achievable. MDCs listed are for STL's alpha spectroscopy methods based on DOE HASL-300 Method A-01-R, and are current as of February 2006.
2. Preliminary Screening Level is concentration above background and determined as described in DGAR, Section 2.6.
3. Estimated background concentration inferred from ORNL (1978) as described in DGAR (USACE, 2006; Section 2.5).
4. Short count is STL default MDC and is based on 1 g sample (solids) or 1 L (water) and count time of about 3 hours.
5. Long count for improved sensitivity (to be performed on limited number of soil samples) requires 2 g sample and count time of about 7-10 hours.
6. Ra-226 inferred from Bi-214; MDC is a function of numerous sample-specific factors. STL will adjust count times to achieve required sensitivity.
7. Ra-228 inferred from Ac-228; MDC is a function of numerous sample-specific factors. STL will adjust count times to achieve required sensitivity.
8. Nuclide does not have good gamma line; activity to be inferred from alternate nuclide.
9. Gamma spec sensitivity is a function of numerous sample-specific factors. STL will adjust count times to achieve required sensitivity.
10. Screening level from Table H.2 of NUREG 1757 (2003), based on 25 mrem/yr limit. For multiple nuclides, sum of fractions rule applies.
11. Aqueous screening levels for Ra isotopes based on MCL of 5 pCi/L total Ra, assuming equal amounts of Ra 226 and 228.
12. Aqueous screening criteria for Th isotopes calculated by USACE based on dose limit of 4 mrem/yr and 2.38 L/day ingestion rate.
13. Aqueous screening criteria for U isotopes based on MCL of 30 ug/l for total U; conversion using specific activities; and EPA-recommended U-234/U-238 ratio of 1.6.
14. If needed to meet project objectives, more rigorous sample prep (total dissolution) can be performed to reduce MDC to below 5 pCi/g.

FIGURES

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FIGURE 3-1
GUTERL RI ON-SITE LABORATORY QUALITY MANAGEMENT PLAN
EXAMPLE TABLE OF CONTENTS

- 1.0 Program Description and Quality Assurance Responsibilities
 - 1.1 Quality System Policies and Objectives
 - 1.2 Organization and Management Structure
 - 1.3 Key Staff Position Descriptions
 - 1.4 Laboratory Approved Signatory
- 2.0 Data Integrity Procedures
 - 2.1 Data Integrity Training
 - 2.2 Signed Data Integrity Documentation
 - 2.3 Periodic Monitoring of Data Integrity
 - 2.4 Data Integrity Procedure Documentation
- 3.0 Training and Qualifications
 - 3.1 Responsibilities
 - 3.2 Requirements
- 4.0 Instrument Quality Control
 - 4.1 Precision
 - 4.2 Accuracy
 - 4.3 Completeness
 - 4.4 Sensitivity
- 5.0 Sample Chain-of-Custody
 - 5.1 Initiation of Sample Custody
 - 5.2 Transfer of Custody
 - 5.3 Sample Security and Transport
 - 5.4 Laboratory Sample Custody
 - 5.5 Sample Archival and Disposal
 - 5.6 Chain-of-Custody Record
- 6.0 Analytical Quality Control
 - 6.1 Responsibilities
 - 6.2 Traceability
 - 6.3 Control Charts
 - 6.4 Trend Analysis
 - 6.5 Matrix Spikes
 - 6.6 Replicates
- 7.0 Data Quality Control
 - 7.1 Responsibilities
 - 7.2 Data review
 - 7.3 Processed and Transcribed Data
 - 7.4 Data Corrections
 - 7.5 Data Record Review
 - 7.6 Data Verification, Validation, and Approval
 - 7.7 Measurement Uncertainty
 - 7.8 Reporting Data
 - 7.9 Audits and Data Review

- 8.0 Document Quality Control
 - 8.1 General
 - 8.2 Document Control and Issue
 - 8.3 Document Changes
- 9.0 Performance Assessment and Corrective Actions
 - 9.1 Responsibilities
 - 9.2 Informal Work Process Assessments
 - 9.3 Internal Quality Assessments
- 10.0 Organizational Support
 - 10.1 Review of Requests, Tenders, and Contracts
 - 10.2 Subcontracting of Environmental Tests
 - 10.3 Purchasing Services and Supplies
 - 10.4 Service to the Client
 - 10.5 Complaints
 - 10.6 Control of Nonconforming Environmental Testing Work
 - 10.7 Corrective Action
 - 10.8 Preventative Action
- 11.0 Critical Record Handling and Storage
 - 11.1 Records Retention
 - 11.2 Control and Maintenance of Documentation
 - 11.3 Records Management and Storage
- 12.0 Internal Audits
 - 12.1 Project File Reviews
 - 12.2 Management Reviews
- 13.0 References

ON-SITE LABORATORY SOPs:

- SOP 1 – Training and Qualification
- SOP 2 – Balance Quality Control
- SOP 3 – Preparation, Control, and Traceability of Standards
- SOP 4 – Control of Laboratory Logbooks
- SOP 5 – Analytical Quality Control and Sample Flow
- SOP 6 – Sample Receipt
- SOP 7 – Sample Log-In
- SOP 8 – Sample Preparation
- SOP 9 – Gamma Spectroscopy
- SOP 10 – Waste Generation Procedure
- SOP 11 – Job Hazard Analyses

ATTACHMENT A

Standard Forms to Be Used

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Figure 2
(Example Condition Upon Receipt Form)

Lot No(s) _____

(Note all associated lot No's)

Condition Upon Receipt Form
St. Louis Laboratory

Client:		COC/RFA No:		Date:	
Quote No:		Initiated By:		Time:	

Shipping Information

Shipper Name:		Multiple Packages:		Y	N	N/A
Shipper No(s):*	1.	Sample Temperature(s):**	1.			
	2.		2.			
	3.		3.			
	4.		4.			
	5.		5.			

*Numbered shipping lines correspond to Numbered Sample Temp lines.

**Sample must be received at 4°C ± 2°C-If not, note contents below.
Temperature variance does NOT affect the following analysis/matrix: Metals-Liquid
Rad tests --

Liquids or Solids.

Condition/Variance (Circle "Y" for yes, "N" for no and "N/A" for not applicable):

1.	Y N	Sample received in undamaged condition?	7.	Y N	Sample received with Chain of Custody?
2.	Y N N/A	Sample received with proper pH ¹ ? (N/A for soil samples) If NO: sample ID _____ Preservative _____ Lot _____ Date _____ Time _____ Sticker applied Y/N	8.	Y N	Chain of Custody matches sample IDs on container(s)?
3.	Y N	If N/A-Was pH taken by original STL Lab?	9.	Y N N/A	Custody seal received intact?
4.	Y N	Sample received in proper containers?	10.	Y N N/A	Custody seal tamper evident?
5.	Y N	Sample volume sufficient for analysis?	11.	Y N N/A	Custody seal on bottles intact?
6.	Y N N/A	Headspace in VOA or TOX liquid samples? (If yes, note sample ID's below)	12.	Y N N/A	Custody seal tamper evident?
¹ For DOE-AL (Pantex, LANL, Sandia) sites, verify pH of all containers received, EXCEPT VOA, TOX, and soils.			13.	Y N N/A	Was Internal COC/CUR rec'd?

Notes:

PM Notified of Short Hold samples: Y N PM Initials:

Corrective Action:

Client's Name:		Informed by:		By:	
Sample(s) processed "as is".					
Sample(s) on hold until:		If released, notify:			
Project Management Review:		Date:			

THIS FORM MUST BE COMPLETED AT THE TIME THE ITEMS ARE BEING CHECKED IN. IF ANY ITEM IS COMPLETED BY SOMEONE OTHER THAN THE INITIATOR, THEN THAT PERSON IS REQUIRED TO APPLY THEIR INITIAL AND THE DATE NEXT TO THAT ITEM.

STL-4124 (0901)
Client

Project Manager

Date

Chain of Custody Number
223734

Address

Telephone Number (Area Code)/Fax Number

Lab Number

Page _____ of _____

City

State

Zip Code

Site Contact

Lab Contact

Analysis (Attach list if more space is needed)

Project Name and Location (State)

Carrier/Waybill Number

Containers & Preservatives

Matrix

Special Instructions/
Conditions of Receipt

Contract/Purchase Order/Quote No.

Date

Time

Sample I.D. No. and Description
(Containers for each sample may be combined on one line)

Containers & Preservatives

Matrix

Analysis (Attach list if more space is needed)

Possible Hazard Identification

Non-Hazard

Flammable

Skin Irritant

Poison B

Unknown

Return To Client

Disposal By Lab

Archive For _____ Months

Sample Disposal

Turn Around Time Required

24 Hours

48 Hours

7 Days

14 Days

21 Days

Other _____

1. Relinquished By

2. Relinquished By

3. Relinquished By

Comments

Comments

LABORATORY NOTIFICATION CHECKLIST

1. Project Name/Location: _____
2. Project Plans Title, Revision Number, and Date: _____
3. Contract Number: _____
4. Data Quality Objectives (DQOs) Summary (intended use of data): _____

5. Lab Specific DQOs (Data quality indicators acceptance limits): _____

6. Name of Person to be Contacted if there are Problems with the Sample Shipment:

Phone Number: _____
FAX Number: _____
6. Name and Address of the Contract/QA Laboratories: _____
8. Project-Specific Requirements
Data Package Turn-Around Time: _____
Sample Retention Time Post-Analysis: _____
Sample Disposition Requirements: _____

MATRIX	SAMPLE NUMBERS	METHODS			REPORTING LIMITS (refer. SAP)
		PREP	CLEANUP	ANALYSIS	

9. Any Special Requirements (i.e., unusual target analytes, sample quick turnaround time (TAT)):

Figure 3-4. Laboratory notification checklist

A - E Daily Quality Control Summary Report (DQCSR)

Date: _____

Week ending: _____

USACE Project Manager: Ray Pilon

Project No.: 86184 (Earth Tech)

Contract No.: W912P4-05-D-001

Task Order No: 001

WEATHER	BRIGHT SUN ✓	CLEAR ✓	OVERCAST ✓	RAIN ✓	SNOW ✓
TEMPERATURE	TO 32 ✓	23 – 50 ✓	50 – 70 ✓	70 – 85 ✓	85 UP ✓
WIND	STILL ✓	MODERATE ✓	HIGH ✓	Report No. 000	
HUMIDITY	DRY ✓	MODERATE ✓	HUMID ✓		

[illegible]

DATE: _____

WEEK ENDING: _____

QUALITY CONTROL ACTIVITIES (INCLUDING FIELD CALIBRATIONS):
HEALTH AND SAFETY ACTIVITIES:
PROBLEMS ENCOUNTERED/CORRECTION ACTION TAKEN:
SPECIAL NOTES:
EXPECTATIONS FOR NEXT WEEK:

BY _____ TITLE _____

ATTACHMENT B

STL Laboratory SOPs

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STL St. Louis
13715 Rider Trail North
Earth City, MO 63045

Tel: 314 298 8566 Fax: 314 298 8757
www.stl-inc.com

STL ST. LOUIS STANDARD OPERATING PROCEDURE

TITLE: ACID DIGESTION OF SOILS, SW846 METHOD 3050B FOR ICP, ICP/MS

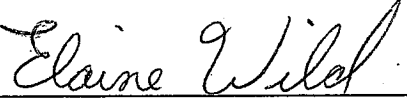
(SUPERSEDES: STL-IP-0002 REV 5)

Prepared by: _____

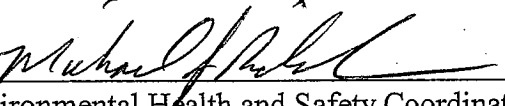
Approved by: _____


Supervisor/Lead Analyst

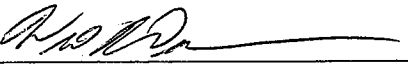
Approved by: _____


Quality Assurance Manager

Approved by: _____


Environmental Health and Safety Coordinator

Approved by: _____


Laboratory Director

Proprietary Information Statement:

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1. SCOPE AND APPLICATION

- 1.1. This procedure describes the preparation of soil samples for the analysis of metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP), and Inductively Coupled Plasma Atomic Emission/Mass Spectrometry (ICP/MS).
- 1.2. This procedure is in accordance with SW-846 Method 3050B.
- 1.3. Additional metals may be processed by this method, assuming that performance criteria of the determinative method are met.
- 1.4. This method is not a total digestion, but will dissolve almost all metals that could become "environmentally available". By design, metals bound in silicate structures are not dissolved by this procedure as they are not usually mobile in the environment. This SOP can be applied to metals in solids, sludges, wastes and sediments.
- 1.5. The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Information Management System (QuantIMS). A copy of the Structure and Analysis Code (SAC), which lists this information, is included in the respective analytical SOPs: STL-MT-0001 (ICP/MS) and STL-MT-0003 (ICP).

2. SUMMARY OF METHOD

- 2.1. A representative 0.5 gram (wet weight) portion of sample is digested in nitric acid and hydrogen peroxide. The digestate is refluxed with hydrochloric acid for ICP, ICP/MS analysis. The digestates are then diluted and filtered to 50ml/50g.

3. DEFINITIONS

- 3.1. See the STL Quality Management Plan (QMP) and STL St. Louis Laboratory Quality Manual (LQM) for a glossary of common laboratory terms and data reporting qualifiers
- 3.2. Total Metals: The concentration determined on an unfiltered sample following digestion.

4. INTERFERENCES

- 4.1. There are numerous routes by which samples may become contaminated. Potential sources of trace metals contamination include: metallic or metal-containing labware (e.g., talc gloves which contain high levels of zinc), containers, impure reagents, dirty glassware, improper sample transfers, dirty work areas, atmospheric inputs such as dirt and dust, etc. Be aware of potential sources of contamination and take appropriate measures to minimize or avoid them.
- 4.2. The entire work area, including the bench top and fume hood, should be thoroughly cleaned on a routine schedule in order to minimize the potential for environmental contamination.

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- 4.3. Boron and silica from the glassware will grow into the sample solution during and following sample processing. For critical low level determinations of boron and silica, only quartz and/or plastic labware is used.
- 4.4. Physical interference effects may contribute to inaccuracies in the determinations of trace elements. Oils, solvents and other matrices may not be digested using these methods if they are not soluble with acids. If physical interferences are present, they should be documented.
- 4.5. Visual interferences or anomalies (such as foaming, emulsions, precipitates, etc.) must be documented.
- 4.6. Specific analytical interferences are discussed in the respective analytical SOPs: STL-MT-0001 (ICP/MS) and STL-MT-0003 (ICP).

5. SAFETY

- 5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, the Waste Management SOP, and this document.

5.2. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

Hydrogen peroxide (H_2O_2) is a strong oxidizer and is corrosive. The digestion must be cooled sufficiently before the addition of H_2O_2 to avoid a reaction and possible violent effervescence, or boiling over of the digestion. A splash/splatter hazard is possible and a face shield should be worn.

5.3. PRIMARY MATERIALS USED

- 5.3.1. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

- 6.1. Hot block, capable of maintaining a temperature of 90°C +/- 5°C.
- 6.2. Thermometer, temperature range of 0-200°C.
- 6.3. Hot block digestion vessels
- 6.4. Watch glasses, ribbed or equivalent
- 6.5. Environmental Express 2.0µ nominal filter unit or equivalent.
- 6.6. Vacuum pump apparatus.
- 6.7. Analytical balance capable weighing to the nearest 0.01 grams.
- 6.8. Calibrated automatic pipettes with corresponding pipet tips
- 6.9. Plastic bottles

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7. REAGENTS AND STANDARDS

- 7.1. All standards and reagent preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002, current revision.
- 7.2. Deionized (reagent) water: Obtained by the use of a commercial ion-exchange deionizing unit which includes a polishing unit (Milli-Q System).
- 7.3. Matrix spike (MS) solutions are purchased as custom STL solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Stock standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the stock solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.
- 7.4. Working ICP, ICP/MS spike solution: The ICP, ICP/MS MS working spike solution is provided directly by the vendor, no further standard preparation is necessary.
- 7.5. ERA soil laboratory control samples (LCS)
- 7.6. Nitric acid (HNO₃), concentrated, trace metal grade
- 7.7. Hydrochloric acid (HCl), concentrated, trace metal grade
- 7.8. 30% Hydrogen peroxide (H₂O₂), reagent grade.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. STL St. Louis supplies sample containers and chemical preservatives in accordance with the method. STL St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in STL-PM-0002.
- 8.2. Samples are to be collected in plastic or glass containers.
- 8.3. All soils must be refrigerated to 4°C ± 2°C.
- 8.4. The analytical holding time for metals is 6 months.

9. QUALITY CONTROL

9.1. Batch

- 9.1.1 Definition: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is

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composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 Each analytical batch may contain up to 20 environmental samples, a method blank, and a single Laboratory Control Sample (LCS) and a Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair. In the event that there is insufficient sample to analyze an MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.
- 9.1.4 Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.2. Method Blank

- 9.2.1. Definition: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.2.2. A method blank must be prepared with every batch (20 or fewer samples of the same matrix).
- 9.2.3. Soil method blanks are prepared by taking 0.5 g of glass beads water through the procedure.

9.3. Laboratory Control Sample

- 9.3.1. Definition: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2. An LCS must be prepared with every batch.
- 9.3.3. A solid reference material containing a standard list of metal target analytes is used.

9.4. Matrix Spike/Matrix Spike Duplicate

- 9.4.1. Definition: Two aliquots of a field sample to which a known amount of target analyte(s) is added.
- 9.4.2. Additional MS/MSDs do not count towards the 20 samples in an analytical batch.
- 9.4.3. An MS/MSD is digested with every batch. If there is insufficient sample to perform an MS/MSD, a duplicate LCS is analyzed.

9.5. Procedural Variations

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- 9.5.1. Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance Memo shall be filed in the project file and incorporated into the report narrative.

9.6. Nonconformance and Corrective Action

- 9.6.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager. See SOP STL-QA-0036 for details regarding the NCM process.

10. CALIBRATION AND STANDARDIZATION

- 10.1. Hot block temperature must be verified daily for each unit used and must be recorded in the hot block temperature logbook. The temperature is verified by measuring the temperature of a vessel of reagent water placed in the hot block.
- 10.2. Instrument calibration is discussed in in the respective analytical SOPs: STL-MT-0001 (ICP/MS) and STL-MT-0003 (ICP).

11. PROCEDURE

- 11.1. Labeling vessels and bottles must be done ensure connection with the proper sample.
- 11.2. When initiating prep examine the sample to see if the sample matches the matrix designation. Contact the lab supervisor or project administrator in some cases where it appears the sample may be more appropriately processed as a liquid.
- 11.3. In some cases, both ICP/MS and ICP digests are required on each sample. One aliquot is used.
- 11.4. Preparation of Soils, Sediments and Sludges for Analysis by ICP, and ICP/MS.
 - 11.4.1. Mix sample thoroughly by stirring with a clean plastic or wooden spatula.
 - 11.4.2. For each digestion procedure, weigh a 0.5 g +/- 0.004g portion of solid and record the weight to the nearest 0.01 g. Larger sample sizes (typically 2 g) may be used if needed to meet the reporting limits.
 - 11.4.3. Measure additional aliquots of the designated samples for the MS and MSD analyses.
 - 11.4.4. Spike each of the MS and MSD aliquots with 0.5 mL of the working LCS/MS spiking solution. ICP/MS or a sample that requires both ICP and ICP/MS analysis is spiked with 0.25ml of the working solution.
 - 11.4.5. Measure 0.5 g of glass beads into a digestion vessel for the method blank.
 - 11.4.6. For the LCS, weigh 0.5 gram of ERA soil LCS +/- 0.004.

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11.4.7. For ICP without ICP/MS, add 5 mL 1:1 HNO₃ mix the sample.

11.4.8. For ICP/MS or a sample requiring ICP and ICP/MS analyses, add 2.5 mL 1:1 HNO₃.

11.4.9. Place digestion vessels in hot block and heat for 10 minutes.

11.4.9.1. **Do not allow the sample to boil or go dry during the digestion.** Allowing so may result in the loss of volatile metals. If this occurs the sample must be reprepared. Antimony is easily lost by volatilization from hydrochloric media.

11.4.10. Take samples out and allow to cool.

11.4.11. For ICP without ICP/MS, add 2.5 ml of concentrated HNO₃.

11.4.12. For ICP/MS or a sample requiring ICP and ICP/MS, add 1.25ml HNO₃.

11.4.13. Place watch glass on digestion vessels and reflux at 95°C for 30 minutes. Add reagent water as needed to ensure that the volume of solution is not reduced to less than 5 mL.

11.4.14. If brown fumes are observed, additional 2.5 mL aliquots of concentrated nitric acid until no more fumes are evolved.

11.4.15. Allow the samples to cool.

11.4.16. Add 1 mL of reagent water and 2 mL of 30 % H₂O₂. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence.

11.4.17. Replace the vapor recovery device and heat sample until effervescence subsides.

11.4.18. Allow the sample to cool.

11.4.19. Continue adding 30% H₂O₂ in 1 mL aliquots with warming until effervescence is minimal or sample appearance is unchanged.

Note: Do not add more than a total of 5 mL of 30 % H₂O₂.

11.4.20. The sample is heated for 2 hours.

11.4.21. For ICP without ICP/MS, add 5 mL of concentrated HCl and reflux for an additional 15 minutes without boiling.

11.4.22. For ICP/MS or a sample requiring ICP and ICP/MS, add 2.5 mL HCL and reflux for an additional 15 minutes without boiling.

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11.4.22.1. **Note:** Antimony and silver have poor solubility in dilute nitric acid solution. Therefore it is strongly recommended that these elements are determined by the ICP or ICP/MS procedure that includes HCl as the final digestion acid.

11.4.23. Allow the sample to cool.

11.4.24. Wash down digestion vessel walls and watchglass with reagent water.

11.4.25. Dilute sample to 50 mL with reagent water. Samples are brought up to volume in original digestion vessel.

11.4.26. Filter sample through 2.0 μ nominal filter unit. The filter is pushed through sample and remains at the bottom of the digestion vessel. The sample is now ready for analysis. In place of filtering, the samples, after dilution and mixing, may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. (The use of a vacuum pump may be required.)

12. DATA ANALYSIS AND CALCULATIONS

12.1. Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the STL St. Louis LQM. Specific calculations are included in the respective analytical SOPs: STL-MT-0001 (ICP/MS) and STL-MT-0003 (ICP).

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1. Data assessment, acceptance criteria and corrective actions are included in the respective analytical SOPs: STL-MT-0001 (ICP/MS) and STL-MT-0003 (ICP).

14. METHOD PERFORMANCE AND DEMONSTRATIONS OF CAPABILITY

14.1. Method performance data, Reporting Limits, MDLs, and QC acceptance limits, are given in the appendix to this SOP.

14.2. Method Detection Limit

14.2.1. Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. See SOP STL-QA-0016 regarding our MDL procedure.

14.3. Demonstration of Capability

14.3.1. Initial and continuing demonstrations of capability requirements are established in STL St. Louis' LQM section 5.1.2.

14.4. Training Qualification

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14.4.1. The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.4.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in STL St. Louis' LQM section 5.1.2.

14.5. Annually the analyst must successfully demonstrate proficiency to continuing to perform this analysis. See requirements in STL St. Louis' LQM section 5.1.2.

15. **VALIDATION DATA**

15.1. Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. STL ST Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16. **WASTE MANAGEMENT AND POLLUTION PREVENTION**

16.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2. Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
- Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17. **REFERENCES**

17.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 3050B.

17.2. STL Quality Management Plan (QMP), current revision

17.3. STL St. Louis Laboratory Quality Manual (LQM), current revision

17.4. STL Corporate Safety Manual and St. Louis Facility Addendum (SOP STL-HS-0002), current revisions

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17.5. Associated SOPs, current revisions

17.5.1. STL-PM-0002, Sample Receipt and Chain of Custody

17.5.2. STL-QA-0002, Standard and Reagent Preparation

17.5.3. STL-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes

17.5.4. STL-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts

17.5.5. STL-QA-0016, IDL/MDL Determination

17.5.6. STL-QA-0036, Non-conformance Memorandum (NCM) Process

17.5.7. STL-IP-0004, Labware Preparation for Inorganic and Trace Metal Analysis

17.5.8. STL-MT-0001, Analysis of Metals by Inductively Coupled Plasma/Mass Spectrometry

17.5.9. STL-MT-0003, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method for Trace Element Analysis

17.6. Modifications to reference method

17.6.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants, as defined in the determinative SOPs, are allowed up to two times the reporting limit in the blank following consultation with the client.

18. **CHANGES TO PREVIOUS REVISION**

18.1. Revised waste management and pollution prevention Section 16.

18.2. Revised SOP reference in Section 8.

18.3. Revised Section 11

18.4. Revised SOP reference in section 17

18.5. Removed references to GFAA analysis.

18.6. Added Low Level MS Spike table

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Low Level DOE Matrix Spike

ELEMENT	Stock MS Standard (mg/L)	Soil MS Level** (mg/Kg)
Arsenic	4	4
Selenium	1	1
Lead	10	10
Thallium	5	5
Antimony	10	10
Cadmium	0.5	0.5

* Levels shown indicate the spike concentration in the final digestate of the low level matrix spike based on the addition of 0.5 mL working spike to 50 mL of sample.

** Final soil spike concentration based on the addition of 0.5 mL working spike to 0.5 g of sample/50 mL final volume (assumes 100% solids).

STL Reference Data Summary

Structured Analysis Code: A-GK-MH-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: METALS, TOTAL - 2% HCL
Method: Inductively Coupled Plasma Mass Spectrometry(6020)
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List			Detection Limits			Check List 6428					Spike List 6225									
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
88	Aluminum	3.0	mg/kg	2.00	mg/kg	20050128	C	Y	6320	mg/kg	58	142	20	C	Y	100	mg/kg	75	125	30
128	Antimony	1.0	mg/kg	0.147	mg/kg	20050128	C	Y	60.9	mg/kg	10	150	20	C	Y	25	mg/kg	75	125	30
140	Arsenic	1.0	mg/kg	0.142	mg/kg	20050128	C	Y	161	mg/kg	80	120	20	C	Y	100	mg/kg	75	125	30
194	Barium	2.0	mg/kg	0.152	mg/kg	20050128	C	Y	252	mg/kg	82	118	20	C	Y	100	mg/kg	75	125	30
222	Beryllium	0.50	mg/kg	0.0310	mg/kg	20050128	C	Y	94.4	mg/kg	82	118	20	C	Y	2.5	mg/kg	75	125	30
313	Boron	5.0	mg/kg	3.200	mg/kg	20050128	C	Y	97.4	mg/kg	56	144	20	C	Y	100	mg/kg	75	125	30
411	Cadmium	0.50	mg/kg	0.0245	mg/kg	20050128	C	Y	128	mg/kg	81	119	20	C	Y	2.5	mg/kg	75	125	30
413	Calcium	50	mg/kg	2.512	mg/kg	20031223	C	Y	3320	mg/kg	79	121	20	C	Y	2500	mg/kg	75	125	30
2952	Chromium	1.0	mg/kg	0.3845	mg/kg	20050128	C	Y	69.5	mg/kg	78	121	20	C	Y	10.0	mg/kg	75	125	30
637	Cobalt	1.0	mg/kg	0.0640	mg/kg	20050128	C	Y	35.2	mg/kg	73	127	20	C	Y	25.0	mg/kg	75	125	30
643	Copper	1.0	mg/kg	0.2205	mg/kg	20050128	C	Y	148	mg/kg	82	118	20	C	Y	12.5	mg/kg	75	125	30
1539	Iron	10.0	mg/kg	1.655	mg/kg	20050128	C	Y	11200	mg/kg	57	143	20	C	Y	50.0	mg/kg	75	125	30
1605	Lead	0.30	mg/kg	0.0930	mg/kg	20050128	C	Y	142	mg/kg	80	120	20	C	Y	25.0	mg/kg	75	125	30
1618	Magnesium	50	mg/kg	1.176	mg/kg	20050128	C	Y	2040	mg/kg	77	123	20	C	Y	2500	mg/kg	75	125	30
1659	Manganese	1.0	mg/kg	0.0131	mg/kg	20050128	C	Y	408	mg/kg	80	120	20	C	Y	25.0	mg/kg	75	125	30
1906	Molybdenum	1.0	mg/kg	0.2410	mg/kg	20050128	C	Y	84.1	mg/kg	79	120	20	C	Y	100	mg/kg	75	125	30
1956	Nickel	1.0	mg/kg	0.1295	mg/kg	20050128	C	Y	147	mg/kg	82	118	20	C	Y	25.0	mg/kg	75	125	30
3924	Niobium	2.5	mg/kg	1.015	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
3925	Palladium	0.1	mg/kg	0.0765	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
2209	Platinum	0.1	mg/kg	0.0435	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
2214	Potassium	50	mg/kg	2.079	mg/kg	20050128	C	Y	1920	mg/kg	71	129	20	C	Y	2500	mg/kg	75	125	30
2281	Selenium	0.50	mg/kg	0.0770	mg/kg	20050128	C	Y	64.2	mg/kg	76	124	20	C	Y	100	mg/kg	75	125	30
2283	Silicon	50.0	mg/kg	4.278	mg/kg	20050128	C	Y	754	mg/kg	80	120	20	C	Y	500	mg/kg	75	125	30
2285	Silver	1.0	mg/kg	0.0618	mg/kg	20050128	C	Y	130	mg/kg	53	147	20	C	Y	2.5	mg/kg	75	125	30
2315	Sodium	50	mg/kg	3.972	mg/kg	20031223	C	Y	445	mg/kg	56	144	20	C	Y	2500	mg/kg	75	125	30
2353	Strontium	1.0	mg/kg	0.0735	mg/kg	20050128	C	Y	84.0	mg/kg	80	120	20	C	Y	50.0	mg/kg	75	125	30
2477	Thallium	1.0	mg/kg	0.0775	mg/kg	20050128	C	Y	84	mg/kg	76	125	20	C	Y	100	mg/kg	75	125	30
3935	Thorium	1.0	mg/kg	0.013	mg/kg	20031223	C	Y	100	mg/kg	80	120	20	C	Y	10.0	mg/kg	75	125	30
2479	Tin	1.0	mg/kg	0.1870	mg/kg	20050128	C	Y	61.0	mg/kg	58	142	20	C	Y	50.0	mg/kg	75	125	30
2482	Titanium	1.0	mg/kg	0.1175	mg/kg	20050128	C	Y	310	mg/kg	40	150	20	C	Y	50.0	mg/kg	75	125	30
2602	Tungsten	0.5	mg/kg	0.0175	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
3827	Uranium	1.0	mg/kg	0.0380	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50.0	mg/kg	75	125	30
5927	Uranium 233	0.005	mg/kg	0.00005	mg/kg	20051202	C	Y	3.86	mg/kg				C	Y	3.86	mg/kg	75	125	30
4129	Uranium 234	0.005	mg/kg	0.00003	mg/kg	20051202	C	Y	1.75	mg/kg				C	Y	1.75	mg/kg	75	125	30
4131	Uranium 235	0.005	mg/kg	0.00013	mg/kg	20051202	Y	Y	2.28	mg/kg				Y	Y	2.28	mg/kg	75	125	30
5385	Uranium 236	0.005	mg/kg	0.00002	mg/kg	20051213	Y	Y	17.5	mg/kg				Y	Y	17.5	mg/kg	75	125	30
4133	Uranium 238	0.005	mg/kg	0.00011	mg/kg	20051202	Y	Y	336	mg/kg				Y	Y	336	mg/kg	75	125	30

Structured Analysis Code: A-GK-MH-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: METALS, TOTAL - 2% HCL

Method: Inductively Coupled Plasma Mass Spectrometry(6020)

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Check List 6428			Spike List 6225		
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	LCL UCL RPD
2607	Vanadium	1.0	mg/kg	0.5535	mg/kg	C	Y	97.3	mg/kg	75 125 20
2649	Zinc	2.0	mg/kg	0.2425	mg/kg	C	Y	165	mg/kg	79 121 20
								25.0	mg/kg	75 125 30
								25.0	mg/kg	75 125 30

STL Reference Data Summary

Structured Analysis Code: A-GK-QO-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: METALS, TOTAL - 2% HCL
Method: Inductively Coupled Plasma (6010B)
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List			Detection Limits			Check List 6224					Spike List 6013								
Syn	Compound	RL	Units	MDL	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
88	Aluminum	20	mg/kg	2.854	20040209	C	N	6320	mg/kg	58	142	20	C	Y	200	mg/kg	75	125	30
128	Antimony	6	mg/kg	2.064	20041228	C	N	60.9	mg/kg	10	150	20	C	Y	50	mg/kg	75	125	30
140	Arsenic	30	mg/kg	2.776	20041228	C	N	161	mg/kg	80	120	20	C	Y	200	mg/kg	75	125	30
194	Barium	20	mg/kg	0.1508	20040520	C	N	252	mg/kg	82	118	20	C	Y	200	mg/kg	75	125	30
222	Beryllium	0.5	mg/kg	0.1119	20040209	C	N	94.4	mg/kg	82	118	20	C	Y	5	mg/kg	75	125	30
307	Bismuth	20	mg/kg	1.845	20040520								C	Y	100	mg/kg	75	125	30
313	Boron	20	mg/kg	0.4685	20040209	C	N	97.4	mg/kg	56	144	20	C	Y	200	mg/kg	75	125	30
411	Cadmium	0.5	mg/kg	0.1658	20040209	C	N	128	mg/kg	81	119	20	C	Y	5	mg/kg	75	125	30
413	Calcium	500	mg/kg	1.887	20040209	C	N	3320	mg/kg	79	121	20	C	Y	5000	mg/kg	75	125	30
2952	Chromium	1	mg/kg	0.4112	20040209	C	N	69.5	mg/kg	78	121	20	C	Y	20	mg/kg	75	125	30
637	Cobalt	5	mg/kg	0.2851	20040209	C	N	35.2	mg/kg	73	127	20	C	Y	50	mg/kg	75	125	30
643	Copper	2.5	mg/kg	0.2990	20040209	C	N	148	mg/kg	82	118	20	C	Y	25	mg/kg	75	125	30
1539	Iron	10	mg/kg	1.789	20040209	C	N	11200	mg/kg	57	143	20	C	Y	100	mg/kg	75	125	30
1605	Lead	10	mg/kg	0.1511	20040209	C	N	142	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
1616	Lithium	5	mg/kg	0.5540	20040520								C	Y	10	mg/kg	75	125	30
1618	Magnesium	500	mg/kg	10.10	20040209	C	N	2040	mg/kg	77	123	20	C	Y	5000	mg/kg	75	125	30
1659	Manganese	1.5	mg/kg	0.1962	20040209	C	N	408	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
1906	Molybdenum	4	mg/kg	0.4661	20040209	C	N	84.1	mg/kg	79	120	20	C	Y	100	mg/kg	75	125	30
1956	Nickel	4	mg/kg	0.2676	20040209	C	N	147	mg/kg	82	118	20	C	Y	50	mg/kg	75	125	30
2214	Potassium	500	mg/kg	155.5	20040520	C	N	1920	mg/kg	71	129	20	C	Y	5000	mg/kg	75	125	30
2281	Selenium	25	mg/kg	0.2259	20040209	C	N	64.2	mg/kg	76	124	20	C	Y	200	mg/kg	75	125	30
2283	Silicon	50	mg/kg	0.6032	20040209	C	N	754	mg/kg	10	150	20	C	Y	1000	mg/kg	75	125	30
2285	Silver	1	mg/kg	0.3060	20040209	C	N	130	mg/kg	53	147	20	C	Y	5	mg/kg	75	125	30
2315	Sodium	500	mg/kg	6.274	20040209	C	N	445	mg/kg	56	144	20	C	Y	5000	mg/kg	75	125	30
2353	Strontium	5	mg/kg	0.0531	20040209	C	N	84.0	mg/kg	80	120	20	C	Y	100	mg/kg	75	125	30
2477	Thallium	200	mg/kg	0.1772	20040209	C	N	84	mg/kg	76	125	20	C	Y	200	mg/kg	75	125	30
3935	Thorium	50	mg/kg	0.5257	20041228	C	N	100	mg/kg	80	120	20	C	Y	100	mg/kg	75	125	30
2479	Tin	10	mg/kg	0.468	20041228	C	N	61.0	mg/kg	58	142	20	C	Y	100	mg/kg	75	125	30
2482	Titanium	5	mg/kg	0.0580	20040209	C	N	310	mg/kg	40	150	20	C	Y	200	mg/kg	75	125	30
3827	Uranium	50	mg/kg	1.060	20040209	C	N	100	mg/kg	80	120	20	C	N	200	mg/kg	75	125	30
2607	Vanadium	2	mg/kg	0.5561	20040209	C	N	97.3	mg/kg	75	125	20	C	Y	50	mg/kg	75	125	30
2649	Zinc	2	mg/kg	0.1923	20040209	C	N	165	mg/kg	79	120	20	C	Y	50	mg/kg	75	125	30
2651	Zirconium	10	mg/kg	0.1013	20040209	C	N	100	mg/kg	80	120	20	C	Y	200	mg/kg	75	125	30

STL Reference Data Summary

Structured Analysis Code: A-GK-MH-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: METALS, TOTAL - 2% HCL
Method: Inductively Coupled Plasma Mass Spectrometry(6020)
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List			Detection Limits				Check List 6428				Spike List 6225									
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
88	Aluminum	3.0	mg/kg	2.00	mg/kg	20050128	C	Y	6320	mg/kg	58	142	20	C	Y	100	mg/kg	75	125	30
128	Antimony	1.0	mg/kg	0.147	mg/kg	20050128	C	Y	60.9	mg/kg	10	150	20	C	Y	25	mg/kg	75	125	30
140	Arsenic	1.0	mg/kg	0.142	mg/kg	20050128	C	Y	161	mg/kg	80	120	20	C	Y	100	mg/kg	75	125	30
194	Barium	2.0	mg/kg	0.152	mg/kg	20050128	C	Y	252	mg/kg	82	118	20	C	Y	100	mg/kg	75	125	30
222	Beryllium	0.50	mg/kg	0.0310	mg/kg	20050128	C	Y	94.4	mg/kg	82	118	20	C	Y	2.5	mg/kg	75	125	30
313	Boron	5.0	mg/kg	3.200	mg/kg	20050128	C	Y	97.4	mg/kg	56	144	20	C	Y	100	mg/kg	75	125	30
411	Cadmium	0.50	mg/kg	0.0245	mg/kg	20050128	C	Y	128	mg/kg	81	119	20	C	Y	2.5	mg/kg	75	125	30
413	Calcium	50	mg/kg	2.512	mg/kg	20031223	C	Y	3320	mg/kg	79	121	20	C	Y	2500	mg/kg	75	125	30
2952	Chromium	1.0	mg/kg	0.3845	mg/kg	20050128	C	Y	69.5	mg/kg	78	121	20	C	Y	10.0	mg/kg	75	125	30
637	Cobalt	1.0	mg/kg	0.0640	mg/kg	20050128	C	Y	35.2	mg/kg	73	127	20	C	Y	25.0	mg/kg	75	125	30
643	Copper	1.0	mg/kg	0.2205	mg/kg	20050128	C	Y	148	mg/kg	82	118	20	C	Y	12.5	mg/kg	75	125	30
1539	Iron	10.0	mg/kg	1.655	mg/kg	20050128	C	Y	11200	mg/kg	57	143	20	C	Y	50.0	mg/kg	75	125	30
1605	Lead	0.30	mg/kg	0.0930	mg/kg	20050128	C	Y	142	mg/kg	80	120	20	C	Y	25.0	mg/kg	75	125	30
1618	Magnesium	50	mg/kg	1.176	mg/kg	20050128	C	Y	2040	mg/kg	77	123	20	C	Y	2500	mg/kg	75	125	30
1659	Manganese	1.0	mg/kg	0.0131	mg/kg	20050128	C	Y	408	mg/kg	80	120	20	C	Y	25.0	mg/kg	75	125	30
1906	Molybdenum	1.0	mg/kg	0.2410	mg/kg	20050128	C	Y	84.1	mg/kg	79	120	20	C	Y	100	mg/kg	75	125	30
1956	Nickel	1.0	mg/kg	0.1295	mg/kg	20050128	C	Y	147	mg/kg	82	118	20	C	Y	25.0	mg/kg	75	125	30
3924	Niobium	2.5	mg/kg	1.015	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
3925	Palladium	0.1	mg/kg	0.0765	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
2209	Platinum	0.1	mg/kg	0.0435	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
2214	Potassium	50	mg/kg	2.079	mg/kg	20050128	C	Y	1920	mg/kg	71	129	20	C	Y	2500	mg/kg	75	125	30
2281	Selenium	0.50	mg/kg	0.0770	mg/kg	20050128	C	Y	64.2	mg/kg	76	124	20	C	Y	100	mg/kg	75	125	30
2283	Silicon	50.0	mg/kg	4.278	mg/kg	20050128	C	Y	754	mg/kg	80	120	20	C	Y	500	mg/kg	75	125	30
2285	Silver	1.0	mg/kg	0.0618	mg/kg	20050128	C	Y	130	mg/kg	53	147	20	C	Y	2.5	mg/kg	75	125	30
2315	Sodium	50	mg/kg	3.972	mg/kg	20031223	C	Y	445	mg/kg	56	144	20	C	Y	2500	mg/kg	75	125	30
2353	Strontium	1.0	mg/kg	0.0735	mg/kg	20050128	C	Y	84.0	mg/kg	80	120	20	C	Y	50.0	mg/kg	75	125	30
2477	Thallium	1.0	mg/kg	0.0775	mg/kg	20050128	C	Y	84	mg/kg	76	125	20	C	Y	100	mg/kg	75	125	30
3935	Thorium	1.0	mg/kg	0.013	mg/kg	20031223	C	Y	100	mg/kg	80	120	20	C	Y	10.0	mg/kg	75	125	30
2479	Tin	1.0	mg/kg	0.1870	mg/kg	20050128	C	Y	61.0	mg/kg	58	142	20	C	Y	50.0	mg/kg	75	125	30
2482	Titanium	1.0	mg/kg	0.1175	mg/kg	20050128	C	Y	310	mg/kg	40	150	20	C	Y	50.0	mg/kg	75	125	30
2602	Tungsten	0.5	mg/kg	0.0175	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
3827	Uranium	1.0	mg/kg	0.0380	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50.0	mg/kg	75	125	30
5927	Uranium 233	0.005	mg/kg	0.00005	mg/kg	20051202	C	Y						C	Y	3.86	mg/kg	75	125	30
4129	Uranium 234	0.005	mg/kg	0.00003	mg/kg	20051202	C	Y						C	Y	1.75	mg/kg	75	125	30
4131	Uranium 235	0.005	mg/kg	0.00013	mg/kg	20051202	Y	Y						Y	Y	2.28	mg/kg	75	125	30
5385	Uranium 236	0.005	mg/kg	0.00002	mg/kg	20051213	Y	Y						Y	Y	17.5	mg/kg	75	125	30
4133	Uranium 238	0.005	mg/kg	0.00011	mg/kg	20051202	Y	Y						Y	Y	336	mg/kg	75	125	30

Structured Analysis Code: A-GK-MH-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: METALS, TOTAL - 2% HCL

Method: Inductively Coupled Plasma Mass Spectrometry(6020)

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List

Syn	Compound	RL	Detection Limits		Run Date	Check List 6428		Spike List 6225	
			Units	MDL		T	A Amt	Units	LCL UCL RPD
2607	Vanadium	1.0	mg/kg	0.5535	20050128	C	Y 97.3	mg/kg	75 125 20
2649	Zinc	2.0	mg/kg	0.2425	20050128	C	Y 165	mg/kg	79 121 20

SOP No.: STL-MT-0001
Revision No.: 7
Revision Date: 12/30/05
Page: 1 of 26
Implementation Date: 01/13/06

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

**TITLE: ANALYSIS OF METALS BY INDUCTIVELY COUPLED
PLASMA/MASS SPECTROMETRY**

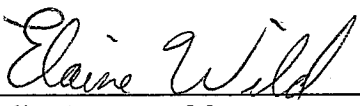
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Prepared by: _____

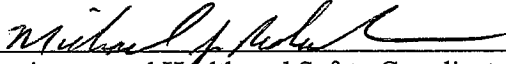
Approved by: _____


Supervisor/Lead Analyst

Approved by: _____


Quality Assurance Manager

Approved by: _____


Environmental Health and Safety Coordinator

Approved by: _____


Laboratory Director

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1. SCOPE AND APPLICATION

- 1.1. This method is applicable to the determination of metals by inductively coupled plasma mass spectrometry (ICP-MS) by EPA SW846 Method 6020A and EPA 200.8.
- 1.2. This method is applicable to drinking, surface, and saline waters; soil and waste samples.
- 1.3. The aqueous sample digestion procedure is found in SOP: STL-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8) and the soil sample digestion procedure is found in SOP: STL-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, ICP/MS.
- 1.4. The analysis time varies depending on the number of analytes and the memory characteristics of those analytes quantitated in an analysis run.
- 1.5. The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Information Management System (QuantIMS). A copy of the Structure and Analysis Code (SAC), which lists this information, is included in the appendix of this SOP.

2. SUMMARY OF METHOD

- 2.1. Aqueous samples, digestates or leachates are nebulized into a spray chamber where a stream of argon carries the sample aerosol through a quartz torch and injects it into a radio frequency plasma. There the sample is decomposed and desolvated. The ions produced are entrained in the plasma gas and by means of a water-cooled, differentially pumped interface, introduced into a high-vacuum chamber that houses a quadrupole mass spectrometer. The ions are sorted according to their mass-to-charge ratio and measured with a channel electron multiplier.

3. DEFINITIONS

- 3.1. See the STL Quality Management Plan (QMP) and STL St. Louis Laboratory Quality Manual (LQM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2. EPA and SW methodology use different terminology. Our SOP references the SW 846 terminology:
 - 3.2.1. The ICV satisfies the QCS requirements found in method 200.8.
 - 3.2.2. The LCS satisfies the requirements of the LFB found in method 200.8.
- 3.3. Dissolved Metals: Those elements which pass through a 0.45 um membrane filter. (Sample is acidified after filtration)
- 3.4. Suspended Metals: Those elements retained by a 0.45 um filter.
- 3.5. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.
- 3.6. CRI: a low level Continuing Calibration Verification Standard of the analyte of interest. CRI concentration is set at approximately the reporting limit and is run immediately following the ICV/ICB.
- 3.7. ICSA/ICSAB: Interference Check Analysis used to validate the interelement correction factors.

4. INTERFERENCES

- 4.1. Isobaric elemental interferences: Isobaric elemental interferences associated with naturally occurring isotopes are automatically corrected by the instrument software.
- 4.2. Isobaric molecular interferences: Corrections for molecular interferences will be applied where appropriate based on known or suspected interferences.
- 4.3. Common molecular ion interferences are listed in Table 1 of this SOP.
- 4.4. Matrix interferences: Internal standards will be used to correct for some matrix interferences.
 - 4.4.1. Internal standards should be added at a level to give approximately 100,000 - 2,000,000 counts of raw signal intensity. The mass of the internal standard used should ideally be within ± 50 amu of the mass of the affected analyte.

4.4.2. Severe matrix effects will be monitored by comparing the internal standard intensity in the sample to the internal standard intensity of the initial calibration blank.

5. SAFETY

5.1. Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, the Waste Management SOP, and this document.

5.2. SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1. None

5.3. PRIMARY MATERIALS USED

5.4. The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

5.5.

6. EQUIPMENT AND SUPPLIES

- 6.1. Perkin Elmer/Sciex ELAN 6100 ICP-MS or equivalent.
- 6.2. Argon gas: High-purity grade (99.99%)
- 6.3. Cool-flow or appropriate water cooling device
- 6.4. Peristaltic Pump
- 6.5. Calibrated automatic pipettes or Class A glass volumetric pipettes
- 6.6. Class A volumetric flasks
- 6.7. Autosampler

7. REAGENTS AND STANDARDS

- 7.1. All standards and reagent preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002, current revision.
- 7.2. Reagents and standards used in sample preparation are found in the sample preparation SOPs, STL-IP-0002 (soils) and STL-IP-0013 (waters).
- 7.3. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks. Reagent water must be shown to have a resistivity greater than or equal to 16.67 Mohm-cm.
- 7.4. Nitric Acid, Optima Grade
- 7.5. Hydrochloric Acid, Trace Metals grade
- 7.6. Internal Standard Solution: Prepare internal standards (Li, Sc, Ge, In, Ho, Y, Rh, Tb, Bi) at 100 ppb concentration when needed.
- 7.7. Intermediate standards are purchased as custom multi-element mixes or as single-element solutions. All standards must be stored in FEP fluorocarbon or unused polyethylene or polypropylene bottles.
- 7.8. Working calibration and calibration verification solutions may be used for up to 1 week and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 1% hydrochloric and 2% nitric acid.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. STL St. Louis supplies sample containers and chemical preservatives in accordance with the method. STL St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in STL-PM-0002.
- 8.2. Sample holding times for metals are six months from time of collection to the time of analysis.
- 8.3. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron and/or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis. For samples analyzed by Method 200.8 for compliance with Safe Drinking Water regulations, the samples must be held for a minimum of 16 hours prior to verifying the pH.
- 8.4. All soils must be refrigerated to $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$.
- 8.5. Aqueous samples for total metals must be digested before analysis using an appropriate digestion procedure, STL-IP-0013.
- 8.6. Soil or waste samples are digested before analysis using an appropriate digestion procedure. Method 3050B of SW846 is the appropriate digestion procedure, STL-IP-0002.

9. QUALITY CONTROL

- 9.1. Batch
 - 9.1.1. Definition: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
 - 9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.

- 9.1.3. Each analytical batch may contain up to 20 environmental samples, a method blank, and a single Laboratory Control Sample (LCS) and a Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair. In the event that there is insufficient sample to analyze an MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed. Certain client projects, replace the MSD requirement with a sample Duplicate. If only these client project samples are in a batch, then a MSD is not performed. If the batch contains routine samples in addition to these client project samples, then a MSD and a sample duplicate are performed.
- 9.1.4. Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.
- 9.2. Method Blank
 - 9.2.1. Definition: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.2.2. A method blank must be prepared with every batch (20 or fewer samples of the same matrix).
 - 9.2.3. A method blank must be included with each batch of samples. The matrix for aqueous is reagent (DI) water. Soil method blanks are glass beads.
 - 9.2.4. For dissolved metals samples that have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.
- 9.3. Laboratory Control Sample
 - 9.3.1. Definition: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
 - 9.3.2. An LCS must be prepared with every batch.
 - 9.3.3. The water LCS is reagent (DI) water fortified with trace metal analytes of interest. The solid LCS is a commercially prepared purchased solid reference material containing a standard list of common trace metals.
- 9.4. Matrix Spike/Matrix Spike Duplicate
 - 9.4.1. Definition: Two aliquots of a field sample to which a known amount of target analyte(s) is added.
 - 9.4.2. Additional MS/MSDs do not count towards the 20 samples in an analytical batch.
 - 9.4.3. An MS/MSD can be digested with every extraction batch, although it is not a method requirement. If there is insufficient sample to perform an MS/MSD, a duplicate LCS is analyzed.
- 9.5. Serial Dilution
 - 9.5.1. Definition: A dilution test is performed to determine whether significant physical or chemical interferences exist due to the sample matrix.
 - 9.5.2. The test is performed by running a sample at a 5x (1:4) dilution.
 - 9.5.3. Samples identified as field blanks cannot be used for dilution tests.
 - 9.5.4. The serial dilution results shall agree within $\pm 10\%$ of the undiluted sample results, if the undiluted sample results and the serial dilution result are both above the reporting limit.
- 9.6. Post Digestion Spike (PDS)

- 9.6.1. Definition: A post digestion spike is a sample which has been fortified with target analytes of interest after the digestion process.
- 9.6.2. The laboratory requires the analysis of a serial dilution for all batches and thus does not perform the intermediate post digestion spike QC step.
- 9.6.3. The method stipulates that a PDS be performed on the sample chosen for MS/MSD and if the PDS fails to proceed to performing a serial dilution on the sample. If the PDS is acceptable, the laboratory is not required to perform a serial dilution. Since the laboratory has elected to perform the serial dilution routinely, the outcome of the PDS is not critical. There is no qualification made to the data based on the performance of the PDS.
- 9.6.4. For client project or programs requiring a PDS, the laboratory will include a PDS in the batch in addition to the serial dilution. This requirement is noted by the Project Manager in the client requirement sheet and/or client summary report.
 - 9.6.4.1. If a PDS is performed, the acceptance criteria is 75%-125%, with a spike concentration between 10-100 times the MDL, UNLESS, the project/program criteria is given.
- 9.7. Method of Standard Addition (MSA)
 - 9.7.1. Definition: This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift.
 - 9.7.2. MSA are not required by the method.
 - 9.7.3. MSAs are not considered normal batch QC and if required by the client, must appear on the client requirement sheet or client summary report.
- 9.8. Linear Range Verification (LR)
 - 9.8.1. Definition: The linear range is determined bi-annually for each element on the standard list, using the 10% criteria.
 - 9.8.2. Standards must be run at increasing concentration until elements are no longer within 10% of true value. The last concentration where the element was within 10 % of true value is considered the upper linear range.
- 9.9. Procedural Variations
 - 9.9.1. Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance Memo shall be filed in the project file and incorporated into the report narrative.
- 9.10. Nonconformance and Corrective Action
 - 9.10.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager. See SOP STL-QA-0036 for details regarding the NCM process.
10. CALIBRATION AND STANDARDIZATION/ INSTRUMENT CONDITIONS
 - 10.1. It is recommended that the instrument be run under the following conditions:

PARAMETER	Recommended Conditions
Neb Flow	0.86 – 1.0L/min
RF Power	950 – 1400 watts
Lens Volatage	4 – 11 volts
Analog Stage Volatage	(-1500 to -2500 volts)
Pulse Stage Voltage	700 – 1300 volts
Number of replicates	3
Flush Time	60 seconds
Wash Time	60 seconds
Default Method Name	EPA ICP
Default Auto Sampler Table	SW846

10.2. Instrument start-up

- 10.2.1. Follow the instrument start-up procedure outlined in the Perkin Elmer Elan 6100 Operator's Manual.
- 10.2.2. (Cone Conditioning) Aspirating a 25% tap water solution for at least 1 hour can enhance instrument performance. This procedure should only be used after a thorough cleaning of the interface cones or the installation of new cones takes place.

10.3. Instrument Tuning

- 10.3.1. Aspirate a 10 ppb tuning solution containing all of the tuning elements. The typical tuning elements are Be, Co, In, Pb, Mg, Ba, Ce and Rh. The instrument manufacturer monitors Mg, Rh, Pb, Ce and He for instrument performance.
- 10.3.2. Mass calibration and resolution checks must be documented and included as part of the raw data package.
 - 10.3.2.1. Resolution must be < 0.9 amu at 10% peak height for the tuning elements of interest. The recommended resolution for the Elan 6100 is around 0.7 amu.
 - 10.3.2.2. Mass calibration must be within ± 0.1 amu from the actual value for the tuning elements of interest or the mass calibration must be adjusted.
 - 10.3.2.3. Using the Tuning Solution, an Auto-lens calibration should be performed to ensure that optimum voltages are being applied to the Auto-lens. The default calibration should range from 4-10 volts in .25 volt increments.
 - 10.3.2.4. A "daily" performance check must be performed. This uses the same tuning solution as above. The tuning elements must have RSDs below 5%. Mg must be at or above 30,000 counts. Pb must be at or above 100,000 counts. Rh must be at or above 150,000 counts and the oxides/polyatomic ions must be below 3.0%. The blank must be less than or equal to 30 counts. If any of these conditions are not met repairs or optimization procedures must be performed until these specifications are met.
 - 10.3.2.5. A dual-detector calibration must be performed daily.

10.4. Initial Calibration

10.4.1. Initial Calibration Sequence

Instrument Calibration
 Cal BLANK
 CAL 1 – CAL 5
 ICV (QCSTD1)
 ICB (QCSTD2)
 CRI (QCSTD3)

ICSAB (QCSTD4)

ICSA (QCSTD5) includes longer rinse after reading (240min)

- 10.4.2. Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all calibration verification (CCV) and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.
 - 10.4.2.1. Instrument calibration consists of a minimum of 3 standards plus a method blank.
 - 10.4.2.2. The coefficient of determination (R^2) for all regression curve must be equal to or greater than 0.995.
 - 10.4.2.3. If the calibration curve does not meet method requirements, the system is evaluated to determine if the failure is due to instrument malfunction or standard preparation. Corrective action is to correct the malfunction or prepare new standards and recalibrate the instrument.
- 10.5. CRI - is a low level CCV, sometimes referenced as the Low-Level Standard (LLS) of the analyte of interest. The CRI concentration is set at approximately the reporting limit and is analyzed immediately following the initial ICV/ICB.
 - 10.5.1. Since the EPA has not set acceptance limits for the CRI, STL has set CRI recovery limits at $\pm 50\%$. However, if the native sample concentration is 10x or greater than the reporting limit and the LCS passes, the sample results will be acceptable due to it's higher range.
 - 10.5.2. Failure to meet this criteria may result in reanalysis and require that the system be evaluated and checked for error.
- 10.6. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). The ICV must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within \pm the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified.
 - 10.6.1. The internal standard intensity in the ICV/CCV and ICB/CCB should be within 20% of the IS intensity in the instrument standardization solution. If not, the analyst should check for any instrument anomalies and continue if none are noted.
- 10.7. Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Custom multielement ICS solutions must be used. All analytes should be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it should be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix.
 - 10.7.1. The ICSA and ICSAB solutions must be run daily at the beginning of the run or every 12 hours, whichever is more frequent.
 - 10.7.2. ICSA results for the non-interfering elements with RLs $< 10 \mu\text{g/L}$ must fall within $\pm 2x$ RL from zero. ICSA results for the non-interfering elements with RLs $> 10 \mu\text{g/L}$ must fall within $\pm 1x$ RL from zero.
 - 10.7.3. ICSAB results must be within 80 - 120% recovery.
 - 10.7.4.
- 10.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV may be a second source or the same source as the calibration. The CCV must fall within 10% of the true value for that solution. A CCB is analyzed immediately following each CCV. The CCB result must fall within \pm

RL from zero. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the CCV or CCB may be reanalyzed once to and accepted if there is a reason for the initial out-of-control event such as carryover from a high concentration sample. Otherwise, if the CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed.

10.9. Closing Calibration

10.9.1. Sequence

Instrument Calibration

ICV

ICB

ICSA

ICSAB

CRI

CCV

CCB

7 samples (analysis runs)

CCV

CCB

10 samples (repeat every 10 instrument runs)

CCV

CCB

10 samples (CCV/CCB pairs as required to complete run)

CCV

CCB

End

11. PROCEDURE

- 11.1. The aqueous sample digestion procedure is found in SOP: STL-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8)
- 11.2. The soil sample digestion procedure is found in SOP: STL-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, ICP/MS.
- 11.3. The mass ions used for determination of the element of interest is given in Table 2 of this SOP.
- 11.4. The Internal Standard recovery should not fall outside 30%-150% (6020A) and 60-125% (200.8) of the IS recoveries obtained in the first calibration standard of each analytical run. If this criteria is not met, the sample should be diluted and re-analyzed until the IS recoveries are within specified limits. If the upper control limit is exceeded for 200.8, the analyst should review the data for the presence of the out-of-control internal standard in the native sample. Narrate any findings.

12. DATA ANALYSIS AND CALCULATIONS

- 12.1. Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the STL St. Louis LQM.
- 12.2. Appropriate factors must be applied to sample values if dilutions are performed.
- 12.3. Sample results should be reported with up to three significant figures in accordance with the significant figure policy.

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1. The data assessment and corrective action process is detailed through the Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: STL-QA-0036. A hardcopy of all the data assessment types and descriptions along with their associated corrective actions is included in the SOP. Below is a subset of the data assessment and QC excursion types within Clouseau; the text in underline is the exact "type" line in Clouseau. For a complete and current listing, please access the software program.
- 13.2. Method Blank
 - 13.2.1. Acceptance Criteria:
 - 13.2.1.1. No target analytes may be present in the method blank above the reporting limit.
 - 13.2.2. Corrective Action for Method Blanks not meeting acceptance criteria:
 - 13.2.2.1. Method Blank Contamination – See Clouseau NCM for corrective action.
- 13.3. Laboratory Control Sample (LCS)
 - 13.3.1. Acceptance Criteria:
 - 13.3.1.1. All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD).
 - 13.3.2. Corrective Action for LCS not meeting acceptance criteria:
 - 13.3.2.1. LCS Spike Recovery excursion (high) – See Clouseau NCM for corrective action.
 - 13.3.2.2. LCS Spike Recovery excursion (low) – See Clouseau NCM for corrective action.
 - 13.3.2.3. RPD excursion for MS/MSD or LCS/LCSD – See Clouseau NCM for corrective action.
- 13.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD)
 - 13.4.1. All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).
 - 13.4.2. Corrective Action for MS/MSD not meeting acceptance criteria:
 - 13.4.2.1. MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. See Clouseau NCM to determine if re-preparation re-analysis is required.
- 13.5. Post-digestion Spike
 - 13.5.1. A post digestion spike should be performed on one sample per prep batch. Spike recovery results that fall outside of the 75-125% recovery window will be narrated in an NCM.
- 13.6. Sample result evaluation
 - 13.6.1. Dilutions
 - 13.6.1.1. If the sample is causing interference due to matrix, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. A NCM is not required if the dilution was performed to bring the result within the linear dynamic range of the instrument.
 - 13.6.1.1.1. Dilution: Sample– See Clouseau NCM for corrective action.
 - 13.6.1.1.2. Dilution: Spike(s) diluted out– See Clouseau NCM for corrective action.
 - 13.6.2. Carryover
 - 13.6.2.1. When a sample has a high response for a compound, there is a real possibility that some of the sample may carry over into the sample analyzed immediately afterward.
 - 13.6.2.2. If a sample analyzed after a sample with high concentrations has negative results, carryover did not occur.

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- 13.6.2.3. If a sample analyzed after a sample with high concentrations has positive results for the same analytes, the results are questionable. This sample must be reanalyzed under conditions in which carryover can be confirmed to not have occurred.

13.7. Insufficient Sample

- 13.7.1. For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis and narrative comment stating such is included in the report narrative. The insufficient sample description is included in the Clouseau NCM within the type defining the excursion.

14. METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1. Method performance data, Reporting Limits, MDLs, and QC acceptance limits, are given in the appendix to this SOP.
- 14.2. Method Detection Limits
- 14.2.1. Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. See SOP STL-QA-0016 regarding our MDL procedure.
- 14.3. Instrument Detection Limits
- 14.3.1. Instrument detection limits are analyzed quarterly using a reagent water solution. Seven replicate analyses are performed on three non-consecutive days, analyzing each replicate as a routine sample. The IDL is calculated by averaging the standard deviations of each of the three runs.
- 14.4. Demonstration of Capability
- 14.4.1. Initial and continuing demonstrations of capability requirements are established in STL St. Louis' LQM section 5.1.2.
- 14.5. Training Qualification
- 14.5.1. The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 14.5.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in STL St. Louis' LQM section 5.1.2.
- 14.5.3. Annually the analyst must successfully demonstrate proficiency to continuing to perform this analysis. See requirements in STL St. Louis' LQM section 5.1.2.

15. VALIDATION DATA

- 15.1. Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. STL ST Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16. WASTE MANAGEMENT AND POLLUTION PREVENTION

- 16.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 16.2. The following waste streams are produced when this method is carried out.

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- Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."
- Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17. REFERENCES

- 17.1. EPA Method 6020A SW846.
- 17.2. Perkin Elmer/Sciex ELAN model 6000 Users Manual
- 17.3. EPA Method 200.8
- 17.4. STL Quality Management Plan (QMP), current revision
- 17.5. STL St. Louis Laboratory Quality Manual (LQM), current revision
- 17.6. STL Corporate Safety Manual and St. Louis Facility Addendum (SOP STL-HS-0002), current revisions.
- 17.7. Associated SOPs, current revisions
 - 17.7.1. STL-IP-0002, Acid Digestion of Soils, SW846 Method 3050B for ICP, ICP/MS, and GFAA
 - 17.7.2. STL-IP-0013, Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy, and ICP/MS (Method 3010A, EPA 200.7 and EPA 200.8)
 - 17.7.3. STL-QA-0002, Standard and Reagent Preparation
 - 17.7.4. STL-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes.
 - 17.7.5. STL-PM-0002, Sample Receipt and Chain of Custody
 - 17.7.6. STL-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.7.7. STL-QA-0016, IDL/MDL Determination
 - 17.7.8. STL-QA-0036, Non-conformance Memorandum (NCM) Process
- 17.8. Clarifications and Modification to the Reference Method
 - 17.8.1. The post spike is not performed per batch. Internal standards are used to monitor matrix interferences in all samples. Post spikes will be done per specific QAPP or program requirements. Post-spikes using analytes other than the internal standards may be used if an analyst encounters a new or unusual matrix.
 - 17.8.2. This SOP may be used to analyze for elements not included in Method 6020A and 200.8, as long as appropriate QC samples spiked with the non-routine analytes are run with acceptable results.
 - 17.8.3. An ICB/CCB is acceptable if the result is <RL. Method 6020A states that the results of the calibration blank (CCB) are to be less than 3x the IDL. If not, terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. The intent of this requirement is to ensure that the calibration is not drifting at the low end. STL St. Louis has adopted an absolute control limit of +/- RL from zero for calibration blank criteria.

18. CHANGES FROM PREVIOUS REVISION

- 18.1. Grade of Hydrochloric acid changed in section 7
- 18.2. SOP reference (Sample Receipt and Chain of Custody) changed in sections 8 and 17.
- 18.3. Run Conditions and run sequence revised in section 10.

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- 18.4. Section 11: changed Internal Standard criteria for method 6020
- 18.5. Section 16 revised.

Table 1
COMMON MOLECULAR ION INTERFERENCES IN ICP-MS
BACKGROUND MOLECULAR IONS

Molecular Ion	Mass	Element Interferences*
NH ⁺	15	
OH ⁺	17	
OH ₂ ⁺	18	
C ₂ ⁺	24	
CN ⁺	26	
CO ⁺	28	
N ₂ ⁺	28	
N ₂ H ⁺	29	
NO ⁺	30	
NOH ⁺	31	
O ₂ ⁺	32	
O ₂ H ⁺	33	
³⁶ ArH ⁺	37	
³⁸ ArH ⁺	39	
⁴⁰ ArH ⁺	41	
CO ₂ ⁺	44	
CO ₂ H ⁺	45	Sc
ArC ⁺ , ArO ⁺	52	Cr
ArN ⁺	54	Cr
ArNH ⁺	55	Mn
ArO ⁺	56	
ArOH ⁺	57	
⁴⁰ Ar ³⁶ Ar ⁺	76	Se
⁴⁰ Ar ³⁸ Ar ⁺	78	Se
⁴⁰ Ar ₂ ⁺	80	Se

* Method elements or internal standards affected by the molecular ions.

Table 1 cont'd
MATRIX MOLECULAR IONS

CHLORIDE

Molecular Ion	Mass	Element Interference
$^{35}\text{ClO}^+$	51	V
$^{35}\text{ClOH}^+$	52	Cr
$^{37}\text{ClO}^+$	53	Cr
$^{37}\text{ClOH}^+$	54	Cr
Ar $^{35}\text{Cl}^+$	75	As
Ar $^{37}\text{Cl}^+$	77	Se

SULFATE

Molecular Ion	Mass	Element Interference
$^{32}\text{SO}^+$	48	
$^{32}\text{SOH}^+$	49	
$^{34}\text{SO}^+$	50	V, Cr
$^{34}\text{SOH}^+$	51	V
$\text{SO}_2^+, \text{S}_2^+$	64	Zn
Ar $^{32}\text{S}^+$	72	
Ar $^{34}\text{S}^+$	74	

PHOSPHATE

Molecular Ion	Mass	Element Interference
PO^+	47	
POH^+	48	
PO_2^+	63	Cu
ArP $^+$	71	

GROUP I, II METALS

Molecular Ion	Mass	Element Interference
ArNa $^+$	63	Cu
ArK $^+$	79	
ArCa $^+$	80	

MATRIX OXIDES*

Molecular Ion	Mass	Element Interference
TiO	62-66	Ni, Cu, Zn
ZrO	106-112	Ag, Cd
MoO	108-116	Cd

* Oxide interferences will normally be very small and will only impact the method elements when present at relatively high concentrations. Some examples of matrix oxides are listed of which the analyst should be aware. It is recommended that Ti and Zr isotopes are monitored in solid waste samples, which are likely to contain high levels of these elements. Mo is monitored as a method analyte.

Table 2
ANALYTICAL ISOTOPES

Isotope	Element of Interest
<u>27</u>	Aluminum
123	Antimony
<u>75</u>	Arsenic
137, <u>135</u>	Barium
<u>9</u>	Beryllium
<u>10</u>	Boron
111	Cadmium
44	Calcium
52	Chromium
59	Cobalt
65	Copper
57	Iron
208	Lead
24	Magnesium
55	Manganese
98, <u>97</u>	Molybdenum
60	Nickel
39	Potassium
82	Selenium
107	Silver
23	Sodium
88	Strontium
203, <u>205</u>	Thallium
232	Thorium
<u>118</u> , 120	Tin
47	Titanium
238	Uranium
51	Vanadium
<u>66</u> , 67	Zinc
99	Technecium
133	Cesium
104, 105	Palladium
194, 195	Platinum
185	Rhenium
28	Silicon
182, 183	Tungsten
233, 234, 235, 236, 238	Uranium
93	Niobium

Mass Ion number “ ” (underlined) indicates primary ion

STL Reference Data Summary

Structured Analysis Code: I-GJ-MH-01-06		Matrix: WATER
Target Analyte List: All Analytes		Extraction: METALS, TOTAL - 2% HCL
		Method: Inductively Coupled Plasma Mass Spectrometry(6020)
		QC Program: STANDARD TEST SET
		Location: STL St. Louis

Analyte List		Detection Limits				Check List 6224				Spike List 6225			
		RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD
Syn	Compound												
88	Aluminum	30	ug/L	8.509	ug/L	20050128	C	Y	500	ug/L	85	115	20
128	Antimony	5	ug/L	0.609	ug/L	20050128	C	Y	500	ug/L	85	115	20
140	Arsenic	10	ug/L	1.809	ug/L	20050128	C	Y	500	ug/L	85	115	20
194	Barium	5	ug/L	0.600	ug/L	20050128	C	Y	500	ug/L	85	115	20
222	Beryllium	1	ug/L	0.1256	ug/L	20050128	C	Y	500	ug/L	85	115	20
313	Boron	50	ug/L	5.50	ug/L	20050128	C	Y	1000	ug/L	85	115	20
411	Cadmium	0.5	ug/L	0.0672	ug/L	20050128	C	Y	500	ug/L	85	115	20
413	Calcium	500	ug/L	100	ug/L	20050128	C	Y	10000	ug/L	85	115	20
5935	Cesium 133	0.5	ug/L	0.00282	ug/L	20051128	C	Y	500	ug/L	85	115	20
2952	Chromium	10	ug/L	3.7	ug/L	20050128	C	Y	500	ug/L	85	115	20
637	Cobalt	5	ug/L	0.519	ug/L	20050128	C	Y	500	ug/L	85	115	20
643	Copper	5	ug/L	0.719	ug/L	20050128	C	Y	500	ug/L	85	115	20
1539	Iron	50	ug/L	7.254	ug/L	20050128	C	Y	500	ug/L	85	115	20
1605	Lead	3	ug/L	0.5652	ug/L	20050128	C	Y	500	ug/L	85	115	20
1618	Magnesium	100	ug/L	13	ug/L	20050128	C	Y	10000	ug/L	85	115	20
1659	Manganese	5	ug/L	0.544	ug/L	20050128	C	Y	500	ug/L	85	115	20
1906	Molybdenum	5	ug/L	0.627	ug/L	20050128	C	Y	500	ug/L	85	115	20
1956	Nickel	5	ug/L	1.150	ug/L	20050128	C	Y	500	ug/L	85	115	20
3924	Niobium	25	ug/L	7.643	ug/L	20050128	C	Y	1000	ug/L	85	115	20
3925	Palladium	1	ug/L	0.2258	ug/L	20050128	C	Y	1000	ug/L	85	115	20
2209	Platinum	1	ug/L	0.1	ug/L	20050128	C	Y	1000	ug/L	85	115	20
2214	Potassium	100	ug/L	18	ug/L	20050128	C	Y	10000	ug/L	85	115	20
3927	Rhenium	1	ug/L	0.214	ug/L	20050127							
5936	Ruthenium 101	0.5	ug/L	0.00155	ug/L	20051128							
2281	Selenium	5	ug/L	0.570	ug/L	20050128	C	Y	500	ug/L	85	115	20
2283	Silicon	250	ug/L	25	ug/L	20050128	C	Y	1000	ug/L	85	115	20
2285	Silver	5	ug/L	1.5	ug/L	20050128	C	Y	125	ug/L	85	115	20
2315	Sodium	100	ug/L	18.94	ug/L	20050128	C	Y	10000	ug/L	85	115	20
2353	Strontium	5	ug/L	0.5338	ug/L	20050128	C	Y	500	ug/L	85	115	20
4113	Technetium 99	0.5	ug/L	0.00050	ug/L	20051118							
2477	Thallium	2	ug/L	0.2198	ug/L	20050128	C	Y	500	ug/L	85	115	20
3935	Thorium	5	ug/L	0.2512	ug/L	20050128	C	Y	1000	ug/L	85	115	20
2479	Tin	10	ug/L	6.2	ug/L	20050128	C	Y	500	ug/L	85	115	20
2482	Titanium	10	ug/L	0.7	ug/L	20050128	C	Y	1000	ug/L	85	115	20
2602	Tungsten	5	ug/L	0.884	ug/L	20050128	C	Y	1000	ug/L	85	115	20
3827	Uranium	1	ug/L	0.1256	ug/L	20050128	C	Y	1000	ug/L	85	115	20

Structured Analysis Code: I-GJ-MH-01-06

Target Analyte List: All Analytes

Matrix: WATER

Extraction: METALS, TOTAL - 2% HCL

Method: Inductively Coupled Plasma Mass Spectrometry(6020)

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List			Detection Limits			Check List 6224						Spike List 6225								
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5927	Uranium 233	0.05	ug/L	0.0007	ug/L	20051201	C	Y	0.386	ug/L	85	115	20	C	Y	0.386	ug/L	75	125	20
4129	Uranium 234	0.05	ug/L	0.0004	ug/L	20050923	C	Y	0.175	ug/L	85	115	20	C	Y	0.175	ug/L	75	125	20
4131	Uranium 235	0.05	ug/L	0.0023	ug/L	20050923	C	Y	0.228	ug/L	85	115	20	C	Y	0.228	ug/L	75	125	20
5385	Uranium 236	0.05	ug/L	0.00012	ug/L	20051213	C	Y	1.75	ug/L	85	115	20	C	Y	1.75	ug/L	75	125	20
4133	Uranium 238	0.05	ug/L	0.0014	ug/L	20050923	C	Y	33.6	ug/L	85	115	20	C	Y	33.6	ug/L	75	125	20
2607	Vanadium	10	ug/L	1.627	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	250	ug/L	75	125	20
2649	Zinc	10	ug/L	7.3	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	250	ug/L	75	125	20

STL Reference Data Summary

Structured Analysis Code: I-JX-MH-01-06		Matrix: WATER
Target Analyte List: All Analytes		Extraction: METALS, FILTERED 2% HCL, DISSOLVED
		Method: Inductively Coupled Plasma Mass Spectrometry(6020)
		QC Program: STANDARD TEST SET
		Location: STL St. Louis

Analyte List		Detection Limits				Check List 6224				Spike List 6225			
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD
88	Aluminum	30	ug/L	8.509	ug/L	20050128	C	Y	500	ug/L	85	115	20
128	Antimony	5	ug/L	0.609	ug/L	20050128	C	Y	500	ug/L	85	115	20
140	Arsenic	10	ug/L	1.809	ug/L	20050128	C	Y	500	ug/L	85	115	20
194	Barium	5	ug/L	0.600	ug/L	20050128	C	Y	500	ug/L	85	115	20
222	Beryllium	1	ug/L	0.1256	ug/L	20050128	C	Y	500	ug/L	85	115	20
313	Boron	50	ug/L	5.50	ug/L	20050128	C	Y	1000	ug/L	85	115	20
411	Cadmium	0.5	ug/L	0.0672	ug/L	20050128	C	Y	500	ug/L	85	115	20
413	Calcium	500	ug/L	100	ug/L	20050128	C	Y	10000	ug/L	85	115	20
2952	Chromium	10	ug/L	3.7	ug/L	20050128	C	Y	500	ug/L	85	115	20
637	Cobalt	5	ug/L	0.519	ug/L	20050128	C	Y	500	ug/L	85	115	20
643	Copper	5	ug/L	0.719	ug/L	20050128	C	Y	500	ug/L	85	115	20
1539	Iron	50	ug/L	7.254	ug/L	20050128	C	Y	500	ug/L	85	115	20
1605	Lead	3	ug/L	0.5652	ug/L	20050128	C	Y	500	ug/L	85	115	20
1618	Magnesium	100	ug/L	13	ug/L	20050128	C	Y	10000	ug/L	85	115	20
1659	Manganese	5	ug/L	0.544	ug/L	20050128	C	Y	500	ug/L	85	115	20
1906	Molybdenum	5	ug/L	0.627	ug/L	20050128	C	Y	500	ug/L	85	115	20
1956	Nickel	5	ug/L	1.150	ug/L	20050128	C	Y	500	ug/L	85	115	20
3924	Niobium	25	ug/L	7.643	ug/L	20050128	C	Y	1000	ug/L	85	115	20
3925	Palladium	1	ug/L	0.2258	ug/L	20050128	C	Y	1000	ug/L	85	115	20
2209	Platinum	1	ug/L	0.1	ug/L	20050128	C	Y	1000	ug/L	85	115	20
2214	Potassium	100	ug/L	18	ug/L	20050128	C	Y	10000	ug/L	85	115	20
3927	Rhenium	1	ug/L	0.214	ug/L	20050127	C	Y	500	ug/L	85	115	20
2281	Selenium	5	ug/L	0.570	ug/L	20050128	C	Y	1000	ug/L	85	115	20
2283	Silicon	250	ug/L	25	ug/L	20050128	C	Y	125	ug/L	85	115	20
2285	Silver	5	ug/L	1.5	ug/L	20050128	C	Y	10000	ug/L	85	115	20
2315	Sodium	100	ug/L	18.94	ug/L	20050128	C	Y	500	ug/L	85	115	20
2353	Strontium	5	ug/L	0.5338	ug/L	20050128	C	Y	500	ug/L	85	115	20
2477	Thallium	2	ug/L	0.2198	ug/L	20050128	C	Y	500	ug/L	85	115	20
3935	Thorium	5	ug/L	0.2512	ug/L	20050128	C	Y	1000	ug/L	85	115	20
2479	Tin	10	ug/L	6.2	ug/L	20050128	C	Y	500	ug/L	85	115	20
2482	Titanium	10	ug/L	0.7	ug/L	20050128	C	Y	1000	ug/L	85	115	20
2602	Tungsten	5	ug/L	0.884	ug/L	20050128	C	Y	1000	ug/L	85	115	20
3827	Uranium	1	ug/L	0.1256	ug/L	20050128	C	Y	1000	ug/L	85	115	20
2607	Vanadium	10	ug/L	1.627	ug/L	20050128	C	Y	500	ug/L	85	115	20
2649	Zinc	10	ug/L	7.3	ug/L	20050128	C	Y	500	ug/L	85	115	20
2651	Zirconium	100	ug/L	0.3330	ug/L	20040520	C	Y	1000	ug/L	85	115	20

STL Reference Data Summary

Structured Analysis Code: A-GK-MH-01-06		Matrix: SOLID
Target Analyte List: All Analytes		Extraction: METALS, TOTAL - 2% HCL
		Method: Inductively Coupled Plasma Mass Spectrometry(6020)
		QC Program: STANDARD TEST SET
		Location: STL St. Louis

Analyte List			Detection Limits			Check List 6428					Spike List 6225									
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
88	Aluminum	3.0	mg/kg	2.00	mg/kg	20050128	C	Y	6320	mg/kg	58	142	20	C	Y	100	mg/kg	75	125	30
128	Antimony	1.0	mg/kg	0.147	mg/kg	20050128	C	Y	60.9	mg/kg	10	150	20	C	Y	25	mg/kg	75	125	30
140	Arsenic	1.0	mg/kg	0.142	mg/kg	20050128	C	Y	161	mg/kg	80	120	20	C	Y	100	mg/kg	75	125	30
194	Barium	2.0	mg/kg	0.152	mg/kg	20050128	C	Y	252	mg/kg	82	118	20	C	Y	100	mg/kg	75	125	30
222	Beryllium	0.50	mg/kg	0.0310	mg/kg	20050128	C	Y	94.4	mg/kg	82	118	20	C	Y	2.5	mg/kg	75	125	30
313	Boron	5.0	mg/kg	3.200	mg/kg	20050128	C	Y	97.4	mg/kg	56	144	20	C	Y	100	mg/kg	75	125	30
411	Cadmium	0.50	mg/kg	0.0245	mg/kg	20050128	C	Y	128	mg/kg	81	119	20	C	Y	2.5	mg/kg	75	125	30
413	Calcium	50	mg/kg	2.512	mg/kg	20031223	C	Y	3320	mg/kg	79	121	20	C	Y	2500	mg/kg	75	125	30
2952	Chromium	1.0	mg/kg	0.3845	mg/kg	20050128	C	Y	69.5	mg/kg	78	121	20	C	Y	10.0	mg/kg	75	125	30
637	Cobalt	1.0	mg/kg	0.0640	mg/kg	20050128	C	Y	35.2	mg/kg	73	127	20	C	Y	25.0	mg/kg	75	125	30
643	Copper	1.0	mg/kg	0.2205	mg/kg	20050128	C	Y	148	mg/kg	82	118	20	C	Y	12.5	mg/kg	75	125	30
1539	Iron	10.0	mg/kg	1.655	mg/kg	20050128	C	Y	11200	mg/kg	57	143	20	C	Y	50.0	mg/kg	75	125	30
1605	Lead	0.30	mg/kg	0.0930	mg/kg	20050128	C	Y	142	mg/kg	80	120	20	C	Y	25.0	mg/kg	75	125	30
1618	Magnesium	50	mg/kg	1.176	mg/kg	20050128	C	Y	2040	mg/kg	77	123	20	C	Y	2500	mg/kg	75	125	30
1659	Manganese	1.0	mg/kg	0.0131	mg/kg	20050128	C	Y	408	mg/kg	80	120	20	C	Y	25.0	mg/kg	75	125	30
1906	Molybdenum	1.0	mg/kg	0.2410	mg/kg	20050128	C	Y	84.1	mg/kg	79	120	20	C	Y	100	mg/kg	75	125	30
1956	Nickel	1.0	mg/kg	0.1295	mg/kg	20050128	C	Y	147	mg/kg	82	118	20	C	Y	25.0	mg/kg	75	125	30
3924	Niobium	2.5	mg/kg	1.015	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
3925	Palladium	0.1	mg/kg	0.0765	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
22209	Platinum	0.1	mg/kg	0.0435	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
22214	Potassium	50	mg/kg	2.079	mg/kg	20050128	C	Y	1920	mg/kg	71	129	20	C	Y	2500	mg/kg	75	125	30
22281	Selenium	0.50	mg/kg	0.0770	mg/kg	20050128	C	Y	64.2	mg/kg	76	124	20	C	Y	100	mg/kg	75	125	30
22283	Silicon	50.0	mg/kg	4.278	mg/kg	20050128	C	Y	754	mg/kg	80	120	20	C	Y	500	mg/kg	75	125	30
22285	Silver	1.0	mg/kg	0.0618	mg/kg	20050128	C	Y	130	mg/kg	53	147	20	C	Y	2.5	mg/kg	75	125	30
2315	Sodium	50	mg/kg	3.972	mg/kg	20031223	C	Y	445	mg/kg	56	144	20	C	Y	2500	mg/kg	75	125	30
2353	Strontium	1.0	mg/kg	0.0735	mg/kg	20050128	C	Y	84.0	mg/kg	80	120	20	C	Y	50.0	mg/kg	75	125	30
2477	Thallium	1.0	mg/kg	0.0775	mg/kg	20050128	C	Y	84	mg/kg	76	125	20	C	Y	100	mg/kg	75	125	30
3935	Thorium	1.0	mg/kg	0.013	mg/kg	20031223	C	Y	100	mg/kg	80	120	20	C	Y	10.0	mg/kg	75	125	30
2479	Tin	1.0	mg/kg	0.1870	mg/kg	20050128	C	Y	61.0	mg/kg	58	142	20	C	Y	50.0	mg/kg	75	125	30
2482	Titanium	1.0	mg/kg	0.1175	mg/kg	20050128	C	Y	310	mg/kg	40	150	20	C	Y	50.0	mg/kg	75	125	30
2602	Tungsten	0.5	mg/kg	0.0175	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
3827	Uranium	1.0	mg/kg	0.0380	mg/kg	20050128	C	Y	100	mg/kg	80	120	20	C	Y	50.0	mg/kg	75	125	30
5927	Uranium 233	0.005	mg/kg	0.00005	mg/kg	20051202	C	Y	3.86	mg/kg	80	120	20	C	Y	3.86	mg/kg	75	125	30
4129	Uranium 234	0.005	mg/kg	0.00003	mg/kg	20051202	C	Y	1.75	mg/kg	80	120	20	C	Y	1.75	mg/kg	75	125	30
4131	Uranium 235	0.005	mg/kg	0.00013	mg/kg	20051202	C	Y	2.28	mg/kg	80	120	20	C	Y	2.28	mg/kg	75	125	30
5385	Uranium 236	0.005	mg/kg	0.00002	mg/kg	20051213	C	Y	17.5	mg/kg	80	120	20	C	Y	17.5	mg/kg	75	125	30

Target Analyte List: All Analytes

Extraction: METALS, TOTAL - 2% HCL

Method: Inductively Coupled Plasma Mass Spectrometry(6020)

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Check List 6428						Spike List 6225									
		RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
	Compound																			
	4133 Uranium 238	0.005	mg/kg	0.00011	mg/kg	20051202	C	Y	336	mg/kg	80	120	20	C	Y	336	mg/kg	75	125	30
	2607 Vanadium	1.0	mg/kg	0.5535	mg/kg	20050128	C	Y	97.3	mg/kg	75	125	20	C	Y	25.0	mg/kg	75	125	30
	2649 Zinc	2.0	mg/kg	0.2425	mg/kg	20050128	C	Y	165	mg/kg	79	121	20	C	Y	25.0	mg/kg	75	125	30

STL Reference Data Summary

Structured Analysis Code: A-JV-MH-01-06		Matrix: SOLID
Target Analyte List: All Analytes	Extraction: SPLP-E -> LOW LEVEL, 2% HCL	
	Method: Inductively Coupled Plasma Mass Spectrometry(6020)	
	QC Program: STANDARD TEST SET	
		Location: STL St. Louis

Analyte List		Detection Limits				Check List 6332				Spike List 6225			
		RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD
Syn	Compound												
88	Aluminum	30	ug/L	8.509	ug/L	20050128	C	Y	6360	mg/kg	57.8	142.	20
128	Antimony	10	ug/L	0.609	ug/L	20050128	C	Y	65.2	mg/kg	1.53	222.	20
140	Arsenic	10	ug/L	1.809	ug/L	20050128	C	Y	110	mg/kg	79.7	120	20
194	Barium	20	ug/L	0.600	ug/L	20050128	C	Y	334	mg/kg	82.0	117.	20
222	Beryllium	5	ug/L	0.1256	ug/L	20050128	C	Y	133	mg/kg	81.9	118.	20
313	Boron	50	ug/L	5.50	ug/L	20050128	C	Y	59.1	mg/kg	41.6	158.	20
411	Cadmium	5	ug/L	0.0672	ug/L	20050128	C	Y	101	mg/kg	81.4	118.	20
413	Calcium	500	ug/L	100	ug/L	20050128	C	Y	3320	mg/kg	79.2	120.	20
2952	Chromium	10	ug/L	3.7	ug/L	20050128	C	Y	167	mg/kg	78.4	121.	20
637	Cobalt	10	ug/L	0.519	ug/L	20050128	C	Y	136	mg/kg	81.6	118.	20
643	Copper	10	ug/L	0.719	ug/L	20050128	C	Y	118	mg/kg	82.2	117.	20
1539	Iron	100	ug/L	7.254	ug/L	20050128	C	Y	11400	mg/kg	57.3	142.	20
1605	Lead	3	ug/L	0.5652	ug/L	20050128	C	Y	102	mg/kg	80.5	119.	20
1618	Magnesium	500	ug/L	13	ug/L	20050128	C	Y	1980	mg/kg	77.2	122.	20
1659	Manganese	10	ug/L	0.544	ug/L	20050128	C	Y	534	mg/kg	79.9	120.	20
1906	Molybdenum	10	ug/L	0.627	ug/L	20050128	C	Y	45.5	mg/kg	79.3	120.	20
1956	Nickel	10	ug/L	1.150	ug/L	20050128	C	Y	127	mg/kg	81.8	118.	20
3924	Niobium	25	ug/L	7.643	ug/L	20050128	C	Y	50	mg/kg	80	120	20
3925	Palladium	1	ug/L	0.2258	ug/L	20050128	C	Y	50	mg/kg	80	120	20
2209	Platinum	1	ug/L	0.1	ug/L	20050128	C	Y	50	mg/kg	80	120	20
2214	Potassium	500	ug/L	18	ug/L	20050128	C	Y	1930	mg/kg	71.5	128.	20
2281	Selenium	5	ug/L	0.570	ug/L	20050128	C	Y	166	mg/kg	75.3	124.	20
2283	Silicon	500	ug/L	25	ug/L	20050128	C	Y	200	mg/kg	80	120	20
2285	Silver	10	ug/L	1.5	ug/L	20050128	C	Y	82.9	mg/kg	61.2	138.	20
2315	Sodium	500	ug/L	18.94	ug/L	20050128	C	Y	452	mg/kg	55.5	144.	20
2353	Strontium	10	ug/L	0.5338	ug/L	20050128	C	Y	74.3	mg/kg	79.8	120.	20
2477	Thallium	10	ug/L	0.2198	ug/L	20050128	C	Y	152	mg/kg	105.	196.	20
3935	Thorium	10	ug/L	0.2512	ug/L	20050128	C	Y	100	mg/kg	80	120	20
2479	Tin	10	ug/L	6.2	ug/L	20050128	C	Y	230	mg/kg	69.5	130	20
2482	Titanium	10	ug/L	0.7	ug/L	20050128	C	Y	299	mg/kg	39.4	160.	20
2602	Tungsten	5	ug/L	0.884	ug/L	20050128	C	Y	50	mg/kg	80	120	20
3827	Uranium	10	ug/L	0.1256	ug/L	20050128	C	Y	200	mg/kg	80	120	20
2607	Vanadium	10	ug/L	1.627	ug/L	20050128	C	Y	118	mg/kg	74.7	125.	20
2649	Zinc	20	ug/L	7.3	ug/L	20050128	C	Y	193	mg/kg	79.2	120.	20
2651	Zirconium	100	ug/L	0.3330	ug/L	20040520	C	Y	100	mg/kg	80	120	20

Structured Analysis Code: A-M6-MH-01-06

Target Analyte List: All Analytes

Matrix:	SOLID
Extraction:	KD leach/2% HCl 3010
Method:	Inductively Coupled Plasma
QC Program:	STANDARD TEST SET
Location:	STL St. Louis

ge number 1

STL Reference Data Summary

Structured Analysis Code: A-3E-MH-01-06		Matrix: SOLID
Target Analyte List: All Analytes		Extraction: LEACHATE, DI (ASTM D3987-85)-18 hour > Digestion/Ino
		Method: Inductively Coupled Plasma Mass Spectrometry(6020)
		QC Program: STANDARD TEST SET
		Location: STL St. Louis

Analyte List			Detection Limits			Check List 6224					Spike List 6225									
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
88	Aluminum	30	ug/L	8.509	ug/L	20040210	C	N	6320	mg/kg	58	142	20	C	Y	100	mg/kg	75	125	30
128	Antimony	10	ug/L	0.6088	ug/L	20040520	C	N	60.9	mg/kg	10	150	20	C	Y	25	mg/kg	75	125	30
140	Arsenic	10	ug/L	0.2826	ug/L	20040210	C	N	161	mg/kg	80	120	20	C	Y	100	mg/kg	75	125	30
194	Barium	20	ug/L	0.0314	ug/L	20040210	C	N	252	mg/kg	82	118	20	C	Y	100	mg/kg	75	125	30
222	Beryllium	5	ug/L	0.1256	ug/L	20040210	C	N	94.4	mg/kg	82	118	20	C	Y	2.5	mg/kg	75	125	30
313	Boron	50	ug/L	5.5	ug/L	20040210	C	N	97.4	mg/kg	56	144	20	C	Y	100	mg/kg	75	125	30
411	Cadmium	5	ug/L	0.5966	ug/L	20040210	C	N	128	mg/kg	81	119	20	C	Y	2.5	mg/kg	75	125	30
413	Calcium	500	ug/L	17.45	ug/L	20040210	C	N	3320	mg/kg	79	121	20	C	Y	2500	mg/kg	75	125	30
2952	Chromium	10	ug/L	0.8792	ug/L	20040210	C	N	69.5	mg/kg	78	121	20	C	Y	10.0	mg/kg	75	125	30
637	Cobalt	10	ug/L	0.0114	ug/L	20040210	C	N	35.2	mg/kg	73	127	20	C	Y	25.0	mg/kg	75	125	30
643	Copper	10	ug/L	0.1884	ug/L	20040210	C	N	148	mg/kg	82	118	20	C	Y	12.5	mg/kg	75	125	30
1539	Iron	100	ug/L	3.3912	ug/L	20040210	C	N	11200	mg/kg	57	143	20	C	Y	50.0	mg/kg	75	125	30
1605	Lead	3	ug/L	0.5652	ug/L	20040210	C	N	142	mg/kg	80	120	20	C	Y	25.0	mg/kg	75	125	30
1618	Magnesium	500	ug/L	1.099	ug/L	20040210	C	N	2040	mg/kg	77	123	20	C	Y	2500	mg/kg	75	125	30
1659	Manganese	10	ug/L	0.0942	ug/L	20040210	C	N	408	mg/kg	80	120	20	C	Y	25.0	mg/kg	75	125	30
1906	Molybdenum	10	ug/L	0.1256	ug/L	20040210	C	N	84.1	mg/kg	79	120	20	C	Y	100	mg/kg	75	125	30
1956	Nickel	10	ug/L	0.0628	ug/L	20040210	C	N	147	mg/kg	82	118	20	C	Y	25.0	mg/kg	75	125	30
3924	Niobium	25	ug/L	7.6430	ug/L	20040412	C	N	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
3925	Palladium	1	ug/L	0.2258	ug/L	20040412	C	N	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
2209	Platinum	1	ug/L	0.0495	ug/L	20040412	C	N	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
2214	Potassium	500	ug/L	6.374	ug/L	20040210	C	N	1920	mg/kg	71	129	20	C	Y	2500	mg/kg	75	125	30
2281	Selenium	5	ug/L	0.5652	ug/L	20040210	C	N	64.2	mg/kg	76	124	20	C	Y	100	mg/kg	75	125	30
2283	Silicon	500	ug/L	5.0	ug/L	20040520	C	N	754	mg/kg	10	150	20	C	Y	500	mg/kg	75	125	30
2285	Silver	10	ug/L	0.0228	ug/L	20040210	C	N	130	mg/kg	53	147	20	C	Y	2.5	mg/kg	75	125	30
2315	Sodium	500	ug/L	3.203	ug/L	20040210	C	N	445	mg/kg	56	144	20	C	Y	2500	mg/kg	75	125	30
2353	Strontium	10	ug/L	0.5338	ug/L	20040210	C	N	84.0	mg/kg	80	120	20	C	Y	50.0	mg/kg	75	125	30
2477	Thallium	10	ug/L	0.2198	ug/L	20040210	C	N	84	mg/kg	76	125	20	C	Y	100	mg/kg	75	125	30
3935	Thorium	10	ug/L	0.2512	ug/L	20040210	C	N	100	mg/kg	80	120	20	C	Y	10.0	mg/kg	75	125	30
2479	Tin	10	ug/L	0.1884	ug/L	20040210	C	N	61.0	mg/kg	58	142	20	C	Y	50.0	mg/kg	75	125	30
2482	Titanium	10	ug/L	0.157	ug/L	20040210	C	N	310	mg/kg	40	150	20	C	Y	50.0	mg/kg	75	125	30
2602	Tungsten	5	ug/L	0.2403	ug/L	20040412	C	N	100	mg/kg	80	120	20	C	Y	50	mg/kg	75	125	30
3827	Uranium	10	ug/L	0.1256	ug/L	20040210	C	N	100	mg/kg	80	120	20	C	Y	50.0	mg/kg	75	125	30
2607	Vanadium	10	ug/L	0.7222	ug/L	20040210	C	N	97.3	mg/kg	75	125	20	C	Y	25.0	mg/kg	75	125	30
2649	Zinc	20	ug/L	0.8943	ug/L	20040520	C	N	165	mg/kg	79	120	20	C	Y	25.0	mg/kg	75	125	30
2651	Zirconium	100	ug/L	0.3330	ug/L	20040520	C	N	100	mg/kg	80	120	20	C	Y	50.0	mg/kg	75	125	30

STL Reference Data Summary

Structured Analysis Code: I-GJ-QV-01-06		Matrix: WATER
Target Analyte List: All Analytes		Extraction: METALS, TOTAL - 2% HCL
		Method: ICP-Mass Spectrometry (200.8)
		QC Program: STANDARD TEST SET
		Location: STL St. Louis

Analyte List		Detection Limits				Check List 6226				Spike List 6227			
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD
88	Aluminum	30	ug/L	8.509	ug/L	20050128	C	Y	200	ug/L	85	115	20
128	Antimony	10	ug/L	0.609	ug/L	20050128	C	Y	200	ug/L	85	115	20
140	Arsenic	10	ug/L	1.809	ug/L	20050128	C	Y	200	ug/L	85	115	20
194	Barium	20	ug/L	0.600	ug/L	20050128	C	Y	200	ug/L	85	115	20
222	Beryllium	5	ug/L	0.1256	ug/L	20050128	C	Y	200	ug/L	85	115	20
313	Boron	50	ug/L	5.50	ug/L	20050128	C	Y	400	ug/L	85	115	20
411	Cadmium	5	ug/L	0.0672	ug/L	20050128	C	Y	200	ug/L	85	115	20
413	Calcium	500	ug/L	100	ug/L	20050128	C	Y	4000	ug/L	85	115	20
2952	Chromium	10	ug/L	3.7	ug/L	20050128	C	Y	200	ug/L	85	115	20
637	Cobalt	10	ug/L	0.519	ug/L	20050128	C	Y	200	ug/L	85	115	20
643	Copper	10	ug/L	0.719	ug/L	20050128	C	Y	200	ug/L	85	115	20
1539	Iron	100	ug/L	7.254	ug/L	20050128	C	Y	200	ug/L	85	115	20
1605	Lead	3	ug/L	0.5652	ug/L	20050128	C	Y	200	ug/L	85	115	20
1618	Magnesium	500	ug/L	13	ug/L	20050128	C	Y	4000	ug/L	85	115	20
1659	Manganese	10	ug/L	0.544	ug/L	20050128	C	Y	200	ug/L	85	115	20
1906	Molybdenum	10	ug/L	0.627	ug/L	20050128	C	Y	200	ug/L	85	115	20
1956	Nickel	10	ug/L	1.150	ug/L	20050128	C	Y	200	ug/L	85	115	20
2214	Potassium	500	ug/L	18	ug/L	20050128	C	Y	4000	ug/L	85	115	20
2281	Selenium	5	ug/L	0.570	ug/L	20050128	C	Y	200	ug/L	85	115	20
2283	Silicon	500	ug/L	25	ug/L	20050128	C	Y	400	ug/L	85	115	20
2285	Silver	10	ug/L	1.5	ug/L	20050128	C	Y	50	ug/L	85	115	20
2315	Sodium	500	ug/L	18.94	ug/L	20050128	C	Y	4000	ug/L	85	115	20
2353	Strontium	10	ug/L	0.5338	ug/L	20050128	C	Y	200	ug/L	85	115	20
2477	Thallium	10	ug/L	0.2198	ug/L	20050128	C	Y	400	ug/L	85	115	20
2479	Tin	10	ug/L	6.2	ug/L	20050128	C	Y	400	ug/L	85	115	20
2482	Titanium	10	ug/L	0.7	ug/L	20050128	C	Y	1000	ug/L	85	115	20
3827	Uranium	10	ug/L	0.1256	ug/L	20050128	C	Y	400	ug/L	85	115	20
2607	Vanadium	10	ug/L	1.627	ug/L	20050128	C	Y	200	ug/L	85	115	20
2649	Zinc	20	ug/L	7.3	ug/L	20050128	C	Y	200	ug/L	85	115	20

STL Reference Data Summary

Structured Analysis Code: I-JX-QV-01-06										Matrix: WATER										
Target Analyte List: All Analytes										Extraction: METALS, FILTERED 2% HCL, DISSOLVED										
										Method: ICP-Mass Spectrometry (200.8)										
										QC Program: STANDARD TEST SET										
										Location: STL St. Louis										
Analyte List					Check List 6224					Spike List 6227										
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
88	Aluminum	30	ug/L	8.509	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
128	Antimony	10	ug/L	0.609	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
140	Arsenic	10	ug/L	1.809	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
194	Barium	20	ug/L	0.600	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
222	Beryllium	5	ug/L	0.1256	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
313	Boron	50	ug/L	5.50	ug/L	20050128	C	Y	1000	ug/L	85	115	20	C	Y	400	ug/L	70	130	20
411	Cadmium	5	ug/L	0.0672	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
413	Calcium	500	ug/L	100	ug/L	20050128	C	Y	10000	ug/L	85	115	20	C	Y	4000	ug/L	70	130	20
2952	Chromium	10	ug/L	3.7	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
637	Cobalt	10	ug/L	0.519	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
643	Copper	10	ug/L	0.719	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
1539	Iron	100	ug/L	7.254	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
1605	Lead	3	ug/L	0.5652	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
1618	Magnesium	500	ug/L	13	ug/L	20050128	C	Y	10000	ug/L	85	115	20	C	Y	4000	ug/L	70	130	20
1659	Manganese	10	ug/L	0.544	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
1906	Molybdenum	10	ug/L	0.627	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
1956	Nickel	10	ug/L	1.150	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
3924	Niobium	25	ug/L	7.643	ug/L	20050128	C	Y	1000	ug/L	85	115	20							
3925	Palladium	1	ug/L	0.2258	ug/L	20050128	C	Y	1000	ug/L	85	115	20							
2209	Platinum	1	ug/L	0.1	ug/L	20050128	C	Y	1000	ug/L	85	115	20							
2214	Potassium	500	ug/L	18	ug/L	20050128	C	Y	10000	ug/L	85	115	20	C	Y	4000	ug/L	70	130	20
2281	Selenium	5	ug/L	0.570	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
2283	Silicon	500	ug/L	25	ug/L	20050128	C	Y	1000	ug/L	85	115	20	C	Y	400	ug/L	70	130	20
2285	Silver	10	ug/L	1.5	ug/L	20050128	C	Y	125	ug/L	85	115	20	C	Y	50	ug/L	70	130	20
2315	Sodium	500	ug/L	18.94	ug/L	20050128	C	Y	10000	ug/L	85	115	20	C	Y	4000	ug/L	70	130	20
2353	Strontium	10	ug/L	0.5338	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
2477	Thallium	10	ug/L	0.2198	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	400	ug/L	70	130	20
3935	Thorium	10	ug/L	0.2512	ug/L	20050128	C	Y	1000	ug/L	85	115	20	C	Y	400	ug/L	70	130	20
2479	Tin	10	ug/L	6.2	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	400	ug/L	70	130	20
2482	Titanium	10	ug/L	0.7	ug/L	20050128	C	Y	1000	ug/L	85	115	20	C	Y	1000	ug/L	70	130	20
2602	Tungsten	5	ug/L	0.884	ug/L	20050128	C	Y	1000	ug/L	85	115	20							
3827	Uranium	10	ug/L	0.1256	ug/L	20050128	C	Y	1000	ug/L	85	115	20	C	Y	400	ug/L	70	130	20
2607	Vanadium	10	ug/L	1.627	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
2649	Zinc	20	ug/L	7.3	ug/L	20050128	C	Y	500	ug/L	85	115	20	C	Y	200	ug/L	70	130	20
2651	Zirconium	100	ug/L	0.333	ug/L	20050128	C	Y	1000	ug/L	85	115	20	C	Y	200	ug/L	70	130	20

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

**TITLE: PREPARATION OF SOIL, SLUDGE, FILTER, BIOTA AND OIL
AND GREASE SAMPLES FOR RADIOCHEMICAL ANALYSIS**

(Supersedes: STL-RC-0004 Rev.8)

Prepared by:

Approved by:

Supervisor/Lead Analyst

Approved by:

Quality Assurance Manager

Approved by:

Environmental Health and Safety Coordinator

Approved by:

Laboratory Director

Proprietary Information Statement:

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1 SCOPE AND APPLICATION

- 1.1 This SOP has destructive procedures, which prepare a sample for radiometric measurement. This procedure is applicable to soils, sludge, filters, biota and oil/grease samples where non-volatile radionuclides are to be determined. This is a procedure for sample preparation only and is used in conjunction with other radiochemical analytical procedures.
- 1.2 The reporting limits and QC limits are maintained in the Information Management System (QuantIMS) and can be found in associated analytical SOPs.

2 SUMMARY OF METHOD

- 2.1 This SOP describes the method that is used to prepare a sample for analysis. After treatment with concentrated acids and/or the muffle furnace, the sample extract or residue is transferred to the applicable procedure for further separation prior to analysis.

3 DEFINITIONS

- 3.1 See the STL Quality Management Plan (QMP) and STL St. Louis Laboratory Quality Manual (LQM) for a glossary of common laboratory terms and data reporting qualifiers.

4 INTERFERENCES

- 4.1 Inherent in this procedure is the assumption that the carrier and/or tracer solution mixes completely with the sample matrix. Incomplete mixing can cause anomalous chemical yield data.
- 4.2 Samples containing naturally high concentrations of the carriers and/or tracers (e.g. Ba, or U-232) can cause chemical yields in excess of 100% for some analyses.

5 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

None.

5.3 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Compound	Hazards	Exposure Limits (1)	Signs of Exposure
Fuming Nitric Acid	Poison Corrosive Oxidizer	2 ppm (TWA) 4 ppm (STEL)	Inhalation symptoms include breathing difficulties, coughing, choking, and irritation of the nose, throat, and respiratory tract. Onset of symptoms may be delayed 4-30 hours. Ingestion of nitric acid can cause immediate pain and burns of the mouth, throat, esophagus and gastrointestinal tract. Skin contact can cause redness, pain, and severe skin burns. Vapors are irritating to the eyes.
Hydrofluoric Acid	Poison Corrosive	3 ppm (TWA)	Inhalation symptoms may include sore throat, coughing, labored breathing and lung congestion/inflammation. Skin contact may cause serious burns which are not immediately apparent or painful. Symptoms of eye contact include redness, pain, and blurred vision.
Nitric Acid	Corrosive Poison Oxidizer	2 ppm, 5 mg/m ³	Inhalation may cause coughing, choking, and irritation of the nose, throat, and respiratory tract. Skin contact can cause redness, pain, and severe skin burns. Concentrated solutions can stain the skin a yellow-brown color. Vapors are irritating to the eyes and contact may cause severe burns.
Ethyl Alcohol (contains methanol)	Flammable	1000 ppm (TWA) (Ethanol)	Symptoms include headache, nausea, dizziness, narcosis. Prolonged contact causes irritation to skin and eyes.
Hydrogen peroxide (30%)	Oxidizer Corrosive Fire (increases flammability of combustible, organic, and readily oxidizable materials)	1 ppm (TWA)	Irritation to respiratory tract and burning of mucous membrane of nose and throat. Pain, redness and blurred vision in eyes.
Hydrochloric Acid	Poison Corrosive	5 ppm Ceiling	Inhalation symptoms include coughing, choking, inflammation of the nose, throat, and upper respiratory tract. Skin contact causes redness, pain, severe skin burns, and discoloration. Vapors are irritating to the eyes. Contact may cause severe burns.

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Compound	Hazards	Exposure Limits (1)	Signs of Exposure
Perchloric acid	Poison Corrosive Oxidizer	None established	Unstable at ordinary temperature and pressure and can undergo explosive decomposition, especially at elevated temperatures or if allowed to dehydrate. Inhalation of vapors or mists will cause irritation with coughing, choking, and inflammation of the nose, throat, and upper respiratory tract. Highly corrosive to tissue. Can cause severe burns with discoloration and pain. Permanent visual damage may occur.
1- Always add acid to water to prevent violent reactions.			
2- Exposure limit refers to the OSHA regulatory exposure limit.			

6 EQUIPMENT AND SUPPLIES

- 6.1 Analytical balance.
- 6.2 Ashless Powder, cellulose filter, Whatman or equivalent.
- 6.3 Disposable aluminum pans.
- 6.4 Disposable digestion vials, 75 ml, Capitol Vial Corporation or equivalent.
- 6.5 Drying oven.
- 6.6 Glassware as appropriate.
- 6.7 Griddle (Hot Plate), heating limit 400 °F.
- 6.8 Grinder.
- 6.9 Hot plates, stirring hotplates.
- 6.10 Mod Block digestion system, CPI International, 48 hole per block or equivalent.
- 6.11 Muffle furnace, programmable.
- 6.12 Perchloric Acid Digestion Hood with water washdown.
- 6.13 Quartz and Porcelain crucibles individually numbered with Tech Pen or permanent marker.
- 6.14 Teflon and Glass beakers, 100, 250, 2000 ml etc.
- 6.15 Tech Pen, for high temperature marking of quartz crucibles.
- 6.16 Teflon beaker covers and glass watch glasses.

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6.17 Teflon stir bars.

7 REAGENTS AND STANDARDS

- 7.1. All standards and reagent preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002, current revision.
- 7.2. L + Ascorbic Acid, $C_6H_8O_6$, powder or crystal.
- 7.3. Barium chloride, $BaCl_2 \cdot 2H_2O$, 0.5% and 0.25% solutions in water.
- 7.4. Boric acid crystals, H_3BO_3 , (Reagent).
- 7.5. Calcium nitrate (1.25 M) - Dissolve 51 g of $Ca(NO_3)_2$ in 100 mL of water and dilute to 250 mL with water.
- 7.6. Deionized Water, obtained from the Milli-Q unit.
- 7.7. Diphonix Resin, Eichrom Technologies, 100-200 mesh.
- 7.8. Eichrom Load solution, Nitric acid (3 M HNO_3) in Aluminum nitrate (1 M $Al(NO_3)_3 \cdot 9 H_2O$), Dissolve 350 g of Aluminum nitrate in 700 ml of water, add 190 ml of concentrated nitric acid and dilute to 1000 ml with DI water. CAUTION - Nitric acid is a strong oxidizer. Contact with other material may cause fire. CORROSIVE. Liquid and mist cause severe burns to all body tissue.
- 7.9. Ferrous Ammonium Sulfate Solution, $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$, Dissolve approximately 1.7 g in approximately 10 ml of DI water.
- 7.10. Fuming nitric acid (90% HNO_3). CAUTION: Strong oxidizer. Contact with other materials may cause fire. Liquid and mist cause severe burns to all body tissue .
 - 7.10.1. Nitric acid (16 M HNO_3) -concentrated, sp. gr. 1.42, 70.4%. Nitric acid is a strong oxidizer. Contact with other material may cause fire. CORROSIVE. Liquid and mist cause severe burns to all body tissue.
 - 7.10.2. Nitric Acid (8 M HNO_3) - To an appropriately sized bottle containing 500 mL deionized water, add 500 mL concentrated HNO_3 . Mix well.
 - 7.10.3. Nitric Acid (4 M HNO_3) - To an appropriately sized bottle containing 500 mL deionized water, add 250 mL concentrated HNO_3 , dilute to 1000 ml with DI water. Mix well.
 - 7.10.4. Nitric Acid (2.5 M HNO_3) - To an appropriately sized bottle containing 500 mL deionized water, add 156 mL concentrated HNO_3 , dilute to 1000 ml with DI water. Mix well.
 - 7.10.5. Nitric Acid (1 M HNO_3) - To an appropriately sized bottle containing 900 mL deionized water, add 62.5 mL concentrated HNO_3 and dilute to 1 liter. Mix well.
- 7.11. Hydrochloric acid (12 M HCl) - concentrated, 37.2%. CAUTION - Hydrochloric acid is a corrosive. Liquid and mist causes severe burns to all body tissue.
 - 7.11.1. Hydrochloric acid (10 M HCl) – Carefully add 833 ml of concentrated hydrochloric acid to 167 ml of Deionized water. Mix well.
 - 7.11.2. Hydrochloric Acid (6 M HCl) - Carefully add 500 mL concentrated hydrochloric acid to 500 mL deionized water. Mix well.

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- 7.12. Hydrofluoric acid (48-52% HF) - concentrated. CAUTION - Hydrofluoric acid is a corrosive and a poison. Extremely hazardous liquid and vapor. Requires special first aid should contact occur. Analysts must be trained in HF acid first aid treatment prior to using this procedure. Reaction with certain metals generates flammable and potentially explosive hydrogen gas.
- 7.13. Hydrogen Peroxide (H₂O₂), 30%. CAUTION - Strong oxidizer. Contact with other materials may cause fire. If allowed to dry on clothing or other combustible materials (i.e., bench paper), evaporation leads to concentration and increased possibility of ignition.
- 7.14. Perchloric acid, HClO₄, concentrated, 70-72%. CAUTION - Strong oxidizer. Contact with other material may cause fire or explosion. Corrosive. Causes severe irritation and burns to every area of contact. Harmful if swallowed or inhaled.
- 7.15. Phenolphthalein Indicator (1%) - In a volumetric flask, dissolve 2.5 grams of phenolphthalein in 125 ml of ethyl alcohol. Dilute to a final volume of 250 ml with ethyl alcohol.
- 7.16. Potassium carbonate, 1 M: Dissolve 138 g of K₂CO₃ in 1 L of water.
- 7.17. Small Ion Exchange Column, 120 x 11.9 mm, Environmental Express # R1010 or equivalent.
- 7.18. Sodium Carbonate (Na₂CO₃·10H₂O), (1 M) - In a 1-L graduated cylinder, dissolve 500 g of sodium carbonate, in deionized water. Dilute to a final volume of 1 L with deionized water. Mix thoroughly and allow to settle overnight.
- 7.19. Sodium sulfate, Na₂SO₄, crystals.
- 7.20. Ethyl alcohol, reagent grade. CAUTION - Flammable liquid and vapor. Vapor harmful.
- 7.21. Refer to Table I for a listing of carriers and tracers. The standard, carrier and/or tracer preparations are described in the applicable analysis procedure.

8 SAMPLE COLLECTION, PRESERVATIVES AND STORAGE

- 8.1 STL St. Louis supplies sample containers and chemical preservatives in accordance with the method. STL St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in STL-PM-0002.
- 8.2 Aqueous samples should be preserved at the time of collection by adding sufficient nitric acid to a pH < 2.
- 8.3 If samples are collected without acidification, they should be brought to the laboratory within 5 days, nitric acid added to bring the pH to 2 or less, the sample shaken, and then held for a minimum of 24 hours in the original container before analysis or transfer of sample. If dissolved or suspended material is to be analyzed separately, do not acidify the sample before filtering the sample. The filtering may be performed in the field by the customer or by the laboratory.
- 8.4 Samples may be collected in either plastic or glass containers.
- 8.5 Samples can be stored for no more than 180 days unless specified by the client.

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9 QUALITY CONTROL

9.1. Batch

- 9.1.1. Definition: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
- 9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3. Each analytical batch may contain up to 20 environmental samples, a method blank, and a single Laboratory Control Sample (LCS) and a Sample Duplicate. For tracer/carrier methods, a Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is performed upon client request. In the event that there is insufficient sample to analyze a Sample Duplicate or MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.
- 9.1.4. Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.2. Method Blank

- 9.1.1 Definition: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.1.2 A method blank must be prepared with every batch (20 or fewer samples of the same matrix).
 - 9.1.2.1 For solid samples, the method blank shall be made using clean sand and analyzed as a similar manner as the client's samples.
 - 9.1.2.2 For liquid samples, deionized water will be used.

9.3 Laboratory Control Sample

- 9.3.1 Definition: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every batch.

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9.4 Matrix Spike/Matrix Spike Duplicate

- 9.4.1 Matrix Spike Definition: An aliquot of a field sample to which a known amount of target analyte(s) is added.
- 9.4.2 Sample Duplicate Definition: An additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.
- 9.4.3 Additional MS and sample duplicates do not count towards the 20 samples in an analytical batch.

9.5 Procedural Variations

- 9.5.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance Memo shall be filed in the project file and incorporated into the report narrative.

9.6 Nonconformance and Corrective Action

- 9.6.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager. See SOP STL-QA-0036 for details regarding the NCM process.
- 9.6.2 The LCS is spiked with all of the standard target isotopes and is used to monitor the accuracy of the analytical process. The matrix for aqueous and solid analyses is deionized (DI) water. Other suitable matrices (i.e., solid reference material for a LCS) can be used as directed by Radiochemistry Supervisors.

10 CALIBRATION AND STANDARDIZATION

- 10.1 Refer to the applicable analytical procedure for standardization of carrier/tracer solutions.
- 10.2 Balances and pipettes calibration must be checked daily when used. Refer to SOP STL-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and pipette".

11 PROCEDURE

11.1 Soil and Sediment - Total Digestion

- 11.1.1 Samples should be dried, ground, ball milled and/or pulverized as necessary, per STL-RC-0003, "Drying and Grinding of Soil and Solid Samples."
- 11.1.2 Samples may be sieved to ensure a uniform particle size.
- 11.1.3 Remove up to a 10 gram aliquot for analysis, and place in a tech pen numbered quartz crucible. Record the exact weight of the sample taken to the nearest 0.0001 gram, when weighed on an analytical balance.

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Note: The amount of sample required will be determined by the MDA requested by the client. For Actinides: Usually a short count will require a 1 gram aliquot and a long count will require a 2 gram aliquot. For Radiums/Strontiums/Nickels/Irons: Usually a 1 gram aliquot will be sufficient. For Leads: Usually a 0.5 gram aliquot will be required. However these amounts are subject to change based on the client requirements, suspected activities of the isotopes, and the discretion of the countroom with regards to count time.

11.1.4 Prepare a LCS and blank using 0.5 ml of 1.25M Calcium nitrate. For an LCS, MS or MSD, add 10 - 40 dpm of standard, depending on the requested target isotopes. A soil reference material can be used for the LCS when the requested target isotopes are present.

11.1.5 Wet samples with enough DI water to saturate the matrix.

11.1.6 Add 5 - 40 dpm of yield tracer to all samples, blanks, LCSs, and MS/MSDs in the analytical batch, depending on the target isotopes (See Table I). If samples are to be diluted, add the tracers after the digestions. (For Sr analysis, add approximately 100 mg of Strontium Nitrate carrier).

11.1.6.1 NOTE: For samples received from DOE-AL sites, add sufficient tracer to yield at least 400 counts.

11.1.7 Gently dry the sample on a hotplate then heat the crucible containing the soil sample to approximately 600°C for at least 4 hours in the muffle furnace (if analyzing for Technetium-99, heat at 450°C). The TEXPEN® ink should turn white and become slightly ashy. This is an indication that the samples have gone to temp. Cool and remove.

11.1.8 Proceed to section 11.2 for digestion instructions.

11.2 Filters and Swipes

Note: The following procedure is for all filters that can be muffled safely via a ramped program without melting and binding to the crucible or beaker. This process is useful for filters (or swipes) that contain organic material as the high temperature of the muffle oven destroys such matter and allows for a much cleaner and simpler digestion. Plastic and Teflon filters should not be prepared using this method. Instead a leach should be performed. For these filter types proceed to section 11.4.

11.2.1 Combine all paper filters which constitute the sample in a 250 mL glass beaker. Make sure the beakers are properly labeled with a TEXPEN® or metal marker to ensure identification post muffling.

11.2.2 If requested prepare a matrix blank using the same number of clean, comparable filters. If no comparable filters are available, a water blank can be used. **Note:** because of the high temperatures in the muffle furnace, most water tracers will be cooked to the beaker and will have low recoveries. To prevent this, 0.5

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mL of 1.25 N Calcium Nitrate should be added to the water controls to keep the tracers from cooking onto the glass. Crucibles can also be used.

- 11.2.3 Prepare an LCS using water, as noted in section 11.1.2.2, or by using a solid reference material as requested by client.

NOTE: For samples received from DOE-AL sites, add sufficient tracer to yield at least 400 counts.

- 11.2.4 Heat samples on hotplate to dry any tracer added.

- 11.2.5 Place sample beakers in muffle furnace and cover with a ribbed watch glass.
NOTE: Use of a ribbed watch glass is vital. If not used organic material may not be destroyed.

- 11.2.6 Heat samples in a muffle furnace using the following sequence.

11.2.6.1 Ramp the heat in the muffle furnace at 3° C a minute up to 160° C.

11.2.6.2 Heat samples for 45 minutes at 160° C.

11.2.6.3 Ramp the heat in the muffle furnace at 0.9° C per minute up to 600° C.

11.2.6.4 Heat to approximately 600° C for 5-6 hours to reduce filters to ash.

- 11.2.7 Allow samples to cool to room temperature.

- 11.2.8 If filters only partially reduced to ash, repeat steps 11.1.2.6 and 11.1.2.7, otherwise proceed to section 11.2 **NOTE: Not all filters will reduce to ash. Glass fiber filters do not ash. As long as the organic material has lightened in color, samples can proceed to digestion.**

11.3 Flora and Fauna

11.3.1 Weigh sample into beaker/crucible. Make sure that the beaker/crucible is properly labeled with a TEXPEN® or metal marker to ensure identification post muffling.

11.3.2 Prepare a Blank in a clean beaker. Also prepare an LCS using water or soil as requested by client. **Note: because of the high temperatures in the muffle furnace, most water tracers will be cooked to the beaker and will have low recoveries. To prevent this, 0.5 mL of 1.25 N Calcium Nitrate can be added to the water controls to keep the tracers from cooking to the glass. NOTE: For samples received from DOE-AL sites, add sufficient tracer to yield at least 400 counts.**

11.3.3 Cover samples with concentrated nitric acid and allow samples to sit overnight. This will help them break down.

11.3.4 Gently heat samples on hotplate to dryness. (This will convert samples to nitrates, which will help destroy the sample in the muffle furnace.)

11.3.5 Place sample beakers in muffle furnace and cover with a watch glass.

11.3.6 Heat samples in a muffle furnace using the following sequence.

11.3.6.1 Ramp the heat in the muffle furnace at 3 ° C a minute up to 160 ° C.

11.3.6.2 Heat samples for 45 minutes at 160 ° C.

11.3.6.3 Ramp the heat in the muffle furnace at 0.9 ° C per minute up to 600 ° C.

11.3.6.4 Heat to approximately 600 ° C for 5-6 hours to reduce samples.

11.3.7 Allow samples to cool to room temperature. **Proceed to section 11.2 for digestion instructions.**

11.4 Oils, greases, and solvents

11.4.1 Properly label beakers/crucibles with a TEXPEN® or metal marker to ensure identification post muffling.

11.4.2 Add enough ashless powder into the bottom of a crucible/beaker to absorb the oil and/or grease of the sample, and the tracer and spikes used. Add the same amount of ashless powder into the Blank and LCS crucibles.

11.4.3 Weigh the sample into the beaker/crucible and cover with more ashless powder.

11.4.4 For the LCS, MS, or MSD add 10-40 dpm of standard, depending on the requested isotopes.

11.4.5 Add 5-40 dpm of yield tracer to all the samples, LCSs and MS/MSDs in the analytical batch, depending on the target isotopes (See Table 1). If samples are to be diluted, add the tracers after the digestion. (For Sr analysis, add approximately 100 mg of Strontium Nitrate carrier).

11.4.6 Place samples on a hotplate. Heat on low to dry.

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11.4.7 Heat samples in a muffle furnace using the following sequence.

11.4.7.1 Ramp the heat in the muffle furnace at 3 ° C a minute up to 160 ° C.

11.4.7.2 Heat samples for 45 minutes at 160 ° C.

11.4.7.3 Ramp the heat in the muffle furnace at 0.9 ° C per minute up to 600 ° C.

11.4.7.4 Heat to approximately 600 ° C for 5-6 hours to reduce samples.

11.4.8 Allow samples to cool to room temperature. **Proceed to section 11.2 for digestion instructions.**

11.5 Mod Block Total Digestion

11.5.1 Add 5 mL of concentrated Hydrochloric acid to the crucibles, cover with a watch glass, and allow to reflux for approximately 30 minutes.

11.5.2 Transfer the samples to properly labeled mod block tubes with concentrated Nitric acid. Try to keep the total amount of acid under 15 mL. **Note: Always label the mod block tubes with a permanent black sharpie. Any other color ink will degrade in the acid fumes and tubes will be unidentifiable.**

11.5.3 Add 10 mL of concentrated Hydrofluoric acid.

11.5.4 Place samples into the Mod Block. Set the temperature setting to 119 degrees Centigrade. Set the timer to 4 hours. Allow samples to cool approximately 30 minutes.

11.5.5 Repeat steps 11.6.7 and 11.6.8. For all samples other than actinides, proceed to the proper separation SOP. For actinides, proceed to 11.2.6.

11.5.6 Add 10 mL of concentrated Nitric acid to the digestion vessel. Add approximately 0.3 grams of boric acid crystals. Set the timer for 2 hours and allow them to go to dryness.

11.5.7 Add 15 mL of Load solution, cover with a plastic watch glass, and set timer for 30 minutes.

11.5.8 Using 3M nitric acid, transfer samples to properly labeled centrifuge tubes and proceed directly to the appropriate column SOP.

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11.6 Wet ashing – Used for samples that were not muffled which have a noticeable amount of organics present.

11.6.1 To wet ash samples, add 20 mL of concentrated Nitric acid. Place samples on a hot plate on low heat.

11.6.2 Slowly add 1-2 mL of peroxide to the samples. **Caution: Samples can bubble vigorously.** Heat for 2- 5 minutes until bubbling slows.

11.6.3 Repeat step 11.6.12.2 until dark samples become lighter in appearance, indicating the destruction of the organics. Heat to dryness.

11.6.4 Proceed with a normal digestion (section 11.2.2).

11.7 Perchloric Acid Digestion- This is an alternative digestion to the one described above. It is useful for samples with complicated matrices. It can be used for samples that were not muffled.

11.7.1 Place samples in Teflon beakers. Include the required QC samples in each batch.

11.7.2 Add the appropriate tracers.

NOTE: For samples received from DOE-AL sites, add sufficient tracer to yield at least 400 counts.

11.7.3 Digest with 10 mL of Concentrated Perchloric acid, 10 mL of Concentrated Nitric acid and 25 mL of Concentrated Hydrofluoric acid to near dryness in fume hood (moist bead). **CAUTION – Perchloric acid is a strong oxidizer. Contact with other material may cause fire or explosion. Corrosive. Causes severe irritation and burns to every area of contact. Harmful if swallowed or inhaled.**

11.7.4 If sample has large amounts of residue repeat step 11.4.3 until the sample residue no longer changes in appearance. For samples with large amounts of dark organics, repeat digestion with 10 mL of concentrated Perchloric acid and cover with a Teflon beaker cover.

11.7.5 Dissolve the residue as needed in the appropriate solution used in the extraction method.

NOTE: Rinse the Perchloric hood down after using perchloric acid. Record the date of washdown in logbook.

11.8 Leaching

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- 11.8.1 Weigh an appropriate amount of sample into a properly labeled 500 mL or 1L poly container complete with lid. Record this weight in the appropriate spreadsheet. If the sample is a filter the weight is 1.
- 11.8.2 Add a known amount nitric acid and record this on the spread sheet as well. Usually the amount of the acid will be either 250mL or 500 mL.
NOTE: The molarity of the acid will vary depending on the purpose of the leach and the matrix. If the purpose is a surface leach, the molarity of the nitric acid should be 2M as long as the matrix is not heavy in metal. If it is heavy in metal, then use a 1M nitric solution. This will prevent excess metals which prove to be interferences for some of the analyses from being dissolved. If the purpose of the leach is dissolution, use an 8M nitric solution.
- 11.8.3 Allow sample to leach for 2 hours, shaking every 15 minutes. Do not completely close lids to allow gases to escape.
- 11.8.4 Samples that do not totally dissolve need to be decanted into another labeled poly.
- 11.8.5 To aliquot the leach, remove a known volume (usually 25 mL). This equates to a percentage of the leach and a percentage of the gram aliquot or filter originally taken.
- 11.8.6 Proceed with a cookdown if the molarity of the acid is too high or in the case of actinides, the samples need to be in load solution. Otherwise proceed to the appropriate separations SOP.

12 DATA ANALYSIS AND CALCULATIONS

- 12.4 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the STL St. Louis LQM.

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 Refer to appropriate analytical SOP.

14 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appropriate analytical SOP.
- 14.2 Initial Demonstration
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in STL St. Louis' LQM section 5.1.2.
- 14.3 Training Qualification

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The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in STL St. Louis' LQM section 5.1.2.

14.3.1 Annually the analyst must successfully demonstrate proficiency to continuing to perform this analysis. See requirements in STL St. Louis' LQM section 5.1.2.

15 VALIDATION DATA

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. STL ST Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
- Sample waste with a Basic pH is generated. All base waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
- Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17 REFERENCES

17.1 STL Quality Management Plan (QMP), current revision.

17.2 STL St. Louis Laboratory Quality Manual (LQM), current revision.

17.3 Associated SOPs:

17.3.1 STL St. Louis Laboratory, STL-QA-0002, "Standards and Reagent Preparation"

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- 17.3.2 STL St. Louis Laboratory, STL-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and pipettes"
- 17.3.3 STL St. Louis Laboratory, STL-PM-0002, "Sample Receipt and Chain-of-Custody."
- 17.3.4 STL St. Louis Laboratory, STL-QA-0036, "Non-conformance Memorandum (NCM) Process."
- 17.3.5 STL St. Louis Laboratory, STL-RC-0003, "Drying and Grinding of Soil and Solid Samples."
- 17.3.6 STL St. Louis Laboratory, STL-RC-0020, "Determination of Gross Alpha/Beta Activity."
- 17.3.7 STL St. Louis Laboratory, STL-RC-0040, "Total Alpha Emitting Isotopes of Radium."
- 17.3.8 STL St. Louis Laboratory, STL-RC-0041, "Radium-228 in Water."
- 17.3.9 STL St. Louis Laboratory, STL-RC-0050, "Preparation of Strontium-89 and -90."
- 17.3.10 STL St. Louis Laboratory, STL-RC-0090, "Preparation of Samples for Sequential Determination of Isotopic Americium, Curium, Neptunium, Plutonium, Thorium, and Uranium in Aqueous Samples."
- 17.3.11 STL St. Louis Laboratory, STL-RC-0110, "Analysis of Total Uranium by Laser-Induced Phosphorimetry."
- 17.3.12 STL St. Louis Laboratory, STL-RC-0120, "Determination of Technetium-99."
- 17.3.13 STL St. Louis Laboratory, STL-RC-0125, "Determination of Technetium-99 Using EIChroM® Teva Resin."
- 17.3.14 STL St. Louis Laboratory, STL-RC-0232, "Isotopic Thorium and/or Neptunium in Water, Soil, Sludge, and Filters by EIChroM® Teva Separation Resin."
- 17.3.15 STL St. Louis Laboratory, STL-RC-0238, "Isotopic Uranium by EIChroM® Uteva Resin For Water, Soil, Sludge and Filters."
- 17.3.16 STL St. Louis Laboratory, STL-RC-0240, "Isotopic Americium, Curium, Plutonium, Thorium, and Uranium in Water, Soil, Sludge and Filters by EIChroM® Separation Resin."
- 17.3.17 STL St. Louis Laboratory, STL-RC-0241, "Americium, Plutonium, Curium and Uranium in Water, Soil, Sludge and Filters by EIChroM® Uteva and Tru resins."
- 17.3.18 STL St. Louis Laboratory, STL-RC-0242, "Isotopic Thorium and Uranium in Water, Soil, Sludge and Filters by EIChroM® Separation Resins."
- 17.3.19 Tables or Figures referenced in body of SOP.

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18 CHANGES FROM PREVIOUS REVISION

18.1 Signature page corrected..

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TABLE I

Analysis①	Tracer	Carrier (Concentration and Volume)
Gross Alpha	None	None
Gross Beta	None	None
Radium-226 or Total Alpha Emitting Radium	Ba-133 (if not performing gravimetric yields)	Ba (~16 mg/ml, 1.000 ml) Pb (~15 mg/ml, 2 ml)
Radium-228	Ba-133 (if not performing gravimetric yields)	Ba (~16 mg/ml, 1.000 ml) Pb (~15 mg/ml, 10 ml) Sr (~10 mg/ml, 2 ml) Y (~18 mg/ml, 1.000 ml)
Strontium-89 and -90	Sr-85 (if not performing gravimetric yields)	Ba (~10 mg/ml, 1 ml) Sr (~50 mg/ml, 1.000 ml)
Lead-210	None	Pb (~20 mg/ml, 1.000 ml)
Polonium-210	Po-209	None
Isotopic Americium	Am-243	None
Isotopic Neptunium	Am-243	None
Isotopic Plutonium	Pu-242	None
Isotopic Thorium	Th-229	None
Isotopic Uranium	U-232	None
Total Uranium	None	None
Technetium-99	Tc-99m	None

① Other analyses may be added which are not listed. Consult with the Radiochemistry Sample Preparation Team Leader or the Radiochemistry Group Leader for tracers/carriers for those procedures.

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

TITLE: Determination of Gross Alpha/Beta Activity

(Supersedes: STL-RC-0020 Revision 7)

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1.0 SCOPE AND APPLICATION

- 1.1 This procedure applies to the preparation and analysis of samples for gross alpha and/or beta radioactivity.
- 1.2 This SOP is applicable to EPA Method 900.0, SW-846, Method 9310.
- 1.3 For water samples containing high concentrations of dissolved solids (> 500 ppm), see SOP STL-RC-0021 for analysis of gross alpha radioactivity.
- 1.4 The reporting limits, method detectable activities and QC limits are maintained in the Information Management System (QuantIMS). Because of their dynamic nature, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools. A copy of the SAC is included in the analytical SOP to demonstrate this information.

2.0 SUMMARY OF METHOD

- 2.1 This SOP is applicable for the preparation and analysis of samples for gross alpha and/or beta radioactivity.
- 2.2 This method is applicable to determination of gross alpha and/or gross beta activity in air filters, water (dissolved solids not > 500 ppm), soil/sediment, and vegetation samples.
 - 2.2.1 For total sample activity, an aliquot of aqueous sample is evaporated to dryness in a glass beaker after the addition of concentrated nitric acid to convert any chlorides to nitrates, and transferred quantitatively to a tarred counting planchet. The sample residue solution is dried, and then counted for alpha and/or beta radioactivity using a Gas Flow Proportional Counter.
 - 2.2.2 For the activity of dissolved matter, an aliquot of aqueous sample is filtered through a 0.45-mm membrane filter. The filtrate is evaporated to dryness in a glass beaker after the addition of concentrated nitric acid to convert any chlorides to nitrates, and transferred quantitatively to a tarred counting planchet. The sample residue solution is dried, and then counted for alpha and/or beta radioactivity using a Gas Flow Proportional Counter.
 - 2.2.3 For the activity of suspended matter, an aliquot of aqueous sample is filtered through a 0.45-mm membrane filter. The filter is transferred to a counting planchet. The sample residue is dried, and then counted for alpha and/or beta radioactivity using a Gas Flow Proportional Counter.
 - 2.2.4 Air filter samples are counted for gross alpha and/or beta activity without further processing if the filter is less than 2 inches diameter. If the filter is greater than 2-inch diameter, the sample is digested per STL-RC-0004, "Preparation of Soil, Sludge and Filter Paper Samples for Radiochemical Analysis," and then an aliquot prepared like a liquid.
 - 2.2.5 Solid samples can be analyzed for gross alpha and/or beta activity as a dry powder. If Method RP710 is required, an acid leach is performed per STL-RC-0004, "Preparation of Soil, Sludge and Filter Paper Samples for Radiochemical Analysis". The digestate is then treated like a liquid.

NOTE: Total Sample Dissolution can be done using HF and Nitric acid as in section 11.9.

- 2.2.6 Oil samples are ashed in a muffle furnace, then dissolved in nitric acid and transferred to a glass beaker where they are converted to nitrate salts using concentrated nitric acid. The sample is then transferred to a tarred planchet using nitric acid, dried, and counted for alpha and/or beta radioactivity using a Gas Flow Proportional Counter.
- 2.2.7 Gross Alpha and Gross Beta activity does not identify the radionuclide that is present. Instead, the activity is referenced as equivalent to Th-230 for Gross Alpha and Sr-90/Y-90 for Gross Beta.

3.0 DEFINITIONS

- 3.1 See the STL Quality Management Plan (QMP) and STL St. Louis Laboratory Quality Manual (LQM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 Minimum Detectable Activity (MDA) - The smallest amount of activity that can be detected given the conditions of a specific sample. It is reported at the 95% confidence interval, meaning that there is a 5% chance that a false signal was reported as activity, and a 5% chance that true activity went undetected. The MDA that is reported is a combination of counting error as well as preparation errors.

4.0 INTERFERENCES

- 4.1 Since, in this method for gross alpha and gross beta measurement, the radioactivity of the sample is not separated from the solids of the sample, the solids concentration is a limiting factor in the sensitivity of the method for any given sample.
- 4.2 For a 2-inch diameter counting planchet (20 cm²), an aliquot containing 100 mg of dissolved solids would be the maximum aliquot size for that sample which should be evaporated and counted for gross alpha or gross beta activity.
- 4.3 Radionuclides that are volatile under the sample preparation conditions of this method can not be measured. Other radioactivities may also be lost during the sample evaporation and drying (such as tritium and some chemical forms of radioiodine). Some radioactivities, such as the cesium and technetium radioisotopes, may be lost when samples are heated to dull red color. Such losses are limitations of the test method.
- 4.4 Moisture absorbed by the sample residue increases self absorption and, if uncorrected, leads to low-biased results. For hygroscopic sample matrices, the nitrated water solids (sample evaporated with nitric acid present) will not remain at a constant weight after being dried and exposed to the atmosphere before and during counting. Those types of water samples need to be heated to a dull red color for a few minutes to convert the salts to oxides.
- 4.5 Heterogeneity of the sample residue in the counting planchet interferes with the accuracy and precision of the method.

5.0 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

None.

5.3 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.**

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Analytical Balance (4 - or 5 - place).
- 6.2 Beakers: pyrex glass and Teflon, various sizes. Teflon beakers must be washed immediately before use. Please consult SOP: STL-RC-5006 "DECONTAMINATION OF LABORATORY GLASSWARE, LABWARE AND EQUIPMENT"
- 6.3 Bottle, wash.
- 6.4 Counting planchets, stainless steel, 5.0 cm (2.0"), cleaned per STL-RC-0002, "Preparation of Stainless Steel Planchets for Radiochemistry Analyses."
- 6.5 Desiccator with desiccant, Dri-Rite or equivalent.

- 6.6 Drying oven with thermostat set at $105^{\circ}\text{C} \pm 5^{\circ}\text{C}$.
- 6.7 Filter paper: ash less, Whatman #41 or ash less paper pulp, and 0.45-mm membrane, 5.0 cm.
- 6.8 Graduated cylinder - size appropriate to sample volume.
- 6.9 Propane torch.
- 6.10 Hot plate-stirrer or heat lamp.
- 6.11 Calibrated pipettes, Eppendorf or equivalent.
- 6.12 Policeman: rubber or plastic
- 6.13 Porcelain crucibles with lids, approximately 30-ml. capacity.
- 6.14 Muffle furnace
- 6.15 Tongs or forceps.

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002, current revision.
- 7.2 Reagents are prepared from reagent grade chemicals, unless otherwise specified below, and reagent water.
- 7.3 Deionized Water, obtained from the Milli-Q unit.
- 7.4 Nitric acid, concentrated (16N HNO_3)
- 7.5 Hydrofluoric acid, concentrated (29N HF)
- 7.6 4N Nitric acid (4N HNO_3) - Add 250 ml of 16N HNO_3 to 750 ml of reagent water and mix well.
- 7.7 Calibration solution: Add 6.75 g $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, 17.5 NaCl, 7.75 g $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$, 2.55 g $\text{MgCl} \cdot 6\text{H}_2\text{O}$, and 1.5 g CaCO_3 to 1900 ml of reagent water, add 50 ml concentrated Nitric acid and dilute to 2 liters. Stir, heat to dissolve.
- 7.8 Salt, NaCl, granular.
- 7.9 Thorium-230 for LCS and matrix spikes, calibrated - NIST traceable, diluted to approximately 20 dpm/ml.
- 7.10 Thorium-230 for attenuation curve, calibrated - NIST traceable, diluted to approximately at least 4000 dpm/ml. Must be a different solution than the one used for the LCS and matrix spikes.
- 7.11 Strontium-90 for LCS and matrix spikes, calibrated - NIST traceable, in equilibrium with Yttrium 90, diluted to approximately 20 dpm/ml.

- 7.12 Strontium-90 for attenuation curve, calibrated - NIST traceable, in equilibrium with Yttrium 90, diluted to at least 4000 dpm/ml. Must be a different solution than the one used for the LCS and matrix spikes.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 STL St. Louis supplies sample containers and chemical preservatives in accordance with the method. STL St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in STL-PM-0002.
- 8.2 Aqueous samples should be preserved at the time of collection by adding sufficient nitric acid to a pH < 2.
- 8.3 If samples are collected without acidification nitric acid is added to bring the pH to 2 or less, the sample shaken, and then held for a minimum of 24 hours in the original container before analysis or transfer of sample. If dissolved or suspended material is to be analyzed separately, do not acidify the sample before filtering the sample. The filtering may be performed in the field by the customer or by the laboratory.
- 8.4 Samples may be collected in either plastic or glass containers. Hold time is 180 days.

9.0 QUALITY CONTROL

9.1 Batch

- 9.1.1 Definition: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. An analytical batch is composed of prepared environmental samples that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 Each analytical batch may contain up to 20 environmental samples, a method blank, a single Laboratory Control Sample (LCS), a Matrix Spike and Sample Duplicate. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
- 9.1.4 Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.2 Method Blank

- 9.2.1 Definition: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.2.2 A method blank must be performed with every batch (20 or fewer samples of the same matrix).

9.2.3 Water method blanks consist of reagent water. Prepare a method blank from an aliquot of reagent water equivalent to the target volume of 200 or 500 ml. Add 1.5 ml of NaCl solution for mass.

9.2.4 Soil method blanks are sand.

9.2.5 For oils, prepare a method blank from shredded filter paper in a crucible.

9.2.5.1 For filters, prepare a method blank for filter samples by securing a blank filter into a planchet.

9.3 Laboratory Control Sample

9.3.1 Definition: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every batch.

9.3.3 For water, oils and filter samples, a separate LCS for gross alpha and a LCS for gross beta are performed.

9.3.3.1 The alpha LCS is reagent water is fortified with a Thorium 230 and the beta LCS is reagent water fortified with Strontium 90.

9.3.4 For waters, prepare an LCS with a similar aliquot of reagent water spiked with 1 ml. of the standard. Add 1.5 ml of NaCl solution for mass.

9.3.5 For solid samples, the matrix is the National Bureau of Standards, SRM 4353, Rocky Flats Soil #1.

9.3.6 For Oils, Prepare a LCS from shredded filter paper that has been spiked with 1 ml. of the spiking solution.

9.3.6.1 For filters, prepare a LCS by securing a blank filter which has been spiked with 1 ml of the spiking solution. Dry the planchets which have been spiked with the aqueous solutions under a heat lamp or in an oven (105 ± 2 °C) before proceeding.

9.4 Matrix Spike

9.4.1 Definition: An aliquot of a field sample to which a known amount of target analyte(s) is added.

9.4.2 An MSD can be prepared with a batch, in lieu of a sample duplicate. If there is insufficient sample to perform an MS/MSD, a duplicate LCS is analyzed..

9.5 Sample Duplicate

9.5.1 Definition: A separate aliquot of a field sample taken through the entire analytical process.

9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed.

9.6 Procedural Variations

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance Memo shall be filed in the project file and incorporated into the report narrative.

9.7 Nonconformance and Corrective Action

- 9.7.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager. See SOP STL-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 At least six standards per alpha and beta curve are prepared in a glass beaker, as in Section 11.5; using varying amounts of calibration solution. The planchet net weights range from approximately 0 to 0.150 g for the alpha curve and approximately 0 to 0.250 g for the beta curve.
- 10.2 Balance calibration must be checked daily when used. Refer to SOP STL-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes Procedure.
- 10.3 For analytical instrumentation calibration, see SOP: STL-RD-0403, "Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System".

11.0 PROCEDURE

- 11.1 For cleaning of glassware/labware prior to sample preparation, refer to SOP: STL-RC-5006.
- 11.2 If the activity of dissolved matter in an aliquot of aqueous sample is to be determined, filter the desired aliquot through a 0.45-mm membrane filter and proceed with aqueous sample preparation.
- 11.3 If the activity of suspended matter of an aliquot of aqueous sample is to be determined, filter the desired aliquot through a 0.45-mm membrane filter, and proceed with aqueous sample preparation.
- 11.4 Aqueous Sample - Total Solid Screen
- 11.4.1 Record all sample preparation data on a sample worksheet or on the Weight file for the batch.
- 11.4.2 Agitate the sample container thoroughly.
- 11.4.2.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply.
- 11.4.3 Pipette a 20 ml. aliquot in to a tared beaker. Add 10 ml of concentrated Nitric acid.
- 11.4.4 Evaporate to dryness using a hot plate or heat lamp, such that the sample does not splatter.

- 11.4.5 Remove from heat and allow to cool to room temperature. Add 10 ml concentrated Nitric acid.
- 11.4.6 Evaporate to dryness using a hot plate or heat lamp, such that the sample does not splatter.
- 11.4.7 Remove from heat and allow to cool in desicator for a minimum of 30 minutes.
- 11.4.8 Reweigh the beaker to estimate solids content of the sample.
- 11.4.9 From the net residue weight and sample volume used, determine the sample volume required to meet the target residue weight using the formula given in step 12.1, with a target weight of 80 mg (sample weights should not exceed 100 mg, if sample weights exceed 100 mg an aliquot of the dried residue should be taken after redissolving in 4 N nitric acid. Dillutions are noted on the worksheet. If it is not practical to redissolve the residue the sample should be redone using less volume. If it is not practical to redissolve or restart the sample, check with the count room supervisor or designee to verify that the sample weight fits on the current alpha curve before counting.) alpha/beta dried residue on the planchet. If only Gross Beta is being performed, the target weight may be increased to 160 mg. Compare the calculated volume to meet the weight limitation with the volume required to ensure that the MDA is below the Reporting Limit. The volume for analysis is the smaller of the two volumes.

11.5 Aqueous Sample Total Activity

- 11.5.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required or begin a Weight file for recording planchet weights.
- 11.5.2 Shake the sample container thoroughly. Measure a volume of sample, previously determined in section 11.4, into an appropriately sized beaker. Record volume of sample used.
- 11.5.3 If it is determined that only a small volume of sample is required, the additional volume may be added in small aliquots directly to the beaker previously used to determine the volume needed to achieve the target sample weight.
- 11.5.4 Add 10 ml of concentrated nitric acid to all samples including the Blank and LCS.
- 11.5.5 Evaporate to dryness using a hot plate or heat lamp, such that the sample does not splatter.
- 11.5.6 Remove from heat and allow to cool to rooom temperature. Add 10 ml concentrated Nitric acid.
- 11.5.7 Evaporate to dryness using a hot plate or heat lamp, such that the sample does not splatter.

Note: Some samples with difficult matrices may require steps 11.5.2 and 11.5.3 to be repeated until the sample residue does not change in appearance.

- 11.5.8 Remove from heat and allow to cool to room temperature. Add 10 ml of 4 N nitric acid. Heat on hot plate to dissolve sample residue and reduce volume.
- 11.5.9 Quantitatively transfer the sample to a tared, stainless steel planchet.
- 11.5.10 Use a policeman, if needed, to complete the transfer. Wash down the beaker with small portions of 4 N HNO₃ and add to the planchet.
- 11.5.11 Evaporate to dryness on a hot plate such that the sample does not splatter. Remove sample from hot plate.

NOTE: Do not allow liquid to splatter.

- 11.5.12 Dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours if sample appears hygroscopic. If not hygroscopic proceed to step 11.5.13.
- 11.5.13 Cool planchets in a desiccator for a minimum of 30 minutes. Weigh the cooled planchets and record final weight.

NOTE: If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; 100 mg (2.0" planchet)

- 11.5.14 Store dry sample in a desiccator until counted for gross alpha and/or beta activity.

11.6 Oil

- 11.6.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required.
- 11.6.2 Fill a 30 ml porcelain crucible ¼ full with confetti made from Whatman No. 41 filter paper or ashless paper pulp.
- 11.6.3 Place crucible on analytical balance, then tare the balance.
- 11.6.4 Weigh to the nearest 0.0001 g, approximately 1 to 2 gm sample of the oil onto the shredded filter paper. Record the sample weight. Cover with a crucible lid.
- 11.6.5 If the sample is a mixture of oil and water, or is a sample spiked with an aqueous solution, evaporate the water on a hot plate or under a heat lamp before muffling. Do not allow residue to "bake" on hot plate. A programmable muffle program may also be used to dry the water before ramping the temperature.

NOTE: Do not allow liquid to splatter.

- 11.6.6 Heat the sample in a muffle oven for one hour at 750° C. Ramp the temperature in increments of 50-75° C starting at 200° C and maintain for 30 minutes.
- 11.6.7 Turn off the muffle oven, crack open the door, and allow the sample to cool to room temperature.
- 11.6.8 Add approximately 2 ml of 4 N HNO₃ to the residue in the crucible.

- 11.6.9 Quantitatively transfer the sample to a glass beaker with 4 N HNO₃.
- 11.6.10 Use a policeman, if needed, to complete the transfer. Wash down the crucible and lid with small portions of dilute HNO₃ and add to beaker.
- 11.6.11 Evaporate to dryness on hot plate or heat lamp such that the sample does not splatter.
- 11.6.12 Remove from heat and allow to cool to room temperature.
- 11.6.13 Add 10 ml of concentrated nitric acid. Evaporate to dryness on a hot plate or heat lamp such that the sample does not splatter.
- 11.6.14 Remove from heat and allow to cool to room temperature.
- 11.6.15 Add 10 ml of 4 N nitric acid and heat to dissolve and reduce volume.
- 11.6.16 Dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours if sample appears hygroscopic. If not hygroscopic proceed to step 11.6.18.
- 11.6.17 Cool planchets in a desiccator for a minimum of 30 minutes.
- 11.6.18 Weigh the cooled planchet and record final weight.

CAUTION: Ensure that the solids content do not exceed the maximum allowed weight for the determination and planchet used.

- 11.6.19 Store dry sample in a desiccator until counted for gross alpha and/or beta activity.

11.7 Filter Samples

- 11.7.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required.
- 11.7.2 If the filter is 2" diameter or less, secure the air filter in a stainless steel planchet with double-sided cellophane tape such that no portion of filter extends above the lip of the planchet. Then proceed to step 11.7.18.
- 11.7.3 If the filter is greater than 2 inches diameter, digest or leach the sample per STL-RC-0004. Prepare a method blank and LCS from blank filters, spiked as above, which are digested in the same manner.
- 11.7.4 Shake the digested sample thoroughly. Measure a volume of sample into an appropriately sized teflon beaker. Record volume of sample used.
- 11.7.5 Add 10 ml of 16N nitric acid.
- 11.7.6 Evaporate to dryness on a warm hot plate such that the sample does not splatter.
- 11.7.7 Remove from heat and allow to cool to room temperature.
- 11.7.8 Add 10 ml of 16N nitric acid.

- 11.7.9 Evaporate to dryness on a warm hot plate such that the sample does not splatter.
- 11.7.10 Remove from heat and allow to cool to room temperature.
- 11.7.11 Add 10 ml of 4 N nitric acid. Heat to dissolve and reduce volume.
- 11.7.12 Quantitatively transfer the sample to a tared, stainless steel planchet.
- 11.7.13 Use a rubber policeman, if needed, to complete the transfer. Wash down the beaker with small portions of 4 N HNO₃ and add to the planchet.
- 11.7.14 Evaporate to dryness on a warm hot plate such that the sample does not splatter.
- 11.7.15 Dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours if sample appears hygroscopic. If not hygroscopic proceed to step 11.7.16.
- 11.7.16 Cool planchets in a desiccator for a minimum of 30 minutes.
- 11.7.17 Weigh the cooled planchet and record final weight.

CAUTION: Ensure that the solids content do not exceed the maximum allowed weight for the determination and planchet used.

- 11.7.18 Store dry sample in a desiccator until counted for gross alpha and/or beta activity.

11.8 Solid and/or Soil Samples

- 11.8.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required.
- 11.8.2 If the sample has already been prepared per STL-RC-0003, "Drying and Grinding of Soil and Solid Samples," proceed to step 11.8.7 for direct sample mounting. If the sample is to be leached per DOE Method RP710, proceed to STL-RC-0004, "Preparation of Soil, Sludge and Filter Paper Samples for Radiochemical Analysis." The digestate is then treated like a liquid (section 11.3).
 - 11.8.2.1 For soils that need to be analyzed using total sample dissolution, proceed to Section 11.9.
- 11.8.3 Remove an aliquot (typically 1 - 5 gm.) with a spatula and place into a clean, labeled aluminum pan." (Aluminum weighing pans work well).
- 11.8.4 Place sample on a hot plate or in a drying oven at approximately 105° C and evaporate any moisture.
- 11.8.5 When dry, remove from hot plate or oven and allow the sample to cool.
- 11.8.6 Using a metal spatula, reduce the solid sample to a fine particle size.

NOTE: Sample size is restricted to 100 mg for alpha/beta analysis.

- 11.8.7 Self adhesive label dots of the chosen planchet size can be used to hold finely divided solid material uniformly for gross alpha and/or beta analysis. Tare the prepared planchet.
- 11.8.8 Distribute the sample evenly in a tared stainless steel planchet.
- 11.8.9 Weigh and record the gross sample weight.
- 11.8.9.1 Use table salt for the blank and a soil standard reference material, i.e. NIST Traceable Rocky Flats Soil, for the LCS. Prepare in the same fashion as the samples.
- 11.8.10 Store dry sample in a desiccator until counted for gross alpha and/or beta activity.
- 11.9 Solid and/or Soil Samples by total dissolution.
 - 11.9.1 Weigh 1 to 2 grams of sample into a recently washed teflon beaker. Wet with deionized water.
 - 11.9.2 Prepare a method blank from an aliquot of reagent water equivalent to the largest sample weight. Prepare an LCS with a similar aliquot of reagent water spiked with 1 ml. of the standards described in 7.5 or use a determined amount of suitable soil standard with known activity. Note: separate LCS's must be prepared for alpha and beta analysis.
 - 11.9.3 Carefully add 5 ml concentrated nitric acid, 5 ml concentrated hydrochloric acid and 10 ml of concentrated Hydrofluoric acid to all samples.
 - 11.9.4 Evaporate to dryness using a hot plate or heat lamp, such that the sample does not splatter. Repeat 11.9.3 and 11.9.4 step once.
 - 11.9.5 Add 10 ml of concentrated nitric acid to all samples including the Blank and LCS.
 - 11.9.6 Evaporate to dryness using a hot plate or heat lamp, such that the sample does not splatter.
 - 11.9.7 Remove from heat and allow to cool to room temperature. Add 10 ml concentrated Nitric acid.
 - 11.9.8 Evaporate to dryness using a hot plate or heat lamp, such that the sample does not splatter.
 - 11.9.8.1 Note: Some samples with difficult matrices may require steps 11.9.6 and 11.9.7 to be repeated until the sample residue does not change in appearance.
 - 11.9.9 Remove from heat and allow to cool to room temperature.
 - 11.9.10 Add 10 ml of 4 N nitric acid. Heat on hot plate to dissolve sample residue and reduce volume.
 - 11.9.11 Quantitatively transfer the sample to a tared, stainless steel planchet.
 - 11.9.12 Use a policeman, if needed, to complete the transfer. Wash down the beaker with small portions of 4 N HNO₃ and add to the planchet.

11.9.13 Evaporate to dryness on a warm hot plate such that the sample does not splatter. Remove sample from hot plate.

NOTE: Do not allow liquid to splatter.

11.9.14 Dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours if sample appears hygroscopic. If not hygroscopic proceed to step 11.9.15.

11.9.15 Cool planchets in a desiccator for a minimum of 30 minutes. Weigh the cooled planchets and record final weight.

11.9.16 **NOTE: If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; 100 mg (2.0" planchet).**

11.9.17 Store dry sample in a desiccator until counted for gross alpha and/or beta activity.

11.10 Reprocessing planchets which are over the weight limit.

11.10.1 Rinse residue from planchet with 4 N HNO₃ into a beaker. Add 4 N HNO₃ to planchet and heat if necessary. Use a policeman, if needed, to complete the transfer.

11.10.2 Redissolve the residue into 4 N HNO₃. Dilute the sample to a known volume.

11.10.3 Remove an aliquot which will keep the residue weight under the limit and transfer to the tared planchet. Record information on sample worksheet.

11.10.4 Evaporate to dryness on a warm hot plate so that the sample does not boil. Remove sample from hot plate. Allow to cool.

11.10.5 Weigh the cooled planchet and record final weight.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery, RPD, MDA) and standard instrument software calculations are given in the STL St. Louis LQM.

12.2 Appropriate factors must be applied to sample values if dilutions are performed.

12.3 Sample volume may need to be adjusted in order not to exceed 100 mg of dried residue on planchet. Volume shown is "typical" maximum volume used provided Total Solids does not exceed 500 ppm for waters and 200 ppm for drinking waters.

12.4 To calculate the aqueous sample volume required (ml), use the following equation:

$$\text{volume required (mL)} = \frac{\text{target net residue weight (mg)} * \text{initial aliquot volume (mL)}}{\text{initial aliquot net residue weight (mg)}}$$

12.5 To calculate the density (mg/cm²), use the following equation:

$$mg/cm^2 = \frac{\text{net residue weight (mg)}}{20.27cm^2 (2" \text{ planchet})}$$

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1. The data assessment and corrective action process is detailed through the Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: STL-QA-0036. A hardcopy of all the data assessment types and descriptions along with their associated corrective actions is included in that SOP.

13.2 See analytical SOP STL-RD-0403.

14. METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1. Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix to this SOP.

14.2. Demonstration of Capability

14.2.1. Initial and continuing demonstrations of capability requirements are established in STL St. Louis' LQM section 5.1.2

14.3. Training Qualification

14.3.1. The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in STL St. Louis' LQM section 5.1.2

14.3.3. Annually the analyst must successfully demonstrate proficiency to continuing to perform this analysis. See requirements in STL St. Louis' LQM section 5.1.2

15. VALIDATION DATA

15.1. Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods.

16. WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2. Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

17. REFERENCES

17.1. "Prescribed Procedures for Measurement of Radioactivity in Drinking Water," Method 900.0, August, 1980.

17.2. "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW846, Method 9310, Rev. 0, September, 1986.

17.3. STL Quality Management Plan (QMP)

17.4. STL St. Louis Laboratory Quality Manual (LQM)

17.5. STL Corporate Safety Manual and St. Louis Facility Addendum (SOP STL-HS-0002), current revisions.

17.6. Associated SOPs

- | | |
|----------|---|
| 17.6.1. | STL-RC-0002, Preparation of Stainless Steel Planchets for Radiochemistry Analyses. |
| 17.6.2. | STL-RC-0003, Drying and Grinding of Soil and Solid Samples |
| 17.6.3. | STL-RC-0004, Preparation of Soil, Sludge and Filter Paper Samples for Radiochemical Analysis |
| 17.6.4. | STL-RC-0021, Gross Alpha Radiation in Water Using Coprecipitation |
| 17.6.5. | STL-RC-5006, Decontamination of Laboratory Glassware. Labware and Equipment |
| 17.6.6. | STL-RD-0403, Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System |
| 17.6.7. | STL-QA-0002, Standards and Reagent Preparation |
| 17.6.8. | STL-QA-0005, STL-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes |
| 17.6.9. | STL-QA-0036, Non-conformance Memorandum (NCM) Process |
| 17.6.10. | STL-PM-0002, Sample Receipt and Chain of Custody |

18. CHANGES FROM PREVIOUS REVISION

- 18.1. Revised volume in sections 11.4.3 and 11.9.1.
- 18.2. Revised section 11 text relating to hygroscopic samples.
- 18.3. Replaced SOP reference for STL-QA-0006 with STL-PM-0002
- 18.4. Revised Safety, section 5 and hazard tables in accordance with CSM.
- 18.5. Merged and revised waste management and pollution prevention sections, Section 16.
- 18.6. Added text to address sample collection references and capabilities, Section 8.
- 18.7. Added text to Section 12 referencing commonly used calculations are in the LQM.
- 18.8. Added DOC reference information to the method performance Section 14.
- 18.9. Created a "Validation Data" section, Section 15.
- 18.10. Revised Quality Control, Section 9.
- 18.11. References, section 17 revised.

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

**TITLE: PREPARATION OF SAMPLES FOR GAMMA
SPECTROSCOPY**

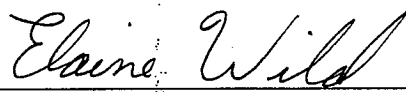
(SUPERSEDES: STL-RC-0025 REV. 4)

Prepared by: _____

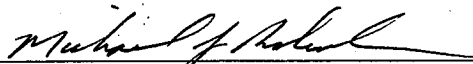
Approved by: _____


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Approved by: _____


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Approved by: _____


Laboratory Director

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1 SCOPE AND APPLICATION

- 1.1 The purpose of this SOP is to provide detailed instructions for the preparation of samples which require gamma spectroscopy analysis.
- 1.2 This SOP is applicable to EPA Method 901.1 and DOE Method GA-01-R.
- 1.3 The laboratory target analytes supported by this method, the reporting limits, and QC limits are maintained in the Information Management System (QuantIMS). A copy of the Structure and Analysis Code (SAC), which lists this information, is included in SOP: STL-RD-0101.

2 SUMMARY OF METHOD

- 2.1 This SOP provides procedure describes methods for preparation of samples of liquid, soil, vegetation, air filter, and core matrix prior to gamma spectroscopy analysis.
- 2.2 Samples are transferred to a standard geometry container for counting on the gamma detectors. Hyper pure germanium (HPGe) gamma detectors are used to detect isotopes with gamma ray energies between 40 and 2000 Kev. Activity concentration is determined using commercially available gamma spectral analysis software. Any sample matrix, which can be mounted in one of the standard geometries, may be analyzed for any of the isotopes included in the radionuclide reference library. Gamma photon energies not identified in the reference library may be identified and evaluated manually.

3 DEFINITIONS

- 3.1 See the STL Quality Management Plan (QMP) and STL St. Louis Laboratory Quality Manual (LQM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 Minimum Detectable Activity (MDA) or Minimum Detectable Concentration (MDC) - A calculated estimate of the minimum activity of a radionuclide which could be measured given the existing conditions. It is an estimate at the 95% confidence interval, meaning that there is a 5% chance that a nuclide could be reported as a false positive, and 5% chance that a nuclide would be reported as a false negative if the result were at the MDA or MDC.

4 INTERFERENCES

- 4.1 Gamma energy emissions identified with scientifically measured probability by some radionuclides are documented by multiple sources. There are some discrepancies between reference sources and attempts are made to evaluate the reference data used in spectral analysis. Gamma emissions at discreet energy and probability are used to identify and quantify specific radionuclides in the sample. Gamma emissions which are completely absorbed by an HPGe detector form photo peaks which are used for identification and quantification of gamma emitting radionuclides. When two or more nuclides emit similar gamma energy the photo peaks cannot be resolved without using complex algorithms. These photo peaks in close proximity can interfere with the identification or quantification of a radionuclide. Knowing this the nuclide reference library, computer software and analyst training are used to minimize the possibility of interference and mis-identification. Although it is not possible to eliminate interferences and misidentification.

5 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.
- 5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
Wear Kevlar or MAPA Blue-Grip gloves when using knives or sharp articles.
- 5.3 PRIMARY MATERIALS USED
The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table**

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contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm- STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6 EQUIPMENT AND SUPPLIES

- 6.1 Calibrated Balance (2 to 5 decimal places as appropriate for measurement.)
- 6.2 Blender
- 6.3 Drying Oven
- 6.4 Mortar and pestle
- 6.5 Pulverizer
- 6.6 Food chopper/grinder
- 6.7 Kevlar Gloves for cutting
- 6.8 Knives appropriate for food preparation
- 6.9 Graduated cylinder
- 6.10 Filter disk, 47 millimeter diameter
- 6.11 Plastic Tape
- 6.12 Marinelli beakers of various sizes (commonly 500 mL and 1000 mL less commonly used 4000 mL or 350 mL)
- 6.13 Petri dishes, 2 inch diameter
- 6.14 Can Sealer
- 6.15 Cans and lids, Ness 307 X 200, 8 oz, 227 mL or equivalent (commonly referred to as tuna cans)
- 6.16 8oz, straight sided polypropylene jars or equivalent; (used for 25mL and 100 mL geometries)

7 STANDARDS and REAGENTS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002, current revision.
- 7.2 Deionized Water, obtained from the Milli-Q unit.
- 7.3 Nitric acid (16M HNO₃)
- 7.4 Sand

8 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 STL St. Louis supplies sample containers and chemical preservatives in accordance with the method. STL St. Louis does not perform sample collection. Samplers should reference the analytical methods and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in STL-PM-0002.

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- 8.2 Preserve water samples by adding 2 ml concentrated nitric acid per liter at the time of sample collection.

Note: samples collected for I-129 or I-131 analysis are not preserved.

Milk samples are also not preserved with acid.

- 8.3 The sample container should be glass or polyethylene. Wide-mouth bottles are preferred for soil, sludge and sediment samples.
- 8.4 Samples can be stored for no more than 180 days unless specified by the client.

9 QUALITY CONTROL

9.1 Batch

- 9.1.1 Definition: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.
- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 Each analytical batch may contain up to 20 environmental samples, a method blank, a single Laboratory Control Sample (LCS) and Sample Duplicate. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
- 9.1.4 Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.2 Method Blank

- 9.2.1 Definition: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.2.2 A method blank must be extracted/digested with every extraction batch (20 or fewer samples of the same matrix).
- 9.2.2.1 For solid samples, the method blank shall be made using clean sand or other clean matrix and analyzed in the same manner as the client's samples.
- 9.2.2.2 For liquid samples, deionized water will be used.
- 9.2.2.3 For filter samples. 47mm filter disks will be used.

9.3 Laboratory Control Sample

- 9.3.1 Definition: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 A geometry specific LCS is run with each batch of samples.

9.4 Matrix Duplicate

- 9.4.1 Sample Duplicate Definition: An additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.
- 9.4.2 Additional sample duplicates do not count towards the 20 samples in an analytical batch.

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9.5 Procedural Variations

- 9.5.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance Memo shall be filed in the project file and incorporated into the report narrative.

9.6 Nonconformance and Corrective Action

- 9.6.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager. See SOP STL-QA-0036 for details regarding the NCM process.

10 CALIBRATION AND STANDARDIZATION

- 10.1 Balance calibrated in accordance with SOP: STL-QA-0005. For gamma spectroscopy calibration, see SOP: STL-RD-0101.

11 PROCEDURE

11.1 Liquid Sample Preparation

- 11.1.1 Liquid samples shall be prepared as a 25 mL, 50 mL, 100 mL, 500 mL, or 1000 mL geometry.
11.1.2 The volume of sample used depends on the amount required to meet the detection limits, the volume of sample supplied by the client, and whether the sample has very high activity. The sample volume may be reduced for high activity samples due to detector dead time considerations. Consult the countroom supervisor or radiochemistry technical director, if the sample has high activity which may require action.
11.1.3 Shake the sample to suspend any residue and to ensure that the sample is homogeneous.
11.1.4 Measure the required sample volume (25, 100, 500, or 1000mL). Record the sample volume on the gamma worksheet.

NOTE: If the client does not provide sufficient sample, and the sample is near a larger geometry, rather than reducing the volume significantly it may be preferable to dilute an aqueous sample with DI water to the correct volume in order to achieve a lower MDC. **ALWAYS CONSULT SUPERVISION BEFORE PERFORMING THIS ACTION.** If the sample is diluted the undiluted volume is recorded as the sample volume the dilution is only for fitting the calibrated geometry. A Nonconformance Memo (NCM) is necessary for this action and a second NCM may be necessary if the contract required detection limit (CRDL) cannot be achieved.

- 11.1.5 Write sample information (i.e. ID #) on the container.
11.1.6 Pour the sample into the appropriately sized container.
11.1.7 Place the lid securely on the Marinelli beaker.
11.1.7.1 Remove excess air from Marinelli.
11.1.8 Seal the lid using plastic tape. Marinelli beakers are prone to leaking liquids; the tape is tightly wrapped around the lid and the beaker in three layers each overlapping the previous layer with half the width of the tape. Make sure there are no creases in the tape which will form a channel for leakage.
11.1.9 Inspect for leakage.
11.1.10 Submit sample for analysis.

11.2 Soil Sample Preparation

- 11.2.1 Soil samples for I-129 or I-131 analysis **are not dried and ground** but rather inserted into an appropriate calibrated geometry. I-131 can be processed in any soil geometry but I-129 analysis uses only a 50 mL or 100 mL straight sided poly jar geometry.
- 11.2.2 Soil samples shall be prepared as 200 mL sealed can, 100 mL, or 25 mL or 500 mL Marinelli (marnsoil) geometry based on the amount of available sample. In both the can and Marnsoil geometries the soil should nearly fill the container.
- 11.2.3 Dry and grind or pulverize the soil sample as described in Procedure STL-RC-0003.
- 11.2.4 Write sample information (ie. ID #) on the sample container.
- 11.2.5 Tare the empty container.
- 11.2.6 Fill the container with the appropriate amount of sample as described below.
 - 11.2.6.1 Fill tuna cans to the ridge mark with sample. If there is insufficient sample to fill the can to the ridge, reduce geometry size.
 - 11.2.6.2 Fill 100 mL geometry to the level as denoted on the reference bottle. If there is insufficient sample to fill, reduce the geometry size.
 - 11.2.6.2.1 A 100mL reference bottle is kept denoting the appropriate fill level.
 - 11.2.6.3 Fill 25 mL geometry to the level as denoted on the reference bottle. If there is insufficient sample to fill the 25 mL geometry, write a NCM stating insufficient sample provided for routine analysis.
 - 11.2.6.3.1 A 25mL reference bottle is kept denoting the appropriate fill level.
 - 11.2.6.4 Fill 500 mL Marinelli beakers to the ridge mark just below the lid with sample. If there is insufficient sample to fill the Marnsoil, to the ridge, reduce geometry size.
- 11.2.7 Record the sample weight/mass on the gamma worksheet.
- 11.2.8 Close the sample container securely.
- 11.2.9 Seal the container with plastic tape, for tuna cans seal with can sealer.
- 11.3 Vegetation Sample Preparation
 - 11.3.1 Vegetation samples shall be prepared in the same geometries as liquid samples and counted directly as dried and chopped matrix, green unprocessed matrix (if directed to do so by the client or if I-131 or I-129 is to be reported) or digested and counted as a liquid. Consult the client requirement information and supervision to determine proper handling.
 - 11.3.2 Vegetation samples **for I-129 or I-131 analysis are not dried and ground** but rather inserted into an appropriate calibrated geometry. I-131 can be processed in any liquid geometry but I-129 analysis uses only a 50 mL or 100 mL straight sided poly jar geometry.
 - 11.3.3 For vegetation samples requiring digestion prior to analysis, refer to SOP STL-RC-0004.
 - 11.3.4 A dry solid sample counted directly shall be counted in a 500 mL Marinelli, 100 mL, or 25 mL geometry. The containers shall be filled to the appropriate level with the dried sample. If there is sufficient sample and a low detection limit is required, a 1 liter Marinelli beaker can be used. Consult supervision to ask if this geometry is appropriate.
 - 11.3.5 Write sample information (i.e. ID #) on the container.
 - 11.3.6 Tare the empty container.
 - 11.3.7 Place sample in the tared container. For dried and chopped vegetation compress the sample when filling a 500 mL Marinelli beaker
 - 11.3.8 Weigh sample and record the weight on the worksheet as DRY weight in grams. If the sample is not dried per client instructions use the percent moisture to convert the mass to dry weight.
 - 11.3.9 Close the sample container securely, seal with plastic tape and submit for analysis.
 - 11.3.10 For vegetation samples which must be digested and counted as a liquid sample.

- 11.3.11 Digest the sample as described in SOP STL-RC-0004. Dissolve or dilute the residue as appropriate for the geometry being used.
- 11.3.12 Shake the sample to suspend any residue and to ensure that the sample is homogeneous.
- 11.3.13 Write sample information (i.e. ID #) on the poly jar or Marinelli.
- 11.3.14 Measure the required sample volume (25 mL, 100 mL, and 500 mL). Record the sample mass / weight as DRY weight in grams on the gamma worksheet. If the sample is not dried per client instructions use the percent moisture to convert the mass to dry weight. If the client requires reporting on a wet weight basis record the mass as measured.
- 11.3.15 Pour the sample into the appropriate size container.
- 11.3.16 Place lid securely on the Marinelli beaker.
 - 11.3.16.1 Remove excess air from Marinelli beaker.
- 11.3.17 Seal the lid using plastic tape. Marinelli beakers are prone to leaking liquids; the tape is tightly wrapped around the lid and the beaker in three layers each overlapping the previous layer with half the width of the tape. Make sure there are no creases in the tape which will form a channel for leakage.
- 11.3.18 Inspect for leakage.
- 11.4 Air Filters/Stripes
 - 11.4.1 Air filters will be counted as single filters or as composite filters.
 - 11.4.2 Air filters shall be counted directly or digested and counted as a liquid.
 - 11.4.3 Direct Filter Preparation
 - 11.4.3.1 Write sample information (i.e. ID #) on petri dish.
 - 11.4.3.2 Load the air filter(s) directly into petri dish.
 - 11.4.3.3 Cover the petri dish.
 - 11.4.3.4 Secure the petri dish lid with plastic tape.
 - 11.4.3.5 Submit sample for counting.
 - 11.4.4 Digested Filter Preparation
 - 11.4.4.1 Refer to SOP STL-RC-0004 for preparation of digested filters.
- 11.5 Core Samples
 - 11.5.1 To obtain sample, cut Shelby tube or sample container into two pieces.
 - 11.5.2 Using a rigid pipe cutter cut the tube completely through.
 - 11.5.3 Using a wire saw, cut through the sample.
 - 11.5.4 Cuts should be made at 2 inch intervals.
 - 11.5.5 If more than 500g of sample is available:
 - 11.5.5.1 Weigh the empty container to be used for counting. Select a Tuna can, 10 mL, or 25 mL.
 - 11.5.5.2 CAREFULLY remove sample from every other sliced section of the Shelby tube.
 - 11.5.5.3 Dry and grind the sample as described in SOP STL-RC-0003.
 - 11.5.5.4 Tare the sample container.
 - 11.5.5.5 Place the dried sample into the container for counting.
 - 11.5.5.6 Record sample weight on worksheet.
 - 11.5.5.7 Secure the lid on the container with plastic tape.
 - 11.5.5.8 Submit sample for analysis.
 - 11.5.5.9 Store unused sample in the labeled sample container.

NOTE: If less than 500 g of sample is available, contact supervision and follow his/her instructions.
 - 11.5.6 Submit sample for analysis, SOP: STL-RD-0101.
- 11.6 Food: vegetables, produce, grain or animal feed:

- 11.6.1 Vegetables, produce, and grain samples shall be prepared in a 500 mL Marinelli beaker or 1 liter Marinelli beaker geometry, due to the lower detection limits for food matrices. These matrices are counted directly as whole grain, chopped or blended produce or vegetable matrices without drying unless directed by the client to dry the matrix. Consult the client requirement information and supervision to determine proper handling.
- 11.6.2 **Warning: Kevlar or MAPA Blue-Grip gloves must be worn when processing the sample.**
- 11.6.3 For vegetables and produce prepare the sample by chopping with a knife on a cutting board or using a food processor, if available.
- 11.6.4 Write sample information (i.e. ID #) on the container.
- 11.6.5 Tare the empty container.
- 11.6.6 Place processed sample in the tared container. For chopped vegetation compress the sample when filling a 500 mL Marinelli beaker
- 11.6.7 Weigh sample and record the weight on the worksheet as WET weight in grams. If the sample is dried per client instructions use the dry weight.
- 11.6.8 Close the sample container securely, seal with plastic tape and submit for analysis. **Consult the countroom concerning use of refrigerated storage.**
- 11.7 Food: meat and fish:
 - 11.7.1 Meat and fish shall be prepared in a 500 mL Marinelli beaker or 1 liter Marinelli beaker geometry due to the lower detection limits for food matrices. Potentially the 100 mL geometry may be utilized if there is insufficient volume to prepare the larger geometry although the detection limits will increase substantially. These matrices are counted directly without drying. Consult the client requirement information and supervision to determine proper handling.
 - 11.7.2 **Warning: Kevlar or MAPA Blue-Grip gloves must be worn when processing the sample.**
 - 11.7.3 For meat and edible portions of fish prepare the sample by chopping with a knife on a cutting board. The fish will need to be filleted prior to chopping.
 - 11.7.4 For analysis of fish **when the whole fish is required to be analyzed.**
 - 11.7.5 Write sample information (i.e. ID #) on the container.
 - 11.7.6 Tare the empty container.
 - 11.7.7 Remove the head with a knife and cut the fish into pieces of appropriate size to easily fit into the Marinelli beaker without air voids. Place the heads in the main portion of the Marinelli and surround it with pieces to eliminate air voids or spaces
 - 11.7.8 Place processed sample in the tared container. Compress the sample evacuating any spaces in the geometry when filling a Marinelli beaker
 - 11.7.9 Weigh sample and record the weight on the worksheet as WET weight in grams.
 - 11.7.10 Close the sample container securely, seal with plastic tape and submit for analysis. **Consult the countroom concerning use of refrigerated storage.**

12 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the STL St. Louis LQM.

13 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: STL-QA-0036. A hardcopy of all the data assessment types and descriptions along with their associated corrective actions is included in the SOP. Below is a subset of the data assessment and QC

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excursion types within Clouseau; the text in underline is the exact "type" line in Clouseau. For a complete and current listing, please access the software program.

- 13.2 Data Assessment and Acceptance Criteria; Corrective Action for Out of Control Data, see SOP: STL-RD-0101.

14 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of the applicable analytical SOP.
- 14.2 Initial and continuing demonstrations of capability requirements are established in STL St. Louis' LQM section 4.1.3.
- 14.3 Training Qualification:
- 14.3.1 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in STL St. Louis' LQM section 4.1.3.
- 14.3.3 Annually the analyst must successfully demonstrate proficiency to continuing to perform this analysis. See requirements in STL St. Louis' LQM section 4.1.3.

15 DATA VALIDATION

- 15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. STL ST Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16 WASTE MANAGEMENT AND POLLUTION PREVENTION

- 15.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where feasible, technological changes have been implemented minimizing the potential for pollution to the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- 15.2 Waste Streams Produced by the Method
- The following waste streams are produced when this method is carried out.
- Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B."
 - Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17 REFERENCES

- 17.1 Method EPA 901.1.
- 17.2 Method DOE GA-01-R.
- 17.3 STL Quality Management Plan (QMP), current revision.
- 17.4 STL St. Louis Laboratory Quality Manual (LQM) current revision.

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- 17.5 STL Corporate Safety Manual and St. Louis Facility Addendum (SOP STL-HS-0002), current revisions.
- 17.6 Associated SOPs
 - 17.6.1 STL-RC-0003, Drying and Grinding of Soil and Solid Samples.
 - 17.6.2 STL-RC-0004, Preparation of Soil, Sludge, and Filter Paper Samples for Radiochemical Analysis.
 - 17.6.3 STL-QA-0002, Standard and Reagent Preparation.
 - 17.6.4 STL-PM-0002, Sample Receipt and Chain of Custody.
 - 17.6.5 STL-QA-0036, Non- conformance Memorandum (NCM) process.

18 CHANGES FROM PREVIOUS REVISION

- 18.1 Summary, Definitions and Interference sections to more accurate descriptions.
- 18.2 Revised Safety section 5 to address use of cut proof gloves.
- 18.3 Modified the Equipment list.
- 18.4 Minor edits to multiple sections.
- 18.5 Modified preservation Section 8 to not preserve samples for Iodine 129 or 131 analysis as well as not preserving Milk samples with acid.
- 18.6 Modifications to Procedure, section 11 giving more explicit detail to soil, vegetation and liquid matrices. Added two sections for food matrices.
- 18.7 Updated SOP reference in Section 8 and Section 17.
- 18.8 Updated DOC LQM reference in Section 14.

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

TITLE: TOTAL ALPHA EMITTING ISOTOPES OF RADIUM

(SUPERSEDES: STL-RC-0040 Rev. 2)

Prepared by: _____

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1. SCOPE AND APPLICATION

- 1.1. This procedure describes the determination of total Radium, for all isotopes emitting alpha radiation, using EPA method 903.0
- 1.2. This procedure also defines a method for the preparation in the determination of Ra226 followed by alpha spec. analysis.
- 1.3. This procedure applies to the analysis of these isotopes in water and in other media where dissolution and carrier exchange are readily available in the laboratory.
- 1.4. The barium sulfate from the last part of the ^{228}Ra procedure (STL-RC-0041) can be counted for total alpha radiation, if a sequential procedure is desired. Care should be taken to ensure even distribution of the precipitate on each planchet prior to counting. The time of the last barium sulfate precipitation should be recorded and used in calculating the ingrowth factor.
- 1.5. The reporting limits and QC limits are maintained in the Information Management System (QuantIMS). A copy of the SAC is included in the analytical SOP: STL-RD-0403 and STL-RD-0210.

2. SUMMARY OF METHOD

- 2.1. Barium and lead are used to coprecipitate radium as the sulfate. Following chelation with EDTA, (Ra-Ba) sulfate is precipitated, purified and counted in a gas flow proportional counter, measuring alpha radiation only. Total radium is quantified by applying correction factors for ingrowth of ^{226}Ra progeny, gravimetric yield and counting efficiency.

3. DEFINITIONS

- 3.1. See the STL Quality Management Plan (QMP) and STL St. Louis Laboratory Quality Manual (LQM) for a glossary of common laboratory terms and data reporting qualifiers.

4. INTERFERENCES

- 4.1. This procedure screens for ^{226}Ra by measuring the alpha emitting radium isotopes. It follows that if there is no detectable radium alpha activity there would be no ^{226}Ra above the specified detection limit.
- 4.2. Waters that contain large amounts of barium will cause a bias to the gravimetric yield.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
None.

5.3 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. **A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.**

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid	Corrosive Poison Cancer Hazard	1 mg/m3	Inhalation may cause irritation of the nose and throat, and labored breathing. Skin contact symptoms include redness, pain, and severe burning. Eye contact can cause blurred vision, redness, pain, and severe tissue burns.

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Acetic Acid, Glacial	Corrosive Flammable	10 PPM (TWA)	Inhalation causes respiratory tract irritation including nasal discharge, hoarseness, coughing, chest pain, and breathing difficulty. Skin contact symptoms may include redness or discoloration, swelling, itching, burning, or blistering of skin. Eye symptoms include irritation, burning sensation, pain, watering, and/or change of vision.
Ammonium Hydroxide	Poison Corrosive	50 PPM (NH3)	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6. EQUIPMENT AND SUPPLIES

- 6.1. Centrifuge tubes, 50 ml
- 6.2. Centrifuge
- 6.3. Hot plate
- 6.4. Analytical balance
- 6.5. Stainless steel planchets
- 6.6. Syringe filters, 20 ml
- 6.7. micro co-precipitation filtration apparatus
- 6.8. 0.1um Eichrom Resolve filters
- 6.9. vacuum pump
- 6.10. heat lamp
- 6.11. plastic adhesive discs

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7. REAGENTS AND STANDARDS

- 7.1. All standards and reagent preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002, current revision.
- 7.2. Distilled or deionized water, ASTM Type II (1991) from the Millipore unit.
- 7.3. Acetic acid (17.4 N, 99.8%), concentrated glacial CH_3COOH , specific gravity 1.05.
- 7.4. Ammonium hydroxide (15 N, 56.6%), concentrated NH_4OH , sp. gr. 0.90.
- 7.5. Ammonium sulfate (200 mg/L) - dissolve 200 grams $(\text{NH}_4)_2\text{SO}_4$ in 300 ml deionized water. Bring to a volume of 1000 ml.
- 7.6. Barium carrier - dissolve 28.46 g $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ in 750 ml deionized water. Add 5 ml 16N HNO_3 . Dilute to 1000 ml.
 - 7.6.1. Standardize the barium carrier solution using the following procedure.
 - 7.6.1.1. Pipette 1.0 ml barium carrier solution (16 mg/ml, Ba) into six separate labeled centrifuge tubes containing 15 ml DI H_2O .
 - 7.6.1.2. Add 1 ml 18 N sulfuric acid with stirring and digest precipitate in a hot water bath for approximately 10 min.
 - 7.6.1.3. Cool, centrifuge and decant the supernate into appropriate waste container.
 - 7.6.1.4. Wash precipitate with 15 ml DI water, centrifuge and decant the supernate.
 - 7.6.1.5. Transfer the precipitate to a tared stainless steel planchet with a minimum amount of DI water.
 - 7.6.1.6. Dry on a heat source. Store in desiccator until cool and weigh as barium sulfate.
 - 7.6.1.7. Record the net weights of the precipitates and calculations in the Rad Standards Preparation Log. Assign the solution a unique number.
- 7.7. Citric acid (1M) - dissolve 19.2g of $\text{C}_6\text{H}_8\text{O}_7 \cdot \text{H}_2\text{O}$ in water and dilute to 100 ml.

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- 7.8. EDTA reagent basic (0.25M) - dissolve 20g NaOH in 750ml water, heat and slowly add 93g [ethylenedinitrilo] tetraacetic disodium salt, (C₁₀H₁₄O₈N₂Na₂·2H₂O) while stirring. Dilute to 1 liter.
- 7.9. Lead carrier (15 mg/ml) - dissolve 2.397g Pb(NO₃)₂ in water, add 0.5 ml 16N HNO₃ and dilute to 100 ml with water.
- 7.10. Methyl orange indicator (0.1%) - dissolve 0.1 g methyl orange indicator in 100 ml water.
- 7.11. Nitric acid (16 N, 70.4%), concentrated HNO₃, sp. gr.
- 7.12. Sulfuric acid (18 N) - Cautiously mix 1 volume 36N H₂SO₄ (concentrated) with 1 volume of water.
- 7.13. Barium carrier (standardized) – 33.9 mg/ml
- 7.14. Barium carrier (0.339 mg/ml) – dilute 0.5ml of standardized barium carrier to 50ml with DI water

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1. STL St. Louis supplies sample containers and chemical preservatives in accordance with the method. STL St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in STL-QA-0006.
- 8.2. All samples may be collected in glass or plastic containers.
- 8.3. Preserve all aqueous samples with nitric acid to a pH of less than 2. within 180 days of the collection date.

9. QUALITY CONTROL

9.1. Batch

- 9.1.1. Definition: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation

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method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

- 9.1.2. Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3. Each analytical batch may contain up to 20 environmental samples, a method blank, and a single Laboratory Control Sample (LCS) and a Sample Duplicate. A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is performed upon client request. In the event that there is insufficient sample to analyze a Sample Duplicate or MS/MSD, an LCS Duplicate (LCSD) is prepared and analyzed.
- 9.1.4. Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.2. Method Blank

- 9.2.1. Definition: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.2.2. A method blank must be prepared with every preparation batch (20 or fewer samples of the same matrix).
- 9.2.2.1. For solid samples, the method blank shall be made using clean sand and analyzed in a similar manner as the client's samples.
- 9.2.2.2. For liquid samples, deionized water will be used.

9.3. Laboratory Control Sample

- 9.3.1. Definition: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

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9.3.2. An LCS must be prepared with every batch.

9.4. Matrix Spike/Matrix Spike Duplicate

9.4.1. Matrix Spike Definition: An aliquot of a field sample to which a known amount of target analyte(s) is added.

9.4.2. Sample Duplicate Definition: An additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.4.3. Additional MS and sample duplicates do not count towards the 20 samples in an analytical batch.

9.5. Procedural Variations

9.5.1. Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance Memo shall be filed in the project file and incorporated into the report narrative.

9.6. Nonconformance and Corrective Action

9.6.1. Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager. See SOP STL-QA-0036 for details regarding the NCM process.

10. CALIBRATION AND STANDARDIZATION

10.1. The Gas Proportional Counting System must be characterized such that the response to (Ra-Ba)SO₄ is firmly established and the appropriate correction factors have been established and documented. See SOP STL-RD-0403.

11. PROCEDURE

11.1. Total Alpha Emitting Isotopes of Radium by GFPC

11.1.2 Ensure that sample container is capped tightly and shake it thoroughly. Transfer to a beaker an aliquot of appropriate size. Label beaker with sample ID number. Record all data on sample worksheet.

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11.1.3 Add 1 M citric acid in the ratio of 5 ml per liter. Add methyl orange indicator until the persistence of a red color. Mix thoroughly.

11.1.4 Add 1.0 ml standardized barium carrier and 2.5 ml of lead carrier, heat and stir until incipient boiling.

11.1.5 Add ammonium hydroxide dropwise until the solution changes from pink to yellow or the pH is > 6.5.

11.1.6 Add 18N sulfuric acid until the red color reappears or the pH is < 2, then add 5 ml ammonium sulfate. Stir the samples for a minimum of 15 minutes and turn off the heat of the stirrer. Remove the stir bar.

11.1.7 Cover the beaker and allow the precipitate to settle for at least four to six hours. Note: the lead and barium sulfate should be clearly separate from the solution.

11.1.8 Remove the clear supernate using suction and discard into the appropriate waste container. Quantitatively transfer the precipitate to a 50 ml centrifuge tube, using a strong jet of deionized H₂O.

11.1.9 Centrifuge for 10 minutes at a speed sufficient to cause the precipitate to form a pellet. Pour off liquid and save the BaSO₄ precipitate.

11.1.10 Carefully add 10 ml 16N HNO₃. Cap tube and vortex to ensure complete mixing. Centrifuge for 10 minutes at a speed determined as in 5.8.10. Pour off the liquid and save the BaSO₄ precipitate.

11.1.11 Repeat preceding step once using deionized water as a rinse. Save the BaSO₄ precipitate and proceed to the next step.

11.1.12 Add 20 ml basic EDTA reagent, vortex and heat in a hot water bath until precipitate dissolves. Add a few drops 10N NaOH if precipitate does not readily dissolve.

11.1.13 Add 1 ml (NH₄)₂SO₄ (200 mg/ml) and stir thoroughly. Add 17.4N CH₃COOH until barium sulfate precipitates, then add 2 ml excess. Note date and time of BaSO₄ precipitation on the sample data sheet. Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate.

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11.1.13.1 Perform a Lead scavenge as follows:

11.1.13.2 Dissolve the precipitate in 20 ml basic EDTA reagent as before, then add 1ml lead carrier (15 mg/ml). If any precipitate forms, dissolve it by adding a few drops of 10N NaOH.

11.1.13.3 Add 0.3 ml ammonium sulfate and stir well. Add 10N sodium hydroxide drop-wise with vigorous stirring until lead sulfide precipitates, then 10 drops excess. Stir intermittently for about 10 minutes. Centrifuge and decant supernatant into a clean tube.

11.1.13.4 Add 1 ml lead carrier (1.5 ng/ml), 0.1 ml ammonium sulfide, and a few drops 10N sodium hydroxide. Repeat precipitation of lead sulfide as before. Centrifuge and filter supernatant through 0.45 mm syringe filter into a clean tube. Wash filter with approximately 5 ml water. Discard residue.

11.1.13.5 Add 20 ml basic EDTA reagent, vortex and heat in a hot water bath until precipitate dissolves. Add a few drops 10N NaOH if precipitate does not readily dissolve.

11.1.14 Wash precipitate with 10 ml water. Centrifuge and discard supernate. Repeat this step once.

11.1.15 Transfer precipitate to a tared stainless steel planchet with a minimum amount of water.

11.1.15.1 Dry on a hot plate on medium heat. Cool in a dessicator, and then weigh planchet.

11.1.15.2 Heat the planchet again using the infrared lamp. Weigh the planchet a second time to confirm that the weight of the planchet has not changed (± 0.005 mg).

11.1.15.3 Repeat steps 11.18.1 and 11.18.2 until the weight of the planchet is constant.

11.1.16 Record the final weight of the planchet to determine the chemical recovery for the barium carrier solution.

11.1.17 Submit planchet for counting at alpha voltage only.

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11.2 Radium 226 by Alpha Spec

- 11.2.1 Ensure that sample container is capped tightly and shake it thoroughly. Transfer to a beaker an aliquot of appropriate size. Label beaker with sample ID number. Record all data on sample worksheet.
- 11.2.2 Add 1 M citric acid in the ratio of 5 ml per liter. Add methyl orange indicator until the persistence of a red color. Mix thoroughly.
- 11.2.3 Add 1 ml of 0.339 mg/ml Barium carrier and 1 ml Ba133 tracer.
- 11.2.4 Add 10 ml of 15 mg/ml lead carrier.
- 11.2.5 Add 1.0 ml standardized barium carrier and 2.5 ml of lead carrier, heat and stir until incipient boiling.
- 11.2.6 Add ammonium hydroxide dropwise until the solution changes from pink to yellow or the pH is > 6.5 .
- 11.2.7 Add 18N sulfuric acid until the red color reappears or the pH is < 2 , then add 5 ml ammonium sulfate. Stir the samples for a minimum of 15 minutes and turn off the heat of the stirrer. Remove the stir bar.
- 11.2.8 Cover the beaker and allow the precipitate to settle for at least four to six hours. Note: the lead and barium sulfate should be clearly separate from the solution.
- 11.2.9 Remove the clear supernate using suction and discard into the appropriate waste container. Quantitatively transfer the precipitate to a 50 ml centrifuge tube, using a strong jet of deionized H_2O .
- 11.2.10 Centrifuge for 10 minutes at a speed sufficient to cause the precipitate to form a pellet. Pour off liquid and save the $BaSO_4$ precipitate.
- 11.2.11 Carefully add 10 ml 16N $HN O_3$. Cap tube and vortex to ensure complete mixing. Centrifuge for 10 minutes at a speed determined as in 5.8.10. Pour off the liquid and save the $BaSO_4$ precipitate.
- 11.2.12 Repeat preceding step once using deionized water as a rinse. Save the $BaSO_4$ precipitate and proceed to the next step.
- 11.2.13 Add 20 ml basic EDTA reagent, vortex and heat in a hot water bath until precipitate dissolves. Add a few drops 10N NaOH if precipitate does not readily dissolve.
- 11.2.14 Add 1 ml $(NH_4)_2SO_4$ (200 mg/ml) and stir thoroughly. Add 17.4N CH_3COOH until barium sulfate precipitates, then add 2 ml excess. Note date and time of $BaSO_4$ precipitation on the sample data sheet. Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate.
- 11.2.15 Set up micro co-precipitator apparatus. Place a 0.1 μm Eichrome Resolve filter on the base stem and lock the filter funnel onto the base. Wet the filter with DI water and apply vacuum.
- 11.2.16 Slurry the precipitate using 5 ml DI water and pour onto filter.
- 11.2.17 Rinse with DI water.
- 11.2.18 Turn off vacuum and remove filter.

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- 11.2.19 Place under heat lamp.
- 11.2.20 Once dry, place the filter on the plastic adhesive disc.
- 11.2.21 Submit for counting.

12 DATA ANALYSIS AND CALCULATIONS

- 12.1. Commonly used calculations (e.g. LCS % recovery and RPD) and standard instrument software calculations are given in the STL St. Louis LQM.
- 12.2. Radium by GFPC calculations are given in SOP: STL-RD-0403.
- 12.3. Radium 226 by alpha spec calculations are given in SOP: STL-RD-0210

13. DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1. The data assessment and corrective action process is detailed through the Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: STL-QA-0036. A hardcopy of all the data assessment types and descriptions along with their associated corrective actions is included in the SOP. Below is a subset of the data assessment and QC excursion types within Clouseau; the text in underline is the exact "type" line in Clouseau. For a complete and current listing, please access the software program.
- 13.2. Data Assessment and Acceptance Criteria; Corrective Action for Out of Control Data, see SOP: STL-RD-0403 and STL-RD-0210.

14. METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

- 14.1. Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix of the applicable analytical SOP.
- 14.2. Initial and continuing demonstrations of capability requirements are established in STL St. Louis' LQM section 5.1.2.
- 14.3. Training Qualification:
 - 14.3.1. The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in STL St. Louis' LQM section 5.1.2.
 - 14.3.3. Annually the analyst must successfully demonstrate proficiency to continuing to perform this analysis. See requirements in STL St. Louis' LQM section 5.1.2.

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15. VALIDATION DATA

15.1. Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. STL ST Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16. WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention.

16.2. Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
- Sample waste with a Basic pH is generated. All base waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
- Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17. REFERENCES

- 17.1. U.S. Nuclear Regulatory Commission, Regulatory Guide 4.15, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment.
- 17.2. Alpha Emitting Radium Isotopes in Drinking Water, Method 903.0, Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA 600/4-30-032, Section 6, Environmental Protection Agency.
- 17.3. STL Quality Management Plan (QMP)

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- 17.4. STL St. Louis Laboratory Quality Manual (LQM)
- 17.5. STL Corporate Safety Manual and St. Louis Facility Addendum (SOP STL-HS-002), current revisions.
- 17.6. Associated SOPs
 - 17.6.1. STL-QA-0002, Standard and Reagent Preparation
 - 17.6.2. STL-QA-0005, Calibration and Verification Procedure for Thermometers, Blances, Weights and Pipettes.
 - 17.6.3. STL-QA-0006, Sample Receipt and Chain of Custody.
 - 17.6.4. STL-QA-0036, Non-conformance Memorandum (NCM) Process.
 - 17.6.5. STL-RC-0002, Planchet Preparation for Radiochemistry and Radiological Screening Analysis.
 - 17.6.6. STL-RD-0403, Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System
 - 17.6.7. STL-RD-0210, Daily Operations of an Alpha Spectroscopy System (using AlphaVision Software)
- 17.7. Clarifications, Modifications to the Reference Method
 - 17.7.1. The initial precipitation of total alpha radiums uses the technique cited in EPA Method 904.0, whereas EPA Method 903.0 uses straight sulfuric acid, and less carriers to bring down the Pb/Ba sulfate.
 - 17.7.2. A Pb scavenge identical to the one found in EPA Method 904.0 has been incorporated into this procedure, as skipping this step could artificially inflate barium yields and thus bias the result.

18. CHANGES TO PREVIOUS REVISION

- 18.1. Added Radium 226 to section 11.

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

TITLE: Radium 228 in Water

(SUPERSEDES: STL-RC-004 Rev. 2)

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1.0 SCOPE AND APPLICATION

- 1.1 This method covers the determination of Radium 228 (^{228}Ra) by direct measurement of its beta emitting progeny, Actinium (^{228}Ac). It is applicable to liquid or other media where complete dissolution and carrier exchange are readily achievable in the laboratory. For media where chemical dissolution is impractical, non-destructive measurement of the three principal photons of ^{228}Ac by gamma spectrometry is better suited.
- 1.2 This SOP is applicable to EPA Method 904.0
- 1.3 The barium sulfate precipitate from this procedure contains all radium isotopes and therefore can be used for ^{226}Ra also.
- 1.4 The reporting limits, method detectable activities and QC limits are maintained in the Information Management System (QuantIMS). Because of their dynamic nature, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools. A copy of the SAC is included in the analytical SOP to demonstrate this information.
- 1.5 Method Variances:
 - 1.5.1 After initial precipitation, STL-St. Louis decants the supernate after precipitation has been allowed to settle for at least six hours, as opposed to the EPA Method 904 which requires filtration to isolate the precipitate.
 - 1.5.2 At the point of ingrowth of Actinium-228, STL St. Louis waits 14 days for wastewater and 21 days for drinking water before finishing the procedure. This allows unsupported Ra-224 to decay away to less than ten percent of initial activity.
 - 1.5.3 STL St. Louis counts the Barium sulfate fraction (minus the Ra-224) by GFPC to report Radium-226; a possibility proscribed in section 10.5 of the EPA Method 903.3, "alpha-emitting Radium Isotopes in Drinking Water."

2.0 SUMMARY OF METHOD

- 2.1 Radium isotopes are collected by coprecipitation with barium and lead sulfate and purified by precipitation from EDTA solution. After a suitable ingrowth period, ^{228}Ac is separated and carried on yttrium oxalate, purified and counted for the presence of total beta radiation. The precipitation and counting are performed in a manner consistent with the time requirements of the 6.13 hour half life of ^{228}Ac . By applying correction factors for ingrowth and decay and appropriately calibrating the beta counter, ^{228}Ra is quantified.

3.0 DEFINITIONS

- 3.1 See the STL Quality Management Plan (QMP) and STL St. Louis Laboratory Quality Manual (LQM) for a glossary of common terms and data reporting qualifiers.

4.0 INTERFERENCES

- 4.1 Strontium 90 (^{90}Sr) or other beta emitting radionuclides that are carried by the yttrium oxalate precipitate (ie. certain mixed fission or activation products) will yield a positive bias to the ^{228}Ra values.

4.2 Samples which contain excess barium could cause inaccurate chemical yield determinations.

4.3 Excessive barium chemical yields may also be caused by improper handling. The BaSO_4 can be redissolved and precipitated a second time to check this.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

None.

5.3 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Ammonium Hydroxide	Poison Corrosive	50 ppm (NH_3)	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
Acetic Acid, Glacial	Corrosive Flammable	10 ppm (TWA)	Inhalation causes respiratory tract irritation including nasal discharge, hoarseness, coughing, chest pain and breathing difficulty. Skin contact symptoms may include redness or discoloration, swelling, itching, burning or blistering of skin. Eye symptoms include irritation, burning sensation, pain, watering, and/or change of vision.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3- TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 EQUIPMENT AND SUPPLIES

- 6.1 Low background gas proportional counter.
- 6.2 50ml Centrifuge tubes
- 6.3 Centrifuge
- 6.4 Hot Plate
- 6.5 Analytical balance
- 6.6 Stainless steel planchettes
- 6.7 Glassware as appropriate
- 6.8 Syringe filters 20ml

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002, current revision.
- 7.2 Distilled or deionized water, ASTM II (1991) from the Millipore unit.
- 7.3 Acetic acid, 17.4N: glacial CH_3COOH (concentrated), specific gravity 1.05, 99.8%.
- 7.4 Ammonium hydroxide, 15N: NH_4OH (concentrated), sp. gr. 0.90, 56.6%.
- 7.5 Ammonium oxalate, 5%: Dissolve 5g $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$ in water and dilute to 100ml.
- 7.6 Ammonium sulfate, 200mg/ml: Dissolve 20g $(\text{NH}_4)_2\text{SO}_4$ in water and dilute to 100ml.
- 7.7 Ammonium sulfide, 2%: Dilute 10ml $(\text{NH}_4)_2\text{S}$, (20-24%), to 90 ml water; total volume 100ml.
- 7.8 Barium carrier, 33.9 mg/ml, standardized.

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- 7.9 Citric acid, 1M: Dissolve 19.2g $C_6H_8O_7 \cdot H_2O$ in water and dilute to 100ml.
- 7.10 EDTA reagent basic (0.25M) - dissolve 20g NaOH in 750ml water, heat and slowly add 93g [ethylenedinitrilo] tetraacetic disodium salt, ($C_{10}H_{14}O_8N_2Na_2 \cdot 2H_2O$) while stirring. Dilute to 1 liter.
- 7.11 Lead carrier, 15mg/ml: Dissolve 2.397g $Pb(NO_3)_2$ in water, add 0.5 ml 16N HNO_3 and dilute to 100ml with water.
- 7.12 Lead carrier, 1.5mg/ml: Dilute 10ml lead carrier, (15mg/ml), to 100ml with water.
- 7.13 Methyl orange indicator, 0.1%: Dissolve 0.1g methyl orange indicator in 100ml water.
- 7.14 Nitric acid, 16N: HNO_3 (concentrated), specific gravity 1.42, 70.4%.
- 7.15 Nitric acid, 6N: Mix 3 volumes 16N HNO_3 (concentrated) with 5 volumes of water.
- 7.16 Nitric acid, 2N: Mix 1 volume 6N HNO_3 with 2 volumes of water.
- 7.17 Sodium hydroxide, 18N: Dissolve 72g NaOH in water and dilute to 100ml.
- 7.18 Sodium hydroxide, 10N: dissolve 40g NaOH in water and dilute to 100ml.
- 7.19 Strontium carrier, 10 mg/ml: Dissolve 24.16g $Sr(NO_3)_2$ in water and dilute to 1 liter.
- 7.20 Sulfuric acid, 18N: Cautiously mix 1 volume 36N H_2SO_4 (conc.) with 1 volume of water.
- 7.21 Yttrium Carrier, 18.2 mg/ml, standardized.
- 7.22 Yttrium carrier, 9 mg/ml: Dilute 50 ml yttrium carrier, (18 mg/ml), to 100 ml with water.
- 7.23 Strontium-yttrium mixed carrier, 0.9 mg/ml Sr^{+2} , 0.9 mg/ml Y^{+3} :
 - 7.23.1 Solution A: Dilute 10.0 ml yttrium carrier, (18 mg/ml), to 100 ml
 - 7.23.2 Solution B: Dissolve 0.4348 g $Sr(NO_3)_2$ in water and dilute to 100 ml.
 - 7.23.3 The mixed carrier is made by adding equal volumes to a flask.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 STL St. Louis supplies sample containers and chemical preservatives in accordance with the method. STL St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in STL-PM-0002.
- 8.2 All samples may be collected in glass or plastic containers.
- 8.3 Preserve all aqueous samples with nitric acid to a pH of less than 2.

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8.4 Samples should be stored for no more than 180 days.

9.0 QUALITY CONTROL

9.1 Batch

9.1.1 Definition: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.1.3 Each analytical batch may contain up to 20 environmental samples, a method blank, and a single Laboratory Control Sample (LCS) and a Sample Duplicate (SD). In the event that there is insufficient sample to analyze a Sample Duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 A matrix spike is not routinely performed for analyses utilizing a tracer or carrier. A matrix spike may be performed by client request.

9.1.4 Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.2 Method Blank

9.2.1 Definition: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.2.2 A method blank must be prepared with every batch (20 or fewer samples of the same matrix).

9.2.3 The matrix for the method blank is DI water.

9.3 Laboratory Control Sample

9.3.1 Definition: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 An LCS must be prepared with every batch.

9.3.3 The matrix for the LCS is DI water.

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9.4 Matrix Spike

- 9.4.1 Matrix Spike Definition: An aliquot of a field sample to which a known amount of target analyte(s) is added.
- 9.4.2 Additional MS and sample duplicates do not count towards the 20 samples in an analytical batch.
- 9.4.3 An MSD can be prepared in lieu of a sample duplicate. If there is insufficient sample to perform an MS/MSD, a duplicate LCS is analyzed.

9.5 Sample Duplicate

- 9.5.1 Sample Duplicate Definition: An additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.
- 9.5.2 If there is insufficient sample to perform a Sample Duplicate, a duplicate LCS is analyzed.

9.6 Procedural Variations

- 9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance Memo shall be filed in the project file and incorporated into the report narrative.

9.7 Nonconformance and Corrective Action

- 9.7.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager. See SOP STL-QA-0036 for details regarding the NCM process.

10.0 CALIBRATION AND STANDARDIZATION

- 10.1 Balance and thermometer calibration must be checked daily when used. Refer to SOP STL-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes Procedure.
- 10.2 See the analytical SOP for instrument calibration; STL-RD-0403, "Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System."

11.0 PROCEDURE

- 11.1 Confirm that sample is acidic, pH less than 2 using pH paper. If not acidic, add 2ml 16N HNO₃ per liter of sample and mix thoroughly. Notify laboratory supervisor and initiate a Nonconformance.
- 11.2 Ensure that sample container is capped tightly and shake it thoroughly. Transfer an aliquot of the sample (typically 1 liter) to an appropriate size beaker. Label beaker with sample ID number and volume. Record all data on sample worksheet.
- 11.2.1 NOTE: The sample volume may vary according to the contract required detection limits. Review the Quality Assurance Summary for additional information.

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- 11.3 Add 1M citric acid in ratio of 5 ml per liter. Add methyl orange indicator until the persistence of a red color. Mix thoroughly.
- 11.4 Add 2.5 ml lead carrier (15 mg/ml), 2ml strontium carrier (10 mg/ml), 1.0 ml barium carrier (33.9 mg/ml), and 1 ml yttrium carrier (18 mg/ml); stir well. Heat to incipient boiling and maintain at this temperature for about 30 minutes.
- 11.5 Add 15N ammonium hydroxide until a definite yellow color is obtained, then add a few drops excess. Precipitate lead and barium sulfates by adding 18N sulfuric acid until the red color reappears, then add 0.25 ml excess. Add 5 ml ammonium sulfate (200 mg/ml) for each liter of sample. Stir frequently and keep at a temperature of approximately 90°C for 30 minutes.
- 11.6 Cool sample for at least 30 minutes. Allow precipitate to settle to the bottom of the beaker for a least 6 hours. Decant the supernatant and discard, taking care to avoid disturbing the precipitate.
- 11.7 Quantitatively transfer precipitate to a 50 ml centrifuge tube, taking care to rinse last particles out of beaker with a strong jet of deionized water. Centrifuge and discard supernatant.
- 11.8 Wash the precipitate with 10ml 16N HNO₃, vortex, centrifuge, and discard supernate.
- 11.9 Repeat Step 11.10 one time.
- 11.10 Wash the precipitate with 10ml D.I.H₂O, vortex, centrifuge, and discard supernate.
- 11.11 Add 20 ml basic EDTA reagent; vortex thoroughly, and heat in a hot water bath (approximately 80°C) until precipitate dissolves.
 - 11.11.1 If insoluble solids remain in the tube after addition of EDTA, confirm that the pH is > 10. If > 10, centrifuge and decant supernate into a clean, labeled 50 mL centrifuge tube. Discard insoluble residue.
- 11.12 Add 1 ml strontium-yttrium mixed carrier and stir thoroughly. Add a few drops 10N NaOH if any precipitate forms.
- 11.13 Add 2ml ammonium sulfate (200mg/ml) and stir thoroughly. Add 17.4N acetic acid until barium sulfate precipitates, then add 2 ml excess. Digest in a hot water bath until precipitate settles. Centrifuge and discard supernatant.
- 11.14 Add 20 ml basic EDTA reagent, vortex thoroughly, and heat in a hot water bath until precipitate dissolves. Repeat steps 11.14 through 11.15. Note the time of last barium sulfate precipitation. This is the beginning of the ²²⁸Ac ingrowth time. Record the date and time on the sample worksheet.
- 11.15 Dissolve the precipitate in 20 ml basic EDTA reagent as before, then add 1.0 ml standardized yttrium carrier and 1 ml lead carrier (1.5 mg/ml). If any precipitate forms, dissolve it by adding a few drops of 10N NaOH. Cap the tube and allow it to age at least 36 hours.

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- 11.16 Add 0.3 ml ammonium sulfide and stir well. Add 10N sodium hydroxide drop-wise with vigorous stirring until lead sulfide precipitates, then 10 drops excess. Stir intermittently for about 10 minutes. Centrifuge and decant supernatant into a clean tube.
- 11.17 Add 1 ml lead carrier (1.5 mg/ml), 0.1 ml ammonium sulfide, and a few drops 10N sodium hydroxide. Repeat precipitation of lead sulfide as before. Centrifuge and filter supernatant through 0.45 mm syringe filter into a clean tube. Wash filter with approximately 5ml water. Discard residue.
- 11.18 Check availability of gas proportional counter.
- 11.19 Once yttrium hydroxide is precipitated, the analysis must be carried to completion to avoid excessive decay of ^{228}Ac .
- 11.20 Ensure that the hot water bath is at the desired temperature, 70-85°C.
- 11.21 Add 7 ml 18N sodium hydroxide, stir well and digest in a hot water bath until yttrium hydroxide coagulates, usually about 5 minutes. Centrifuge and decant supernatant into a clean, labeled 50 ml centrifuge tube. Save for barium yield determination, Step 11.29.
- 11.22 Note time of yttrium hydroxide precipitation; this is the end of the ^{228}Ac ingrowth time and beginning of ^{228}Ac decay time. Record time on the sample data sheet. (End of Ingrowth).
- 11.23 Dissolve the precipitate in 2ml 6N nitric acid. Vortex and add 5ml water and precipitate yttrium hydroxide with 3 ml 10N sodium hydroxide. Heat and stir in a hot water bath until precipitate coagulates. Centrifuge and discard supernate.
- 11.24 Dissolve precipitate with 1 ml 2N nitric acid. Vortex, if solution is still cloudy add 2N nitric acid dropwise until the solution clears. Dilute to 5ml with DI water and add 2ml 5% ammonium oxalate.
- 11.25 Centrifuge and discard supernate.
- 11.26 To determine yttrium yield, quantitatively transfer the precipitate to a tared stainless steel planchet using a minimum amount of water. Dry with a heat source to constant weight and count in a gas flow proportional counter for total beta radiation. Record tare and gross weights on sample worksheets.
- 11.27 To the supernatant from Step 11.23; add 5 ml 16N nitric acid and 2ml ammonium sulfate (200mg/ml), stirring well after each addition. Add 17.4N acetic acid until barium sulfate precipitates, then add 2ml excess. Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate.
- 11.28 Add 20ml basic EDTA reagent, vortex and heat in a hot water bath until precipitate dissolves. Add a few drops 10N NaOH if precipitates does not readily dissolve.
- 11.29 Add 2 ml ammonium sulfate (200 mg/ml) and stir thoroughly. Add 17.4N acetic acid until barium sulfate precipitates, then add 2 ml excess.

11.29.1 NOTE: If ^{226}Ra is requested, record date and time of BaSO_4 on ^{226}Ra data sheet.

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- 11.30 Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate.
- 11.31 Wash precipitate with 10ml water. Vortex, centrifuge and discard supernate. Repeat this step once.
- 11.32 Transfer precipitate to a tared stainless steel planchet with a minimum amount of water.
- 11.33 Dry on a hot plate on medium heat. Cool planchets in a dessicator. Weigh the planchet.
- 11.34 Heat the planchet again using the hot plate. Weigh the planchet a second time to confirm that the weight of the planchet has not changed ($\pm 5\%$).
- 11.35 Repeat steps 11.31 and 11.32 until the weight of the planchet is constant.
- 11.36 Record the final weight of the planchet to determine the chemical recovery for the barium carrier solution.
- 11.37 Submit the planchets to the counting room for total alpha radiation.

12.0 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery, RPD, MDA) and standard instrument software calculations are given in the STL St. Louis LQM.

13.0 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF DATE DATA

- 13.1 The data assessment and corrective action process is detailed through the Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: STL-QA-0036. A hardcopy of all the data assessment types and descriptions along with their associated corrective actions is included in that SOP.
- 13.2 See Analytical SOP STL-RD-0403, "Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System."

14.0 METHOD PERFORMANCE

- 14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix to this SOP.
- 14.2 Demonstration of Capability
 - 14.2.1 Initial and continuing demonstrations of capability requirements are established in STL St. Louis' LQM section 5.1.2
- 14.3 Training Qualification
 - 14.3.1 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
 - 14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in STL St. Louis' LQM section 5.1.2

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14.3.3 Annually the analyst must successfully demonstrate proficiency to continuing to perform this analysis. See requirements in STL St. Louis' LQM section 5.1.2

15.0 VALIDATION

- 15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods.

16.0 WASTE MANGEMENT AND POLLUTION PREVENTION

- 16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
- Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the labware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the labware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17.0 REFERENCES

- 17.1 Radium 228 in Drinking Water, Method 904.0, Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 8, EPA 600/4-30-032 (1980).
- 17.2 Percival, D. R. and Martin, D. B., "Sequential Determination of Radium-226, Radium-228, Actinium-227, and Thorium Isotopes in Environmental and Process Waste Samples," Analytical Chemistry, 46-1742-2749, (1974).
- 17.3 STL Quality Management Plan (QMP)
- 17.4 STL St. Louis Laboratory Quality Manual (LQM)
- 17.5 STL Corporate Safety Manual and St. Louis Facility Addendum (SOP STL-HS-0002), current revision.
- 17.6 Associated SOPs:
- 17.6.1 STL-QA-0002, Standard and Reagent Preparation
- 17.6.2 STL-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
- 17.6.3 STL-RC-0002, Planchet Preparation for Radiochemistry and Radiological Screening Analysis
- 17.6.4 STL-QA-0036, Non-conformance Memorandum (NCM) Process

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17.6.5 STL-RD-0403, Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System

17.6.6 STL-PM-0002, Sample receipt and Chain of Custody

17.6.7 STL-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes

18.0 CHANGES FROM PREVIOUS REVISION

- 18.1 Revised Safety, section 5 and hazard tables in accordance with CSM.
- 18.2 Merged and revised waste management and pollution prevention sections, Section 16.
- 18.3 Added text to address sample collection references and capabilities, Section 8.
- 18.4 Added text to Section 12 referencing commonly used calculations are in the LQM.
- 18.5 Added DOC reference information to the method performance Section 14.
- 18.6 Created a "Validation Data" section, Section 15.
- 18.7 Revised Quality Control, Section 9.
- 18.8 References, section 17 revised.
- 18.9 Added method reference to Scope and Application section

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

TITLE: Isotopic Americium, Curium, Plutonium, Thorium and Uranium in Various Matrices by EICrom® Separation Resins

(Supersedes: STL-RC-0240 Rev 4)

Prepared by:

Approved by:

Supervisor/Lead Analyst

Approved by:

Quality Assurance Manager

Approved by:

Environmental Health and Safety Coordinator

Approved by:

Laboratory Director

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1 SCOPE AND APPLICATION

- 1.1 This SOP provides rapid, reliable method for measurement of Thorium and Uranium in water, soil, sludge, filter, biota and oil/grease samples. This method is more cost-effective and more efficient than traditional ion exchange, solvent extraction and precipitation techniques.
- 1.2 This method is based on Eichrom Technologies Inc. analytical procedures "ACS04: Americium/Lanthanide Separation in Soil" and "ACW01 Uranium and Thorium in Water".
- 1.3 Method detection limits are not applicable to this procedure.
- 1.4 The reporting limits, method detectable activities and QC limits are maintained in the Information Management System (QuantIMS). Because of their dynamic nature, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools. A copy of the SAC is included in the analytical SOP to demonstrate this information.

2 SUMMARY OF METHOD

- 2.1 This SOP describes the method for separation of Americium, Curium, Plutonium, Thorium and Uranium using Eichrom resin prior to measurement by alpha spectrometry. A calcium phosphate precipitation technique is used to concentrate and remove actinides from water samples. Soils, Sludge and Filters are prepared for analysis using STL-RC-0004, Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis. Tracers are used to correct for chemical recovery and correct results to improve precision and accuracy.

3 DEFINITIONS

- 3.1 See the STL Quality Management Plan (QMP) and STL St. Louis Laboratory Quality Manual (LQM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 Minimum Detectable Activity (MDA) - The smallest amount of activity that can be detected given the conditions of a specific sample. It is reported at the 95% confidence interval, meaning that there is a 5% chance that a false signal was reported as activity and a 5% chance that true activity went undetected.
- 3.3 Tracer - A known amount of either ^{232}U , ^{229}Th , ^{241}Am , ^{243}Am , ^{242}Pu , or ^{236}Pu , individually or in combination, is added to each sample to determine chemical yield. The tracer serves as an internal standard, which is used to calculate the activity of the target isotopes.

4 INTERFERENCES

- 4.1 Actinides with unresolvable alpha energies such as Am-241, U-232, Th-228 and Pu-238 must be chemically separated to enable measurement. This method separates these isotopes effectively.
- 4.2 Very high levels of phosphate in the sample may cause an interference. Adjusting the amount of phosphate added to coprecipitate the actinides may be necessary in these cases.
- 4.3 Np-237 can interfere with the Pu-242 peak if not removed from the column before eluting Plutonium.
- 4.4 Samples with high amounts of Fe^{3+} (soils) will interfere with Am recoveries on Tru resin if not reduced to Fe^{2+} with sulfamic and ascorbic acid after Pu and Th are loaded on TEVA.

5 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

None.

5.3 PRIMARY MATERIALS USED

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Ammonium Hydroxide	Poison Corrosive	50 ppm (NH ₃)	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
Ammonium oxalate	Poison Corrosive	None established	Symptoms of inhalation exposure include nervousness, cramps and central nervous system depression. Skin contact causes redness, itching, and pain. Eye contact causes irritation, redness, and pain.
Calcium nitrate	Oxidizer	None established	Inhalation symptoms include coughing and shortness of breath. Skin contact symptoms include redness, itching, and pain. Eye contact causes irritation, redness and pain.
Formic acid	Corrosive	5 ppm (TWA)	Inhalation of vapors can cause severe irritation of nose, throat, and upper respiratory tract. Skin contact symptoms are redness, pain and severe burning. Vapors are irritating to the eyes and may cause damage.
Hydrochloric Acid	Poison Corrosive	5 ppm Ceiling	Inhalation symptoms include coughing, choking, inflammation of the nose, throat, and upper respiratory tract. Skin contact can cause redness, pain, severe skin burns, and discoloration. Vapors are irritating to the eyes. Contact may cause severe burns.
Nitric Acid	Corrosive Poison Oxidizer	2 ppm, 5 mg/m ³	Inhalation may cause coughing, choking, and irritation of the nose, throat, and respiratory tract. Skin contact can cause redness, pain, and severe skin burns. Concentrated solutions can stain the skin a yellow-brown color. Vapors are irritating to the eyes and contact may cause severe burns.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6 EQUIPMENT AND SUPPLIES

6.1 Beakers, 150-2000 mL

6.2 Analytical balance - 0.0001 g sensitivity

6.3 Centrifuge

- 6.4 Centrifuge tubes, poly, 50 mL with cap
- 6.5 Pipets, glass or plastic, disposable
- 6.6 Pipetter, mechanical
- 6.7 Fume hood
- 6.8 Hotplate with stirrer
- 6.9 Vortex mixer
- 6.10 pH strips, narrow range
- 6.11 Vacuum Box, Eichrom part number AC-24-BOX, or equivalent
- 6.12 Syringe filter, 25 mm acrodisc, 0.45 or 0.70 μm
- 6.13 Cartridge reservoirs/syringe/funnel-20 mL B-D Luer Lok syringe Part Number 301625 (Fisher part number 14-823-2B), or equivalent.

7 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002, current revision.
- 7.2 Deionized (DI) Water, obtained from the Milli-Q unit.
- 7.3 Am rare earth load (AmLoad), 2M ammonium thiocyanate in 0.1M formic acid: 304.5 g of NH_4SCN + 8.5 mL HCOOH diluted to 2 L with water.
- 7.4 Ammonium bioxalate, $(\text{NH}_4)\text{HC}_2\text{O}_4$, (0.1M): Dissolve 14.2 g of ammonium oxalate, $(\text{NH}_4)_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$, and 12.6 g of oxalic acid, $\text{HOCCOOH} \cdot 2\text{H}_2\text{O}$ in 2 L of water. **CAUTION – Ammonium oxalate is harmful if inhaled. Causes severe irritation to eyes, skin, and respiratory tract. Oxalic acid is corrosive. Causes severe irritation and burns to skin, eyes, and respiratory tract.**
- 7.5 Ammonium hydrogen phosphate (3.2M) - Dissolve 106 g of $(\text{NH}_4)_2\text{HPO}_4$ in 200 mL of water, heat gently to dissolve, and dilute to 250 mL with water.
- 7.6 Ammonium hydroxide (14.7M) - concentrated (sp gr 0.9)
 - 7.6.1 Ammonium hydroxide (5 wt %) - Mix 50 mL concentrated ammonium hydroxide into 950 mL water. **CAUTION - Corrosive. Mist and vapor cause burns to every area of contact.**
- 7.7 Ammonium Oxalate, reagent crystals.
- 7.8 Ammonium Thiocyanate, reagent crystals.
- 7.9 L (+) Ascorbic Acid, reagent powder.
- 7.10 L (+) Ascorbic Acid solution, 2.5 g dissolved in 10 mL of DI water.

- 7.11 Bromocresol Purple indicator solution – Dissolve 0.20 g of Bromocresol Purple (520.24 F.W.) in 250 mL of water, add one mL of concentrated Ammonium Hydroxide.
- 7.12 Calcium nitrate (1.25M) - Dissolve 51 g of $\text{Ca}(\text{NO}_3)_2$ in 100 mL of water and dilute to 250 mL with water.
- 7.13 Cresol Red indicator solution – Dissolve 0.10g of Cresol Red indicator in 75ml of water, add 2.65ml of 0.1N Sodium Hydroxide, mix and dilute to 100ml.
- 7.14 Formic acid, 90 %, laboratory grade.
- 7.15 Hydrochloric acid (12M) - concentrated HCl (sp gr 1.19).
 - 7.15.1 Hydrochloric acid (9M) - Add 1500 mL of concentrated HCl (sp gr 1.19) to 500 mL of water.
 - 7.15.2 Hydrochloric acid (6M) - Add 1000 mL of concentrated HCl (sp gr 1.19) to 1000mL of water.
 - 7.15.3 Hydrochloric acid (4M) - Add 667 mL of concentrated HCl (sp gr 1.19) to 1330mL of water.
 - 7.15.4 Hydrochloric acid (1M) - Add 167 mL of concentrated HCl (sp gr 1.19) to 1833mL of water.
 - 7.15.5 Hydrochloric acid (0.24M) - Add 20 mL of concentrated HCl (sp gr 1.19) to 900mL of water and dilute to 1L with water.
 - 7.15.6 Hydrochloric acid (0.10M) - Add 0.80 mL of concentrated HCl (sp gr 1.19) to 900 mL of water and dilute to 1 L with water.
- 7.16 Hydrochloric acid (5M) - 0.05M oxalic acid solution - Add 12.6 grams of oxalic acid dihydrate in approximately 800 mL of water. Add 834 mL of concentrated hydrochloric acid. Dilute to 2 liters, add a stir bar and place on a stir plate until oxalic is completely dissolved.
- 7.17 Hydrochloric acid (1M) - 0.03M Oxalic acid solution - Add 167 mL of concentrated HCl to approximately 1500 mL of H_2O and mix; then add 7.6 g of oxalic acid dihydrate, $\text{HOOC-COOH} \cdot 2\text{H}_2\text{O}$, and dilute to 2 L. Shake to dissolve the oxalic acid.
- 7.18 Lead Nitrate (Reagent, crystals).
 - 7.18.1 Lead Nitrate 1 % solution. Dissolve 1 g of lead nitrate crystals in 100 mL of DI water.
- 7.19 Nitric acid (16M) – concentrated HNO_3 (sp gr 1.42)
 - 7.19.1 Nitric acid (3M) – add 382ml of concnetrated nitric acid to 1618ml of water and dilute to 2L
 - 7.19.2 Load Solution [Nitric acid (3M) –aluminum nitrate (1M)] – weight 1500g $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ in a 4 liter beaker. Add 800ml of water (first) and 764ml of concentrated nitric acid. Dilute to 4L with water. Add stir bar, cover with watch glass and place on stir plate until aluminum nitrate is dissolved.
- 7.20 Potassium Hydroxide pellets

- 7.20.1 Potassium Hydroxide (2M) - dissolve 11.22 g of potassium hydroxide pellets in 100 mL of DI water.
- 7.21 Potassium Sulfate (Reagent Crystals).
- 7.22 Oxalic acid, reagent, crystals.
- 7.23 Sodium Nitrite, NaNO_2 reagent crystals.
 - 7.23.1 Sodium Nitrite solution, Dissolve 1.0 grams NaNO_2 in 10 mL of DI water.
- 7.24 Sulfamic acid, $\text{NH}_2\text{SO}_3\text{H}$.
 - 7.24.1 Sulfamic acid solution, $\text{NH}_2\text{SO}_3\text{H}$, Dissolve 2.0 grams sulfamic acid in 10 mL of DI water.
- 7.25 Sulfuric acid (18M).
 - 7.25.1 Sulfuric acid (1M)-slowly add 2.8 mL of 18M sulfuric acid to 90 mL of water, dilute to 100 mL.
- 7.26 Titanium trichloride, TiCl_3 , 10% solution, commercially available.
- 7.27 TEVA Resin - prepacked column, 100-150 micron resin, or 50-100 micron prepacked cartridges.
- 7.28 TRU Resin-prepacked column, 100-150 micron resin, or 50-100 micron prepacked cartridges.
- 7.29 Am-243 tracer, NIST traceable, approximately 10-20 dpm/mL.
- 7.30 Am-243 spike, NIST traceable, approximately 10-20 dpm/mL.
- 7.31 Am-241 spike, NIST traceable, approximately 10-20 dpm/mL.
- 7.32 Curium-244 tracer, NIST tracable, approximately 10-20 dpm/mL.
- 7.33 Plutonium 242 tracer (Pu-236 can also be used), NIST tracable, , approximately 10-20 dpm/mL.
- 7.34 Plutonium 238 spike, NIST traceable, approximately 10-20 dpm/mL.
- 7.35 Plutonium 239/240 spike, NIST traceable, approximately 10-20 dpm/mL.
- 7.36 Thorium-229, NIST traceable 10-20 dpm/mL.
- 7.37 Natural Thorium spike standard (Th-232/Th-228), NIST traceable, approximately 10-20 dpm/mL.
- 7.38 Th-230 spike standard, NIST traceable, approximately 10-20 dpm/mL.
- 7.39 Clean Uranium-232 tracer standard (free of Th-228 daughter, removed by lead sulfate precipitation, activity verified before use), NIST traceable, approximately 10-20 dpm/mL.
- 7.40 Clean U-232 tracer standard: U-232 is used as a tracer for Uranium isotopic analysis. Th-228 is the first daughter of U-232. Thorium isotopic analysis includes Th-232, Th-230 and Th-228. Due to the presence of the Th-228 daughter (Th contamination) in a U-232 solution, a sequential

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Uranium/Thorium analysis requires a Thorium free U-232 standard. A Th-228 free U-232 standard may be made as described below:

- 7.40.1 Dilute the appropriate aliquot of stock standard to about 40 mL with water.
- 7.40.2 Add 3 grams of potassium sulfate.
- 7.40.3 Add a few drops of Cresol Red solution.
- 7.40.4 Adjust the pH to 1.5 using narrow range pH strips with either 2M H₂SO₄ or 2M KOH.
- 7.40.5 While mixing, slowly add 25 mL of 1% Pb(NO₃)₂.
- 7.40.6 Adjust the pH to 1.5 using narrow range pH strips with either 2M H₂SO₄ or 2M KOH.
- 7.40.7 Dilute to 100 mL with water, and mix well. Solution should be spun (at a rate fast enough to form a vortex) continuously for at least 30 minutes to remove any Thorium that maybe in solution.
- 7.40.8 Let stand for at least 1 hour. Centrifuge the solution for 30 minutes. Use the clean U-232 solution as soon as possible after removing the Th-228.
- 7.40.9 Before each use, shake the standard at least 30 minutes (to absorb any ingrown Th-228 onto the sulfate precipitate), and let the precipitate settle (centrifuge). Do not disturb the precipitate while using the standard.

8 SAMPLE COLLECTION, PRESERVATIVES AND STORAGE

- 8.1 STL St. Louis supplies sample containers and chemical preservatives in accordance with the method. STL St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in STL-PM-0002.
- 8.2 Aqueous samples should be preserved at the time of collection by adding sufficient nitric acid to a pH < 2.
- 8.3 If samples are collected without acidification, they should be brought to the laboratory within 5 days, nitric acid added to bring the pH to 2 or less, the sample shaken, and then held for a minimum of 24 hours in the original container before analysis or transfer of sample. If dissolved or suspended material is to be analyzed separately, do not acidify the sample before filtering the sample. The filtering may be performed in the field by the customer or by the laboratory.
- 8.4 Samples may be collected in either plastic or glass containers.
- 8.5 Samples can be stored for no more than 180 days unless specified by the client.
- 8.6 Solid sample requirements are found in SOP STL-RC-0004, "Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis".

9 QUALITY CONTROL

9.1 Batch

- 9.1.1 Definition: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one

to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, volatile organics, water) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

- 9.1.2 Instrument conditions must be the same for all standards, samples and QC samples.
- 9.1.3 Each analytical batch may contain up to 20 environmental samples, a method blank, a single Laboratory Control Sample (LCS), and Sample Duplicate. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
- 9.1.4 Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.2 Method Blank

- 9.2.1 Definition: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.2.2 A method blank must be prepared with every batch (20 or fewer samples of the same matrix).
- 9.2.3 A method blank consists of DI water. For non-aqueous method blanks see soil preparation SOP, STL-RC-0004, for details.

9.3 Laboratory Control Sample

- 9.3.1 Definition: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.
- 9.3.2 An LCS must be prepared with every batch.
- 9.3.3 The LCS consists of DI water fortified with tracer and spikes. For soils, either a solid reference material or a fortified solution of calcium nitrate is used. See soil preparation SOP, STL-RC-0004, for details.

9.4 Matrix Spike/Sample Duplicate

- 9.4.1 Yield monitors (radiological tracers) are used to determine analyte recovery during sample analysis. Matrix Spike (MS) samples typically are not required when radiometric tracers are used as yield monitors. Matrix Spikes may be performed if specifically requested by the client.
- 9.4.2 Matrix Spike Definition: An aliquot of a field sample to which a known amount of target analyte(s) is added.

9.4.3 Sample Duplicate Definition: An additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.4.4 Additional MS and sample duplicates do not count towards the 20 samples in an analytical batch.

9.5 Procedural Variations

9.5.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance Memo shall be filed in the project file and incorporated into the report narrative.

9.6 Nonconformance and Corrective Action

9.6.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager. See SOP STL-QA-0036 for details regarding the NCM process.

10 CALIBRATION AND STANDARDIZATION

10.1 Balance and automatic pipetter calibrations must be checked daily when used. Refer to SOP STL-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."

11 PROCEDURE

11.1 For NON-AQUEOUS matrices (soil, oil, biota, etc) see SOP: STL-RC-0004 for initial sample preparation and proceed to section 11.5 of this SOP.

11.2 Water Sample Preparation:

11.2.1 If not already pre-filtered, and the client requested analysis on a filtered fraction, filter the sample through a 0.45 micron filter. If the sample contains a large amount of sediment which would not be possible to work with, filter through a Whatman GF/A (1.6 micron) filter.

11.2.2 Aliquot 500 to 1000 mL of the filtered sample (or enough to meet required detection limit) into an appropriate size beaker. Verify acid pH less than 2 while in original sample bottle. If not, acidify sample pH to less than 2 with concentrated nitric acid. Allow acidified sample to sit in original bottle at least 24 hours before proceeding with analysis.

11.2.3 Add appropriate tracers or standards. Generally 10 – 20 dpm of the Thorium, Americium, Curium, Uranium and/or Plutonium tracers are added. Check radscreen results or customer/site history in determining sample size.

11.3 Evaporation option to reduce volume:

11.3.1 This option may be used when large sample volumes are needed to achieve low level reporting limits.

11.3.2 Evaporate sample to less than 50 mL and transfer to a 100 mL beaker. Note: For some water samples, calcium sulfate formation may occur during evaporation. If this occurs, use the calcium phosphate precipitation option, step 11.4.1

11.3.3 Gently evaporate the sample to dryness and redissolve in approximately 5 mL of concentrated HNO_3 (sp gr 1.42).

11.3.4 Repeat step 11.3.3 two more times, evaporate to dryness and proceed step 11.5.1.

11.4 Calcium phosphate precipitation:

11.4.1 Add 0.5 mL of 1.25M $\text{Ca}(\text{NO}_3)_2$ to each beaker, then add 200 μL of 3.2M $(\text{NH}_4)_2\text{HPO}_4$ solution to each beaker. Add 3-5 drops Bromocresol Purple indicator to each beaker. Place each beaker on a hotplate. Cover each beaker with a watch glass. Allow the samples to heat to near boiling (a stirbar can be added to help samples boil without bumping) approximately 30 minutes.

11.4.2 Once the samples reach near boiling, take the watch glass off the beaker and turn the heat down to medium.

11.4.3 Add enough concentrated NH_4OH with a squirt bottle to reach the bromocresol purple indicator end point and form $\text{Ca}_3(\text{PO}_4)_2$ precipitate. NH_4OH should be added very slowly, such that the bromocresol purple endpoint is just reached. The 5 % ammonium hydroxide solution can be used as the endpoint is approached. Stir the solution. Allow samples to heat uncovered for another 30 minutes. Remove from the hot plate, allow sample to cool and the precipitate to settle.

11.4.4 If the sample volume is too large to centrifuge the entire sample, allow precipitate to settle until solution can be decanted.

11.4.5 Decant supernatant and discard to waste.

11.4.6 Transfer the precipitate to a centrifuge tube and centrifuge the precipitate for approximately 10 minutes at 2000 rpm. Decant supernatant and discard to waste.

11.4.7 Using a disposable pipette, add 3 drops of concentrated nitric acid to the precipitate for every 1 mL of precipitate in bottom of centrifuge tube. (Note: This is to keep from neutralizing the load solution.) Proceed to 11.5.1.

11.5 Thorium/Plutonium/Americium/Uranium Separation using Eichrom resins.

11.5.1 Dissolve precipitate (calcium phosphate or iron hydroxide or calcium oxalate), soil dissolution residue or evaporated water sample with 15 mL of 3M HNO_3 - 1M $\text{Al}(\text{NO}_3)_3$ (Load Solution). Vortex the sample.

Note: Additional 5 mL load solution aliquots may be necessary to dissolve the sample residue. Do not use more than 30 mL of load solution.

Note: If particles are observed or the solution is cloudy, centrifuge the sample at approximately 2000 rpm for 10 minutes. The supernatant will be transferred to a clean labeled centrifuge tube. The precipitates will be discarded. **The use of filtration is also permitted, e.g. syringe filter if the solution is still cloudy.**

Note: Steps 11.5.2 through 11.5.4 are applicable for Plutonium analysis only. If analyzing for Thorium and/or Americium/Uranium (which do not require a valence adjustment), skip to step 11.5.5.

11.5.2 Add 1 mL ascorbic acid solution to the sample load solution in the centrifuge tube and heat in hot water bath for approximately 5 minutes.

- 11.5.3 Add 1 mL NaNO_2 solution to the sample load solution in the centrifuge tube and heat in hot water bath for approximately 5 minutes.
- 11.5.4 Remove samples from hot water bath and let cool in cold water bath until samples are at or slightly below room temperature.
- 11.5.5 **FOR SAMPLES CONTAINING METAL PIECES OR SOILS HIGH IN Fe^{3+} :** Samples such as soils that are high in Fe^{3+} cannot be done sequentially through the TEVA and then the TRU column. The iron will stay oxidized at +3 with the addition of sodium nitrite and interfere with Americium recoveries on TRU. Proceed to section 11.6.

If no high Fe^{3+} adjustment is necessary, continue with step 11.5.6.

- 11.5.6 For each sample dissolved in load solution, place a TRU resin cartridge in the vacuum box. Lock a TEVA Resin cartridge, onto the top of the TRU cartridge. Attach a plastic syringe funnel to the top of the TEVA cartridge.
- 11.5.7 Just prior to loading the sample, pipet 5 mL of 3M HNO_3 into each funnel. Turn on the vacuum pump and attach it to the vacuum box. Open the valves on the box for each cartridge and allow the solution to be pulled through the column. This will condition the resin. The flow rate should be approximately 3 mL per minute. Discard to waste.

Approximately 20 drops equals 1 mL. Use the valve to adjust the flow for each individual sample. Adjust the flow for each solution added!

- 11.5.8 Transfer each sample load solution into the appropriate TEVA/TRU Resin cartridge funnel. Allow to drain. The flow rate should be approximately 1 ml per minute. **Note: the TEVA and the TRU cartridge can turn blue green as the load solution drains through it.**
- 11.5.9 Rinse the funnel with 15 ml of 3M HNO_3 and allow to drain. **Dispose to waste.** (Adjust flow to approximately 3 ml per minute.)
- 11.5.10 Separate TEVA cartridge from TRU cartridge. Place new syringe on the TRU cartridge. **For Thorium and/or Plutonium separation with TEVA cartridge continue with steps 11.6.10. For Americium/Curium and/or Uranium separation proceed to section 11.6.2.**

11.6 Thorium/Plutonium/Americium/Uranium separation via TEVA/TRU with high Fe^{3+} present:

In order to get good recovery on the TRU column for Americium, the samples must first be run through the TEVA column only (no TRU). The the load solution and a 5 mL 3M nitric acid rinse which are collected is then separated by following this section.

Following the Plutonium valence adjustment (section 11.4.2 - 11.4.4):

- 11.6.1 For each sample dissolved in load solution, place a TEVA resin cartridge on the vacuum box. Attach a plastic syringe funnel to the top of the TEVA cartridge.

- 11.6.2 Just prior to loading the sample, pipet 5 mL of 3M HNO₃ into each funnel. Turn on the vacuum pump and attach it to the vacuum box. Open the valves on the box for each cartridge and allow the solution to be pulled through the column. This will condition the resin. The flow rate should be approximately 3 mL per minute. Discard to waste.

Approximately 20 drops equals 1 mL. Use the valve to adjust the flow for each individual sample. Adjust the flow for each solution added!

- 11.6.3 Place a clean labeled empty centrifuge tube into the holder inside the vacuum box to catch the solution.
- 11.6.4 Load sample onto TEVA cartridge,
- 11.6.5 Rinse with 5 mL of 3M nitric acid. Remove labeled tubes (from 11.5.4 and 11.5.5) and proceed to 11.5.7.
- 11.6.6 Move TEVA cartridge to the back row and rinse with 10mL 3M nitric acid. Discard the waste from the other tubes and proceed to Section 11.6.1 for the thorium/plutonium separation.
- 11.6.7 To the load solution and 5 mL rinse removed from the box add 2.5 mL of Sulfamic acid solution slowly while swirling. Solution will bubble as the Nitrite in the sample reacts with the Sulfamic acid.
- 11.6.8 After bubbling subsides, add 3 mL of the ascorbic acid solution.
- 11.6.9 Heat the solution for approximately 3 minutes.
- 11.6.10 Cool the solution to room temperature in a cold water bath.
- 11.6.11 For each sample place a TRU resin cartridge on the vacuum box. Attach a plastic syringe funnel to the top of the TRU cartridge.
- 11.6.12 Just prior to loading the sample, precondition the TRU column by pipeting 5 mL of 3M HNO₃ into each funnel. Turn on the vacuum pump and attach it to the vacuum box. Open the valves on the box for each cartridge and allow the solution to be pulled through the column. The flow rate should be approximately 3 mL per minute. Discard to waste.

Approximately 20 drops equals 1 mL. Use the valve to adjust the flow for each individual sample. Adjust the flow for each solution added!

- 11.6.13 Transfer each sample load solution into the appropriate TRU Resin cartridge funnel. Allow to drain. The flow rate should be approximately 1 mL per minute.
- 11.6.14 Rinse the funnel with 5 mL of 3M HNO₃ and allow to drain. (Adjust flow to approximately 3 mL per minute.)

11.6.15 Proceed to section 11.6.2 to separate Americium and/or Curium and Uranium from TRU resin.

11.7 Thorium/Plutonium/Americium/Uranium elution from Eichrom resins.

11.7.1 Thorium/Plutonium from TEVA

11.7.1.1 Place a clean, labeled 50 mL centrifuge tube below each TEVA cartridge.

11.7.1.2 **Elute Thorium** with 20 mL of 9M HCl into each cartridge into a clean, labeled centrifuge tube. (Adjust flow to approximately 1 mL per minute.)

11.7.1.3 Pipet 5 mL of 6M HCl in each cartridge and collect in the same centrifuge tube as in the previous step. This 6M HCl rinse will strip any residual traces of Th from the cartridge. (Adjust flow to approximately 3 mL per minute.)

11.7.1.4 Transfer the Thorium HCl solution to a clean labeled beaker (save centrifuge tubes) and evaporate to near dryness. Add 5-10mL 1M HCl to beaker and let sit for 15 minutes. Transfer sample back to original centrifuge tube. **To co-precipitate the Thorium**, proceed to STL-RC-0100, "Actinide Coprecipitation."

11.7.1.5 Place a clean, labeled 50 mL centrifuge tube below each TEVA cartridge.

NOTE: For samples high in Neptunium, go to 11.6.1.7, otherwise proceed to 11.6.1.6.

11.7.1.6 **For routine Plutonium**, close the flow control valve. **Elute the Plutonium** with 20 mL of 1M HCl and 0.25 mL of TiCl_3 . Mix the HCl and TiCl_3 by adding 10 mL of the 1M HCl to the column, pipetting 0.25 mL of the TiCl_3 , and then adding the remaining 10 mL 1M HCl. Open the flow control valve and adjust flow to approximately 1 mL per minute. Collect the eluant and proceed to 11.6.1.8.

11.7.1.7 **For samples high in Neptunium-237**, **elute the Plutonium** with 20 mL of 8M HCl and 0.4 mL of TiCl_3 . Mix the HCl and TiCl_3 by adding 10 mL of the 8M HCl to the column, pipetting 0.4 mL of the TiCl_3 , and then adding the remaining 10 mL of 8M HCl. Collect the eluant and proceed to 11.6.1.8.

11.7.1.8 **To co-precipitate the Plutonium**, proceed to STL-RC-0100, "Actinide Coprecipitation."

11.7.2 Americium/Uranium from TRU

- 11.7.2.1 Place a clean, labeled 50 mL centrifuge tube below each TRU cartridge.
- 11.7.2.2 **Elute Americium and/or Curium** from TRU columns with 18 mL of 4M HCl. Collect eluant for Am and/or Curium analysis. (Adjust flow to approximately 1 mL per minute).

To co-precipitate the Americium and/or Curium analysis, proceed to STL-RC-0100, "Actinide Coprecipitation."

NOTE: Samples containing greater than 100 µg total rare earths require further purification for Am and Cm (usually only soil samples of more than 2 grams). Save for the Am and/or Curium purification. Dry samples on hot plate and proceed to section 11.6.3.

- 11.7.2.3 Rinse the TRU columns with 10 mL of 8M nitric acid. This will remove any polonium in the samples that will interfere with the U-232 tracer recoveries.
- 11.7.2.4 Rinse the TRU columns with 20-mL of 1M HCl - 0.03M oxalic acid. Discard rinse. (Adjust flow to approximately 3 mL per minute).
- 11.7.2.5 Place a clean, labeled 50 mL centrifuge tube below each TRU cartridge.
- 11.7.2.6 **Elute Uranium** from TRU columns with 20 mL of 0.1M ammonium bioxalate. Collect eluate in centrifuge tube. (Adjust flow to approximately 1 mL per minute.)

To co-precipitate the Uranium, proceed to STL-RC-0100, "Actinide Coprecipitation."

11.7.3 Lanthanide purification via TEVA resin

- 11.7.3.1 Dissolve the residue in 20 mL of the AmLoad solution by gently heating. Allow the samples to cool to room temperature.
- 11.7.3.2 Load the solution onto an Eichrom TEVA column previously conditioned with 10 mL of AmLoad solution. (Adjust flow to approximately 1 mL per minute.)
- 11.7.3.3 Add 10 mL of AmLoad solution to the original beakers, and heat until just boiling. Allow to cool to room temperature, and load onto the resin. (Adjust flow to approximately 1 mL per minute.)
- 11.7.3.4 Wash the resin with 10 mL of AmLoad solution. (Adjust flow to approximately 1 mL per minute.)

11.7.3.5 **Elute Americium** with 15 mL of 0.24 M HCl. (Adjust flow to approximately 1 mL per minute.) Collect the eluate in centrifuge tubes.

To co-precipitate the Americium and/or Curium analysis, proceed to STL-RC-0100, "Actinide Coprecipitation."

12 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT-OF-CONTROL DATA

12.1 Commonly used calculations (e.g. % recovery, RPD, MDA) and standard instrument software calculations are given in the STL St. Louis LQM.

13 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: STL-QA-0036. A hardcopy of all the data assessment types and descriptions along with their associated corrective actions is included in that SOP.

13.2 See analytical SOP STL-RD-0403.

14 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the appendix to this SOP.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in STL St. Louis' LQM section 5.1.2

14.3 Training Qualification

14.3.1 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in STL St. Louis' LQM section 5.1.2

14.4 Annually the analyst must successfully demonstrate proficiency to continuing to perform this analysis. See requirements in STL St. Louis' LQM section 5.1.2

15 VALIDATION DATA

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods.

16 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".
- Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17 REFERENCES

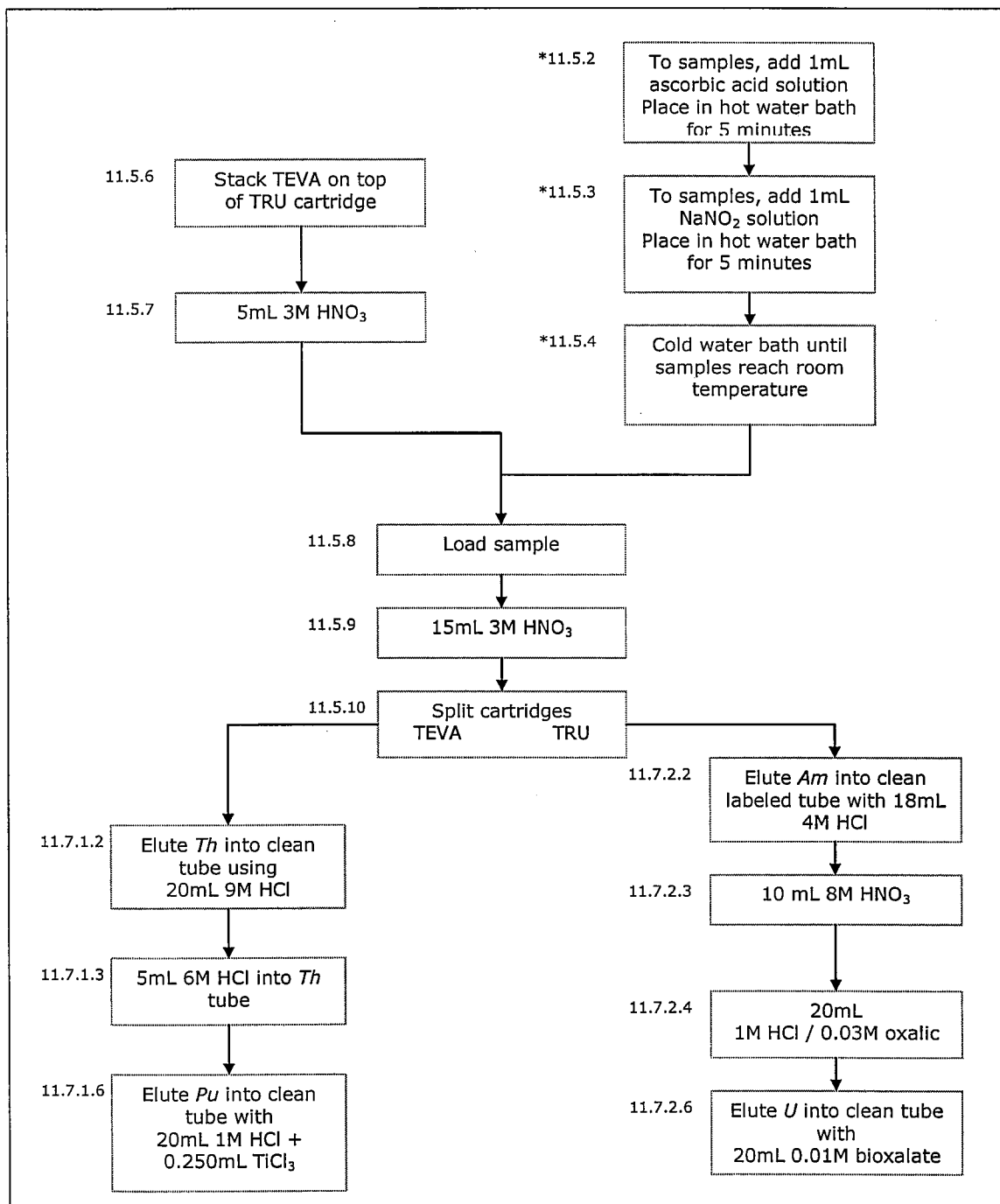
- 17.1 Eichrom Industries, Inc. Analytical Procedures, "ACS04 Americium/Lanthanide Separations in Soil, July 1999
- 17.2 Eichrom Industries, Inc. Analytical Procedures, "ACW01 Uranium and Thorium in Water".
- 17.3 STL Quality Management Plan, current revision.
- 17.4 STL St. Louis Laboratory Quality Manual, current revision.
- 17.5 STL Corporate Safety Manual and St. Louis Facility Addendum (SOP STL-HS-002), current revisions.
- 17.6 Associated SOPs (current revisions)
 - 17.6.1 STL-RD-0210, Daily Operations of an Alpha Spectroscopy System (using AlphaVision Software)
 - 17.6.2 STL-RC-0004, Preparation of Soil Samples for Radiochemical Analysis
 - 17.6.3 STL-RC-0100, Actinide Coprecipitation
 - 17.6.4 STL-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
 - 17.6.5 STL-QA-0002, Standards and Reagent Preparation
 - 17.6.6 STL-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
 - 17.6.7 STL-QA-0036, Non-conformance Memorandum (NCM) Process

18 CHANGES TO PREVIOUS REVISION

- 18.1 Method reference added.
- 18.2 Replaced SOP reference for STL-QA-0006 with STL-PM-0002
- 18.3 Revised Safety, section 5 and hazard tables in accordance with CSM.
- 18.4 Merged and revised waste management and pollution prevention sections, Section 16.
- 18.5 Added text to address sample collection references and capabilities, Section 8.
- 18.6 Added text to Section 12 referencing commonly used calculations are in the LQM.
- 18.7 Added DOC reference information to the method performance Section 14.
- 18.8 Created a "Validation Data" section, Section 15.
- 18.9 Revised procedure in section 11.

Sequential Thorium, Plutonium/Uranium, Americium via TEVA/TRU

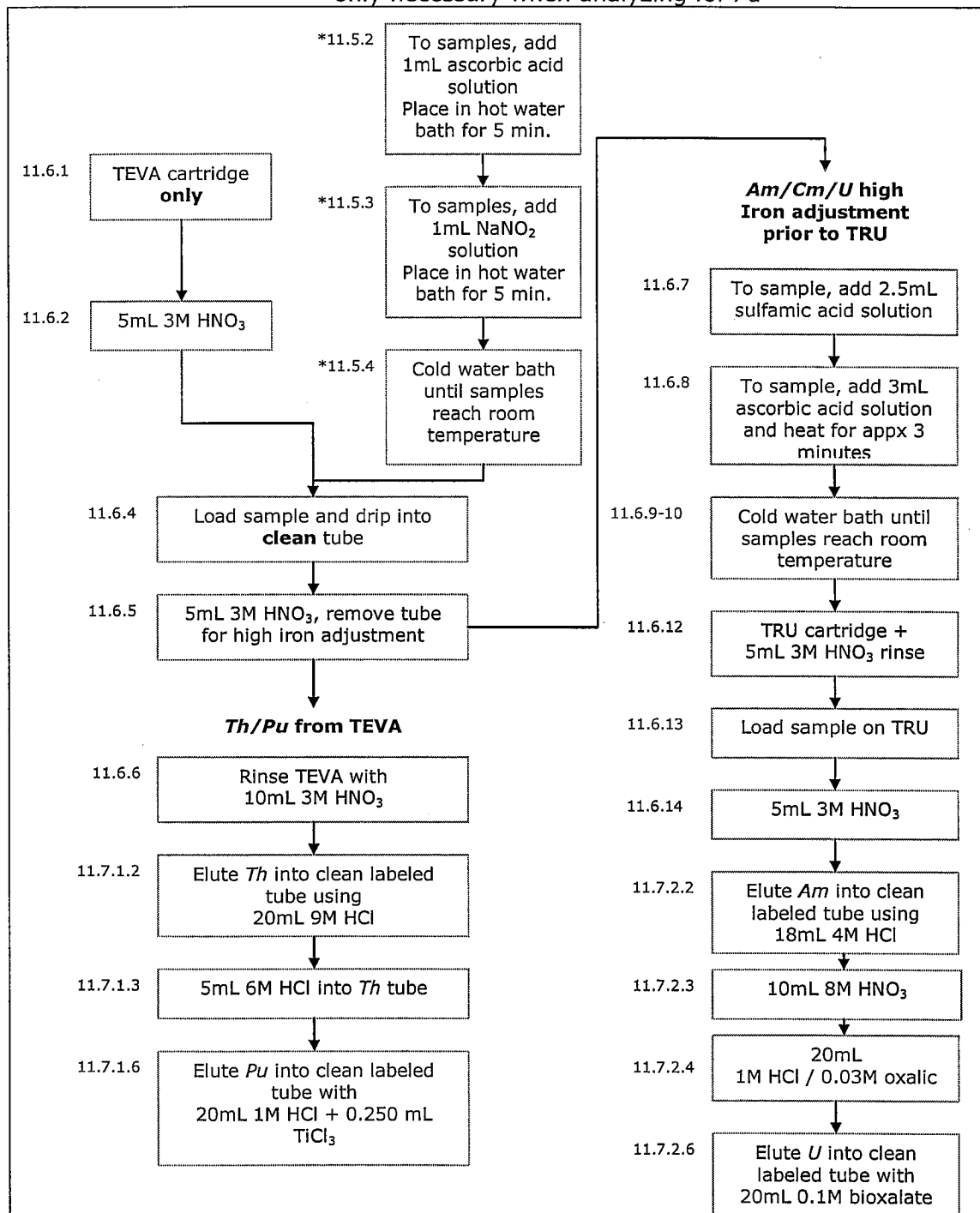
All rinses should flow at 1mL/minute (only 3M HNO₃ may be done at 3mL/minute)
*only necessary when analyzing for *Pu*



Sequential Thorium, Plutonium/Americium, Uranium via TEVA/TRU with high Iron adjustment

All rinses should flow at 1mL/minute (only 3M HNO₃ may be done at 3mL/minute)

*only necessary when analyzing for Pu



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TRENT**

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

**TITLE: DAILY OPERATION, CALIBRATION AND MAINTENANCE OF A
GERMANIUM SPECTROSCOPY SYSTEM**

(Supersedes STL-RD-0101 Rev. 5)

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1 SCOPE AND APPLICATION

- 1.1 This procedure applies to all germanium detectors and the computer assisted germanium spectroscopy analysis system.
- 1.2 Due to the nature of gamma spectroscopy, once the system is calibrated to a particular geometry any matrix can be run as long as it is prepared to match a calibrated geometry.
- 1.3 This SOP is based on EPA Method 901.1 and DOE EML HASL 300 Method GA-01-R.
- 1.4 The reporting limits, method detectable activities and QC limits are maintained in the Information Management System (QuantIMS). Because of their dynamic nature, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools. A copy of the SACs are included in this SOP to demonstrate this information.

2 SUMMARY OF METHOD

- 2.1 This procedure provides detailed instructions for energy calibration, efficiency determination, quality control checks, background and sample counting of the germanium spectroscopy system.

3 DEFINITIONS

- 3.1 See the STL Quality Management Plan (QMP) and STL St. Louis Laboratory Quality Manual (LQM) for a glossary of common laboratory terms and data reporting qualifiers.

4 INTERFERENCES

- 4.1 Germanium spectrometry has much potential interference. Interferences are usually in the form of radionuclides with unresolved photon emissions. These interferences are limited by the careful design/construction of the gamma spectral identification and interference libraries.

5 SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual, and the Facility Addendum to the Corporate Safety Manual.

6 EQUIPMENT AND SUPPLIES

- 6.1 Germanium spectroscopy system utilizing a computer based data acquisition system.

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7 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002, current revision.
- 7.2 Commercially prepared mixed gamma standards in reproducible geometries, with all appropriate NIST Source Certificate information.

8 SAMPLE COLLECTION, PRESERVATION AND STORAGE

- 8.1 STL St. Louis supplies sample containers and chemical preservatives in accordance with the method. STL St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in STL-QA-0006.
- 8.2 See radiochemistry sample preparation SOPs: STL-RC-0004 and STL-RC-0025.

9 QUALITY CONTROL

9.1 Batch

- 9.1.1 Definition: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists (example, pH) the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples.
- 9.1.2 Each analytical batch may contain up to 20 environmental samples, LCS and a Sample Duplicate.

9.2 Laboratory Control Sample

- 9.2.1 Definition: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3 Sample Duplicates

- 9.3.1 Definition: Sample Duplicate – a separate aliquot of a field sample taken through the entire analytical process.
- 9.3.2 Additional Sample Duplicates do not count towards the 20 samples in an analytical batch.

9.4 Procedural Variations

- 9.4.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance

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Memo shall be filed in the project file and incorporated into the report narrative.

9.5 Nonconformance and Corrective Action

- 9.5.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager. See SOP STL-QA-0036 for details regarding the NCM process.

10 CALIBRATION AND STANDARDIZATION

10.1 Calibration and QC Counting Schedule

- 10.1.1 Energy calibrations shall be established for the germanium spectroscopy systems **annually**, or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters.
- 10.1.2 FWHM calibrations shall be established for the germanium spectroscopy systems **annually**, or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters.
- 10.1.3 Efficiency Calibration Criteria
- 10.1.3.1 The curve should have eight calibration points used to determine the energy relationship of the calibration.
- 10.1.3.2 The calibration source must have radionuclides that "blanket" the intended range of calibration.
- 10.1.3.3 The energy difference should be less than 0.1 for all points or within 0.0005 of the energy keV for at least 10 points.
- 10.1.3.4 Computed efficiency test for all points should have a percent difference less than 5%.
- 10.1.3.5 The efficiency must be compared to the previous efficiency for the same calibration comparison performed. All values should have a percent difference that is less than 10%.
- 10.1.3.6 The FWHM must be less than 3.0 keV at 1332 keV.
- 10.1.3.7 FWHM difference should be less than 0.500 for all points.
- 10.1.3.8 Where applicable, the efficiency curve should be bounded by at least 8 data points (includes uncertainty).
- 10.1.3.9 The average deviation of efficiency should be less than 5% and the reduced Chi Square should be less than 5%.

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10.1.3.10 At least one point should be hand calculated to verify the calibration.

10.1.4 Background subtraction spectrum shall be established for the germanium spectroscopy systems **monthly**, or when the background quality control check indicates an unacceptable change in the daily background parameters, or as needed per client requirements.

10.1.4.1 Background count time is 12 hours.

10.1.4.1.1 If a client project requires a longer count time, then the background must be performed at the longer time before initiating analysis.

10.2 Daily Checks

10.2.1 The energy, resolution and efficiency calibrations for a detector shall be checked with its respective source each day that the germanium spectroscopy system is used.

10.2.2 The detector background shall be checked each day that the germanium spectroscopy system is used.

10.2.3 Calibration and background checks are acceptable if the value is less than the action (3σ) limit. The routine calibration and background quality control parameters that will be monitored are:

- Peak Energy for energy alignment (low-, mid-, and high-energy),
- Activity for efficiency check (low-, mid-, and high-energy),
- Full-Width at the Half Maximum (FWHM) for peak shape monitoring (low-, mid-, and high-energy),
- Background Count Rate

10.2.3.1 Calibration (efficiency, resolution, energy alignment, and background) quality control parameters will be found **acceptable** if the result is within the established investigate limits (2σ to 3σ range).

10.2.3.2 Calibration (efficiency, resolution, energy alignment, and background) quality control parameters will be found **not acceptable** if the result is outside the established limits (2σ to 3σ range) and marked as "action". In the case of an action, the daily QC check may be counted again or tagged out. The Daily QC check may only be recounted once without corrective action.

10.2.3.2.1 If the errant parameter is found acceptable for the rerun, the instrument can be used for the analysis of samples. No corrective action is necessary for this situation since the uncertainty can be attributed to the stochastic uncertainty of decay process (statistics), uncertainty of the sources, or a known uncorrected trend.

10.2.3.2.2 Entry is necessary in the instrument calibration and maintenance log of this situation.

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10.2.3.2.3 If the instrument fails to meet the acceptance criteria for the rerun for peak centroid and activity, the instrument must be declared "Out of Service". The detector/instrument must be "tagged". (See STL-QA-0036 for NCM details regarding tagging out of service). Note this action in the instrument calibration and maintenance log.

10.2.3.2.4 If the instrument fails to meet the acceptance criteria for FWHM, but the activity and centroid for that nuclide are within limits, then that detector may be used. However, if FWHM exceeds 1.45 for Am-241(low), 2.4 for Cs-137(med) or 3.25 for Co-60(high) regardless of activity, the instrument must be declared "Out of Service". The detector/instrument must be "tagged". (See STL-QA-0036 for NCM details regarding tagging out of service). Note this action in the instrument calibration and maintenance log and notify the Rad supervisor.

10.2.3.2.5 If the QC check fails for a second time, the analyst may want to:

10.2.3.2.5.1 Check the expiration date of the radioactive standard to confirm the material is current, for the isotopes being utilized.

10.2.3.2.5.2 Check source positioning and all instrument settings.

10.2.3.2.5.3 Check all cables for any apparent damage and to confirm that all cables are routed to proper connectors and are in good working order.

10.2.3.2.5.4 Check the seating of the Nuclear Instrumentation Module (NIM) electronics..

10.2.3.3 The instrument may be returned to service once the malfunction has been corrected and the above acceptance criteria have been met. This situation could be corrected by modifying the control chart to reflect the correct and current limits. Corrective actions must be noted in the instrument calibration and maintenance log.

10.2.3.4 If a parameter has two successive values in the warning/investigate limits, the system will be examined for a trend and noted in the maintenance log. Decisions will be based upon the Data Quality Objectives (DQO) and the degree of the bias in relation to the parameter.

10.2.3.4.1

10.3 Calibration Software Handling

10.3.1 Gamma Detector System Energy and Shape Calibration

10.3.1.1 Select "Calibrate" from the main menu.

10.3.1.2 Select "Count Calibration Spectrum".

10.3.1.3 Select the detector to be calibrated.

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- 10.3.1.4. Place the calibration standard on the selected gamma detector, and start acquisition by selecting "OK".
- 10.3.1.5. When the calibration standard has finished counting, select "Calibrate" from the menu.
- 10.3.1.6. Select "Recalibrate".
- 10.3.1.7. Select "Energy and Shape Calibration".
- 10.3.1.8. Select the detector and then the certificate that corresponds to the calibration standard.
- 10.3.1.9. The energy calibration curve is displayed. Select "OK" and then "Print" the calibration report.
- 10.3.2 Gamma Detector System Efficiency Calibration
 - 10.3.2.1. Select "Calibrate" from the main menu.
 - 10.3.2.2. Select "Count Calibration Spectrum".
 - 10.3.2.3. Select the detector to be calibrated.
 - 10.3.2.4. If the calibration standard has been counted already, proceed to 10.2.5. If not, enter the duration time for the acquisition. The count time should be long enough to accumulate approximately 10,000 counts for the major peaks found in the calibration standard. Place the calibration standard on the selected gamma detector, and start acquisition by selecting "OK".
 - 10.3.2.5. Select the geometry being calibrated or "Create a New Geometry".
 - 10.3.2.6. Select the certificate file that corresponds to the calibration standard.
 - 10.3.2.7. The efficiency curve is displayed. Select "OK" and print the efficiency calibration report.
 - 10.3.2.8. The user is asked to save the efficiency file. Select "OK".
- 10.3.3 Detector Background Counting
 - 10.3.4. Select "Count".
 - 10.3.5. Select "Start a Background Count".
 - 10.3.6. Select the gamma detector to be used for the background acquisition.
 - 10.3.7. Select the analysis sequence file for long background counts.
 - 10.3.8. Select the "No Specific Geometry" for the background count geometry.
 - 10.3.9. Remove any samples from the detector and close the shield, and start acquisition.
 - 10.3.10. After the acquisition has been started, the program prompts the user to enter the sample description parameters.
 - 10.3.10.1. Enter the sample information in the following format:
 - 10.3.10.2. Batch Number: "Long"
 - 10.3.10.3. Project Number: "Background"
 - 10.3.10.4. Sample ID: "mmdd_year"
 - 10.3.10.5. After entering the sample parameters, select "OK".
 - 10.3.11. Note this action in the runlog.

11 PROCEDURE

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11.1 Calibration Quality Control

- 11.1.1 Select "QC" from the main menu.
- 11.1.2 Select "Calibration Check" from the quality control menu.
- 11.1.3 Select the gamma detector to be used for the calibration quality control acquisition.
- 11.1.4 Select the specific geometry for the calibration quality control sample.
- 11.1.5 Select the certificate that describes the standard source used in the calibration check.
- 11.1.6 Place the calibration quality control sample on the detector, and start acquisition.
- 11.1.7 Note this action in the runlog.

11.2 Background Quality Control

- 11.2.1 Select "QC" from the main menu.
- 11.2.2 Select "Background Check" from the quality control menu.
- 11.2.3 Select the gamma detector to be used for the background quality control acquisition.
- 11.2.4 Remove any samples from the detector, and start acquisition.
- 11.2.5 Note this action in the instrument calibration and maintenance log.

11.3 Sample Counting

- 11.3.1 Select "Count" from the main menu.
- 11.3.2 Select "Start a Count".
- 11.3.3 Select the gamma detector to be used for the sample acquisition.
- 11.3.4 Select the analysis sequence file to use for the specific analysis of the unknown sample.
- 11.3.5 Select the specific geometry for the unknown sample.
- 11.3.6 Place the sample on the detector and select "OK".
- 11.3.7 After the acquisition has been started, the program prompts the user to enter the sample description parameters.
- 11.3.8 After entering the sample parameters, select "OK".
- 11.3.9 Note this action in the instrument run log.

12 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the STL St. Louis LQM.
- 12.2 All calculations are performed automatically by the Canberra Spectroscopy software routines.
- 12.3 Gamma Activity Concentration

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The activity concentration of a sample will be calculated using the following equation.

$$ACT_s = \frac{Net_Counts}{2.22 * E * t_s * Ab * V_A * D_c * D_s}$$

where:

ACT_s = the activity in pCi/(units of the volume)
Net_Counts = the net area of a peak
2.22 = the correction factor to pCi
E = the efficiency – corrected for transmission
t_s = the count time in minutes
Ab = the gamma abundance factor
V_A = the sample aliquot volume
D_c = the decay correction during the analysis
D_s = the decay correction from collection date to start of analysis

13 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

13.1 The data assessment and corrective action process is detailed through the Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: STL-QA-0036. A hardcopy of all the data assessment types and descriptions along with their associated corrective actions is included in the SOP. Below is a subset of the data assessment and QC excursion types within Clouseau; the text in underline is the exact "type" line in Clouseau. For a complete and current listing, please access the software program.

13.2 Method Blank

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit..

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – See Clouseau NCM for corrective action. Note certain analytes are common laboratory contaminants which require special narrative comment. These compounds are so designated in Clouseau.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD).

13.3.2 Corrective Action for LCS not meeting acceptance criteria:

13.3.2.1 LCS Spike Recovery excursion (high) – See Clouseau NCM for corrective action.

13.3.2.2 LCS Spike Recovery excursion (low) – See Clouseau NCM for corrective action.

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13.4 Duplicate

13.4.1 Acceptance Criteria:

13.4.1.1 RPD less than 40%. If RPD greater than 40%, RER must be less than 1.

13.4.1.2 If one or both samples below MDA, evaluate RER.

13.4.2 Corrective Action for RPD/RER not meeting acceptance criteria:

13.4.2.1 RPD/RER excursion for LCS/LCSD or Sample/Sample Duplicate – See Clouseau NCM for corrective action.

14 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits and QC acceptance limits, are given in the appendix to this SOP.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in STL St. Louis' LQM section 5.1.2.

14.3 Training Qualification

14.3.1 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.3.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in STL St. Louis' LQM section 5.1.2.

14.3.3 Annually the analyst must successfully demonstrate proficiency to continuing to perform this analysis. See requirements in STL St. Louis' LQM section 5.1.2.

15 VALIDATION DATA

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. STL St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

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17 REFERENCES

- 17.1 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, method GA-01-R
- 17.2 EPA Prescribed Procedures for Measurement of Radioactivity in Drinking Water Method 901.1
- 17.3 PROcount Menu Software Package User's Manual, 07-0513, Canberra Industries, Inc. (latest version)
- 17.4 VMS Spectroscopy Applications Package User's Manual, 07-0196, Canberra Industries, Inc. (latest version)
- 17.5 Canberra Industries, "Nuclide Identification Algorithms and Software Verification and Validation Manual 07-0464".
- 17.6 Canberra Industries, "Spectroscopy Applications Algorithms and Software Verification and Validation Manual 07-0368".
- 17.7 STL Quality Management Plan (QMP), current revision.
- 17.8 STL St. Louis Laboratory Quality Manual (LQM), current revision.
- 17.9 STL Corporate Safety Manual and St. Louis Facility Addendum (SOP STL-HS-0002), current revisions
- 17.10 Associated SOPs:
 - 17.10.1 STL-RC-0004, current revision, Preparation of Soil Samples for Radiochemical Analysis
 - 17.10.2 STL-RC-0025, current revision, Preparation of Samples for Gamma Spectroscopy
 - 17.10.3 STL-QA-0002, current revision, Standards and Reagent Preparation
 - 17.10.4 STL-QA-0014, current revision, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.10.5 STL-QA-0036, current revision, Non-Conformance Memorandum (NCM) Process

18 CHANGES FROM PREVIOUS REVISION

- 18.1 Updated method and SOP references

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18.2 Clarified calibration terminology

18.3 Updated NELAC format

STL Reference Data Summary

Structured Analysis Code: I-G7-Z7-01-06

Target Analyte List: All Analytes

Matrix: WATER
 Extraction: Direct Addition of Sample to Geometry
 Method: Gamma Cs-137 & Hlls by EPA 901.1-MOD
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Syn	Compound	Analyte List	RL	Detection Limits			Run Date	Check List 6506			Spike List 6506		
				Units	MDL	Units		T	A	Amt	Units	LCL	UCL
3997	Actinium 228			pCi/L			0						
3984	Americium 241			pCi/L			0	C	Y			90	110
4280	Antimony 124			pCi/L			0					90	110
4103	Antimony 125			pCi/L			0						
5556	Barium-137			pCi/L			0						
4211	Barium/Lanthanum-140			pCi/L			0						
4168	Barium 133			pCi/L			0						
3999	Barium 140			pCi/L			0						
4001	Beryllium 7			pCi/L			0						
4798	Bismuth 211 (assumes equilibrium w/			pCi/L			0						
4800	Bismuth 212			pCi/L			0						
4005	Bismuth 214			pCi/L			0						
5557	Calcium-45			pCi/L			0						
4009	Cerium 141			pCi/L			0						
4804	Cerium 139			pCi/L			0						
4011	Cerium 144			pCi/L			0						
4031	Cesium 134			pCi/L			0						
4033	Cesium 137		20	pCi/L			0	C	Y			90	110
4023	Cobalt 57			pCi/L			0						
4025	Cobalt 58			pCi/L			0						
4027	Cobalt 60			pCi/L			0	C	Y			90	110
4035	Europium 152			pCi/L			0						
4037	Europium 154			pCi/L			0						
4039	Europium 155			pCi/L			0						
4213	Hafnium 181			pCi/L			0						
4049	Iodine 131			pCi/L			0						
4043	Iron 59			pCi/L			0						
4156	Lead 210			pCi/L			0						
4077	Lead 212			pCi/L			0						
4079	Lead 214			pCi/L			0						
5558	Manganese-56			pCi/L			0						
4055	Manganese 54			pCi/L			0						
4806	Mercury 203			pCi/L			0						
4069	Neptunium 237			pCi/L			0						
4172	Neptunium 239			pCi/L			0						
5877	Niobium 83			pCi/L			0						
4051	Potassium 40			pCi/L			0						

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Structured Analysis Code: I-G7-Z7-01-06

Target Analyte List: All Analytes

Matrix: WATER

Extraction: Direct Addition of Sample to Geometry
 Method: Gamma Cs-137 & Hlts by EPA 901.1 MOD
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List		Detection Limits			Check List 6506				Spike List 6506							
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	T	A	Amt	Units	LCL	UCL	RPD
4081	Promethium 144		pCi/L			0										
4083	Promethium 146		pCi/L			0										
4085	Promethium 147		pCi/L			0										
5225	Protactinium 234M		pCi/L			0										
4073	Protactinium 234		pCi/L			0										
2257	Radium (226)		pCi/L			0										
2259	Radium 228		pCi/L			0										
4810	Radium 223 (assumes equilibrium w/		pCi/L			0										
4095	Radium 224		pCi/L			0										
4101	Ruthenium 106		pCi/L			0										
5044	Scandium 46		pCi/L			0										
4057	Sodium 22		pCi/L			0										
4059	Sodium 24		pCi/L			0										
4107	Strontium 85		pCi/L			0										
4125	Thallium 208		pCi/L			0										
4816	Thorium 227		pCi/L			0										
4115	Thorium 228		pCi/L			0										
4117	Thorium 230		pCi/L			0										
4119	Thorium 231		pCi/L			0										
4121	Thorium 232		pCi/L			0										
4123	Thorium 234		pCi/L			0										
4278	Tin 113		pCi/L			0										
4129	Uranium 234		pCi/L			0										
4131	Uranium 235		pCi/L			0										
4133	Uranium 238		pCi/L			0										
5559	Vanadium-48		pCi/L			0										
4137	Yttrium 88		pCi/L			0										
4141	Zinc 65		pCi/L			0										
4143	Zirconium 95		pCi/L			0										

STL Reference Data Summary

Structured Analysis Code: A-G6-0A-01-06

Target Analyte List: All Analytes

Matrix: SOLID
 Extraction: Dry, Grind, and Fill Geometry
 Method: Gamma Cs-137 & Hits by DOE GA-01-R MOD.
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List			Detection Limits		Check List 6506				Spike List 6506											
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3995	Actinium 227		pCi/g			0														
3997	Actinium 228		pCi/g			0														
3984	Americium 241		pCi/g			0	C	Y			90	115	40	C	Y			90	115	40
4280	Antimony 124		pCi/g			0														
4103	Antimony 125		pCi/g			0														
5556	Barium-137		pCi/g			0														
4211	Barium/Lanthanum-140		pCi/g			0														
4168	Barium 133		pCi/g			0														
3999	Barium 140		pCi/g			0														
4001	Beryllium 7		pCi/g			0														
5067	Bismuth-207		pCi/g			0														
5068	Bismuth-210M		pCi/g			0														
4798	Bismuth 211 (assumes equilibrium w/		pCi/g			0														
4800	Bismuth 212		pCi/g			0														
4005	Bismuth 214		pCi/g			0														
4802	Cadmium 109		pCi/g			0														
5557	Calcium-45		pCi/g			0														
4009	Cerium 141		pCi/g			0														
4804	Cerium 139		pCi/g			0														
4011	Cerium 144		pCi/g			0														
4031	Cesium 134		pCi/g			0														
4033	Cesium 137	0.2	pCi/g			0	C	Y			90	115	40	C	Y			90	115	40
5399	Cobalt 56		pCi/g			0														
4023	Cobalt 57		pCi/g			0														
4025	Cobalt 58		pCi/g			0														
4027	Cobalt 60		pCi/g			0	C	Y			90	111	40	C	Y			90	111	40
4035	Europium 152		pCi/g			0														
4037	Europium 154		pCi/g			0														
4039	Europium 155		pCi/g			0														
5415	Gadolinium 153		pCi/g			0														
4213	Hafnium 181		pCi/g			0														
4049	Iodine 131		pCi/g			0														
5416	Iridium 192		pCi/g			0														
4043	Iron 59		pCi/g			0														
5438	Kr-85		pCi/g			0														
4156	Lead 210		pCi/g			0														
4077	Lead 212		pCi/g			0														

number 1

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Structured Analysis Code: A-G6-0A-01-06

Target Analyte List: All Analytes

Matrix: SOLID
 Extraction: Dry, Grind, and Fill Geometry
 Method: Gamma Cs-137 & Hits by DOE GA-01-R MOD.
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List		Detection Limits		Check List 6506				Spike List 6506												
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4079	Lead 214		pCi/g			0														
5558	Manganese-56		pCi/g			0														
4055	Manganese 54		pCi/g			0														
4806	Mercury 203		pCi/g			0														
4089	Neptunium 237		pCi/g			0														
4172	Neptunium 239		pCi/g			0														
5877	Niobium 83		pCi/g			0														
4061	Niobium 94		pCi/g			0														
4063	Niobium 95		pCi/g			0														
4051	Potassium 40		pCi/g			0														
4081	Promethium 144		pCi/g			0														
4083	Promethium 146		pCi/g			0														
4085	Promethium 147		pCi/g			0														
5225	Protactinium 234M		pCi/g			0														
4071	Protactinium 231		pCi/g			0														
4073	Protactinium 234		pCi/g			0														
2257	Radium (226)		pCi/g			0														
2259	Radium 228		pCi/g			0														
5094	Radium-225		pCi/g			0														
4810	Radium 223 (assumes equilibrium w/		pCi/g			0														
4095	Radium 224		pCi/g			0														
5220	Rhodium 106		pCi/g			0														
5846	Rh102m		pCi/g			0														
4099	Ruthenium 103		pCi/g			0														
4101	Ruthenium 106		pCi/g			0														
5044	Scandium 46		pCi/g			0														
5404	Silver 108m		pCi/g			0														
4779	Silver 110m		pCi/g			0														
4057	Sodium 22		pCi/g			0														
4059	Sodium 24		pCi/g			0														
4107	Strontium 85		pCi/g			0														
5553	Tantalum 182		pCi/g			0														
5554	Terbium 160		pCi/g			0														
4125	Thallium 208		pCi/g			0														
4816	Thorium 227		pCi/g			0														
4115	Thorium 228		pCi/g			0														
4117	Thorium 230		pCi/g			0														
4119	Thorium 231		pCi/g			0														
4121	Thorium 232		pCi/g			0														

Structured Analysis Code: A-G6-0A-01-06

Target Analyte List: All Analytes

Matrix: SOLID
 Extraction: Dry, Grind, and Fill Geometry
 Method: Gamma Cs-137 & Hits by DOE GA-01-R MOD.
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List		Detection Limits		Check List 6506				Spike List 6506					
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD
4123	Thorium 234		pCi/g			0							
4278	Tin 113		pCi/g			0							
4129	Uranium 234		pCi/g			0							
4131	Uranium 235		pCi/g			0							
4133	Uranium 238		pCi/g			0							
5559	Vanadium-48		pCi/g			0							
4137	Yttrium 88		pCi/g			0							
4141	Zinc 65		pCi/g			0							
4143	Zirconium 95		pCi/g			0							

STL Reference Data Summary

Structured Analysis Code: A-K1-0A-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: As Received, Fill Geometry

Method: Gamma Cs-137 & HIs by DOE GA-01-R MOD.

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		RL	Detection Limits		Units	Run Date	Check List 6506				Spike List 6506			
Syn	Compound		Units	MDL			T	A	Amt	Units	LCL	UCL	RPD	Units
3995	Actinium 227		pCi/g			0								
3997	Actinium 228		pCi/g			0								
3984	Americium 241		pCi/g			0	C	Y			90	115	40	90
4280	Antimony 124		pCi/g			0								115 40
4103	Antimony 125		pCi/g			0								
5556	Barium-137		pCi/g			0								
4211	Barium/Lanthanum-140		pCi/g			0								
4168	Barium 133		pCi/g			0								
3999	Barium 140		pCi/g			0								
4001	Beryllium 7		pCi/g			0								
5068	Bismuth-210M		pCi/g			0								
4798	Bismuth 211 (assumes equilibrium w/		pCi/g			0								
4800	Bismuth 212		pCi/g			0								
4005	Bismuth 214		pCi/g			0								
4802	Cadmium 109		pCi/g			0								
5557	Calcium-45		pCi/g			0								
4009	Cerium 141		pCi/g			0								
4804	Cerium 139		pCi/g			0								
4011	Cerium 144		pCi/g			0								
4031	Cesium 134		pCi/g			0								
4033	Cesium 137	0.2	pCi/g			0	C	Y			90	115	40	90
5399	Cobalt 56		pCi/g			0								
4023	Cobalt 57		pCi/g			0								
4025	Cobalt 58		pCi/g			0								
4027	Cobalt 60		pCi/g			0	C	Y			90	111	40	90
4035	Europium 152		pCi/g			0								
4037	Europium 154		pCi/g			0								
4039	Europium 155		pCi/g			0								
5415	Gadolinium 153		pCi/g			0								
4213	Hafnium 181		pCi/g			0								
4049	Iodine 131		pCi/g			0								
5416	Iridium 192		pCi/g			0								
4043	Iron 59		pCi/g			0								
5438	Kr-85		pCi/g			0								
4156	Lead 210		pCi/g			0								
4077	Lead 212		pCi/g			0								
4079	Lead 214		pCi/g			0								

a number 1

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Structured Analysis Code: A-K1-0A-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Fill Geometry
Method: Gamma Cs-137 & Hils by DOE GA-01-R MOD.
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List			Detection Limits			Check List 6506			Spike List 6506											
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5558	Manganese-56		pCi/g			0														
4055	Manganese 54		pCi/g			0														
4806	Mercury 203		pCi/g			0														
4069	Neptunium 237		pCi/g			0														
4172	Neptunium 239		pCi/g			0														
5877	Niobium 83		pCi/g			0														
4061	Niobium 94		pCi/g			0														
4063	Niobium 95		pCi/g			0														
4051	Potassium 40		pCi/g			0														
4081	Promethium 144		pCi/g			0														
4083	Promethium 146		pCi/g			0														
4085	Promethium 147		pCi/g			0														
5225	Protactinium 234M		pCi/g			0														
4071	Protactinium 231		pCi/g			0														
4073	Protactinium 234		pCi/g			0														
2257	Radium (226)		pCi/g			0														
2259	Radium 228		pCi/g			0														
5094	Radium-225		pCi/g			0														
4810	Radium 223 (assumes equilibrium w/		pCi/g			0														
4095	Radium 224		pCi/g			0														
5220	Rhodium 106		pCi/g			0														
4099	Ruthenium 103		pCi/g			0														
4101	Ruthenium 106		pCi/g			0														
5044	Scandium 46		pCi/g			0														
5404	Silver 108m		pCi/g			0														
4779	Silver 110m		pCi/g			0														
4057	Sodium 22		pCi/g			0														
4059	Sodium 24		pCi/g			0														
4107	Strontium 85		pCi/g			0														
5553	Tantalum 182		pCi/g			0														
5554	Terbium 160		pCi/g			0														
4125	Thallium 208		pCi/g			0														
4816	Thorium 227		pCi/g			0														
4115	Thorium 228		pCi/g			0														
4117	Thorium 230		pCi/g			0														
4119	Thorium 231		pCi/g			0														
4121	Thorium 232		pCi/g			0														
4123	Thorium 234		pCi/g			0														
4278	Tin 113		pCi/g			0														

Structured Analysis Code: A-K1-0A-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: As Received, Fill Geometry
Method: Gamma Cs-137 & Hils by DOE GA-01-R MOD.
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits		Run Date		Check List 6506		Spike List 6506	
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units
4129	Uranium 234		pCi/g		0				
4131	Uranium 235		pCi/g		0				
4133	Uranium 238		pCi/g		0				
5559	Vanadium-48		pCi/g		0				
4137	Yttrium 88		pCi/g		0				
4141	Zinc 65		pCi/g		0				
4143	Zirconium 95		pCi/g		0				

STL Reference Data Summary

Structured Analysis Code: A-1C-0A-01-06

Target Analyte List: All Analytes

Matrix: SOLID
 Extraction: LEACHATE, DI (ASTM D3987-85) - 18 hour
 Method: Gamma Cs-137 & Hils by DOE GA-01-R MOD.
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List		RL	Detection Limits		Units	Run Date	Check List 6341				Spike List 6342								
Syn	Compound		Units	MDL			T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL
3995	Actinium 227		pCi/L			0													
3997	Actinium 228		pCi/L			0													
3984	Americium 241		pCi/L			0													
4280	Antimony 124		pCi/L			0	C	Y		90	110	40		C	Y		90	110	40
4103	Antimony 125		pCi/L			0													
5556	Barium-137		pCi/L			0													
4211	Barium/Lanthanum-140		pCi/L			0													
4168	Barium 133		pCi/L			0													
3999	Barium 140		pCi/L			0													
4001	Beryllium 7		pCi/L			0													
4798	Bismuth 211 (assumes equilibrium w/		pCi/L			0													
4800	Bismuth 212		pCi/L			0													
4005	Bismuth 214		pCi/L			0													
4802	Cadmium 109		pCi/L			0													
5557	Calcium-45		pCi/L			0													
4009	Cerium 141		pCi/L			0													
4804	Cerium 139		pCi/L			0													
4011	Cerium 144		pCi/L			0													
4031	Cesium 134		pCi/L			0													
4033	Cesium 137	20	pCi/L			0													
5399	Cobalt 56		pCi/L			0	C	Y		90	110	40		C	Y		90	110	40
4023	Cobalt 57		pCi/L			0													
4025	Cobalt 58		pCi/L			0													
4027	Cobalt 60		pCi/L			0													
4035	Europium 152		pCi/L			0	C	Y		90	110	40		C	Y		90	110	40
4037	Europium 154		pCi/L			0													
4039	Europium 155		pCi/L			0													
5415	Gadolinium 153		pCi/L			0													
4213	Hafnium 181		pCi/L			0													
4049	Iodine 131		pCi/L			0													
5416	Iridium 192		pCi/L			0													
4043	Iron 59		pCi/L			0													
5438	Kr-85		pCi/L			0													
4156	Lead 210		pCi/L			0													
4077	Lead 212		pCi/L			0													
4079	Lead 214		pCi/L			0													
5558	Manganese-56		pCi/L			0													

a number 1

Structured Analysis Code: A-1C-0A-01-06

Target Analyte List: All Analytes

Matrix: SOLID
 Extraction: LEACHATE, DI (ASTM D3987-85) - 18 hour
 Method: Gamma Cs-137 & Hils by DOE GA-01-R MOD.
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List		Detection Limits			Check List 6341			Spike List 6342		
Syn	Compound	RL	Units	MDL	Units	T	A	Units	T	A
			pCi/L			Run Date	RPD		Run Date	RPD
4055	Manganese 54		pCi/L			0			0	
4806	Mercury 203		pCi/L			0			0	
4069	Neptunium 237		pCi/L			0			0	
4172	Neptunium 239		pCi/L			0			0	
4061	Niobium 94		pCi/L			0			0	
4063	Niobium 95		pCi/L			0			0	
4051	Potassium 40		pCi/L			0			0	
4081	Promethium 144		pCi/L			0			0	
4083	Promethium 146		pCi/L			0			0	
4085	Promethium 147		pCi/L			0			0	
5225	Protactinium 234M		pCi/L			0			0	
4071	Protactinium 231		pCi/L			0			0	
2257	Radium (226)		pCi/L			0			0	
2259	Radium 228		pCi/L			0			0	
5094	Radium-225		pCi/L			0			0	
4810	Radium 223 (assumes equilibrium w/		pCi/L			0			0	
4095	Radium 224		pCi/L			0			0	
5220	Rhodium 106		pCi/L			0			0	
4099	Ruthenium 103		pCi/L			0			0	
4101	Ruthenium 106		pCi/L			0			0	
5044	Scandium 46		pCi/L			0			0	
5404	Silver 108m		pCi/L			0			0	
4779	Silver 110m		pCi/L			0			0	
4057	Sodium 22		pCi/L			0			0	
4059	Sodium 24		pCi/L			0			0	
4107	Strontium 85		pCi/L			0			0	
5553	Tantalum 182		pCi/L			0			0	
5554	Terbium 160		pCi/L			0			0	
4125	Thallium 208		pCi/L			0			0	
4816	Thorium 227		pCi/L			0			0	
4115	Thorium 228		pCi/L			0			0	
4117	Thorium 230		pCi/L			0			0	
4119	Thorium 231		pCi/L			0			0	
4121	Thorium 232		pCi/L			0			0	
4123	Thorium 234		pCi/L			0			0	
4278	Tin 113		pCi/L			0			0	
4129	Uranium 234		pCi/L			0			0	
4131	Uranium 235		pCi/L			0			0	
4133	Uranium 238		pCi/L			0			0	

Structured Analysis Code: A-1C-0A-01-06

Target Analyte List: All Analytes

Matrix: SOLID
 Extraction: LEACHATE, DI (ASTM D3987-85) - 18 hour
 Method: Gamma Cs-137 & Hits by DOE GA-01-R MOD.
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List

Syn	Compound	RL	Detection Limits	Units	Run Date	T	A	Amt	Check List 6341	Units	LCL	UCL	RPD	T	A	Amt	Spike List 6342	Units	LCL	UCL	RPD	
5559	Vanadium-48		pCi/L		0																	
4137	Yttrium 88		pCi/L		0																	
4141	Zinc 65		pCi/L		0																	
4143	Zirconium 95		pCi/L		0																	

STL Reference Data Summary

Structured Analysis Code: A-GM-0B-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: Dry, Grind, Fill Geometry - 10-DAY INGROWTH

Method: Gamma Ra-226 & HIs By DOE GA-01-R Mod.

QIC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits		Run Date		Check List 6547		Spike List 6547	
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units
3995	Actinium 227		pCi/g		0				
3997	Actinium 228		pCi/g		0				
3984	Americium 241		pCi/g		0				
4280	Antimony 124		pCi/g		0				
4103	Antimony 125		pCi/g		0				
4211	Barium/Lanthanum-140		pCi/g		0				
4168	Barium 133		pCi/g		0				
3999	Barium 140		pCi/g		0				
4001	Beryllium 7		pCi/g		0				
5053	Bismuth 207		pCi/g		0				
5068	Bismuth-210M		pCi/g		0				
4800	Bismuth 212		pCi/g		0				
4005	Bismuth 214		pCi/g		0				
4009	Cerium 141		pCi/g		0				
4011	Cerium 144		pCi/g		0				
4031	Cesium 134		pCi/g		0				
4033	Cesium 137		pCi/g		0				
4023	Cobalt 57		pCi/g		0				
4025	Cobalt 58		pCi/g		0				
4027	Cobalt 60		pCi/g		0				
4035	Europium 152		pCi/g		0				
4037	Europium 154		pCi/g		0				
4039	Europium 155		pCi/g		0				
4213	Hafnium 181		pCi/g		0				
4049	Iodine 131		pCi/g		0				
4043	Iron 59		pCi/g		0				
4156	Lead 210		pCi/g		0				
4077	Lead 212		pCi/g		0				
4079	Lead 214		pCi/g		0				
4055	Manganese 54		pCi/g		0				
4069	Neptunium 237		pCi/g		0				
4172	Neptunium 239		pCi/g		0				
4051	Potassium 40		pCi/g		0				
4081	Promethium 144		pCi/g		0				
4083	Promethium 146		pCi/g		0				
4085	Promethium 147		pCi/g		0				
4071	Protactinium 231		pCi/g		0				

C N 9.3 pCi/g 75 135 40 C N 9.3 pCi/g 75 135 40

STL Reference Data Summary

Structured Analysis Code: A-J9-0B-01-06
Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, and Fill Geometry -> 21 day In-growth
Method: Gamma Ra-226 & HIs By DOE GA-01-R Mod.
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits		Check List 6547		Spike List 6547														
Syn	Compound	RL	Units	MDL	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD	
3995	Actinium 227		pCi/g		0															
3997	Actinium 228		pCi/g		0															
3984	Americium 241		pCi/g		0															
4280	Antimony 124		pCi/g		0															
4103	Antimony 125		pCi/g		0															
4211	Barium/Lanthanum-140		pCi/g		0															
4168	Barium 133		pCi/g		0															
3999	Barium 140		pCi/g		0															
4001	Beryllium 7		pCi/g		0															
5053	Bismuth 207		pCi/g		0															
5068	Bismuth-210M		pCi/g		0															
4800	Bismuth 212		pCi/g		0															
4005	Bismuth 214		pCi/g		0															
4009	Cerium 141		pCi/g		0															
4011	Cerium 144		pCi/g		0															
4031	Cesium 134		pCi/g		0															
4033	Cesium 137		pCi/g		0															
4023	Cobalt 57		pCi/g		0															
4025	Cobalt 58		pCi/g		0															
4027	Cobalt 60		pCi/g		0															
4035	Europium 152		pCi/g		0															
4037	Europium 154		pCi/g		0															
4039	Europium 155		pCi/g		0															
4213	Hafnium 181		pCi/g		0															
4049	Iodine 131		pCi/g		0															
4043	Iron 59		pCi/g		0															
4156	Lead 210		pCi/g		0															
4077	Lead 212		pCi/g		0															
4079	Lead 214		pCi/g		0															
4055	Manganese 54		pCi/g		0															
4069	Neptunium 237		pCi/g		0															
4172	Neptunium 239		pCi/g		0															
4051	Potassium 40		pCi/g		0															
4081	Promethium 144		pCi/g		0															
4083	Promethium 146		pCi/g		0															
4085	Promethium 147		pCi/g		0															
5225	Protactinium 234M		pCi/g		0															
											C	N	9.3	C	N	9.3	pCi/g	75	135	40

Printed at 11/3/20

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Structured Analysis Code: A-J9-0B-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: Dry, Grind, and Fill Geometry -> 21 day In-growth
Method: Gamma Ra-226 & Hills By DOE GA-01-R Mod.

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List

Analyte List		Detection Limits			Check List 6547				Spike List 6547											
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4071	Protactinium 231	1.0	pCi/g			0														
2257	Radium (226)		pCi/g			0	C	Y	12.2	pCi/g	75	135	40	C	Y	12.2	pCi/g	75	135	40
2259	Radium 228		pCi/g			0														
4095	Radium 224		pCi/g			0														
4101	Ruthenium 106		pCi/g			0														
4057	Sodium 22		pCi/g			0														
4059	Sodium 24		pCi/g			0														
4125	Thallium 208		pCi/g			0														
4121	Thorium 232		pCi/g			0	C	Y	9.5	pCi/g	75	135	40	C	Y	9.5	pCi/g	75	135	40
4123	Thorium 234		pCi/g			0														
4278	Tin 113	pCi/g			0															
4131	Uranium 235	pCi/g			0															
4133	Uranium 238	pCi/g			0	C	N	11.9	pCi/g	75	135	40	C	N	11.9	pCi/g	75	135	40	
4137	Yttrium 88	pCi/g			0															
4141	Zinc 65	pCi/g			0															
4143	Zirconium 95	pCi/g			0															

STL Reference Data Summary

Structured Analysis Code: A-K5-4F-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: As Received, Direct Addition of Sample to Geometry

Method: Gamma Iodine by GA-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List

Syn	Compound	RL	Detection Limits Units	MDL	Units	Run Date	T	A	Amt	Check List 6581 Units	LCL	UCL	RPD	T	A	Amt	Spike List 6581 Units	LCL	UCL	RPD
5409	Iodine 125	30	pCi/g			0					90	110	40	C	Y			90	110	40
4047	Iodine 129	30	pCi/g			0	C	Y												
4049	Iodine 131	30	pCi/g			0														

STL Reference Data Summary

Structured Analysis Code: A-G6-4F-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: Dry, Grind, and Fill Geometry

Method: Gamma Iodine by GA-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List

Syn	Compound	RL	Detection Limits	Units	Run Date	T	A	Amt	Check List 6581	Units	LCL	UCL	RPD	T	A	Amt	Spike List 6581	Units	LCL	UCL	RPD
5409	Iodine 125	30	pCi/g		0																
4047	Iodine 129	30	pCi/g		0	C	Y		90	110	40			C	Y				90	110	40
4049	Iodine 131	30	pCi/g		0																

STL Reference Data Summary

Structured Analysis Code: I-G7-4F-01-06

Target Analyte List: All Analytes

Matrix: WATER

Extraction: Direct Addition of Sample to Geometry

Method: Gamma Iodine by GA-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St Louis

Analyte List

Syn	Compound	RL	Detection Limits	Units	MDL	Run Date	T	A	Amt	Check List 6581	LCL	UCL	RPD	T	A	Amt	Spike List 6581	LCL	UCL	RPD
5409	Iodine 125	10	pCi/L			0														
4047	Iodine 129	10	pCi/L			0	C	Y			90	110	40	C	Y			90	110	40
4049	Iodine 131	10	pCi/L			0														

SOP No.: STL-RD-0210
Revision No.: 1
Revision Date: 01/15/06
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Implementation Date: 01/19/06

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

**TITLE: Daily Operations of an Alpha Spectroscopy System (using
AlphaVision Software)**

(Supersedes STL-RD-0210 Rev. 0)

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1 SCOPE AND APPLICATION

- 1.1 This procedure applies to alpha spectroscopy detectors and the computer assisted alpha spectroscopy analysis systems, using AlphaVision software.
- 1.2 The reporting limits, method detectable activities and QC limits are maintained in the Information Management System (QuantIMS). Because of their dynamic nature, they are not specifically listed in this document, but can be retrieved at any time using TraQAr tools. A copy of the SACs are included in this SOP to demonstrate this information.

2 SUMMARY OF METHOD

- 2.1 This SOP provides detailed instructions for energy calibration, efficiency determination, quality control checks, background and sample counting of the alpha spectroscopy system.

3 DEFINITIONS

- 3.1 See STL Quality Management Plan (QMP) and STL St. Louis Laboratory Quality Manual (LQM) for glossary of common terms and data qualifiers.
- 3.2 Minimum Detectable Activity (MDA) - The smallest amount of activity that can be detected given the conditions of a specific sample. It is reported at the 95% confidence interval, meaning that there is a 5% chance that a false signal was reported as activity and a 5% chance that true activity went undetected.
- 3.3 Tracer - A known amount of ^{232}U , ^{242}Pu or ^{236}Pu , ^{243}Am , ^{209}Po , ^{237}Np or ^{229}Th (depending on analyte(s) required) added to each sample to determine chemical yield. The tracer serves as an internal standard, which is used to calculate the activity of the target isotopes.
- 3.4 Region of Interest (ROI): The KeV range through which the target isotope peak signal responds.
- 3.5 Tailing: Tailing is a delayed return of a peak to chromatographic baseline or continuation of response beyond its normal response window (RT window, ROI) due to high concentration of the analyte or a matrix interference.

4 INTERFERENCES

- 4.1 Alpha spectrometry has many potential interferences. These are usually in the form of radionuclides with unresolved alpha emissions. Poorly resolved alpha peaks are often due to high alpha activity rates or attenuation of the alpha emissions.
- 4.2 Isotope peak responses, when sufficiently high, may tail into other isotope ROIs. Th229 tailing into the Th230 region of interest is a recognized example. This interference is minimized by maintaining low activities of the Th229 tracer and monitoring of the separation of the ROIs for Th229 and Th230. The use of manual integration may be required.
- 4.3 Some isotopic elements are not distinguishable and are reported as an isotopic pair, unless specifically directed by the client not to do so. These pairs may be reported separately depending on the client's DQOs and the use-ability of the data. When reported separately, the narrative must describe the technical aspects of how the isotopic pair was divided.

4.3.1 Recognized Isotopic Pairs:

- 4.3.1.1 Plutonium 239/240
- 4.3.1.2 Uranium 235/236
- 4.3.1.3 Uranium 233/234
- 4.3.1.4 Curium 245/246
- 4.3.1.5 Curium 247/248

5 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

None.

5.3 PRIMARY MATERIALS USED

None.

6 EQUIPMENT AND SUPPLIES

6.1 Alpha spectroscopy system utilizing a computer based data acquisition system.

7 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002, current revision.

7.2 Commercially prepared alpha standards with all appropriate NIST Source Certificate information.

8 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 STL St. Louis supplies sample containers and chemical preservatives in accordance with the method. STL St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures. Sample volumes and preservative information is given in STL-PM-0002.

9 QUALITY CONTROL

9.1 See actinide preparation SOPs for additional information regarding QC types, frequency and preparation

9.2 Batch

9.2.1 Definition: environmental samples, which are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of a similar matrix, meeting the above mentioned criteria. Where no preparation method exists the batch is defined as environmental samples that are analyzed together with the same process and personnel, using the same lots of reagents, not to exceed 20 environmental samples. An analytical batch is composed of prepared environmental samples, extracts, digestates or concentrates that are analyzed

together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.

9.2.3 Each analytical batch may contain up to 20 environmental samples, a method blank, a single Laboratory Control Sample (LCS), a Matrix Spike and Sample Duplicate. In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2.4 Samples that have assigned QC limits different than the standard limits contained in QuantIMS QC code 01 must be batched separately, but can share the same QC samples.

9.3 Method Blank

9.3.1 Definition: a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.3.2 A method blank must be prepared with every batch (20 or fewer samples of the same matrix).

9.4 Laboratory Control Sample

9.4.1 Definition: a blank matrix spiked with a known amount of analyte(s), processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 An LCS must be prepared with every batch.

9.5 Matrix Spike/Sample Duplicate

9.5.1 Matrix Spike Definition: An aliquot of a field sample to which a known amount of target analyte(s) is added.

9.5.2 Sample Duplicate Definition: An additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.5.3 Additional MS and sample duplicates do not count towards the 20 samples in an analytical batch.

9.6 Procedural Variations

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. The Nonconformance Memo shall be filed in the project file and incorporated into the report narrative.

9.7 Nonconformance and Corrective Action

9.7.1 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager. See SOP

STL-QA-0036 for details regarding the NCM process.

10 CALIBRATION AND STANDARDIZATION

- 10.1 Initial calibrations are to performed according to the following schedule
 - 10.1.1 Energy calibrations shall be established for the alpha spectroscopy systems monthly, or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters.
 - 10.1.1.1 Energy Calibrations shall be performed using at least three isotopes within the energy range of 3-6 meV. Final peak energy positions of all oabserved isotopes shall be within +/- 40 leV of expected energy.
 - 10.1.2 Efficiency calibrations shall be established for the alpha spectroscopy systems monthly, or when the calibration quality control check indicates an unacceptable change in the efficiency calibration parameters.
 - 10.1.3 Background subtraction spectrum shall be established for the alpha spectroscopy systems monthly, or when the background quality control check indicates an unacceptable change in the daily background parameters.
- 10.2 Daily Checks
 - 10.2.1 Routine pulser quality control verifications are to performed each day of use.
 - 10.2.1.1 The pulser energy, peak centroid, peak resolution, peak area quality control for a detector shall be checked each day that the alpha spectroscopy system is used.
 - 10.2.2 Routine calibration, background and pulser quality control parameters using the "Boundary" out-of-range test will be found unacceptable if the value is outside reasonable parameter tolerance.
 - 10.2.2.1 The routine quality control check should be rerun to determine the statistical significance of the errant parameter.
 - 10.2.2.2 If the errant parameter is found acceptable for the rerun, the investigation will be noted in the instrument calibration and maintenance log.
 - 10.2.2.3 Check the expiration date of the radioactive standard to confirm the material is current.
 - 10.2.2.4 Check source positioning and all instrument settings.
 - 10.2.2.5 Check all cables for any apparent damage and to confirm that all cables are routed to proper connectors and are in good working order.
 - 10.2.3 If the instrument fails to meet the acceptance criteria, and the corrective actions above do not resolve the problem, the instrument must be "tagged" out of service, and documented in instrument calibration and maintenance log.
 - 10.2.4 The instrument may be returned to service once the malfunction has been corrected and the above acceptance criteria have been met. Note this action in the instrument calibration and maintenance log.
- 10.3 Calibration process in the Software
 - 10.3.1 Alpha Detector System Energy and Efficiency Calibration
 - 10.3.1.1 Place the correct source into the detector.
 - 10.3.1.2 In the Alphavision software, click on Calibration Icon.
 - 10.3.1.3 Click on detector to be calibrated.
 - 10.3.1.4 Select Calibration from the Tool Bar.

10.3.1.5 Select Process

10.3.1.5.1 The Calibration Explorer Window will appear.

10.3.1.6 In the General Window, name the Calibration with the month, year_detector format. (JAN2001_AV1)

10.3.1.7 Choose correct template for source to count

10.3.1.8 Click next

10.3.1.9 In the Acquisition window, confirm count time of 140 minutes

10.3.1.10 Click next

10.3.1.11 In the Energy/Efficiency Calibration Window, confirm the correct source is used, and select which shelf the source is on. (This will either be 1 or 2)

10.3.1.12 Click next

10.3.1.13 In the Report Window, select print on completion

10.3.1.14 Click finish

10.3.1.15 When count is complete, the Manual Energy and Efficiency Calibration Window will appear. In this window, select Calibration ROI, select Calibrate, and Save.

10.3.1.16 Repeat for each detector

10.3.1.17 Record the calibration in the Alpha Calibration & Maintenance Log Book.

10.3.2 Detector Background Counting

10.3.2.1 Select the Batch Icon

10.3.2.2 Select backgrounds from the Tool Bar

10.3.2.3 Select Process.

10.3.2.3.1 This will open the General Window in Batch Wizard

10.3.2.4 Name the background with month_year format. (JAN_04)

10.3.2.5 Select correct template (provided by analyst)

10.3.2.6 Click next.

10.3.2.7 In the Sample Window, add all detector names.

10.3.2.8 Click next

- 10.3.2.9 In the Acquisition Window, confirm count time is set at 800 minutes
- 10.3.2.10 Click next
- 10.3.2.11 In the Analysis Set Up Page, select Background Library and Background ROI.
- 10.3.2.12 Click next
- 10.3.2.13 In the Report Window, select print on completion
- 10.3.2.14 Click finish
- 10.3.2.15 The Detector Assignment worksheet will appear, select start now.
- 10.3.2.16 Record the backgrounds in the instrument calibration and maintenance log.
- 10.3.2.17 The background spectrum will be processed by the software
- 10.3.2.18 The detectors shall be "categorized" after each monthly background. (0-4 Counts in Region of Interest (i.e. Th230, Th232, U234, U238, Pu238, Pu239) will be labeled as Pantex. Low Level Detectors will be marked for the reaming detectors).

11 PROCEDURE

- 11.1 For sample preparation reference the applicable actinide SOPs: STL-RC-0040, STL-RC-0210, STL-RC-0232, STL-RC-0238, STL-RC-0241, STL-RC-0242, and STL-RC-0246.
- 11.2 Initial Setup
 - 11.2.1 Establish the normal instrument settings for all controls. Suggested settings are tabulated in Attachment 1.
 - 11.2.1.1 Detector specific high voltage settings and required polarity are listed on the detector manufacture's certificate.
 - 11.2.2 Pulser quality controls shall be checked before each use of the instrument.
- 11.3 Counting Samples
 - 11.3.1 In Radcapture, go to Alpha/Gamma, enter batch # and click Export Prep to Alphavision.
 - 11.3.2 In Alphavision, go to Process, select Batch to open the Batch Wizard:
 - 11.3.3 Choose Load from LIMS, and pick the batch.
 - 11.3.4 Choose test by clicking on the correct isotope test
 - 11.3.5 Select Next
 - 11.3.6 Click on blank, and then pick blank type (Uu blank, Pu Blank, ect)

- 11.3.7 Click on LCS, and then pick LCS type with correct spike number. For amount, use the spike aliquot amount (0.1, 0.2mL, 0.1326g, etc).
- 11.3.8 Select Next
- 11.3.9 Live time is count time. Enter correct count time for the batch, select Nuclide Library, choose correct ROI and tracer
- 11.3.10 Select next
- 11.3.11 Change TPU Sigma to 2 (unless otherwise noted in client requirements), select correct activity units (DPM, pCi, etc), select Activity concentration.
- 11.3.12 Select Next, two times
- 11.3.13 Select Print on Completion
- 11.3.14 Select Finish
- 11.3.15 Click and drag correct detectors to the correct sample id and select Start Now.
- 11.3.16 The spectrum will be processed by the software.
- 11.3.17 Backgrounds are checked after high activity samples
- 11.3.18 For DOE: the FWHM of each tracer peak shall be $\leq 100\text{keV}$; the tracer peak energy for each sample shall be within $\pm 50\text{keV}$ of the expected energy.

12 DATA ANALYSIS AND CALCULATIONS

- 12.1 Commonly used calculations (e.g. % recovery, RPD, uncertainty, MDC, tracer recovery) and standard instrument software calculations are given in the STL St. Louis LQM.
- 12.2 Isotope ROIs are given in Tables 1 in this SOP.
- 12.3 Any manual integration of a peak or group of peaks must be documented. In all instances where the data system report has been edited or where manual integration has been performed, the operator must clearly identify such edits or manual procedures. Reference SOP STL-QA-0040 for details.
- 12.4 The following data must be entered into this program or the default value verified:
 - 12.4.1 Sample Identification Number
 - 12.4.2 Sample aliquot used
 - 12.4.3 Tracer Identification Number
 - 12.4.4 Tracer volume used
 - 12.4.5 MDA constant (4.66)
 - 12.4.6 Currie's constant (2.71)
 - 12.4.7 Isotope of interest (library and regions)
 - 12.4.8 Matrix (water, soil, liquid, solid)
 - 12.4.9 Alpha Activity Concentration for each region of interest (ROI) in pCi/unit volume.

13 DATA ASSESSMENT AND ACCEPTANCE CRITERIA; CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

- 13.1 The data assessment and corrective action process is detailed through the Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: STL-QA-0036. A hardcopy of all the data assessment types and descriptions along with their associated corrective actions is included in the SOP. Below is a subset of the data assessment and QC excursion types within Clouseau; the text in underline is the exact "type" line in Clouseau. For a complete and current listing, please access the software program.
- 13.2 Method Blank
- 13.2.1 Acceptance Criteria:
- 13.2.1.1 No target analytes may be present in the method blank above the reporting limit.
- 13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:
- 13.2.2.1 Method Blank Contamination – See Clouseau NCM for corrective action. Note certain analytes are common laboratory contaminants which require special narrative comment. These compounds are so designated in Clouseau.
- 13.3 Laboratory Control Sample (LCS)
- 13.3.1 Acceptance Criteria:
- 13.3.1.1 All control analytes must be within established control limits for accuracy (%Recovery) and precision (RPD).
- 13.3.2 Corrective Action for LCS not meeting acceptance criteria:
- 13.3.2.1 LCS Spike Recovery excursion (high) – See Clouseau NCM for corrective action.
- 13.3.2.2 LCS Spike Recovery excursion (low) – See Clouseau NCM for corrective action.
- 13.3.2.3 RPD/RER Duplicate excursion – See Clouseau NCM for corrective action.
- 13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)
- 13.4.1 All analytes should be within established control limits for accuracy (%Recovery) and precision (RPD).
- 13.4.2 Corrective Action for MS/MSD not meeting acceptance criteria:
- 13.4.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration. See Clouseau NCM to determine if re-preparation re-analysis is required.
- 13.5 Sample result evaluation
- 13.5.1 Tracer/Carrier recovery low– See Clouseau NCM for corrective action.
- 13.5.2 Tracer/Carrier recovery high– See Clouseau NCM for corrective action.
- 13.5.3 Tracer recovery limits are given in the analytical SAC. See attached.
- 13.5.4 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:
- 13.5.4.1 minimum number of tracer counts
- 13.5.4.2 level of uncertainty
- 13.5.4.3 client project requirements/approval

13.5.5 These expectations will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.

13.6 Insufficient Sample

13.6.1 For each prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis and narrative comment stating such is included in the report narrative. The insufficient sample description is included in the the Clouseau NCM within the type defining the excursion.

14 **METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY**

14.1 Initial Demonstration

14.1.1 Initial and continuing demonstrations of capability requirements are established in STL St. Louis' LQM section 5.1.2

14.2 Training Qualification

14.2.1 The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14.2.2 The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in STL St. Louis' LQM section 5.1.2

14.2.3 Annually the analyst must successfully demonstrate proficiency to continuing to perform this analysis. See requirements in STL St. Louis' LQM section 5.1.2

15 **VALIDATION DATA**

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. STL ST Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method

16 **WASTE MANAGEMENT AND POLLUTION PREVENTION**

16.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method

The following waste streams are produced when this method is carried out.

- Contaminated disposable glass or plastic materials utilized in the analysis are disposed of in the sanitary trash. If the lab ware was used for the analysis of radioactive samples and contains radioactivity at a level of 100 cpm over background as determined by a GM meter, the lab ware will be collected in waste barrels designated for solid rad waste for disposal by the EH&S Coordinator.

17 **REFERENCES**

17.1 AlphaVision-32, Alpha Particle Spectrum Acquisition and Analysis for Microsoft Windows and NT, Software Version 5.0 Installation, User Interface and Reference Guide, Ortec (latest version)

- 17.2 OCTETE Plus, Integrated Alpha-Spectroscopy System Hardware Operating Manual, 777720, Ortec (latest version)
- 17.3 MAESTRO-32, MCA Emulator for Microsoft Windows, A65-B32 Software User's Manual, 777800, Ortec (latest version)
- 17.4 U.S. Nuclear Regulatory Commission, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment, Regulatory Guide 4.15.
- 17.5 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).
- 17.6 STL Quality Assurance Manual, current revision.
- 17.7 STL, St. Louis Laboratory Quality Manual, current revision
- 17.8 STL Corporate Safety Manual and St. Louis Facility Addendum (SOP STL-HS-0002), current revisions.
- 17.9 Associated SOPs, current revisions
 - 17.9.1 STL-PM-0002, Chain of Custody
 - 17.9.2 STL-QA-0002, Standard and Reagent Preparation
 - 17.9.3 STL-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
 - 17.9.4 STL-QA-0036 Non-Conformance Memorandum (NCM) Procedure
 - 17.9.5 STL-QA-0040, Manual Integration Procedure
 - 17.9.6 STL-RC-0040, Total Alpha Emitting Isotopes of Radium
 - 17.9.7 STL-RC-0238, ISOTOPIC URANIUM BY EICHROM® UTEVA RESIN FOR VARIOUS MATRICES
 - 17.9.8 STL-RC-0210, DETERMINATION OF POLONIUM-210 BY ALPHA SPECTROMETRY
 - 17.9.9 STL-RC-0232, ISOTOPIC THORIUM AND/OR NEPTUNIUM IN VARIOUS MATRICES BY EICHROM® TEVA SEPARATION RESIN
 - 17.9.10 STL-RC-0240, ISOTOPIC AMERICIUM, CURIUM, PLUTONIUM, THORIUM, AND URANIUM IN VARIOUS MATRICES BY EICHROM® SEPARATION RESIN
 - 17.9.11 STL-RC-0241, AMERICIUM, PLUTONIUM, CURIUM, AND URANIUM IN VARIOUS MATRICES BY EICHROM® UTEVA AND TRU RESINS (WITH VACUUM BOX SYSTEM)
 - 17.9.12 STL-RC-0242, ISOTOPIC THORIUM, PLUTONIUM AND URANIUM IN VARIOUS MATRICES BY EICHROM® SEPARATION RESINS
 - 17.9.13 STL-RC-0246, ISOTOPIC AMERICIUM, CURIUM, URANIUM IN VARIOUS MATRICES BY EICHROM® SEPARATION RESINS

18 Changes to Previous Revision

- 18.1 Revised Safety Section 5 and hazard tables in accordance with CSM.
- 18.2 Merged and revised waste management and pollution prevention sections, Section 16.
- 18.3 Added text to address sample collection references and capabilities, Section 8.
- 18.4 Added text to Section 12 referencing commonly used calculations are in the LQM.
- 18.5 Added DOC reference information to the method performance Section 14.
- 18.6 Added "definition of qualifiers" to Section 3.
- 18.7 Created a "Validation Data" section, Section 15.
- 18.8 Revised Quality Control, Section 9.
- 18.9 References, section 17 revised.

Table 1

Primary α Emissions for Selected Radionuclides

Isotope	a (keV)	a2 (keV)	a3 (keV)	a4 (keV)	a5 (keV)	a6 (keV)	a7 (keV)	a8 (keV)	a9 (keV)
²¹⁰ Polonium	5297								
²²⁶ Radium	4784	4602							
²²⁷ Thorium	5600	6308							
²²⁸ Thorium	5423	5340	5211	5177	5138				
²²⁹ Thorium	5052	5050	4978	4868	4901	4845	4838	4814	4798
²³⁰ Thorium	4688	4621	4480	4438	4372				
²³² Thorium	4010	3952	3830						
²³² Uranium	5320	5264	5137						
²³³ Uranium	4825	4804	4796	4783	4754	4729	4701	4664	
²³⁴ Uranium	4776	4724	4605						
²³⁵ Uranium	4597	4556	4414	4395	4370	4364	4344	4324	4216
²³⁸ Uranium	4196	4147	4040						
²³⁷ Neptunium	4873	4818	4804	4788	4772	4766	4707	4665	4640
²³⁶ Plutonium	5768	5721	5614						
²³⁸ Plutonium	5499	5456	5358						
^{239/240} Plutonium	5105	5143	5105						
²⁴² Plutonium	4901	4856							
²⁴⁴ Plutonium	4546	4589							
²⁴¹ Americium	5544	5512	5486	5443	5388				
²⁴³ Americium	5350	5319	5277	5324	5180				
²⁴² Curium	6069	6113							

Table 1

Primary α Emissions for Selected Radionuclides

Isotope	a (keV)	a2 (keV)	a3 (keV)	a4 (keV)	a5 (keV)	a6 (keV)	a7 (keV)	a8 (keV)	a9 (keV)
²⁴⁴ Curium	5805	5763							
²⁴⁵ Curium	5303	5362							
^{246/247} Curium	4868	5145							

STL Reference Data Summary

Structured Analysis Code: I-J2-2N-01-06

Target Analyte List: All Analytes

Matrix: WATER

Extraction: Extraction Chromatography - Sequential Actinides

Method: Am241, Cm243/244 (LONG CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Check List 6512						Spike List 6556								
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3984	Americium 241	0.1	pCi/L																
5618	Curium 243/244	0.1	pCi/L			C	Y			70	113	40	C	Y			70	130	40

STL Reference Data Summary

Structured Analysis Code: A-J2-2N-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: Extraction Chromatography - Sequential Actinides

Method: Am241, Cm243/244 (LONG CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Check List 6512				Spike List 6556				
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD
3984	Americium 241	0.1	pCi/g			0	C	Y			70	112	40
5818	Curium 243/244	0.1	pCi/g			0	C	Y			70	130	40

STL Reference Data Summary

Structured Analysis Code: A-K7-2N-01-06
Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Am241, Cm243/244 (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Run Date			Check List 6512			Spike List 6556		
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD
3984	Americium 241	0.1	pCi/g			0	C	Y			70	112	40
5618	Curium 243/244	0.1	pCi/g			0							
3986	Curium 242	0.1	pCi/g			0							
												70	130
													40

STL Reference Data Summary

Structured Analysis Code: A-1C-2N-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: LEACHATE, DI (ASTM D3987-85) - 18 hour
Method: Am241, Cm243/244 (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits		Run Date		T A		Check List 6353		Spike List 6354	
Syn	Compound	RL	Units	MDL	Units			Amt	Units	Amt	Units
3984	Americium 241	0.1	pCi/L		0	C	Y	73	117	40	50
5618	Curium 243/244	0.1	pCi/L		0	C	Y	70	130	40	50
											150
											150
											35

STL Reference Data Summary

Structured Analysis Code: I-J2-2J-01-06

Target Analyte List: All Analytes

Matrix: WATER

Extraction: Extraction Chromatography - Sequential Actinides

Method: Am241, Cm243/244 (SHORT CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

[illegible]

STL Reference Data Summary

Structured Analysis Code: A-J2-2J-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Am241, Cm243/244 (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits		Run Date		T A		Check List 6512		Spike List 6556	
								Amt	Units	Amt	Units
Syn	Compound	RL	Units	MDL	Units						
3984	Americium 241	1.0	pCi/g		0	C	Y				
5618	Curium 243/244	1.0	pCi/g		0						
3986	Curium 242	1.0	pCi/g		0						

STL Reference Data Summary

Structured Analysis Code: A-K7-2J-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Am241, Cm243/244 (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Check List 6512			Spike List 6556		
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	LCL UCL RPD
3984	Americium 241	1.0	pCi/g			C	Y			70 112 40
5618	Curium 243/244	1.0	pCi/g							70 130 40
3986	Curium 242	1.0	pCi/g							

STL Reference Data Summary

Structured Analysis Code: I-J2-3L-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: ISO NEPTUNIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Check List 6526			Spike List 6558		
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	LCL UCL RPD
4069	Neptunium 237	0.10	pCi/L			C	Y			79 106 40
4070	Np-237	0.10	pCi/L							86 110 40

STL Reference Data Summary

Structured Analysis Code: A-J2-3L-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: Sequential Actinides

Method: ISO NEPTUNIUM (LONG CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Check List 6526						Spike List 6558								
Syn	Compound	RL	Units	MDL	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4069	Neptunium 237	0.1	pCi/g		0	C	Y			80	120	40	C	Y			75	107	40

STL Reference Data Summary

Structured Analysis Code: A-K7-3L-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction:

Method:

QC Program: STANDARD TEST SET

Location: STL St. Louis

Extraction:

Method:

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List	Detection Limits					Check List 6526	Spike List 6558
	Units MDL	Run Date	T A Amt Units LCL UCL RPD T A Amt Units LCL UCL RPD				
RXN Compound	RL	Units	C Y C Y C Y				
4069 Neptunium 237	0.10 pCi/g	0					
4070 Np-237	0.10 pCi/g	0					

STL Reference Data Summary

Structured Analysis Code: I-J2-3K-01-06		Matrix: WATER
Target Analyte List: All Analytes		Extraction: Extraction Chromatography - Sequential Actinides
		Method: ISO NEPTUNIUM (SHORT CT) DOE A-01-R MOD
		QC Program: STANDARD TEST SET
		Location: STL St. Louis

Analyte List		Detection Limits				Check List 6526				Spike List 6558										
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4069	Neptunium 237	1	pCi/L			0		C	Y		79	106	40	C	Y			86	110	40

STL Reference Data Summary

Structured Analysis Code: A-J2-3K-01-06

Target Analyte List: All Analytes

Matrix:	SOLID
Extraction:	Extraction Chromatography - Sequential Actinides
Method:	ISO NEPTUNIUM (SHORT CT) DOE A-01-R MOD
QC Program:	STANDARD TEST SET
Location:	STL St. Louis

Analyte List		Detection Limits			Check List 6526					Spike List 6558										
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4069	Neptunium 237	1	pCi/g			0	C	Y			80	120	40	C	Y			75	107	40

STL Reference Data Summary

Structured Analysis Code: A-K7-3K-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: ISO NEPTUNIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits		Run Date		Check List 6526		Spike List 6558	
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units
4069	Neptunium 237	1	pCi/g		0	C	Y		
								80	120
								40	75
								C	107
								Y	40

STL Reference Data Summary

Structured Analysis Code: I-J2-2L-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso PLUTONIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Check List 6510			Spike List 6554		
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	Amt
5463	Plutonium 244	0.1	pCi/L							
3989	Plutonium 238	0.1	pCi/L			C	Y	80	124	40
4093	Plutonium 239/40	0.1	pCi/L			C	Y	80	118	40
4091	Plutonium 242	0.1	pCi/L							

STL Reference Data Summary

Structured Analysis Code: A-J2-2L-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: Extraction Chromatography - Sequential Actinides

Method: Iso PLUTONIUM (LONG CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List					
Syn	Compound	RL	Detection Limits Units MDL	Run Date	
T	A	Units	LCL	UCL	RPD
Check List 6510					
T	A	Amt	Units	LCL	UCL RPD
Spike List 6554					
T	A	Amt	Units	LCL	UCL RPD
5463	Plutonium 244	0.1	pCi/g		
3989	Plutonium 238	0.1	pCi/g		
4093	Plutonium 239/40	0.1	pCi/g		
4091	Plutonium 242	0.1	pCi/g		

STL Reference Data Summary

Structured Analysis Code: A-K7-2L-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: As Received, Extraction Chromatography - Seq. Actinides

Method: Iso PLUTONIUM (LONG CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits				Check List 6510				Spike List 6554										
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3989	Plutonium 238	0.1	pCi/g			0	C	Y			74	124	40	C	Y			67	138	40
4093	Plutonium 239/40	0.1	pCi/g			0	C	Y			75	120	40	C	Y			73	131	40
4091	Plutonium 242	0.1	pCi/g			0														

STL Reference Data Summary

Structured Analysis Code: I-J2-2H-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso PLUTONIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List			Detection Limits			Check List 6510			Spike List 6554		
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL UCL RPD
5463	Plutonium 244	1.0	pCi/L			0					
3989	Plutonium 238	1.0	pCi/L			0	C	Y	80	124	40
4093	Plutonium 239/40	1.0	pCi/L			0	C	Y	80	118	40
4091	Plutonium 242	1.0	pCi/L			0					

STL Reference Data Summary

Structured Analysis Code: A-J2-2H-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: Extraction Chromatography - Sequential Actinides

Method: Iso PLUTONIUM (SHORT CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

[illegible]

STL Reference Data Summary

Structured Analysis Code: A-K7-2H-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Iso PLUTONIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List			Detection Limits			Check List 6510					Spike List 6554									
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3989	Plutonium 238	1.0	pCi/g			0	C	Y			74	124	40	C	Y			67	138	40
4093	Plutonium 239/40	1.0	pCi/g			0	C	Y			75	120	40	C	Y			73	131	40
4091	Plutonium 242	1.0	pCi/g			0														

STL Reference Data Summary

Structured Analysis Code: A-1C-2H-01-06

Target Analyte List: All Analytes

Matrix:	SOLID
Extraction:	LEACHATE, DI (ASTM D3987-85) - 18 hour
Method:	Iso PLUTONIUM (SHORT CT) DOE A-01-R MOD
QC Program:	STANDARD TEST SET
Location:	STL St. Louis

Analyte List		Detection Limits			Check List 6349					Spike List 6350										
		RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3989	Plutonium 238	1.0	pCi/L			0	C	Y			72	109	40	C	Y			50	150	40
4093	Plutonium 239/40	1.0	pCi/L			0	C	Y			79	122	40	C	Y			50	150	40
4091	Plutonium 242	1.0	pCi/L			0	T	Y			30	110	0	T	Y			30	110	0

STL Reference Data Summary

Structured Analysis Code: I-J2-2O-01-06

Target Analyte List: All Analytes

Matrix: WATER

Extraction: Extraction Chromatography - Sequential Actinides

Method: Iso THORIUM (LONG CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Check List 6513					Spike List 6557										
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4115	Thorium 228	0.1	pCi/L			0	C	Y			81	130	40	C	Y			74	113	40
4117	Thorium 230	0.1	pCi/L			0	C	Y			75	128	40	C	Y			72	120	40
4121	Thorium 232	0.1	pCi/L			0	C	Y			77	131	40	C	Y			70	120	40

STL Reference Data Summary

Structured Analysis Code: I-88-20-01-06

Target Analyte List: All Analytes

Matrix:	WATER
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Extraction:	NO SAMPLE PREPARATION PERFORMED / DIRECT INJ
Method:	Iso THORIUM (LONG CT) DOE A-01-R MOD
QC Program:	STANDARD TEST SET
Location:	STL St. Louis

Analyte List		Detection Limits			Check List 6513						Spike List 6557									
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4115	Thorium 228		ug/L			0	C	Y			81	130	40	C	Y			74	113	40
4117	Thorium 230		ug/L			0	C	Y			75	128	40	C	Y			72	120	40
4121	Thorium 232		ug/L			0	C	Y			77	131	40	C	Y			70	120	40

STL Reference Data Summary

Structured Analysis Code: A-J2-2O-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: Extraction Chromatography - Sequential Actinides

Method: Iso THORIUM (LONG CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Check List 6513					Spike List 6557										
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4115	Thorium 228	0.1	pCi/g			0	C	Y			76	131	40	C	Y			75	150	40
4117	Thorium 230	0.1	pCi/g			0	C	Y			73	120	40	C	Y			78	150	40
4121	Thorium 232	0.1	pCi/g			0	C	Y			75	129	40	C	Y			76	150	40

STL Reference Data Summary

Structured Analysis Code: A-K7-2O-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: As Received, Extraction Chromatography - Seq. Actinides

Method: Iso THORIUM (LONG CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Run Date	Check List 6513				Spike List 6557									
		RL	Units	MDL		Units	T	A	Amt	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
Syn	Compound																		
4115	Thorium 228	0.1	pCi/g		0		C	Y			76	131	40		C	Y	75	150	40
4117	Thorium 230	0.1	pCi/g		0		C	Y			73	120	40		C	Y	78	150	40
4121	Thorium 232	0.1	pCi/g		0		C	Y			75	129	40		C	Y	76	150	40

STL Reference Data Summary

Structured Analysis Code: A-88-2O-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJ

Method: Iso THORIUM (LONG CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Check List 6513						Spike List 6557									
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4115	Thorium 228		mg/kg			0	C	Y			76	131	40	C	Y			75	150	40
4117	Thorium 230		mg/kg			0	C	Y			73	120	40	C	Y			78	150	40
4121	Thorium 232		mg/kg			0	C	Y			75	129	40	C	Y			76	150	40

STL Reference Data Summary

Structured Analysis Code: I-J2-2K-01-06

Target Analyte List: All Analytes

MATRIX: WATER

Extraction: Extraction Chromatography - Sequential Actinides

Method: Iso THORIUM (SHORT CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Check List 6513			Spike List 6557		
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	RPD
5768	Thorium 228, dissolved	1.0	pCi/L		0					
5769	Thorium 230, dissolved	1.0	pCi/L		0					
5770	Thorium 232, dissolved	1.0	pCi/L		0					
4115	Thorium 228	1.0	pCi/L		0	C	Y	81	130	40
4117	Thorium 230	1.0	pCi/L		0	C	Y	75	128	40
4121	Thorium 232	1.0	pCi/L		0	C	Y	77	131	40
									74	113
									72	120
									70	120

STL Reference Data Summary

Structured Analysis Code: A-J2-2K-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso THORIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Check List 6513			Spike List 6557		
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	LCL UCL RPD
4115	Thorium 228	1.0	pCi/g		0	C	Y	C	Y	75 131 40
4117	Thorium 230	1.0	pCi/g		0	C	Y	C	Y	73 120 40
4121	Thorium 232	1.0	pCi/g		0	C	Y	C	Y	75 129 40
										76 150 40
										78 150 40
										76 150 40

STL Reference Data Summary

Structured Analysis Code: A-K7-2K-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction:

Method:

QC Program: STANDARD TEST SET

Location: STL St. Louis

As Received, Extraction Chromatography - Seq. Actinides

ISO THORIUM (SHORT CT) DOE A-01-R MOD

STANDARD TEST SET

STL St. Louis

Analyte List		Detection Limits				Check List 6513				Spike List 6557										
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4115	Thorium 228	1.0	pCi/g			0	C	Y			76	131	40	C	Y			75	150	40
4117	Thorium 230	1.0	pCi/g			0	C	Y			73	120	40	C	Y			78	150	40
4121	Thorium 232	1.0	pCi/g			0	C	Y			75	129	40	C	Y			76	150	40

STL Reference Data Summary

Structured Analysis Code: I-J2-2M-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Check List 6511				Spike List 6555								
		RL	Units	MDL	Units	T	A	Amt	Units	LCL	UCL	RPD					
Syn	Compound				Run Date												
4119	Thorium 231	0.1	pCi/L		0												
4123	Thorium 234	0.1	pCi/L		0												
5779	Uranium 234, dissolved	0.1	pCi/L		0												
5773	Uranium 238, dissolved	0.1	pCi/L		0												
5789	Uranium 233/234	0.1	pCi/L		0												
4129	Uranium 234	0.1	pCi/L		0	C	Y		76	117	40	C	Y	59	150	40	
5790	Uranium 235/236	0.1	pCi/L		0												
4133	Uranium 238	0.1	pCi/L		0	C	Y		79	117	40	C	Y	63	150	40	

STL Reference Data Summary

Structured Analysis Code: I-88-2M-01-06		Matrix: WATER	
Target Analyte List: All Analytes		Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJI	
		Method: Iso URANIUM (LONG CT) DOE A-01-R MOD	
		QC Program: STANDARD TEST SET	
		Location: STL St. Louis	

Analyte List		Detection Limits			Check List 6511			Spike List 6555		
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	LCL UCL RPD
3743	Total Uranium		ug/ml							
4129	Uranium 234		ug/ml		0	C	Y	76	117	40
4131	Uranium 235		ug/ml		0	C	Y	79	117	40
4133	Uranium 238		ug/ml		0	C	Y	63	150	40

STL Reference Data Summary

Structured Analysis Code: A-IB-2M-01-06

Target Analyte List: All Analytes

Matrix: SOLID
 Extraction: Uranium (ONLY) by Ion Ex and/or Extraction Chromatogra
 Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List		Detection Limits		Check List 6511		Spike List 6555			
Syn	Compound	RL	Units	MDL	Run Date	T	A	Amt	Units
4129	Uranium 234	0.1	pCi/g		0	C	Y	70	127 40 C Y
4131	Uranium 235	0.1	pCi/g		0				81 150 40
5385	Uranium 236	0.1	pCi/g		0				
4133	Uranium 238	0.1	pCi/g		0	C	Y	70	126 40 C Y
									73 165 40

STL Reference Data Summary

Structured Analysis Code: A-J2-2M-01-06

Target Analyte List: All Analytes

Matrix: SOLID
 Extraction: Extraction Chromatography - Sequential Actinides
 Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List		Detection Limits			Check List 6511			Spike List 6555		
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	LCL UCL RPD
4119	Thorium 231	0.1	pCi/g		0					
4123	Thorium 234	0.1	pCi/g		0					
5789	Uranium 233/234	0.1	pCi/g		0					
4129	Uranium 234	0.1	pCi/g		0	C	Y	70	127	40
5790	Uranium 235/236	0.1	pCi/g		0					81 150 40
4133	Uranium 238	0.1	pCi/g		0	C	Y	70	126	40
										73 165 40

STL Reference Data Summary

Structured Analysis Code: A-K7-2M-01-06

Target Analyte List: All Analytes

Matrix: SOLID
 Extraction: As Received, Extraction Chromatography - Seq. Actinides
 Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List		Detection Limits			Check List 6511			Spike List 6555		
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	LCL UCL RPD
4119	Thorium 231	0.1	pCi/g		0					
4123	Thorium 234	0.1	pCi/g		0					
5789	Uranium 233/234	0.1	pCi/g		0					
4129	Uranium 234	0.1	pCi/g		0	C	Y	70	127	40
4131	Uranium 235	0.1	pCi/g		0					81 150 40
5790	Uranium 235/236	0.1	pCi/g		0					
5385	Uranium 236	0.1	pCi/g		0					
4133	Uranium 238	0.1	pCi/g		0	C	Y	70	126	40
										73 165 40

STL Reference Data Summary

Structured Analysis Code: A-88-2M-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction:	NO SAMPLE PREPARATION PERFORMED / DIRECT INJ
Method:	Iso URANIUM (LONG CT) DOE A-01-R MOD
QC Program:	STANDARD TEST SET
Location:	STL St. Louis

Analyte List		Detection Limits			Check List 6511						Spike List 6555									
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3743	Total Uranium		ug/g			0														
4129	Uranium 234		ug/g			0	C	Y		70	127	40	C	Y				81	150	40
4131	Uranium 235		ug/g			0														
4133	Uranium 238		ug/g			0	C	Y		70	126	40	C	Y				73	165	40

STL Reference Data Summary

Structured Analysis Code: I-J2-21-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Check List 6511			Spike List 6555		
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units
4119	Thorium 231	1	pCi/L			0				
4123	Thorium 234	1	pCi/L			0				
5773	Uranium 238, dissolved	1	pCi/L			0				
5789	Uranium 233/234	1	pCi/L			0				
4129	Uranium 234	1	pCi/L			0	C	Y	76	117 40
5781	Uranium 234, Dissolved	1	pCi/L			0				59 150 40
5790	Uranium 235/236	1	pCi/L			0				
5784	Uranium 238, Dissolved	1	pCi/L			0				
4133	Uranium 238	1	pCi/L			0	C	Y	79	117 40
										63 150 40

STL Reference Data Summary

Structured Analysis Code: A-IB-21-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: Uranium (ONLY) by Ion Ex and/or Extraction Chromatogra

Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits				Check List 6511				Spike List 6555			
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	LCL	UCL	RPD	T A Amt Units LCL UCL RPD
4129	Uranium 234	1	pCi/g			0	C	Y		70	127	40	C Y 81 150 40
4131	Uranium 235	1	pCi/g			0							
5385	Uranium 236	1	pCi/g			0							
4133	Uranium 238	1	pCi/g			0	C	Y		70	126	40	C Y 73 165 40

STL Reference Data Summary

Structured Analysis Code: A-J2-2I-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD

Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Run Date	Check List 6511				Spike List 6555									
Syn	Compound	RL	Units	MDL		Units	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL
4119	Thorium 231	1	pCi/g		0														
4123	Thorium 234	1	pCi/g		0														
5789	Uranium 233/234	1	pCi/g		0														
4129	Uranium 234	1	pCi/g		0	C	Y		70	127	40	C	Y			81	150	40	
5790	Uranium 235/236	1	pCi/g		0														
4133	Uranium 238	1	pCi/g		0	C	Y		70	126	40	C	Y			73	165	40	

STL Reference Data Summary

Structured Analysis Code: A-K7-2I-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Check List 6511			Spike List 6555		
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units
4119	Thorium 231	1	pCi/g			0				
4123	Thorium 234	1	pCi/g			0				
5789	Uranium 233/234	1	pCi/g			0				
4129	Uranium 234	1	pCi/g			0	C	Y		81 150 40
4131	Uranium 235	1	pCi/g			0				
5790	Uranium 235/236	1	pCi/g			0				
5385	Uranium 236	1	pCi/g			0				
4133	Uranium 238	1	pCi/g			0	C	Y		73 165 40

STL Reference Data Summary

Structured Analysis Code: A-1C-2I-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: LEACHATE, DI (ASTM D3987-85) - 18 hour
Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Check List 6351			Spike List 6352		
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	LCL UCL RPD
3743	Total Uranium		pCi/L		0					
5780	Total Uranium, dissolved		pCi/L		0					
4129	Uranium 234		pCi/L		0	C	Y	73	115	40
4131	Uranium 235		pCi/L		0					50 150 40
4133	Uranium 238		pCi/L		0	C	Y	70	130	40
										50 150 40

STL Reference Data Summary

Structured Analysis Code: I-JB-3P-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Pu-242
Method: Plutonium-242 by DOE A-01-R Mod
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Check List 6541			Spike List 6541		
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	LCL UCL RPD
5463	Plutonium 244	1	pCi/L							
4091	Plutonium 242	1	pCi/L			C	Y			

STL Reference Data Summary

Structured Analysis Code: A-JB-3P-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Pu-242
Method: Plutonium-242 by DOE A-01-R Mod
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits		Run Date		Check List 6541		Spike List 6541	
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units
5463	Plutonium 244	1	pCi/g		0				
4091	Plutonium 242	1	pCi/g		0	C	Y		
								75	128
								40	75
									128
									40

STL Reference Data Summary

Structured Analysis Code: I-JC-3Q-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - IsoCm
Method: Cm245/246, Cm 247/248, Am243 DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Check List 6586						Spike List 6542									
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3993	Americium 243	1	pCi/L			0	C	Y			82	129	40	C	Y			75	125	40
5551	Curium-245/246	1	pCi/L			0														
5619	Curium 247/248	1	pCi/L			0														

STL Reference Data Summary

Structured Analysis Code: A-JC-3Q-01-06

Target Analyte List: All Analytes

Matrix:	SOLID
Extraction:	Extraction Chromatography - IsoCm
Method:	Cm245/246, Cm 247/248, Am243 DOE A-01-R MOD
QC Program:	STANDARD TEST SET
Location:	STL St. Louis

Analyte List					
Syn	Compound	RL	Detection Limits		
			Units	MDL	Units
			Run Date		
			T	A	Amt
			C	Y	
Check List 6586					
			LCL	UCL	RPD
			89	129	40
			C	Y	
Spike List 6542					
			LCL	UCL	RPD
			75	125	40

STL Reference Data Summary

Structured Analysis Code: I-JD-3R-01-06

Target Analyte List: All Analytes

Matrix:	WATER
Extraction:	Extraction Chromatography - U-232
Method:	Iso URANIUM-232 by DOE A-01-R Mod
QC Program:	STANDARD TEST SET
Location:	STL St. Louis

Analyte List		Detection Limits			Check List 6587						Spike List 6587									
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4166	Uranium-232	1	pCi/L			0	C	Y			70	96	40	C	Y			70	96	40

STL Reference Data Summary

Structured Analysis Code: A-JD-3R-01-06

Target Analyte List: All Analytes

Matrix:	SOLID
Extraction:	Extraction Chromatography - U-232
Method:	Iso URANIUM-232 by DOE A-01-R Mod
QC Program:	STANDARD TEST SET
Location:	STL St. Louis

Analyte List		Detection Limits			Check List 6587						Spike List 6587								
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4166	Uranium-232	1	pCi/g			0	C	Y		74	100	40	C	Y			74	100	40

STL Reference Data Summary

Structured Analysis Code: I-J2-2M-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Run Date			Check List 6511			Spike List 6555		
Syn	Compound	RL	Units	MDL	Units		T	A	Amt	Units	LCL	UCL	RPD
4119	Thorium 231	0.1	pCi/L		0								
4123	Thorium 234	0.1	pCi/L		0								
5779	Uranium 234, dissolved	0.1	pCi/L		0								
5773	Uranium 238, dissolved	0.1	pCi/L		0								
5789	Uranium 233/234	0.1	pCi/L		0								
4129	Uranium 234	0.1	pCi/L		0	C	Y		76		117	40	C
5790	Uranium 235/236	0.1	pCi/L		0								59
4133	Uranium 238	0.1	pCi/L		0	C	Y		79		117	40	C
													63
													150
													40

STL Reference Data Summary

Structured Analysis Code: I-88-2M-01-06

Target Analyte List: All Analytes

Matrix: WATER

Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJ
 Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List		Detection Limits			Check List 6511				Spike List 6555											
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3743	Total Uranium		ug/ml			0														
4129	Uranium 234		ug/ml			0	C	Y			76	117	40	C	Y			59	150	40
4131	Uranium 235		ug/ml			0														
4133	Uranium 238		ug/ml			0	C	Y			79	117	40	C	Y			63	150	40

STL Reference Data Summary

Structured Analysis Code: A-IB-2M-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: Uranium (ONLY) by Ion Ex and/or Extraction Chromatography
 Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List		Detection Limits			Run Date	Check List 6511				Spike List 6555										
Syn	Compound	RL	Units	MDL		Units	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4129	Uranium 234	0.1	pCi/g		0		C	Y		70	127	40	C	Y			81	150	40	
4131	Uranium 235	0.1	pCi/g		0															
5385	Uranium 236	0.1	pCi/g		0															
4133	Uranium 238	0.1	pCi/g		0		C	Y		70	126	40	C	Y			73	165	40	

STL Reference Data Summary

Structured Analysis Code: A-J2-2M-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Run Date	Check List 6511			Spike List 6555								
Syn	Compound	RL	Units	MDL		T	A	Units	LCL	UCL	RPD	T	A	Units	LCL	UCL	RPD
4119	Thorium 231	0.1	pCi/g		0												
4123	Thorium 234	0.1	pCi/g		0												
5789	Uranium 233/234	0.1	pCi/g		0												
4129	Uranium 234	0.1	pCi/g		0	C	Y		70	127	40	C	Y		81	150	40
5790	Uranium 235/236	0.1	pCi/g		0												
4133	Uranium 238	0.1	pCi/g		0	C	Y		70	126	40	C	Y		73	165	40

STL Reference Data Summary

Structured Analysis Code: A-K7-2M-01-06

Target Analyte List: All Analytes

Matrix: SOLID
 Extraction: As Received, Extraction Chromatography - Seq. Actinides
 Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
 QC Program: STANDARD TEST SET
 Location: STL St. Louis

Analyte List		Detection Limits			Check List 6511			Spike List 6555		
Syn	Compound	RL	Units	MDL	Units	Run Date	T A	Amt	Units	LCL UCL RPD
4119	Thorium 231	0.1	pCi/g			0				
4123	Thorium 234	0.1	pCi/g			0				
5789	Uranium 233/234	0.1	pCi/g			0				
4129	Uranium 234	0.1	pCi/g			0	C Y	70	127 40	C Y 81 150 40
4131	Uranium 235	0.1	pCi/g			0				
5790	Uranium 235/236	0.1	pCi/g			0				
5385	Uranium 236	0.1	pCi/g			0				
4133	Uranium 238	0.1	pCi/g			0	C Y	70	126 40	C Y 73 165 40

STL Reference Data Summary

Structured Analysis Code: A-88-2M-01-06

Target Analyte List: All Analytes

Matrix: SOLID

Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJI

Method: Iso URANIUM (LONG CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Run Date	Check List 6511				Spike List 6555									
Syn	Compound	Units	MDL	Units		T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3743	Total Uranium	ug/g			0														
4129	Uranium 234	ug/g			0	C	Y		70	127	40	C	Y			81	150	40	
4131	Uranium 235	ug/g			0														
4133	Uranium 238	ug/g			0	C	Y		70	126	40	C	Y			73	165	40	

STL Reference Data Summary

Structured Analysis Code: A-IB-2I-01-06

Target Analyte List: All Analytes

Matrix:	SOLID
Extraction:	Uranium (ONLY) by Ion Ex and/or Extraction Chromatogra
Method:	Iso URANIUM (SHORT CT) DOE A-01-R MOD
QC Program:	STANDARD TEST SET
Location:	STL St. Louis

Analyte List		Detection Limits			Run Date	Check List 6511				Spike List 6555									
Syn	Compound	Units	MDL	Units		T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4129	Uranium 234	1	pCi/g		0				C	Y							81	150	40
4131	Uranium 235	1	pCi/g		0														
5385	Uranium 236	1	pCi/g		0														
4133	Uranium 238	1	pCi/g		0				C	Y							73	165	40

STL Reference Data Summary

Structured Analysis Code: A-J2-21-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Run Date			Check List 6511			Spike List 6555		
Syn	Compound	RL	Units	MDL	Units		T	A	Amt	Units	LCL	UCL	RPD
4119	Thorium 231	1	pCi/g		0								
4123	Thorium 234	1	pCi/g		0								
5789	Uranium 233/234	1	pCi/g		0								
4129	Uranium 234	1	pCi/g		0		C	Y			70	127	40
5790	Uranium 235/236	1	pCi/g		0								
4133	Uranium 238	1	pCi/g		0		C	Y			70	126	40
												81	150
												73	165

STL Reference Data Summary

Structured Analysis Code: A-K7-2I-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: STL St. Louis

Analyte List		Detection Limits			Check List 6511			Spike List 6555		
Syn	Compound	RL	Units	MDL	Units	T	A	Amt	Units	Units
4119	Thorium 231	1	pCi/g							
4123	Thorium 234	1	pCi/g							
5789	Uranium 233/234	1	pCi/g							
4129	Uranium 234	1	pCi/g			C	Y		81	150 40
4131	Uranium 235	1	pCi/g							
5790	Uranium 235/236	1	pCi/g							
5385	Uranium 236	1	pCi/g							
4133	Uranium 238	1	pCi/g			C	Y		73	165 40

STL Reference Data Summary

Structured Analysis Code: A-1C-2I-01-06

Target Analyte List: All Analytes

SOLID

Matrix:

Extraction: LEACHATE, DI (ASTM D3987-85) - 18 hour

Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		Detection Limits			Check List 6351					Spike List 6352										
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3743	Total Uranium		pCi/L			0														
5780	Total Uranium, dissolved		pCi/L			0														
4129	Uranium 234		pCi/L			0	C	Y			73	115	40	C	Y			50	150	40
4131	Uranium 235		pCi/L			0														
4133	Uranium 238		pCi/L			0	C	Y			70	130	40	C	Y			50	150	40

STL Reference Data Summary

Structured Analysis Code: I-J2-2I-01-06

Target Analyte List: All Analytes

WATER

Matrix:

Extraction: Extraction Chromatography - Sequential Actinides

Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD

QC Program: STANDARD TEST SET

Location: STL St. Louis

Analyte List		RL	Detection Limits		Run Date	Check List 6511				Spike List 6555								
Syn	Compound		Units	MDL		T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL
4119	Thorium 231	1	pCi/L		0													
4123	Thorium 234	1	pCi/L		0													
5773	Uranium 238, dissolved	1	pCi/L		0													
5789	Uranium 233/234	1	pCi/L		0													
4129	Uranium 234	1	pCi/L		0	C	Y		76	117	40	C	Y		59	150	40	
5781	Uranium 234, Dissolved	1	pCi/L		0													
5790	Uranium 235/236	1	pCi/L		0													
5784	Uranium 238, Dissolved	1	pCi/L		0													
4133	Uranium 238	1	pCi/L		0	C	Y		79	117	40	C	Y		63	150	40	

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TRENT

STL

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STL ST. LOUIS STANDARD OPERATING PROCEDURE

TITLE: SAMPLE RECEIPT AND CHAIN OF CUSTODY

(SUPERSEDES: STL-PM-0002 Rev 3)

Prepared by: _____

Approved by: Jill Clarke
Supervisor/Lead Analyst

Approved by: Elaine Wild
Quality Assurance Manager

Approved by: Michael J. Rohlf
Environmental Health and Safety Coordinator

Approved by: John M. ...
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1.0 SCOPE AND APPLICATION

- 1.1 The purpose of this procedure is to describe the receipt of samples by the Sample Control Department and to describe the intra-laboratory custody transfer of samples.
- 1.2 This procedure applies to all samples arriving at the STL St. Louis laboratory.
- 1.3 Radiological screening of samples received is an integral part of the sample receipt process. The SOPs on the screening and classifying of samples must be used in conjunction with this procedure, STL-RC-0010.
- 1.4 NELAC (National Environmental Laboratory Accreditation Conference) specifies requirements under which any NELAC accredited laboratory will accept samples.

2.0 SUMMARY

- 2.1 All samples received by the STL St. Louis laboratory, will be received by the Sample Control Department or authorized designates. Upon receipt, samples will be checked for completeness of associated paperwork, leakage/breakage, proper preservation and sample integrity.
- 2.2 All discrepancies will be noted on the Condition Upon Receipt (CUR) Form. This form must be completed at the time the items are being checked. If any item is completed by someone other than the initiator, then that person is required to apply his/her initials and the date next to that item. The number of the CUR Form is written on the client Chain of Custody for traceability purposes.
- 2.3 Samples are entered into the laboratory information management system (QuantIMS). Internal sample ID numbers are assigned by QuantIMS.
- 2.4 Custody documentation and proper storage conditions are maintained until samples are returned to client or disposal authorization is granted.
- 2.5 All associates involved in the sample receipt process must read, understand and perform according to this Standard Operating Procedure. Any questions should be brought to the attention of the Sample Control Department or the QA Department.
- 2.6 Responsibilities
 - 2.6.1 Sample Control Department: Accepts initial custody of samples received. Are responsible for the integrity and security of samples while in storage. Follow the sample login initiation procedures stipulated in this SOP. Are responsible to regard any precautions associated with a sample.

3.0 DEFINITIONS

- 3.1 See the STL Quality Management Plan (QMP) and STL St. Louis Laboratory Quality Manual (LQM) for a glossary of common laboratory terms and data reporting qualifiers.
- 3.2 Chain of Custody (COC): Documentation of physical possession of a sample. The hardcopy form that begins the documentation process and is initiated by client. The signature of the laboratory Sample Control personnel or authorized designates denotes release of custody by the client and possession by the laboratory. **Figure 1.**
- 3.3 Condition Upon Receipt (CUR) Form: Generated at time of sample receipt. (Documents the items checked during the receipt of samples prior to login.) **Figure 2.**
- 3.4 QuantIMS: The laboratory information management system that tracks sample information.

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4.0 INTERFERENCES

4.1 Not Applicable

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 The preparation of reagents for the sample receiving area will be conducted in a fume hood with the sash closed as far as the operations will permit. If an operation requires the hood sash to be raised to above face level, a face shield shall be used.

5.2.2 All employees receiving samples shall be trained in accordance with the applicable DOT regulations. Personnel authorized to receive samples shall be designated in writing.

5.2.3 Exposure to chemicals will be maintained as low as reasonably achievable. Sample coolers/shipping containers shall be opened in a well-ventilated area (i.e., sample receiving area). If a broken container is found, the cooler/shipping container shall be transferred to a fumehood and carefully unpacked. All sample coolers/shipping containers which show signs of damage will be opened in front of or in an operating fume hood.

5.2.4 Cut resistant gloves must be worn while initially inspecting coolers that are received or while cleaning coolers for re-use.

5.3 PRIMARY MATERIALS USED

5.3.1 The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limits (2)	Signs of Exposure	PPE Required
Nitric Acid	Corrosive Poison Oxidizer	2 ppm, 5 mg/m ³	Inhalation may cause coughing, choking, and irritation of the nose, throat, and respiratory tract. Skin contact can cause redness, pain, and severe skin burns. Concentrated solutions can stain the skin a yellow-brown color. Vapors are irritating to the eyes and contact may cause severe burns.	Safety Glasses, Labcoat, Gloves, Hood or Faceshield

Notes: Nitric acid is a strong oxidizer. Contact with other material may cause fire. CORROSIVE. Liquid and mist cause severe burns to all body tissue.

Sodium Hydroxide	Corrosive Poison	2 mg/m ³ TWA	Nose irritation; pneumonitis; eye, skin burns; temporary loss of hair	Safety Glasses, Labcoat, Gloves, Hood or Faceshield
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Notes: Sodium hydroxide is corrosive. Causes burns to any area of contact. Reacts with water, acids and other materials.

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Material (1)	Hazards	Exposure Limits (2)	Signs of Exposure	PPE Required
Sulfuric Acid	Corrosive Poison Cancer Hazard	1 mg/m3	Inhalation may cause irritation of the nose and throat, and labored breathing. Skin contact symptoms include redness, pain, and severe burning. Eye contact can cause blurred vision, redness, pain, and severe tissue burns.	Safety Glasses, Labcoat, Gloves, Hood or Faceshield
Notes: Sulfuric acid is extremely corrosive. Liquid and mist cause severe burns to all body tissue.				
Hydrochloric Acid	Poison Corrosive	5 ppm Ceiling	Inhalation symptoms include coughing, choking, inflammation of the nose, throat, and upper respiratory tract. Skin contact can cause redness, pain, severe skin burns, and discoloration. Vapors are irritating to the eyes. Contact may cause severe burns.	Safety Glasses, Labcoat, Gloves, Hood or Faceshield
Notes: Hydrochloric acid is a corrosive. Liquid and mist causes severe burns to all body tissue.				
1 - Always add acid to water to prevent violent reactions.				
2 - Exposure limit refers to the OSHA regulatory exposure limit.				

6.0 EQUIPMENT AND SUPPLIES

- 6.1 pH paper, wide range.
- 6.2 Disposable pipettes for sampling liquids.
- 6.3 Tongue depressors, plastic vials.
- 6.4 Thermometers, electronic, calibrated.
- 6.5 Survey Meter, sensitive to Alpha and/or Beta/Gamma. Calibrated.
- 6.6 Protective clothing, safety glasses, gloves (including cut resistant), lab coats.
- 6.7 Swipes for surveying for loose surface contamination.

7.0 REAGENTS AND STANDARDS

- 7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP STL-QA-0002, current revision.
- 7.2 Reagent water, obtained from the Milli-Q system.
- 7.3 Sulfuric acid, concentrated (36N), ACS grade, STL certified.
 - 7.3.1 Sulfuric acid, 18N (1:1) – Carefully add 500 ml of concentrated H₂SO₄ to 500 ml of reagent water while stirring. Mix well.
- 7.4 Nitric acid, concentrated (16N), ACS grade, STL certified (or trace metal grade if preserving aqueous metals samples).
 - 7.4.1 Nitric acid (ACS or trace metals grade), 8N (1:1) – Carefully add 500 ml of concentrated HNO₃ to 500 ml of reagent water while stirring. Mix well.
- 7.5 Hydrochloric acid, concentrated (12N), ACS grade, STL certified.
 - 7.5.1 Hydrochloric acid, 6N (1:1) – Carefully add 500 ml of concentrated HCl to 500 ml of reagent water while stirring. Mix well.
- 7.6 Sodium hydroxide, approximately 10N (50% w/w), reagent grade. CAUTION - Sodium hydroxide is corrosive. Causes burns to any area of contact. Reacts with water, acids and other materials.

8.0 SAMPLE COLLECTION PRESERVATION AND STORAGE

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- 8.1 STL St. Louis supplies sample containers and chemical preservatives in accordance with the method. STL St. Louis does not perform sample collection. Samplers should reference the methods referenced and other applicable sample collection documents for detailed collection procedures.

9.0 CALIBRATION AND STANDARDIZATION

- 9.1 Thermometers used for sample storage cooler and sample arrival cooler measurement will be calibrated as described in SOP STL-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."
- 9.2 Survey meters used for the detection of radiological contamination will be calibrated as described in SOP STL-RP-0032, "Instrumentation and Surveillance."

10.0 QUALITY CONTROL

- 10.1 Matrix Spike/Matrix Spike Duplicates and/or Sample Duplicates are logged-in on samples as designated by the client on the COC, or instruction from the Project Manager.
- 10.2 Method Blanks and LCS QC samples are initiated by the sample preparation analysts and are not part of the log-in process.
- 10.3 Percent moisture determination is logged-in for all inorganic and organic soil samples, except if wet weight ("as is") results are requested by the client, there is limited sample volume, or performing % moisture is determined to be hazardous on the given matrix. Radiological samples are dried and ground as part of the routine procedure and % moisture is performed upon request.

11.0 PROCEDURE

11.1 Sample Acceptance Policy

- 11.1.1 NELAC specifies requirements under which any NELAC accredited laboratory will accept samples. STL St. Louis will review your sample shipment against those requirements listed below, and will communicate any discrepancies to you. Your project manager will assist you in the appropriate resolution of any issues related to sample receipt. Please contact your project manager with any questions.
- 11.1.2 When completing the chain of custody form, sign your name in the "relinquished by" box.
- 11.1.3 NELAC requirements are as follows:
- 11.1.3.1 Proper, full and complete documentation, which includes sample identification, the location, date and time of collection, the collector's name, the preservation type, the sample matrix type, the requested testing method, and any special remarks concerning the samples shall be provided.
 - 11.1.3.2 Each sample shall be labeled with unique, durable and indelible identification.
 - 11.1.3.3 The samples shall be collected in the appropriate sample containers.
 - 11.1.3.4 The samples shall arrive at the laboratory within the specified holding time for the analyses requested.
 - 11.1.3.5 Sufficient sample volume must be available to perform the requested analyses.
 - 11.1.3.6 The laboratory will notify the client upon sample receipt if the samples exhibit obvious signs of damage, contamination or inadequate preservation.

11.2 Sample Handling and Storage

- 11.2.1 Upon receipt, laboratory samples shall be handled and stored in such a manner as: (a) to ensure the safety of all personnel, (b) to maintain the integrity of the samples in accordance with the requested analytical methods, and (c) to prevent potential cross contamination of samples.

11.2.2 Sample Segregation

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- 11.2.2.1 Samples are stored in locations remote from standard reference materials, calibration standards and any other chemicals or reagents. These locations are designated for sample storage only and include walk-in type coolers located in Sample Control or other designated areas within the laboratory.
- 11.2.2.2 To prevent contamination from other samples, samples to be analyzed for volatile organics (VOAs) are stored in locations designated for these samples only. VOA refrigerator/cooler storage blanks are analyzed to monitor air contaminants. Refer to SOP STL-QA-0031, "VOA Holding Blank Analysis."
- 11.2.2.3 Samples received for volatiles analysis are stored in the interim storage refrigerator SCV1, located in Sample Control, until retrieval by the volatiles lab and placed in refrigerators in the volatiles laboratory.
 - 11.2.2.3.1 Actual drinking water samples submitted to the laboratory for analysis by EPA Method 524.2 are stored separate from EPA Method 8260 samples.
- 11.2.2.4 Radioactive samples may be stored in any refrigerator or walk in cooler.
- 11.2.2.5 Coolers or refrigerators designated for sample storage are maintained at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$. Samples that have been submitted to the laboratory that do not require cold preservation are stored in ambient storage areas.
- 11.2.2.6 Samples that have been submitted to the laboratory which require locked storage (e.g. litigation), and samples requiring cold preservation will be stored in a locking refrigerator. Keys to access the cabinet or refrigerator will be maintained by a member of the Sample Control Department.
- 11.2.2.7 Samples that have been submitted to the laboratory that need quarantine (e.g. quarantine due to foreign or known-regulated domestic soil) will be stored on a segregated shelf marked as "Quarantined Soil", within a walk-in cooler. Each sample must be labeled as regulated soil under USDA permit. A sign must be placed on the outside of the walk-in cooler which states: "Foreign soil and/or regulated domestic soil to be used in accordance with USDA, APHIS, PPQ Soil Permit and Compliance Agreement."
- 11.2.2.8 Samples requiring screening for radioactivity have their activities reviewed by the Radiation Safety Officer (RSO) or designee. Based on the activity present, samples will be categorized as Rad Category 1, 2, 3, or 4 (Categories 2, 3 or 4 require a Radiation Work Permit (RWP)). This radioactivity level designation is entered into QuantIMS after sample login.
- 11.2.2.9 Protective clothing is worn at all times when handling sample containers. Gloves must be changed when necessary to prevent cross contamination.
- 11.2.2.10 Samples are handled to ensure that labels or markings on each container remain intact.
- 11.2.2.11 Samples are kept securely capped except when checking pH or when removing aliquots for the screening of radioactive material.

11.3 Sample Receipt Condition

- 11.3.1 Airbills will be checked upon sample arrival with the time and date of arrival noted as verified by the signature of a Sample Control Associate or designate. Airbill numbers and the courier name will be listed on the CUR Form. Receipt of the shipment is documented in the sample receipt log maintained in Sample Control. Airbills or shipping papers, if available, are maintained in the Project Files. If the samples have been designated a Proper

Shipping Name listed below or are DOT class 7 primary or subsidiary hazard code, the samples shall be received per STL-RP-0050, "Purchase, Receipt, Handling, and Identification of Radioactive Material."

- Radioactive Material, Excepted Package, Limited Quantity of Material
- Radioactive Material, n.o.s.
- Radioactive Material, Low Specific Activity (LSA), n.o.s.
- Radioactive Material, Surface Contaminated Object (SCO)

- 11.3.2 When the sample cooler is opened and a Limited Quantity warning is in the package or the samples have radiation stickers/labels, the shipment shall be received per STL-RP-0050, "Purchase, Receipt, Handling, and Identification of Radioactive Material."

Note: No packing material shall be discarded until the completion of step 11.3.8.

- 11.3.3 Evidential (custody) seals on shipping container(s) are inspected. Any evidence of tampering (i.e., broken seals), or if the seals are of a non-tamper evident type, is noted on the CUR Form.
- 11.3.4 The temperature of the coolers is checked upon arrival. This is done by quickly opening the cooler, inserting the probe of a calibrated electronic thermometer, closing the cooler, and leaving it until the thermometer reaches a constant temperature. The probe shall be placed in close proximity to the samples (i.e., between the sample container and its surrounding bubble wrap). The temperature is then noted and documented on the CUR Form. (Note: When multiple coolers are received in a shipment, the specific contents of a cooler that was outside acceptance criteria must be listed.) The Project Manager is notified of unacceptable temperatures. The acceptable temperature is $4^{\circ} \pm 2^{\circ}$ C. Alternatively, the temperature of a cooler blank, if provided, will be recorded. Temperatures are recorded as whole numbers.
- 11.3.5 If the shipment is from a known or suspected radiological site, the sample cooler/shipping container is surveyed for loose surface contamination. Refer to STL-RP-0032, "Instrumentation and Surveillance," for instructions on the proper use of radiacs.
- 11.3.6 If the shipment is of a foreign or known-regulated domestic soil, all soil residue on packaging must be treated with one of the schedules below:

11.3.6.1 Dry Heat Temperatures	Exposure Period
110.0 – 120.5 degrees Celsius (230-240 degrees F)	16 hours
121.0 – 154.0 degrees Celsius (250-309 degrees F)	2 hours
154.4 – 192.5 degrees Celsius (310-379 degrees F)	30 minutes
193.0 – 220.0 degrees Celsius (380-429 degrees F)	4 minutes
221.0 – 232.0 degrees Celsius (430-450 degrees F)	2 minutes

Do not start counting time until entire mass reaches the required temperature.

- 11.3.6.2 Used shipping containers must be decontaminated by one of the treatments approved for soil or destroyed by incineration. The coolers used to ship soil samples must be generously sprayed with an approved disinfectant such as bleach, quaternary ammonia, or 70% alcohol solution to the point of runoff, allowed to drain into a municipal water system and air dried. No stockpiling of used empty containers allowed.
- 11.3.7 Sample coolers/shipping containers with loose surface contamination greater than 100 cpm above background on a beta/gamma frisker require the immediate notification of the RSO or designate. The shipment shall be received per STL-RP-0050, "Purchase, Receipt, Handling, and Identification of Radioactive Material."

11.3.8 Samples are checked against the Client COC. All discrepancies are noted on the Client COC and detailed on the CUR Form. The Client COC should indicate:

- 11.3.8.1 Client Name
- 11.3.8.2 Unique sample number for each sample
- 11.3.8.3 Date of sampling
- 11.3.8.4 Time of sampling
- 11.3.8.5 Matrix sampled
- 11.3.8.6 Chemical preservatives added
- 11.3.8.7 Analyses requested
- 11.3.8.8 Numbers of containers collected for each analysis
- 11.3.8.9 Pertinent observations (odor, hazards, etc.)

11.3.9 COC's will be checked for any requested tests that may have short hold times as indicated by Figure 2, (ex. BOD, pH, etc.).

11.3.10 Samples determined to have short holding times will be removed from cooler/shipping container and will be placed in pass through window to wet chem. along with a copy of COC.

11.3.10.1 Note: it is the responsibility of Wet Chem department to check for short hold time samples.

11.3.11 The pH is taken on all samples requiring acid/base preservation (with the exception of volatile organics samples and TOX samples, see Section 11.3.10). All discrepancies are noted on the CUR Form. A scant amount of sample is removed by dipping a clean pipette into the sample and applying a drop to pH paper.

11.3.11.1 When aqueous samples from DOE-Albuquerque SMOs are received, the pH of both the preserved and nonpreserved samples must be taken. The pH readings must be documented and communicated, via the CUR, to the Project Manager as soon as possible so that the client may have the opportunity to request corrective action, if necessary.

11.3.11.2 When samples are received with improper preservation and when the Project Manager requests, after corresponding with the client, Sample Control will preserve the samples with the appropriate preservative. Only preservatives from STL certified lots shall be used.

11.3.11.2.1 Sufficient acid or base is added to the sample container to adjust the sample to the required pH. The following guidelines should be used for the initial pH adjustment:

11.3.11.2.1.1 Sulfuric acid: 4 ml (1:1) for a 1 liter container.

11.3.11.2.1.2 Nitric acid: 6 ml (1:1) for a 1 liter container.

11.3.11.2.1.3 Hydrochloric acid: 7.8 ml (1:1) for a 1 liter container.

11.3.11.2.1.4 Sodium hydroxide: 1.25 ml (50% w/v) for a 500 ml container.

11.3.11.2.2 Verify pH after addition of acid or base. If the pH is still outside preservation requirements, the client must be contacted before adding additional acid or base.

11.3.11.2.3 For metals analysis, sample aliquots may not be removed for analysis until 16 hours after preservation. For radiological analyses, sample aliquots may not be removed for analysis until 24 hours after preservation.

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- 11.3.11.2.4 Preservation by the lab must be documented on both the Condition Upon Receipt (CUR) Form and on the sample container itself. Documentation on the CUR Forms consists of the date and time of preservation and the lot number of the acid used. Required documentation on the container is the date and time of preservation and the date and time aliquots can be removed for analysis.
- 11.3.12 The pH of samples to be analyzed for volatile organics or TOX is measured by the analyst after removing the aliquot for analysis.
- 11.3.13 After completion by the Sample Control Department, the CUR Form is forwarded to the applicable Project Manager for review and client notification, if necessary. Client notification and any required corrective action will be documented on the CUR Form.
- 11.4 Sample Log in
 - 11.4.1 Samples are logged into QuantIMS which assigns a lot number to the group of samples being entered. Quotes specifying the required analyses and any specific instructions are created in QuantIMS by the Project Manager and are used by the Sample Control Department during login.
 - 11.4.1.1 When entering a composite sample which lists multiple collection times, the latest time will be used as the time of sample collection.
 - 11.4.2 Composite Samples
 - 11.4.2.1 There are instances where the client requests the laboratory to perform sample compositing after collection/receipt. The instructions for how compositing is to be performed (namely what containers to put together) must be communicated to Sample Control prior to the time of receipt.
 - 11.4.2.1.1 Project Managers will request the client to denote "compositing" on the COC instructions.
 - 11.4.2.1.2 The Project Manager will put Composite Notification and Instructions in the specific client Quote comments. These comments will appear first to highlight it for Sample Control.
 - 11.4.2.1.3 If the PM receives advanced notice of samples or copies of COCs, the PM will inform their Sample Control contact.
 - 11.4.2.2 Samples are logged in for all requested analyses and put in a separate storage bin to await compositing. This bin is identified as being "on hold" until compositing is complete.
 - 11.4.2.3 The samples awaiting compositing are stored in C Cooler and the applicable laboratory supervisor is notified of samples needing compositing and forwarded a copy of the compositing instructions from the Project Manager.
 - 11.4.2.3.1 Organic Only Analyses – Contact Organic Prep Supervisor
 - 11.4.2.3.2 Inorganic Only Analyses – Contact Metals Supervisor
 - 11.4.2.3.3 Radiochemical Only Analyses – Contact Separation Supervisor
 - 11.4.2.3.4 Cross Department Analyses – Contact Metals Supervisor
 - 11.4.2.4 After samples are composited, the department supervisor, or designee, will return samples to Sample Control for distribution to appropriate storage locations.
 - 11.4.3 PM will include a rad screening test in the quote, if samples are from a potential radioactive site. Sample control will choose this test code and aliquot accordingly.
 - 11.4.3.1.1 Aliquoting of rad screen will be done by using a clean wooden tongue depressor for solids, or a disposable transfer pipette for liquids.
 - 11.4.3.1.2 The aliquot will be placed in clean plastic vial and be labeled with a copy of the original sample label.
 - 11.4.3.1.3 Screen vial(s) will then be placed in a designated spot for the Rad Department to collect daily and analyze.

- 11.4.4 Samples are placed into the appropriate refrigerated or ambient sample storage area. Storage location is entered into QuantIMS.
- 11.4.5 Upon completion of the login, a Client Analysis Summary is printed using the Worksheet Generation Utility or directly from QuantIMS. This summary is reviewed for accuracy by the Project Manager. Electronic mail notifications of rush samples are sent automatically by the Worksheet Generation Utility to appropriate personnel.
- 11.5 Receipt of Samples after normal working hours
 - 11.5.1 Associates other than those in the Sample Control Department may receive non-radiological samples after normal working hours provided they have been trained in DOT awareness by the EH&S Coordinator.
 - 11.5.2 The following actions will be performed when samples are received after normal working hours:
 - 11.5.2.1 The chain-of-custody (COC) will be signed and dated by both the receiving Associate and the courier. The COC is maintained in the Sample Control office.
 - 11.5.2.2 The evidential (custody) seals on the container(s) are checked per section 11.3.3. Discrepancies regarding the evidential seals are noted on the COC.
 - 11.5.2.3 The temperature of the cooler(s) is taken per section 11.3.4 and recorded on the COC. Thermometers are kept in Sample Control.
 - 11.5.2.4 The cooler is then placed into the walk-in cooler designated "C". Put the COC in Sample Control by the first log-in PC.
- 11.6 Internal Custody Transfer
 - 11.6.1 Custody transfer to the individual laboratories is accomplished using the Sample Transfer Utility (STU). Alternate methods of documenting custody transfers may be utilized for short holdtime samples which have not been logged into QuantIMS. Reference SOP STL-QA-0039 regarding the STU process and operation.
 - 11.6.2 When one of the individual laboratories is prepared to perform an analysis, an analyst will retrieve the necessary sample(s) from the applicable walk-in cooler or ambient storage area. The analyst is directed to the correct storage location by the cooler/shelf number listed on their backlog. After retrieval, the analyst will accept custody of the sample(s) using the Sample Transfer Utility.
 - 11.6.3 After aliquots are removed for analysis, sample containers may be returned to the Sample Control Department for disposal or return to the client. The Sample Transfer Utility (STU) documents this custody transfer. If sample disposal is to be performed by the lab analyst (i.e., for metals water samples), the analyst retains final custody of the sample.
 - 11.6.4 If specific project plans require a person-to-person transfer of sample custody, an Internal Chain of Custody Form can be printed. In this case, a member of Sample Control and the analyst will sign the "Relinquished" and "Received" boxes upon custody transfer.
 - 11.6.5 For samples submitted to the laboratory for specific projects requiring person-to-person custody transfer, samples must be returned to the special storage area immediately after the sample aliquot is taken.

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12.1 Preservation and Holding Time

12.1.1 Preservation requirements and holding times are listed in the specific analytical methods and are summarized in the Preservation Table maintained in Sample Control and included in this SOP, Table 1.

12.2 Documentation concerning the receipt, login, and transfer of samples within the laboratory are maintained in the Project Files and contains the following:

- 12.2.1.1 Original airbill or courier shipping papers (if available),
- 12.2.1.2 Original CUR Form,
- 12.2.1.3 Original client Chain of Custody,
- 12.2.1.4 Client Analysis Summary,
- 12.2.1.5 Sample disposal documentation once samples have been disposed. Please refer to STL-HS-0004, "Hazardous Waste Management Plan", for the procedure used for sample disposal.

13.0 REFERENCES

- 13.1 STL Quality Management Plan (QMP), current revision.
- 13.2 STL St. Louis Laboratory Quality Manual (LQM), current revision
- 13.3 STL Corporate Safety Manual and St. Louis Facility Addendum (SOP STL-HS-0002), current revisions.
- 13.4 National Environmental Laboratory Accreditation Conference, Constitution, Bylaws, and Standards, EPA 600/R-98/151, US of Research and Development, May 2001.
- 13.5 United States Dept. of Agriculture, Foreign and Known-Regulated Domestic Soil Compliance Agreement, Agreement No. SC 04
- 13.6 Associated SOPs:
 - 13.6.1 STL-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
 - 13.6.2 STL-QA-0025, Temperature Monitoring
 - 13.6.3 STL-QA-0031, VOA Holding Blank Analysis
 - 13.6.4 STL-QA-0039, Sample Transfer Utility (STU)
 - 13.6.5 STL-RP-0031, Radiation Work Permits
 - 13.6.6 STL-RC-0010, Screening Samples for the Presence of Radioactive Material
 - 13.6.7 STL-RP-0050, Purchase, Receipt, Handling, and Identification of Radioactive Material
 - 13.6.8 STL-RP-0032, Instrumentation and Surveillance
 - 13.6.9 STL-HS-0004, Hazardous Waste Management Plan
 - 13.6.10 STL-HS-0006, Quarantine Soil Procedure

14.0 CHANGES FROM PREVIOUS REVISION

- 14.1 Revised Section 13, added 13.6.10
- 14.2 Revised Section 11.2.2.7

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Figure 1
(Example Chain of Custody Form)

[illegible]

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Figure 2
(Example Condition Upon Receipt Form)

Lot No(s) _____

(Note all associated lot No's)

Condition Upon Receipt Form
St. Louis Laboratory

Client:		COC/RFA No:		Date:	
Quote No:		Initiated By:		Time:	

Shipping Information

Shipper Name:		Multiple Packages:		Y	N	N/A
Shipper No(s):*	1.	Sample Temperature(s):**	1.			
	2.		2.			
	3.		3.			
	4.		4.			
	5.		5.			

*Numbered shipping lines correspond to Numbered Sample Temp lines.

**Sample must be received at 4°C ± 2°C-If not, note contents below.

Temperature variance does NOT affect the following analysis/matrix: Metals-Liquid
Rad tests –

Liquids or Solids.

Condition/Variance (Circle "Y for yes, "N" for no and "N/A" for not applicable):

1.	Y N	Sample received in undamaged condition?	7.	Y N	Sample received with Chain of Custody?
2.	Y N N/A	Sample received with proper pH ¹ ? (N/A for soil samples) If NO: sample ID _____ Preservative _____ Lot _____ Date _____ Time _____ Sticker applied Y/N	8.	Y N	Chain of Custody matches sample IDs on container(s)?
3.	Y N	If N/A-Was pH taken by original STL Lab?	9.	Y N N/A	Custody seal received intact?
4.	Y N	Sample received in proper containers?	10.	Y N N/A	Custody seal tamper evident?
5.	Y N	Sample volume sufficient for analysis?	11.	Y N N/A	Custody seal on bottles intact?
6.	Y N N/A	Headspace in VOA or TOX liquid samples? (If yes, note sample ID's below)	12.	Y N N/A	Custody seal tamper evident?
¹ For DOE-AL (Pantex, LANL, Sandia) sites, verify pH of all containers received, EXCEPT VOA, TOX, and soils.			13.	Y N N/A	Was Internal COC/CUR rec'd?

Notes:

PM Notified of Short Hold samples: Y N PM Initials:

Corrective Action:

Client's Name:		Informed by:		By:	
Sample(s) processed "as is".					
Sample(s) on hold until:		If released, notify:			
Project Management Review:		Date:			

THIS FORM MUST BE COMPLETED AT THE TIME THE ITEMS ARE BEING CHECKED IN. IF ANY ITEM IS COMPLETED BY SOMEONE OTHER THAN THE INITIATOR, THEN THAT PERSON IS REQUIRED TO APPLY THEIR INITIAL AND THE DATE NEXT TO THAT ITEM.

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Table 1
Sample Container, Volumes, and Preservation Requirements

Analysis	Method	Matrix	Volume	Preservative	Hold Time
Wet Chemistry					
Alkalinity: Total, Carbonate, Bicarbonate	310.1	Water	100 mL P,G 20 g	Cool, 4 deg. C	28 days
		Soil	P,G	Cool, 4 deg. C	14 days
Ammonia	350.1	Water	20 mL P,G 5 g	H2SO4 (pH <2), Cool	28 days
		Soil	P,G	Cool, 4 deg. C	28 days
BOD	405.1	Water	500 mL P,G	Cool, 4 deg. C	48 hrs
Bromide	300.0, 9056A	Water	50 mL P,G 5 g	Cool, 4 deg. C	28 days
		Soil	P,G	Cool, 4 deg. C	28 days
CBOD	5210B	Water	500 mL P,G	Cool, 4 deg. C	48 hrs
Chloride	300.0, 9056A	Water	50 mL P,G 5 g	Cool, 4 deg. C	28 days
		Soil	P,G	Cool, 4 deg. C	28 days
Cyanide	9010A, 9012, 335.2	Water	50 mL P,G 5 g	NaOH (pH >12), Cool	14 days
		Soil	P,G	Cool, 4 deg. C	14 days
COD	410.4	Water	50 mL P,G	H2SO4 (pH, 2), Cool	28 days
Conductivity	120.1, 9050	Water	50 mL P,G 20 g	Cool, 4 deg. C	28 days
		Soil	P,G	Cool, 4 deg. C	28 days
Ferrous Iron	SM 3500D	Water	100 mL P,G 10 g	Cool, 4 deg. C	24 hrs
		Soil	P,G	Cool, 4 deg. C	24 hrs
Fluoride	300.0, 9056A (IC)	Water	50 mL P,G 5 g	Cool, 4 deg. C	28 days
		Soil	P,G	Cool, 4 deg. C	28 days

	340.2 (probe)	Water	50 mL	Cool, 4 deg. C	28 days
Flashpoint (Ignitability)	1010	Water	100 mL	Cool, 4 deg. C	180 days
			P,G		
		Soil	100 g	Cool, 4 deg. C	180 days
			P,G		
			100 mL		
Hardness	130.2	Water		HNO3 (pH <2)	180 days
Hexavalent Chromium	7196A	Water	50 mL	Cool, 4 deg. C	24 hrs
			P,G		
		Soil	20 g	Cool, 4 deg. C	28 days
			P,G		
Iodide	300.0	Water	50 mL	Cool, 4 deg. C	7 days
Nitrate	300.0, 353.1, 9056A	Water	50 mL	Cool, 4 deg. C	48 hrs
			P,G		
		Soil	5 g	Cool, 4 deg. C	48 hrs
			P,G		
Nitrate/Nitrite	353.1	Water	50 mL	H2SO4 (pH <2), Cool	28 days
			P,G		
		Soil	5 g	Cool, 4 deg. C	28 days
			P,G		
Nitrite	300.0, 353.1, 9056A	Water	100 mL	Cool, 4 deg. C	48 hrs
			P,G		
		Soil	5 g	Cool, 4 deg. C	48 hrs
			P,G		
Oil & Grease	9070, 413.1, 1664	Water	1000 mL	HCl (pH <2), Cool	28 days
			G		
	9071	Soil	50 g	Cool, 4 deg. C	28 days
			G		
Orthophosphate	300.0, 365.1, 9056A	Water	50 mL	Cool, 4 deg. C	48 hrs
			P,G		
		Soil	5 g	Cool, 4 deg. C	48 hrs
			P,G		
Paint Filter	9095	Soil	100 g	Cool, 4 deg. C	28 days
			P,G		
Perchlorate	314	Water	50 mL	Cool, 4 deg. C	28 days
			P,G		
		Soil	5 g	Cool, 4 deg. C	28 days
			P,G		
pH	9040, 150.1	Water	50 mL	Cool, 4 deg. C	48 hrs
			P,G		
	9045	Soil	20 g	Cool, 4 deg. C	48 hrs
			P,G		
Phenols	9066, 420.2	Water	50 mL	H2SO4 (pH <2), Cool	28 days
			P,G		
		Soil	5 g	Cool, 4 deg. C	28 days
			G		
Phosphorus	365.1	Water	50 mL	H2SO4 (pH <2), Cool	28 days
			P,G		
		Soil	10 g	Cool, 4 deg. C	28 days

			P,G		
Reactive Cyanide	SW846 Chapter 7	Water	50 mL P,G	NaOH (pH >12), Cool	14 days
		Soil	10 g P,G	Cool, 4 deg. C	None
Reactive Sulfide	SW846 Chapter 7	Water	50 mL P,G	NaOH,Zn Ac. (pH >9), Cool	7 days
		Soil	10 g P,G	Cool, 4 deg. C	None
Residual Chlorine	330.3	Water	200 mL P,G	Light Resistant Container	24 hrs
Settleable Solids	160.4	Water	1000 mL P,G	Cool, 4 deg. C	48 hrs
Sulfate	300.0, 9056A	Water	50 mL P,G	Cool, 4 deg. C	28 days
		Soil	5 g P,G	Cool, 4 deg. C	28 days
Sulfide	9030, 376.1	Water	200 mL P,G	NaOH,Zn Ac. (pH >9), Cool	7 days
	9030, 376.1	Soil	25 g P,G	Cool, 4 deg. C	7 days
Sulfite	377.1	Water	50 mL P,G	Cool, 4 deg. C	24 hrs
		Soil	25 g P,G	Cool, 4 deg. C	24 hrs
Surfactants	425.1	Water	1000 mL P,G	Cool, 4 deg. C	48 hrs
TDS	160.1	Water	100 mL P,G	Cool, 4 deg. C	7 days
TOC	415.1, 9060	Water	100 mL P,G	H2SO4 (pH <2), Cool	28 days
		Soil	5 g P,G	Cool, 4 deg. C	28 days
TKN	351.1	Water	20 mL P,G	H2SO4 (pH <2), Cool	28 days
		Soil	0.1 g	Cool, 4 deg. C	28 days
Total Solids	160.3	Water	100 mL P,G	Cool, 4 deg. C	7 days
TOX (EOX)	450.1, 9020	Water	500 mL G	H2SO4 (pH <2), Cool	28 days
		Soil	1 g G	Cool, 4 deg. C	28 days
TSS	160.2	Water	100 mL P,G	Cool, 4 deg. C	7 days
Turbidity	180.1	Water	100 mL P,G	Cool, 4 deg. C	48 hrs

Microbial

Fecal Coliform	Water	200 mL G	Sterile container, Sodium thiosulfate tablet Cool, 4 deg. C	24 hrs
E-Coli	Water	200 mL G	Sterile container, Sodium thiosulfate tablet Cool, 4 deg. C	24 hrs
Chlorophyll A	Water	500 mL G	Light resistant container (e.g. amber glass) Cool, 4 deg. C	24 hrs

VOA Organics

BTEX	8020/8021, 8260, OA-1	Water	2 X 40 mL G	HCl (pH <2), Cool, 4 deg. C	14 days
		Soil	5g G	Cool, 4 deg. C	14 days
TCLP ZHE Volatiles	1311/ 8260	Solid	2 X 120 mL G	Cool, 4 deg. C	14 days
TPH, Gasoline	8015, OA-1	Water	2 X 40 mL G	HCl (pH <2), Cool, 4 deg. C	14 days
		Soil	5g G	Cool, 4 deg. C	14 days
Volatiles	624, 8260 (5mL purge)	Water	2 X 40 mL G	HCl (pH <2), Cool, 4 deg.C	14 days/ 7 days, if not preserved w/ HCl
	524.2, 624, 8260 (25mL purge)	Water	2 X 40 mL G	HCl (pH <2), Cool, 4 deg.C	14 days/ 7 days, if not preserved w/ HCl
	8260 (5030)	Soil	5g G	Cool, 4 deg. C	14 days
	8260 (5035)	Soil	Encore Sampler x 2	Cool, 4 deg. C	14 days/ 14 days/ 48 hrs (if not rec'd in Sodium Bisulfate preservative)

Extractable Organics

1 L					
Dioxin	8280, 8290, 613	Water	G	Cool, 4 deg. C	30 days
		Soil	30 g G	Cool, 4 deg. C	30 days
Explosives	8330	Water	1 L	Cool, 4 deg. C	7 days

		Soil	G 30 g G 1 L	Cool, 4 deg. C	14 days
PAHs	8310	Water	G 30 g	Cool, 4 deg. C	7 days
		Soil	G 1 L	Cool, 4 deg. C	14 days
Herbicides	8151	Water	G 50 g	Cool, 4 deg. C	7 days
		Soil	G 1 L	Cool, 4 deg. C	14 days
Pesticides	608, 8081	Water	G 30 g	Cool, 4 deg. C	7 days
	8081	Soil	G 1 L	Cool, 4 deg. C	14 days
PCBs	608, 8082	Water	G 30 g	Cool, 4 deg. C	7 days
	8082	Soil	G 1 L	Cool, 4 deg. C	14 days
Phenol	8040	Water	G 30 g	Cool, 4 deg. C	7 days
		Soil	G 1 L	Cool, 4 deg. C	14 days
Semivolatiles	625, 8270	Water	G 30 g	Cool, 4 deg. C	7 days
	8270	Soil	G 100 g	Cool, 4 deg. C	14 days
TCLP Herbicide	1311/ 8151	Solid	G 100 g	Cool, 4 deg. C	14 days
TCLP Pesticide	1311/ 8081	Solid	G 100 g	Cool, 4 deg. C	14 days
TCLP Semivolatile	1311/ 8270	Solid	G 1 L	Cool, 4 deg. C	14 days
TPH, Diesel	8015, OA-2	Water	G 30 g	HCl (pH <2), Cool, 4 deg. C	7 days
		Soil	G	Cool, 4 deg. C	14 days

Metals

Mercury	7470	Water	30ml P,G	HNO3 (pH <2)	28 days
	7471	Soil	0.6 g G	Cool, 4 deg. C	28 days
Metals	200.7, 200.8, 6010, 6020	Water	50mL P,G	HNO3 (pH <2)	180 days
		Soil	1 g G	Cool, 4 deg. C	180 days
					28 days (mercury),
TCLP Metals	1311/ 6010, 7470	Solid	100 g G	Cool, 4 deg. C	180 days

TCLP

CWET, TTLC, and SPLP are the same containers and preservatives as TCLP.

TCLP ZHE Volatiles	1311/ 8260	Solid	2 X 120 mL G	Cool, 4 deg. C	14 days
Full Extraction	1311	Solid	100 g G	Cool, 4 deg. C	14 days
TCLP ZHE Volatiles	1311/ 8260	Liquid	3 X 40 mL G	Cool, 4 deg. C	14 days
Full Extraction	1311	Liquid	2 Liters G	Cool, 4 deg. C	14 days

* Notes:

1. Clients are required to do the phase separation when a liquid sample is in multiple phases.
2. When a liquid sample has a % solid content of less than 25 % of the volume, more sample volume will be required to provide an adequate amount of solids for extraction.
3. For samples requiring Matrix QC, 3 times the volume is required.

Radiochemistry

Carbon-14 (C-14)	EERF C-01	Water	500 mL P,G	None	180 days
		Soil	5 g P,G	None	180 days
Chlorine-36 (Cl-36)		Water			180 days
Gross Alpha/Beta	900.0, 9310	Water	200 mL P,G	HNO3 (pH <2)	180 days
		Soil	1 g P,G	None	180 days
Iodine-129	GA-01-R MOD	Water	1 L P,G	None	180 days
		Soil	650 g P,G	None	180 days
	Liquid Scint	Water	2 L P,G	None	180 days

Iron-55		Water	500 ml	None	180 days
		Soil	5 g	None	180 days
Gamma Scan	901, HASL 300	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	650 g P,G	None	180 days
Americium 241/Curium 243 244	HASL 300 A-R-01	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days
Americium 243/Curium 245,246,247,248	HASL 300 A-R-01	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days
Isotopic Plutonium	HASL 300 A-R-01	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days
Isotopic Thorium	HASL 300 A-R-01	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days
Isotopic Uranium	HASL 300 A-R-01, DOE U-02	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days
Lead 210	EERF PB-01	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days
Nickel-59/63		Soil	5 g P,G	None	180 days
		Water	500 mL P,G	None	180 days
Polonium 210	HASL 300 PO-01	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days
Radium 226 and 228	903.0 / 904.0	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days
Radium 226	903.0	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days
Radium 228	904.0	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days

Total Alpha Radium	903.0	Water	1 L P,G	HNO3 (pH <2)	180 days
	HASL 300	Soil	5 g P,G	None	180 days
Strontium 89 / 90	DOE Sr-02	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days
Strontium 90	DOE Sr-02	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days
Strontium 89	DOE Sr-02	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	5 g P,G	None	180 days
Technetium 99	HASL 300 TC-02	Water	1 L P,G	HNO3 (pH <2)	180 days
		Soil	10 g P,G	None	180 days
Total Uranium	ASTM 5174-91	Water	5 mL P,G	HNO3 (pH <2)	180 days
		Soil	1 g P,G	None	180 days
Tritium	906.0 (distilled)	Water	120 mL G	None	180 days
		Soil	100 g G	None	180 days

- * Sample volumes are based on dry weights, volumes need to be increased if soil is wet/moist.
For samples requiring Matrix QC, 3 times the volume is required.
For normal samples, 2 or more times the volume may be required for re-extracts/digestions.
- ** Gross Alpha MDA is achievable only when solids are less than 500 ppm.

This table should be used for guidance only. If there is **any** doubt or question, please consult the appropriate group leader. Some analyses may be able to be combined, such as the anions, with no additional volume required. Check with group leader if there are changes in the method or the manner in which the lab performs an analysis.

ATTACHMENT C

Other Documents and Forms

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CERTIFICATE OF CALIBRATION

Standard Radionuclide Source

72745-762

500 mL High Density Solid in 538G GA-MA Beaker

This standard radionuclide source was prepared using aliquots measured gravimetrically from master radionuclide solution sources. The Am-241 was calibrated by 4 pi alpha liquid scintillation counting. All other radionuclides were calibrated using a germanium gamma spectrometer system. Calibration and purity were checked using a germanium gamma spectrometer system. At the time of calibration no interfering gamma-ray emitting impurities were detected. The gamma-ray emission rates for the most intense gamma-ray lines are given. Analytix maintains traceability to the National Institute of Standards and Technology through a Measurements Assurance Program as described in USNRC Regulatory Guide 4.15, Rev. 1, February, 1979.

Calibration date: April 1, 2006 12:00 EST

ISOTOPE	GAMMA-RAY ENERGY	HALF-LIFE	GAMMA-RAYS PER SECOND	TOTAL UNCERTAINTY %
Am-241	59.5	432 y	1031	3.0
Cd-109	88	462.6 d	1445	3.3
Co-57	122	271.79 d	751.5	3.0
Ce-139	166	137.6 d	1057	2.8
Hg-203	279	46.61 d	2320	2.7
Sn-113	392	115.1 d	1579	2.6
Cs-137	662	30.07 y	936.9	3.0
Y-88	898	106.6 d	3760	2.6
Co-60	1173	5.2714 y	1791	2.7
Co-60	1332	5.2714 y	1809	2.6
Y-88	1836	106.6 d	3912	2.6

815 gram solid. Density 1.6 g/cc.

P O NUMBER SNIDER 2/22/06, Item 2

SOURCE PREPARED BY:

M. Taskaeva
M. Taskaeva, Radiochemist

Q A APPROVED:

M. May 4-25-06

This standard will expire one year after the calibration date.

USACE Candidate Environmental Laboratory
Self-Declaration Form

STL St. Louis
13715 Rider Trail North
Earth City, MO 63045

Tel: 314 298 8566 Fax: 314 298 8757
www.stl-inc.com

Legal name of laboratory:	Severn Trent Laboratories, Inc. (STL St. Louis)
Street address:	13715 Rider Trail North Earth City, MO 63045
Name of Owner:	Severn Trent Laboratories, Inc.
Owner address (if different):	The Founders Building, Suite 300 580 Virginia Drive Ft. Washington, PA 19034-2707
Phone number:	215-646-9201
E-Mail address:	
Web site:	www.stl-inc.com
Laboratory director:	William R. Deckelmann
Phone number:	314-298-8566 ext 223
E-Mail address:	bdeckelmann@stl-inc.com
Quality Assurance Officer:	Elaine Wild
Phone number:	314-298-8566 ext 225
E-Mail address:	ewild@stl-inc.com

The undersigned persons understand and acknowledge that:

- a. Laboratory operations, which will be utilized for testing in support of environmental analytical testing for USACE, are in full compliance with the most recent version of the DOD Quality Systems Manual (including NELAC Standard Chapter 5 and Appendix requirements). All written documentation provided to USACE, accompanying this declaration, accurately reflect policy and practices implemented by laboratory staff.
- b. The Laboratory will notify USACE immediately of change in status of laboratory operations that may affect on-going compliance as declared per item a.
- c. The Laboratory acknowledges that USACE may audit the laboratory, relative to policy compliance at any time deemed appropriate; and will allow a designated COR fill access to information and facilities to conduct such audit operations.
- d. Signatories are authorized to sign this form on behalf of the owner and that there are no misrepresentations in the information provided in the initial laboratory assessment package.

USACE Candidate Environmental Laboratory Self-Declaration Form

STL St. Louis
13715 Rider Trail North
Earth City, MO 63045

Tel: 314 298 8566 Fax: 314 298 8757
www.stl-inc.com

Signature of Quality Assurance Officer:	Elaine Wild
Date:	July 15, 2005
Signature of Laboratory Director:	Kella Dahl
Date:	July 15, 2005

Note: Minimally, the laboratory evaluator should receive the completed declaration form with the following material (to verify compliance with the QSM):

- A copy of the laboratory's most current Quality Assurance Manual (e.g., the laboratory's ethics program policies and quality system procedures), and select QA SOPs (minimally, the laboratory's SOPs for MDL studies and LCS control chart limits).
- The determinative and preparatory method SOP for the parameters for which testing will be performed.
- Method performance demonstration for the parameters of interest—minimally, MDL studies and LCS control ranges for the preparatory-determinative method combinations and PT sample results (from a NELAP accredited PT provider) for the parameters of interest (e.g., or NELAP accreditation for these parameters, when NELAP accreditation is offered for the parameter of interest).

STL St. Louis provides analytical services to a number of USACE sites which may be within a single district's authority. Our apologies if you have received this Self-Declaration form multiple times. STL St. Louis will provide these documents, along with any other desired documentation, to the laboratory evaluator and/or the COR. To eliminate redundancy in individuals receiving this information from us, we will submit these upon request.