TETRA TECH

May 27, 2014

Mr. James Johnson
On-Scene Coordinator
U.S. Environmental Protection Agency, Region 7
11201 Renner Boulevard
Lenexa, Kansas 66219

Subject: Quality Assurance Project Plan for Baseline Off-Site Air Monitoring and Sampling
West Lake Landfill Site, Bridgeton, Missouri
CERCLIS ID: MOD079900932
EPA Region 7, START 4, Contract No. EP-S7-13-06, Task Order No. 0058
Task Monitor: James Johnson, On-Scene Coordinator

Dear Mr. Johnson:

Tetra Tech, Inc. is submitting the attached Quality Assurance Project Plan regarding air monitoring and sampling at locations off-site of the West Lake Landfill Site (WLLS) in Bridgeton, Missouri. This monitoring will be conducted during a baseline period prior to start of construction of an isolation barrier at the WLLS. If you have any questions or comments, please contact me at (816) 412-1775.

Sincerely,

for Dave Kinroth
START Project Manager

for Ted Faile, PG, CHMM
START Program Manager

Enclosures

X9025.14.0058.000

415 Oak Street, Kansas City, MO 64106
Tel 816.412.1741 Fax 816.410.1748 www.tetratech.com

QUALITY ASSURANCE PROJECT PLAN FOR BASELINE OFF-SITE AIR MONITORING

WEST LAKE LANDFILL SITE

Superfund Technical Assessment and Response Team (START) 4
Contract No. EP-S7-13-06, Task Order No. 0058

Prepared For:

U.S. Environmental Protection Agency
Region 7
Superfund Division
11201 Renner Blvd.
Lenexa, Kansas 66219

May 27, 2014

Prepared By:

Tetra Tech, Inc.
415 Oak Street
Kansas City, Missouri 64106
(816) 412-1741

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Addendum for the Generic QAPP for Superfund Site Assessment and Targeted Brownfields Assessment Activities (October 2012) for the West Lake Landfill Site

Project Information:			
Site Name: West Lake Landfill Site		**City:** Bridgeton	**State:** Missouri
EPA Project Manager: James Johnson		START Project Manager: Dave Kinroth	
Approved By:	for Dave Kinroth	**Prepared For:** EPA Region 7 Superfund Division	
Title: START Project Manager	Date: 05/27/14		
Approved By:			
Title: START Program Manager	Date: 5/27/14		
Approved By:	Cathy Nomes	**Prepared By:** Rob Monnig **Date:** May 2014	
Title: START QA Manager	Date: 5/27/14		
Approved By:			
Title: EPA Project Manager	Date:	Tetra Tech START Project Number: X9025.14.0058.000	
Approved By:			
Title: EPA Region 7 QA Manager	Date:		

1.0 Project Management:

1.1 Distribution List

EPA—Region 7: James Johnson, EPA On-Scene Coordinator
Diane Harris, Region 7 QA Manager

START: Dave Kinroth, Project Manager

1.2 Project/Task Organization

James Johnson, of the EPA Region 7 Superfund Division, will serve as the EPA Project Manager for the activities described in this QAPP. Dave Kinroth, of Seagull Environmental Technologies, Inc. (SETI), will serve as the START Project Manager.

1.3 Problem Definition/Background:

Description: This site-specific Quality Assurance Project Plan (QAPP) form is prepared as an addendum to the Generic QAPP for Superfund Site Assessment and Targeted Brownfields Assessment Programs (updated October 2012), and contains site-specific data quality objectives for the sampling activities described herein.

☒ Description attached.

☐ Description in referenced report: _____ Title _____ Date

1.4 Project/Task Description:

☐ CERCLA PA
☐ Other (description attached):
☐ CERCLA SI
☐ Pre-CERCLIS Site Screening
☐ Brownfields Assessment
☒ Removal Assessment (baseline sampling)

Schedule: Baseline sampling is anticipated from June 2014 through the start of isolation barrier construction. Pre-sampling activities occurred in April and May 2014.

☐ Description in referenced report: _____ Title _____ Date

1.5 Quality Objectives and Criteria for Measurement Data:

Accuracy:	☒ Identified in attached table.
Precision:	☒ Identified in attached table.
Representativeness:	☒ Identified in attached table.
Completeness*:	☒ Identified in attached table.
Comparability:	☒ Identified in attached table.

Other Description:

*A completeness goal of 100 percent has been established for this project. However, if the completeness goal is not met, EPA may still be able to make site decisions based on any or all of the remaining validated data.

1.6 Special Training/Certification Requirements:

☒ OSHA 1910
☐ Special Equipment/Instrument Operator:
☐ Other (describe below):

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1.7 Documentation and Records:

☒ Field Sheets ☒ Site Log ☐ Trip Report ☒ Site Maps ☐ Video
☒ Chain of Custody ☒ Health and Safety Plan ☒ Letter Report ☒ Photos

☒ Sample documentation will follow EPA Region 7 SOP 2420.05.

☒ Other: Analytical information will be handled according to procedures identified in Table 2.

2.0 Measurement and Data Acquisition:

2.1 Sampling Process Design:

☐ Random Sampling ☐ Transect Sampling ☒ Biased/Judgmental Sampling ☐ Stratified Random Sampling
☐ Search Sampling ☐ Systematic Grid ☐ Systematic Random Sampling ☒ Definitive Sampling
☐ Screening w/o Definitive Confirmation ☒ Screening w/ Definitive Confirmation
☒ Sample Map Attached

The proposed sampling scheme will be biased/judgmental with definitive laboratory analysis, in accordance with procedures included in the *Guidance for Performing Site Inspections Under CERCLA*, Office of Solid Waste and Emergency Response (OSWER) Directive #9345.1-05, September 1992, and *Removal Program Representative Sampling Guidance, Volume 1: Soil*, OSWER Directive 9360.4-10, November 1991. Samples will be submitted for analysis by a START-contracted laboratory. See Appendices A and B for additional site-specific information.

Sample Summary Location	Matrix	# of Samples*	Analysis
Off-site Air Monitoring Stations (see Appendix B, Figure 1)	Air	Continuous/weekly/monthly monitoring and sampling (see Tables 1A & 1B)	Radionuclides, radon, gamma exposure rate, carbon monoxide, sulfur dioxide, hydrogen sulfide, volatile organic compounds, and particulates

*NOTE: Quality Control (QC) samples are not included with these totals. See Table 1 for a complete sample summary.

2.2 Sample Methods Requirements:

Matrix	Sampling Method	EPA SOP(s)/Methods
Air	various (see Tables 1A & 1B)	various (see Tables 1A & 1B)

2.3 Sample Handling and Custody Requirements:

☐ Samples will be packaged and preserved in accordance with procedures defined in Region 7 EPA SOP 2420.06.
☒ COC will be maintained as directed by Region 7 EPA SOP 2420.04.
☐ Samples will be accepted according to Region 7 EPA SOP 2420.01.

☒ Other (Describe): Samples will be packaged and accepted according to procedures established by the START-contracted laboratory.

2.4 Analytical Methods Requirements:

☒ Identified in attached table.
☒ Rationale: The requested analyses have been selected based on the historical information on the site and program experience with similar types of sites.
☐ Other (Describe):

2.5 Quality Control Requirements:

☐ Not Applicable
☒ Identified in attached table.
☒ In accordance with the Generic QAPP for Superfund Site Assessment and Targeted Brownfields Assessment Programs (updated October 2012).
☒ Describe Field QC Samples: Field blanks, trip blanks, and field duplicate samples are specified in Tables 1A and 1B. Trip blanks will be used to evaluate contamination introduced during transportation of the containers/samples. Field blanks will be collected to evaluate contamination of sampling containers and to assess contamination potentially introduced during the sampling and laboratory procedure(s). Evaluation of blank samples depends on the levels of contamination found in environmental samples to determine whether the environmental samples are representative. Analytical results from blank samples will be evaluated qualitatively by the EPA Project Manager and EPA contractor(s) to determine a general indication of field-introduced and/or lab-introduced contamination. Field duplicate samples will be collected to evaluate sampling and laboratory precision.

☐ Other (Describe):

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2.6 Instrument/Equipment Testing, Inspection, and Maintenance Requirements:

☐ Not Applicable
☒ In accordance with the Generic QAPP for Superfund Site Assessment and Targeted Brownfields Assessment Programs (updated October 2012).
☒ Other (Describe): Testing, inspection, and maintenance of instrumentation will accord with the SOPs and/or manufacturers' recommendations referenced in Tables 1A and 1B.

2.7 Instrument Calibration and Frequency:

☐ Not Applicable
☒ Inspection/acceptance requirements accord with the Generic QAPP for Superfund Site Assessment and Targeted Brownfields Assessment Programs (updated October 2012).
☒ Calibration of laboratory equipment will be performed as described in the SOPs and/or manufacturers' recommendations referenced in Tables 1A and 1B.
☒ Other (Describe): Calibration of field instruments will be performed as described in the SOPs and equipment operating guides referenced in Tables 1A and 1B.

2.8 Inspection/Acceptance Requirements for Supplies and Consumables:

☐ Not Applicable
☒ In accordance with the Generic QAPP for Superfund Site Assessment and Targeted Brownfields Assessment Programs (updated October 2012).
☐ All sample containers will meet EPA criteria for cleaning procedures for low-level chemical analysis. Sample containers will have Level II certifications provided by the manufacturer in accordance with pre-cleaning criteria established by EPA in *Specifications and Guidelines for Obtaining Contaminant-Free Containers.*
☒ Other (Describe): Air filter media will meet criteria in *Code of Federal Regulations* (CFR) Title 21, Part 177.2260. Summa canisters for VOC analysis will be certified clean by the START-contracted laboratory per the laboratory's SOP referenced in Table 1B.

2.9 Data Acquisition Requirements:

☐ Not Applicable
☒ In accordance with the Generic QAPP for Superfund Site Assessment and Targeted Brownfields Assessment Programs (updated October 2012).
☐ Previous data/information pertaining to the site (including other analytical data, reports, photos, maps, etc., which are referenced in this QAPP) have been compiled by EPA and/or its contractor(s) from other sources. Some of that data has not been verified by EPA and/or its contractor(s); however, the information will not be used for decision-making purposes by EPA without verification by an independent professional qualified to verify such data/information.
☐ Other (Describe):

2.10 Data Management:

☐ All laboratory data acquired will be managed in accordance with Region 7 EPA SOP 2410.01.
☒ Other (Describe): All laboratory data acquired will be managed according to procedures established by the START-contracted laboratory.

3.0 Assessment and Oversight:

3.1 Assessment and Response Actions:

☒ Peer Review ☒ Management Review ☐ Field Audit ☐ Lab Audit

☐ Assessment and response actions pertaining to analytical phases of the project are addressed in Region 7 EPA SOPs 2430.06 and 2430.12.

☐ Other (Describe):

3.1A Corrective Action:

☒ Corrective actions will be taken at the discretion of the EPA Project Manager whenever there appear to be problems that could adversely affect data quality and/or resulting decisions affecting future response actions pertaining to the site.

☐ Other (Describe):

3.2 Reports to Management:

☐ Audit Report ☒ Data Validation Report ☐ Project Status Report ☐ None Required

☒ A letter report describing the sampling techniques, locations, problems encountered (with resolutions to those problems), and interpretation of analytical results will be prepared by Tetra Tech START and submitted to the EPA.
☒ Reports will be prepared in accordance with the Generic QAPP for Superfund Site Assessment and Targeted Brownfields Assessment Programs (updated October 2012).
☐ Other (Describe):

4.0 Data Validation and Usability:

4.1 Data Review, Validation, and Verification Requirements:

☐ Identified in attached table:
☒ Data review and verification will accord with the Generic QAPP for Superfund Site Assessment and Targeted Brownfields Assessment Programs (updated October 2012).
☐ Data review and verification will be performed by a qualified analyst and the laboratory's section manager as described in Region 7 EPA SOPs 2430.06, 2410.10, and 2430.12.
☒ Other (Describe): The analytical data package from the START-contracted laboratory will be validated internally by the contracted laboratory in accordance with the laboratory's established SOPs. A START chemist will conduct an external verification and validation of the laboratory data package.

4.2 Validation and Verification Methods:

☐ Identified in attached table:
☐ The data will be validated in accordance with Region 7 EPA SOPs 2430.06, 2410.10, and 2430.12.
☒ Other (Describe): The data will be validated using methods consistent with a Stage 2B validation, as described in the EPA Contract Laboratory Program (CLP) *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use* (EPA 2009). A Stage 2B validation includes verification and validation based on a completeness and compliance check of sample receipt conditions and sample-related and instrument-related QC results. The EPA Project Manager will be responsible for overall validation and final approval of the data, in accordance with the projected use of the results.

4.3 Reconciliation with User Requirements:

☐ Identified in attached table:
☒ If data quality indicators do not meet the project's requirements as outlined in this QAPP, the data may be discarded and re-sampling or re-analysis of the subject samples may be required by the EPA Project Manager.
☐ Other (Describe):

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Table 1A: Sample Summary – Radiological Parameters

Site Name: West Lake Landfill Site | **Location:** Bridgeton, Missouri
START Project Manager: Dave Kinroth | **Activity/ASR #:** NA | **Date:** May 2014

No. of Samples	Matrix	Location	Purpose	Requested Analysis	Sampling Method	Analytical Method/SOP
Samples Submitted for Laboratory Analysis						
1 sample per station per week	Radionuclides in airborne particulates	5 off-site monitoring stations	Assess concentrations of radionuclides present on airborne particulates	Isotopic Th (including Th-230)	EPA NCRFO SOP RPR-250: Operation of Air Samplers without Flow Measurement Capability	Alpha spec. per lab SOP[1]
				Total alpha-emitting Ra		EPA 903.0 & SW-846 9315 as modified by lab SOP[1]
				Isotopic U		Alpha spec. per lab SOP[1]
				Gross alpha/beta		Low background GFPC per lab SOP[1]
				Gamma spectroscopy		Gamma spec. per lab SOP[1]
				Ra-226[2]		EPA 903.0 & SW-846 9315 as modified by lab SOP[1] preceded by 21-day in-growth of Ra-226 progeny
3 badges per station, submitted monthly	Gamma exposure rate by environmental TLD	5 off-site monitoring stations	Assess gamma exposure rates	Gamma exposure rate	Per vendor-provided instructions and Service Guide[4]	NRC Regulatory Guide 4.13
Field Measurements						
3 E-Perms per station, read weekly	Radon in ambient air	5 off-site monitoring stations	Assess concentrations of radon in air	Radon	EPA Region 7 E-PERM Radon Detection System Equipment Guide	NA *(field measurement only)*
1 continuous instrument per station	Gamma exposure rate by Saphymo GammaTRACER (G-M tube)	5 off-site monitoring stations	Assess gamma exposure rates	Gamma exposure rate	Per EPA ERT procedures	NA *(field measurement only)*
1 continuous sensor per station	Gamma exposure rate by RAE Systems AreaRAE sensor	5 off-site monitoring stations	Assess gamma exposure rates	Gamma exposure rate	EPA Region 7 AreaRAE EOG	NA *(field measurement only)*
QC Samples/Measurements						
1 per weekly field blank submittal	Radionuclides on filter media	Field blank	Assess contamination of the filter from field handling	Same as the requested analyses for filter samples	Filter will be handled in the field[2]	Same as the analyses for filter samples
1 each per batch of TLDs	Gamma exposure rate by environmental TLD	Transit/control badge	Assess contributions to gamma exposure rates related to background and badge transit	Gamma exposure rate	Per vendor-provided instructions and Service Guide[4]	NRC Regulatory Guide 4.13
2 replicates per station[5]	Radon in ambient air	5 off-site monitoring stations	Assess total method precision	Radon	EPA Region 7 E-PERM EOG	NA *(field measurement only)*
2 replicates[5]	Gamma exposure rate by environmental TLD	5 off-site monitoring stations	Assess total method precision	Gamma exposure rate	Per vendor-provided instructions and Service Guide	NRC Regulatory Guide 4.13

Alpha spec. = alpha spectroscopy; EPA = U.S. Environmental Protection Agency; EOG = Equipment Operating Guide; ERT = Environmental Response Team; gamma spec. = gamma spectroscopy; GFPC = gas flow proportional counting; G-M = Geiger-Mueller; NA = not applicable; lab = laboratory; NCRFO = National Center for Radiation Field Operations; NRC = U.S. Nuclear Regulatory Commission; Ra = radium; SOP = Standard Operating Procedure; TLD = thermoluminescence dosimeters; Th = thorium; U = uranium

Notes:
[1] See Appendix C
[2] Analyzed only if total alpha-emitting radium is greater than 5 picoCuries/filter
[3] A filter will be handled in the field in a manner similar to that for the primary filter samples, except no sampling onto the filter will occur
[4] See http://www.landauer.com/uploadedFiles/About_Us/LDR%20Service%20Guide%20-%20012013.pdf
[5] These replicates are included in the per station number of samples

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Table 1B: Sample Summary – Chemical and Particulate Parameters						
Site Name: West Lake Landfill Site				**Location:** Bridgeton, Missouri		
START Project Manager: Dave Kinroth				**Activity/ASR #:** NA	**Date:** May 2014	
No. of Samples	**Matrix**	**Location**	**Purpose**	**Requested Analysis**	**Sampling Method**	**Analytical Method/SOP**
Samples Submitted for Laboratory Analysis						
1 sample per station per week	Outdoor air	5 off-site monitoring stations	Assess VOCs	VOCs	EPA ERT SOP 4231.1704 and EPA Region 7 SOP 2313.04	EPA Method T0-15 and lab SOP[1]
Field Measurements						
1 continuous sensor per station	Outdoor air	5 off-site monitoring stations	Assess for typical landfill gasses of concern	CO, SO_2, H_2S, VOCs[2]	EPA Region 7 AreaRAE EOG	NA *(field measurement only)*
1 continuous sensor per station	Outdoor air	5 off-site monitoring stations	Assess for airborne particulate concentrations	$PM_{2.5}$	EPA Region 7 DataRAM EOG	NA *(field measurement only)*
QC Samples						
1 per week	Outdoor air	Trip blank	Assess contamination of the Summa canister from field handling	VOCs	Trip blank will be handled in the field[3]	EPA Method T0-15 and lab SOP[1]
1 per week	Outdoor air	Field duplicate	Assess total method precision	VOCs	Field duplicate will be co-located with a primary Summa canister and will be sampled concurrent with the primary Summa canister	EPA Method T0-15 and lab SOP[1]

CO = carbon monoxide; EPA = U.S. Environmental Protection Agency; EOG = Equipment Operating Guide; ERT = Environmental Response Team; H_2S = hydrogen sulfide; NA = not applicable; lab = laboratory; $PM_{2.5}$ = particulates less than 2.5 micrometers in diameter; SOP = Standard Operating Procedure; VOC = volatile organic compound

Notes:
[1] See Appendix C
[2] Measures VOCs as a relative instrument response to a 10.6 electron volt lamp calibrated to isobutylene
[3] A Summa canister will be handled in the field in a manner similar to that for the sampled Summa canisters, except no sampling with the trip blank canister will occur

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Table 2: Data Quality Objective Summary								
Site Name: West Lake Landfill Site				**Location:** Bridgeton, Missouri				
START Project Manager: Dave Kinroth				**Activity/ASR #:** N/A (START-contracted laboratory)			**Date:** May 2014	
Analysis	**Analytical Method**	**Data Quality Measurements**					**Sample Handling Procedures**	**Data Management Procedures**
		Accuracy	**Precision**	**Representativeness**	**Completeness**	**Comparability**		
Radionuclides in airborne particulates collected on filters	see Table 1A	per analytical method	per analytical method	Biased/judgmental sampling based on professional judgment of the sampling team	The completeness goal is 100%; however, no individual samples have been identified as critical samples.	Standardized procedures for sample collection and analysis will be used.	See Section 2.3 of QAPP form.	See Section 2.10 of QAPP form.
Gamma exposure rate by environmental TLD	see Table 1A	per analytical method	per analytical method	Biased/judgmental sampling based on professional judgment of the sampling team	The completeness goal is 100%; however, no individual samples have been identified as critical samples.	Standardized procedures for sample collection and analysis will be used.	See Section 2.3 of QAPP form.	See Section 2.10 of QAPP form.
VOCs by EPA Method TO-15	see Table 1B	per analytical method	per analytical method	Biased/judgmental sampling based on professional judgment of the sampling team	The completeness goal is 100%; however, no individual samples have been identified as critical samples.	Standardized procedures for sample collection and analysis will be used.	See Section 2.3 of QAPP form.	See Section 2.10 of QAPP form.

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APPENDIX A

SITE-SPECIFIC INFORMATION REGARDING BASELINE OFF-SITE AIR MONITORING AND SAMPLING AT LOCATIONS AROUND THE WEST LAKE LANDFILL SITE IN BRIDGETON, MISSOURI

INTRODUCTION

The Tetra Tech, Inc. (Tetra Tech) Superfund Technical Assessment and Response Team (START) has been tasked by the U.S. Environmental Protection Agency (EPA) to assist with baseline monitoring at off-site locations around the West Lake Landfill site (WLLS) in Bridgeton, Missouri. Dave Kinroth of Seagull Environmental Technologies, Inc. (SETI) will serve as the START Project Manager. He will be responsible for ensuring that air monitoring and sampling proceeds as described in this Quality Assurance Project Plan (QAPP), and for providing periodic updates to the client concerning the status of the project, as needed. James Johnson will be the EPA Project Manager for this activity.

START's tasks will include, but are not limited to: (1) assembling and maintaining a network of off-site air monitoring stations with instrumentation and sampling devices to measure radiological and chemical parameters of potential concern, (2) collecting samples and coordinating laboratory analysis, (3) assisting EPA with data acquisition and management, and (4) documenting the off-site air monitoring efforts. The Tetra Tech START quality assurance (QA) manager will provide technical assistance, as needed, to ensure that necessary QA issues are adequately addressed.

START will adhere to this QAPP as much as possible, but may alter proposed activities in the field if warranted by site-specific conditions and unforeseen hindrances that prevent implementation of any aspect of this QAPP in a feasible manner. Such deviations will be recorded in the site logbook, as necessary. This QAPP will be available to the field team at all times during sampling activities to serve as a key reference for the proposed activities described herein.

PROBLEM DEFINITION, BACKGROUND, AND SITE DESCRIPTION

This QAPP was prepared by Tetra Tech START to support the off-site air monitoring program during a baseline monitoring period prior to initiation of construction of a planned isolation barrier at WLLS. Air monitoring will be conducted during the baseline period to provide data that will be used to (1) evaluate pre-construction concentrations of chemical and radiological parameters of potential concern in outdoor air, and (2) provide data that will be used to optimize the sampling and monitoring plan for the off-site air monitoring to occur during construction of the isolation barrier.

West Lake Landfill is an approximately 200-acre property that includes several closed solid waste landfill units that accepted wastes for landfilling from the 1940s or 1950s through 2004, plus a solid waste transfer station, a concrete plant, and an asphalt batch plant. The WLLS is at 13570 St. Charles Rock Road in Bridgeton, St. Louis County, Missouri, approximately 1 mile north of the intersection of

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Interstate 70 and Interstate 270 (see Appendix B, Figure 1). The WLLS was used for limestone quarrying and crushing operations from 1939 through 1988. Beginning in the late 1940s or early 1950s, portions of the quarried areas and adjacent areas were used for landfilling municipal refuse, industrial solid wastes, and construction/demolition debris. In 1973, approximately 8,700 tons of leached barium sulfate residues (a remnant from the Manhattan Engineer District/Atomic Energy Commission project) were reportedly mixed with approximately 39,000 tons of soil from the 9200 Latty Avenue site in Hazelwood, Missouri, transported to the WLLS, and used as daily or intermediate cover material. In December 2004, the Bridgeton Sanitary Landfill—the last landfill unit to receive solid waste—stopped receiving waste pursuant to an agreement with the City of St. Louis to reduce potential for birds to interfere with Lambert Field International Airport operations. In December 2010, Bridgeton Landfill detected changes—elevated temperatures and elevated carbon monoxide levels—in its landfill gas extraction system in use at the South Quarry of the Bridgeton Sanitary Landfill portion of the Site (a landfill portion not associated with known radiologically-impacted materials (RIM)). Further investigation indicated that the South Quarry Pit landfill was undergoing an exothermic subsurface smoldering event (SSE). In 2013, potentially responsible parties committed to constructing an isolation barrier that would separate the Bridgeton Landfill undergoing the SSE from the RIM-containing WLLS (EPA 2014).

Before construction of the isolation barrier and during construction activities, START will assist EPA with air monitoring at locations off-site of the WLLS to characterize current ambient air conditions. Monitoring will be conducted for radiological parameters (including alpha-, beta-, and gamma-emitting radionuclides on particulates; radon; and external gamma exposure), as well as typical solid waste landfill gases (including sulfur dioxide [SO_2], hydrogen sulfide [H_2S], carbon monoxide [CO], and volatile organic compounds [VOC]) and particulate matter.

EPA has arranged for placement of the air monitoring stations at the following locations (see Appendix B, Figure 1):

Station 1 – Robertson Fire Protection District Station 2, 3820 Taussig Rd., Bridgeton, Missouri

Station 2 – Pattonville Fire Department District, 13900 St Charles Rock Rd., Bridgeton, Missouri

Station 3 – Pattonville Fire Department District Station 2, 3365 McKelvey Rd., Bridgeton, Missouri

Station 4 – Spanish Village Park, 12827 Spanish Village Dr., Bridgeton, Missouri

Station 5 – St. Charles Fire Department Station #2, 1550 S. Main St., St. Charles, Missouri.

These locations were selected to ensure coverage around the perimeter of the WLLS and are placed in areas near residential populations.

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SAMPLING STRATEGY AND METHODOLOGY

EPA and START began initial evaluation of the five off-site monitoring stations in April 2014; these activities included installation of electrical service, instrument weather housings, monitoring and sampling devices (including particulate air samplers, RAE Systems AreaRAEs, Saphymo GammaTRACERs, E-Perm radon detectors, and thermoluminescence dosimeters), and a continuous remote monitoring network. The baseline sampling period is anticipated to begin in early June 2014, and will end prior to initiation of the isolation barrier construction, when a second phase of air monitoring and sampling will begin.

Baseline period off-site air monitoring and sampling will proceed according to the following sampling process design, including selection of parameters of interest and associated sampling procedures:

Parameters of Interest

The following radiological and chemical parameters of potential concern were identified based on historical information regarding the site and program experience with similar types of sites:

Radiological Parameters of Potential Concern

Presence of naturally occurring alpha-, beta-, and gamma-emitting radionuclides on airborne particulates will be assessed. The radionuclides of potential concern based on the characteristics of the West Lake RIM that will be assessed are thorium-230, radium-226, and radon. Gross gamma activity at each of the monitoring stations will also be assessed.

Chemical Parameters of Potential Concern

Chemical parameters of potential concern selected for assessment include CO, H_2S, SO_2, and VOCs.

Airborne Particulate Matter

Because the isolation barrier construction activities could release airborne particulate matter, $PM_{2.5}$ (particulates less than 2.5 micrometers in diameter) is also being assessed.

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Sampling Procedures

Samples will be collected in a manner consistent with EPA methods and standard operating procedures (SOP). The following are summaries of the project-specific sampling methods. Tables 1A and 1B summarize the sampling method requirements.

Radionuclides in Airborne Particulates

To determine airborne concentrations of radionuclides transported via airborne particulates, airborne particulates will be collected onto borosilicate glass fiber filter media using high-volume air samplers. One air sampler will be operated at each off-site monitoring station and will collect airborne particulates continuously onto the filter media for durations of 7 days. At the end of the sampling period, the sampled filter will be submitted for laboratory analysis, a new filter will be installed, and a new 7-day sampling period will begin. The air samplers will be operated at a flow rate of at least 2.0 cubic feet per minute to yield a minimum air sample volume of 20,160 cubic feet (571 cubic meters [m^3]). With an anticipated laboratory detection limit of 1 picoCurie (pCi) per filter for thorium-230 and radium-226, this sample volume corresponds to a detection limit, in terms of an air concentration, of 1.75E-2 pCi/m^3 for those radionuclides. Calibration and operation of the high-volume air samplers will accord with the EPA National Center for Radiation Field Operations (NCRFO) SOP RPR-250: *Operation of Air Samplers without Flow Measurement Capability.*

Radon

Electret ion chamber radon detectors (E-PERM®) equipped with a high-volume chamber ("H-chamber") short-term ("ST") electrets will be used to assess radon levels at each off-site monitoring station. E-PERM® measurements are performed by use of an Electret Voltage Reader to measure a beginning and final electrical charge on the electret that is exposed for a specified time period. The E-PERM® will be read weekly to yield a radon measurement that is continuously integrated (averaged) over the week-long exposure duration. Three E-PERMs® will be deployed per off-site monitoring station to provide redundant measurements in case of a device failure, and to provide an indication of total method precision.

Gross Gamma Activity

Continuous gross gamma activity at each off-site monitoring station will be assessed using Saphymo GammaTRACER and RAE Systems AreaRAE instruments. The continuous gross gamma measurements

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from these instruments will be remotely transmitted via Safe Environment Engineering's Lifeline remote telemetry system and logged by EPA's Viper data management software.

Thermoluminescence dosimeters (TLD) will also record gross gamma activity at the off-site monitoring stations. TLDs are passive detection devices that require analysis by the dosimeter provider. The TLDs will be deployed for continuous periods of approximately 30 days. Three TLDs will be deployed per off-site monitoring station to provide redundant measurements allowing determination of total method precision.

Continuous Monitoring for CO, H_2S, SO_2, and VOCs

RAE Systems AreaRAEs equipped with CO, H_2S, SO_2, and photo-ionization (for VOC detection) sensors will be deployed at each off-site air monitoring station for continuous air monitoring. These AreaRAE measurements will be remotely transmitted via Safe Environment Engineering's Lifeline remote telemetry system and logged by EPA's Viper data management software. Typical AreaRAE response parameters for the gases listed above are as follows:

Gaseous Parameter Measured	AreaRAE Detection Range	AreaRAE Resolution
Carbon Monoxide (CO)	0-500 PPM	$\pm$ 1 PPM
Hydrogen Sulfide (H_2S)	0-11 PPM	$\pm$ 1 PPM
Sulfur Dioxide (SO_2)	0-20 PPM	$\pm$ 0.1 PPM
Volatile Organic Compounds (VOC)	0-199 PPM	$\pm$ 0.1 PPM

Air Sampling for VOCs

Sampling for VOCs via Summa® canisters will occur each week at the air monitoring stations. The Summa® canister will be fitted with a passive flow regulator to enable collection of an air sample for a continuous 24-hour period. The sampled Summa canisters will be submitted to a START-contracted laboratory for VOC analysis. All Summa® sampling will accord with EPA Environmental Response Team SOP 4231.1704 – Summa® Canister Sampling, and with EPA Region 7 SOP 2313.04 – Air Sampling with Stainless Steel Canisters. During the weekly sampling, a field duplicate sample will be collected at one of the off-site air monitoring stations. In addition, an un-sampled Summa canister will be handled in the field and will be submitted as a trip blank.

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Airborne Particulate Matter

A DataRAM air particulate monitor will be deployed at each off-site air monitoring station to continuously monitor concentrations and median particle sizes. The DataRAM instruments will be equipped with particle discriminators to yield measurements correlated with $PM_{2.5}$. The continuous DataRAM measurements will be remotely transmitted via Safe Environment Engineering's Lifeline remote telemetry system and logged by EPA's Viper data management software.

Quality Control Samples

To evaluate sample quality control (QC), field blank, trip blank, and field duplicate samples will be collected, as specified in Section 2.5 of the QAPP form.

ANALYTICAL METHODS

All samples will be submitted to a START-contracted laboratory for analysis. All samples will be analyzed according to SOPs and methods referenced on the QAPP Form.

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REFERENCES

U.S. Environmental Protection Agency (EPA). 2014. Administrative Settlement Agreement and Order on A Consent For Removal Action – Preconstruction Work. EPA Docket No. CERCLA-07-2014-0002. April 20.

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APPENDIX B

FIGURE

Legend

- Proposed off-site air monitoring station
- Approximate site boundary
- Operational Unit 1 (radiological area)

N

0 1,000 2,000

Feet

Source: ArcGIS Online Aerial Imagery, 2013

West Lake Landfill
Bridgeton, Missouri

Figure 1
Proposed Off-Site
Air Monitoring Stations

TETRA TECH

Date: 5/23/2014 Drawn By: Bill Spiking Project No: X9025.14.0058.000

APPENDIX C

ANALYTICAL LABORATORY STANDARD OPERATING PROCEDURES

Title: DETERMINATION OF GROSS ALPHA/BETA ACTIVITY

Approvals (Signature/Date):

Sarah C. Bernsen 2·12·14	Michael Ridenhower 2/12/14
Sarah Bernsen Date	Michael Ridenhower Date
Radiochemistry Prep Supervisor	Health & Safety Manager / Coordinator
Marti Ward 2·11·14	Elaine Wild 2/13/14
Marti Ward Date	Elaine Wild Date
Quality Assurance Manager	Laboratory Director

This SOP was previously identified as SOP No. ST-RC-0020 Rev. 17

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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 in air filters, water, soil/sediment, oil and vegetation samples.

1.2 This SOP is based on EPA Method 900.0, SW-846 Method 9310 and DOE RP-710.

1.3 For water samples containing high concentrations of dissolved solids (> 500 ppm), see SOP ST-RC-0021 for analysis of gross alpha radioactivity.

1.4 The reporting limits, method detectable activities and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 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 tared counting planchet.

2.2 For the activity of dissolved matter, an aliquot of aqueous sample is filtered through a 0.45-µm 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.

2.3 For the activity of suspended matter, an aliquot of aqueous sample is filtered through a 0.45-µm membrane filter. The filter is transferred to a counting planchet.

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 ST-RC-0004, "Preparation of Soil, Sludge, Filter, Biota , Oil and Grease Samples for Actinide Analysis" and then an aliquot prepared like a liquid.

2.5 Solid samples can be analyzed for gross alpha and/or beta activity as a dry powder. If Method RP710 (for total dissolution) is required, an acid leach is performed per ST-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 also be done using Hydrofluoric acid, Hydrochloric acid and Nitric acid as in section 11.8.

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 planchet using 4 M nitric acid.

2.7 The sample residue is dried, and then counted for alpha and/or beta radioactivity using a Gas Flow Proportional Counter

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 There are no specific definitions for this procedure.

4.0 INTERFERENCES

4.1 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 solid concentration may adversely affect sensitivity of the method.

4.2 For a 2-inch diameter counting planchet (20 cm^2), 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 radionuclides may also be lost during the sample evaporation and drying (such as tritium and some chemical forms of radioiodine). Some radionuclides, 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. Hygroscopic sample matrices may 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.

4.6 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.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
None.

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 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.
Hydrofluoric Acid	Poison Corrosive Dehydrator	3 ppm-(TWA)	Severely corrosive to the respiratory tract. Corrosive to the skin and eyes. Permanent eye damage may occur. Skin contact causes serious skin burns, which may not be immediately apparent or painful. Symptoms may be delayed 8 hours or longer. **THE FLUORIDE ION READILY PENETRATES THE SKIN CAUSING DESTRUCTION OF DEEP TISSUE LAYERS AND BONE DAMAGE.**
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			
Ceiling – At no time should this exposure limit be exceeded			

6.0 EQUIPMENT AND SUPPLIES

6.1 Analytical Balance (4 - or 5 - place).

6.2 Beakers: Glass and Teflon, various sizes. Please consult SOP: ST-RC-5006 "Decontamination of Laboratory Glassware, Labware, and Equiptment."

6.3 Counting planchets, stainless steel, flat and ridged, 5.0 cm (2.0"), cleaned per ST-RC-0002, "Preparation of Stainless Steel Planchets for Radiochemistry Analyses."

6.4 Desiccator with desiccant, Dri-Rite or equivalent.

6.5 Drying oven with thermostat set at 105° C ± 5 °C.

6.6 Filter paper: ashless, Whatman #41 or ashless paper pulp, and 0.45-µm membrane.

6.7 Hot plate

6.8 Pipettes

6.9 Muffle oven

6.10 Mod block

6.11 Tongs or forceps

6.12 Double sided tape or Self-adhesive dots

6.13 Spatula

6.14 Aluminum weighing pans

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-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)

7.4.1 4 N Nitric acid (4N HNO_3) - Add 250 ml of 16 N HNO_3 to 750 ml of reagent water and mix well.

7.5 Hydrofluoric acid, concentrated (29 N HF)

7.6 Hydrochloric acid, concentrated (12 N HCl)

7.7 Salt Solution: $NaHCO_3$ – 22 g, KCL – 0.80 g, $MgCl_2 \cdot 6H_2O$ – 22 g, Na_2SO_4 – 34.2 g add to 500 mL of DI water. Stir on stir plate until dissolved. Bring final volume up to 1 L with DI.

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 Strontium-90 for LCS and matrix spikes, calibrated - NIST traceable, in equilibrium with Yttrium 90, diluted to approximately 20 dpm/ml.

7.11 Sodium Bicarbonate, $NaHCO_3$ powder.

7.12 Potassium Chloride, KCl

7.13 Sodium Sulfate, $NaSO_4$ crystals

7.14 Magnesium Chloride Hexahydrate, $MgCl_2 \cdot 6H_2O$, crystals

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica 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 ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2.

8.3.1 The pH of aqueous samples is checked upon receipt by the Sample Control Department. The pH does not require re-checking prior to analysis.

8.3.2 Aqueous samples acidified upon receipt (designated by label on the bottle) do require a check of the pH prior to analysis.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

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

9.1.3 For this analysis, batch QC consists of a method blank (MB), a 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.

9.1.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank (MB)**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Water analyses, the method blank is comprised of DI water. Prepare a method blank of DI water equivalent to the target volume of 200 mL.

9.2.4 For Soil analyses, the method blank is comprised of salt.

9.2.5 For Oil analyses, the method blank is comprised of shredded filter paper in a crucilble.

9.2.6 For non-digested filters, a prepared method blank is provided by the count room.

9.2.7 For leached analyses, the method blank is comprised of the leaching acid.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 A LCS is 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 sample batch.

9.3.3 For Water analyses, the LCS is comprised of DI water fortified with Strontium 90 for beta and Thorium 230 for alpha. Add 0.7 mL of salt solution for mass.

9.3.4 For Soil analyses, the LCS is comprised of a known solid reference material :National Bureau of Standards, SRM 4353, Rocky Flats Soil #1.

9.3.5 For Oil analyses, the LCS is comprised of shredded filter paper fortified with Strontium 90 for beta and Thorium 230 for alpha.

9.3.6 For non-digested filters, the LCS is provided by the count room.

9.4 **Matrix Spike (MS)/Matrix Spike Duplicate (MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate (SD)**

9.5.1 A Sample Duplicate is 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. A NCM is written to document the insufficient volume and utilizing of a LCSD for demonstration of precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

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

10.0 CALIBRATION AND STANDARDIZATION

10.1 Balance calibration must be checked daily when used. Refer to SOP ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes Procedure.

10.2 For analytical instrumentation calibration, see SOP: ST-RD-0403, "Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System".

11.0 PROCEDURE

11.1 If the activity of dissolved matter in an aliquot of aqueous sample is to be determined.

11.1.1 Filter the desired aliquot through a 0.45-µm membrane filter and proceed with aqueous sample preparation.

11.2 If the activity of suspended matter of an aliquot of aqueous sample is to be determined.

11.2.1 Filter the desired aliquot through a 0.45-µm membrane filter, and proceed with filter sample preparation.

11.3 Aqueous Sample - Total Solid Screen

11.3.1 Record sample preparation data in Gross Alpha/Beta (GAB) Solid Screen Excel program (RAD-0052). Weigh the empty beaker and record weight (under the tare weight header)

11.3.2 Shake the sample container thoroughly.

11.3.2.1 If alpha and beta are to be determined simultaneously from a single aliquot, the lowest net residue weight limit applies.

11.3.3 Measure a 20 mL aliquot into a pre-weighed beaker.

11.3.4 Add 10 mL of concentrated Nitric acid.

11.3.5 Evaporate to dryness using a hot plate, do not allow the sample to splatter.

11.3.6 Remove from heat and allow to cool to room temperature.

11.3.7 Add 10 mL concentrated Nitric acid.

11.3.8 Evaporate to dryness using a hot plate. Do not allow the sample to splatter.

11.3.9 Remove from heat and allow to cool in desicator for a minimum of 30 minutes.

11.3.10 Reweigh the beaker in GAB Solid Screen Excel (RAD-0052) program and record weight (under the gross weight header)

11.3.10.1 By estimating the solid content of the sample, the program will provide the target aliquot .

11.3.10.2 If GAB Solid Screen Excel program is not available use the formula found in 12.2.

11.3.10.3 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 12.2, with a target weight of 80 mg alpha/beta dried residue on the planchet **(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. Dilutions 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.)**. If only Gross Beta is being performed, the target weight is 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.

11.4 Aqueous Sample Gross Alpha/Beta

11.4.1 Initiate sample preparation worksheet.

11.4.2 Shake the sample container thoroughly.

11.4.3 Measure a volume of sample, previously determined in section 11.3, into an appropriately sized beaker. Record volume of sample used.

11.4.3.1 If it is determined (in step 11.3) that only a small volume of sample is required, additional volume may be added in small aliquots directly to the beaker used to determine the volume needed to achieve the target sample weight.

11.4.4 Prepare a method blank, LCS and MS.

11.4.5 Add 10 mL of concentrated Nitric acid to all samples and QC.

11.4.6 Evaporate to near dryness using a hot plate. Do not allow the sample to splatter.

11.4.7 Remove from heat and allow to cool to room temperature.

11.4.8 Add 10 mL concentrated Nitric acid.

11.4.9 Evaporate to near dryness using a hot plate. Do not allow the sample to splatter.
NOTE: Some samples with difficult matrices may require steps 11.4.7 through 11.4.9 to be repeated until the sample residue does not change in appearance.

11.4.10 Remove from heat and allow to cool to room temperature.

11.4.11 Add 10 mL of 4 N nitric acid to wash down the sides of the beaker.

11.4.12 Heat on hot plate to dissolve sample residue and reduce volume to approximately 5-7 mL.

11.4.13 Transfer the sample to a ridged stainless steel planchet.

11.4.14 Wash down the beaker with small portions of 4 N HNO_3 and add to the planchet.

11.4.15 Evaporate planchets to dryness on a hot plate. Do not allow the sample to splatter.

11.4.16 Remove sample planchets from hot plate.

11.4.17 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 11.4.18.
11.4.18 Cool planchets in a desiccator for a minimum of 30 minutes.
11.4.19 Weigh the cooled planchets and record final weight(s).
11.4.19.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.4.19.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.4.19.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).
11.4.20 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.5 Oil Sample
11.5.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required.
11.5.2 Fill a 50 mL beaker ¼ full with confetti made from Whatman No. 41 filter paper or ashless paper pulp.
11.5.3 Place beaker on analytical balance, then record the weight in appropriate sample worksheet.
11.5.4 Weigh to the nearest 0.0001 g, approximately 0.1-1gram of the oil sample onto the shredded filter paper. Record the sample weight.
11.5.5 Cover with a crucible lid.
11.5.6 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 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.
11.5.7 Ramp oven to approximately 600° C and hold there for four hours.
11.5.8 Turn off the muffle oven, crack open the door, and allow the sample to cool to room temperature.
11.5.9 Add approximately 7 mL of 4 N HNO_3 to the residue in the beaker.
11.5.10 Transfer the sample to a glass beaker with 4 N HNO_3.
11.5.11 Wash down the beaker and lid with small portions of 4 N HNO_3 and add to beaker.
11.5.12 Evaporate to dryness on hot plate. Do not allow sample to splatter. Remove from heat and allow to cool to room temperature.
11.5.13 Add 10 mL of concentrated nitric acid.
11.5.14 Evaporate to dryness on a hot plate. Do not allow sample to splatter.
11.5.15 Remove from heat and allow to cool to room temperature.
11.5.16 Add 10 mL of 4 N nitric acid. Heat to dissolve and then to reduce volume to approximately 5-7 mL.
11.5.17 Transfer sample to a ridged stainless steel planchet.
11.5.18 Wash down the beaker with small portions of 4 N HNO_3 and add to the planchet
11.5.19 Evaporate to dryness on a hot plate, do not allow the sample to splatter.
11.5.20 Remove sample from hot plate.
11.5.21 If sample appears hygroscopic, dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours. If not hygroscopic proceed to step 11.5.22.
11.5.22 Cool planchets in a desiccator for a minimum of 30 minutes.
11.5.23 Weigh the cooled planchets and record final weight.

11.5.23.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.5.23.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.5.23.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).

11.5.24 Store dry sample planchets in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.6 Filter Samples

11.6.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required.

11.6.2 If the filter is less than 2" diameter, 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 11.6.20.

11.6.3 If the filter is 2" diameter, the sample can be placed directly in the dectector.

11.6.4 If the filter is greater than 2" diameter, digest or leach the sample per ST-RC-0004 for shared filters. Prepare a method blank and LCS from blank filters, spiked as per 9.3.3, which are digested in the same manner as the samples.

11.6.5 Shake the digested sample thoroughly. Measure a volume of sample into an appropriately sized teflon beaker. Record volume of sample used.

11.6.6 Add 10 mL of 16 N nitric acid.

11.6.7 Evaporate to dryness on a warm hot plate. Do not allow the sample to splatter.

11.6.8 Remove from heat and allow to cool to room temperature.

11.6.9 Add 10 mL of 16 N nitric acid.

11.6.10 Evaporate to dryness on a warm hot plate, do not allow the sample to splatter.

11.6.11 Remove from heat and allow to cool to room temperature.

11.6.12 Add 10 mL of 4 N nitric acid.

11.6.13 Heat to dissolve and then to reduce volume to approximately 5-7 mL

11.6.14 Transfer the sample to a pre-weighed, stainless steel planchet.

11.6.15 Wash down the beaker with small portions of 4 N HNO_3 and add to the planchet.

11.6.16 Evaporate to dryness on a warm hot plate. Do not allow the sample to splatter.

11.6.17 If sample appears hygroscopic dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours. If not hygroscopic proceed to step 11.6.18.

11.6.18 Cool planchets in a desiccator for a minimum of 30 minutes.

11.6.19 Weigh the cooled planchets and record final weight.

11.6.19.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.6.19.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).

11.6.19.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).

11.6.20 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.7 Solid and/or Soil Samples by Dry, Grind Sprinkle

11.7.1 Initiate appropriate sample worksheet for the samples to be analyzed and complete as required.

11.7.2 If the sample has already been prepared per ST-RC-0003, "Drying and Grinding of Soil and Solid Samples," proceed to step 11.7.8 for direct sample mounting.
11.7.3 Use table salt for the blank and a soil standard reference material, e.g. NIST Traceable Rocky Flats Soil, for the LCS. Prepare in the same fashion as the samples.
11.7.4 Remove an aliquot (typically 1 - 5 g.) with a spatula and place into a clean, labeled aluminum weighing pan.
11.7.5 Place sample on a hot plate or in a drying oven at approximately 105° C and evaporate any moisture.
11.7.6 When dry, remove from hot plate or oven and allow the sample to cool.
11.7.7 If necessary, using a metal spatula, reduce the solid sample to a fine particle size.
11.7.8 Use double sided tape to secure the self-adhesive dots (adhesive side up) to a flat stainless steel planchet. Self adhesive label dots are used to hold finely divided solid material uniformly for gross alpha and/or beta analysis. Weigh and record the prepared planchet.
11.7.9 Distribute the sample evenly in the stainless steel planchet.
11.7.10 Record final weight. The target mass is 40-100 mg.
11.7.10.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.7.10.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.7.10.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).
11.7.11 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.8 Solid and/or Soil Samples by total dissolution.
11.8.1 Initiate sample preparation sheet.
11.8.2 Weigh 1.0g sample into a 50 mL beaker and record weight.
11.8.3 Place in oven at 600° and allow to muffle for four hours. Allow to cool.
11.8.4 Transfer to digestion tube using 4 M HNO_3.
11.8.5 Carefully add 5 mL concentrated nitric acid, 5 mL concentrated hydrochloric acid and 10 mL concentrated Hydrofluoric acid.
11.8.6 Digest in mod block at > 110°C for approximately four hours or until dry.
11.8.7 Carefully add 5 mL concnetrated nitric acid, 5 mL concentrated hydrochloric acid and 10 mL concentrated Hydrofluoric acid.
11.8.8 Digest in mod block at > 110°C for approximately four hours or until dry.
11.8.9 Add 10 mL HNO_3 and digest in mod block at >110°C for approximately 4 hours or until dry.
11.8.10 Reflux sample with 10 mL 4M HNO_3 for 20 minutes using a watchglass over digestion vessel.
11.8.11 Bring up to 20 mL with 4M HNO_3 in the digestion vessel.
11.8.12 Transfer 1 mL of sample to a tared planchet and cook to dryness.
11.8.13 Cool in descicator for 30 minutes.
11.8.14 Reweigh the planchet to determine the mass of 1 mL.
11.8.15 Determine the total amount of sample needed to reach the target mass of 100 mg on the planchet.
11.8.16 Transfer amount of sample to a 250 mL beaker.
11.8.17 Prepare blank, MS and LCS.
11.8.18 Add 10 mL of HNO_3 and cook to dryness. Allow to cool.
11.8.19 Add 10 mL of 4 N nitric acid.

11.8.20 Heat on hot plate to dissolve sample residue and then to reduce volume to approximately 5-7 mL.
11.8.21 Transfer the sample to a ridged stainless steel planchet.
11.8.22 Wash down the beaker with small portions of 4 N HNO_3 and add to the planchet.
11.8.23 Evaporate to dryness on a warm hot plate. Do not allow liquid to splatter.
11.8.24 Remove sample from hot plate.
11.8.24.1 If sample appears hygroscopic, dry planchets in an oven at 105 ± 5 °C for a minimum of 2 hours.
11.8.25 Weigh the cooled planchets and record final weight.
11.8.25.1 If alpha and beta are to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.8.25.2 If alpha only is to be determined simultaneously from a single aliquot, the net residue weights for alpha apply; mass should not exceed 100 mg (2.0" planchet).
11.8.25.3 If beta only is to be determined simultaneously from a single aliquot, the net residue weights for beta apply; mass should not exceed 200 mg (2.0" planchet).
11.8.26 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

11.9 Reprocessing planchets which are over the weight limit.
11.9.1 Rinse residue from planchet with 4 N HNO_3 into a beaker. Add 4 N HNO_3 to planchet and heat if necessary to complete the transfer.
11.9.2 Redissolve the residue into 4 N HNO_3. Dilute the sample to a known volume.
11.9.3 Remove an aliquot which will keep the residue weight under the limit (100 mg) and transfer to the pre-weighed planchet. Record information on sample worksheet.
11.9.4 Evaporate to dryness on a warm hot plate so that the sample does not boil.
11.9.5 Remove sample from hot plate. Allow to cool.
11.9.6 Weigh the cooled planchet and record final weight.
11.9.7 Store dry sample in a desiccator. The sample is ready for gross alpha and/or beta activity analysis by GFPC.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analytical SOP.
12.2 To calculate the aqueous sample volume required (ml), use the following equation:

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

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

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analytical SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in LIMS

14.2 Demonstration of Capability
- 14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification
- 14.3.1 The manager/supervisor 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 the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 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 the Corporate Safety Manual for "Waste Management and Pollution Prevention."

16.2 Waste Streams Produced by the Method
- 16.2.1 The following waste streams are produced when this method is carried out.
  - 16.2.1.1 Acidic sample waste generated. All acidic waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

17.0 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 DOE Method RP-710, "Laboratory Method for Gross Alpha and Beta Activity Determination, 1997

17.4 TestAmerica St. Louis Laboratory Quality Assurance Manual (ST-QAM)

17.5 Corporate Environmental Health and Safety Manual (CW-E-M-001) and Facility addendum.

17.6 Associated SOPs

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

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Updated section 9.2.7: added leached analyses use of a method blank that is comprised of leaching acid.
19.2 Replaced piptte with pre-weighted beaker to measure sample aliquot in section 11.3.3.

19.3 Rev 14:
19.3.1 Updated the total dissolution procedure for solid/soil samples in Section 11.8.

19.4 Revision 15:
19.4.1 Added required pH checking for all aqueous samples prior to analysis in section 8.3.
19.5 Revision 16:
19.5.1 Added reference to DOE Method RP-710 to Sections 1 and 17

19.6 Revision 17:
19.6.1 Updated section 15.
19.6.2 Removed Structure and Analysis Codes from SOP and referenced LIMS as the new source to recover that information in section 1.0.
19.6.3 Removed references to 'Clouseau" and "Quantims", replaced with LIMS.
19.6.4 Updated method requirements for Air Filter samples in section 2.0.
19.6.5 Updated supplies in section 6.0.
19.6.6 Updated reagents and standards in section 7.0.
19.6.7 Replaced the use of a porcelain crucible with a beaker throughout section 11.0.
19.7 Rev.18:
19.7.1 Section 8, removed sample hold time
19.7.2 Section 11.3.1, added GAB solid screen form Rad-0052
19.7.3 Section 11.1.10, added GAB solid screen form Rad-0052
19.7.4 Grammatical errors fixed throughout

Title: PREPARATION OF SAMPLES FOR GAMMA SPECTROSCOPY [EPA 901.1 and DOE GA-01-R]

Approvals (Signature/Date):

Sarah Bernsen 8/29/13 Sarah Bernsen — Date Radiochemistry Prep Supervisor	Terry Romanko 8/30/13 Terry Romanko *for* Michael Ridenhower — Date Health & Safety Manager / Coordinator
8-29-13 Tony L. Byrd *for Marti Ward* — Date Quality Assurance Specialist	Elaine Wild 8/29/13 Elaine Wild — Date Laboratory Director

This SOP was previously identified as SOP No. ST-RC-0025 Rev. 14

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1.0 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 describes methods for the preparation of samples of liquid, soil, vegetation, air filter, and core matrices prior to gamma spectroscopy analysis.

1.3 This SOP is based on EPA Method 901.1 and DOE Method GA-01-R.

1.4 The laboratory target analytes supported by this method, the reporting limits, and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Samples are transferred to a standard geometry container for counting on the gamma detectors. High purity 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. A 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. Detection limits may be affected by the sample size. Gamma photon energies not identified in the reference library may be identified and evaluated manually.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

3.2 Replicate Analyses – Two or more analysis of the same sample whose independent measurements are used to determine the precision of equipments analytical procedure.

4.0 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 misidentification. It is not possible to eliminate all interferences and misidentification.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to

follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 **Wear Kevlar or MAPA Blue-Grip gloves when using knives or sharp articles.**

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 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. Nitric acid 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.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

6.0 EQUIPMENT AND SUPPLIES

6.1 Balance, top loader

6.2 Blender

6.3 Food chopper/grinder

6.4 Knives appropriate for food preparation

6.5 Graduated cylinder

6.6 Filter disk, 47 millimeter diameter

6.7 Plastic Tape

6.8 Marinelli beakers of various sizes (500-mL and 1000-mL)

6.9 Petri dishes, 2-inch diameter

6.10 Can Sealer

6.11 Cans and lids (commonly referred to as tuna cans)

6.12 8 oz, straight sided polypropylene jars or equivalent; (used for 25 mL and 100 mL geometries)

6.13 Teflon® or glass beakers (250-mL, 400-mL)

6.14 Disposable digestion vessels

6.15 Muffle furnace (programmable)

6.16 TEXPEN®

6.17 Teflon® beaker covers

6.18 Watch glasses

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002.

7.2 DI Water obtained from the Milli-Q® unit.

7.3 Nitric acid (16 N HNO_3) concentrated

7.3.1 Nitric acid (4 N HNO_3) – to an appropriately sized bottle containing 1500 mL of DI water; add 500 mL of 16 N HNO_3.

7.4 Hydrochloric acid (12 N HCL) – concentrated, 37.2%

7.5 Hydrofluoric acid (HF 48.52%) – concentrated

7.6 Radiacwash™ solution 10% - add 100 mL of Radiac to 1 L of water

7.7 Bleach solution 10% - add 100 mL of bleach to 1 L of water

7.8 Sodium Sulfate

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica 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 ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2, unless I-129 or I-131 is requested. Samples collected for I-129 or I-131 analysis are ***not preserved***.

8.3.1 The pH of aqueous samples are checked upon receipt by Sample Control, therefore, the pH does not require checking prior to analysis

8.3.1.1 Aqueous samples acidified upon receipt (designated by a label on the bottle) do require checking the pH prior to analysis.

8.4 Milk samples are not chemically preserved.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (i.e. water sample volatile organics, water sample anion analysis) the batch is comprised of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

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

9.1.3 For this analysis, batch QC consists of a Method Blank (MB), a Laboratory Control Sample (LCS), and Sample Replicate.

9.1.3.1 A Sample Duplicate may be performed at the request of the client. See client requirements.

9.2 **Method Blank**

9.2.1 For **Water** and **Liquid** analyses, the method blank is comprised of DI water.

9.2.2 For **Soil** and **Solids** analyses, the method blank is comprised of sodium sulfate.

9.2.3 For Filter analyses, the method blank is comprised of a Petri dish.

9.2.4 A Method Blank must be prepared with every sample batch.

9.3 **Laboratory Control Sample**

9.3.1 An LCS is 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 The LCS is a purchased sealed source standard in the prescribed geometry for the sample analysis.

9.3.3 An LCS must be prepared with every sample batch.

9.4 **Sample Duplicate**

9.4.1 A sample duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision (a replicate analysis of the original sample counted on a different detector will be performed as the duplicate).

9.5 **Procedural Variations/ Nonconformance and Corrective Action**

9.5.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

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

9.6 **Decontamination of Tuna Can Sealer**

9.6.1 The sealer must be wiped down with 10% Radiacwash™ solution daily

9.6.1.1 Analyst must record the information in the daily logbook.

9.6.2 Sealer will be monitored for contamination as part of the monthly contamination survey, as per SOP ST-RP-0032

10.0 CALIBRATION AND STANDARDIZATION

10.1 The balance must be calibrated in accordance with ST-QA-0005.

10.2 For Gamma Spectroscopy calibration requirements, see ST-RD-0102.

11.0 PROCEDURE

11.1 Liquid Sample Preparation

11.1.1 Liquid samples shall be prepared as a 25 mL, 100 mL, 500 mL, or 1000 mL geometry.

11.1.2 Determine the proper geometry.

11.1.2.1 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 count room supervisor or radiochemistry technical director, if the sample has high activity which may require such consideration.

11.1.3 Label the top of the container with the sample Id

11.1.4 Shake the sample to suspend any residue and to ensure that the sample is homogeneous.

11.1.5 Write sample information (i.e. ID #) on the container.

11.1.6 Measure the required sample volume (25, 100, 500 or 1000 mL) by comparing the sample to the reference container

11.1.6.1 Reference containers are pre-made geometries comprised of DI water measured volumetrically.

11.1.6.2 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. Consultant Supervisor/Manager to determine which action is preferable.

11.1.6.2.1 If the sample is diluted the undiluted volume is recorded as the sample volume. The dilution is only for fitting the calibrated geometry.

11.1.7 Place the lid securely on the container.

11.1.7.1 Remove excess air from Marinelli.

11.1.7.2 If the density is suspected to be greater than 1.2 g/ mL or less than 0.98 g/mL, generate a NCM. To determine the density use form RAD-0075_Density.xls (include form with batch paper work if utilized) Attachment 1

11.1.7.2.1 Form directory: \\slsvr01\QA\FORMS\ST-LOUIS\RAD

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 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

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. Proceed to step 11.2.3.

11.2.2 Soil samples, which do not require I-129 or I-131 analyses, are prepared in accordance with SOP ST-RC-0003 or ST-RC-0014.

11.2.3 Soil samples shall be prepared as 200 mL sealed (tuna) can, 100 mL, or 25 mL, or 500 mL Marinelli (marnsoil) geometry based on the amount of available sample. In both the tuna can and marnsoil geometries, the soil should nearly fill the container.

11.2.3.1 For I-129 analysis only a 25 mL or 100 mL straight sided poly jar geometry may be used (check with count room analyst on which geometry I-129/I-131 is calibrated for and prep the sample using a matching geometry).

11.2.4 Write sample information (i.e. ID #) on the sample container.

11.2.5 Pre-weigh the empty container (tare weight) and record weight in TALS and on the container lid.

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 container. If there is insufficient sample to fill, reduce the geometry size.

11.2.6.2.1 A 100 mL "reference" bottle is marked with 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 25 mL "reference" container is marked with 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 Close the sample container securely.

11.2.8 Seal the container with plastic tape. For tuna cans, seal with can sealer, wipe samples clean with paper towel and DI water.

11.2.9 Place the sample on the balance; record the weight of the sample on the gamma worksheet (total weight of container plus sample minus the tare weight of the empty container).

11.2.10 Generate a label and proper paperwork then submit to count room for analysis by gamma spec.

11.3 Vegetation Sample Preparation (No digestion)

11.3.1 Vegetation samples may be prepared in an appropriate calibrated geometry counted directly as dried and chopped matrix or green unprocessed matrix (if directed to do so by the client or if I-131 or I-129 is to be reported).

11.3.1.1 Green unprocessed samples can be reported on a wet or dry basis, determined by client or Project Manager.

11.3.1.1.1 Vegetation samples **for I-129 or I-131 analysis are not dried** but rather inserted into an appropriate calibrated geometry.

11.3.1.1.2 Dry weight can be determined on sample(s) not dried by using the percent moisture.

11.3.1.2 Consult the client requirements, client requirement memorandums or the Supervisor/Manager to determine proper sample handling.

11.3.2 The sample shall be counted in a 500 mL Marinelli, 100 mL, or 25 mL geometry. The container is filled to the appropriate level with the sample.

11.3.2.1 I-129 analysis uses only a 25 mL or 100 mL straight sided poly jar geometry (check with count room analyst on which geometry I-129/I-131 is calibrated for and prep the sample using a matching geometry).

11.3.3 Write sample information (i.e. ID #) on the container.

11.3.4 Pre-weigh the empty container and record weight in TALS and on lid of container.

11.3.5 Place sample in the tared container.

11.3.5.1 Compress the sample when filling a 500 mL Marinelli beaker

11.3.6 Place the sample on the balance; record the weight of the sample on the gamma worksheet (total weight of container plus sample minus the tare weight of the empty container).
11.3.6.1 Verify with the Project Manager if sample is to be reported on a wet or dry basis.
11.3.7 Close the sample container securely, seal with plastic tape
11.3.8 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.4 Vegetation Sample Preparation (with digestion)
11.4.1 Vegetation samples may be digested and then placed into a appropriate calibrated geometry and counted as a liquid matrix.
11.4.1.1 Green unprocessed samples can be reported on a wet or dry basis, determined by the client or project manager.
11.4.1.1.1 **Iodine isotopes (e.g. I-125, I-129 or I-131) cannot be prepared using digestion technique** due to volatility.
11.4.1.2 Consult the client requirements and Supervisor/Manager to determine proper handling.
11.4.2 Label an appropriate sized beaker with a TEXPEN® to ensure identification post muffling
11.4.3 Transfer sample into beaker and record the weight on the gamma worksheet
11.4.3.1 Check with the Project Manager to determine if sample is to be reported on a wet or dry basis.
11.4.4 Cover sample with 8 N HNO_3 and allow to sit overnight.
11.4.5 Place beaker on hot plate and gently heat so that sample does not splatter. Periodically stir sample
11.4.6 Wash down sides of the beaker with small amounts of 30% H_2O_2 and small amounts of 8M HNO3. Continue this process periodically as sample digests on hot plate.
11.4.7 Cook sample down to dryness.
11.4.7.1 Upon visual inspection if sample is orange in color, cover sample with 16M HNO_3 and continue to wet ash with 30% H_2O_2.
11.4.7.2 Repeat 11.4.7 and 11.4.7.1 until sample is light yellow or white in color.
11.4.8 Cover beaker with a watch glass and place into the muffle oven.
11.4.9 Heat at 600□C for a minimum of 4 hours to reduce the sample
11.4.10 Allow beakers to cool to room temperature before removing from the oven.
11.4.10.1 The sample may be wet ashed again if yellow color remains.
11.4.11 Reflux samples with enough of 4 N HNO_3 to cover sample for approximately 30 minutes, using a watch glass to cover the beaker.
11.4.12 Quantitatively transfer sample to a labeled digestion vessel or Teflon® beaker with a minimal amount of 4 N HNO_3.
11.4.13 Add 5 mL of 16 N HNO_3, 5 mL of 12 N HCl and 10 mL of concentrated HF
11.4.14 Bring sample to dryness
11.4.14.1 If using a digestion vessel, place in MOD Block
11.4.14.2 If using a Teflon® beaker, heat on a hotplate
11.4.15 Repeat steps 11.4.10 and 11.4.11
NOTE: The amount of acid will vary depending on the sample aliquot. When increasing the aliquot, increased acid will be required
11.4.16 Reflux the sample in 10 to 20 mL of 4 N HNO_3. Allow to reflux with a watch glass over the beaker for approximately 30 minutes to ensure samples goes into solution.
11.4.16.1 If sample does not go into solution additional digestions may be required (see Supervisor/Manager/Technical Director for guidance)
11.4.17 Label appropriate size container (25 or 100 mL)
11.4.18 Quantitatively transfer sample to container by rinsing with minimal amount of 4 N HNO_3
11.4.19 Bring to volume to match the reference container
11.4.20 Close the sample container lid securely and seal with plastic tape.
11.4.21 Inspect for leakage.

11.4.22 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.5 Air Filters/Swipes (no digestion)

11.5.1 Air filters may be counted as single filters or as composite filters.

11.5.1.1 Consult client requirements and Supervisor/Manager for instruction.

11.5.1.2 Air filters are reported as pCi/sample or pCi/g. The weight to the air filter is required if the reporting units is pCi/g.

11.5.2 Write sample information (i.e. ID#) on Petri dish.

11.5.3 Filters with reporting units of

11.5.3.1 pCi/g proceed to 11.5.4

11.5.3.2 pCi/sample proceed to 11.5.5

11.5.4 Pre-weigh empty Petri dish, record the weight in TALS and then on the lid of the Petri dish.

11.5.5 Load air filter(s) directly into Petri dish

11.5.6 Place lid on Petri dish, for pCi/g record the weight of the sample (container and sample weight minus container weight) in TALS.

11.5.7 Secure the Petri dish lid with plastic tape

11.5.8 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.6 Air Filters/Swipes (with digestion)

11.6.1 Air filters are digested and counted as a liquid.

11.6.2 Label a 250 mL beaker with a TEXPEN® to ensure identification post muffling.

11.6.3 Place sample into beaker

11.6.4 Place sample beaker in muffle oven and cover with a watch glass.

11.6.5 Ramp air filter in muffle oven

11.6.5.1 Refer to ST-RC-004 for ramping settings

11.6.6 Allow beakers to cool to room temperature

11.6.7 Digest filter in accordance with sections 11.4.9 to 11.4.12

11.6.8 Label appropriate size container (25 to 100 mL)

11.6.9 Transfer sample to container with minimal amount of 4 N HNO_3.

11.6.10 Bring to volume using reference container as a guide

11.6.11 Place lid securely on the container

11.6.12 Seal the lid using plastic tape

11.6.13 Inspect for leakage.

11.6.14 Generate a label and proper paperwork then submit to count room for analysis by gamma spec.

11.7 Core Samples

11.7.1 To obtain sample, cut Shelby tube or sample container into two pieces.

11.7.1.1 Using a rigid pipe cutter cut the tube completely through.

11.7.1.2 Using a wire saw, cut through the sample.

11.7.1.3 Cuts should be made at 2 inch intervals.

11.7.1.4 Remove sample from every other sliced section of the Shelby tube.

11.7.1.5 Dry and grind the sample as described in SOP ST-RC-0003.

11.7.2 Aliquot 500 g of sample

11.7.2.1 If less than 500 g of sample is available, contact Supervisor/Manager for instruction.

11.7.2.2 Soil samples shall be prepared as 200 mL sealed (tuna) can, 100 ml, or 25 ml, or 500 mL Marinelli (marnsoil) geometry based on the amount of available sample. In both the tuna can and marnsoil geometries, the soil should nearly fill the container.

11.7.3 Pre-weigh the empty container and record the weight on the container lid and in TALS

11.7.4 Writer sample information (i.e. ID#) on the sample container
11.7.5 Place the dried sample into the container for counting.
11.7.6 Weigh and record sample weight in TALS .
11.7.7 Secure the lid on the container with plastic tape.
11.7.8 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.8 Food: vegetables, produce, grain or animal feed:
11.8.1 Vegetables, produce and grain samples may be prepared in a 500 mL Marinelli beaker or 1 liter Marinelli beaker geometry depending on the requested reporting limit. 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.
11.8.1.1 Consult the client requirements and Supervisor/Manager for instruction.
11.8.2 For vegetables and produce, prepare the sample by chopping with a knife on a cutting board or using a food processor.
11.8.3 Write sample information (i.e. ID #) on the appropriate container.
11.8.4 Pre-weigh the empty container and record weight on container and in TALS.
11.8.5 Place processed sample in the pre-weighed container.
11.8.6 Compress the sample when filling a 500 mL Marinelli beaker
11.8.7 Weigh sample and record the weight in TALS as WET weight in grams.
11.8.7.1 If the sample is dried, record the DRY weight.
11.8.8 Close the sample container securely, seal with plastic tape.
11.8.9 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.9 Food: meat and fish:
11.9.1 Meat and fish may be prepared in a 500 mL Marinelli beaker or 1 liter Marinelli beaker geometry depending on the requested reporting limit. These matrices are counted directly **without drying.**
11.9.1.1 Consult the client requirements and Supervisor/Manager for instruction.
11.9.2 For meat and edible portions of fish, prepare the sample by chopping with a knife on a cutting board.
11.9.2.1 Fish sample are to be filleted prior to chopping.
11.9.2.2 For analysis of fish **when the whole fish is required to be analyzed**, 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.
11.9.3 Write sample information (i.e. ID #) on the container.
11.9.4 Pre-weigh the empty container and record weight on container and in TALS.
11.9.5 Place processed sample in the pre-weighed container.
11.9.6 Compress the sample evacuating any space in the geometry when filling a Marinelli beaker
11.9.7 Weigh sample and record the weight in TALS as WET weight in grams.
11.9.8 Close the sample container securely, seal with plastic tape.
11.9.9 Generate a label and print proper paperwork then submit to count room for analysis by gamma spec.

11.10 Store unused portions of sample in appropriately sized poly containers. Food and Vegetation samples are to be refrigerated.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 There are no calculations pertaining to this sample preparation procedure.

12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analysis SOP.

12.3 Percent Moisture: Mass of original sample minus mass of dried sample divided by mass of dried sample.

12.3.1 $$p = \frac{W - D}{D}$$

p = fraction of total evaporable moisture content of sample
W = mass of the original sample
D = mass of dried sample

12.4 Density: Sample weight divided by the volume of said sample weight

12.4.1 $$d = \frac{m}{v}$$

d = density
m = mass
v = volume

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

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability
- 14.2.1 Initial and continuing demonstrations of capability requirements are established in ST-QAM.

14.3 Training Qualification
- 14.3.1 The Supervisor/Manager 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 ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in ST-QAM

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 ST-QAM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. TestAmerica St. Louis will include this information in the SOP when accreditation is sought for a performance based measurement system or non-standard method.

16.0 WASTE MANAGEMENT AND POLLUTION PREVENTION

16.1 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."

16.2 Waste Streams Produced by the Method
The following waste streams are produced when this method is carried out.

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

16.2.2 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.

17.0 REFERENCES

17.1 Prescribed Procedures for Measurement of Radioactivity in Drinking Water Method EPA 901.1.

17.2 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, GA-01-R.

17.3 TestAmerica St. Louis Quality Assurance Manual (ST-QAM), current revision.

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revision.

17.5 Associated SOPs

17.5.1 ST-RC-0003, Drying and Grinding of Soil and Solid Samples
17.5.2 ST-RC-0004, Preparation of Soil, Sludge, and Filter Paper Samples for Radiochemical Analysis
17.5.3 ST-RD-0102, Gamma Spectroscopy Analysis
17.5.4 ST-PM-0002, Sample Receipt and Chain of Custody
17.5.5 ST-QA-0002, Standard and Reagent Preparation
17.5.6 ST-QA-0036, Non-conformance Memorandum (NCM) process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 No Changes, Annual Review

19.2 Rev 11:

- 19.2.1 Inserted instructions regarding requirements for checking the pH of samples upon receipt and or prior to analysis in section 8.3.1.
- 19.2.2 Updated when sample ID's should be written on the container in section 11.0.
- 19.2.3 Inserted instructions for generating container ID labels throughout section 11.0.
- 19.2.4 Updated when weighing and recording sample weights/mass should be documented in gamma worksheet in section 11.0.
- 19.2.5 Added instructions for properly cleaning tuna cans before storage in section 11.2.9.

19.3 Rev 12:

- 19.3.1 Extensive revision to entire procedure
- 19.3.2 Addition of attachment 1

19.4 Rev 13:

- 19.4.1 Added sodium sulfate to section 7.0.
- 19.4.2 Updated section 9.2 regarding the matrix of method blanks.

19.5 Rev 14:

- 19.5.1 Updated vegetation sample with and without digestion throughout section 11.3 and 11.4.

19.6 Rev. 15:

- 19.6.1 Grammatical corrections and removal of references to QuantIMS through out
- 19.6.2 Section 3, added definition of replicate analyses
- 19.6.3 Section 8, removal of 180 day holding time
- 19.6.4 Section 9, explained that replicate will be used as duplicate for this procedure
- 19.6.5 Section 9.6, added "record in daily logbook"
- 19.6.6 Deleted record tare weight on lid in section 11.1.2
- 19.6.7 Deleted record weight of sample (container and sample weight minus container weight) on lid in section 11.1.8 , 11.2.9, 11.3.6
- 19.6.8 Added record weight in TALS throughout SOP
- 19.6.9 Added print proper paperwork throughout SOP

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Attachment 1

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

DENSITY CORRECTION

Date: _____ **Digi Tube Lot #:** _____

Analyst: _____ **Balance ID:** _____

	Sample ID:	Container Wt (g)	Sample Volume (mL)	Sample + Container Wt (g)	Density (g/mL)	Original Sample Aliquot	Adjust Sample Aliquot
1							
2							
3							
4							
5							
6							

Instructions:
Record the weight of a Class A digi tube in "Container Wt(g)"; record the volume of the sample added to the digit tube in "Sample Volume (mL)"; record the sample and digi tube in "Sample + Container Wt(g)." "Original Sample Aliquot" is the full sample size used for the actual analysis.

This density is to be used for sample aliquot correction when a sample is suspected of density less than or greater than 1. Include this document with batch paperwork.

Review is to be completed by a peer (someone other than the analyst handling the samples).

Reviewed by: _____ Date: _____

\\slsvr01\QA\FORMS\ST-LOUIS RAD-0075 REV (0)

Title: TOTAL ALPHA EMITTING ISOTOPES OF RADIUM (TAR) [EPA 903.0 & SW-846 9315]

Approvals (Signature/Date):

Sarah Bernsen 8-6-13	Michael Ridenhower 8/6/13
Sarah Bernsen — Date	Michael Ridenhower — Date
Radiochemistry Prep Supervisor	Health & Safety Manager / Coordinator
Marti Ward 8-6-13	Elaine Wild 8/6/13
Marti Ward — Date	Elaine Wild — Date
Quality Assurance Manager	Laboratory Director

This SOP was previously identified as SOP No. ST-RC-0040 Rev. 11

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1.0 SCOPE AND APPLICATION

1.1 This procedure describes the determination of total radium, for all isotopes emitting alpha radiation, using EPA method 903.0, SW846 Method 9315.

1.2 This procedure applies to the analysis of these isotopes in liquid and in other media where dissolution and carrier exchange are readily available in the laboratory.

1.3 The barium sulfate can be counted for total alpha radiation. The time of the last barium sulfate precipitation should be recorded and used in calculating the in-growth factor.

1.4 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 Barium and lead are used to coprecipitate radium as the sulfate. Following chelation with EDTA, barium 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 in-growth of radium-226 progeny, gravimetric yield and counting efficiency.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers .

3.2 There are no specific definitions for this procedure.

4.0 INTERFERENCES

4.1. This procedure screens for radium-226 by measuring the alpha emitting radium isotopes. It follows that if there is no detectable radium alpha activity there would be no radium-226 above the specified detection limit.

4.2. Samples which contain natural barium cause inaccurate chemical yield determinations.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

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) 4ppm (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 (TWA)	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.
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 (TWA)	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.			
TWA – Time Weighted Average			
STEL – Short Term Exposure Limit			

6.0 EQUIPMENT AND SUPPLIES

6.1 Centrifuge tubes, 50 mL

6.2 Centrifuge

6.3 Hot plate

6.4 Analytical balance

6.5 Stainless steel planchet

6.6 Syringe, 20 mL, .45mm filter

6.7 Glassware, beakers

6.8 Water Bath

6.9 Desiccator

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI water 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 $(NH_4)_2SO_4$ in 300 mL DI water. Bring to a volume of 1000 mL.

7.6 Ammonium sulfide, 2%: Dilute 10 mL $(NH_4)_2S$, (20-24%), to 90 mL water; total volume 100 mL.

7.7 Barium carrier (standardized) – 33.9 mg/mL, NIST traceable

7.7.1 If the barium carrier is not already standardized, standardize the barium carrier solution using the following procedure.

7.7.1.1 Pipette 1.0 mL barium carrier solution (16 mg/mL, Ba) into six separate labeled centrifuge tubes containing 15 mL DI H_2O.

7.7.1.2 To each tube, add 1 mL 18 N sulfuric acid while stirring and digest precipitate in a hot water bath for approximately 10 min.

7.7.1.3 Cool, centrifuge and decant the supernate into appropriate waste container.

7.7.1.4 Wash the precipitate with 15 mL DI water, centrifuge and decant the supernate.

7.7.1.5 Transfer the precipitate to a pre-weighed stainless steel planchet with a minimal amount of DI water.

7.7.1.6 Dry on a heat source. Store in desiccator until cool and weigh as barium sulfate.

7.7.1.7 Record the net weights of the precipitates and calculations in the Rad Standards Preparation Log.

7.8 Citric acid (1M) - dissolve 19.2g of $C_6H_8O_7 \cdot H_2O$ in water and dilute to 100 mL.

7.9 EDTA reagent basic (0.25M) - dissolve 20g NaOH in 750 mL 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.

7.10 Lead carrier (15 mg/mL) - dissolve 2.397g $Pb(NO_3)_2$ in water, add 0.5 mL 16N $HN0_3$ and dilute to 100 mL with water.

7.11 Methyl orange indicator (0.1%) - dissolve 0.1 g methyl orange indicator in 100 mL water.

7.12 Nitric acid (16 N, 70.4%), concentrated $HN0_3$, sp. gr.

7.13 Sulfuric acid (18 N) - Cautiously mix 1 volume 36N H_2S0_4 (concentrated) with 1 volume of water.

7.14 Radium-226, standard 20-25dpm, NIST traceable

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica 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 ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2.

8.3.1 The pH of aqueous samples is checked upon receipt by the Sample Control Department. The pH does not require re-checking prior to analysis.

8.3.2 Aqueous samples acidified upon receipt (designated by a label on the bottle) do require a check of the pH prior to analysis.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch comprises of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

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

9.1.3 For this analysis, batch QC consists of a method blank, a 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 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.1.5 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Liquid analyses, the method blank is comprised of DI water.

9.2.4 For Soil analyses, the method blank is comprised of DI water acidified with 2ml of nitric acid.

9.3 **Laboratory Control Sample**

9.3.1 An LCS is 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 sample batch.

9.3.3 For Liquid analyses, the LCS is comprised of DI water fortified with radium-226.

9.3.4 For Soil analyses, the LCS is comprised of radium-226.

9.4 **Matrix Spike (MS) /Matrix Spike Duplicate (MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate**

9.5.1 A Sample Duplicate is 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. A NCM is written to document the insufficient volume and utilizing of an LCSD for demonstration of precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

9.6.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA Manager. See SOP ST-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 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes Procedure.

10.2 See the analytical SOP for instrument calibration; ST-RD-0403, "Daily Calibration Verification and Maintenance of the Low Background Gas Flow Proportional Counting System."

11.0 PROCEDURE

11.1 Water Samples

11.1.1 Ensure that the sample container is capped tightly and shake it thoroughly. Transfer a sample aliquot to a beaker.

11.1.2 Sample aliquot size is 1 liter.

11.1.2.1 For client requesting a reporting limit less than 1pCi/L, a larger sample volume may be required. Contact Radiochemistry manager/supervisor for instruction.

11.1.2.2 If less than 1 liter of sample was provided by the client, write the NCM noting insufficient volume.

11.2 Soil Samples

11.2.1 For soil samples prepare per SOP STL-RC-003, "Drying and Grinding of Soil and Solid Samples", and weigh 1 to 2 grams into a labeled crucible.

11.2.2 Place in oven at 600° and muffle for four hours. Allow to cool.

11.2.3 Transfer to digestion tube using 4M HNO_3.

11.2.4 Add 1 mL of standardized barium carrier to samples and QC. Add radium-226 spike to LCS and MS/MSD, if applicable.

11.2.5 Carefully add 5 mL concentrated nitric acid, 5 mL concentrated hydrochloric acid and 10 mL concentrated hydrofluoric acid.

11.2.6 Digest in mod block at >110° for four hours or until dry.

11.2.7 Carefully add 10 mL concentrated nitric acid, 10 mL concentrated hydrochloric acid and 5 mL concentrated hydrofluoric acid.

11.2.8 Digest in mod block at >110° for four hours or until dry.

11.2.9 Dissolve with 10 mL HNO_3 and 10 mL HCl, return to mod block for 30min.

11.2.10 Transfer to 250 mL beakers with 4M HNO_3. Dilute to a final volume of 200 mL with DI water.

11.3 Precipitation

11.3.1 Add methyl orange indicator until red color persists. Add 1M citric acid in ratio of 5 mL per liter; mix thoroughly.

11.3.2 For Waters, perform steps 11.3.2.1 through 11.3.2.3; for soil perform steps 11.3.2.1.

11.3.2.1 2.5 mL of lead carrier.

11.3.2.2 Add 1.0 mL Standardized barium carrier. (33.9 mg/mL)

11.3.2.3 Spike LCS and MS/MSD (if applicable) with radium-226. Document the spike volume and concentration on the prep sheet.

11.3.3 Heat and stir until just beginning to boil.

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

11.3.5 **A face shield is required for this step.** Add 10 mL18N sulfuric acid until the red color reappears or the pH is < 2, and then add 5 mL ammonium sulfate.

11.3.6 Stir and heat the samples for a minimum of 15 minutes.

11.3.7 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.

11.4 In-growth

11.4.1 Decant the supernate and discard into the appropriate waste container, taking care to avoid disturbing the precipitate.

11.4.2 Transfer the precipitate to a 50 mL centrifuge tube, taking care to rinse the last particles out of the beaker with DI water.

11.4.3 Centrifuge until precipitate appears to be compacted at the bottom of the tube. (5-10 minutes). Pour off liquid and save the $BaS0_4$ precipitate.

11.4.4 Add 10 mL 16N $HN0_3$. Cap tube and vortex to ensure complete mixing. Centrifuge for 5-10 minutes at a maximum of 2000 rpm. Pour off the liquid and save the $BaSO_4$ precipitate. Repeat this step if the precipitate visually appears larger than that in the Blank and LCS.

11.4.5 Wash the precipitate with 10 mL DI water. Vortex, centrifuge and discard supernate. Save the $BaSO_4$ precipitate.

11.4.6 Add 20 mL basic EDTA reagent, vortex and heat in a hot water bath until precipitate dissolves.

11.4.6.1 If insoluble solids remain in the tube after the addition of EDTA, confirm the pH is > 10.

11.4.6.2 If pH is >10, centrifuge and syringe filter supernatant into a clean labeled 50 mL centrifuge tube. Discard insoluble residue.

11.4.7 Add 2 mL $(NH_4)_2SO_4$ (200 mg/mL) and stir thoroughly.

11.4.8 Add 3 mL 17.4N Acetic acid or until barium sulfate precipitates.

11.4.9 Digest in a hot water bath until precipitate settles. Centrifuge and discard supernatant.

11.4.10 Repeat steps 11.4.6 to 11.4.10.

11.4.11 Record date and time of the last $BaSO_4$ precipitation on the sample data sheet.

11.4.12 Add 1 mL Lead carrier (1.5 mg/mL)

11.4.13 Dissolve the precipitate in 20 mL basic EDTA.

11.4.14 Vortex, heat in hot bath until the precipitate dissolves.

11.5 Lead Scavenge Clean-up

11.5.1 Add 0.3 mL ammonium sulfide and stir well.

11.5.2 Add 10N sodium hydroxide drop-wise (approximately 0.5 mL) with vigorous stirring until lead sulfide precipitates, then add 10 drops more. Stir intermittently. Centrifuge

11.5.3 Add 1 mL lead carrier (1.5 ng/mL), 0.1 mL ammonium sulfide, and 0.1 mL 10N sodium hydroxide. Centrifuge and filter supernatant through 0.45 mm syringe filter into a clean labeled tube.

11.6 Barium Yield

11.6.1 Add 2 mL Ammonium sulfate and 3 mL Acetic acid or until barium sulfate forms.

11.6.2 Vortex.
11.6.3 Heat in hot water bath.
11.6.4 Centrifuge and decant.
11.6.4.1 If barium sulfate does not form after 2 additions; consult the radiochemistry manager/supervisor for further instruction.
11.6.5 To the precipitate, 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.
11.6.6 Add 2 mL Ammonium sulfate and 3 mL Acetic acid until barium sulfate forms.
11.6.7 Heat in hot water bath.
11.6.8 Centrifuge and decant.
11.6.9 Record time as "T3".
11.6.10 Wash precipitate with 10 to 20 mL DI. Centrifuge and discard supernate. Repeat this step.

11.7 Plating
11.7.1 Transfer precipitate to a pre-weighed stainless steel cleaned planchet with a minimal amount of water.
11.7.1.1 The cleaned planchet has been processed in accordance with SOP: ST-RC-0002. See SOP for additional information.
11.7.2 Heat the planchet again using the hot plate, let cool in a desiccator for a minimum of 15 minutes and then weigh the planchet.
11.7.3 Record the final weight of the planchet to determine the chemical recovery for the barium carrier solution.
11.8 Submit planchet to the count room for analysis.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Commonly used calculations (e.g. LCS % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM.

12.2 There are no calculations pertaining to this sample preparation procedure.

12.3 Total Alpha Radium (TAR) by GFPC calculations are given in SOP: ST-RD-0403.

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

13.1 Data assessment does not pertain to this sample preparation procedure.

13.2 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the associated analytical SOP

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor 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 the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

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 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 6, Method 903.0, Alpha-Emitting Radium Isotopes in Drinking Water

17.2 SW-846,"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", Method 9315, Alpha Emitting Radium Isotopes

17.3 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.5 Associated SOPs, current revisions:

17.5.1 ST-PM-0002, Sample receipt and Chain of Custody

17.5.2 ST-QA-0002, Standard and Reagent Preparation

17.5.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.5.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.5.5 ST-RC-0002, Planchet Preparation for Radiochemistry and Radiological Screening Analysis
17.5.6 ST-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
17.5.7 ST-RD-0403, Gas Flow Proportional Counting (GFPC) Analysis

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 The initial precipitation of total alpha radium uses the technique cited in EPA Method 904.0 instead of 903.0 which uses straight sulfuric acid and fewer carriers to bring down the Pb/Ba sulfate.

18.2 A lead (Pb) scavenge identical to the one found in EPA Method 904.0 has been incorporated into this procedure. Skipping this step could artificially inflate barium yields and thus bias the result.

19.0 CHANGES TO PREVIOUS REVISION

19.1 Updated section 11.4 and 11.5 regarding amount of mL used of Acetic Acid
19.1. Rev 10,
19.1.1. Updated sample collection, preservation and storage times in section 8.0.
19.2. Rev 11:
19.2.1. Annual Review, No Changes
19.3. Rev 12
19.3.1. Grammatical errors fixed throughout SOP
19.3.2. Section 11.4.6.2 added syringe filter supernate
19.3.3. Section 11.4.8 added 3 mL
19.3.4. Section 11.4.11 moved record date and time of last $BaSO_4$ precipitation on the sample date sheet
19.3.5. Section 11.5.2 Deleted “decant supernate into clean tube”
19.3.6. Section 11.6.1 deleted “in increments”

Title: ISOTOPIC THORIUM, PLUTONIUM AND URANIUM IN VARIOUS MATRICES BY EICHROM® SEPARATION RESINS

Approvals (Signature/Date):

Sarah Bernsen 6/20/13 Sarah Bernsen — Date Separations Supervisor	Michael Ridenhower 6/18/13 Michael Ridenhower — Date Health & Safety Manager / Coordinator
Marti Ward 6.18.13 Marti Ward — Date Quality Assurance Manager	Elaine Wild 6/17/13 Elaine Wild — Date Laboratory Director

This SOP was previously identified as SOP No. ST-RC-0242 Rev. 15

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1.0 SCOPE AND APPLICATION

1.1 This SOP provides a rapid, reliable method for separation of Thorium, Plutonium and Uranium in various matrices.
- 1.1.1 If only Uranium analysis is requested, see SOP: ST-RC-0238.
- 1.1.2 If other actinides in addition to Thorium, Plutonium and Uranium are requested, please see SOP index to determine applicable SOP.

1.2 This SOP is based on Eichrom Technologies, Inc. Analytical Procedures "ACW13 VBS Thorium, Plutonium and Uranium in Water (with Vacuum Box System)" and "ACW01 Uranium and Thorium in Water".

1.3 This procedure is applicable to water, soil, filter, biota, and oil.
- 1.3.1 Soil, filter, biota and oils are pre-prepared in accordance with SOP, ST-RC-0004.
- 1.3.2 Water preparation is contained within this SOP.

1.4 The requested limits, minimum detection amounts and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This SOP describes the method for separation of Thorium, Plutonium 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. Tracers are used to correct for chemical recovery and correct results to improve precision and accuracy.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common terms and data qualififers.

3.2 Tracer - A known amount of ^{232}Uranium, ^{229}Thorium, ^{242}Plutonium, (or ^{236}Plutonium), 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.

4.0 INTERFERENCES

4.1 Actinides with unresolvable alpha energies, such as Americium-241 and Plutonium-238, must be chemically separated to enable measurement of the target actinide(s). This method separates these isotopes effectively.

4.2 Samples that are high in carbonates and phosphates, as indicated by a violent and vigorous reaction during the initial phases of digestion, need to be loaded in a minimum of 40 mL of load solution. The increased amount of load increases the amount of aluminum nitrate that the samples are exposed to. The extra aluminum nitrate helps to bind phosphates which interfere with thorium uptake.

4.3 Neptunium-237 can interfere with the Plutonium-242. This interference can be avoided by increasing the normality of the hydrochloric acid rinse and increasing the concentration of the titanium trichloride eluant.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of

the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.1.1 None.

5.2 PRIMARY MATERIALS USED

5.2.1 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 Limit[2]	Signs and symptoms of exposure
Ammonium Hydroxide	Poison Corrosive	50 ppm (TWA)	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.
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.
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.
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.
Lead nitrate	Poison Oxidizer	0.05 mg/m^3 (TWA)	Inhalation of lead can produce local irritation of bronchia and lungs with acute exposure causing a metallic taste in the mouth and chest and abdominal pain. Ingestion symptoms can include abdominal pain and spasms, nausea, vomiting, and headache. Absorption through skin can occur causing symptoms similar to ingestion. Skin contact may cause local irritation, redness and pain. Absorption can also occur through eye tissue.
Nitric Acid	Corrosive Poison Oxidizer	2 ppm (TWA) 4 ppm (STEL)	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.
Sulfuric Acid	Corrosive Poison Cancer Hazard	1 mg/m^3 (TWA)	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.

1 – Always add acid to water to prevent violent reactions.
2 – Exposure limit refers to the OSHA regulatory exposure limit.
TWA – Time Weighted Average
STEL – Short term exposure limit
Ceiling – At no time should this exposure limit be exceeded.

6.0 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 Pipettes, glass or plastic, disposable

6.6 Pipettes, mechanical

6.7 Fume hood

6.8 Hotplate

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.

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 DI Water, obtained from the Milli-Q unit.

7.3 Aluminum Nitrate, solid.

7.4 Ammonium hydrogen phosphate (3.2M)

7.4.1 Dissolve 104 g of $(NH_4)_2HPO_4$ in 200 mL of water, heat gently to dissolve, and dilute to 250 mL with water.

7.5 Ammonium hydroxide (NH4OH), Reagent.

7.6 Ammonium Thiocyanate, crystals

7.6.1 Dissolve 7.6g of ammonium thiocyanate crystals in 90mL of DI water. Dilute to 100mL.

7.7 L (+) Ascorbic Acid, reagent powder.

7.7.1 L (+) Ascorbic Acid solution, 2.5 g dissolved in 10 mL of DI water.

7.8 Bromocresol Purple indicator solution
7.8.1 Dissolve 0.20 g of Bromocresol Purple (520.24 F.W.) in 250 mL of water, add one mL of concentrated Ammonium Hydroxide.

7.9 Calcium nitrate (1.25M)
7.9.1 Dissolve 51 g of $Ca(NO_3)_2$ in 100 mL of water and dilute to 250 mL with water.

7.10 Hydrochloric acid (12M) - concentrated HCl (sp gr 1.19).
7.10.1 Hydrochloric acid (9M) - Add 1500 mL of concentrated HCl (sp gr 1.19) to 200 mL of water and dilute to 2 liters.
7.10.2 Hydrochloric acid (6M) - Add 1000 mL of concentrated HCl (sp gr 1.19) to 200 mL of water and dilute to 2 liters
7.10.3 Hydrochloric acid (1M) - Add 167 mL of concentrated HCl (sp gr 1.19) to 200 mL of water and dilute to 2 liters

7.11 Hydrochloric acid (5M) - 0.05M oxalic acid solution
7.11.1 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 stir plate until oxalic is completely dissolved

7.12 Lead Nitrate (Reagent, crystals).
7.12.1 Lead Nitrate 1 % wt/vol. solution. Dissolve 1 g of lead nitrate crystals in 100 mL of DI water.

7.13 Nitric acid (16M) - concentrated HNO_3 (sp gr 1.42).
7.13.1 Nitric acid (3M) – Add 375mL of concentrated nitric acid to 1500mL of DI water and dilute to 2L.

7.14 Load solution [Nitric acid (3 M) - aluminum nitrate (1 M)]
7.14.1 Weigh 1500 g $Al(NO_3)_3 \cdot 9H_2O$ in a 4 liter beaker. Add 800 mL of water(first) and 764 mL of concentrated nitric acid. Dilute to 4 L with water. Add stir bar, cover with watch glass and place on stir plate until aluminum nitrate is dissolved.

7.15 Potassium Hydroxide, KOH

7.16 Potassium Sulfate (Reagent Crystals).

7.17 Oxalic Acid, reagent, crystals.

7.18 Sodium Nitrite, $NaNO_2$ reagent crystals.
7.18.1 Sodium Nitrite Solution – dissolve 1.0 grams of sodium nitrite crystals in 10 mL of DI water.

7.19 Titanium trichloride, $TiCl_3$, 10% solution, commercially available.

7.20 TEVA Resin - prepacked column, 100-150 micron resin, or 50-100 micron prepacked cartridges.

7.21 UTEVA Resin-prepacked column, 100-150 micron resin, or 50-100 micron prepacked cartridges.

7.22 Plutonium-242 tracer standard, 10-20 dpm/mL (Plutonium-236 can also be used).

7.23 Plutonium-238 and/or Plutonium-239.

7.24 Thorium-229, 10-20 dpm/mL.

7.25 Natural Thorium spike standard (Thorium-232/Thorium-228), approximately 10-20 dpm/mL.

7.26 Thorium-230 spike standard, approximately 10-20 dpm/mL.

7.27 TRM solid reference material.

7.28 Uranium-232 tracer, approximatly 10-20dpm/mL.

7.28.1 Clean uranium free of Th-228 daughter, removed by lead sulfate precipitation, activity verified prior to use. A Th-228 free Uranium-232 standard may be made as descirbed below.

7.28.2 Dilute the appropriate aliquot of stock to about 40mL with DI water.

7.28.3 Add 3 grams of potassium sulfate.

7.28.4 Adjust the pH to 1.5 using narrow range pH strips with either 2M H_2SO_4 or 2M KOH.

7.28.5 While mixing, slowly add 25 mL of 1% $Pb(NO_3)_2$.

7.28.6 Adjust the pH to 1.5 using narrow range pH strips with either 2M H_2SO_4 or 2M KOH.

7.28.7 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.

7.28.8 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.

7.28.9 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.

8.0 SAMPLE COLLECTION, PRESERVATIVES AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica 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 ST-PM-0002.

8.2 Samples may be collected in glass or plastic containers.

8.3 Aqueous samples are preserved with nitric acid to a pH of less than 2.

8.3.1 The pH of aqueous samples are checked upon receipt by sample control, therefore, the pH does not require checking prior to analysis.

8.3.1.1 Aqueous samples acidified upon receipt (designated by label on the bottle) do require checking the pH prior to analysis.

8.4 Solid sample requirements are found in SOP ST-RC-0004, “Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Actinide Analysis”.

9.0 QUALITY CONTROL

9.1 **Batch**

9.1.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents. Where no preparation method exists (e.g. water sample volatile organics, water sample anion analysis) the batch comprises of a maximum of 20 environmental samples which are analyzed together with the same process, lots of reagents and personnel.

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

9.1.3 For this analysis, batch QC consists of a <u>method blank</u> (MB), a <u>Laboratory Control Sample</u> (LCS), and <u>Sample Duplicate</u>. 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 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.1.4 Samples having different QC codes, due to non-standard client specific QC requirements, must be batched separately in the LIMS. A method blank and LCS may be shared across QC codes provided the actual "sample batch" does not exceed 20 environmental samples. Duplicates (and MS/MSD if applicable) must be performed for each separate QC code.

9.2 **Method Blank (MB)**

9.2.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.2.2 A method blank must be prepared with every sample batch.

9.2.3 For Water analyses, the method blank is comprised of DI water with Nitric Acid.

9.2.4 For non-aqueous analyses,the method blank is comprised of 1.25M Calcium Nitrate. See the soil preparation SOP ST-RC-0004.

9.3 **Laboratory Control Sample (LCS)**

9.3.1 An LCS is 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 sample batch.

9.3.3 For Water analyses, the LCS is comprised of DI water with Nitric acid fortified with the isotopes of interest.

9.3.4 For non-aqueous analyses, the LCS is comprised of a TRM sold reference material or 1.25M Calcium Nitrate fortified with the isotopes of interest. See the soil prep SOP ST-RC-0004.

9.3.4.1 For Am, Cm, Pu, Uranium only, use Calcium nitrate

9.3.4.2 For Thorium use the TRM standard.

9.4 **Matrix Spike(MS)/Matrix Spike Duplicate(MSD)**

9.4.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.4.2 MS/MSD samples do not count towards the 20 environmental samples in a sample batch.

9.4.3 MS/MSD samples, when requested, must be performed with every sample batch and every LIMS batch.

9.5 **Sample Duplicate (SD)**

9.5.1 A Sample Duplicate is 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. A NCM is written to document the insufficient volume and utilizing of a LCSD for demonstration of precision.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

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

10.0 CALIBRATION AND STANDARDIZATION

10.1 Balance and pipette calibrations must be checked daily when used. Refer to SOP ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes."

11.0 PROCEDURE

11.1 **For NON-AQUEOUS matrices (soil, oil, biota, etc) see SOP: ST-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 has 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, contact the manager/supervisior.
- 11.2.2 Prepare method blank and LCS using 500ml or 1000ml DI water (match to volume of associated samples).
  - 11.2.2.1 Acidify with nitric acid to a pH < 2.
- 11.2.3 Shake the sample to suspend any residue and to ensure that the sample is homogeneous.
- 11.2.4 Weigh approximately 500 to 100mL (depending on the dection limit) of sample into an appropriate size beaker. Record weight.
  - 11.2.4.1 Aqueous sample aliquot volumes are determined by mass and use an assumed density of 1g/mL.
  - 11.2.4.2 If upon visual inspection a sample is suspected to have a high density (>1.2g/mL, e.g. brine or waste) or a low density (<0.98g/mL, e.g. mixed solvent) the sample density will be measured and the volume determined arithemetically (sample mass divided by density equals volume).
- 11.2.5 Add appropriate tracers or standards. Generally 10 – 20 dpm of each of the Thorium, Uranium and Plutonium tracers are added
  - 11.2.5.1 Spike LCS and MS (if applicable) with isotopes of interest.

11.3 Evaporation (Alternative option to Calcium Phosphate precipitation):

- 11.3.1 This option may be used when large sample volumes are needed to achieve low level reporting limits.
  - 11.3.1.1 Consult Manager/Supervisor to determine when this option should be used.
- 11.3.2 Evaporate sample on a hot plate to less than 50 mL and transfer to a 100 mL beaker.
  - 11.3.2.1 Note: For some water samples, calcium sulfate formation may occur during evaporation. If this occurs, use the calcium phosphate precipitation option in step 11.4
- 11.3.3 Gently evaporate the sample to dryness and redissolve in approximately 5 mL of concentrated HNO_3 (sp gr 1.42). Repeat step two more times, evaporate to dryness and proceed to step 11.5.

11.4 Calcium phosphate precipitation:

- 11.4.1 Add 0.5 mL of 1.25M $Ca(NO_3)_2$ to each beaker.
- 11.4.2 Add 0.200 mL of 3.2 M $(NH_4)_2HPO_4$ solution to each beaker
- 11.4.3 Add 3-5 drops Bromocresol Purple indicator to each beaker.
- 11.4.4 Stir using the bulb of a transfer pipette and place beaker on a hot plate.
- 11.4.5 Allow the samples to heat to near boiling approximately 30 minutes.
- 11.4.6 Once the samples reach near boiling, turn the heat down to medium.
- 11.4.7 While stirring, add enough concentrated NH_4OH with a squirt bottle to reach the bromocresol purple indicator end point and form $Ca_3(PO_4)_2$ precipitate.
- 11.4.8 Allow the sample to heat for another 20-30 minutes.
- 11.4.9 Remove from the hot plate, allow sample to cool and precipitate to settle.
- 11.4.10 Decant.
- 11.4.11 Transfer the precipitate to a centrifuge tube and centrifuge the precipitate for approximately 5 minutes at 2000 rpm.
- 11.4.12 Decant supernatant and discard to waste.
- 11.4.13 Proceed to section 11.5

11.5 Thorium/Plutonium/Uranium Separation using Eichrom resins.

11.5.1 Dissolve calcium phosphate precipitate, soil dissolution residue or evaporated water sample with 15 mL of Load solution. Vortex the sample.

11.5.1.1 **For waters**:

11.5.1.1.1 A additional 5 mL load solution aliqouts may be necessary to dissolve the sample residue. Do not use more than 30 mL of load solution.

NOTE: Samples that are high in carbonates and phosphates, as indicated by a violent and /or vigorous reaction during the initial phases of digestion, need to be loaded in a minimum of 40 mL of load solution.

11.5.1.1.2 If particles are observed or the solution is cloudy, centrifuge the sample at approximately 2000 rpm for 5 minutes.

NOTE: The use of filtration is also permitted, e.g. syringe filter if the solution is still cloudy.

11.5.2 For each sample dissolved in load solution, place a UTEVA resin cartridge in the vacuum box. Lock a TEVA Resin cartridge onto the top of the UTEVA cartridge. Attach a plastic syringe funnel to the top of the TEVA cartidge.

11.5.2.1 **If samples do not require Uranium analysis, the UTEVA cartridge is omitted.**

11.5.3 Place a waste collection reservoir inside the vacuum box to catch the column effluent.

11.5.4 Just prior to loading the sample condition the resin:

11.5.4.1 Turn on the vacuum pump.

11.5.4.2 Add 5 mL of 3M HNO_3 into each funnel.

11.5.4.3 Allow the solution to be pulled through the columns by adjusting the flow rate on top of the vacuum box.

11.5.4.4 The flow rate should be approximately 3 mL per minute. Discard effluent to waste.

NOTE: Approximately 20 drops equals 1 mL. Use the valve to adjust the flow for each individual sample. Adjust the flow for each solution added.

11.5.5 For samples requiring Plutonium analysis (If samples do not require Plutonium, proceed to step 11.5.8):

11.5.5.1 Add 1 drop of Ammonium Thiocyanate and 1 mL ascorbic acid solution to the sample load solution in the centrifuge tube and heat in hot water bath for approximately 5 minutes.

11.5.5.1.1 After heating, if samples are still red in color (indication of iron present in sample), add ascorbic acid drop wise unitl red color disappears. Heat in hot bath for 3 minutes.

11.5.5.2 Add 1mL of $NaNO_2$ solution to the sample load solution in the centrifuge tube and heat in hot water bath for approximately 5 minutes.

11.5.5.3 Remove samples from hot water bath and let cool in cold water bath until samples are at or slightly below room temperature.

11.5.6 Transfer each sample load solution into the appropriate TEVA/UTEVA Resin cartridge funnel. Allow to drain. Adjust the flow rate to approximately 1 mL per minute.

NOTE: the TEVA and the UTEVA cartridge can turn blueish green as the load solution drains through it.

11.5.7 Rinse the funnel with 20 mL of 3M HNO_3 and allow to drain. Adjust flow to approximately 3 mL per minute.

11.5.8 Separate TEVA cartridge from UTEVA cartridge. Place new syringe on the UTEVA cartridge.

NOTE: If Uranium analysis is not requested, the UTEVA cartridge is omitted.

11.5.9 Thorium Elution

11.5.9.1 Place a new clean labeled centrifuge tube in the rack beneath the TEVA cartridge.

11.5.9.2 Add 20 mL of 9M HCl into each cartridge and collect eluant. Adjust flow to approximately 1 mL per minute.

11.5.9.3 Add 5 mL of 6M HCl into each funnel and collect in the same centrifuge tube as in the previous step. This 6M HCl rinse will strip any residual traces of Thorium from the cartridge. Adjust flow to approximately 1 mL per minute.

11.5.9.4 For Thorium analysis:

11.5.9.4.1 Transfer the Thorium HCl solution to a clean labeled beaker (save centrifuge tubes) and evaporate to near dryness.

11.5.9.4.2 Add 5-10mL of DI water in beaker and let sit for 15 minutes.

11.5.9.4.3 Transfer sample back to original centrifuge tube.

11.5.9.4.4 **To co-precipitate the Thorium**, proceed to ST-RC-0100, "Actinide Coprecipitation."

11.5.10 Plutonium Elution (**Thorium Elution step must be completed before this step- even if Thorium is not requested)**

11.5.10.1 Place a clean, labeled 50 mL centrifuge tube below each TEVA cartridge.

11.5.10.2 **For samples not suspected of containing Neptunium:**

11.5.10.2.1 **Plutonium Elution:**

11.5.10.2.1.1 Mix the HCl and $TiCl_3$ after closing the flow control valve by adding 10 mL of the 1M HCl to the column and pipetting 0.25 mL of the $TiCl_3$ into the column.

11.5.10.2.1.2 Add 10 mL of 1M HCl.

11.5.10.2.1.3 Adjust the flow control valve so that the flow is approximately 1 mL per minute.

11.5.10.2.2 **Collect the plutonium eluant,** and proceed to ST-RC-0100, "Actinide Coprecipitation."

11.5.10.3 **For samples suspected of containing Neptunium:**

11.5.10.3.1 **Plutonium Elution:**

11.5.10.3.1.1 Mix the HCl and $TiCl_3$ after closing the flow control valve by adding 10 mL of 9M HCl to the column and pipetting 0.4 mL of the $TiCl_3$ into the column.

11.5.10.3.1.2 Add 10 mL of 9M HCl.

11.5.10.3.1.3 Adjust the flow control valve so that the flow is approximately 1 mL per minute.

11.5.10.3.1.4 **Collect the plutonium eluant.**

11.5.10.3.1.5 **To co-precipitate the Plutonium** proceed to ST-RC-0100, "Actinide Coprecipitation."

11.5.11 Uranium Elution (if requested)

11.5.11.1 Place a waste 50 mL centrifuge tube below each UTEVA cartridge.

11.5.11.2 Add 5 mL of 3M HNO_3 into each cartridge, adjust flow to approximately 3 mL per minute. Dispose to waste.

11.5.11.3 Add 5 mL of 9M HCl into each UTEVA cartridge and allow to drain. Adjust the flow rate to 1mL per/minute.

11.5.11.4 Discard this rinse.
NOTE: This rinse converts the resin to the chloride system. Some Neptunium may be removed here.

11.5.12 Add 20 mL of 5M HCl- 0.05M oxalic acid into each cartridge. Adjust flow rate to 1mL per/minute and allow to drain. Discard this rinse.
Note: This rinse removes neptunium and thorium from the cartridge. The 9M HCl and the oxalic acid remove any residual ferrous ion.

11.5.13 Ensure that clean, labeled tubes are placed in the tube rack under the appropriate cartridge.

11.5.14 Add 15 mL of 1M HCl into each cartridge to strip the **Uranium**. Adjust flow rate to 1mL per/minute and allow to drain.

11.5.15 **To co-precipitate the Uranium**, proceed to ST-RC-0100, "Actinide Coprecipitation."

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Density: Sample weight divided by the volume of said sample weight.

12.1.1 D=M/V D=Density, M=Mass and V=Volume

12.2 Commonly used calculations (e.g. % recovery and RPD) and standard instrument software calculations are given in the TestAmerica St. Louis ST-QAM. Specific analysis calculations are given in the applicable analytical SOP.

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

13.1 Data assessment does not pertain to this sample preparation procedure.

13.1 Samples requiring re-preparation are submitted to the preparation lab with a NCM detailing the issue. The NCM process is described in SOP: ST-QA-0036. Specific information is given in the applicable analysis SOP.

14.0 METHOD PERFORMANCE

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in the associated analytical SOP

14.2 Demonstration of Capability

1.2.1. Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

1.3.1. The manager/supervisor 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.

1.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 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

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

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

16.2.1.2 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.

17.0 REFERENCES

17.1 Eichrom Technologies, Inc Analytical Procedure "ACW13 VBS Thorium, Plutonium and Uranium in Water (with Vacuum Box System". January 2003

17.2 Eichrom Technologies, Inc Analytical Procedure "ACW01 Uranium and Thorium, in Water". April, 2001

17.3 TestAmerica Quality Assurance Manual (ST-QAM), current revision

17.4 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.5 Associated SOPs (current revisions)

17.5.1 ST-PM-0002, Sample Receipt and Chain of Custody
17.5.2 ST-QA-0002, Standards and Reagent Preparation
17.5.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.5.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.5.5 ST-RC-0004, Preparation of Soil Samples for Radiochemical Analysis
17.5.6 ST-RC-0100, Actinide Coprecipitation
17.5.7 ST-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
17.5.8 ST-RD-0210, Daily Operations of an Alpha Spectroscopy System (using AlphaVision Software)

18.0 CLARIFICATIONS AND MODIFICATIONS TO REFERENCED METHODS

18.1 Ascorbic acid is used in place of ferrous sulfate to do the Plutonium valance adjustment.

18.1.1 We use ascorbic acid which provides the oxidation without introducing any iron, which is a known interference.

18.2 A 20ml rinse is done instead of a 5ml to ensure that all Uranium is rinsed from the cartridge.

18.3 The rinse steps are eliminated in the Plutonium separation step due to the larger rinse prior to the separation of the cartridge.

18.4 Plutonium is eluted with 20ml 1M HCL to 25ml $TiCl_3$ which serves to oxidize the Plutonium and strip it from the column in place of 25ml 0f 0.05M HNO3/0.05M HF/0.02M $TiCl_3$.

18.5 Bromocresol purple indicator is used throughout, where as the Eichrome method switches from Bromocresol purple to phenolphthalien indicator in mid-process.

19.0 CHANGES TO PREVIOUS REVISION

19.1 Updated to TestAmerica format.
19.2 Updated standards and reagents in section 7.0 and removed Sulfuric Acid from list.
19.3 Updated procedure in section 11.0 regarding centrifuge time length and removed "Pipet" replacing it with "Add" throughout the section.
19.4 Rev. 13:
- 19.4.1 Updated the section 11.4 in reference to the amount of time sample must remain in centrifuge.

19.5 Rev. 14:
- 19.5.1 Added Hydrochloric Acid to Cresol Red indicator solution in section 7.9.
- 19.5.2 Added a note to section 11.5.1.1.3 stipulating that samples with 5mL or more of gel/sediment need to go though a clean-up process.
- 19.5.3 Added section 11.6 for Actinide Extraction from Gel/Sediment formation.

19.6 Rev. 15:
- 19.6.1 Updated the summary of method in section 2.0.
- 19.6.2 Updated equipment used in section 6.0.
- 19.6.3 Updated reagents & standards throughout section 7.0.
- 19.6.4 Removed the use of Cresol Red Indicator solution from section 7.0.
- 19.6.5 Updated section 8.0 regarding storage, collection, pH testing and preservatives of samples.
- 19.6.6 Added uranium to the list of analytes used for laboratory control samples in section 9.3.
- 19.6.7 Updated section 11.0 regarding water sample prep, calcium phosphate precipitation and thorium/plutonium/uranium separation using Eichrom resins.
- 19.6.8 Removed instruction for actinide extraction from gel/sediment formation in section 11.0.
- 19.6.9 Updated calculation for sample density in section 12.0

19.7 Rev. 16:
- 19.7.1 Section 7, removed reference to NIST traceable
- 19.7.2 Section 8, removed holding time requirement
- 19.7.3 Section 9, MB composition updated and LCS requirements updated.
- 19.7.4 Section 11.5.5 updated
- 19.7.5 Section 15 updated

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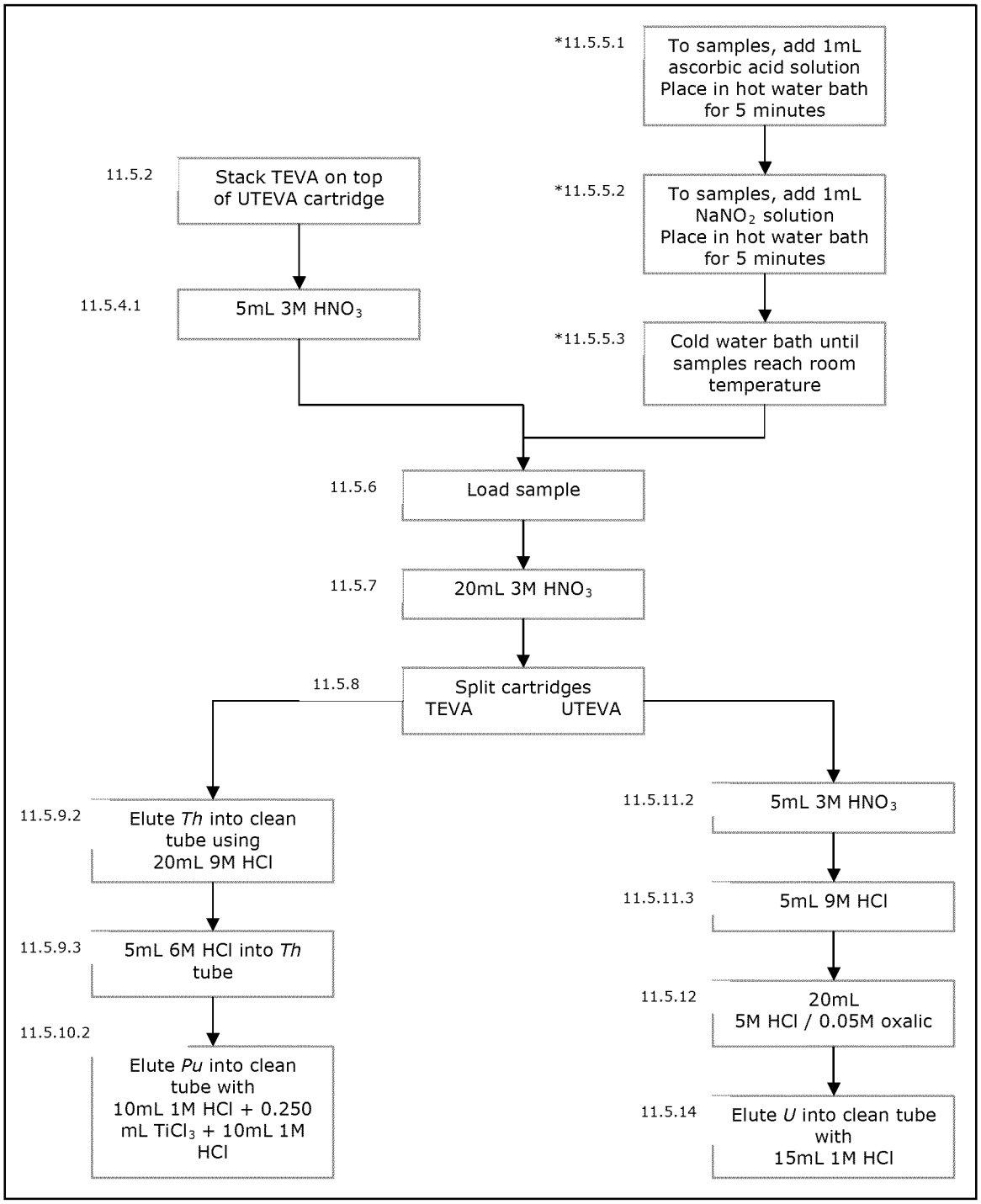
Sequential Thorium, Plutonium and Uranium via TEVA/UTEVA

All rinses should flow at 1mL/minute (only 3M HNO_3 may be done at 3mL/minute)
*only necessary when analyzing for *Plutonium*



Title: GAMMAVISION® ANALYSIS

Approvals (Signature/Date):

Chris Hough — 4/17/14 Chris Hough — Date Radiochemistry Manager	Michael Ridenhower — 4-18-14 Michael Ridenhower — Date Health & Safety Manager / Coordinator
4-15-14 Tony L. Byrd — Date Quality Assurance Specialist	Elaine Wild — 4/15/14 Elaine Wild — Date Laboratory Director

This SOP was previously identified as SOP No. ST-RD-0102 Rev. 11

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1.0 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 a similar matrix may 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 requested limits (**RL**), minimum detectable amount (**MDA**) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 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.

3.0 DEFINITIONS

3.1 See the TestAmerica Quality Assurance Manual (ST-QAM) for a glossary of common laboratory terms and data reporting qualifiers.

4.0 INTERFERENCES

4.1 Germanium spectrometry has 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.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

6.0 EQUIPMENT AND SUPPLIES

6.1 Germanium spectroscopy system utilizing a computer based data acquisition system (GammaVision®-32).

6.2 GammaVision®-32 (know as GammaVision) is a comprehensive, all-in-one package, for the analysis of gamma-ray spectra acquired with HPGe detectors.

6.3 Global Value software is an optimization tool for automation, custom reporting, quality assurance and data management (GammaVision productivity add-on software).

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002.

7.2 Commercially prepared mixed gamma standards in reproducible geometries, with all appropriate NIST Source Certificate information.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica 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 ST-PM-0002. Samples may be collected in glass or plastic containers.

8.2 Aqueous samples are preserved with nitric acid to a pH of less than 2.

9.0 QUALITY CONTROL

9.1 See actinide preparation SOPs for additional informati on regarding QC types, frequency and preparation.

9.2 **Batch**

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

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

9.2.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample , and Sample Duplicate.

9.3 **Method Blank (MB)**

9.3.1 A method blank must be counted with every sample batch.

9.3.1.1 For soils, a method blank is sodium sulfate filled in the specified geometry.

9.3.1.2 For waters, a method blank is DI water filled in the specified geometry.

9.3.1.3 For filters, a method blank is a blank petri dish.

9.4 **Laboratory Control Sample (LCS)**

9.4.1 An LCS must be counted with every sample batch.

9.4.1.1 For water, a purchased mixed nuclide source in the specified geometry.

9.4.1.2 For soil, a purchased mixed nuclide source in the specified geometry.

9.4.1.3 For filters, a purchased mixed nuclide source in a petri dish.

9.5 **Sample Duplicate**

9.5.1 A Sample Duplicate is a recounted field sample to demonstrate instrument precision, since there is no sample preparation (required to count on a different detector than the sample).

9.5.1.1 If requested, the laboratory may perform a Sample Duplicate which is an additional aliquot of a field sample.

9.6 **Procedural Variations/ Nonconformance and Corrective Action**

9.6.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

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

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10.0 CALIBRATION AND STANDARDIZATION

10.1 There are two types of Calibrations performed for Gamma: Energy and Efficiency

10.1.1 Energy Calibrations

10.1.1.1 Frequency: the energy calibration is performed once per detector. The source is not geometry specific.

10.1.1.2 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include significant changes in instrument operating parameters, and major instrument maintenance (e.g. replacing the detector)

10.1.1.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

10.1.1.4 Range: the energy range, is 46.54 to 1836.1 keV for air filter and solid.

10.1.1.5 Criteria:

10.1.1.5.1 The curve should have, at minimum, eight calibration points used to determine the energy relationship of the calibration.

10.1.1.5.2 The energy difference (delta Δ) should be within 0.05% for all calibration points or within 0.2 keV for the calibration points.

10.1.1.5.3 The FWHM must be less than 3.0 keV at 1332 keV.

10.1.1.5.4 FWHM difference (delta Δ) should be within 8% for all calibration points.

10.1.2 Efficiency Calibrations

10.1.2.1 Frequency: the efficiency calibration is performed per geometry.

10.1.2.2 A new calibration curve must be generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include significant changes in instrument operating parameters, and major instrument maintenance (e.g. replacing the detector)

10.1.2.3 Except in specific instances, it is NOT acceptable to remove points from a calibration curve for the purpose of meeting criteria. Refer to the TestAmerica Policy CA-T-P-0002, Selection of Calibration Points

10.1.2.4 Range: the energy range of the calibration is dependent on the matrix that is calibrated. i.e. 46.54 to 1836.1 keV for air filter and solid, 59.5 keV to 1836.1 keV for water.

10.1.2.5 Criteria:

10.1.2.5.1 The curve should have at least eight points to determine the efficiency

10.1.2.5.2 The calibration source must have radionuclides that "bracket" the intended range of calibration

10.1.2.5.3 A minimum of 10,000 counts will be accumulated for each data point

10.1.2.5.4 The efficiency difference (delta Δ) should be within 8% of the true value for each point

10.2 Initial Calibration Verification (ICV) [Frequency: Once]

10.2.1 An initial calibration verification standard must be a different standard source than the one used for the initial calibration.

10.2.1.1 The ICV check does not include short half-life nuclides which may exist in the purchased standard. At a minimum, the ICV will always contain Am-241 (low), Cs-137 (medium) and Co-60 (high).

10.2.2 An ICV must be performed with every initial calibration.

10.2.3 The ICV percent recovery must be within ± 10% of the true value for each nuclide.

10.2.4 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ICV, or analysis of a

different ICV). Any decision to proceed with analysis of samples when the ICV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.3 Annual Calibration Verification (ACV) [Frequency: Annually] not geometry specific

10.3.1 An annual calibration verification check will be performed on each detector.

10.3.2 Two verification standards (second source independent from the initial calibration source) will be used for the verification checks.

10.3.2.1 One from a water source that surrounds the detector

10.3.2.2 One from a solid source that rests on top of the detector

10.3.3 The checks will include isotopes from the low (Am-241), medium (Cs-137) and high (Co-60) energy range.

10.3.4 The verification can be accomplished by using LCS samples counted on each detector.

10.3.5 The ACV percent recovery must be within ± 10% of the true value for each nuclide.

10.3.6 Not meeting this requirement may be indicative of serious system malfunction or inaccuracies in the standards used for the initial calibration curve or ICV standard. Corrective action must be taken (including reanalysis of the ACV, or analysis of a different ACV). Any decision to proceed with analysis of samples when the ACV is out-of-control must be taken with great care and in consultation with the QA department and the laboratory director. Any such action must be documented in an NCM.

10.4 Daily Checks

10.4.1 The detector **background** shall be checked each day that the germanium spectroscopy system is used. Limits are set at 2 sigma and 3 sigma.

10.4.1.1 <u>Bkgd Countrate (background count rate for entire spectrum)</u>

Tolerance (warning)	=	± 2 σ
Control (out)	=	± 3 σ

10.4.2 The instrument **Channel**, **Energy**, **FWHM** (resolution) and **Activity Difference** (efficiency) for a detector shall be checked each day the germanium spectroscopy system is used (using a check source that is non-geometry specific).

10.4.2.1 **Channel** – (low and high energy) is monitored for channel alignment. Limits are set around the target Channel

10.4.2.1.1	QA-60	<u>Low Energy</u>		
		Tolerance (warning)	=	± 1 channel
		Control (out)	=	± 2 channels
10.4.2.1.2	QA-1332	<u>High Energy</u>		
		Tolerance (warning)	=	± 2 channels
		Control (out)	=	± 3 channels

10.4.2.2 **Energy** – (low and high energy) is monitored for energy alignment. Limits are set around a target energy

10.4.2.2.1	QA-60	<u>Low Energy</u>		
		Tolerance (warning)	=	± 0.25 keV
		Control (out)	=	± 0.50 keV
10.4.2.2.2	QA-1332	<u>High Energy</u>		
		Tolerance (warning)	=	± 0.5 keV
		Control (out)	=	± 0.75 keV

10.4.2.3 Full-Width at the Half Maximum (**FWHM**) - (low, mid, and high energy) is monitored for peak shape There are no limits compared to a target FWHM. There are no lower limits (–) set for FWHM.

10.4.2.3.1	QA-60	<u>Low Energy</u>		
		Tolerance (warning)	=	+ 1.1
		Control (out)	=	+ 1.2
10.4.2.3.2	QA-662	<u>Mid Energy</u>		
		Tolerance (warning)	=	+ 1.7
		Control (out)	=	+ 1.8
10.4.2.3.3	QA-1332	<u>High Energy</u>		

Tolerance (warning)	=	+ 2.2
Control (out)	=	+ 2.3

10.4.2.4 **Activity Difference** (low, mid, and high energy) – is monitored to check the percent difference between the source activity and the reported activity. Limits are set around the target activity.

10.4.2.4.1 QA-60/662/1332 Low/Mid/High Energy

Tolerance (warning)	=	± 4
Control (out)	=	± 5

10.4.3 If the daily check is outside of the control limits, it may be recounted or tagged out with red tag (or with an NCM). The Daily QC check may only be recounted once without corrective action.

10.4.3.1 If the out of control parameter is found acceptable for the rerun, the instrument can be used for the analysis of samples. ***Note***: *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.*

10.4.3.2 If the instrument fails to meet the acceptance criteria for the rerun, the instrument must be declared "Out of Service". The detector/instrument must be "tagged out". (See ST-QA-0036 for NCM details regarding tagging out of service).

10.4.3.3 If the QC check fails the following day for the same detector for the same specific parameter as the day before, the instrument must be declared "Out of Service". The detector/instrument must be "tagged out" until the detector can be evaluated and/or maintenance can be performed.

10.4.3.4 The analyst may want to:

10.4.3.4.1 Check the expiration date of the radioactive standard to confirm the material is current, for the isotopes being utilized.

10.4.3.4.2 Check source positioning and all instrument settings.

10.4.3.4.3 Check all cables for any apparent damage and confirm that all cables are routed to proper connectors and are in good working order.

10.4.3.4.4 The instrument may be returned to service once the malfunction has been corrected and the above acceptance criteria have been met. Corrective actions must be noted in the instrument maintenance log.

10.4.3.4.5 If a parameter has two successive values in the warning 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.

10.5 Background

10.5.1 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.

10.5.1.1 Background count time is 12 hours.

10.5.1.1.1 If a client project requires a longer count time, then the background must be performed at the longer time before initiating analysis.

10.5.1.1.2 After review of the monthly background, the analyst will mark each detector complete on the "Monthly Background Complete" sheet located on each gamma cave (see attachment 2)

10.5.1.2 Monthly Background limits are set at 2 sigma and 3 sigma.

10.5.1.2.1 Bkgd Countrate (background count rate for entire spectrum)

Tolerance (warning)	=	± 2 σ
Control (out)	=	± 3 σ

10.6 Calibration Software Handling

10.6.1 Gamma Detector System Energy and Shape Calibration

10.6.1.1 Acquire a spectrum from a calibration standard in the manual mode for an appropriate duration. Save the spectrum to the path "C:\User\Cal\Spectra\DetX\OriginalCountfileName.spc" where:

10.6.1.1.1 X = Detector Number
10.6.1.1.2 Analysis method
10.6.1.1.3 Select library
10.6.1.1.4 Enter correct sample data.
10.6.1.1.5 Enter correct conversion time.

10.6.1.2 Close all detectors windows in the current instance of gamma vision, then recall the appropriate calibration spectrum into the buffer window.

10.6.1.3 Select the menu "Analyze\Setting\Sample type…."

10.6.1.4 Select the browse button next to the "File" field and open the file. Click the "OK" button of the window to close it.

10.6.1.5 Recall the application Calibration File from the menu "Calibration \Recall Calibration…"

10.6.1.6 Select the menu "Calibrate\Calibration wizard…"

10.6.1.7 Select the option to create new energy calibrations. Select the next button.

10.6.1.8 On the energy calibration wizard page, select the file "DET_EnergyStandardMix Lib" or appropriate library for mixed gamma used the browser button if desired. Select the next button.

10.6.1.9 Select the next button to perform the energy, FWHM.

10.6.1.10 Select the edit energy button to review the energy.

10.6.1.10.1 Close the energy calibration sidebar window.

10.6.1.11 Select the save calibration button and save the calibration to "Cal\Energy\X_Energy.clb" where X is the detector.

10.6.1.12 Enter the calibration description in the format "X_ENERGY_GEOMETRY" where X is the detector number and Geometry is an appropriate geometry description when prompted. Select the Finish button to close the calibration wizard.

10.6.1.13 Print the calibration report from the menu "Calibrate \print calibration.

10.6.2 Gamma Detector System Efficiency Calibration

10.6.2.1 Acquire a spectrum from a calibration standard in the manual mode for an appropriate duration. Save the spectrum to the path "C:\User\Cal\Spectra\DetX\OriginalCountfileName.spc" where:

10.6.2.1.1 X = Detector Number
10.6.2.1.2 Analysis method
10.6.2.1.3 Select library
10.6.2.1.4 Enter correct sample data.
10.6.2.1.5 Enter correct conversion time.

10.6.2.2 Close all detector windows in the current instance of Gamma Vision, then recall the appropriate calibration spectrum into the buffer window.

10.6.2.3 Select the menu "Analyze\Setting\Sample Type"

10.6.2.4 Select the browse button next to the "File", field and open the file. Click the "OK" button at the bottom of the window to close it.

10.6.2.5 Recall the applicable calibration file from the menu "Calibration\Recall Calibration" (if the geometry file currently exists)

10.6.2.6 Select the menu "Calibrate\Calibration Wizard"

10.6.2.7 Select the option to create new energy and efficiency calibration. Select next button.

10.6.2.8 On the Energy Calibration Wizard page select the file "EnergyStandardMix Lib" or appropriate library for mixed gamma used the browser button if desired. Select the Next button.

10.6.2.9 On the Efficiency Calibration Wizard page, select library file, "DET_EfficiencyCalibration.Lib" for mixed gamma sources.

10.6.2.10 On the Efficiency Calibration Wizard page, select the appropriate Certification file from the directory.

10.6.2.11 Select the next button to perform the energy FWHM and efficiency calibration.

10.6.2.12 Select the Edit Energy button to review the energy and FWHM Calibration.

10.6.2.12.1 Close the Efficiency Calibration side window.

10.6.2.13 Select the save calibration button and save the calibration to Cal\X_Geometry.clb" where X is the detector and geometry is an appropriate geometry name.

10.6.2.14 Enter the calibration description in the format "x_Geometry_Source number_date counted" where X is the detector number and geometry is an appropriate geometry description when prompted. Select the finish button to close the calibration wizard.

10.6.2.15 Print calibration report from the menu "Calibrate\Print Calibration"

10.6.2.16 Select "Analyze", select "Entire spectrum in memory" and file print.

10.6.2.17 Close the spectrum Buffer window and save the spectrum when prompted.

10.6.3 Detector Long Background Counting

10.6.3.1 Remove any samples from the detector, clean the detector, close the shield lid and start acquisition.

10.6.3.2 Select detector 1 in Global Value Quick Start

10.6.3.3 Select Monthly Background PBC under Automation Groups

10.6.3.4 Select Background PBC Long Count under Automation Jobs.

10.6.3.5 Login using name and password.

10.6.3.6 Select "OK", ensure detector cave is empty.

10.6.3.7 Repeat for each detector which background you would like to start.

10.6.3.8 After the background is complete it will save as a PBC file.

11.0 PROCEDURE

11.1 Calibration Quality Control (**Daily Check**)

11.1.1 Place the calibration quality control sample on the detector, and start acquisition.

11.1.2 Select detector from Global Value Quick Start.

11.1.3 Select Quality Control under Automation Groups.

11.1.4 Select Daily Quality Control Check under Automation Jobs.

11.1.5 Login with user name and password.

11.1.6 Select "OK", ensure source is on detector.

11.1.7 Repeat for each detector.

11.1.8 Record in the instrument run log.

11.2 Background Quality Control (**Daily Background**)

11.2.1 Remove any samples from the detector, and start acquisition.

11.2.2 Select detector global value quick start.

11.2.3 Select quality control under automation groups.

11.2.4 Select daily background check under automation jobs.

11.2.5 Login with username and password.

11.2.6 Select "OK", ensure detector cave is empty.

11.2.7 Repeat for each detector.

11.2.8 Record in the instrument run log.

11.3 Sample Counting
- 11.3.1 Place the sample on the detector.
- 11.3.2 Select detector from GlobalValue quick start.
- 11.3.3 Select analyze samples under automation groups.
- 11.3.4 Select count sample under automation jobs.
- 11.3.5 Login with username and password.
- 11.3.6 Scan sample description from barcode report.
- 11.3.7 Select analysis method, sample type, geometry, library, correct date, count time, continue.
- 11.3.8 Select "OK", ensure sample is on detector.
- 11.3.9 Record in the instrument run log.

12.0 DATA ANALYSIS AND CALCULATIONS

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

12.2 All calculations are performed in GammaVision-32 software; conversions are performed in RadCapture. Calculations are found in ST-QAM.

13.0 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 LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

13.2 Method Blank (**MB**)
- 13.2.1 Acceptance Criteria:
  - 13.2.1.1 No target analytes may be present in the method blank above the requested limit.
  - 13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target anlaytes present above ½ RL), will be noted on the client requirements sheet.
- 13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:
  - 13.2.2.1 <u>Method Blank Contamination</u> – The blank may be re-counted once to confirm the activity (in the same detector). If the re-counted MB activity exceeds the MDA and/or the requested limit, samples with less than 10 times the activity found in the blank are recounted. An NCM is written to document the excursion. Note certain analytes are common laboratory contaminants which require special narrative comment. These compounds are so designated in LIMS.

13.3 Laboratory Control Sample (**LCS**)
- 13.3.1 Acceptance Criteria:
  - 13.3.1.1 All control analytes must be within the specified control limits for accuracy (%Recovery) and precision (RPD).
- 13.3.2 Corrective Action for LCS not meeting acceptance criteria:
  - 13.3.2.1 <u>LCS Spike Recovery excursion (high)</u> – The LCS may be re-counted once to confirm the result. If the re-counted LCS exceeds the control limit, samples that are non-detect may be reported with an NCM.
  - 13.3.2.2 <u>LCS Spike Recovery excursion (low)</u> – The LCS may be re-counted once to confirm the result. <u>If the low recovery is confirmed, the batch is recounted</u>.

13.3.2.3 RPD/RER Duplicate excursion – The LCS is recounted if both RPD and RER exceed criteria.

13.4 Duplicate

13.4.1 Acceptance Criteria:

13.4.1.1 All control analytes must be within the specified control limits for precision (RPD), max. 40% RPD, RER < 1.

13.4.2 Corrective Action for Duplicate not meeting acceptance criteria:

13.4.2.1 RPD/RER Duplicate excursion – The sample is recounted if both RPD and RER exceed criteria.

13.5 Insufficient Sample

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

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

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

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification

14.3.1 The manager/supervisor 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 of capability requirements prior to working independently. See requirements in the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 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 Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

17.0 REFERENCES

17.1 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 28th Edition, method GA-01-R, Gamma Radioassay
17.2 EPA Prescribed Procedures for Measurement of Radioactivity in Drinking Water Method 901.1
17.3 American National Standards Institute (ANSI) Accredited Standards Committee on Radiation Instrumentation, N42; ANSI N42.14-1999, American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides
17.4 Ortec MCB Connections-32, Hardware Property Dialogs Manual, current version
17.5 MAESTRO–32, MCA Emulator, current version
17.6 GammaVision–32, Gamma-Ray Spectrum Analysis and MCA Emulator, current version
17.7 Master library Source: Gerhard Erdtmann, Werner Soyka
17.8 TestAmerica Quality Assurance Manual (ST-QAM), current revision
17.9 TestAmerica Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.
17.10 TestAmerica Policy CA-T-P-0002, Selection of Calibration Points
17.11 Associated SOPs, Current Revision:
- 17.11.1 ST-RC-0003, Drying and Grinding of Soil and Solid Samples
- 17.11.2 ST-RC-0004, Preparation of Soil Samples for Radiochemical Analysis
- 17.11.3 ST-RC-0025, Preparation of Samples for Gamma Spectroscopy
- 17.11.4 ST-QA-0002, Standards and Reagent Preparation
- 17.11.5 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
- 17.11.6 ST-QA-0036, Non-Conformance Memorandum (NCM) Process

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Annual Review, No Changes.
19.2 Revision 8:
- 19.2.1 Increased background count times from 12 to 36 hours in section 10.3.1.1.
- 19.2.2 Updated the procedure for detector long background counting in section 10.5 to reflect new software.
- 19.2.3 Updated daily calibration checks, daily background and sample counting procedures in section 11.0 to reflect new software.

19.3 Revision 9:
- 19.3.1 Replaced quartz sand with sodium sulfate to be used for soil method blanks in section 9.2.
- 19.3.2 Updated section 10.4 regarding instrument daily checks.
- 19.3.3 Updated data assessment and acceptance criteria in section 13.0
- 19.3.4 Updated section 9.0 regarding batch, method blank and laboratory control samples.
- 19.3.5 Updated the calibation points for an internal calibation in section 10.1.
- 19.3.6 Updated the percent recovery regarding the ICV in section 10.2.
- 19.3.7 Updated software storage file name throughout section 10.5.

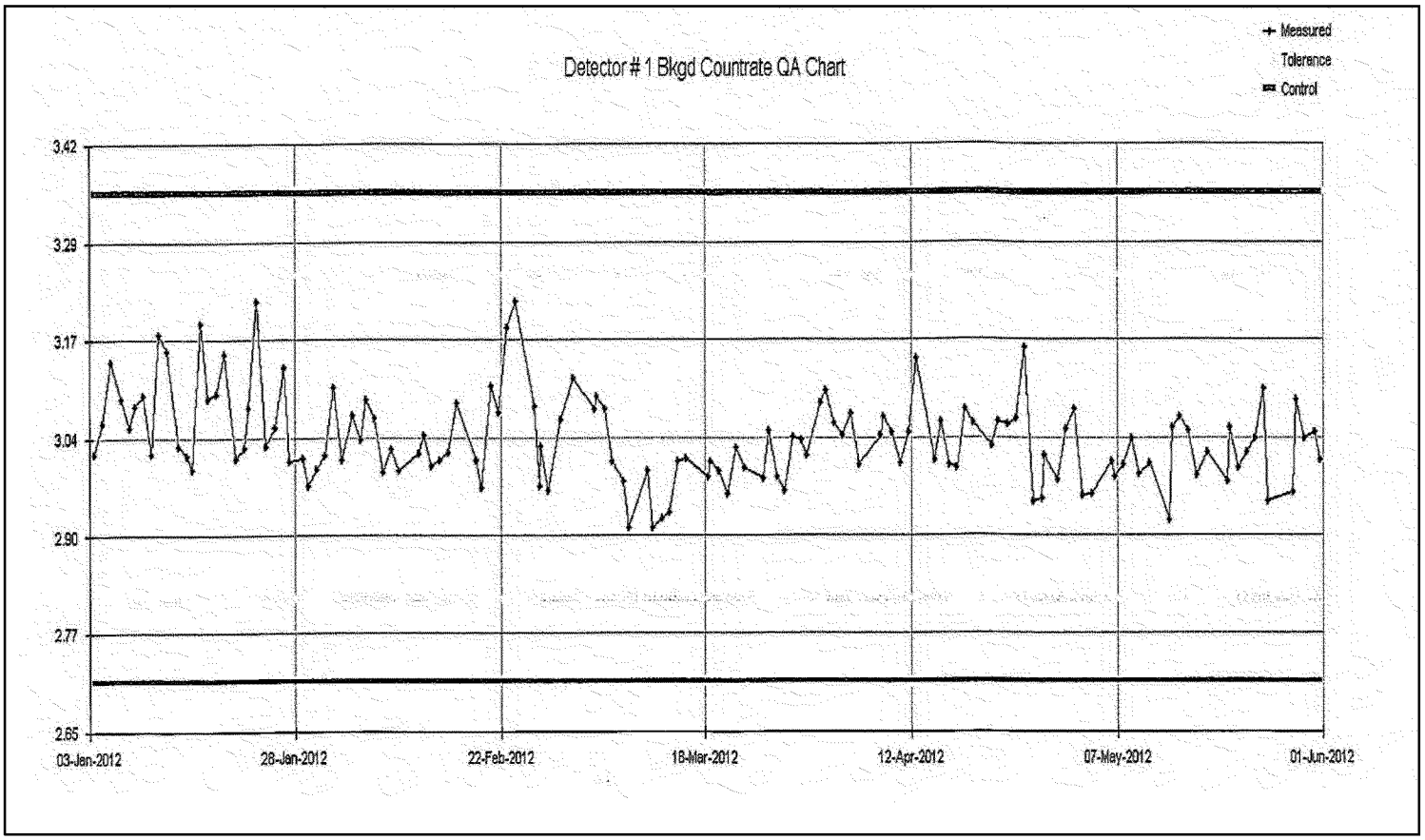
19.4 Revision 10:
- 19.4.1 Updated references to QuantIMS through out
- 19.4.2 Update §10.1
- 19.4.3 Added §10.3 Annual Calibration Verification
- 19.4.4 Updated §10.4: 36 hour background changed to 12 hour and requirement to complete Attachment 2

19.4.5 Added Attachment 2, "Monthly Background Complete" example
19.4.6 Updated §13 references to Clouseau changed to LIMS
19.4.7 Added §17 reference to ANSI 42.14-1999

19.5 Revision 11:
19.5.1 Updated §1.4 with corrected termonolgy
19.5.2 Updated §6.0 software details
19.5.3 Additon/Update §10.0 major change in calibration
19.5.4 Updated §13.0 additional corrective action steps
19.5.5 Updated §15.0 with new verbiage

19.6 Revision 12:
19.6.1 Spelling and grammar corrections made throughout SOP.
19.6.2 Sections 10.2.3 and 10.2.5 had wording changed to common text.
19.6.3 Section 10.4.3.3 was updated to add „for the same specific parameter as the day before" and „until the detector can be evaluated and/or maintenance can be performed.".
19.6.4 Section 10.5.1.2.1 was added to provide limits for monthly backgrounds, which were not previously provided.
19.6.5 Section 13.4.2 had „LCS" changed to „duplicate" since it is the duplicate section and LCS was incorrectly referenced.

Attachment 1



Attachment 2

Example of the form, actual form in use may have slight variations.

2013 Monthly Background Complete

Jan	Reveiwed by_____ Initials & Date
Feb	Reveiwed by_____ Initials & Date
Mar	Reveiwed by_____ Initials & Date
April	Reveiwed by_____ Initials & Date
May	Reveiwed by_____ Initials & Date
June	Reveiwed by_____ Initials & Date
July	Reveiwed by_____ Initials & Date
Aug	Reveiwed by_____ Initials & Date
Sept	Reveiwed by_____ Initials & Date
Oct	Reveiwed by_____ Initials & Date
Nov	Reveiwed by_____ Initials & Date
Dec	Reveiwed by_____ Initials & Date

Title: ALPHA SPECTROSCOPY ANALYSIS [DOE HASL-300 A-01-R]

Approvals (Signature/Date):

Chris Hough — Date: 8/21/13 Radiochemistry Manager	Michael Ridenhower — Date: 8/21/13 Health & Safety Manager / Coordinator
Marti Ward — Date: 8-19-13 Quality Assurance Manager	Elaine Wild — Date: 8/21/13 Laboratory Director

This SOP was previously identified as SOP No. ST-RD-0210 Rev. 10

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1.0 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 This SOP is based on DOE method A-01-R

1.3 This SOP is applicable to both liquid and solid matrices.

1.4 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 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.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for glossary of common terms and data qualifiers.

3.2 Tracer – A known amount of ^{232}U, ^{242}Pu or ^{236}Pu, ^{243}Am, ^{209}Po, ^{244}Cm 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.

3.3 Region of Interest (ROI) – The keV range through which the target isotope peak signal responds.

3.4 keV - (kilo electron Volt) – electron volt is a unit of energy defined as the amount of energy gained (or lost) by the charge of a single electron moved across an electric potential difference of one volt.

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 matrix interference.

3.6 AlphaVision – The Alpha Spectrometer data collection and processing software.

4.0 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 ROI. Th-229 tailing into the Th-230 region of interest is a recognized example. This interference is minimized by maintaining low activities of the Th-229 tracer and monitoring of the separation of the ROI for Th-229 and Th-230. 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 DQO 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

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS
None.

5.3 PRIMARY MATERIALS USED
None.

6.0 EQUIPMENT AND SUPPLIES

6.1 Alpha spectroscopy system utilizing a computer based data acquisition system.

6.1.1 Hardware – Octete Plus and Alpha Ensemble
6.1.2 Software – AlphaVision and Radcapture

7.0 REAGENTS AND STANDARDS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision.

7.2 Commercially prepared alpha standards for the isotopes Th-230, Pu-239 and Am-243.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica 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 ST-PM-0002.

9.0 QUALITY CONTROL

9.1 See actinide preparation SOPs for additional information regarding QC types, frequency and preparation

9.2 **Batch**

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.
9.2.2 Instrument conditions must be the same for all standards, samples and QC samples.
9.2.3 For this analysis, batch QC consists of a Method Blank (MB), a Laboratory Control Sample (LCS), and Sample Duplicate (Dup). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.
9.2.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.3 **Method Blank (MB)**
9.3.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.
9.3.2 A method blank must be prepared with every sample batch.

9.4 **Laboratory Control Sample (LCS)**
9.4.1 An LCS is 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 sample batch.

9.5 **Matrix Spike (MS)**
9.5.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.6 **Sample Duplicate (Dup)**
9.6.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.7 **Procedural Variations/ Nonconformance and Corrective Action**
9.7.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA, see SOP ST-QA-0036 for details regarding the NCM process.
9.7.2 Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the Supervisor and QA, see SOP ST-QA-0036 for details regarding the NCM process.

10.0 INSTRUMENT SETUP, CALIBRATION, AND STANDARDIZATION

10.1 Initial instrument setup is performed when instrument is first installed, when a detector or chamber is changed/replaced, when a chamber is returned from the manufacturer after servicing, or other such circumstances. The following steps should be taken to ensure proper setup. Steps may be accomplished either through hardware knobs/potentiometers or through software settings (depending upon the system hardware/software). See the hardware and/or software manual to determine further detailed instructions:
10.1.1 Set the conversion gain to 1024 channels.
10.1.2 Adjust the coarse and fine gain as well as the offset to adjust the location of the three peaks of the alpha source such that the lower energy peak (Th-230 at 4688 keV) falls into channel 176, the mid-energy peak (Pu-239 at 5155 keV) falls into channel 239, and the higher energy peak (Am-241 at 5486 keV) falls into channel 283. Note that this results in 107 channels between the low energy and high energy peaks (about 7.46 keV/channel with offset of approximately 3375 keV). Ensure each peak is within 2 channels of the desired channel before beginning energy and efficiency calibrations.

10.1.2.1 Gain adjustment:

10.1.2.1.1 Turning the knob counter-clockwise decreases the gain (decrease the value in the fine gain for software adjustment), causing the spectrum peaks to move closer to each other (smaller keV/channel slope) and toward the lower energy.

10.1.2.1.2 Turning the knob clockwise increases the gain (increase the value in the fine gain for software adjustment), causing the spectrum peaks to spread apart (larger keV/channel slope) and toward higher energy.

10.1.2.2 Offset adjustment:

10.1.2.2.1 Increasing the offset moves the peak/spectra toward the the left (lower channel number) without altering the keV/channel (slope).

10.1.2.2.2 Decreasing the offset moves the peak/spectra toward the right (higher channel number) without altering the keV/channel (slope).

10.1.3 Adjust the pulser setting such that the pulser peak is centered at about channel 222 (approximately 5 MeV).

10.2 Calibrations, see 10.6 for procedure.

10.2.1 Energy calibrations shall be performed for the alpha spectroscopy systems yearly, or when a 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. Typical isotopes used are Th-230, Pu-239, and Am-241. Final peak energy positions of all observed isotopes shall be within ± 5 channels (~40 keV) of expected channel/energy (see 10.1.2). The actual energy vs. channel and the equation with the slope is not calculated. Setting the peaks to within 5 channels of expected will ensure calculations utilizing fixed Regions of Interest (ROI) for each isotope will provide accurate results with minimal need for manual adjustment of ROI. Routine pulser checks and continuing calibration verifications (see below) will help control/monitor for drift.

10.2.2 Efficiency calibrations shall be established for the alpha spectroscopy systems yearly, or when a calibration quality control check indicates an unacceptable change in the efficiency calibration parameters.

10.2.2.1 Calibrated efficiency should fall between 20% and 32%. Values outside this range do not constitute a failure. However, if the calibrated efficiency does fall outside this range, an evaluation of the suitability of the detector for use should be performed and documented.

10.2.3 Initial calibration verifications (ICV) shall be performed utilizing an independent second source following the initial calibration.

10.2.3.1 The efficiency of the ICV must fall withing 95%-105% of the initial calibration efficiency value.

10.2.3.2 A second level review will be perfomed before detectors are placed into service and will be noted as acceptable in the electronic monthly maintenance log.

10.3 Continuing Calibration Verification (CCV)

10.3.1 A continuing calibration verification shall be performed on a monthly basis.

10.3.1.1 The Final peak energy positions for the isotopes should fall within ± 5 channels of the expected channel/energy.

10.3.1.2 The efficiency should fall within 95%-105% of the calibrated efficiency.

10.3.1.3 A second level review will be performed before detectors are placed into service and will be noted as accepable in the electronic monthly maintenance log.

10.4 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.

10.5 Daily Checks (Pulsers)

10.5.1 Routine pulser quality control verifications are performed each day of use.

10.5.1.1 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. The limits for pulser centroid and pulser energy will be as below:

10.5.1.1.1 Gross counts must be within 5% of the average (20-point minimum) for each detector.

10.5.1.1.2 The peak resolution (FWHM) must fall within 10-20 keV.

10.5.1.1.3 The pulser centroid must fall within ± 5 channels of the average (20-point minimum) for each detector.

10.5.1.1.4 The pulser energy must fall within ± 40 keV of the average (20-point minimum) for each detector.

10.5.2 Routine calibration, background and pulser quality control parameters using the "Boundry" out-of-range test will be found unacceptable if the value is outside parameter tolerance.

10.5.2.1 The routine quality control check should be rerun to determine the statistical significance of the out of control parameter.

10.5.2.2 If the out of control parameter is found acceptable in the rerun, the investigation will be noted in the instrument maintenance log.

10.5.2.3 Check the integrity of the radioactive standard.

10.5.2.4 Check source positioning and all instrument settings.

10.5.2.5 Check all cables for any apparent damage and to confirm that all cables are routed to proper connectors and are in good working order.

10.5.3 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 (OOS) for the day see Attachment 1 for OOS tag example.

10.5.3.1 This is noted on the Alpa Spec Daily report by marking the report (The report will display FAIL for criteria not met). The detector will be marked out of service with the date and initials of the analyst performing the daily check.

10.5.3.2 If a detector fails three consecutive days for the same criteria, the detector will be taken out of service until the problem is resolved. This is done by clicking on the detector in Alphavision. Right click on the the detector, select detector properties, check the "out of service" box and fill in the description field briefly explaining the problem. Mark the detector with an OOS tag as a visual indicator of its status.

10.5.3.3 The instrument may be returned to service once the malfunction has been corrected and the above acceptance criteria have been met. Note any repairs in the maintenance log.

10.6 Calibration process in the Software

10.6.1 Alpha Detector System Energy and Efficiency Calibration

10.6.1.1 Print out the standards sheet for calibration

10.6.1.1.1 Location: \\slsvr01\rad\alpha\Calibration_Sources .

10.6.1.2 Load sources into the detector

10.6.1.3 In the AlphaVision software, click on the Calibration icon.

10.6.1.4 Click on the detector to be calibrated.

10.6.1.5 Select Process and then select calibration from the dropdown menu.

10.6.1.5.1 The Calibration Explorer Window will appear.

10.6.1.6 In the General Window: Scan the source name;AV(detector)#-#_date (with year month day format YYYYMMDD).

10.6.1.7 Choose the correct source template.

10.6.1.8 Click next

10.6.1.9 In the Acquisition window, confirm count time of 140 minutes

10.6.1.10 Click next

10.6.1.11 In the Energy/Efficiency Calibration Window, confirm the correct source is used, and select which shelf the source is on. (This will be shelf 1) Make sure the

'Active' box is checked so the calibratoin is put in use immediately after calibration is processed.

10.6.1.12 Click next
10.6.1.13 In the Report Window, select print on completion
10.6.1.14 Click finish
10.6.1.15 When count is complete, the Manual Energy and Efficiency Calibration Window will appear. In this window, select Calibration ROI, Click Calibrate, click Save.
10.6.1.16 Verify the efficiency is above 20% and below 32%
10.6.1.17 Repeat for each detector
10.6.1.18 Record the calibration in the Alpha Maintenance Log.

10.6.2 ICV Procedure

10.6.2.1 Print out the standards sheet for ICV
10.6.2.1.1 Location: \\slsvr01\rad\alpha\Calibration_Sources
10.6.2.2 Place the correct source into the detector.
10.6.2.3 In The AlphaVision software, click on the Calibration Icon
10.6.2.4 Click on the detector to be calibrated
10.6.2.5 Select Process and then select calibration from the dropdown menu
10.6.2.5.2 The Calibration Explorer Window will appear.
10.6.2.6 In the General Window; Scan the source name;AV(detector)#-#_date (with year month day format YYYYMMDD).
10.6.2.7 Chose the correct source template
10.6.2.8 Click next
10.6.2.9 In the Energy/Efficiency Calibration Window, confirm the correct source is used, and select which shelf the source is on. (This will be shelf 1)
10.6.2.10 Click next
10.6.2.11 In the Report Window, select print on completion
10.6.2.12 Click finish
10.6.2.13 When the count is complete, the Manual Energy and Efficiency Calibration Window will appear. In this window, select Calibration ROI, click Calibrate and click Save.
10.6.2.14 Verify the efficiency is above 20% and below 32%
10.6.2.15 Repeat for each detector
10.6.2.16 Record the ICV in the Alpha Maintenance Log.
10.6.2.17 Open the AlphaVision Access database program on computer slrad18
10.6.2.18 Select QC main from the form tab
10.6.2.19 Enter date range
10.6.2.20 Select system 1 for AlphaVision or system 2 for AlphaVision 1
10.6.2.21 Select Get Cal Data.
10.6.2.22 Exit
10.6.2.23 Select Check Ver to run the report and verify the ICV passes criteria.
10.6.2.24 For the Intitial Calibratin (IC), the detectors must have the box checked in the Cal Data window for that specific calibration and the previous year's IC must be unchecked and the 'do not use' box must be checked to ensure the correct calibration is being used.

10.6.3 CCV Procedure

10.6.3.1 Print out standards sheet for CCV on the network
10.6.3.1.1 Location: \\slsvr01\rad\alpha\Calibration_Sources
10.6.3.2 Load sources into the detectors
10.6.3.3 In the AlphaVision software, click on the Calibration Icon.
10.6.3.4 Click on the detector to be calibrated
10.6.3.5 Select process and then select calibration from the dropdown menu
10.6.3.5.1 The Calibration Explorer Window will appear.

10.6.3.6 In the General Window: "Scan the source name;AV(detector)#-#_date (with year month day format YYYYMMDD)."
10.6.3.7 Select correct source template
10.6.3.8 Click Next
10.6.3.9 In the Acquisition window, confirm count time of 60 mins
10.6.3.10 Click Next
10.6.3.11 In the Energy/Efficiency Calibration Window, confirm the correct source is used and select which shelf the source is on. (This will be shelf 1)
10.6.3.12 Click next
10.6.3.13 In the Report Window, select print on completion
10.6.3.14 Click finish
10.6.3.15 When the count is complete, the Manual Energy and Efficiency Calibration window will appear. In this window, select Calibration ROI, select Calibrate and Save.
10.6.3.16 Verify the efficiency is above 20% and below 32%.
10.6.3.17 Repeat for each detector
10.6.3.18 Record the CCV in the Alpha Maintenace Log.Efficiency must be greater than 20% and less than 32%.
10.6.3.19 Open the AlphaVision Access database program on computer slrad18.
10.6.3.20 Select QC main from the form tab.
10.6.3.21 Enter date range.
10.6.3.22 Select system 1 for AlphaVision or system 2 for AlphaVision 1.
10.6.3.23 Select Get Cal Data.
10.6.3.24 Exit.
10.6.3.25 Select Check Ver to run the report and verify the CCV passes criteria.

10.6.4 Detector Background Process (See Section 10.4)

10.6.4.1 Select the Batch Icon
10.6.4.2 Select backgrounds from the Tool Bar
10.6.4.3 Select Process.
10.6.4.3.1 This will open the General Window in Batch Wizard
10.6.4.4 Name the background with month_y ear format MonthYYMMM_YY (e.g. JAN_04)
10.6.4.5 Select correct template (provided by analyst)
10.6.4.6 Click next.
10.6.4.7 In the Sample Window, add all detector names with the format: ICB;AV(detector)#.
10.6.4.8 Click next
10.6.4.9 In the Acquisition Window, confirm count time is set at 960 minutes (or as long as the longest sample count time)
10.6.4.10 Click next
10.6.4.11 In the Analysis Set Up Page, select Background Library and Background ROI, check the Use ROI box.
10.6.4.12 Click next
10.6.4.13 In the Report Window, select print on completion
10.6.4.14 Click finish
10.6.4.15 The Detector Assignment worksheet will appear, assign detectors, and select start now.
10.6.4.16 Record the backgrounds in the instrument maintenance log.
10.6.4.17 The background spectrum will be processed by the software
10.6.4.18 The detectors shall be "categorized" after each monthly background. The detectors will be labelled as follows:

Counts in Region of Interest (i.e. Th230, Th232, U234, U238, Pu238, Pu239):

- 0-2 counts – Blue – Ultra Low Level
- 0-4 counts – Yellow – Intermediate Low Level
- 0-20 counts – Green – Low Level
- 0-40 counts –Red – Always designated for Routine analysis when the RL=1 or the activity is from a known radioactive site.

See Attachment 1 Detector Color Key

10.6.4.19 Detectors with backgrounds above the counts listed above are taken out of service for cleaning.
10.6.4.20 Detectors may also be removed from service when there is a visible peak present or at analyst judgment.
10.6.4.21 Backgrounds will be 2nd reviewed before placing into service and a notation of acceptable will be listed in the electronic monthly maintenance log.

10.7 Detector Cleaning (This should be done before starting Backgrounds)
10.7.1 Clean detector surface with ethanol and a clean cotton ball.
10.7.2 Clean the sample tray and place a clean background planchette on the tray.
10.7.2.1 A passing background count is required before returning the detector to service.

10.6 Standard Verification Procedure
10.6.2 Receive manual batch from prep
10.6.3 Count for 960 minutes (make sure batch is set up correctly).
10.6.4 After count, open decay corrector (located in Rad Dive, LSC, decay corrector) to see if isotope you are verifying needs to be decay corrected. (if the isotope verifying is located in this spreadsheet, it needs to be decay corrected). Note, new activity on the prep sheet.
10.6.5 Open new spreadsheet verification folder (located in Rad Drive) and select master 3 or 6 point verification (depending on how many standards are made)
10.6.6 Enter calculated value from Decay Corrector (as True value) and value from the spectra print outs (activity on the spectra for the Isotope you are verifying).
10.6.7 Make sure units match.
10.6.8 Standard passes if the mean value is within 5% of certified (true) value, the 1.96 sigma value is within 10% of mean value and the standard reverification acceptablity evaluations are all yes.
10.6.9 Sign bottom of prep sheet and calculation page.
10.6.10 Give to prep supervisior.

11 PROCEDURE

11.6 For sample preparation reference the applicable preparation SOP.

11.7 Initial Setup
11.7.2 Establish the normal instrument settings for all controls.
11.7.2.6 Detector specific high voltage settings and required polarity are listed in the method software settings.
11.7.3 Pulser quality controls shall be checked before each use of the instrument.

11.8 Counting Samples

11.8.2 In Radcapture, go to Utilities > Export>Choose 'Alphavision' or 'Alphavision1' depending on which instrument the samples were set up on>, enter batch # in the window that pops up, and click ok to export to Alphavision.
11.8.3 In Alphavision, go to Process, select Batch to open the Batch Wizard.
11.8.4 Choose Load from LIMS, and pick the batch.
11.8.5 Choose proper analysis by clicking on the correct isotope
11.8.6 Select Next
11.8.7 Click on blank, and then pick blank type (Uu blank, Pu Blank, etc…)
11.8.8 Click on LCS, and then pick LCS type with correct spike number. For amount, use the spike aliquot amount (0.1, 0.2 mL, 0.1326 g, etc) which is printed on the batch paper work.
11.8.9 Select Next
11.8.10 Live time is count time. Enter correct count time for the batch, select next.
11.8.11 Select Nuclide Library, choose correct ROI and the specified tracer that is printed on the batch paper work.
11.8.12 Select next
10.7.3 Select correct activity units (DPM, pCi, etc), select Activity concentration,
10.7.3.1 QuantIMS: change TPU Sigma to 2 (unless otherwise noted in client requirements), add 5% systematic uncertainty and check the negative activity box. .
10.7.3.2 TALS: always select TPU Sigma 1, add 5% systematic uncertainty and check the negative activity box. .
11.8.13 Select Next, two times.
11.8.14 Select Print on Completion.
11.8.15 Select Finish.
11.8.16 Click and drag correct detectors to the correct sample ID and select Start Now.
11.8.17 The spectrum will be processed by the software.
10.7.4 For DOE:
10.7.4.1 FWHM of each tracer peak shall be ≤ 100 keV
10.7.4.2 Tracer peak energy for each sample shall be within ± 50keV of the expected energy.
11.8.18 Backgrounds are checked after high activity samples by counting an 180 minute background with an empty chamber (see 11.9.2).

11.9 Samples with a count rate of greater than 1 CPS should be removed from the alpha counting system to prevent contamination of detector(s).
11.9.2 Alpha detectors exposed to samples with count rates greater than 1 CPS should be tagged out-of-service until an empty chamber check can be performed. To perform an empty chamber check, place a clean stainless steel disc in the chamber, establish vacuum, turn on bias and start acquisition for the pre-set time (180 minutes). Note this in the instrument and maintenance log.

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 TestAmerica St. Louis QAM.

12.2 Isotope ROI and libraries are derived from the PCNudat master nuclide library:.

12.2.1 http://www.nndc.bnl.gov/nudat2/indx_dec.jsp

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 ST-QA-0040 for details.

13.0 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 LIMS Nonconformance Memorandum (NCM) module. The NCM process is described in SOP: ST-QA-0036.

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.

13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target anlaytes present above ½ RL), will be noted on the client requirements sheet.

13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:

13.2.2.1 Method Blank Contamination – If the MB concentration exceeds the applicable criteria, the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within the specified 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) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the isotopes with a high bias in the LCS are re-prepped and re-analyzed.

13.3.2.2 LCS Spike Recovery excursion (low) – The batch is re-prepped and re-analyzed for the affected isotope.

13.3.2.3 RPD/RER Duplicate excursion – For the RPD/RER one or both must be with in acceptance limits. The RPD limit is 40% or less. The RER limit is 1 or less depending on the significant digits. Not meeting the criteria requires a reprep of the samples. If samples have a physical matrix issue (ie, nonhomogenous), results can be reported with an NCM. If samples fail RPD/RER criteria after the reprep and no matrix issue is observed sample may be reported with client approval and narrated in an NCM.

13.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.4.1 Analytes should be within 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

13.5 Sample Result Evaluation

13.5.1 Tracer recovery must be within specified limits. Tracer limits are 30% - 110% unless other wise specified by the client

13.5.2 Tracer/Carrier recovery (low) – Re-extract using a reduced volume or recount for maximum count time to achieve 400 tracer counts

13.5.2.1 Note: QSAS allows for reporting results as quantitative when tracer recoveries are below 30% if:

13.5.2.1.1 the relative uncertainty associated with the tracer recovery is less than 10% (2 sigma)

13.5.2.1.2 spectral resolution requirements are met and there are no indications of spectral interferences (resolution of <100 keV).

13.5.2.1.3 detection limit requirements have been met

13.5.3 Tracer/Carrier recovery (high) – If the blank and LCS are **within limits**, have the sample logged in for native analyisis if not already logged in for native. If the blank and or LCS has **high recovery**, a reprep is required.

13.5.3.1 Truncation to 100%: Truncation can be done at the clients discretion, or with approval from manager or technical director or based on sample history.

13.5.3.2 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:

13.5.3.2.1 minimum number of tracer counts

13.5.3.2.2 level of uncertainty

13.5.3.2.3 client project requirements/approval

13.5.4 These expections will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.

13.5.5 The following occurances require a dilution to be performed:

13.5.5.1 Dilution level is determined by taking the highest gross counts divided by the count time divided by a factor of 2. (ie: 7200/180/2=1:20)

$$DL \square GCts_{High} / t_{count} / 2$$

$$DL \square \text{Dilution Level}$$

$$GCts_{High} \square \text{Highest Gross Counts}$$

$$t_{count} \square \text{Count Time}$$

13.5.5.1.1 Tailing – A peak is significantly tailing out side its region of interest (ROI)

13.5.5.1.2 The tracer recovery is low due the high activity of the sample

13.5.5.1.3 Peak Interference – A Peak is observed which is identified as an interference

13.6 Insufficient Sample

13.6.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis an NCM is written and a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are maintained in the LIMS.

14.2 Demonstration of Capability

14.2.1 Initial and continuing demonstrations of capability requirements are established in ST-QAM

14.3 Training Qualification

14.3.1 The manager/supervisor 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 ST-QAM.

14.1 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 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

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

16.2.1.1 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.

17.0 REFERENCES

17.1 Department of Energy (DOE) Environmental Measurement Laboratory (EML) HASL 300 28th Edition method A-01-R, Alpha Radioassay

17.2 AlphaVision-32, Alpha Particle Spectrum Acquistion and Analysis for Microsoft Windows and NT, Software Version 5.0 Installation, User Interface and Reference Guide, Ortec (latest version)

17.3 OCTETE Plus, Integrated Alpha-Spectroscopy System Hardware Operating Manual, 777720, Ortec (latest version)

17.4 MAESTRO-32, MCA Emulator for Microsoft Windows, A65-B32 Software User's Manual, 777800, Ortec (latest version)

17.5 U.S. Nuclear Regulatory Commission, Quality Assurance for Radiological Monitoring Programs (Normal Operations) - Effluent Streams and the Environment, Regulatory Guide 4.15.

17.6 "Quality Assurance Program Requirements for Nuclear Facilities", ANSI/ASME NQA-1 (latest edition).

17.7 TestAmerica, St. Louis Quality Assurance Manual, current revision

17.8 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions.

17.9 Decay Radiation Datebase, Version of 5/8/2013; http://www.nndc.bnl.gov/nudat2/indx_dec.jsp

17.10 Associated SOPs, current revisions
- 17.10.1 ST-PM-0002, Chain of Custody
- 17.10.2 ST-QA-0002, Standard and Reagent Preparation
- 17.10.3 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
- 17.10.4 ST-QA-0036 Non-Conformance Memorandum (NCM) Procedure
- 17.10.5 ST-QA-0040, Manual Integration Procedure
- 17.10.6 ST-RC-0040, Total Alpha Emitting Isotopes of Radium
- 17.10.7 ST-RC-0238, Isotopic Uranium By Eichrom□ Uteva Resin For Various Matrices
- 17.10.8 ST-RC-0210, Determination Of Polonium-210 By Alpha Spectrometry
- 17.10.9 ST-RC-0232, Isotopic Thorium And/Or Neptunium In Various Matrices By Eichrom□ Teva Separation Resin
- 17.10.10 ST-RC-0240, Isotopic Americium, Curium, Plutionium, Thorium, And Uranium In Various Matrices By Eichrom□ Separation Resin
- 17.10.11 ST-RC-0241, Americium, Plutonium, Curium, And Uranium In Various Matrices By Eichrom□ Uteva And Tru Resins (With Vacuum Box System)
- 17.10.12 ST-RC-0242, Isotopic Thorium, Plutonium And Uranium In Various Matrices By Eichrom□ Separation Resins
- 17.10.13 ST-RC-0246, Isotopic Americium, Curium, Uranium In Various Matrices By Eichrom□ Separation Resins

18.0 MODIFICATIONS TO REFERENCE METHOD

18.1 Energy calibrations checks are performed monthly. Daily pulsar checks are performed in place of the weekly energy calibration checks.
18.2 Backgrounds are determined monthly rather weekly
18.3 CCV's are determined monthly rather that before and after each measurement.

19.0 CHANGES TO PREVIOUS REVISION

19.1 No Changes- Annual Review
19.2 Rev. 8:
- 19.2.1 Section 10 additions
  - 19.2.1.1 Addition of Instrument setup as §10.1
  - 19.2.1.2 Addition of Calibration Quality Control Check as §10.3
  - 19.2.1.3 Addition of calibration acceptance criteria

19.3 Rev. 9:
- 19.3.1 Section 10.5, addition of limits for pulser centroid and pulser energy

19.4 Rev. 10:
- 19.4.1 Section 10:
  - 19.4.1.1 2nd level review for ICV and CCV added to section 10
  - 19.4.1.2 1200 minute setting for acquisition window for special projects
  - 19.4.1.3 Upper control limits for long backgrounds
  - 19.4.1.4 Detector cleaning
- 19.4.2 Section 12: addition of ROI and library reference
- 19.4.3 Section 13: Occurances that require dilution
- 19.4.4 Addition of Attachement 1

19.5 Rev. 11:
- 19.5.1 Grammatical corrections through out and removal of referencest to QuantIMS and Clouseau

19.5.2 Section 10, updated internal calibrations information in section and the calibration process in the lab software throughout
19.5.3 Section 10, added new standards verification procedure
19.5.4 Section 13, added corrective actions and equation for dilution level
19.5.5 Section 15, updated with new verbiage
19.5.6 Attachment 1 revised
19.5.7 Section 13,removed "See Clouseau NCM for Corrective Action"and added specfic Corrective Actions to the SOP
19.5.8 Section 18, added backgrounds will be done monthly and added CCV's will be done monthly.
19.5.9 Section 3, Added "keV" definition
19.5.10 Section 6, Added Hardware and Software specifics

Attachment 1

AlphaVision Detector Key

Red – Routine only 0-40 counts

Green – Low Level 0-20 counts

Yellow – Intermediate Low Level 0 – 4 counts
Special Projects and QC samples (Method Blanks and Lab Control Samples) Only

Blue – Ultra Low Level (for Pu/Am/Np)
Special Projects Only

OOS Tag – Out of Service (OOS)

Title: LOW BACKGROUND GAS FLOW PROPORTIONAL COUNTING (GFPC) SYSTEM ANALYSIS

Approvals (Signature/Date):

Chris Hough 9/11/13 Chris Hough Date Radiochemistry Department Manager	Michael Ridenhower 9/11/13 Michael Ridenhower Date Health & Safety Manager / Coordinator
Marti Ward 9-11-13 Marti Ward Date Quality Assurance Manager	Elaine Wild 9/11/13 Elaine Wild Date Laboratory Director

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1.0 SCOPE AND APPLICATION

1.1 This SOP is applicable to all Low Background Proportional Counting instruments. TestAmerica St. Louis performs radium-226/228, strontium-89/90, gross alpha/beta, neptunium-36 and chlorine 36.

1.2 This SOP is based on SW846 method 9310, 9315 and 9320; EPA methods 900.0, 903.0, 904.0, 905.0; and DOE EML HASL 300 method, Ba-01-R, Sr-02 and Sr-03-RC.

1.3 The SOP applies to GFPC analysis of liquid and solid matrices.

1.4 The requested limits (RL), minimum detectable amount (MDA) and QC limits are maintained in the Laboratory Information Management System (LIMS).

2.0 SUMMARY OF METHOD

2.1 This procedure provides instructions for the daily calibration and maintenance of the Low Background Proportional Counting instrumentation.

3.0 DEFINITIONS

3.1 See the TestAmerica St. Louis Quality Assurance Manual (ST-QAM) for a glossary of common terms and data qualifiers.

3.2 IQC - a computerized Quality Control Program where the counting results of Daily Radioactive check sources and Daily Background checks are entered and compared to statistical average data. A measurement within ± 3 standard deviations indicates the detector is operating within acceptable parameters.

3.3 αLL - discriminator setting indicating the alpha lower voltage limit.

3.4 Alpha Voltage Only - detector voltage capable of collecting ions created by alpha radiation only. Ion pairs created by beta radiation are not collected.

3.5 αUL - discriminator setting indicating the instruments alpha upper voltage limit.

3.6 βLL - discriminator setting indicating the beta lower voltage limit.

3.7 βUL - discriminator setting indicating the beta upper voltage limit.

3.8 Crosstalk - a measure of the amount of beta radiation that is collected in the alpha radiation channel; it is also a measure of alpha radiation collected in the beta channel.

3.9 Plateau - a point on a graph of count rate vs. detector bias voltage where further increases in bias will not result in an increase in measured counting rate.

3.10 LB4100 – LBPC (Low background Gas Flow Proportional Counting instrument).

4.0 INTERFERENCES

4.1 A detector contaminated with radioactive material will result in a high background and interfere with the correct measurement of a sample.

4.1.1 If a sample "times out" reaching 10000 counts before the allotted time, and the sample count rate is 60 cpm or greater, then another daily background check is performed on that detector. If the detector background check is unacceptable, the detector is taken Out Of Service until action is taken to bring the background check within acceptable limits. If the chamber requires action to remove contamination and a new background check is acceptable, then a 30 minute empty chamber count should be performed to determine if a new long background needs to be performed on that detector.

4.2 The actual counting efficiency for alpha radiation decreases greatly with a density > 6.0 mg/cm2. Therefore, the maximum acceptable mass density is typically 5 mg/cm 2 or less that 100 mg for a 2" planchet.

4.3 For beta radiation, reliable data may be obtained counting samples with a density as high as 10 mg/cm 2 or greater.

4.4 Sample thickness as well as moisture content may impact the alpha and/or beta results.

5.0 SAFETY

5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001), Radiation Safety Manual and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1.1 None.

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 Limit (2)	Signs and symptoms of exposure
Silver Nitrate	Poison Corrosive Oxidizer	$0.01^{g}/_{m3}$ (TWA) for silver, metal dust, and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. Skin contact may cause redness, pain, and sever burning. Eye contact can cause blurred vision, redness, and pain.
Ammonium Hydroxide	Poison Corrosive	50 ppm (NH_3)	Inhalation symptoms may include irritation to the respiratory tract. Ingestion symptoms may 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.			
TWA – Time Weighted Average			

6.0 EQUIPMENT AND SUPPLIES

6.1 Low Background Proportional Counter, equivalent to the Canberra/Oxford/Tennelec LB4100, or Protean MPC9604.

6.2 Gas mixture, 90% argon, 10% Methane
6.3 Blank planchets
6.4 PC based data acquisition system, OSUM software, IQC software
6.5 Centrifuge tubes
6.6 Centrifuge
6.7 Vortex
6.8 Pipettes, Eppendorf or equivalent
6.9 Pipette, disposable

7.0 STANDARDS AND REAGENTS

7.1 All standards and reagent preparation, documentation and labeling must follow the requirements of SOP ST-QA-0002, current revision

7.2 Radioactive sources to measure beta radiation,: Sr-90 and Ra-228 sources.

7.3 Radioactive sources to measure alpha radiation: Am-241, Th-230 and Ra-226

7.4 Deionized Water (DI), obtained from the Milli-Q unit.

7.5 Silver nitrate ($AgNO_3$), 0.5 N

7.6 Sodium chloride (NaCl), crystals

7.7 Sodium chloride (NaCl), 0.5 N

7.7.1 Add 50 mL of DI water to a 100 mL volumetric, add 5.84 g of NaCl, dilute to 100 mL, cap and shake to dissolve. Adjust volume to 100 mL with DI water.

7.8 Ammonium hydroxide (NH_4OH), concentrated, 28 N

7.9 Ammonium hydroxide (NH_4OH), 5 %

7.9.1 Add 25 mL of concentrated Ammonium Hydroxide to 475 mL of DI water. **CAUTION – Ammonium hydroxide is corrosive. Mist and vapor cause burns to every area of contact.**

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 TestAmerica St. Louis supplies sample containers and chemical preservatives in accordance with the method. TestAmerica 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 ST-PM-0002.

8.2 See associated sample preparation SOPs ST-RC-0020, ST-RC -0021, ST-RC -0036, ST-RC -0040, ST-RC -0041and ST-RC -0050, for more detailed information.

9.0 QUALITY CONTROL

9.1 See actinide preparation SOPs for additional information regarding QC types, frequency and preparation.

9.2 **Batch**

9.2.1 A sample batch is a maximum of 20 environmental samples, which are prepared together using the same process and same lot(s) of reagents.

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

9.2.3 For this analysis, batch QC consists of a method blank, a Laboratory Control Sample (LCS), and Matrix Spike (MS)/ Sample Duplicate (Dup). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.2.3.1 Matrix Spike (MS) and Matrix Spike Duplicate (MSD) may be performed upon client request, and are noted in the Client Requirement Sheets and Log-in.

9.3 **Method Blank (MB)**

9.3.1 A method blank is a blank matrix processed simultaneously with, and under the same conditions as, samples through all steps of the procedure.

9.3.2 A method blank must be prepared with every sample batch.

9.4 **Laboratory Control Sample (LCS)**

9.4.1 An LCS is 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 sample batch.

9.5 **Matrix Spike**

9.5.1 A Matrix Spike is an aliquot of a field sample to which a known amount of target analyte(s) is added, and is processed simultaneously with, and under the same conditions as, samples through all steps of the analytical procedure.

9.6 **Sample Duplicate**

9.6.1 A Sample Duplicate is an additional aliquot of a field sample taken through the entire analytical process to demonstrate precision.

9.7 **Procedural Variations/ Nonconformance and Corrective Action**

9.7.1 Any variation shall be completely documented using a Nonconformance Memo and approved by the Supervisor and QA Manager. See SOP ST-QA-0036 for details regarding the NCM process.

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

10.0 CALIBRATION AND STANDARDIZATION

10.1 Additional preventative maintenance can be found in ST-QA-0024.

10.2 Voltage Plateau Determination

10.2.1 **Frequency**:

10.2.1.1 Performed as a part of the Intial Calibration.

10.2.2 **Voltage Plateau Determination on LB4110 Red**

10.2.2.1 Place the Am-241 sources in Drawers A and B.

10.2.2.2 Select the auto-sequence file "Plateau"

10.2.2.3 Highlight the detectors in Drawers A and B

10.2.2.4 Click on 'Run"

10.2.2.5 Type 'Alpha' for the sample ID for the detectors with the alpha source

10.2.2.6 Click 'Done'. The voltage plateau will begin automatically.

10.2.2.7 When the alpha counts are complete, the highlighted detectors will flash. Click 'Unit status' and highlight the voltage plateau file name. Click on 'Re-load'. Enter 'Beta" as the sample ID.

10.2.2.8 Place the Sr^{90} beta source on the detector.

10.2.2.9 Click 'Done'. The voltage plateau will complete automatically.

10.2.2.10 When the plateau is complete click on 'Data Output'. Select the plateau file. Print the graphs and data for calibration packages. Archive the data file.

10.2.2.11 Repeat the steps above for drawers C and D.

10.2.3 **Criteria for Plateaus for LB4110 Red**

10.2.3.1 Voltage range used to determine the plateau is 300-1500V

10.2.3.2 Voltage increase per step is <50V per step

10.2.4 **Voltage Plateau Determination on Protean MPC 9604**

10.2.4.1 The manufacturer has counted plateaus and permanently set the discriminator voltages. The manufacturer does not recommend recounting plateaus. The detectors are manufactured to have a high dead time. However, a cross talk test may be performed using different nuclide sources to indicate acceptable discriminator settings. This procedure is outlined in the discriminator setting section of this SOP.

10.2.5 **Criteria for Plateaus for Protean MPC 9604**

10.1.5.1 Plateaus are permanently set for this instrument from the manufactuer.

10.3 **Discriminator Settings**

10.3.1 **Frequency:**

10.3.1.1 Performed as a part of the Intial Calibration.

10.3.2 **Discrimator Settings on LB4110 Red**

10.3.2.1 From the unit menu, select 'Change ROI'

10.3.2.2 Place a beta source in each detector

10.3.2.3 Highligh a detector with a beta source

10.3.2.4 The alpha upper limit should be set at 100% and the beta lower limit should be set a 0%. The alpha lower limit and the beta upperlimit should both be set at 50%.

10.3.2.5 Select 'Count'. Accumulate at least 100,000 beta counts

10.3.2.6 Reduce the beta upper limit/alpha lower limit until there is 3.5% beta into alpha crosstalk.

10.3.2.7 Raise the alpha lower limit until there is 0.10% beta into alpha crosstalk.

10.3.2.8 Raise the beta upper limit until it is 5% less than the alpha lower limit.

10.3.2.9 Select 'Halt'.

10.3.2.10 Repeat steps above until all detectors have been set.

10.3.2.11 Select 'Close'. Discriminator settings are updated automatically.

10.3.3 **Discrimator Settings on Protean MPC 9604**

10.3.3.1 Collect a minimum of 10,000 counts for each of Am-241, Th-230 and Po-210 sources

10.3.3.2 Calculate the percentage of crosstalk and compare the results to historical and expected values. Consult the Technical director if the values fall out of range.

10.4 **Initial Calibration**:

10.4.1 **Frequency**:

10.4.1.1 The Gas Flow Proportional Counter (GFPC) is calibrated initially and verified each year thereafter. Recalibration may be required if indicated during the operation of the instrument.

10.4.2 The specific calibration source preparations can be found in the file containing the previous calibration.

10.4.3 All nuclide sources shall be NIST traceable.

10.4.4 The efficiency calibration shall consist of at least seven single or dual sets of mass attenuated calibration standards, unless a single point source efficiency is to be determined.

10.4.5 The standards shall have enough activity to generate at least 10000 counts in 90 minutes of count time for the most highly attenuated source. The count rate shall not exceed 5000 counts per second.

10.4.5.1 For alpha and beta analysis, separate sets of calibration sources shall be prepared.

10.4.6 The mass attenuation is accomplished by utilization of a salt solution with comparable make up to the majority of samples seen in the laboratory.

10.4.6.1 Alternatively, the mass attenuation may be accomplished by using the same carrier solution used in a specific analysis.

10.4.7 Each standard shall be counted in every detector to be calibrated.

10.4.8 **Criteria for a Single or Dual Set Calibration**

10.4.8.1 The efficiency of the detector (the dependent variable) shall be plotted on a single graph against the masses (the independent variable) for all data points.

10.4.8.2 The equation of the calibration curve shall be determined using polynomial functions and be included on the plot of the curve. The curve shall be continuous and smooth.

10.4.8.3 The degree of the polynomial shall not exceed three. The number of discreet source pairs shall be two more than the degree of the polynomial.

10.4.8.4 The percent difference of the measured efficiency and theoretical efficiency shall be calculated for all data points.

10.4.8.5 Points that are visual outliers or demonstrate less than 15 percent difference between the measured efficiency and theoretical efficiency may be removed at the analyst's discretion. Low residual mass sources and samples are difficult to plate with acceptable duplicate precision. Therefore, high outliers may not necessarily be removed from the calibration if they mimic live sample masses. In any case outliers above 15 percent shall be removed from the calibration curve. No more than 20 percent of the data points may be removed. Reasons for removal or inclusion of outliers shall be documented in the calibration narrative. Once outliers are removed, the percent difference between the measured efficiency and theoretical efficiency must be recalculated using the new polynomial coefficients generated from removal of data points. If outliers over 15 percent difference remain between the measured efficiency and theoretical efficiency the Radiochemistry Manager/QA must be consulted before calibration may continue.

10.4.8.6 The coefficient of determination (r^2) shall be calculated and displayed on the plot with the equation of the trend line. An r^2 greater than or equal to 0.9 is required to proceed to counting of verification sources.

10.4.8.7 If the coefficient of determination (r^2) is not greater than or equal to 0.9 on the plot of all data points (with or without outliers removed), the data may be plotted using the mean of the paired values for both the efficiencies and the masses. This action is acceptable to reduce the variability caused when plating low mass sources. Calculate the percent difference of each datum value from the mean of the paired points. If the percent difference for any datum value is greater than 10 percent for alpha or 7.5 percent for beta delete the data pair and perform another statistical fit to the data. More than 20 percent of all data points may not be removed from the curve. The coefficient of determination (r^2)

shall be calculated and displayed on the plot with the equation of the trend line. An r^2 greater than or equal to 0.9 is required to proceed to counting of verification sources.

10.5 **Independent Calibration Verification (ICV)**

10.5.1 **Frequency**:

10.5.1.1 Performed with every intial calibration

10.5.2 GFPC initial calibrations must be verified by a second source standard.

10.5.3 The ICV standard is NIST traceable.

10.5.4 The ICV is counted to accumulate at least 5,000 counts.

10.5.5 **ICV for Dual Set Calibrations**:

10.5.5.1 Prepare at least one set of calibration verification sources consisting of a low, medium and high residual mass within the calibration range of the curve.

10.5.5.2 Prepare a blank at the same time.

10.5.5.3 The sources shall contain radionuclide from a second source. Second sources may include a second dilution from the same primary source used for the calibration curve.

10.5.5.3.1 Alternatively, verification source nuclides may consist of different nuclides than the calibration curve if it is customary to do so.

10.5.5.4 Count each secondary source and the blank in all detectors that were calibrated.

10.5.5.5 Calculate the results in terms of percentage recovery.

10.5.5.6 Calculate the mean results of all masses across each detector.

10.5.5.7 Criteria:

10.5.5.7.1 Individual points are within 30 percent of the true value

10.5.5.7.2 The mean result of all masses across all detectors is less than 10 percent.

10.5.5.7.3 If any detector fails the validation tests the Technical Director must be consulted to provide corrective action.

10.5.6 **ICV for Single Set Calibrations**:

10.5.6.1 Prepare at least one set of verification sources varying in expected mass within the calibration range of the curve.

10.5.6.2 Prepare a blank at the same time.

10.5.6.3 The sources shall contain radionuclide from a second source. Second sources may include a second dilution from the same primary source used for the calibration curve.

10.5.6.3.1 Alternatively, verification source nuclides may consist of different nuclides than the calibration curve if it is customary to do so.

10.5.6.4 Count the secondary source and the blank in all detectors that were calibrated.

10.5.6.5 Calculate the results in terms of percentage recovery.

10.5.6.6 Calculate the mean results of all masses across each detector.

10.5.6.7 Criteria:

10.5.6.7.1 Individual points are within 30 percent of the true value

10.5.6.7.2 The mean result of all masses across all detectors is less than 10 percent.

10.5.6.7.3 If any detector fails the validation tests the Technical Director must be consulted to provide corrective action.

10.6 **Setting Performance Check Criteria After Calibration**

10.6.1 Twenty background check samples are counted and used to establish quality control limits for the daily background checks.

10.6.2 Twenty alpha/beta check sources are counted after calibration and used to establish quality control limits for the daily source checks.

10.6.3 The limits will be a running average of the four months post calibration.
10.6.3.1 The limits are to be documented.
10.6.3.2 The limits will be re-established monthly at the following frequency
10.6.3.2.1 1st month – take first five data points from the new month and fifteen data points from the initial calibration.
10.6.3.2.2 2nd month – take first five points from new month, five from prior month and ten from initial calibration.
10.6.3.2.3 3rd month – take first five points from new month, five points each from the previous two months and five from the initial calibration.
10.6.3.2.4 4th month – take first five data points from new month and five points each from the previous three months.
10.6.3.3 Limits are set.

10.7 **Alpha to Beta Crosstalk Determination**
10.7.1 The mean mass is determined for each data point used to calculate the mass attenuation curve.
10.7.1.1 These curves are calculated and plotted and the percent of alpha into beta crosstalk is determined. This is done by dividing the beta counts per minute as observed in the beta channel from the alpha calibration source counts by the sum of the alpha and beta counts per minute.
10.7.1.2 The mean percent of alpha into beta is determined for each mass point by using the count data accumulated for two sets of alpha sources.
10.7.1.3 The crosstalk curve is plotted as mean crosstalk values relative to the mean mass for the two sets of data.
10.7.1.3.1 In this manner the crosstalk factor can be determined for any given mass.
10.7.1.4 The equation of the curve shall be determined using polynomial functions.
10.7.1.5 The coefficient of determination (R^2) shall be calculated and displayed on the plot as well as the equation for the trendline.

10.8 **Beta to Alpha Crosstalk Determination**
10.8.1 Since beta to alpha crosstalk does not vary across mass, a mean beta to alpha crosstalk correction factor is calculated.
10.8.2 The percent of beta into alpha is determined by dividing the alpha counts per minute as observed in the alpha channel from the beta calibration source counts by the sum of the alpha and beta counts per minute.
10.8.3 The mean percent of beta into alpha is determined for all mass points. The mean percent is insignificant in calculating results, therefore is not applied to the result calculation.

10.9 **Long Background**
10.9.1 **Frequency**:
10.9.1.1 Monthly or whenever instrument conditions have significantly changed since the previous background was performed (e.g. detector replaced, etc.)
10.9.1.2 Minimum count time: 1000 minutes.
10.9.2 Wash the planchet holder and clean the drawers with a 20% radiac wash or ethyl alcohol.
10.9.2.1 Do not spray cleaner directly onto the drawers. Spray cleaner on a Kimwipe, a cotton ball, or paper towel and wipe out the drawers.
10.9.3 Check that instrument settings are as specified in 11.1.

10.9.4 **Red Long Background Count Set Up**
10.9.4.1 Place the cursor on the red box in the upper left hand corner of the screen and right click on the mouse.

10.9.4.2 Select 'edit parameters'. Verify the count time on the screen is set to 1000 minutes and the iterations is set to 1. If they are different than 1000 min and 1iteration, change them to 1000 min and 1 iteration. Then select 'close' to exit this window.
10.9.4.3 Place clean empty planchets in instruments.
10.9.4.4 Place the cursor on the red box in the upper left hand corner of the screen and right click on the mouse.
10.9.4.5 Select 'create batch' from the instrument menu.
10.9.4.6 Select 'background'
10.9.4.7 Select the detectors that are to be counted by double clicking the mouse on the drawer desired which selects all detectors in that drawer or on each individual detector in the display.
10.9.4.8 Select 'run'
10.9.4.9 Select 'done'
10.9.4.10 When backgrounds are complete, review the printouts for acceptance.

10.9.5 **Protean Long Background Count Set Up**
10.9.5.1 Create Manual batch in RadCapture
10.9.5.2 Export Manual batch from RadCapture
10.9.5.3 At Protean instrument:
10.9.5.4 Select 'Detector'
10.9.5.5 Select 'Sample Log'
10.9.5.6 Select appropriate Long Background (ex: Sept_Lng_Bkg_00) you want to start under sample ID
10.9.5.7 Change count time to 1000min
10.9.5.8 Select 'Start'
10.9.5.9 Continue these steps with detectors 1-23.
10.9.5.10 Review the data for acceptance when the backgrounds are complete.

10.9.6 **Printing Protean Long Backgrounds**
10.9.6.1 Select 'Print Protean data icon' on the desk top
10.9.6.2 Select OK
10.9.6.3 Enter Batch #
10.9.6.4 Print

10.9.7 **Protean Long Background Entry into Protean**
10.9.7.1 Select Input data
10.9.7.2 Select Definitions
10.9.7.3 Select Calibrations
10.9.7.4 Select Properties
10.9.7.5 Select References 0-7 for Detectors 0 thru 7 and 8-15 for Detectors 8 thru 15
10.9.7.6 Enter Background CPM's for Alpha and Beta from printed data sheet

10.9.8 **Orange and Purple Long Background Count Set-Up**
10.9.8.1 Select detector 0
10.9.8.2 Select 'source log'
10.9.8.3 Select 'monthly long background' by clicking on the file list arrow.
10.9.8.4 Ensure count time is set to 1000 minutes.
10.9.8.5 Select 'start'
10.9.8.6 Continue these steps with detectors 1-23.
10.9.8.7 Review the data for acceptance when the backgrounds are complete.

10.9.9 **Printing Orange and Purple Long Backgrounds**
10.9.9.1 Select 'Data

10.9.9.2 Select 'Source Count Data'
10.9.9.3 Select 'Source Name' Monthly Long BKG
10.9.9.4 Select 'This Range' enter your date range that Long Backgrounds were performed.
10.9.9.5 Select 'Refresh'
10.9.9.6 Select 'Source Count Summary' under Reports
10.9.9.7 Select 'Print'
10.9.9.8 Select 'Landscape' under Orientation
10.9.9.9 Select 'OK'

10.9.10 **Long Background Criteria:**
10.9.10.1 The CPM for the alpha are <0.2 and the beta < 2.0, the detector may be used.
10.9.10.1.1 The data printout must include initials and date to indicate that has been reviewed.
10.9.10.1.2 If a detector is above this limit, discard planchet.
10.9.10.1.3 Clean the planchet holder with radiac wash, ethyl alcohol or a detergent spray cleaner and dry thoroughly.
10.9.10.1.4 Place a clean planchet in the holder and repeat steps for that detector (s) only.
10.9.10.1.5 Perform a new background.
10.9.10.1.5.1 Note: the detector is tagged out of service until a successful background has been achieved.

10.10 **Cl-36**: At least four sodium chloride standards are prepared for calibration.
10.10.1.1 Add 10 mL of DI water to 4 centrifuge tubes.
10.10.1.2 Add 0.500 mL of 0.5 N sodium chloride carrier solutions to each centrifuge tube. Swirl to mix.
10.10.1.3 Add 2 drops of 5 % ammonium hydroxide solution, swirl to mix.
10.10.1.4 Add 12 mL of 0.5 N silver nitrate solution to each centrifuge tube.
10.10.1.5 Vortex for 30 seconds.
10.10.1.6 Centrifuge and decant supernate to waste.
10.10.1.7 Proceed to section 11.4, Planchet Preparation of Silver Chloride Precipitation of SOP ST-RC-0036.
10.10.1.8 Average the four weights for the sodium chloride carrier solution, record the standardized weight in the log book and on the bottle.
10.10.1.9 NOTE: It may be necessary to use more than 0.500 mL of carrier in some large water samples or calibrate a 4 N sodium chloride carrier solution. The efficiency of the detectors will have to be calculated using the heavier sodium chloride carrier solution.

10.10.2 Prepare four sodium chloride calibration samples as in Section 10.9 but add a known amount of Cl-36 to each tube before the sodium chloride carrier is added. Analyze samples by GFPC and determine detector efficiency as per Section 12, Data Analysis and Calculations.

11.0 PROCEDURES

11.1 Initial Setup
11.1.1 Check the normal instrument settings for all controls as described below:
11.1.1.1 Tank Flow 8 psi
11.1.1.2 Flow Cells >/= 0.3 SCFH, the flow will vary, the target range is 0.15 to 0.20 SCFH.
11.1.2 The High Voltage is set as indicated in the Manuals for the LB4000/LB4100 located in the count room file cabinet. The Protean remains as set by the manufacturer and does not require adjustment.

11.1.3 If counting gas has just been changed or turned on, allow a minimum purge time of 30 minutes prior to operation. Record gas tank changes on document on separate sheet.

11.2 Record date of Daily Background and Check Source Data in runlog logbook.

11.3 Maintenance

11.3.1 Change out the counting gas when the gauge reads under 500 psi. This usually occurs every 1 to 2 weeks. Record in the instrument maintanence logbook.

11.3.2 Allow gas to purge a minimum of 30 minutes prior to operation.

11.4 Data Acquisition: Daily Background Check and Source Check

11.4.1 **Daily Background Check**:

11.4.1.1 **Red Instrument**:

11.4.1.2 Open the drawer by rotating the knob at the front of the drawer to the 'DOWN' position and pull the sample drawer out slowly. Place clean empty planchets in the sample holders.

11.4.1.3 **Before inserting the drawer, confirm that none of the planchets extend above the sample holder. Failure to observe this note can result in damage to the detector.**

11.4.1.4 Slowly insert sample drawer into the instrument and slowly rotate the positioning knob into the 'UP' position.

11.4.1.5 Place the cursor on the red box in the upper left hand corner of the screen and <u>right click</u> on the mouse.

11.4.1.6 Select 'create batch'

11.4.1.7 Select 'daily background check'

11.4.1.8 Select the detectors that are to be loaded by double clicking the mouse on the drawer desired or on each individual detector in the display.

11.4.1.9 Select 'run'

11.4.1.10 Select 'done'

11.4.1.11 Measure the detector background for 200 minutes. The count time is predetermined by the protocol selected. i.e. 'daily background check'.

11.4.1.12 The detector display will be yellow when the detector is counting. The detector display will turn green when the count is complete.

11.4.1.13 When counting is complete, place the cursor on the red box in the upper left hand corner of the screen and <u>right click</u> on the mouse. Select 'data output' and select the data file generated by your background counts for that instrument, "DAY- ###". Select 'ok' to print data.

11.4.1.14 On any work station, i.e. "PC computer in the count room", double click on the IQC icon.

11.4.1.15 Select 'import data'

11.4.1.16 Select 'Red'. Enter the current date. Click on the file list arrow.

11.4.1.17 From the file list select <u>each</u> file generated above <u>individually</u>, and then select import data. i.e. import each "Day" or "DQC" file for that instrument individually.

11.4.1.18 Select 'close'

11.4.1.19 Select 'reporting'. Verfiy the current date in both the 'start' and 'end' date fields. Select 'print' to generate the report.

11.4.2 **Protean Instrument**:

11.4.2.1 Open each detector drawer. Place clean empty planchets into each sample holder and slowly insert each sample drawer into the instrument.

11.4.2.2 Double click detector 0 on the Protean computer screen.
11.4.2.3 Select 'source log'
11.4.2.4 Set the time for 200 minutes.
11.4.2.5 Type 'DB0' in the sample id box. (D for daily. B for background. 0 for detector.)
11.4.2.6 Select 'start'
11.4.2.7 Double click detector 1 on the computer screen. Repeat steps 11.4.2.3 through 11.4.2.5 for each detector, making sure to change the number to coincide with the detector the background is counting for.
11.4.2.8 Remove planchets from detector drawers when counting is complete.
11.4.2.9 On any work station, i.e. "PC computer in the count room", double click on the IQC icon.
11.4.2.10 Select 'import data'
11.4.2.11 Select 'Protean'. Enter the current date. Click on the file list arrow.
11.4.2.12 Select 'close'
11.4.2.13 Select 'reporting'. Verfiy the current date in both the 'start' and 'end' date fields. Select 'print' to generate the report.

11.4.3 **Orange and Purple Instrument**:
11.4.3.1 Open each detector drawer. Place clean empty planchets into each sample holder and slowly insert each sample drawer into the instrument.
11.4.3.2 Select detector 0.
11.4.3.3 Select 'source log'.
11.4.3.4 Select 'DB' by clicking on the file list arrows for orange.
11.4.3.5 Select 'Daily BKG' by clicking on the file list arrows for purple.
11.4.3.6 Select 'start'
11.4.3.7 Repeat these steps with detectors 1-23.

11.4.4 **Daily Background Criteria:**
11.4.4.1 Review the IQC printouts for each detector.
11.4.4.1.1 If a detector fails background criteria (3 sigma), clean the detector with radiac wash or ethyl alcohol and re-run.
11.4.4.1.2 If the detector fails a second time, but the CPM for the alpha are <0.2 and the beta < 2.0, the detector may be used. The data printout must include initials and date to indicate that it was checked.
11.4.4.1.3 Circle any failed detector on the printout.
11.4.4.1.4 Place a planchet upside down in the planchet holder to indicate that the detector is out of service for that day.

11.5 **Daily Source Check**
11.5.1 **Red Instrument**:
11.5.1.1 Open the drawer by rotating the knob at the front of the drawer to the 'DOWN' position and pull the sample drawer out slowly.
11.5.1.2 Place alpha sources in the sample holders of the A and B drawer. Place beta sources in the sample holders of the C and D drawer.
11.5.1.3 Place the cursor on the red box in the upper left hand corner of the screen and right click on the mouse.
11.5.1.4 Select 'create batch'
11.5.1.5 Select 'daily source check'
11.5.1.6 Select the detectors that are loaded by double clicking the mouse on the desired drawer or on each individual detector in the display.
11.5.1.7 Select 'run'

11.5.1.8 Select 'done'
11.5.1.9 Measure the detector source for 2 min. The count time is predetermined by the protocol selected. i.e. 'daily source check'
11.5.1.10 The detector display will be yellow when the detector is counting. The detector display will turn green when the count is complete.
11.5.1.11 When counting is complete, place the cursor on the red box in the upper left hand corner of the screen and right click on the mouse. Select 'data output' and select the data file generated by your background counts for that instrument, "DQC- ###". Select 'ok' to print data.
11.5.1.12 Open the drawer by rotating the knob at the front of the drawer to the 'DOWN' position and pull the sample drawer out slowly. Place beta sources in the sample holders of the A and B drawer. Place alpha sources in the sample holders of the C and D drawer.
11.5.1.13 Repeat steps 11.5.1.3 to 11.5.1.11.
11.5.1.14 On any work station, i.e. "PC computer in the count room", double click on the IQC icon.
11.5.1.15 Select 'import data'
11.5.1.16 Select 'Red'. Enter the current date. Click on the file list arrow.
11.5.1.17 From the file list select each file generated above individually, and then select import data. i.e. import each "Day" or "DQC" file for that instrument individually.
11.5.1.18 Select 'close'
11.5.1.19 Select 'reporting button'. Verify the current date in both the 'start' and 'end' date fields. Select 'print'to generate the report

11.5.2 **Protean Instrument:**
11.5.2.1 Slowly open each detector drawer. Place alpha sources in sample holders of detectors 0-7. Place beta sources in sample holders of detectors 8-15 and slowly insert each drawer into the instrument.
11.5.2.2 Double click detector 0 on the Protean computer screen.
11.5.2.3 Select 'source log'.
11.5.2.4 Set the time for 2 minutes.
11.5.2.5 Type "SA0" in the sample id box. (S for source. A for alpha. 0 for detector.)
11.5.2.6 Select 'start'
11.5.2.7 Double click detector 1 on the computer screen. Repeat steps 11.5.2.3 to 11.5.2.6 for each detector, making sure to change A to B when starting the beta sources on detectors 8-15 and changing the number to coincide with the detector the source is on.
11.5.2.8 When the counting is complete, slowly open each detector drawer. Place beta sources in detectors 1-7. Place alpha sources in detectors 8-15.
11.5.2.9 Double click detector 0 on the Protean computer screen.
11.5.2.10 Type "SB0" in the sample id box. (S for source. B for beta. 0 for the detector.)
11.5.2.11 Double click detector 1 on the computer screen. Repeat steps 11.5.2.10 for each detector, making sure B to A when starting the alpha sources on detectors 8-15.
11.5.3 Remove sources from detector drawers when counting is complete.

11.5.4 **Orange and Purple Instrument**:
11.5.4.1 Slowly open each detector drawer. Place alpha sources in sample holders of detectors 0-7. Place beta sources in sample holders of detectors 8-15. Slowly insert each drawer into the instrument.
11.5.4.2 Select detector 0.
11.5.4.3 Select 'source log'.
11.5.4.4 Select 'SA0'.

11.5.4.5 Select 'start'
11.5.4.6 Repeat these steps for detectors 1-7 using the correlating detector number. For detectors 8-15 select 'SB8', 'SB9', and so on for each correlating detector number.
11.5.4.7 Slowly open each detector drawer when counting is complete. Place beta sources in detectors 1-7 and place alpha sources in detectors 8-15.
11.5.4.8 Select detector 0.
11.5.4.9 Select 'SB0'.
11.5.4.10 Select 'start'.
11.5.4.11 Repeat these steps for detectors 1-7 using the correlating detector number. For detectors 8-15, select 'SB8', 'SB9' and so on for each correlating detector number.
11.5.4.12 Repeat steps 11.5.4.1 to 11.5.4.11 for detectors 16-23.
11.5.4.13 Remove sources from detector drawers when counting is complete.

12.0 DATA ANALYSIS AND CALCULATIONS

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

12.2 Result calculations are performed by TestAmerica St. Louis' Rad Capture software program. These calculations are found in the TestAmerica St. Louis ST-QAM.

12.3 To calculate the efficiency of the detectors for Cl-36, divide the net counts determined of the spiked Sodium Chloride, by the known dpm of the Standard used.

$$\frac{\textit{Net Counts of Spiked Silver Chloride}}{\textit{Known dpm of Cl} \square 36\,(\textit{decay corrected to day counted})} \square \textit{Efficiency}$$

13.0 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 LIMS Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036.

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.
13.2.1.2 Project specific requirements if more stringent than our routine procedure (e.g. no target anlaytes present above ½ RL), will be noted on the client requirements sheet.
13.2.2 Corrective Action for Method Blanks not meeting acceptance criteria:
13.2.2.1 Method Blank Contamination – (e.g. reprep/reanalysis, narration). If the Method Blank concentration exceeds the applicable criteria, the batch must be re-prepped unless the concentration of all associated samples is less than the RL or greater than ten times the concentration found in the blank.

13.3 Laboratory Control Sample (LCS)
13.3.1 Acceptance Criteria:
13.3.1.1 All control analytes must be within the specified 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) – Samples with results less than the RL may be reported with an NCM (unless prohibited by client requirements). Samples with detects for the isotopes with a high bias in the LCS are re-prepped and re-analyzed..

13.3.2.2 LCS Spike Recovery excursion (low) the batch is re-prepped and re-analyzed for the affected isotope.

13.4 RPD/RER Duplicate excursion – For the RPD/RER One or both must be with in acceptance limits. The RPD limit is 40% or less. The RER limit is 1 or less depending on the significant digits. Not meeting the criteria requires a reprep of the samples. If samples have a physical matrix issue (ie, nonhomogenous), results can be reported with an NCM. If samples fail RPD/RER criteria after the reprep and no matrix issue is observed sample may be reported with client approval and narated in an NCM.

13.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

13.5.1 Analytes should be within control limits for accuracy (%Recovery) and precision (RPD).

13.5.2 Corrective Action for MS/MSD not meeting acceptance criteria:

13.5.2.1 MS/MSD Spike Rec. excursion may not necessarily warrant corrective action other than narration.

13.6 Sample Result Evaluation

13.6.1 Tracer/Carrier recovery must be within specified limits.

13.6.2 Tracer/Carrier recovery low– Samples must be reextracted. Exceptions can be made and results reported with approval from the technical director, manager, or client and approvpriate NCM included.

13.6.3 Tracer/Carrier recovery high

13.6.3.1 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:

13.6.3.1.1 minimum number of tracer counts

13.6.3.1.2 level of uncertainty

13.6.3.1.3 client project requirements/approval

13.6.4 If the sample carrier recovery is significantly higher than normal, the native concentration in the sample of the carrier analyte may be present causing a high bias to the carrier recovery. This high bias to the carrier analyte would in turn cause a low bias to the samples result. The laboratory defines significant to be an additional 20% above the average LCS/MB carrier recovery (as determined from a population of LCS and MB data), with a maximum of 110%. The table below shows the limits determined for each carrier analyte. The analyst should ensure that the carrier analysis is requested to determine native concentration for samples exceeding the limit.

Radium	Strontium	Chloride
110%	109%	109%

13.6.5 These expections will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.

13.7 Insufficient Sample

13.7.1 For any prescribed re-preparation corrective action, if there is insufficient sample to repeat the analysis a narrative comment stating such is included in the report narrative.

14.0 METHOD PERFORMANCE AND DEMONSTRATION OF CAPABILITY

14.1 Method performance data, Reporting Limits, and QC acceptance limits, are given in LIMS.

14.2 Demonstration of Capability
14.2.1 Initial and continuing demonstrations of capability requirements are established in the ST-QAM.

14.3 Training Qualification
14.3.1 The manager/supervisor 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 the ST-QAM.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in the ST-QAM.

15.0 VALIDATION

15.1 Laboratory SOPs are based on published methods (EPA, DOE, ASTM, Eichrom, Standard Methods) and do not require validation by the laboratory. The requirements for laboratory demonstration of capability are included in the ST-QAM. Laboratory validation data would be appropriate for performance based measurement systems, non-standard methods and significant modifications to published methods. Data from said validations is held in the QA department.

16.0 WASTE MANAGEMENT AND POLLUTION CONTROL

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
16.2.1 The following waste streams are produced when this method is carried out.
16.2.1.1 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.

17.0 REFERENCES

17.1 Department of Energy (DOE) Environmental Monitoring Laboratory (EML) HASL-300 Procedures Manual, method Ba-01-R, Beta Radioassay, Sr-02 Strontium 90, Sr-03-RC Strontium-90 in Environmental Samples.
17.2 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 1, Method 900.0 Gross Alpha and Gross Beta Radiochemistry
17.3 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 6, Method 903.0 Alpha-Emitting Radium Isotopes
17.4 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 8, Method 904.0 Radium-228
17.5 Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Section 9, Method 905 Radioactive Strontium in Drinking Water

17.6 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9310, Gross Alpha and Gross Beta
17.7 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9315, Alpha-Emitting Radium Isotopes
17.8 Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, Method 9320, Radium-228
17.9 TestAmerica St. Louis Quality Assurance Manual, current revision
17.10 Corporate Environmental Health and Safety Manual (CW-E-M-001) and St. Louis Facility Addendum (SOP ST-HS-0002), current revisions
17.11 Associated SOPs, current revisions:
- 17.11.1 ST-PM-0002 "Sample Receipt and Chain of Custody"
- 17.11.2 ST-QA-0002, "Standards and Reagent Preparation."
- 17.11.3 ST-QA-0024, "Preventative Maintenance"
- 17.11.4 ST-QA-0036, "Non-Conformance Memorandum (NCM) Process"
- 17.11.5 ST-RC-0004, "Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis".
- 17.11.6 ST-RC-0020, "Determination of Gross Alpha/Beta Activity"
- 17.11.7 ST-RC-0021, "Gross Alpha Radition in Water using Copreciptation"
- 17.11.8 ST-RC-0036, "Determination of Chlorine-36 in Various Matrices by GFPC"
- 17.11.9 ST-RC-0040, 'Total Alpha Emitting Isotopes of Radium"
- 17.11.10 ST-RC-0041, "Radium 228 in Water"
- 17.11.11 ST-RC-0050, "Preparation of Strontium-89 and 90"
- 17.11.12 ST-RC-0300, "New Jersey 48-hour Gross Alpha Testing for Private Well Testing ACT (PWTA)

18.0 MODIFICATIONS TO THE REFERENCE METHOD

18.1 TestAmerica St. Louis uses thorium-230 to calibrate the GFPC system for Ra-226. Th-230 has similar alpha energies and a sufficiently long half life to eliminate the need for purification. The laboratory has historically performed well on PE programs for Ra-226, demonstrating the laboratory's ability to accurately calibrate for this isotope. Calibrating with a Ra-226 source presents a severe bias in the quantitated result. Ra-226 can be purified and separated from all other alpha emitting isotopes, but the moment after separation, alpha emitting daughters begin to grow (i.e. radon-222, polonium-28 and polonium-214). As the daughter's in-growth alpha activity changes and due to the higher alpha energies of these daughters, the measured efficiency of the GFPC changes as well. After three weeks the alpha activity from purified Ra-226 increases by a factor of four. Due to their short half lives, these daughters can not be isolated long enough to mathematically correct for the bias brought on by them. Calibrating the GFPC with Ra-226 is actually calibrating with a mix of the four isotopes and not a legitimate calibration under the cited regulation.

18.2 Strontium-89 short half life makes it impractical to use as a calibration standard for both radium-228 analysis, as stated in EPA method 904 and SW method 9310, and strontium-89 analysis, as stated in EPA method 905. TestAmerica St. Louis uses a mixed strontium-90/yittrium-90 standard for its' GFPC beta calibration used in Gross Beta, strontium-90, strontium-89, and radium-228 analyses. TestAmerica St. Louis has selected the strontium-90/yittrium-90 standard because it produces a stable beta emission which can be reliably used for initial and continuing calibration. By using this standard mix, we have beta emissions at the lower and upper energetic spectrum whose average is in the middle of the beta range.

18.3 For Ra-228 analysis, TestAmerica St. Louis uses chemical separation techniques to eliminate other potential beta emitters.

18.4 TestAmerica St. Louis does not perform a direct strontium-89 analysis. TestAmerica St. Louis provides calculated results based on the difference between Total strontium and strontium-90.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Updated Section 10 to address voltage increase per step, plateau slope and QC check count requirements (5000 counts)

19.2 Rev. 11;
- 19.2.1 Added instument Purple throughout section 10 and 11.
- 19.2.2 Adjusted procedure steps throughout section 11.

19.3 Rev. 12,
- 19.3.1 Added Sr-02-RC and Sr-03-RC to sections 1.0 and 17.0.

19.4 Rev. 13:
- 19.4.1 Added Neptunium to scope in section 1.0.
- 19.4.2 Updated the Quality Control Program for counting daily rad checks and daily background checks in section 3.0.
- 19.4.3 Updated background count set-up, printing and entering protean data in section 10.8.

19.5 Rev. 14:
- 19.5.1 Removed references to Clouseau, SAC and QuantIMS
- 19.5.2 Section 5.0 added silver nitrate and ammonium hydroxide
- 19.5.3 Section 6.0 updated to include additional equipment
- 19.5.4 Section 7.0 updated to include addition reagents
- 19.5.5 Section 9.0 added reference to prep SOPs for additional information
- 19.5.6 Section 10.0 added sodium cloride standard preparation & reference to ST-QA-0024
- 19.5.7 Section 12.0 added Cl-36 detector efficiency calculation
- 19.5.8 Section 13.0 updated to include actual corrective actions and native concentration carrier requirements
- 19.5.9 Section 13.0 updated to include corrective actions
- 19.5.10 Section 17.0 added reference to ST-QA-0024

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