DEPARTMENT OF THE AIR FORCE
HEADQUARTERS 49TH WING (ACC)
HOLLOMAN AIR FORCE BASE, NEW MEXICO

ENTERED

APR 2011

APR 25 2011

Lieutenant Colonel Christian J. Knutson
Base Civil Engineer
550 Tabosa Avenue
Holloman AFB NM 88330-5840

New Mexico Environment Department
Attn: Mr. James Bearzi
Hazardous Waste Bureau
2905 Rodeo Park Drive East
Santa Fe NM 87105-6303

Dear New Mexico Environment Department

Holloman AFB is pleased to address the base-wide background study comments regarding radiological data from your contractor AQS.

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision according to a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

If you have any questions, please contact Mr. David Scruggs of our Asset Management Flight at (575) 572-5395.

Sincerely

CHRISTIAN J. KNUTSON, Lt Col, USAF

Attachment:
Response to Comments

cc:

Mr. David Strasser
Hazardous Waste Bureau
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Response to Comments from NMED on Base-wide Background Study
Holloman Air Force Base
Comments Received via Email on 18 March 2011

Responses to the additional comments provided on 18 March 2011 are presented below. All HAFB responses are provided in blue text.

The review of each response was based upon the information provided in the written response from Holloman Air Force Base (HAFB) as well as discussions from the January 4, 2011 conference call. Since most of the data needed to resolve the radiochemical NODs are similar for each comment, an attached matrix is not being provided, to reduce redundancy. The initial responses as provided by HAFB are not adequate as provided. However, the outstanding issues may be resolved by providing the requested information contained in the following discussion.

Additional Clarification Required from HAFB

The responses to NMED NOD Comments Part 1, No. 5 and all of Part 2 (Comment Nos. 1 - 9) were not adequate as provided. In order to resolve data quality issues and render a determination on the usability of the radiochemical data, the following must be addressed.

For the HAFB Base-wide Background Study Report, radiological data for one sample delivery group and its associated data validation report were reviewed (SDG D8I090195). Additional cursory reviews showed that the issues presented apply to all other radiochemical data (as outlined in Part 1 Comment 5 and Part 2 Comments 1 - 9). In order to approve the background values provided, NMED seeks to further understand the analytical methods that were used, the extent and effect of matrix interferences observed, and the overall usability of the radiochemical data.

1. For all non-Environmental Protection Agency (EPA) or modified methods used (C-01-1, E903.0, E904.0, STL-RC-0211, A-01-R), please provide a copy of the method including details regarding modifications and non-standard approaches. For example, describe how samples were prepared and results calculated for soil samples that were analyzed using drinking water methods such as Methods 903.0/904.0.

The table presented below provides a cross-reference of analytical methods and laboratory standard operating procedures (SOPs) that demonstrated how the internal SOPs apply to EPA and non EPA methods. The SOPs reference the EPA methods upon which they are based and provide a description of sample preparation (Nine SOPs are attached). Labs routinely use internal SOPs to document methodology and system performance and with radiochemistry as the practice is dynamic due to advances in technology and instrumentation. For example, Test America used their SOP ST-RD-0403 to analyze for Radium 226 and 228 and it applies to both soil and water matrices. 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. Additionally, the soil prep method specific to this SOP has also been provided (ST-RC-0003/ST-RC-0004).

Requested SOP / Method	SOP used by Test America	Analyte	Prep Method	Soil Prep Method
C-01-1	ST-RD-0302	Carbon 14	ST-RC-0057	
E903/9320/E904	ST-RD-0403	Ra 226 and 228	ST-RC-0041	ST-RC-0003/ST-RC-0004
ST-RC-0211	ST-RD-0302	Pb210	ST-RC-0211	
A-01-R	ST-RD-0210	Isotopic Th and U	ST-RC-0241	ST-RC-0004

Below is an email received from the director of the radiochemical laboratory with Test America/St. Louis explaining the methods used for the Holloman samples that helps explain the various methods used vs. those in the SOW.

"The SOP used for preparation of Lead-210 would have been the "ST-RC-0211" (we have dropped the "L" in "STL" since that time). "ST-RD-0302" is the analysis SOP for Liquid Scintillation Counting (both C-14 and Pb-210 are analyzed on the LSC instrumentation). Our internal SOPs are generally separated by preparation for the specific element/analytes and analysis by instrument type.

In regards to our SOPs versus "published" methods (e.g. EPA 903.0 for Ra-226), our SOPs are written such that they are compliant with multiple method references. For example "ST-RC-0041" covers the prep for Ra-226 and Ra-228. The chemistry in this internal SOP is consistent with EPA 903.0, SW846 9315, DOE HASL 300 Ra-06-RC, and SM 7500-Ra to name a few. Thus, we typically reference the method requested by the client by following our SOP. Other methods may not have a "published" reference. This would be the case for the Pb-210, so we reference the SOP# in the report.

Method 907 is an "older" technology method which utilizes ion exchange resins. Most laboratories have moved away from this technology toward solid-phase resins manufactured by EiChrom Technologies. While the technology is slightly different, the results should be comparable. We reference DOE HASL-300 A-01-R for our alpha spec, and have for quite some time. We normally would state the equivalence in a proposal – however, this does not mean the client updates the SOW to reflect our method reference. Our SOP for the prep would have been "ST-RC-0241".

In regards to the "MOD" reference, note that there are not many methods written specifically with soil in mind. Thus, we utilize the basic water/groundwater/waste method references with "MOD". The main modification is that we have to get the solid into a liquid form. This is normally accomplished using a total dissolution technique. For some analyses this is not practical/advisable, so a digestion or leach is performed. Once the solid is "in solution", we can then follow the rest of the chemistry steps based upon the referenced method. The "MOD" could also be present due to a slight change/improvement to the method to accommodate different matrices or more recent technological improvements. Many older methods utilize organic extractants/materials that are a burden to the environment from a waste generation perspective. We have made improvements where practical to address this.

Thanks,

Terry Romanko
(314)298-8566 (office)

2. For all radiological data, provide a detailed assessment of the matrix interference issues. Provide a detailed review of all radiochemical data for failed matrix spike/matrix spike duplicate (MS/MSD) recovery, carrier recovery, or other evidence of significant matrix interference. Acceptable laboratory control sample (LCS) recovery is not a valid rationale to overlook other quality control (QC) failures. This assessment should include:

    a. Specific data that are affected for each method, matrix, and analyte,

    b. Severity of the matrix effects and likely bias,

    c. Usability of affected data for the background study, and

    d. Suggested method improvements to reduce matrix effects.

Qualify or reject radiochemical data as necessary. Reissue data validation (DV) reports, amended data tables, and completeness reports as necessary.

A detailed review and assessment of the matrix interference issues was made in each of the DV reports which have been reissued and are provided as Attachments. Additionally, a detailed review of all radiochemical data including all QC deficiencies is addressed in each reissued DV report. For example, if the MS/MSD were taken from a different sample batch (a common laboratory practice when a relatively low number of samples are received in a sample delivery group), precision and accuracy were more readily gauged by the carrier recoveries AND LCS recoveries. Since MS/MSD results are applicable only to the **spiked** sample, the relative value for evaluation purposes is lower than the value placed on LCS/LCSD results which apply to all the samples in the batch. Therefore, LCS/LCSD results for each batch are a more effective measure of laboratory accuracy and precision. According to the *Evaluation of Radiochemical Data Usability Guidelines*, "matrix spikes may not be required for methods where a carrier or tracer is used...matrix effect will be detected through tracer (carrier) recovery". A thorough review of the carrier recoveries was also made in the reissued DV reports. Therefore, the systematic process of all QC criteria was weighed and validated against the parameters set forth in the *Evaluation Radiochemical Data Usability Guidelines* and if multiple deficiencies were present they were handled according to the statistical procedure outlined in Appendix B of the guidelines.

a) Specific data that are affected for each method, matrix, and analyte are addressed in the revised DV reports provided. Reason codes for DV qualifiers have been added as requested.
b) Severity of the matrix effect and likely bias has been accounted for with the use of additional qualifiers on the data as well acknowledgment of bias in the DV reports.
c) Usability of the data remains unchanged as no data has been rejected.
d) Suggested method improvements to reduce matrix effects: Given the high salinity of groundwater and soil in the Tularosa basin the matrix effects will persist. It is beyond the scope the background study to perform research and development of analytical methods.

3. Either reject the Radium-226 result for sample BWBG-SB20-20 or provide additional rationale for its inclusion. Review all radiochemical data for similar co-precipitation failures and reissue the DV reports, amended data tables, and completeness reports as necessary.

   Please refer to the attached DV report D8I090195Rev1 for more details on this sample. The following text is excerpted from the DV report: "Sample BWBG-SB20-20 was double traced with barium carrier and would not precipitate for Radium 226. The recovery for Radium 226 (57%) was within control limits therefore no qualification was necessary."

4. Provide a written description of the "truncation" procedure. It must be sufficiently detailed to allow an independent laboratory to reproduce the data. Include available references to EPA or other peer-reviewed scientific literature approving this practice. The discussion must indicate when and how the procedure is to be used and the impact on resulting data.

   A written explanation of the truncation procedure is attached in the letter from TestAmerica to Bhate dated 15 April 2011.

5. In addition, to ensure the consistency and usability of project data, future work plans and investigation reports should address the following:

   a. Evaluation of method alternatives to reduce matrix interferences.

      By definition matrix interference refers to sample characteristics that interfere with the test method execution. Examples of matrix interference include samples with extreme pH, high alkalinity or acidity, and chemical constituents that react with target analytes. Common matrix interference is the presence of a non-target compound in high concentrations. Given the highly saline content of both soil and groundwater matrices at Holloman, removal of the interferences would be by default remove the target analytes. Discussion with the largest analytical services provider in the country (TestAmerica) regarding the subject of

alternative methods for analysis in saline-rich matrices has determined that the current commercially available technology cannot be improved at this time. Method modifications are continuously being attempted by labs to improve system performance but when matrices such as soil and groundwater from Holloman are subject to analysis, they are bound to interfere with target analytes and can only be addressed through dilution of the sample.

b. Comparability of background and site data from different labs using proposed method modifications.

See response above regarding method modifications. It is beyond the scope the background study to perform research and development of analytical methods.

c. Specific data validation guidelines including:

i. Treatment of MS/MSD and other QC failures,

Treatment of MS/MSD and other QC failures are discussed in item 2 as well as in the reissued DV reports.

ii. Use of "Reason codes" for DV qualifiers, and

Reason codes were added to the DV reports as requested.

iii. Use of statistical tests for the evaluation of quality control sample outcomes as described in industry standard radiological guides.

The use of statistical tests for the evaluation of quality control sample outcomes was addressed by using Appendix B of *Evaluation of Radiochemical Data Usability Guidelines.*

List of attachments provided on CD:

Attachment	Description
ST-RC-0003	Drying and Grinding of Soil and Solid Samples
ST-RC-0004	Preparation of Soil, Sludge, Filter, Biota and Oil & Grease Samples for Actinide Analyses
ST-RC-0041	Radium 226 and Radium 228 by Chemical Separation Preparation
ST-RC-0057	Carbon 14 Determination Using Positive Inert Gas Flow
ST-RC-0211	Determination of Lead 210 by Liquid Scintillation Counting
ST-RC-0241	Americium, Plutonium, Curium and Uranium in Various Matrices by EiChroM Uteva and Tru Resins (with vacuum box systems)
ST-RD-0210	Alpha Spectroscopy Analysis
ST-RD-0302	Liquid Scintillation Counter Analysis
ST-RD-0403	Low Background Gas Flow Proportional Counting (GFPC) System Analysis
D8I040185REV1	Re-issued DV report
D8I050142REV1	Re-issued DV report
D8I060139REV1	Re-issued DV report
D8I090195REV1	Re-issued DV report
D8I100172REV1	Re-issued DV report
D8I110184REV1	Re-issued DV report
D8I120256REV1	Re-issued DV report
D8I190223REV1	Re-issued DV report
D8I200171REV1	Re-issued DV report
D8I230152REV1	Re-issued DV report
Bhate Truncation Letter 041511	Letter from Test America describing the truncation practice

Title: DRYING AND GRINDING OF SOIL AND SOLID SAMPLES

Approvals (Signature/Date):

Rhonda Ridenhower 8/12/10 Rhonda Ridenhower Date Radiochemistry Dept Manager	Michael Ridenhower 8/13/10 Michael Ridenhower Date Health & Safety Manager / Coordinator
Marti Ward 8·13·10 Marti Ward Date Quality Assurance Manager	Elaine Wild MAW 8·12·10 Elaine Wild Date Laboratory Director

This SOP was previously identified as SOP No. ST-RC-0003 Rev. 11

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1.0 SCOPE AND APPLICATION

1.1 This procedure describes the method for drying, reducing particle size and homogenizing samples for radiochemical analysis.

1.2 This method is applicable to solid samples including sediment and soil.

1.3 This method is not applicable to solid samples such as oils, filters, fauna, flora and certain types of "destruction and debris" materials. Consult the radiochemistry manager or supervisor to determine if the "destruction and debris" material is applicable to this procedure.

2.0 SUMMARY OF METHOD

2.1 This SOP describes the method for drying and grinding samples to a uniform particle size so that radiochemical analyses can be performed.

3.0 DEFINITIONS

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

3.2 Rolling Rocks –inert grinding cylinders used to homogenize samples on the ball mill.

4.0 INTERFERENCES

4.1 This method may cause evaporation of volatile radionuclides such as iodine and tritium. This method is not to be used for samples that require volatile chemical analysis.

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
Hearing protection is required when using the pulverizer or ball mill in the event that noise levels exceed 90 dB(A) within 3 feet of the grinder. Contact the EH&S Coordinator to determine the results of the noise surveys.

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.

6.0 EQUIPMENT AND SUPPLIES

6.1 Ball mill

6.2 Heat resistant metal pan (Bread loaf pan)

6.3 Drying Oven

6.4 GM 44-9 beta/gamma probe

6.5 Paint cans

6.6 Pulverizer, including dish and puck

6.7 Rolling Rocks

6.8 Vacuum cleaner (hand held)

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 Quartz sand

7.3 Deionized water

7.4 Ethanol

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 cited and other applicable sample collection documents for detailed collection procedures. Sample volume and preservative information is given in ST-PM-0002.

8.2 Soil samples may be collected in either plastic or glass containers.

9.0 QUALITY CONTROL

9.1 **Decontamination Blank (Pulverizer)**

9.1.1 A pulverizer decontamination blank is processed at the beginning of each day of use and between every sample.

9.1.2 The pulverizer decontamination blank consists of quartz sand, processed for a minimum of 10 minutes in the pulverizer, after the dish and puck have been decontaminated by a previous grinding of quartz sand.

9.1.3 See section 11.4.12 of this SOP for cleaning and collecting of pulverizer decontamination blanks.

9.1.4 The pulverizer decontamination blank is processed for gamma and alpha/beta analysis.

9.2 **Decontamination Blank (Ball Mill)**

9.2.1 Ball Mill decontamination blanks are processed with each use of the rolling rocks, after decontamination steps are completed, as per section 9.2.2 of this SOP.

9.2.2 The rolling rocks are soaked in radiac wash 2-4 hours, and then washed in a dishwasher. After cleaning, the stones are laid on paper to air dry.

9.2.3 The dried, washed rolling rocks are place in a paint can with quartz sand and ground for 8 hours.

9.2.4 The decontamination blank (sand from 9.2.3) is processed for gamma and gross alpha / beta analysis. On a quarterly bases, three of the decontamination blanks are selected to have isotopic analyses performed including strontium, radium, uranium, thorium, and plutonium.

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

9.3.1 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.

9.3.2 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 There are no calibration requirements for this preparation procedure.

11.0 PROCEDURE

11.1 **Sizing of Sample**

11.1.1 For solid samples with various particle sizes, including pebbles or small rocks, processing by ball milling may be adequate to achieve a uniform particle size.

11.1.2 For solid samples that must be reduced to a fine mesh size (i.e. 200 mesh), a pulverizer will be necessary.

11.1.3 For solid samples that are composed of large particles such as rock, cement, stone, etc., it may be necessary to fracture or reduce the size of the sample before pulverizing.

11.1.4 Refer to the Client Requirement sheet to determine if there are client specific sizing requirements.

11.1.5 In an effort to avoid cross contamination, the order of the samples processed should be from low activity to high radiological activity, if the sample activty is known. Screening results or surveying with a GM 44-9 beta/gamma probe may be used for this purpose.

11.2 **Drying of Samples - Non-volatile Isotopes**

11.2.1 Place sample in labeled, bread loaf pan.

11.2.1.1 The amount of sample dried must be sufficient to cover the minimum sample amounts needed for the analyses requested. See ST-PM-0002 for minimum sample amounts.

11.2.2 If the sample has not been dried, perform the following steps. Otherwise, proceed to step 11.3.

11.2.3 Place sample in a drying oven for **8-24** hours at approximately 105°C +/- 5°C. Record time, date, lot and initials into logbook.

11.2.3.1 Visually inspect the sample at the end of the drying period. If the sample does not appear to be dry, return sample to oven to continue drying.

11.2.3.2 When the sample is dry, remove the sample from the oven. Record the oven temperature, date, time and analyst initials in the Oven Temperature Logbook.

11.2.3.3 Allow the sample to cool to room temperature.

11.3 **Ball Mill Procedure**

11.3.1 Start with dried sample as specified in the Drying section of this procedure.

11.3.2 Document the sample ID number on the labeled paint can lid and side. Load the sample into paint cans.

11.3.3 Place 4-5 rolling rocks into the paint can. Seal the lid on the paint can.

11.3.4 Load each can onto the ball mill.

11.3.5 Start the ball mill.

11.3.6 Record the date, analyst initial in oven temperature logbook for when sample were put onto a ball mill.

11.3.7 The sample should tumble for at least 1 hour to provide adequate **mixing**; to ensure adequate **grinding**, the sample should roll for **8** hours.

11.3.8 At the end of the processing cycle, turn off the ball mill and remove the paint cans from the ball mill.

11.3.9 In a hood, open the paint cans and visually inspect the sample to ensure that the sample has been adequately ground.

11.3.10 Print labels for the samples and place them on the paint cans. If labels are not available be sure the written label on the can is still legible and store in the appropriate location for laboratory analysis.

11.4 **Pulverizer procedure**

11.4.1 Pulverize large solid samples such as rocks and cement samples, or samples which must be reduced to 200 mesh.

11.4.2 Fragment the sample to a size appropriate for the pulverizer dish.

11.4.3 Separate the larger particles that are not amenable to the pulverizer. Continue to fracture or fragment the larger portions until the entire sample can be added to the pulverizer.

11.4.4 In a hood, add sample to dish and puck. **Do not overfill the dish and puck thereby preventing the lid from sealing securely.**

11.4.5 Place the dish securely in the pulverizer holder and close the door on the machine.

11.4.6 Open the air cylinder valve and regulate outgoing pressure to 80 psi for operation of the pulverizer.

11.4.7 Press and hold the green start button on the pulverizer, until it begins to operate at the 80 psi level.

11.4.8 To achieve 200 mesh sample size run pulverizer for 10 minutes.

11.4.9 After pulverizing the sample, press the red stop button to stop the pulverizer.

11.4.10 Lift the pulverizer door and remove the dish.

11.4.11 In a hood, carefully transfer the pulverized sample from the dish to a labeled secondary sample container.

11.4.11.1 Transfer the pulverized sample into a labeled paint can, add small mixing cylinders or ball bearings and mix for at least 1 hour on the ball mill.

11.4.12 Cleaning the pulverizer

11.4.12.1 The dish and puck must be decontaminated before pulverizing the next soil sample, to prevent cross contamination.

11.4.12.2 Vacuum the pulverizer and wipe with a damp paper towel. Frisk the paper towel and dispose to appropriate waste stream.

11.4.12.3 Decontaminate the dish and puck by filling it with approximately 4 oz. of quartz sand and replacing the lid; then place it in the pulverizer and operate the machine a minimum of 10 minutes.

11.4.12.4 Empty the sand (be sure to prepare gamma and alpha/beta analysis).

11.4.12.5 Vacuum off the dish, puck and lid. Dampen a clean paper towel with DI water. Wipe the dish, puck, and lid ensuring that all traces of sand are removed.

11.4.12.6 Wipe with Ethanol and let air dry. Frisk with a GM 44-9 beta/gamma probe to scan for contamination.

11.5 Proceed to sample preparation SOP, ST-RC-0004, "Preparation of Soil, Sludge, Biota and Oil/Grease samples for Radiochemical Analysis."

12.0 DATA ANALYSIS AND CALCULATIONS

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

12.2 Include copies of the applicable pages of the pulverizer logbook with the data package.

12.3 The paint can containing the rolling rocks for decontamination is labeled prior to rolling.
- 12.3.1 The naming convention is as follows:
  - 12.3.1.1 #run, month ID (in QuanTIMS), date, year, sample# (e.g. 1K2806-1)
    - 12.3.1.1.1 a second can on the same day would be identified 1K2806-2

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

13.2 Decontamination Blank
- 13.2.1 No activty above the standard reporting limit should be observed in the decontamination blank.
  - 13.2.1.1 Gross Alpha less than 5 pCi/gram
  - 13.2.1.2 Gross Beta less than 8 pCi/gram
  - 13.2.1.3 Gamma results less than MDA
- 13.2.2 The samples processed with the decontamination blank are evaluated.
- 13.2.3 Decontamination blank assessment and corrective action details are given in the Gas Flow Proportional Counter SOP: ST-RD-0403 and the Gamma Spectroscopy SOP: ST-RD-0101

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 QAM section 18.3.

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 QAM section 18.3.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in QAM section 18.3.

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 QAM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods.

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."

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 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 TestAmerica Quality Assurance Manual (QAM), current revision

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

17.3 Associated SOPs

17.3.1 ST-PM-0002, "Sample Receipt and Chain of Custody".
17.3.2 ST-QA-0002, "Standards and Reagent Preparation."
17.3.3 ST-QA-0036, "Non-conformance Memorandum (NCM) Process."
17.3.4 ST-RC-0004, "Preparation of Soil, Sludge and Filter Paper Samples for Radiochemical Analysis."
17.3.5 ST-RD-0102, "Gamma Spectroscopy Analysis"
17.3.6 ST-RD-0403, "Gas Flow Proportional Counting Analysis"

18.0 CHANGES FROM PREVIOUS REVISION

18.1 Added section 11.3.6, referering to recording steps for dates and analyes of oven temerature for samples put in the ball mill in section 11.3.

Title: PREPARATION OF SOIL, SLUDGE, FILTER, BIOTA AND OIL & GREASE SAMPLES FOR ACTINIDE ANALYSES

Approvals (Signature/Date):

3-14-11	3/16/11
Sarah Hurst — Date	Michael Ridenhower — Date
Actinides Supervisor	Health & Safety Manager / Coordinator
3-16-11	3/16/11
Marti Ward — Date	Elaine Wild — Date
Quality Assurance Manager	Laboratory Director

This SOP was previously identified as SOP No. ST-RC-0004 Rev. 17

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1.0 SCOPE AND APPLICATION

1.1 This SOP has destructive procedures, which prepare a sample for radiometric measurement. This procedure is applicable to soils, sludge, filters, biota and oil/grease samples for Actinide analyses, namely: isotopic americium, curium, neptunium, plutonium, thorium and uranium.

1.2 For samples requiring an acid leaching, the entire sample may be leached and a leachate aliquot taken for all radiochemistry analyses. Method Blanks and QC samples are initiated after the leaching process.

1.3 The reporting limits and QC limits are maintained in the Information Management System (QuantIMS) and can be found in associated analytical SOPs.

2.0 SUMMARY OF METHOD

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

3.0 DEFINITIONS

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

4.0 INTERFERENCES

4.1 Inherent in this procedure is the assumption that the carrier and/or tracer solution mixes completely with the sample matrix. Incomplete mixing can cause anomalous chemical yield data.

4.2 Samples containing naturally high concentrations of the carriers and/or tracers (e.g. Ba, or U-232) can cause chemical yields in excess of 100% for some analyses.

5.0 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 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.

Compound	Hazards	Exposure Limits (1)	Signs of Exposure
Hydrofluoric Acid	Poison Corrosive	3 ppm (TWA)	Inhalation symptoms may include sore throat, coughing, labored breathing and lung congestion/inflammation. Skin contact may cause serious burns which are not immediately apparent or painful. Symptoms of eye contact include redness, pain, and blurred vision.
Nitric Acid	Corrosive Poison Oxidizer	2 ppm (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.
Hydrogen peroxide (30%)	Oxidizer Corrosive Fire (increases flammability of combustible, organic, and readily oxidizable materials)	1 ppm (TWA)	Irritation to respiratory tract and burning of mucous membrane of nose and throat. Pain, redness and blurred vision in eyes.
Hydrochloric Acid	Poison Corrosive	5 ppm (Ceiling)	Inhalation symptoms include coughing, choking, inflammation of the nose, throat, and upper respiratory tract. Skin contact can cause redness, pain, severe skin burns, and discoloration. Vapors are irritating to the eyes. Contact may cause severe burns.
Perchloric acid	Poison Corrosive Oxidizer	None established	**Unstable at ordinary temperature and pressure and can undergo explosive decomposition, especially at elevated temperatures or if allowed to dehydrate.** Inhalation of vapors or mists will cause irritation with coughing, choking, and inflammation of the nose, throat, and upper respiratory tract. Highly corrosive to tissue. Can cause severe burns with discoloration and pain. Permanent visual damage may occur.
1- Always add acid to water to prevent violent reactions.			
2- Exposure limit refers to the OSHA regulatory exposure limit.			
TWA – Time Weighted Average			
Ceiling – At no time should this exposure limit be exceeded.			
STEL – Short term exposure limit			

6.0 EQUIPMENT AND SUPPLIES

6.1 Analytical balance.

6.2 Ashless Powder, cellulose filter, Whatman or equivalent.

6.3 Disposable aluminum pans.

6.4 Disposable digestion vials, 75 ml, Capitol Vial Corporation or equivalent.

6.5 Drying oven.

6.6 Glassware as appropriate.

6.7 Griddle (Hot Plate), heating limit 400 °F.

6.8 Grinder.

6.9 Hot plates, stirring hotplates.

6.10 Mod Block digestion system, CPI International, 48 hole per block or equivalent.

6.11 Muffle furnace, programmable.

6.12 Perchloric Acid Digestion Hood with water washdown.

6.13 Quartz and Porcelain crucibles individually numbered with TEXPEN® or permanent marker.

6.14 Teflon and Glass beakers, 100, 250, 2000 ml etc.

6.15 TEXPEN®, for high temperature marking of quartz crucibles.

6.16 Teflon beaker covers and glass watch glasses.

6.17 Teflon stir bars.

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. Boric acid crystals, H_3BO_3, (Reagent).

7.4. Calcium nitrate (1.25 M) - Dissolve 51 g of $Ca(NO_3)2$ in 100 mL of water and dilute to 250 mL with water.

7.5. Eichrom Load solution, Nitric acid (3 M HNO_3) in Aluminum nitrate (1 M $Al(NO_3)_3 \bullet 9\ H_2O$)

7.5.1 Dissolve 350 g of Aluminum nitrate in 700 ml of water, add 190 ml of concentrated nitric acid and dilute to 1000 ml with DI water.

7.6. Nitric acid (16 M HNO_3) -concentrated, sp. gr. 1.42, 70.4%.

7.6.1 Nitric Acid (8 M HNO_3) - To an appropriately sized bottle containing 500 mL DI water, add 500 mL concentrated HNO_3. Mix well.

7.6.2 Nitric Acid (2 M HNO_3) - To an appropriately sized bottle containing 500 mL DI water, add 127 mL concentrated HNO_3, dilute to 1000 ml with DI water. Mix well.

7.6.3 Nitric Acid (1 M HNO_3) - To an appropriately sized bottle containing 900 mL DI water, add 62.5 mL concentrated HNO_3 and dilute to 1 liter. Mix well.

7.6.4 Nitric Acid (4M HNO_3) – to an appropriately sized bottle containing 1500 mL DI water, add 500 mL concentrated HNO_3.

7.7. Hydrochloric acid (12 M HCl) - concentrated, 37.2%.

7.8. Hydrofluoric acid (48-52% HF) - concentrated.

7.9. Hydrogen Peroxide (H_2O_2), 30%.

7.10. Perchloric acid, $HClO_4$, concentrated, 70-72%.

7.11. Sodium Carbonate ($Na_2CO_3 \cdot 10H_2O$), (1 M)
7.11.1 In a 1-L graduated cylinder, dissolve 500 g of sodium carbonate, in DI water. Dilute to a final volume of 1 L with DI water. Mix thoroughly and allow to settle overnight.

7.12. Americium 243 tracer, NIST traceable, approximately 10-20 dpm/mL.

7.13. Americium 241 spike, NIST traceable, approximately 10-20 dpm/mL.

7.14. Curium 244 tracer, NIST tracable, approximately 10-20 dpm/mL.

7.15. Neptunium 237 spike standard, 10-25 dpm/mL.

7.16. Plutonium 242 tracer (Pu-236 can also be used), NIST tracable, approximately 10-20 dpm/mL.

7.17. Plutonium 238 spike, NIST traceable, approximately 10-20 dpm/mL.

7.18. Plutonium 239/240 spike, NIST traceable, approximately 10-20 dpm/mL.

7.19. Thorium 229, NIST traceable 10-20 dpm/mL.

7.20. Natural Thorium spike standard (Th-232/Th-228), NIST traceable, approximately 10-20 dpm/mL.

7.21. Thorium 230 spike standard, NIST traceable, approximately 10-20 dpm/mL.

7.22. TRM solid reference material.

7.23. Clean Uranium-232 tracer standard (free of Th-228 daughter, removed by lead sulfate precipitation, activity verified before use), NIST traceable, approximately 10-20 dpm/mL.
7.23.1 See SOP: STL-RC-0242 for the clean uranium procedure.

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.

8.4 Sample hold time is 180 days from collection.

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 existed (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)MB), a Laboratory Control Sample (LCS), and Sample Duplicate (SD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 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 Oil analyses, the method blank is comprised of 1.25M Calcium Nitrate.

9.2.4 For Soil and other solid (non-filter) matrices, the method blank is comprised of 1.25M Calcium Nitrate.

9.2.5 For Filter analyses, the method blank is comprised of a blank filter, supplied either by the laboratory or the client.

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

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 Oil analyses, the LCS is comprised of 1.25M Calcium Nitrate fortified with the isotopes of interest.

9.3.4 For Soil, and other solid (non-filter) matrices, the LCS may consist of a TRM solid reference material or 1.25M Calcium Nitrate fortified with the isotopes of interest.

9.3.4.1 If Americium, Curium and/or Plutonium are requested, fortify calcium nitrate with the requested isotopes.

9.3.4.2 If Uranium or Thorium are requested, either singly or in combination with other actinides, use the TRM reference material.

9.3.5 For Filter analysis, the LCS is comprised of a blank filter, supplied either by the laboratory or the client, fortified with the isotopes of interest.

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, are 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 Refer to the applicable analytical procedure for standardization of carrier/tracer solutions.

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

11.0 PROCEDURE

11.1 This SOP addresses multiple sample preparation options based on the matrix and behavior of the samples. Samples are muffled prior to digestion unless the sample matrix is not conducive to muffling (e.g. high organic matter, construction debris). Consultant manager/supervisor prior to initiating sample preparation to determine which preparation is to be performed.

11.2 Soil and Sediment - Muffling

11.2.1 Initiate sample preparation worksheet.

11.2.2 Samples should be dried, ground, ball milled and/or pulverized as necessary, per ST-RC-0003, "Drying and Grinding of Soil and Solid Samples."

11.2.3 Samples may be sieved to ensure a uniform particle size.

11.2.4 Pre-weigh crucible.

11.2.5 Remove up to a 10 gram aliquot for analysis, and place in a TEXPEN® numbered quartz crucible. Record the exact weight of the sample taken to the nearest 0.0001 gram. Weigh on an analytical balance.

11.2.5.1 **The amount of sample required is determined by the reporting limit requested and can be up to 10 grams. Usually a short count analysis requires a 1 gram aliquot and a long count analysis requires a 2 gram aliquot. These amounts are subject to change based on the client requirements, suspected activities of the isotopes, and count time requirements.**

11.2.6 Prepare a Method Blank and LCS using 0.5 ml of 1.25M Calcium nitrate.

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

11.2.8 Spike the LCS and MS or MSD, with 10 - 40 dpm of the appropriate standard, for the requested target isotope(s).

11.2.8.1 A soil reference material can be used for the LCS when the requested target isotopes are present.

11.2.8.2 See Client Requirement sheet to determine which LCS is needed.

11.2.9 Add 5 - 40 dpm of each tracer to all samples, blanks, LCSs, and MS/MSDs in the analytical batch that corresponds to target isotopes requested.

11.2.9.1 If samples are to be analyzed at a dilution, add the tracer after the digestions, unless client requirements specifically stipulate otherwise.

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

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

11.2.12 Proceed to section 11.9 for digestion instructions.

11.3 Filters and Swipes - Muffling

NOTE: The following procedure is for all filters that can be muffled safely via a ramped program without melting and binding to the crucible or beaker. This process is useful for filters (or swipes) that contain organic material as the high temperature of the muffle oven destroys such matter and allows for a much cleaner and simpler digestion. Plastic and Teflon filters should not be prepared using this method. Instead a leach should be performed. For these filter types proceed to section 11.8.

11.3.1 Weigh beaker and record.

11.3.2 Combine all paper filters which constitute the sample in a 250 mL glass beaker.

11.3.3 Label beakers with a TEXPEN® or metal marker to ensure identification post muffling.

11.3.4 Prepare a Method blank and LCS using the same number of clean, comparable filters.

11.3.4.1 If no comparable filters are available, a DI water blank can be used.

11.3.5 Spike LCS and, MS or MSD, with 10 - 40 dpm of appropriate standard, for the requested target isotopes.

11.3.5.1 A soil reference material can be used for the LCS when the requested target isotopes are present.

11.3.5.2 See Client Requirement sheet to determine which LCS is needed.

11.3.6 Add 5 - 40 dpm of each tracer to all samples, blanks, LCSs, and MS/MSDs in the analytical batch that corresponds to target isotopes requested.

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

11.3.7.1 Ribbed watch glasses help ensure organic material is being destroyed.

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

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

11.3.8.2 Heat samples for 45 minutes at approximately160° C.

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

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

11.3.9 If filters only partially reduced to ash, repeat steps 11.3.8.3 and 11.3.8.4, otherwise proceed to section 11.9

11.3.9.1 NOTE: Not all filters will reduce to ash. As long as the organic material has lightened in color, samples can proceed to digestion.

11.3.9.2 If filter has not reduced to ash after repetition and does not appear lightened in color, consult manager/supervisor for instruction.

11.3.10 Allow samples to cool to room temperature.

11.3.11 Proceed to section 11.9 for digestion instructions.

11.4 Glass Fiber Filter Muffling Preparation

NOTE: Glass fiber filters are not ramped like other filters. Glass fiber filters also require a Hydrofluoric pre-digestion, which cannot take place in a glass beaker.

11.4.1 Glass filters are placed in either 250 mL or 400 mL glass beakers. Make sure to change gloves in between handling samples.

11.4.2 Beakers are then muffled at 600° C for four hours.

11.4.3 Remove beakers from muffle oven and allow to cool.

11.4.4 Wash Teflon beaker with radiac soap.

11.4.5 Reflux Teflon beaker on a hot plate for approximately thirty minutes with ~ 20mL of 4M HNO_3, cover with a watch glass

11.4.6 Dump 4M HNO_3 in waste and remove Teflon beaker from hotplate

11.4.7 Transfer samples from glass beaker to Teflon beaker. This can be done using hands with clean gloves for each sample.
11.4.8 Place Teflon beakers on hotplate (temperature ~ 300° C)
11.4.9 Slowly add between 50 and 100 mL of HF. Acid should be added until the filter is dissolved.
11.4.9.1 Caution: this is an extremely exothermic reaction.
11.4.10 Cook to dryness.
11.4.11 Proceed to section 11.9 for digestion instructions.

11.5 Flora and Fauna - Muffling
11.5.1 Label beaker/crucible is a TEXPEN® or metal marker to ensure identification post muffling.
11.5.2 Pre-weigh labeled beaker/crucible and record weight.
11.5.3 Weigh sample into beaker/crucible.
11.5.4 Prepare a Method Blank and LCS using 0.5 ml of 1.25M Calcium nitrate in a beaker.
11.5.5 Spike LCS and, MS or MSD, with 10 - 40 dpm of standard, depending on the requested target isotopes.
11.5.5.1 A soil reference material can be used for the LCS when the requested target isotopes are present.
11.5.5.2 See Client Requirement sheet to determine which LCS is needed.
11.5.6 Add 5 - 40 dpm of each tracer to all samples, blanks, LCSs, and MS/MSDs in the analytical batch, that corresponds to target isotopes requested.
11.5.6.1 If samples are to be analyzed at a dilution, add the tracer after the digestions, unless client requirements specifically stipulate otherwise.
11.5.7 Cover samples with concentrated nitric acid and allow samples to sit overnight.
11.5.7.1 This will help them break down.
11.5.8 Gently heat samples on hotplate to dryness.
11.5.8.1 This converts samples to nitrates, which help destroy the sample in the muffle furnace in step 11.5.9.
11.5.9 Place sample beakers in muffle furnace and cover with a ribbed watch glass.
11.5.9.1 Ribbed watch glasses help ensure organic material is being destroyed.
11.5.10 Heat samples in a muffle furnace at approximately 600 ° C for 4 hours to reduce samples.
11.5.11 Allow samples to cool to room temperature.
11.5.12 Proceed to section 11.9 for digestion instructions.

11.6 Oils, greases, and solvents - Muffling
11.6.1 Label beakers/crucibles with a TEXPEN® or metal marker to ensure identification post muffling.
11.6.2 Pre-weigh labeled beaker/crucible and record weight.
11.6.3 Add enough ashless powder into the bottom of a crucible/beaker to absorb the oil and/or grease of the sample, and the tracer and spikes used.
11.6.4 Weigh the sample into the beaker/crucible and cover with more ashless powder.
11.6.5 Prepare Blank and LCS crucible with the same amount of ashless powder.
11.6.6 For the LCS, MS, or MSD add 10-40 dpm of standard, depending on the requested isotopes.
11.6.7 Add 5 - 40 dpm of each tracer to all samples and QC in the analytical batch that corresponds to target isotopes requested.
11.6.7.1 If samples are to be performed at dilution, add the tracer after the digestions, unless client requirements specifically stipulate otherwise.
11.6.8 Heat samples in a muffle furnace using the following sequence.
11.6.8.1 Ramp the heat in the muffle furnace at 3 ° C a minute up to approximately 160 ° C.
11.6.8.2 Heat samples for 45 minutes at approximately 160 ° C.

11.6.8.3 Ramp the heat in the muffle furnace at 0.9 ° C per minute up to 600 ° C.
11.6.8.4 Heat to approximately 600 ° C for 5-6 hours to reduce samples.
11.6.9 Allow samples to cool to room temperature.
11.6.10 Proceed to section 11.9 for digestion instructions.

11.7 Wet Ashing – Non Muffling Preparation
11.7.1 Wet Ashing is used for samples that were not muffled which have a noticeable amount of organics present.
11.7.2 Initiate sample preparation worksheet.
11.7.3 Pre-weigh empty labeled beaker.
11.7.4 Aliquot an appropriate amount of sample.
11.7.4.1 The amount of sample utilized is determined by the reporting limit and availability. Contact manager/supervisor for instruction.
11.7.5 Weigh sample aliquot and record weight.
11.7.6 Prepare a Method Blank and LCS using 0.5 ml of 1.25M Calcium nitrate.
11.7.7 Spike LCS and, MS or MSD, with 10 - 40 dpm of standard, depending on the requested target isotopes.
11.7.7.1 A soil reference material can be used for the LCS when the requested target isotopes are present.
11.7.7.2 See Client Requirement sheet to determine which LCS is needed.
11.7.8 Add 5 - 40 dpm of each tracer to all samples, blanks, LCSs, and MS/MSDs in the analytical batch, that corresponds to target isotopes requested.
11.7.8.1 If samples are to be performed at dilution, add the tracer after the digestions, unless client requirements specifically stipulate otherwise.
11.7.9 Add 20 mL of concentrated Nitric acid.
11.7.10 Place samples on a hot plate on low heat.
11.7.11 Slowly add 1-2 mL of peroxide to the samples.
CAUTION: Samples can bubble vigorously.
11.7.12 Heat for 2- 5 minutes until bubbling slows.
11.7.13 Repeat steps 11.7.11 and 11.7.12 until dark samples become lighter in appearance, indicating the destruction of the organics.
11.7.14 Heat to dryness.
11.7.15 Proceed to section 11.9 for digestion instructions.

11.8 Leaching – Non Muffling Preparation
11.8.1 The Leaching procedure is used for sample matrices not identifiably high in organic matter and not conducive to muffling. Method Blanks and QC samples are initiated after the leaching procedure.
11.8.2 Aliquot an appropriate amount of sample into a labeled 500 mL or 1L poly container complete with lid. Alternatively, the entire sample content may be acid leached. Contact manager/supervisor for instruction.
11.8.3 Record sample weight on RadCapture spreadsheet.
11.8.3.1 If the sample is a filter the weight is 1.
11.8.4 Add a known amount of nitric acid.
11.8.4.1 Typically, the amount of the acid is 250mL or 500 mL.
11.8.4.1.1 The molarity of the acid will vary depending on the purpose of the leach and the matrix. If the purpose is a surface leach, the molarity of the nitric acid should be 1M.
11.8.5 Record the amount of nitric acid used on the RadCapture spreadsheet.
11.8.6 Allow sample to leach for 2 hours, shaking every 15 minutes. Do not completely close lids, in order to allow gases to escape.
11.8.7 Samples that do not totally dissolve need to be decanted into another labeled poly container.
11.8.8 Aliquot the equivalent amount of leachate to represent 1-2 grams of sample.

11.8.8.1 For example, if 50 grams of sample is leached with 500ml of leaching solution, the aliquot would be 10-20 ml for each radiological analysis requested.

11.8.9 Prepare a Method Blank and LCS using 0.5 ml of 1.25M Calcium nitrate.

11.8.10 Spike LCS and, MS or MSD, with 10 - 40 dpm of standard, depending on the requested target isotopes.

11.8.10.1 The MS/MSD is performed by taking an additional aliquot of leachate and fortifying it w/ the requested target isotopes.

11.8.11 Add 5 - 40 dpm of each tracer to all samples, blanks, LCSs, and MS/MSDs in the analytical batch, that corresponds to target isotopes requested.

11.8.12 Proceed to section 11.9 for digestion instructions

11.9 Mod Block Total Digestion

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

11.9.1.1 For samples that require dilutions, where there has been no spiking and tracing prior to muffling, the 5ml reflux with HCL is not needed. Transfer samples into labeled digestion vessel using concentrated Nitric acid. Add 5ml concentrated Hydrochloric Acid and proceed to 11.9.3.

11.9.2 Transfer the samples to properly labeled mod block tubes with concentrated Nitric acid. Try to keep the total amount of acid under 15 mL.

NOTE: Label the mod block tubes with a permanent black sharpie. Any other color ink degrades in the acid fumes and tubes will be unidentifiable.

11.9.3 Add 10 mL of concentrated Hydrofluoric acid.

11.9.4 Place samples into the Mod Block. Set the temperature setting to 119˚C. Set the timer to 4 hours. Allow samples to cool approximately 30 minutes.

11.9.5 Repeat steps 11.9.3 and 11.9.4.

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

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

11.9.8 Using 3M nitric acid, transfer samples to properly labeled centrifuge tubes.

11.9.9 Proceed directly to the appropriate column SOP.

11.10 Perchloric Acid Digestion

11.10.1 This is an alternative digestion to the Mod Block digestion in section 11.9. Consult manager/supervisor to determine when appropriate to use this alternative.

11.10.2 Place samples and QC in Teflon beakers.

11.10.3 Add 10 mL of Concentrated Perchloric acid, 10 mL of Concentrated Nitric acid and 25 mL of Concentrated Hydrofluoric acid.

11.10.4 Digest to near dryness in fume hood (moist bead).

CAUTION – Perchloric acid is a strong oxidizer. Contact with other material may cause fire or explosion. Corrosive. Causes severe irritation and burns to every area of contact. Harmful if swallowed or inhaled.

11.10.4.1 If sample has large amounts of residue repeat step 11.10.3 and 11.10.4 until the sample residue no longer changes in appearance.

11.10.4.2 For samples with large amounts of dark organics, repeat digestion with 10 mL of concentrated Perchloric acid and cover with a Teflon beaker cover.

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

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

11.10.6 Proceed directly to the appropriate column SOP.

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 LQM. Specific analysis calculations are given in the applicable analysis 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.

13.2 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

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

13.2. Demonstration of Capability
13.2.1. Initial and continuing demonstrations of capability requirements are established in QAM section 18.3.

13.3. Training Qualification
13.3.1. 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.
13.3.2. The analyst must have successfully completed the initial demonstration capability requirements prior to working independently. See requirements in QAM section 18.3.

13.4. Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in QAM section 18.3.

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 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 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 is 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 Sample waste with a Basic pH is generated. All base waste will be accumulated in the appropriate waste accumulation container, labeled as Drum Type "A" or "B".

16.2.1.3 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 TestAmerica Quality Assurance Manual (QAM), current revision.

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

17.3 Associated SOPs:

17.3.1 ST-PM-0002, "Sample Receipt and Chain-of-Custody."
17.3.2 ST-QA-0002, "Standards and Reagent Preparation"
17.3.3 ST-QA-0005, "Calibration and Verification Procedure for Thermometers, Balances, Weights and pipettes"
17.3.4 ST-QA-0036, "Non-conformance Memorandum (NCM) Process."
17.3.5 ST-RC-0003, "Drying and Grinding of Soil and Solid Samples."
17.3.6 ST-RC-0100, "Actinide Coprecipiation"
17.3.7 ST-RC-0232, "Isotopic Thorium and/or Neptunium in Water, Soil, Sludge, and Filters by EIChroM® Teva Separation Resin."
17.3.8 ST-RC-0238, "Isotopic Uranium by EIChroM® Uteva Resin For Water, Soil, Sludge and Filters."
17.3.9 ST-RC-0240, "Isotopic Americium, Curium, Plutonium, Thorium, and Uranium in Water, Soil, Sludge and Filters by EIChroM® Separation Resin."
17.3.10 ST-RC-0241, "Americium, Plutonium, Curium and Uranium in Water, Soil, Sludge and Filters by EIChroM® Uteva and Tru resins."
17.3.11 ST-RC-0242, "Isotopic Thorium and Uranium in Water, Soil, Sludge and Filters by EIChroM® Separation Resins."

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 None.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 The removal of section 11.6.8 regarding hotplates in section 11.0

19.2 Rev. 18; In section 9.2, removed analysis reference for "other solids and non-filters" and inserted instructions for leached analysis.

19.3 Rev. 18; Changed the morality of nitric acid need for a surface leach from 2M to 1M in section 11.8.4.1.1 and removed the stipulation regarding using the 1M solution if there are not heavy metals present.

Title: RADIUM 226 AND RADIUM 228 BY CHEMICAL SEPARATION PREPARATION

Approvals (Signature/Date):

Sarah Hurst 5/3/10 Sarah Hurst — Date Radiochemistry Prep Supervisor	Michael Ridenhower 5/3/10 Michael Ridenhower — Date Health & Safety Manager / Coordinator
Marti Ward 5-3-10 Marti Ward — Date Quality Assurance Manager	Mwland for 5-3-10 Elaine Wild — Date Laboratory Director

This SOP was previously identified as SOP No. ST-RC-0041 Rev. 8

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1.0 SCOPE AND APPLICATION

1.1 This method covers the chemical separation preparation for Radium 226 and Radium 228 by direct measurement of its beta emitting progeny, Actinium (^{228}Ac). It is applicable to liquid (e.g water and wastewater) where complete dissolution and carrier exchange are readily achievable in the laboratory. For media where chemical dissolution is impractical, non-destructive measurement of the three principal photons of ^{228}Ac by gamma spectrometry is better suited.

1.2 This SOP is based on EPA Method 904.0, 903.0, SW9315, and SW9320.

1.3 The barium sulfate precipitate from this procedure contains all radium isotopes.

1.4 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.

1.5 The reporting limits and QC limits are maintained in the Information Management System (QuantIMS). A copy of the SAC containing this information is included in the analytical SOP: ST-RD-0403.

2.0 SUMMARY OF METHOD

2.1 Radium isotopes are collected by coprecipitation with barium and lead sulfate and purified by precipitation from EDTA solution. After a suitable ingrowth period, ^{228}Ac is separated and carried on yttrium oxalate, purified and counted for the presence of total beta radiation. The precipitation and counting are performed in a manner consistent with the time requirements of the 6.13 hour half life of ^{228}Ac. By applying correction factors for ingrowth and decay and appropriately calibrating the beta counter, Radium 228 is quantified. The barium sulfate fraction, minus Radium 224, is counted by Gas Flow Proportional Counting (GFPC) to report Radium 226.

3.0 DEFINITIONS

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

3.2 There are no specific definitions for this procedure.

4.0 INTERFERENCES

4.1 Strontium 90 or other beta emitting radionuclides that are carried by the yttrium oxalate precipitate (i.e. certain mixed fission or activation products) will yield a positive bias to the Radium 228 values.

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.

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5.2 SPECIFIC SAFETY CONCERNS OR REQUIREMENTS

5.2.1 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
Ammonium Hydroxide	Poison Corrosive	50 ppm	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
Acetic Acid, Glacial	Corrosive Flammable	10 ppm (TWA)	Inhalation causes respiratory tract irritation including nasal discharge, hoarseness, coughing, chest pain and breathing difficulty. Skin contact symptoms may include redness or discoloration, swelling, itching, burning or blistering of skin. Eye symptoms include irritation, burning sensation, pain, watering, and/or change of vision.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-(TWA) 4 ppm-(STEL)	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-(TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			
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 planchets

6.6 Glassware, Beakers

6.7 Syringe, 20ml; .45mm filter

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, 17.4N: glacial CH_3COOH (concentrated), specific gravity 1.05, 99.8%.

7.4 Ammonium hydroxide, 15N: NH_4OH (concentrated), sp. gr. 0.90, 56.6%.

7.5 Ammonium oxalate, 5%: Dissolve 2g $(NH_4)_2C_2O{\cdot}H_2O$ in water and dilute to 40ml.

7.6 Ammonium sulfate, 200mg/ml: 400g $(NH_4)_2SO_4$ in water and dilute to 2000ml.

7.7 Ammonium sulfide, 2%: Dilute 5ml $(NH_4)_2S$, (20-24%), to 50 ml water; total volume 50ml.

7.8 Barium carrier (standardized), 33.9 mg/ml, NIST Traceable

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

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

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

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

7.8.5 Wash precipitate with 15 ml DI water, centrifuge and decant the supernate.

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

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

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

7.9 Citric acid, 1M: Dissolve 192g $C_6H_8O_7{\cdot}H_2O$ in water and dilute to 1000ml.

7.10 EDTA reagent basic (0.25M) - dissolve 20g NaOH in 750ml water, heat and slowly add 93g [ethylenedinitrilo] tetraacetic disodium salt, $(C_{10}H_{14}O_8N_2Na_2{\cdot}2H_2O)$ while stirring. Dilute to 1 liter.

7.11 Lead carrier, 15mg/ml: Dissolve 23.97g $Pb(NO_3)_2$ in water, add 5ml 16N HNO_3 and dilute to 1000ml with water.

7.12 Lead carrier, 1.5mg/ml: Dilute 10ml lead carrier, (15mg/ml), to 100ml with water.

7.13 Methyl orange indicator, 0.1%: Dissolve 0.1g methyl orange indicator in 100ml water.

7.14 Nitric acid, 16N: HNO_3 (concentrated), specific gravity 1.42, 70.4%.

7.14.1 Nitric acid, 6N: Mix 3 volumes 16N HNO_3 (concentrated) with 5 volumes of water.
7.14.1.1 Nitric acid, 2N: Mix 1 volume 6N HNO_3 with 2 volumes of water.

7.15 Sodium hydroxide, 18N: Dissolve 72g NaOH in water and dilute to 100ml.

7.16 Sodium hydroxide, 10N: dissolve 40g NaOH in water and dilute to 100ml.

7.17 Strontium carrier, 9 mg/ml: Dissolve 24.16g $Sr(NO_3)_2$ in water and dilute to 1 liter.

7.18 Sulfuric acid, 18N: concentrated.

7.19 Yttrium carrier(standardized), NIST traceable

7.19.1 If the Yttrium carrier is not already standardized, standardize the yttrium carrier solution using the following procedure.

7.19.1.1 Prepare 10 samples of the carrier solution. Perform the following steps on each vial.
7.19.1.2 Add 5 ml DI water to a 50 ml centrifuge tube.
7.19.1.3 Add 2 ml concentrated nitric acid.
7.19.1.4 Add 1 drop of MCP indicator (0.1%).
7.19.1.5 Add 1.0 ml of yttrium carrier (10 mg/ml, Y) pipette.
7.19.1.6 Add 5 ml of 0.4 M oxalic acid.
CAREFULLY add concentrated ammonium hydroxide dropwise until a reddish yellow end point is obtained.
NOTE: The pH must be between 1.7 - 1.9 to assure a uniform 9·H_2O hydrate yttrium oxalate precipitate. The yttrium oxalate will have precipitated.
7.19.1.7 Heat the solution for approximately 5 –10 minutes in a hot water bath.
7.19.1.8 Cool the solution for approximately 5 – 10 minutes in an ice water bath.
7.19.1.9 Centrifuge.
7.19.1.10 Decant supernate into the appropriate waste container.
7.19.1.11 Add approximately 20 ml of DI water to the precipitate.
7.19.1.12 Vortex and centrifuge.
7.19.1.13 Decant supernate into the appropriate waste container.
7.19.1.14 Add approximately 20 ml of DI water to the precipitate.
7.19.1.15 Vortex and Centrifuge.
7.19.1.16 Decant supernate into the appropriate waste container.
7.19.1.17 Add approximately 5 ml of D.I. water, slurry the precipitate.
7.19.1.18 Pre-weigh a planchet and record the tare weight on the sample worksheet.
7.19.1.19 Transfer the slurry to the planchet.
7.19.1.20 Dry the planchet on a hotplate.
7.19.1.21 Remove from heat and cool to room temperature in a desiccator.
7.19.1.22 Weigh sample to determine yttrium yield.
7.19.1.23 Repeat heating and cooling in the desiccator until a constant weight is obtained as determined by two consecutive measurements where the weight differences are ± 5% or less.
7.19.1.24 Record gross and final weights in the Rad Standards Log.

7.20 Yttrium carrier, 9 mg/ml: Dilute 50 ml yttrium carrier, to 100 ml with water.

7.21 Strontium-yttrium mixed carrier, 0.9 mg/ml Sr^{+2}; 0.9 mg/ml Y^{+3}:
- 7.21.1 Solution A: Dilute 10.0 ml yttrium carrier, (18 mg/ml), to 100 ml
- 7.21.2 Solution B: Dissolve 0.4348 g $Sr(NO_3)_2$ in water and dilute to 100 ml.
- 7.21.3 The mixed carrier is made by adding equal volumes to a flask.

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

7.23 Radium 228, standard 20-25 dpm, 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.

8.4 Sample hold time is 180 days from collection.

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>, 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**
- 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 acidified with 2mL of nitric acid.
- 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 and Radium 228.
9.3.4 For Soil analyses, the LCS is comprised with Radium 226 and Radium 228.

9.4 **Matrix Spike/Matrix Spike Duplicate**
9.4.1 A Matrix Spike (MS) 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. 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.
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. Ensure that the sample is at or below a pH of 2. 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 ST-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 allow to 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 and Radium 228 spike to LCS and MS/MSD, if applicable.
11.2.5 Carefully add 5ml concentrated nitric acid, 5ml concentrated hydrochloric acid and 10ml concentrated Hydrofluoric acid.
11.2.6 Digest in mod block at >110° for four hours or until dry.
11.2.7 Carefully add 10ml concentrated nitric acid, 10ml concentrated hydrochloric acid and 5ml concentrated Hydrofluoric acid.
11.2.8 Digest in mod block at >110° for four hours or until dry.
11.2.9 Dissolve with 10ml HNO_3 and 10ml HCl, return to mod block for 30min.
11.2.10 Transfer to 250 ml beakers with 4M HNO_3. Dilute to 200mL with DI water.

11.3 Precipitation
11.3.1 Add 1M citric acid in ratio of 5 ml per liter. Mix thoroughly. Add methyl orange indicator until red color persists.
11.3.2 For Waters perform steps 11.3.2.1, 11.3.2.2 and 11.3.2.3; for soils perform step 11.3.2.1
11.3.2.1 Add 2.5 ml lead carrier (15 mg/ml), 2ml strontium carrier (10 mg/ml), and 1 ml yttrium carrier (9 mg/ml); stir well.
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 and Radium 228.
11.3.3 Heat to just boiling and maintain at this temperature for about 30 minutes.
11.3.4 Add 15N ammonium hydroxide until a definite yellow color is obtained, then add a few drops more.
11.3.5 **Face shield must be worn during this process.** Precipitate lead and barium sulfates by adding 18N sulfuric acid until the red color reappears, then add 0.25 ml more.
11.3.6 Add 5 ml ammonium sulfate (200 mg/ml) for each liter of sample. Stir frequently and keep at a temperature of approximately 90°C for 30 minutes.
11.3.7 Allow precipitate to settle to the bottom of the beaker for a least 6 hours.

11.4 In-growth
11.4.1 Decant the supernate and discard, taking care to avoid disturbing the precipitate. Transfer precipitate to a 50 ml centrifuge tube, taking care to rinse last particles out of beaker with DI water. Centrifuge and discard supernate.
11.4.2 Wash the precipitate with 10ml 16N HNO_3, vortex, centrifuge, and discard supernate. Repeat this step if precipitate visually appears larger than that in the Blank and LCS.
11.4.3 Wash the precipitate with 10ml D.I.H_2O, vortex, centrifuge, and discard supernate.
11.4.4 Add 20 ml basic EDTA reagent; vortex thoroughly, and heat in a hot water bath (approximately 80°C) until precipitate dissolves. There should be no precipitate remaining in the tube.
11.4.4.1 If insoluble solids remain in the tube after addition of EDTA, confirm that the pH is > 10.
11.4.4.2 If pH > 10, centrifuge and decant supernate into a clean, labeled 50 mL centrifuge tube. Discard insoluble residue.
11.4.5 Add 1 ml strontium-yttrium mixed carrier and stir thoroughly.
11.4.5.1 Add a few drops 10N NaOH if any precipitate forms.
11.4.6 Add 2ml ammonium sulfate (200mg/ml) and stir thoroughly.
11.4.7 Add 17.4N acetic acid until barium sulfate precipitates, then add 2 ml more.
11.4.8 Digest in a hot water bath until precipitate settles.
11.4.9 Centrifuge and discard supernate.
11.4.10 Add 20 ml basic EDTA reagent, vortex thoroughly, and heat in a hot water bath until precipitate dissolves.

11.4.11 Add 2ml ammonium sulfate (200mg/ml) and stir thoroughly.
11.4.12 Add 17.4N acetic acid until barium sulfate precipitates, then add 2 ml more.
11.4.13 Digest in a hot water bath until precipitate settles.
11.4.14 Centrifuge and discard supernate.
11.4.15 Record the date and time of last barium sulfate precipitation. This is the beginning of the ^{228}Ac in-growth time.
11.4.16 Add 20 ml basic EDTA reagent, vortex thoroughly, and heat in a hot water bath until precipitate dissolves.
11.4.17 Add 1.0 ml standardized yttrium carrier and 1 ml lead carrier (1.5 mg/ml).
11.4.17.1 If any precipitate forms, dissolve it by adding a few drops of 10N NaOH.
11.4.17.2 If Radium 228 analysis is requested, cap the tube and allow it to age at least 36 hours.
11.4.17.3 If Radium 226 analysis is requested, cap the tube and allow it to age (in-growth period is 14 or 21 days, see sample log in sheet).

11.5 Lead Scavenge Clean-up
11.5.1 Add 0.3 ml ammonium sulfide and stir well. Add 10N sodium hydroxide drop-wise with vigorous stirring until lead sulfide precipitates, then add 10 drops more. Vortex. Centrifuge and decant supernate into a clean labeled tube.
11.5.2 Add 1 ml lead carrier (1.5 mg/ml), 0.1 ml ammonium sulfide, and a few drops 10N sodium hydroxide.
11.5.3 Centrifuge and filter supernate through 0.45 mm syringe filter into a clean labeled tube. Discard filter.
11.5.3.1 The half life ^{228}Ac is very short. Check with count room staff on scheduling of the GFPC. **Do NOT proceed with the remaining steps of this procedure until authorized by the count room GFPC analysts or Radiochemistry manager.**

11.6 Out of In-growth
11.6.1 Once yttrium hydroxide is precipitated, the analysis must be carried to completion to avoid excessive decay of ^{228}Ac.
11.6.2 Add 10 ml 18N sodium hydroxide, stir well and digest in a hot water bath, 70-85°C, until yttrium hydroxide coagulates, usually about 5 minutes. Centrifuge and decant supernate into a clean, labeled 50 ml centrifuge tube. Save for barium yield determination, Step 11.7.1

11.7 Yttrium Yield (for Radium 228)
11.7.1 Weigh a stainless steel cleaned planchet. Record weight in the RadCapture spreadsheet.
11.6.1.1 The cleaned planchet has been processed in accordance with SOP: ST-RC-0002. See SOP for additional information.
11.7.2 Record time of yttrium hydroxide precipitation; this is the end of the ^{228}Ac in-growth time and beginning of ^{228}Ac decay time.
11.7.3 Dissolve the precipitate from step 11.5.2 in 2ml 6N nitric acid. Vortex and add 5ml water and precipitate yttrium hydroxide with 3 ml 10N sodium hydroxide. Heat and stir in a hot water bath until precipitate coagulates. Centrifuge and discard supernate.
11.7.4 Dissolve precipitate with 1 ml 2N nitric acid. Vortex.
11.7.4.1 If solution is still cloudy adds 2N nitric acid drop wise until the solution clears.
11.7.5 Dilute to 5ml with DI water and add 2ml 5% ammonium oxalate. Centrifuge and discard supernate.
11.7.6 Wash precipitate with 5 mL DI water.
11.7.7 Vortex, centrifuge and discard supernate.
11.7.8 Repeat sections 11.7.6 and 11.7.7.
11.7.9 Plating

11.7.9.1 To determine yttrium yield, quantitatively transfer the precipitate to the previously weighed stainless steel cleaned planchet using a minimal amount of water.
11.7.9.2 Dry on a hot plate.
11.7.9.3 Upon dryness, let cool in a desiccator for a minimum of 15 minutes and then weigh planchet.
11.7.9.4 Record the weight in the RadCapture worksheet to determine the chemical yield of the yttrium carrier solution.

11.8 Barium Yield (for Radium 226)
11.8.1 Weigh a stainless steel cleaned planchet. Record weight in the RadCapture spreadsheet.
11.8.1.1 The cleaned planchet has been processed in accordance with SOP: ST-RC-0002. See SOP for additional information.
11.8.2 To the supernate from Step 11.5.2, add 7 ml 16N nitric acid and 2ml ammonium sulfate (200mg/ml), stirring well after each addition. Add 17.4N acetic acid until barium sulfate precipitates, then add 2mls more. Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate.
11.8.3 Add 20ml basic EDTA reagent, vortex and heat in a hot water bath until precipitate dissolves. Add a few drops 10N NaOH if precipitates does not readily dissolve.
11.8.4 Add 2 ml ammonium sulfate (200 mg/ml) and stir thoroughly. Add 17.4N acetic acid until barium sulfate precipitates, then add 2 ml more.
11.8.5 Record date and time of $BaSO_4$ precipitate in the RadCapture data sheet.
11.8.6 Digest in a hot water bath until precipitate settles. Centrifuge and discard supernate.
11.8.7 Wash precipitate with 10ml water. Vortex, centrifuge and discard supernate.
11.8.8 Repeat wash step.
11.8.9 Plating
11.8.9.1 Transfer the precipitate to a pre-weighed stainless steel cleaned planchet with a minimal amount of water.
11.8.9.2 Heat the planchet again using the hot plate, let cool in a desiccator for a minimum of 15 minutes and then weigh planchet.
11.8.9.3 Record the final weight of the planchet in RadCapture to determine the chemical recovery for the barium carrier solution.

11.9 Submit the planchets to the counting 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 QAM.

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

12.3 Radium 226 and Radium 228 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 QAM section 18.3.

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 QAM section 18.3.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in QAM section 18.3.

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 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 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
- 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 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 8, Method 904.0, Radium 228 in Drinking Water

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

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

17.4 SW-846,"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", Method 9320, Radium 228

17.5 TestAmerica St. Louis Quality Assurance Manual (QAM), current revision.

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

17.7 Associated SOPs:
- 17.7.1 ST-PM-0002, Sample receipt and Chain of Custody
- 17.7.2 ST-QA-0002, Standard and Reagent Preparation
- 17.7.3 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
- 17.7.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process
- 17.7.5 ST-RC-0002, Planchet Preparation for Radiochemistry and Radiological Screening Analysis
- 17.7.6 ST-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
- 17.7.7 ST-RD-0403, Gas Flow Proportional Counting (GFPC) Analysis

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 After initial precipitation, TestAmercica St. Louis decants the supernate after the precipitate has been allowed to settle for at least six hours, as opposed to the EPA Method 904 which requires filtration to isolate the precipitate.

18.2 At the point of in-growth of Actinium-228, TestAmerica St. Louis utilizes either a 14 days or 21 day in-growth depending on the client's request for wastewater and 21 days for drinking water before finishing the procedure.

18.3 TestAmerica St. Louis counts the Barium sulfate fraction (minus the Ra-224) by GFPC to report Radium-226; an option documented in section 10.5 of the EPA Method 903.0.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Annual Review, No Changes.

Title: CARBON-14 DETERMINATION USING POSITIVE INERT GAS FLOW

Approvals (Signature/Date):

01-19-11	1-19-11
Sarah Hurst — Date	Michael Ridenhower — Date
Separations Prep Supervisor	Health & Safety Manager / Coordinator
1-19-11	1/19/11
Marti Ward — Date	Elaine Wild — Date
Quality Assurance Manager	Laboratory Director

No previous revision

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1.0 SCOPE AND APPLICATION

1.1 This SOP provides detailed instructions for the determination of Carbon-14 by Liquid Scintillation Spectrometry in waters, soils and filters.

1.2 This SOP is based on method EERF C-01-1.

1.3 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Information Management System (QuantIMS). A copy of the Structure and Analysis Code (SAC), which lists this information, is included in the appendix of SOP ST-RD-0302.

2.0 SUMMARY OF METHOD

2.1 Carbon present in the sample matrix is oxidized to carbon dioxide by addition of permanganates and sulfuric acid. The carbon dioxide passes through a water trap to cleanse the gas of any potential interference from tritium. An inert gas is used to purge the sample of any remaining carbon. The carbon dioxide is then trapped in Carbosorb scintillation cocktail. After addition of Permafluor cocktail to the vial the samples are ready for counting.

3.0 DEFINITIONS

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

4.0 INTERFERENCES

4.1 Tritium is a potential interference, but should be collected in the water trap. Also there is a small potential that the sample could contain more carbon than can be loaded into the cocktail, in this event a low bias may be realized. To aid in detecting such overloading, the Carbosorb is loaded only up to approximately 60% of its capacity when generating the quench curve. Samples resulting in a quench parameter off of the quench curve (high tSIE) may then be re-prepped using a smaller aliquot.

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
Nitromethane is extremely flammable. Store and use only in a well ventilated area away from heat, sparks, or flame. Can be an explosion hazard, especially when heated. Can react violently with shock, friction or heat.

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
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3 (TWA)	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Potassium Permanganate	Oxidizer	5 Mg/M3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
Potassium Persulfate	Oxidizer	None	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Causes irritation to skin and eyes. Symptoms include redness, itching, and pain. May cause dermatitis, burns, and moderate skin necrosis.
Nitromethane	Flammable Explosive	100 ppm, (TWA)	Eye, skin, respiratory tract 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 (see attachment for letter references)

6.1 Glass stopper (A)
6.2 Addition Funnel (B)
6.3 Straight adapter (C)
6.4 Round Bottom Flask (D)
6.5 Gas Washing Tube (E)
6.6 Plastic Tubing (F)
6.7 Pipettes, glass disposable
6.8 Adjustable dispensers
6.9 Analytical Balance
6.10 Pipettes, variable
6.11 Liquid Scintillation vials (plastic).
6.12 Plastic glassware joint clamps
6.13 Heating mantles
6.14 Inert gas (Nitrogen, Helium of other carbon/water free gas

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 DI water, obtained from the Milli-Q unit.

7.3 Carbo-Sorb E, 6013729 Packard Bioscience.

7.4 Perma-Fluor E+, 6013187 Packard Bioiscience.

7.5 Potassium Permanganate 5%,
7.5.1 Add 5g of potassium permanganate to 100mL of DI water. Mix.

7.6 Potassium Persulfate 5%, LC20080-2 LabChem Inc.

7.7 Sulfuric Acid 10%
7.7.1 Dilute 100mL ofconcentrated Sulfuric Acid to 500mL of DI water. Mix.

7.8 Oxidizing Cocktail
7.8.1 Mix equal parts 5% potassium persulfate, 5% potassium permanganate and 10% sulfuric acid.
7.8.2 Prepare fresh daily.

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 not chemically preserved.

8.4 Sample hold time is 180 days from collection.

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 <u>method blank</u>, a <u>Laboratory Control Sample</u> (LCS), <u>Matrix Spike</u> (MS) and <u>Sample Duplicate</u> (SD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 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.

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

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 Carbon 14.

9.3.4 For Soil analyses, the LCS is comprised of DI water fortified with Carbon 14.

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 the utilization of a LCSD to demonstrate 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 A background sample is submitted for counting with each batch.

10.1.1 The background sample consists 10mL of Carbo-Sorb E and 10mL of Permafluor E+.

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

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

10.4 For analytical instrumentation calibration see procedure in ST-RD-0302 for Liquid Scintillation Counting.

11.0 PROCEDURE

11.1 Water Preparation
- 11.1.1 Ensure that the sample container is capped tightly and shake it thoroughly, in accordance with SOP, ST-QA-0038.
- 11.1.2 Weigh sample into round bottom flask (D).
  - 11.1.2.1 Record sample volume on laboratory prep spreadsheet

11.2 Soil Preparation:
- 11.2.1 Homogenize sample in accordance with SOP, ST-QA-0038.
- 11.2.2 Weigh sample into a round bottom flask (D).
  - 11.2.2.1 Record sample volume on laboratory prep spreadsheet.

11.3 Spike the LCS and Matrix spike aliquots

11.4 Set up the glassware system (see attachment) on a secured lattice using metal clamps.

11.5 Place heating mantle under the round bottom flask (D).

11.6 Connect addition funnel to inert gas tank.

11.7 Plastic tubing will connect adapter (C) to the gas washing tube (E) and secure with clamps. Add approximately 5mL DI water to the gas washing tube. The adapter needs to extend well into the sample.

11.8 Attach more plastic tubing to the lower arm of the gas washing tube. This will connect toa glass pipette that will be placed in a plastic scintillation vial with 10mL of Carbo-Sorb E to collect carbon gas. For full apparatus set up see Attachment 1.

11.9 Add oxidizing cocktail to the addition funnel (B). The amount of oxidizing cocktail needed will depend on the amount of sample used.

- 11.9.1 For 75mL or 1g of sample, use 25mL of oxidizing cocktail
- 11.9.2 For 250mL or 5g of sample use 45mL of oxidizing cocktail.

11.10 Slowly turn the know on the addition funnel to release the oxidizing cocktail into the flask. Turn the inert gas on to help push the oxidizing cocktail into the flask. Turn gas off once all the cocktail is transferred.

11.11 Turn the heating mantle on and bring sample to a light boil for approximately 20 minutes.

- 11.11.1 The heat setting may need to be turned down partway through to keep a gentle boil. Soils may not need the full 20 minutes to boil.

11.12 Turn the inert gas on again to purge the sample of the remaining carbon that may be trapped.

11.13 Turn off the inert gas and heating mantle. Allow all bubbling to cease before dismantling the apparatus. Removing any part will relieve pressure too quickly.

11.14 Take pipette out of the scintillation vial and tap the tip to remove excess Carbo-Sorb

11.15 Add 10mL of Permafluor E+ to labeled scintillation vial

11.16 Screw cap onto vial and shake well.

11.17 Submit vials and all paperwork to the count room.

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 QAM. Specific analysis calculations are given in the applicable analysis 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.

13.2 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.

13.3 Refer to SOP ST-RD-0302, “Calibration and Opertation of a Liquid Scintillation Counter.”

14.0 METHOD PERFORMANCE 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
- 14.2.1 Initial and continuing demonstrations of capability requirements are established in QAM section 18.3.

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 QAM section 18.3.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in QAM section 18.3.

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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 QAM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods.

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 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 Radiochemical Determination of Carbon-14 in Aqueous Samples, `Method C-01-1, Eastern Environmental Radiation Facility Radiochemistry Procedure Manual, EPA 520/5-84-006 (1984)

17.2 TestAmerica Quality Assurance Manual (QAM), current revision

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

17.4 Associated SOPs, current revisions:

17.4.1 ST-QA-0002, Standards and Reagent Preparation
17.4.2 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.4.3 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.4.4 ST-QA-0038, Procedure for Compositing and Subsampling
17.4.5 ST-PM-0002, Sample Receipt and Chain of Custody
17.4.6 ST-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
17.4.7 ST-RD-0302, Calibration and Opertation of a Liquid Scintillation Counter

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 Potasium persulfate is added to the reaction mixture in addition to potassium permanganate.

18.2 A yield monitor is not employed for Carbon-14 analysis.

18.3 The trap used to capture carbon dioxide is Carbo-Sorb E. This product is utilized in Perkin Elmer equiptment designed for Carbon-14 analysis and is a very efficient carbon dioxide trap.

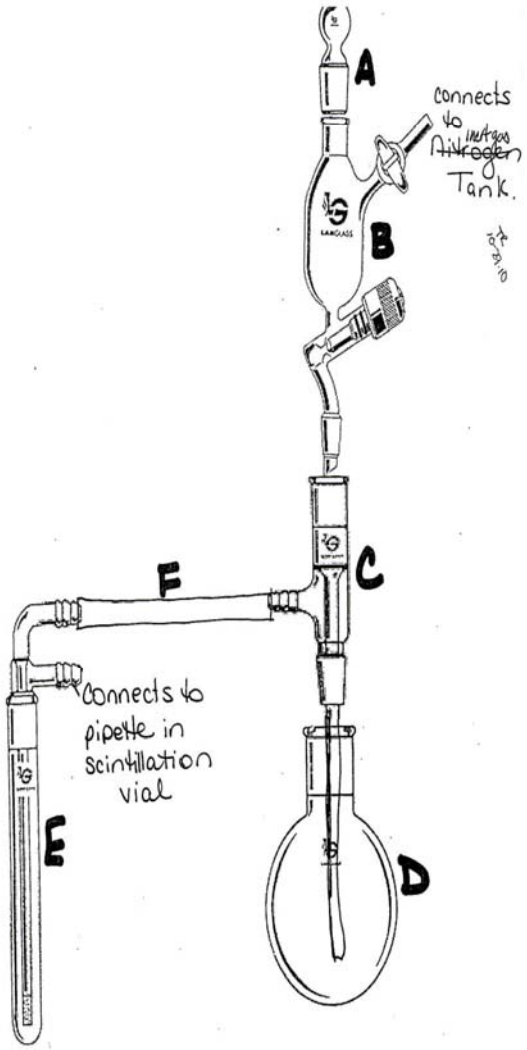
18.4 When necessary a tritium trap is employed to prevent contamination of the Carobsorb E with tritium.

19.0 CHANGES FROM PREVIOUS REVISION

19.1 Initial version of SOP.

ATTACHMENT 1

Company Confidential & Proprietary


A
connects to ~~Nitrogen~~ inert gas Tank.
B
C
F
Connects to pipette in scintillation vial
E
D

Title: AMERICIUM, PLUTONIUM, CURIUM, AND URANIUM IN VARIOUS MATRICIES BY ElChroM® Uteva AND TRU RESINS (WITH VACUUM BOX SYSTEM)

Approvals (Signature/Date):

Sarah Hurst 11-29-10	Michael Ridenhower 11-29-10
Sarah Hurst — Date	Michael Ridenhower — Date
Separations Supervisor	Health & Safety Manager / Coordinator
Marti Ward 11-29-10	Elaine Wild 11/29/10
Marti Ward — Date	Elaine Wild — Date
Quality Assurance Manager	Laboratory Director

This SOP was previously identified as SOP No. ST-RC-0241 Rev. 7

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes an **alternate** sequential analysis technique which may be used for certain project samples. For **routine** Americium, Plutonium, Curium and Uranium actinide sample preparation see SOP: **ST-RC-0240**.

1.1.1 If only Uranium analysis is requested, see SOP: ST-RC-0238.

1.2 This method is based on Eichrom Technologies Inc. analytical procedure "ACS03 VBS: Americium, Plutonium and Uranium in Soil (with Vacuum Box System)" and "ACS04 Americium/Lanthanide Separation in Soil".

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 reporting limits, minimum detectable activities and QC limits are maintained in the Information Management System (QuantIMS). A copy of the SAC is included in the analytical SOPs to demonstrate this information.

2.0 SUMMARY OF METHOD

2.1 This SOP describes the method for separation of americium, 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 ST-RC-0004, Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis. Tracers are used to monitor chemical recoveries and correct results to improve precision and accuracy.

3.0 DEFINITIONS

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

3.2 Tracer - A known amount of either ^{232}Uranium, ^{241}Americium, ^{243}Americium, ^{242}Plutonium, or ^{236}Plutonium is added to each sample to determine chemical yield. The tracer serves as an internal standard, which is used to calculate the activity of the target isotopes.

4.0 INTERFERENCES

4.1 Actinides with unresolvable alpha energies such as Americium-241and Plutonium-238 or Neptunium-237 and Uranium-234 must be chemically separated to enable measurement. This method separates these isotopes effectively.

4.2 Very high levels of phosphate in the sample may cause interference. Adjusting the amount of phosphate added to coprecipitate the actinides may be necessary in these cases.

4.3 Polonium-210 decays in the Uranium-232 region, and can cause high tracer recoveries if not removed from the Uteva cartridge/column, using a 25 ml 3 M HNO_3 wash after the load solution has run through the cartridge/column.

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 Hydrofluoric Acid is a corrosive and a poison. Extremely hazardous as a liquid or vapor. Requires special first aid should contact occur. Analysts must be trained in HF acid first aid prior to using this procedure. Reaction with certain metals generates flammable and potentially explosive hydrogen gas.

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
Ammonium Hydroxide	Poison Corrosive	50 ppm (NH3)	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
Aluminum nitrate	Oxidizer	2 mg/m3 (TWA) Soluble salts as Al	Inhalation symptoms may include coughing, shortness of breath. Symptoms of skin contact include redness, itching, and pain. Eye contact causes irritation, redness, and pain.
Ammonium oxalate	Poison Corrosive	None established	Symptoms of inhalation exposure include nervousness, cramps and central nervous system depression. Skin contact causes redness, itching, and pain. Eye contact causes irritation, redness, and pain.
Calcium nitrate	Oxidizer	None established	Inhalation symptoms include coughing and shortness of breath. Skin contact symptoms include redness, itching, and pain. Eye contact causes irritation, redness and pain.
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.

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.
Oxalic Acid	Corrosive	1mg/m3 (TWA) 2mg/m3 (STEL)	Inhalation symptoms include severe irritation and burns of nose, throat, and respiratory tract. Ingestion symptoms include burns, nausea, severe gastroenteritis and vomiting. Skin contact causes severe irritation and burns. Oxalic Acid is an eye irritant.
Sulfamic Acid	Corrosive	No specific exposure limits established.	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. Skin contact produces redness, pain, and severe burns. Eye contact can cause blurred vision, redness, pain, burns, and 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			
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 Pipetter, mechanical

6.7 Fume hood

6.8 Hotplate with stirrer

6.9 Vortex mixer

6.10 pH strips, narrow range

6.11 Vacuum Box, Eichrom part number AC-24-BOX, or equivalent

6.12 Syringe filter, 25 mm acrodisc, 0.45 or 0.70 µm

6.13 Cartridge reservoirs/syringe/funnel-20 ml B-D Luer Lok syringe Part Number 301625 (Fisher part number 14-823-2B), or equivalent.

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

7.3 L (+) Ascorbic acid, reagent grade.
7.3.1 L (+) Ascorbic Acid solution, 2.5g dissolved in 10ml of DI water.

7.4 Ammonium hydrogen phosphate, dibasic (3.2M)
7.4.1 Dissolve 104 grams of $(NH_4)_2HPO_4$ in approximately 200 mL of water, heating gently to dissolve. Dilute to 250 mL with water and mix well.

7.5 Ammonium hydroxide (NH4OH) Reagent.

7.6 Ammonium Bioxalate (hydrogen oxalate) (0.1 M)
7.6.1 Dissolve 6.31 g of $H_2C_2O_4 \bullet 2H_2O$ (oxalic acid) and 7.11 g of $(NH_4)2C2O4 \bullet H2O$ (ammonium oxalate) in 900 mL of water, and dilute to 1 L with water.

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

7.8 Sulfamic Acid
7.8.1 Sulfamic acid solution, NH_2SO_3H, Dissolve 2.0 grams sulfamic acid in 10 mL of DI water

7.9 Hydrochloric acid (12M) - concentrated HCl (sp gr 1.19).
7.9.1 Hydrochloric acid (9M) - Add 1500 mL of concentrated HCl (sp gr 1.19) to 200 mL of water and dilute to 2 liters.
7.9.2 Hydrochloric acid (4M) - Add 667 mL of concentrated HCl to 200mL of water and dilute to 2 liters

7.10 Hydrochloric acid (5M) - 0.05M oxalic acid solution
7.10.1 Dissolve 6.3 grams of oxalic acid dihydrate in approximately 400 mL of water. Add 417 mL of concentrated hydrochloric acid. Cool to room temperature. Dilute to 1 liter with water and mix well.
7.10.2 Hydrochloric acid (1M)- Add 167mL of concentrated HCl (sp gr 1.19) to 200mL of water and dilute to 2 liters.

7.11 Hydrofluoric acid (48-52%) (29M)- concentrated hydrofluoric acid.

7.12 Hydrochloric acid (4 M)- hydrofluoric acid (0.1 M)- Add 333 mL of concentrated HCl (sp gr 1.19) and 3.6 mL of concentrated HF (sp gr 1.2) to 500 mL of water and dilute to 1 L with water. Prepare fresh daily.

7.13 Nitric acid (16M) - concentrated HNO_3 (sp gr 1.42).
7.13.1 Nitric acid solution (3.0M) - Add 382 mL of concentrated HNO_3 (sp gr 1.42) to 200 mL of water and dilute to 2 liters.
7.13.2 Nitric acid solution (0.5M)- Add 32mL of concentrated HNO_3 to 900mL of water and dilute to 1 L with water.
7.13.3 Nitric acid solution (2 M)- Add 127 mL of concentrated HNO_3 to 800 mL of water and dilute to 1 L with water.

7.14 Nitric acid (2M)-sodium nitrite (0.1M) solution- Add 32 mL of concentrated HNO_3 to 200 mL of water, dissolve 1.72 g of sodium nitrite in the solution and dilute to 250 mL with water. Prepare fresh daily.

7.15 Sodium Nitrite, NaNo2 reagent crystals.

7.16 Load solution [Nitric acid (3 M) - aluminum nitrate (1 M)] – Weigh 1500 g $Al(NO_3)_3 \cdot 9H_2O$ in a 4 liter beaker. Add 800 mLof 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.

7.17 Bromocresol Purple indicator solution
7.17.1 Disso1ve 0.20 g of Bromocresol Purple (520.24 F.W.) in 250 mL of water, add one ml of concentrated Ammonium Hydroxide.

7.18 Pu-242 tracer standard, NIST traceable, 10-20 dpm/ml (Pu-236 can also be used).

7.19 Pu-238 and/or Pu-239, NIST traceable.

7.20 Uranium-232 standard, NIST traceable 10-20 dpm/ml.

7.21 Natural Uranium standard, NIST traceable approximately 10-20 dpm/ml.

7.22 Am-243 tracer, NIST traceable, approximately 10-20 dpm/ml.

7.23 Am-243 spike, NIST traceable, approximately 10-20 dpm/ml.

7.24 Am-241 spike, NIST traceable, approximately 10-20 dpm/ml.

7.25 Curium-244 tracer, NIST traceable, approximately 10-20 dpm/ml.

7.26 TRU Resin – 2mL cartridge, 50-100 micron, part number TR-R50-S and TR-R200-S.

7.27 UTEVA Resin- 2mL cartridge, 50-100 micron, part number UT-R50-S and UT-R200-S.

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.

8.4 Sample hold time is 180 days from collection.

8.5 Solid sample requirements are found in SOP ST-RC-0004, “Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical 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 (MB), a Laboratory Control Sample (LCS), and Sample Duplicate (SD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

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

9.2.4 For non-aqueous method blanks, 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 and Pu only, use Calcium nitrate

9.3.4.2 Otherwise 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, filter through a Whatman GF/A (1.6 micron) filter.
- 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 Check radscreen results and customer/site history, if available, to determine sample size.
- 11.2.4 Aliquot 500 to 1000 mL of the filtered sample (or enough to meet required detection limit) into an appropriate size beaker.
  - 11.2.4.1 Typically 500ml for short count and 1000ml for long count
- 11.2.5 Add appropriate tracers or standards. Generally 10 – 20 dpm of each of the 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 Cover each beaker with a watch glass, and place each beaker on a hotplate.
- 11.4.5 Allow the samples to heat to near boiling approximately 30 minutes.
- 11.4.6 Once the samples reach near boiling, take the watch glass off the beaker and turn the heat down to medium.
- 11.4.7 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 uncovered 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 Actinide Separations using Eichrom Resins:

11.5.1 Re-dissolve samples in load solution:

11.5.1.1 Dissolve each calcium phosphate precipitate, soil dissolution residue or evaporated water samples with 15 mL of (load solution) 3 M HNO_3-1.0M $Al(NO_3)_3$,. Vortex sample.

NOTE: Additional aliquots of 5-mL may be necessary to dissolve the sample residue. Do not use more than 30 ml of load solution.

NOTE: If particles are observed or the solution is cloudy, centrifuge the sample at approximately 2000 rpm for 5 minutes. Transfer to a clean labeled centrifuge tube. The precipitates will be discarded.

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

11.5.1.2 Add 2.5 mL of sulfamic acid solution to each solution.

NOTE: If additional load solution was used to dissolve the sample in step 11.5.1.1, add a total of 3 mL of sulfamic acid solution. Swirl to mix.

11.5.1.3 Add 3ml of ascorbic acid to each solution, swirling to mix. Place in hot water bath for approximately 3 minutes. Then a cool water bath until slightly below room temperature.

NOTE: If particles are observed or the solution is cloudy, centrifuge the sample at 2000 rpm for 5 minutes. The supernatant will be transferred to the cartridge in step 11.6. The precipitates will be discarded.

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

NOTE: The Iron in the samples needs to be reduced from +3 to +2, in order to prevent it from interfering with the Am recovery off the Tru column. If samples appear yellow in color after the addition of the ascorbic acid, repeat step 11.5.1.3.

11.5.2 Set up UTEVA and TRU cartridges in tandem on the vacuum box system

11.5.2.1 Place the inner tube rack into the vacuum box with the centrifuge tubes in the rack. Fit the lid to the vacuum system box.

11.5.2.2 For each sample solution, put the TRU cartridge into one of the vacuum box's adjustable on/off valves. Luer lock the UTEVA cartridge to the top end of the TRU cartridge. To the top of the UTEVA cartridge connect a Luer lock 20 ml syringe/funnel.

11.5.2.3 Connect the vacuum pump to the box. Turn the vacuum pump on and ensure proper fitting of the lid.

IMPORTANT: The unused openings on the vacuum box should be turned off.

11.5.2.4 Add 5mL of 3M HNO_3 to the funnel and precondition the UTEVA and TRU cartridges. Adjust the vacuum pressure to achieve a flow-rate of approximately 1.0 mL/minute. IMPORTANT: Unless otherwise specified in the procedure, use a flow rate of approximately 1 mL/min for load and strip solutions and approximately 3 mL/min for rinse solutions. (Twenty drops is approximately equal to 1 ml). Discard conditioning rinse to waste.

11.6 U Separation from Pu, Am and Cm using UTEVA and TRU Resin

11.6.1 Transfer each sample load solution into the appropriate syringe/funnel. Allow solution to pass through both cartridges at a flow rate of approximately 1.0mL/minute.

11.6.2 Add 5 mL of 3M HNO_3 into each funnel and discard rinse (flow rate approximately 3 mL/minute).

11.6.3 Separate UTEVA cartridge from TRU cartridge. Place new syringe on the TRU cartridge. **For uranium separation with UTEVA cartridge continue with steps 11.6.4 through 11.6.9. For americium and plutonium proceed to section 11.7.1.**

11.6.4 Place UTEVA cartridge along with the funnel on a previously unused opening on the vacuum box lid. Pipet 25 ml of 3M HNO_3 into each funnel. Discard this rinse.

11.6.5 Pipet 5 mL of 9M HCl into each UTEVA cartridge and allow to drain. Discard this rinse. Note: This rinse converts the resin to the chloride system. Some Np may be removed here.

11.6.6 Pipet 20 mL of 5 M HCl- 0.05 M oxalic acid into each cartridge and allow it to drain. Discard this rinse. Note: This rinse removes neptunium and thorium from the cartridge. The 9 M HCl and the oxalic acid remove any residual ferrous ion.

11.6.7 Ensure that clean, labeled tubes are placed in the tube rack.

11.6.8 Pipet 15 mL of 1M HCl into each cartridge to strip the uranium. Allow to drain.

11.6.9 Cap and set labeled tubes aside. To co-precipitate the uranium, proceed to ST-RC-0100, "Actinide Coprecipitation."

11.7 Pu, Am Separation Using TRU Cartridge:

11.7.1 Pipet 5 mL of 2M HNO_3 into each TRU cartridge from step 11.6.3.

11.7.2 Rinse each funnel with 5 ml of the 2M HNO_3 – 0.01M $NaNO_2$. **IMPORTANT**: The flow rate for the cartridge should be adjusted to approximately 1.0mL/minute for this step. **NOTE:** Sodium nitrite is used to oxidize Pu^{+3} to Pu^{+4} and enhance the Pu/Am separation.

11.7.3 Allow the rinse solution to drain through each cartridge.

11.7.4 Add 5 mL of 0.5M HNO_3 to each cartridge and allow to drain. **NOTE**: 0.5M HNO_3 is used to lower the nitrate concentration prior to conversion to the chloride system.

11.7.5 Discard the rinse solutions to waste.

11.7.6 Ensure that clean, labeled tubes are placed in the tube rack.

11.7.7 Add 18 mL of 4M HCl to elute americium. Collect eluant. Remove and cap samples.

11.7.8 To co-precipitate the americium, proceed to STL-RC-0100, "Actinide Coprecipitation".

11.7.9 Rinse the TRU cartridge with 25 mL of 4M HCl-0.1M HF. Discard eluate. **Note:** 4M HCl – 0.1M HF is used to selectively to remove any residual Th that be present on the Tru Cartridge and leave the Pu.

11.7.10 Ensure that clean, labeled tubes are placed in the tube rack under each cartridge.

11.7.11 Add 12 mL of 0.1M $NH_4HC_2O_4$ (ammonium bioxalte) to elute plutonium from each cartridge. Collect eluant.

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

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 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.

13.2 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 QAM section 18.3.

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 QAM section 18.3.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in QAM section 18.3.

15.0 VALIDATION

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

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 Industries, Inc. Analystical Procedures, "ACS03 VBS Americium, Plutonium and Uranium in Soil (with Vacuum Box)", October 2003, and "ACS04 Americium/Lanthanide Separation in Soil". July 1999

17.2 TestAmerica Quality Assurance Manual (QAM), current revision

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

17.4 Associated SOPs (current revisions)

17.4.1 ST-RD-0210, Daily Operations of an Alpha Spectroscopy System (using AlphaVision Software)
17.4.2 ST-RC-0004, Preparation of Soil Samples for Radiochemical Analysis
17.4.3 ST-RC-0100, Actinide Coprecipitation
17.4.4 ST-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
17.4.5 ST-QA-0002, Standards and Reagent Preparation
17.4.6 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.4.7 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.4.8 ST-PM-0002, Sample Receipt and Chain of Custody

18.0 CLARIFICATIONS AND MODIFICATION TO THE REFERENCE METHOD

18.1 Sulfamic acid is used instead of ferrous sulfamate to redissolve the digested soil residue in the Actinide separation by Eichrom Resins due to Americium interferences created by the ferrous sulfamate. (section 11.4.1.2)

18.2 For the plutonium/americium separation using a TRU cartridge, the laboratory does not add the 3ml of 9M HCL to each cartridge to convert to chloride system. The laboratory adds 18ml of 4M HCL and this is sufficient for this step (section 11.6.7).

18.3 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 TestAmerica format.

19.2 Updated Standards and Reagent list in section 7.0.

19.3 Updated testing procedure in section 11.0 regarding centrifuge time lengths and water bath lengths/times.

19.4 11/30/2010, Rev 8, added sodium nitrite to section 7.0.

19.5 Updated the amount of time a sample will be in a centrifuge from 10 to 5 minutes in section 11.0.

Title: AMERICIUM, PLUTONIUM, CURIUM, AND URANIUM IN VARIOUS MATRICIES BY ElChroM® Uteva AND TRU RESINS (WITH VACUUM BOX SYSTEM)

Approvals (Signature/Date):

Sarah Hurst 11-29-10	Michael Ridenhower 11-29-10
Sarah Hurst — Date	Michael Ridenhower — Date
Separations Supervisor	Health & Safety Manager / Coordinator
Marti Ward 11-29-10	Elaine Wild 11/29/10
Marti Ward — Date	Elaine Wild — Date
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This SOP was previously identified as SOP No. ST-RC-0241 Rev. 7

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1.0 SCOPE AND APPLICATION

1.1 This SOP describes an **alternate** sequential analysis technique which may be used for certain project samples. For **routine** Americium, Plutonium, Curium and Uranium actinide sample preparation see SOP: **ST-RC-0240**.

1.1.1 If only Uranium analysis is requested, see SOP: ST-RC-0238.

1.2 This method is based on Eichrom Technologies Inc. analytical procedure "ACS03 VBS: Americium, Plutonium and Uranium in Soil (with Vacuum Box System)" and "ACS04 Americium/Lanthanide Separation in Soil".

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 reporting limits, minimum detectable activities and QC limits are maintained in the Information Management System (QuantIMS). A copy of the SAC is included in the analytical SOPs to demonstrate this information.

2.0 SUMMARY OF METHOD

2.1 This SOP describes the method for separation of americium, 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 ST-RC-0004, Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis. Tracers are used to monitor chemical recoveries and correct results to improve precision and accuracy.

3.0 DEFINITIONS

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

3.2 Tracer - A known amount of either ^{232}Uranium, ^{241}Americium, ^{243}Americium, ^{242}Plutonium, or ^{236}Plutonium is added to each sample to determine chemical yield. The tracer serves as an internal standard, which is used to calculate the activity of the target isotopes.

4.0 INTERFERENCES

4.1 Actinides with unresolvable alpha energies such as Americium-241and Plutonium-238 or Neptunium-237 and Uranium-234 must be chemically separated to enable measurement. This method separates these isotopes effectively.

4.2 Very high levels of phosphate in the sample may cause interference. Adjusting the amount of phosphate added to coprecipitate the actinides may be necessary in these cases.

4.3 Polonium-210 decays in the Uranium-232 region, and can cause high tracer recoveries if not removed from the Uteva cartridge/column, using a 25 ml 3 M HNO_3 wash after the load solution has run through the cartridge/column.

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 Hydrofluoric Acid is a corrosive and a poison. Extremely hazardous as a liquid or vapor. Requires special first aid should contact occur. Analysts must be trained in HF acid first aid prior to using this procedure. Reaction with certain metals generates flammable and potentially explosive hydrogen gas.

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
Ammonium Hydroxide	Poison Corrosive	50 ppm (NH3)	Inhalation symptoms include irritation to the respiratory tract. Ingestion symptoms include pain in the mouth, chest, and abdomen, with coughing, vomiting and collapse. Skin contact causes irritation and burns. Eye contact with vapors causes irritation.
Aluminum nitrate	Oxidizer	2 mg/m3 (TWA) Soluble salts as Al	Inhalation symptoms may include coughing, shortness of breath. Symptoms of skin contact include redness, itching, and pain. Eye contact causes irritation, redness, and pain.
Ammonium oxalate	Poison Corrosive	None established	Symptoms of inhalation exposure include nervousness, cramps and central nervous system depression. Skin contact causes redness, itching, and pain. Eye contact causes irritation, redness, and pain.
Calcium nitrate	Oxidizer	None established	Inhalation symptoms include coughing and shortness of breath. Skin contact symptoms include redness, itching, and pain. Eye contact causes irritation, redness and pain.
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.

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.
Oxalic Acid	Corrosive	1mg/m3 (TWA) 2mg/m3 (STEL)	Inhalation symptoms include severe irritation and burns of nose, throat, and respiratory tract. Ingestion symptoms include burns, nausea, severe gastroenteritis and vomiting. Skin contact causes severe irritation and burns. Oxalic Acid is an eye irritant.
Sulfamic Acid	Corrosive	No specific exposure limits established.	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea and vomiting. Skin contact produces redness, pain, and severe burns. Eye contact can cause blurred vision, redness, pain, burns, and 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			
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 Pipetter, mechanical

6.7 Fume hood

6.8 Hotplate with stirrer

6.9 Vortex mixer

6.10 pH strips, narrow range

6.11 Vacuum Box, Eichrom part number AC-24-BOX, or equivalent

6.12 Syringe filter, 25 mm acrodisc, 0.45 or 0.70 µm

6.13 Cartridge reservoirs/syringe/funnel-20 ml B-D Luer Lok syringe Part Number 301625 (Fisher part number 14-823-2B), or equivalent.

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

7.3 L (+) Ascorbic acid, reagent grade.
7.3.1 L (+) Ascorbic Acid solution, 2.5g dissolved in 10ml of DI water.

7.4 Ammonium hydrogen phosphate, dibasic (3.2M)
7.4.1 Dissolve 104 grams of $(NH_4)_2HPO_4$ in approximately 200 mL of water, heating gently to dissolve. Dilute to 250 mL with water and mix well.

7.5 Ammonium hydroxide (NH4OH) Reagent.

7.6 Ammonium Bioxalate (hydrogen oxalate) (0.1 M)
7.6.1 Dissolve 6.31 g of $H_2C_2O_4 \bullet 2H_2O$ (oxalic acid) and 7.11 g of $(NH_4)2C2O4 \bullet H2O$ (ammonium oxalate) in 900 mL of water, and dilute to 1 L with water.

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

7.8 Sulfamic Acid
7.8.1 Sulfamic acid solution, NH_2SO_3H, Dissolve 2.0 grams sulfamic acid in 10 mL of DI water

7.9 Hydrochloric acid (12M) - concentrated HCl (sp gr 1.19).
7.9.1 Hydrochloric acid (9M) - Add 1500 mL of concentrated HCl (sp gr 1.19) to 200 mL of water and dilute to 2 liters.
7.9.2 Hydrochloric acid (4M) - Add 667 mL of concentrated HCl to 200mL of water and dilute to 2 liters

7.10 Hydrochloric acid (5M) - 0.05M oxalic acid solution
7.10.1 Dissolve 6.3 grams of oxalic acid dihydrate in approximately 400 mL of water. Add 417 mL of concentrated hydrochloric acid. Cool to room temperature. Dilute to 1 liter with water and mix well.
7.10.2 Hydrochloric acid (1M)- Add 167mL of concentrated HCl (sp gr 1.19) to 200mL of water and dilute to 2 liters.

7.11 Hydrofluoric acid (48-52%) (29M)- concentrated hydrofluoric acid.

7.12 Hydrochloric acid (4 M)- hydrofluoric acid (0.1 M)- Add 333 mL of concentrated HCl (sp gr 1.19) and 3.6 mL of concentrated HF (sp gr 1.2) to 500 mL of water and dilute to 1 L with water. Prepare fresh daily.

7.13 Nitric acid (16M) - concentrated HNO_3 (sp gr 1.42).
7.13.1 Nitric acid solution (3.0M) - Add 382 mL of concentrated HNO_3 (sp gr 1.42) to 200 mL of water and dilute to 2 liters.
7.13.2 Nitric acid solution (0.5M)- Add 32mL of concentrated HNO_3 to 900mL of water and dilute to 1 L with water.
7.13.3 Nitric acid solution (2 M)- Add 127 mL of concentrated HNO_3 to 800 mL of water and dilute to 1 L with water.

7.14 Nitric acid (2M)-sodium nitrite (0.1M) solution- Add 32 mL of concentrated HNO_3 to 200 mL of water, dissolve 1.72 g of sodium nitrite in the solution and dilute to 250 mL with water. Prepare fresh daily.

7.15 Sodium Nitrite, NaNo2 reagent crystals.

7.16 Load solution [Nitric acid (3 M) - aluminum nitrate (1 M)] – Weigh 1500 g $Al(NO_3)_3 \cdot 9H_2O$ in a 4 liter beaker. Add 800 mLof 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.

7.17 Bromocresol Purple indicator solution
7.17.1 Disso1ve 0.20 g of Bromocresol Purple (520.24 F.W.) in 250 mL of water, add one ml of concentrated Ammonium Hydroxide.

7.18 Pu-242 tracer standard, NIST traceable, 10-20 dpm/ml (Pu-236 can also be used).

7.19 Pu-238 and/or Pu-239, NIST traceable.

7.20 Uranium-232 standard, NIST traceable 10-20 dpm/ml.

7.21 Natural Uranium standard, NIST traceable approximately 10-20 dpm/ml.

7.22 Am-243 tracer, NIST traceable, approximately 10-20 dpm/ml.

7.23 Am-243 spike, NIST traceable, approximately 10-20 dpm/ml.

7.24 Am-241 spike, NIST traceable, approximately 10-20 dpm/ml.

7.25 Curium-244 tracer, NIST traceable, approximately 10-20 dpm/ml.

7.26 TRU Resin – 2mL cartridge, 50-100 micron, part number TR-R50-S and TR-R200-S.

7.27 UTEVA Resin- 2mL cartridge, 50-100 micron, part number UT-R50-S and UT-R200-S.

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.

8.4 Sample hold time is 180 days from collection.

8.5 Solid sample requirements are found in SOP ST-RC-0004, “Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical 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 (MB), a Laboratory Control Sample (LCS), and Sample Duplicate (SD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

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

9.2.4 For non-aqueous method blanks, 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 and Pu only, use Calcium nitrate

9.3.4.2 Otherwise 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, filter through a Whatman GF/A (1.6 micron) filter.
- 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 Check radscreen results and customer/site history, if available, to determine sample size.
- 11.2.4 Aliquot 500 to 1000 mL of the filtered sample (or enough to meet required detection limit) into an appropriate size beaker.
  - 11.2.4.1 Typically 500ml for short count and 1000ml for long count
- 11.2.5 Add appropriate tracers or standards. Generally 10 – 20 dpm of each of the 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 Cover each beaker with a watch glass, and place each beaker on a hotplate.
- 11.4.5 Allow the samples to heat to near boiling approximately 30 minutes.
- 11.4.6 Once the samples reach near boiling, take the watch glass off the beaker and turn the heat down to medium.
- 11.4.7 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 uncovered 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 Actinide Separations using Eichrom Resins:

11.5.1 Re-dissolve samples in load solution:

11.5.1.1 Dissolve each calcium phosphate precipitate, soil dissolution residue or evaporated water samples with 15 mL of (load solution) 3 M HNO_3-1.0M $Al(NO_3)_3$,. Vortex sample.

NOTE: Additional aliquots of 5-mL may be necessary to dissolve the sample residue. Do not use more than 30 ml of load solution.

NOTE: If particles are observed or the solution is cloudy, centrifuge the sample at approximately 2000 rpm for 5 minutes. Transfer to a clean labeled centrifuge tube. The precipitates will be discarded.

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

11.5.1.2 Add 2.5 mL of sulfamic acid solution to each solution.

NOTE: If additional load solution was used to dissolve the sample in step 11.5.1.1, add a total of 3 mL of sulfamic acid solution. Swirl to mix.

11.5.1.3 Add 3ml of ascorbic acid to each solution, swirling to mix. Place in hot water bath for approximately 3 minutes. Then a cool water bath until slightly below room temperature.

NOTE: If particles are observed or the solution is cloudy, centrifuge the sample at 2000 rpm for 5 minutes. The supernatant will be transferred to the cartridge in step 11.6. The precipitates will be discarded.

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

NOTE: The Iron in the samples needs to be reduced from +3 to +2, in order to prevent it from interfering with the Am recovery off the Tru column. If samples appear yellow in color after the addition of the ascorbic acid, repeat step 11.5.1.3.

11.5.2 Set up UTEVA and TRU cartridges in tandem on the vacuum box system

11.5.2.1 Place the inner tube rack into the vacuum box with the centrifuge tubes in the rack. Fit the lid to the vacuum system box.

11.5.2.2 For each sample solution, put the TRU cartridge into one of the vacuum box's adjustable on/off valves. Luer lock the UTEVA cartridge to the top end of the TRU cartridge. To the top of the UTEVA cartridge connect a Luer lock 20 ml syringe/funnel.

11.5.2.3 Connect the vacuum pump to the box. Turn the vacuum pump on and ensure proper fitting of the lid.

IMPORTANT: The unused openings on the vacuum box should be turned off.

11.5.2.4 Add 5mL of 3M HNO_3 to the funnel and precondition the UTEVA and TRU cartridges. Adjust the vacuum pressure to achieve a flow-rate of approximately 1.0 mL/minute. IMPORTANT: Unless otherwise specified in the procedure, use a flow rate of approximately 1 mL/min for load and strip solutions and approximately 3 mL/min for rinse solutions. (Twenty drops is approximately equal to 1 ml). Discard conditioning rinse to waste.

11.6 U Separation from Pu, Am and Cm using UTEVA and TRU Resin

11.6.1 Transfer each sample load solution into the appropriate syringe/funnel. Allow solution to pass through both cartridges at a flow rate of approximately 1.0mL/minute.

11.6.2 Add 5 mL of 3M HNO_3 into each funnel and discard rinse (flow rate approximately 3 mL/minute).

11.6.3 Separate UTEVA cartridge from TRU cartridge. Place new syringe on the TRU cartridge. **For uranium separation with UTEVA cartridge continue with steps 11.6.4 through 11.6.9. For americium and plutonium proceed to section 11.7.1.**

11.6.4 Place UTEVA cartridge along with the funnel on a previously unused opening on the vacuum box lid. Pipet 25 ml of 3M HNO_3 into each funnel. Discard this rinse.

11.6.5 Pipet 5 mL of 9M HCl into each UTEVA cartridge and allow to drain. Discard this rinse. Note: This rinse converts the resin to the chloride system. Some Np may be removed here.

11.6.6 Pipet 20 mL of 5 M HCl- 0.05 M oxalic acid into each cartridge and allow it to drain. Discard this rinse. Note: This rinse removes neptunium and thorium from the cartridge. The 9 M HCl and the oxalic acid remove any residual ferrous ion.

11.6.7 Ensure that clean, labeled tubes are placed in the tube rack.

11.6.8 Pipet 15 mL of 1M HCl into each cartridge to strip the uranium. Allow to drain.

11.6.9 Cap and set labeled tubes aside. To co-precipitate the uranium, proceed to ST-RC-0100, "Actinide Coprecipitation."

11.7 Pu, Am Separation Using TRU Cartridge:

11.7.1 Pipet 5 mL of 2M HNO_3 into each TRU cartridge from step 11.6.3.

11.7.2 Rinse each funnel with 5 ml of the 2M HNO_3 – 0.01M $NaNO_2$. **IMPORTANT**: The flow rate for the cartridge should be adjusted to approximately 1.0mL/minute for this step. **NOTE:** Sodium nitrite is used to oxidize Pu^{+3} to Pu^{+4} and enhance the Pu/Am separation.

11.7.3 Allow the rinse solution to drain through each cartridge.

11.7.4 Add 5 mL of 0.5M HNO_3 to each cartridge and allow to drain. **NOTE**: 0.5M HNO_3 is used to lower the nitrate concentration prior to conversion to the chloride system.

11.7.5 Discard the rinse solutions to waste.

11.7.6 Ensure that clean, labeled tubes are placed in the tube rack.

11.7.7 Add 18 mL of 4M HCl to elute americium. Collect eluant. Remove and cap samples.

11.7.8 To co-precipitate the americium, proceed to STL-RC-0100, "Actinide Coprecipitation".

11.7.9 Rinse the TRU cartridge with 25 mL of 4M HCl-0.1M HF. Discard eluate. **Note:** 4M HCl – 0.1M HF is used to selectively to remove any residual Th that be present on the Tru Cartridge and leave the Pu.

11.7.10 Ensure that clean, labeled tubes are placed in the tube rack under each cartridge.

11.7.11 Add 12 mL of 0.1M $NH_4HC_2O_4$ (ammonium bioxalte) to elute plutonium from each cartridge. Collect eluant.

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

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 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.

13.2 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 QAM section 18.3.

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 QAM section 18.3.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in QAM section 18.3.

15.0 VALIDATION

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

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 Industries, Inc. Analystical Procedures, "ACS03 VBS Americium, Plutonium and Uranium in Soil (with Vacuum Box)", October 2003, and "ACS04 Americium/Lanthanide Separation in Soil". July 1999

17.2 TestAmerica Quality Assurance Manual (QAM), current revision

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

17.4 Associated SOPs (current revisions)

17.4.1 ST-RD-0210, Daily Operations of an Alpha Spectroscopy System (using AlphaVision Software)
17.4.2 ST-RC-0004, Preparation of Soil Samples for Radiochemical Analysis
17.4.3 ST-RC-0100, Actinide Coprecipitation
17.4.4 ST-RC-5006, Decontamination of Laboratory Glassware, Labware and Equipment
17.4.5 ST-QA-0002, Standards and Reagent Preparation
17.4.6 ST-QA-0005, Calibration and Verification Procedure for Thermometers, Balances, Weights and Pipettes
17.4.7 ST-QA-0036, Non-conformance Memorandum (NCM) Process
17.4.8 ST-PM-0002, Sample Receipt and Chain of Custody

18.0 CLARIFICATIONS AND MODIFICATION TO THE REFERENCE METHOD

18.1 Sulfamic acid is used instead of ferrous sulfamate to redissolve the digested soil residue in the Actinide separation by Eichrom Resins due to Americium interferences created by the ferrous sulfamate. (section 11.4.1.2)

18.2 For the plutonium/americium separation using a TRU cartridge, the laboratory does not add the 3ml of 9M HCL to each cartridge to convert to chloride system. The laboratory adds 18ml of 4M HCL and this is sufficient for this step (section 11.6.7).

18.3 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 TestAmerica format.

19.2 Updated Standards and Reagent list in section 7.0.

19.3 Updated testing procedure in section 11.0 regarding centrifuge time lengths and water bath lengths/times.

19.4 11/30/2010, Rev 8, added sodium nitrite to section 7.0.

19.5 Updated the amount of time a sample will be in a centrifuge from 10 to 5 minutes in section 11.0.

Title: ALPHA SPECTROSCOPY ANALYSIS

Approvals (Signature/Date):

Rhonda Ridenhower 6/30/10 Rhonda Ridenhower Date Radiochemistry Manager	Michael Ridenhower 6/30/10 Michael Ridenhower Date Health & Safety Manager / Coordinator
Marti Ward 6-30-10 Marti Ward Date Quality Assurance Manager	Elaine Wild 6/30/10 Elaine Wild Date Laboratory Director

This SOP was previously identified as SOP No. STL-RD-0210 Rev. 6

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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 laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Information Management System (QuantIMS). A copy of the Structure and Analysis Code (SAC), which lists this information, is included in the appendix of this SOP.

2.0 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 (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 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 Intrest (ROI): The KeV range through which the target isotope peak signal responds.

3.4 Tailing: Tailing is a delayed return of a peak to chromatographic baseline or continuation of response beyond its normal response window (RT window, ROI) due to high concentration of the analyte or a matrix interference.

3.5 Alpha Vision – 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 ROIs. Th229 tailing into the Th230 region of interest is a recognized example. This interference is minimized by maintaining low activities of the Th229 tracer and monitoring of the separation of the ROIs for Th229 and Th230. The use of manual integration may be required.

4.3 Some isotopic elements are not distinguishable and are reported as an isotopic pair, unless specifically directed by the client not to do so. These pairs may be reported separately depending on the client's DQOs and the use-ability of the data. When reported separately, the narrative must describe the technical aspects of how the isotopic pair was divided.

4.3.1 Recognized Isotopic Pairs:

4.3.1.1 Plutonium 239/240

4.3.1.2 Uranium 235/236
4.3.1.3 Uranium 233/234
4.3.1.4 Curium 245/246
4.3.1.5 Curium 247/248

5.0 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.

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 Th230, Pu239 and Am243.

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 (listed in Section 1.4) 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 <u>method blank</u>, a <u>Laboratory Control Sample</u> (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 Initial calibrations are performed according to the following schedule

10.1.1 Energy calibrations shall be performed for the alpha spectroscopy systems <u>monthly</u>, or when the calibration quality control check indicates an unacceptable change in the energy calibration parameters.

10.1.1.1 Energy Calibrations shall be performed using at least three isotopes within the energy range of 3-6 MeV. Final peak energy positions of all observed isotopes shall be within +/- 40 KeV of expected energy. The energy vs. channel and the equation with the slope is documented within the software. The slope of the equation is less than or equal to 15 KeV/channel.

10.1.2 Efficiency calibrations shall be established for the alpha spectroscopy systems <u>monthly</u>, or when the calibration quality control check indicates an unacceptable change in the efficiency calibration parameters.

10.1.3 Background subtraction spectrum shall be established for the alpha spectroscopy systems <u>monthly</u>, or when the background quality control check indicates an unacceptable change in the daily background parameters. A background check will be performed weekly and compared to the monthly backgrounds to monitor possible contamination.

10.2 Daily Checks

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

10.2.1.1 The pulser energy, peak centroid, peak resolution, peak area quality control for a detector shall be checked each day that the alpha spectroscopy system is used.

10.2.2 Routine calibration, background and pulser quality control parameters using the "Boundry" out-of-range test will be found unacceptable if the value is outside parameter tolerance.

10.2.2.1 The routine quality control check should be rerun to determine the statistical significance of the out of control parameter.

10.2.2.2 If the out of control parameter is found acceptable in the rerun, the investigation will be noted in the instrument maintenance log.

10.2.2.3 Check the integrity of the radioactive standard.

10.2.2.4 Check source positioning and all instrument settings.

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

10.2.3 If the instrument fails to meet the acceptance criteria, and the corrective actions above do not resolve the problem, the instrument must be "tagged" out of service for the day.

10.2.3.1 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.

10.2.3.2 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 orange sticker as a visual indicator of its status.

10.2.3.3 The instrument may be returned to service once the malfunction has been corrected and the above acceptance criteria have been met. Note any repairs in the maintenance log.

10.3 Calibration process in the Software

10.3.1 Alpha Detector System Energy and Efficiency Calibration

10.3.1.1 Place the correct source into the detector.

10.3.1.2 In the Alphavision software, click on Calibration Icon.

10.3.1.3 Click on detector to be calibrated.

10.3.1.4 Select Calibration from the Tool Bar.

10.3.1.5 Select Process

10.3.1.5.1 The Calibration Explorer Window will appear.

10.3.1.6 In the General Window, name the Calibration with the month, year_detector format. (JAN2001_AV1)

10.3.1.7 Choose correct template for source to count

10.3.1.8 Click next

10.3.1.9 In the Acquisition window, confirm count time of 140 minutes

10.3.1.10 Click next

10.3.1.11 In the Energy/Efficiency Calibration Window, confirm the correct source is used, and select which shelf the source is on. (This will either be 1 or 2)

10.3.1.12 Click next

10.3.1.13 In the Report Window, select print on completion

10.3.1.14 Click finish

10.3.1.15 When count is complete, the Manual Energy and Efficiency Calibration Window will appear. In this window, select Calibration ROI, select Calibrate, and Save.

10.3.1.16 Repeat for each detector

10.3.1.17 Record the calibration in the Alpha Maintenance Log Book.

10.3.2 **Detector Background Counting**

10.3.2.1 Select the Batch Icon

10.3.2.2 Select backgrounds from the Tool Bar

10.3.2.3 Select Process.

10.3.2.3.1 This will open the General Window in Batch Wizard
10.3.2.4 Name the background with month_year format. (JAN_04)
10.3.2.5 Select correct template (provided by analyst)
10.3.2.6 Click next.
10.3.2.7 In the Sample Window, add all detector names.
10.3.2.8 Click next
10.3.2.9 In the Acquisition Window, confirm count time is set at 960 minutes (or as long as the longest sample count time, not to exceed 1000 minutes)
10.3.2.10 Click next
10.3.2.11 In the Analysis Set Up Page, select Background Library and Background ROI, check the Use ROI box.
10.3.2.12 Click next
10.3.2.13 In the Report Window, select print on completion
10.3.2.14 Click finish
10.3.2.15 The Detector Assignment worksheet will appear, assign detectors, and select start now.
10.3.2.16 Record the backgrounds in the instrument maintenance log.
10.3.2.17 The background spectrum will be processed by the software
10.3.2.18 The detectors shall be "categorized" after each monthly background. The detectors will be labelled as follows:

Counts in Region of Interest (i.e. Th230, Th232, U234, U238, Pu238, Pu239):
0-2 counts- Blue Sticker-Ultra Low Level
0-4 counts- Yellow Sticker- Intermediate Low Level
4+ counts- Green Sticker Low Level
Detectors 1-8 are always designated for Routine analysis when the RL=1 or the activity is from a known radioactive site.

11.0 PROCEDURE

11.1 For sample preparation reference the applicable actinide SOPs: ST-RC-0040, ST-RC-0210, ST-RC-0232, ST-RC-0238, ST-RC-0241, ST-RC-0242 and ST-RC-0246.

11.2 Initial Setup
11.2.1 Establish the normal instrument settings for all controls.
11.2.1.1 Detector specific high voltage settings and required polarity are listed in the method software settings.
11.2.2 Pulser quality controls shall be checked before each use of the instrument.

11.3 Counting Samples
11.3.1 In Radcapture, go to Utilities,export, Alphavision batch data, enter batch #, and ok export to Alphavision.
11.3.2 In Alphavision, go to Process, select Batch to open the Batch Wizard.
11.3.3 Choose Load from LIMS, and pick the batch.
11.3.4 Choose test by clicking on the correct isotope test
11.3.5 Select Next
11.3.6 Click on blank, and then pick blank type (Uu blank, Pu Blank, ect)
11.3.7 Click on LCS, and then pick LCS type with correct spike number. For amount, use the spike aliquot amount (0.1, 0.2mL, 0.1326g, etc).
11.3.8 Select Next
11.3.9 Live time is count time. Enter correct count time for the batch, select next,
11.3.10 Select Nuclide Library, choose correct ROI and tracer
11.3.11 Select next

11.3.12 Change TPU Sigma to 2 (unless otherwise noted in client requirements), select correct activity units (DPM, pCi, etc), select Activity concentration.
11.3.13 Select Next, two times
11.3.14 Select Print on Completion
11.3.15 Select Finish
11.3.16 Click and drag correct detectors to the correct sample id and select Start Now.
11.3.17 The spectrum will be processed by the software.
11.3.18 For DOE: the FWHM of each tracer peak shall be <= 100keV; the tracer peak energy for each sample shall be within +/-50keV of the expected energy.
11.3.19 Backgrounds are checked after high activity samples

11.4 Samples with a count rate of greater than 1 cps should be removed from the alpha counting system to prevent contamination of detector(s).
11.4.1 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.

12.0 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 LQM.

12.2 Isotope ROIs are given in Tables 1 in this SOP.

12.3 Any manual integration of a peak or group of peaks must be documented. In all instances where the data system report has been edited or where manual integration has been performed, the operator must clearly identify such edits or manual procedures. Reference SOP 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 Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036. Below is a subset of the data assessment and QC excursion types within Clouseau; the text in underline is the exact "type" line in Clouseau. For a complete and current listing, please access the software program.

13.2 Method Blank
13.2.1 Acceptance Criteria:
13.2.1.1 No target analytes may be present in the method blank above the reporting limit.
13.2.1.2 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> – See Clouseau NCM for corrective action (e.g. reprep/reanalysis, narration). Note certain analytes are common laboratory contaminants which require special narrative comment. These compounds are also designated in Clouseau.

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) – See Clouseau NCM for corrective action (e.g. , reanalysis, narration).
13.3.2.2 LCS Spike Recovery excursion (low) – See Clouseau NCM for corrective action (e.g. , reanalysis, narration).
13.3.2.3 RPD/RER Duplicate excursion – See Clouseau NCM for corrective action (e.g. reanalysis, narration).

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. See Clouseau NCM to determine if re-preparation re-analysis is required.

13.5 Sample result evaluation
13.5.1 Tracer recovery must be within specified limits.
13.5.2 Tracer/Carrier recovery low– See Clouseau NCM for corrective action.
13.5.3 Tracer/Carrier recovery high– See Clouseau NCM for corrective action.
13.5.3.1 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:
13.5.3.2 minimum number of tracer counts
13.5.3.3 level of uncertainty
13.5.3.4 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.

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

14.0 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 QAM section 18.3.

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 QAM section 18.3.

14.1 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in QAM section 18.3.

15.0 VALIDATION

15.1 Laboratory SOPs are based on standard reference EPA Methods that have been validated by the EPA and the lab is not required to perform validation for these methods. The requirements for lab demonstration of capability are included in LQM. Lab validation data would be appropriate for performance based measurement systems or non-standard methods. 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 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 3000 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 Associated SOPs, current revisions

17.9.1 ST-PM-0002, Chain of Custody

17.9.2 ST-QA-0002, Standard and Reagent Preparation

17.9.3 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts
17.9.4 ST-QA-0036 Non-Conformance Memorandum (NCM) Procedure
17.9.5 ST-QA-0040, Manual Integration Procedure
17.9.6 ST-RC-0040, Total Alpha Emitting Isotopes of Radium
17.9.7 ST-RC-0238, ISOTOPIC URANIUM BY EICHROM® UTEVA RESIN FOR VARIOUS MATRICES
17.9.8 ST-RC-0210, DETERMINATION OF POLONIUM-210 BY ALPHA SPECTROMETRY
17.9.9 ST-RC-0232, ISOTOPIC THORIUM AND/OR NEPTUNIUM IN VARIOUS MATRICES BY EICHROM® TEVA SEPARATION RESIN
17.9.10 ST-RC-0240, SOTOPIC AMERICIUM, CURIUM, PLUTIONIUM, THORIUM, AND URANIUM IN VARIOUS MATRICES BY EICHROM® SEPARATION RESIN
17.9.11 ST-RC-0241, AMERICIUM, PLUTONIUM, CURIUM, AND URANIUM IN VARIOUS MATRICES BY EICHROM® UTEVA AND TRU RESINS (WITH VACUUM BOX SYSTEM)
17.9.12 ST-RC-0242, ISOTOPIC THORIUM, PLUTONIUM AND URANIUM IN VARIOUS MATRICES BY EICHROM® SEPARATION RESINS
17.9.13 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.

19.0 CHANGES TO PREVIOUS REVISION

19.1 No Changes- Annual Review

Table 1

Primary a Emissions for Selected Radionuclides

Isotope	a (keV)	a2 (keV)	a3 (keV)	a4 (keV)	a5 (keV)	a6 (keV)	a7 (keV)	a8 (keV)	a9 (keV)
^{210}Polunium	5297								
^{226}Radium	4784	4602							
^{227}Thorium	5600	6308							
^{228}Thorium	5423	5340	5211	5177	5138				
^{229}Thorium	5052	5050	4978	4868	4901	4845	4838	4814	4798
^{230}Thorium	4688	4621	4480	4438	4372				
^{232}Thorium	4010	3952	3830						
^{232}Uranium	5320	5264	5137						
^{233}Uranium	4825	4804	4796	4783	4754	4729	4701	4664	
^{234}Uranium	4776	4724	4605						
^{235}Uranium	4597	4556	4414	4395	4370	4364	4344	4324	4216
^{238}Uranium	4196	4147	4040						
^{237}Neptunium	4873	4818	4804	4788	4772	4766	4707	4665	4640
^{236}Plutonium	5768	5721	5614						
^{238}Plutonium	5499	5456	5358						
$^{239/240}$Plutonium	5105	5143	5105						
^{242}Plutonium	4901	4856							
^{244}Plutonium	4546	4589							
^{241}Americium	5544	5512	5486	5443	5388				
^{243}Americium	5350	5319	5277	5324	5180				
^{242}Curium	6069	6113							
^{244}Curium	5805	5763							
^{245}Curium	5303	5362							
$^{246/247}$Curium	4868	5145							

TAL Reference Data Summary

:ructured Analysis Code: I-J2-2M-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6511							Spike List 6555						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
) Thorium 231	0.1	pCi/L			0														
3 Thorium 234	0.1	pCi/L			0														
) Uranium 234, dissolved	0.1	pCi/L			0														
3 Uranium 238, dissolved	0.1	pCi/L			0														
) Uranium 233/234	0.1	pCi/L			0														
) Uranium 234	0.1	pCi/L			0	C	Y			82	123	40	C	Y			65	146	40
) Uranium 235/236	0.1	pCi/L			0														
3 Uranium 238	0.1	pCi/L			0	C	Y			80	124	40	C	Y			68	143	40

TAL Reference Data Summary

ructured Analysis Code: I-88-2M-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJI
Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6511							Spike List 6555						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3 Total Uranium		ug/ml			0														
3 Uranium 234		ug/ml			0	C	Y			82	123	40	C	Y			65	146	40
1 Uranium 235		ug/ml			0														
3 Uranium 238		ug/ml			0	C	Y			80	124	40	C	Y			68	143	40

Printed at 6/30/2010 4:50:13 P

TAL Reference Data Summary

tructured Analysis Code: A-IB-2M-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Uranium (ONLY) by Ion Ex and/or Extraction Chromatogra
Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6511							Spike List 6555						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9 Uranium 234	0.1	pCi/g			0	C	Y			79	121	40	C	Y			70	130	40
1 Uranium 235	0.1	pCi/g			0														
5 Uranium 236	0.1	pCi/g			0														
3 Uranium 238	0.1	pCi/g			0	C	Y			79	122	40	C	Y			70	130	40

Printed at 6/30/2010 4:50:55 P

TAL Reference Data Summary

tructured Analysis Code: A-J2-2M-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List			Detection Limits					Check List 6511							Spike List 6555						
	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9	Thorium 231	0.1	pCi/g			0														
3	Thorium 234	0.1	pCi/g			0														
9	Uranium 233/234	0.1	pCi/g			0														
9	Uranium 234	0.1	pCi/g			0	C	Y			79	121	40	C	Y			70	130	40
0	Uranium 235/236	0.1	pCi/g			0														
3	Uranium 238	0.1	pCi/g			0	C	Y			79	122	40	C	Y			70	130	40

Printed at 6/30/2010 4:54:48 F

TAL Reference Data Summary

tructured Analysis Code: A-K7-2M-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6511							Spike List 6555						
I	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9	Thorium 231	0.1	pCi/g			0														
3	Thorium 234	0.1	pCi/g			0														
9	Uranium 233/234	0.1	pCi/g			0														
9	Uranium 234	0.1	pCi/g			0	C	Y			79	121	40	C	Y			70	130	40
1	Uranium 235	0.1	pCi/g			0														
0	Uranium 235/236	0.1	pCi/g			0														
5	Uranium 236	0.1	pCi/g			0														
3	Uranium 238	0.1	pCi/g			0	C	Y			79	122	40	C	Y			70	130	40

Printed at 6/30/2010 4:55:28 F

TAL Reference Data Summary

ructured Analysis Code: A-K7-2M-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6511							Spike List 6555						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
Thorium 231	0.1	pCi/g			0														
Thorium 234	0.1	pCi/g			0														
Uranium 233/234	0.1	pCi/g			0														
Uranium 234	0.1	pCi/g			0	C	Y			79	121	40	C	Y			70	130	40
Uranium 235	0.1	pCi/g			0														
Uranium 235/236	0.1	pCi/g			0														
Uranium 236	0.1	pCi/g			0														
Uranium 238	0.1	pCi/g			0	C	Y			79	122	40	C	Y			70	130	40

Printed at 6/30/2010 4:56:58 P

TAL Reference Data Summary

tructured Analysis Code: **A-88-2M-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: NO SAMPLE PREPARATION PERFORMED / DIRECT INJI
Method: Iso URANIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6511							Spike List 6555						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3 Total Uranium		ug/g			0														
9 Uranium 234		ug/g			0	C	Y			79	121	40	C	Y			70	130	40
1 Uranium 235		ug/g			0														
3 Uranium 238		ug/g			0	C	Y			79	122	40	C	Y			70	130	40

Printed at 6/30/2010 4:57:40 P

TAL Reference Data Summary

tructured Analysis Code: A-IB-2I-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Uranium (ONLY) by Ion Ex and/or Extraction Chromatogra
Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6511							Spike List 6555						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9 Uranium 234	1	pCi/g			0	C	Y			79	121	40	C	Y			70	130	40
1 Uranium 235	1	pCi/g			0														
5 Uranium 236	1	pCi/g			0														
3 Uranium 238	1	pCi/g			0	C	Y			79	122	40	C	Y			70	130	40

Printed at 6/30/2010 4:58:18 F

TAL Reference Data Summary

:ructured Analysis Code: A-J2-2I-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6511							Spike List 6555						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9 Uranium 233/234	1	pCi/g			0														
9 Uranium 234	1	pCi/g			0	C	Y			79	121	40	C	Y			70	130	40
) Uranium 235/236	1	pCi/g			0														
3 Uranium 238	1	pCi/g			0	C	Y			79	122	40	C	Y			70	130	40

Printed at 6/30/2010 4:58:53 P

TAL Reference Data Summary

tructured Analysis Code: A-K7-2I-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6511							Spike List 6555						
	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9	Thorium 231	1	pCi/g			0														
3	Thorium 234	1	pCi/g			0														
9	Uranium 233/234	1	pCi/g			0														
9	Uranium 234	1	pCi/g			0	C	Y			79	121	40	C	Y			70	130	40
1	Uranium 235	1	pCi/g			0														
0	Uranium 235/236	1	pCi/g			0														
5	Uranium 236	1	pCi/g			0														
3	Uranium 238	1	pCi/g			0	C	Y			79	122	40	C	Y			70	130	40

Printed at 6/30/2010 4:59:36 P

TAL Reference Data Summary

tructured Analysis Code: A-J2-2I-DO-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

	Analyte List		Detection Limits					Check List 6870							Spike List 6871						
	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9	Uranium 233/234	1	pCi/g			0														
9	Uranium 234	1	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25
0	Uranium 235/236	1	pCi/g			0														
3	Uranium 238	1	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

Printed at 6/30/2010 5:00:27 P

TAL Reference Data Summary

ructured Analysis Code: I-J2-2I-DO-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso URANIUM (SHORT CT) DOE A-01-R MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6870							Spike List 6871						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
Uranium 238, dissolved	1	pCi/L			0														
Uranium 233/234	1	pCi/L			0														
Uranium 234	1	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25
Uranium 234, Dissolved	1	pCi/L			0														
Uranium 235/236	1	pCi/L			0														
Uranium 238, Dissolved	1	pCi/L			0														
Uranium 238	1	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

Printed at 6/30/2010 5:01:05 P

TAL Reference Data Summary

ructured Analysis Code: I-JD-3R-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - U-232
Method: Iso URANIUM-232 by DOE A-01-R Mod
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6543							Spike List 6587						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
Uranium-232	1	pCi/L			0	C	Y			70	130	40	C	Y			70	96	40

Printed at 6/30/2010 5:01:54 P

TAL Reference Data Summary

tructured Analysis Code: A-JD-3R-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - U-232
Method: Iso URANIUM-232 by DOE A-01-R Mod
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

	Analyte List		Detection Limits							Check List 6543							Spike List 6587					
	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
6	Uranium-232	1	pCi/g			0	C	Y			73	115	40	C	Y			74	100	40

TAL Reference Data Summary

tructured Analysis Code: **I-J2-2O-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso THORIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6513							Spike List 6557						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5 Thorium 228	0.1	pCi/L			0	C	Y			70	130	40	C	Y			70	130	40
7 Thorium 230	0.1	pCi/L			0	C	Y			79	120	40	C	Y			82	139	40
1 Thorium 232	0.1	pCi/L			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

:ructured Analysis Code: A-J2-2O-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso THORIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6513							Spike List 6557						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5 Thorium 228	0.1	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40
7 Thorium 230	0.1	pCi/g			0	C	Y			80	118	40	C	Y			76	115	40
1 Thorium 232	0.1	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

tructured Analysis Code: A-K7-2O-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Iso THORIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6513							Spike List 6557						
	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5	Thorium 228	0.1	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40
7	Thorium 230	0.1	pCi/g			0	C	Y			80	118	40	C	Y			76	115	40
1	Thorium 232	0.1	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40

Printed at 6/30/2010 5:03:48 F

TAL Reference Data Summary

tructured Analysis Code: I-J2-2K-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso THORIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6513							Spike List 6557						
n	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
15	Thorium 228	1.0	pCi/L			0	C	Y			70	130	40	C	Y			70	130	40
17	Thorium 230	1.0	pCi/L			0	C	Y			79	120	40	C	Y			82	139	40
21	Thorium 232	1.0	pCi/L			0	C	Y			70	130	40	C	Y			70	130	40

Printed at 6/30/2010 5:04:17 F

TAL Reference Data Summary

Structured Analysis Code: **A-J2-2K-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso THORIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6513							Spike List 6557						
1	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5	Thorium 228	1.0	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40
7	Thorium 230	1.0	pCi/g			0	C	Y			80	118	40	C	Y			76	115	40
1	Thorium 232	1.0	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40

Printed at 6/30/2010 5:04:40 F

TAL Reference Data Summary

Structured Analysis Code: **A-K7-2K-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Iso THORIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6513							Spike List 6557						
n	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
15	Thorium 228	1.0	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40
17	Thorium 230	1.0	pCi/g			0	C	Y			80	118	40	C	Y			76	115	40
21	Thorium 232	1.0	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

tructured Analysis Code: **A-J2-2K-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso THORIUM (SHORT CT) DOE A-01-R MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6868							Spike List 6869						
ı	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5	Thorium 228	1.0	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25
7	Thorium 230	1.0	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25
:1	Thorium 232	1.0	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

Printed at 6/30/2010 5:05:25 F

TAL Reference Data Summary

tructured Analysis Code: I-J2-2K-DO-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso THORIUM (SHORT CT) DOE A-01-R MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6868							Spike List 6869						
	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
8	Thorium 228, dissolved	1.0	pCi/L			0														
9	Thorium 230, dissolved	1.0	pCi/L			0														
0	Thorium 232, dissolved	1.0	pCi/L			0														
5	Thorium 228	1.0	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25
7	Thorium 230	1.0	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25
1	Thorium 232	1.0	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

Printed at 6/30/2010 5:05:46 P

TAL Reference Data Summary

:ructured Analysis Code: I-JZ-5G-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Th-229
Method: Thorium-229 by DOE A-01-R Mod
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6514							Spike List 6515						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5 Thorium 228	1.0	pCi/L			0	C	Y			85	125	40	C	Y			85	125	40
2 Thorium 229	1.0	pCi/L			0	C	Y			70	130	0	C	Y			70	130	0
' Thorium 230	1.0	pCi/L			0	T	Y			30	110	40	T	Y			30	110	40
I Thorium 232	1.0	pCi/L			0	C	Y			81	124	40	C	Y			81	124	40

Printed at 6/30/2010 5:06:14 PM

TAL Reference Data Summary

tructured Analysis Code: A-JZ-5G-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Th-229
Method: Thorium-229 by DOE A-01-R Mod
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

	Analyte List	Detection Limits					Check List 6514							Spike List 6515						
	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5	Thorium 228	1.0	pCi/g			0														
2	Thorium 229	1.0	pCi/g			0	C	Y			70	130	0	C	Y			70	130	0
7	Thorium 230	1.0	pCi/g			0	T	Y			69	108	40	T	Y			30	110	40
1	Thorium 232	1.0	pCi/g			0														

Printed at 6/30/2010 5:06:33 P

TAL Reference Data Summary

tructured Analysis Code: I-J2-2L-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso PLUTONIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6510							Spike List 6554						
1 Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
53 Plutonium 244	0.1	pCi/L			0														
39 Plutonium 238	0.1	pCi/L			0	C	Y			79	115	40	C	Y			70	130	40
33 Plutonium 239/40	0.1	pCi/L			0	C	Y			85	120	40	C	Y			70	130	40
31 Plutonium 242	0.1	pCi/L			0														

Printed at 6/30/2010 5:07:03 I

TAL Reference Data Summary

tructured Analysis Code: **A-J2-2L-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso PLUTONIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

	Analyte List		Detection Limits					Check List 6510							Spike List 6554						
	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3	Plutonium 244	0.1	pCi/g			0														
9	Plutonium 238	0.1	pCi/g			0	C	Y			75	112	40	C	Y			75	110	40
3	Plutonium 239/40	0.1	pCi/g			0	C	Y			83	119	40	C	Y			82	113	40
1	Plutonium 242	0.1	pCi/g			0														

Printed at 6/30/2010 5:07:27 P

TAL Reference Data Summary

ructured Analysis Code: A-K7-2L-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Iso PLUTONIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6510							Spike List 6554						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9 Plutonium 238	0.1	pCi/g			0	C	Y			75	112	40	C	Y			75	110	40
3 Plutonium 239/40	0.1	pCi/g			0	C	Y			83	119	40	C	Y			82	113	40
1 Plutonium 242	0.1	pCi/g			0														

Printed at 6/30/2010 5:07:41 F

TAL Reference Data Summary

tructured Analysis Code: I-J2-2H-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso PLUTONIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List			Detection Limits					Check List 6510							Spike List 6554						
ı	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3	Plutonium 244	1.0	pCi/L			0														
9	Plutonium 238	1.0	pCi/L			0	C	Y			79	115	40	C	Y			70	130	40
3	Plutonium 239/40	1.0	pCi/L			0	C	Y			85	120	40	C	Y			70	130	40
1	Plutonium 242	1.0	pCi/L			0														

Printed at 6/30/2010 5:09:21 F

TAL Reference Data Summary

ructured Analysis Code: **A-J2-2H-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso PLUTONIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6510							Spike List 6554						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3 Plutonium 244	1.0	pCi/g			0														
3 Plutonium 238	1.0	pCi/g			0	C	Y			75	112	40	C	Y			75	110	40
3 Plutonium 239/40	1.0	pCi/g			0	C	Y			83	119	40	C	Y			82	113	40
1 Plutonium 242	1.0	pCi/g			0														

Printed at 6/30/2010 5:09:41 P

TAL Reference Data Summary

ructured Analysis Code: **A-K7-2H-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Iso PLUTONIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6510							Spike List 6554						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
Plutonium 238	1.0	pCi/g			0	C	Y			75	112	40	C	Y			75	110	40
Plutonium 239/40	1.0	pCi/g			0	C	Y			83	119	40	C	Y			82	113	40
Plutonium 242	1.0	pCi/g			0														

Printed at 6/30/2010 5:10:05 PM

TAL Reference Data Summary

tructured Analysis Code: **A-J2-2H-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso PLUTONIUM (SHORT CT) DOE A-01-R MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6866							Spike List 6867						
ı	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3	Plutonium 244	1.0	pCi/g			0														
9	Plutonium 238	1.0	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25
3	Plutonium 239/40	1.0	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25
1	Plutonium 242	1.0	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

uctured Analysis Code: I-J2-2H-DO-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Iso PLUTONIUM (SHORT CT) DOE A-01-R MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits						Check List 6866						Spike List 6867					
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
Plutonium 244	1.0	pCi/L			0														
Plutonium 238	1.0	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25
Plutonium 239/40	1.0	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25
Plutonium 242	1.0	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

Printed at 6/30/2010 5:10:54 PI

TAL Reference Data Summary

tructured Analysis Code: I-JB-3P-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Pu-242
Method: Plutonium-242 by DOE A-01-R Mod
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6537							Spike List 6538						
ı	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3	Plutonium 244	1	pCi/L			0														
1	Plutonium 242	1	pCi/L			0	C	Y			90	114	40	C	Y			90	114	40

Printed at 6/30/2010 5:11:25 F

TAL Reference Data Summary

ructured Analysis Code: A-JB-3P-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Pu-242
Method: Plutonium-242 by DOE A-01-R Mod
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6537							Spike List 6538						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
Plutonium 244	1	pCi/g			0														
Plutonium 242	1	pCi/g			0	C	Y			75	128	40	C	Y			75	128	40

TAL Reference Data Summary

tructured Analysis Code: I-J2-3K-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: ISO NEPTUNIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6526							Spike List 6558						
ı Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9 Neptunium 237	1	pCi/L			0	C	Y			58	126	40	C	Y			70	130	40

Printed at 6/30/2010 5:12:01 I

TAL Reference Data Summary

tructured Analysis Code: A-J2-3K-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: ISO NEPTUNIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6526							Spike List 6558						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9 Neptunium 237	1	pCi/g			0	C	Y			41	140	40	C	Y			68	112	40

Printed at 6/30/2010 5:14:23 F

TAL Reference Data Summary

tructured Analysis Code: A-K7-3K-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: ISO NEPTUNIUM (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6526							Spike List 6558						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9 Neptunium 237	1	pCi/g			0	C	Y			41	140	40	C	Y			68	112	40

Printed at 6/30/2010 5:15:01 F

TAL Reference Data Summary

tructured Analysis Code: **I-J2-3L-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: ISO NEPTUNIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6526							Spike List 6558						
ı Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
;9 Neptunium 237	0.10	pCi/L			0	C	Y			58	126	40	C	Y			70	130	40
'0 Np-237	0.10	pCi/L			0														

Printed at 6/30/2010 5:15:33 F

TAL Reference Data Summary

:ructured Analysis Code: **A-J2-3L-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: ISO NEPTUNIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits						Check List 6526						Spike List 6558					
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
) Neptunium 237	0.1	pCi/g			0	C	Y			41	140	40	C	Y			68	112	40

Printed at 6/30/2010 5:15:47 P

TAL Reference Data Summary

tructured Analysis Code: A-K7-3L-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: ISO NEPTUNIUM (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6526							Spike List 6558						
ı Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9 Neptunium 237	0.10	pCi/g			0	C	Y			41	140	40	C	Y			68	112	40
0 Np-237	0.10	pCi/g			0														

Printed at 6/30/2010 5:16:05 F

TAL Reference Data Summary

tructured Analysis Code: A-J2-3K-DO-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: ISO NEPTUNIUM (SHORT CT) DOE A-01-R MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6864							Spike List 6865						
ı	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
59	Neptunium 237	1	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

tructured Analysis Code: **I-J2-3K-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: ISO NEPTUNIUM (SHORT CT) DOE A-01-R MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6864							Spike List 6865						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
9 Neptunium 237	1	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

Printed at 6/30/2010 5:16:49 P

TAL Reference Data Summary

tructured Analysis Code: I-J2-2N-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Am241, Cm243/244 (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6512							Spike List 6556						
1	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4	Americium 241	0.1	pCi/L			0	C	Y			77	121	40	C	Y			70	130	40
8	Curium 243/244	0.1	pCi/L			0														
6	Curium 242	0.1	pCi/L			0														

Printed at 6/30/2010 5:17:13 F

TAL Reference Data Summary

Structured Analysis Code: **A-J2-2N-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Am241, Cm243/244 (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6512							Spike List 6556						
n	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
34	Americium 241	0.1	pCi/g			0	C	Y			73	130	40	C	Y			76	127	40
18	Curium 243/244	0.1	pCi/g			0														
36	Curium 242	0.1	pCi/g			0														

Printed at 6/30/2010 5:17:35

TAL Reference Data Summary

Structured Analysis Code: **A-K7-2N-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Am241, Cm243/244 (LONG CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6512							Spike List 6556						
n	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
34	Americium 241	0.1	pCi/g			0	C	Y			73	130	40	C	Y			76	127	40
18	Curium 243/244	0.1	pCi/g			0														
36	Curium 242	0.1	pCi/g			0														

Printed at 6/30/2010 5:17:52

TAL Reference Data Summary

tructured Analysis Code: I-J2-2J-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Am241, Cm243/244 (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6512							Spike List 6556						
1 Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
34 Americium 241	1.0	pCi/L			0	C	Y			77	121	40	C	Y			70	130	40
8 Curium 243/244	1.0	pCi/L			0														
36 Curium 242	1.0	pCi/L			0														

Printed at 6/30/2010 5:18:22 F

TAL Reference Data Summary

ructured Analysis Code: **A-J2-2J-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Am241, Cm243/244 (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6512							Spike List 6556						
Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
Americium 241	1.0	pCi/g			0	C	Y			73	130	40	C	Y			76	127	40
Curium 243/244	1.0	pCi/g			0														
Curium 242	1.0	pCi/g			0														

Printed at 6/30/2010 5:18:40 P

TAL Reference Data Summary

tructured Analysis Code: A-K7-2J-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Extraction Chromatography - Seq. Actinides
Method: Am241, Cm243/244 (SHORT CT) DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6512							Spike List 6556						
ı Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
34 Americium 241	1.0	pCi/g			0	C	Y			73	130	40	C	Y			76	127	40
8 Curium 243/244	1.0	pCi/g			0														
36 Curium 242	1.0	pCi/g			0														

Printed at 6/30/2010 5:18:55 I

TAL Reference Data Summary

tructured Analysis Code: **A-J2-2J-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - Sequential Actinides
Method: Am241, Cm243/244 (SHORT CT) DOE A-01-R MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6860							Spike List 6861						
1 Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
34 Americium 241	1.0	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25
8 Curium 243/244	1.0	pCi/g			0														
36 Curium 242	1.0	pCi/g			0														

Printed at 6/30/2010 5:19:17

TAL Reference Data Summary

Structured Analysis Code: I-J2-2J-DO-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - Sequential Actinides
Method: Am241, Cm243/244 (SHORT CT) DOE A-01-R MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6860							Spike List 6861						
n	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
84	Americium 241	1.0	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25
18	Curium 243/244	1.0	pCi/L			0														
36	Curium 242	1.0	pCi/L			0														

Printed at 6/30/2010 5:19:33

TAL Reference Data Summary

tructured Analysis Code: I-JC-3Q-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - IsoCm
Method: Cm245/246, Cm 247/248, Am243 DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6586							Spike List 6542						
ı Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
)3 Americium 243	1	pCi/L			0	C	Y			70	130	40	C	Y			70	130	40
;1 Curium-245/246	1	pCi/L			0														
9 Curium 247/248	1	pCi/L			0														

Printed at 6/30/2010 5:19:57 I

TAL Reference Data Summary

tructured Analysis Code: A-JC-3Q-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography - IsoCm
Method: Cm245/246, Cm 247/248, Am243 DOE A-01-R MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6586							Spike List 6542						
n	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
93	Americium 243	1	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40
51	Curium-245/246	1	pCi/g			0														
19	Curium 247/248	1	pCi/g			0														

Printed at 6/30/2010 5:20:25

TAL Reference Data Summary

tructured Analysis Code: **I-JC-3Q-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography - IsoCm
Method: Cm245/246, Cm 247/248, Am243 DOE A-01-R MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6862							Spike List 6863						
ı Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
3 Americium 243	1	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25
1 Curium-245/246	1	pCi/L			0	C	N			75	125	25	C	N			60	140	25
9 Curium 247/248	1	pCi/L			0														

Printed at 6/30/2010 5:21:08

Title: LIQUID SCINTILLATION COUNTER ANALYSIS

Approvals (Signature/Date):

Rhonda Ridenhower 7-15-10	Michael Ridenhower 7-15-10
Rhonda Ridenhower — Date	Michael Ridenhower — Date
Radiochemistry Manager	Health & Safety Manager / Coordinator
Marti Ward 7.16.10	Elaine Wild 7/15/10
Marti Ward — Date	Elaine Wild — Date
Quality Assurance Manager	Laboratory Director

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1.0 SCOPE AND APPLICATION

1.1 This procedure provides instruction for the operation of the Packard Tri-Carb series3170, Packard Tri-Carb series 3180, and Packard Tri-Carb 2550 CA/LL Liquid Scintillation Analyzer.

1.2 This procedure applies to all samples that are analyzed using the liquid scintillation counter, namely:

- Carbon 14
- Iodine 129
- Iron 55
- Lead 210
- Nickel 59 and 63
- Plutonium 241
- Technetium 99
- Tritium

1.3 The reporting limits, method detectable activities and QC limits are maintained in the Information Management System (QuantIMS). A copy of the Structure and Analysis Code (SAC), which lists this information, is included in the appendix of this SOP.

2.0 SUMMARY OF METHOD

2.1 The liquid scintillation counter is calibrated with standards containing a known quantity of H^3, C-14, Fe-55, I-129, Ni-59/63, Pb-210, Pu-241, and Tc-99. Not every instrument is calibrated for every nuclide. However, any instrument used to analyze a given method is calibrated for the nuclide requested.

2.2 Prepared samples are loaded into the sample tray and counted.

3.0 DEFINITIONS

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

3.2 Quenching - The interference with the conversion of decay energy to electronic signal in the photo multiplier tubes resulting in a reduction in counting efficiency. This can be caused by materials present in the scintillation solution that interfere with the process leading to the production of light as well as materials present that absorb the light before it reaches the photo multiplier tubes.

4.0 INTERFERENCES

4.1 See individual sample preparation SOPs.

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.

6.0 EQUIPMENT AND SUPPLIES

6.1 Packard Tri-Carb Series 3170, Packard 3180, Packard Tri-Carb 2550 CA/LL Liquid Scintillation Analyzers.

6.2 Liquid Scintillation Counter vials

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 Type obtained from the Milli-Q unit.

7.3 Unquenched standards:
- 7.3.1 Background Standard
- 7.3.2 Unquenched C -14 standard
- 7.3.3 Unquenched Fe-55 standard
- 7.3.4 Unquenched I-129 standard
- 7.3.5 Unquenched Ni-59/63 standard
- 7.3.6 Unquenched Pb-210 standard
- 7.3.7 Unquenched Pu-241 standard
- 7.3.8 Unquenched Tc-99 standard
- 7.3.9 Unquenched Tc-99 for Total Activity standard
- 7.3.10 Unquenched H3 standard
- 7.3.11 Unquenched Pm-147 standard

7.4 For Quench curves:
- 7.4.1 Quenched C14 standard
- 7.4.2 Quenched Fe-55 standard
- 7.4.3 Quenched I-129 standard
- 7.4.4 Quenched Ni-59/63 standard
- 7.4.5 Quenched Pb-210 standard
- 7.4.6 Quenched Pu-241 standard
- 7.4.7 Quenched Tc-99 standard
- 7.4.8 Quenched Tc-99 for Total Activity standard
- 7.4.9 Quenched H3 standard
- 7.4.10 Quenched Pm-147 standard

7.5 Standards may contain varying activities depending on the supplier and concentration availability of the nuclide of interest. However, all standards in a quench curve are counted until a minimum of 10,000 counts are collected.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

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

8.2 See individual sample preparation SOP for specific sample handling and storage requirements.

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.

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), Matrix Spike (MS) and a sample duplicate (SD). In the event that there is insufficient sample to analyze a sample duplicate, an LCS Duplicate (LCSD) is prepared and analyzed.

9.1.3.1 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.

9.2 **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.

9.3 **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.

9.4 **Matrix Spike**

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.

9.5 **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.

9.6 **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 Initial Calibration:

10.1.1 Prepare a quench curve for the nuclide of interest.

10.1.1.1 The process for quench curve preparation can be found on the print out of the previous calibration file.

10.1.1.1.1 See Attachment 1.

10.1.2 Frequency:

10.1.2.1 Quench curves are performed minimally on an annual basis.

10.1.2.1.1 A new calibration curve is generated after major changes to the system or when the continuing calibration criteria cannot be met. Major changes include any significant changes in instrument operating parameters, and major instrument maintenance.

10.1.3 Acceptance Criteria:

10.1.3.1 A minimum of 10,000 counts must be obtained for each data point

10.1.3.2 Correlation Coefficient (r^2) > 0.995 or each data point less than 7.5% relative to the calibrated value calculated from the fitted curve.

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

10.2 Initial Calibration Verification (ICV)

10.2.1 The initial calibration verification standard is a different standard source than the one used for the initial calibration.

10.2.2 Validate the quench curve by counting at least three second source standards.

10.2.2.1 The process of preparing validation standards is found on the print out of the previous calibration file. The verification standards should contain an unquenched standard as well as quenched standards that represent the quench range of live samples.

10.2.3 Frequency:

10.2.3.1 An ICV is performed with every initial calibration.

10.2.4 Acceptance Criteria:

10.2.4.1 Recovery +/- 10% of the known value.

10.2.4.1.1 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.

10.2.4.1.2 Corrective action must be taken (including reanalysis of the ICV or analysis of a different ICV).

10.3 Continuing Calibration:

10.3.1 Load standards and use the appropriate protocol flag as listed on the status page "Daily QC". The instrument will perform the required tests and the results are printed and saved to the Quality Control Table.

10.3.2 Frequency:

10.3.2.1 Quality control checks are performed for each day of use for the nuclides which measurements are being performed.

10.3.3 Acceptance Criteria:

10.3.3.1 The instruments are set at 2 sigma for a warning and 3 sigma for out of control limits.

10.3.3.2 If a Continuing Check fails and the analyst can document the reason for failure (e.g. carryover from the previous sample, etc.) then a second check may be analyzed without any adjustments to the instrument. If this check meets criteria then sample analysis may continue.

10.3.3.3 If this second check does not meet criteria, the analysis run is terminated. Instrument maintenance is performed and the instrument may require calibration.

11.0 PROCEDURE

11.1 Confirm the following:
- 11.1.1 Computer software is loaded and operational.
- 11.1.2 Printer is on-line.

11.2 Load sample vials by lifting the instrument cover and placing samples in tray with flag facing towards the left.

11.3 Samples are counted from left to right. QC checks are run daily. Prior to starting samples, make sure the daily QC has run and is acceptable.

11.4 Position 1 in the sample tray is reserved for the background vial.

11.5 Log the following information into the Run Log:
- 11.5.1 Date
- 11.5.2 Batch number
- 11.5.3 Sample I.D.
- 11.5.4 Protocol No.
- 11.5.5 Count length
- 11.5.6 Type of analysis
- 11.5.7 Operator's initials

11.6 Close instrument cover and allow sample vials to dark-adapt per the cocktail manufacturer's instructions, typically 30 minutes.

11.7 Spectrum are assessed for interferences, peak shape, etc. against a known source either the LCS or the instruments reference manual.

11.8 For the Packard 2550 LSC:
- 11.8.1 Press F1 key until Status Page is displayed on monitor. Enter the protocol number and press Enter.
- 11.8.2 Press F3 Key and verify the desired count time is entered. If needed, use the arrow keys to go to the count time parameter and type in the correct count time. Then press the enter key. Press F1 key to return to the Status Page.
- 11.8.3 Verify sample tray is in the correct order, starting with the background, blank, LCS, followed by the samples. Label the prep sheet with the correct counting order.
- 11.8.4 Press F10 key to go to the Worklist page.
- 11.8.4 Use the arrow keys to go to the beginning of the Worklist. Place the cursor on the second line of the Worklist. Press F3 key repeatedly until all the lot numbers from the previous analysis have been deleted.
- 11.8.5 Scan current batch in correct sample order. Press enter twice between each sample ID Press F1 to exit worklist. Press F2 to confirm exit.
- 11.8.6 Press F11 key to start the analysis.
- 11.8.7 Data is sent to the printer
- 11.8.8 When the batch is done counting, save the data file and transfer to a disk.
  - 11.8.8.1 To save the data file, select F5 (DOS Exit).
  - 11.8.8.2 Place disk in drive and change directory and save data as follows: Type command *CD\spectrum* (this will change to the spectrum directory where the data is retrieved)
  - 11.8.8.3 Type command *copy s.data X.XXX a:* (X.XXX will be found on the first page of the data printout). (This will copy the data to the disk)
  - 11.8.8.4 Type command rename sdata.X.XXX batch number i.e. *rename sdata1.123 875413.* (this will rename the file in the spectrum directory)
  - 11.8.8.5 Type command *exit* which will return to the status page. (The data is now saved to the disk)

11.8.8.6 Transfer the file the Upload folder, 2550 on SLSVR01.

11.9 For the Packard 3170 LSC and 3180 LSC:

11.9.1 Maximize the instrument window. Double click on the flag icon which has the protocol number that needs to be run. Click on the count conditions tab at the top of the analysis window. Enter the correct count time in the count time field.

11.9.2 Click on the Worklist tab at the top of the analysis window. Scan the samples from the Rad Capture paperwork.

11.9.3 Verify sample tray is in the correct order, starting with the background, blank, LCS, followed by the samples. Check tray order with the order appearing on the screen version of the worklist.

11.9.4 Click on the green start flag in the upper left corner of the instrument window.

11.9.5 Upon completion of counting, results will be sent to printer. The data is sent to the Upload folder, 3170 or 3180 on SLSVR01.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Calculations are performed in RadCapture (TestAmerica St. Louis custom software). These calculations are printed directly from the software and are included the TestAmerica St. Louis QAM.

12.2 Commonly used calculations (e.g. % recovery, RPD, uncertainty, MDC, tracer recovery) and standard instrument software calculations are given in the TestAmerica St. Louis 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 Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036. Below is a subset of the data assessment and QC excursion types within Clouseau; the text in underline is the exact "type" line in Clouseau. For a complete and current listing, please access the software program.

13.2 Method Blank

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit.

13.2.1.2 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> – See Clouseau NCM for corrective action (e.g. reprep/reanalysis, narration). Note certain analytes are common laboratory contaminants which require special narrative comment. These compounds are so designated in Clouseau.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within 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> – See Clouseau NCM for corrective action (e.g. , reanalysis, narration).

13.3.2.2 LCS Spike Recovery excursion (low) – See Clouseau NCM for corrective action (e.g. , reanalysis, narration).

13.3.2.3 RPD/RER Duplicate excursion – See Clouseau NCM for corrective action (e.g. , reanalysis, narration).

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. See Clouseau NCM to determine if re-preparation re-analysis is required.

13.5 Sample result evaluation

13.5.1 Tracer recovery must be within specified limits.
Tracer/Carrier recovery low– See Clouseau NCM for corrective action.

13.5.2 Tracer/Carrier recovery high– See Clouseau NCM for corrective action.

13.5.2.1 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:

13.5.2.2 Minimum number of tracer counts

13.5.2.3 Level of uncertainty

13.5.2.4 Client project requirements/approval

13.5.3 These expectations will be documented using the NCM process. The NCM will narrate the conditions upon which the sample results were accepted with tracer recovery excursions.

13.6 Insufficient Sample

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

14.0 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 QAM section 18.3.

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 QAM section 18.3.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in QAM section 18.3.

15.0 VALIDATION

15.1 Laboratory SOPs are based on standard reference EPA, DOE, and ASTM methodologies 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 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

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."

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 EPA-600/4-80-032. Prescribed Procedures for Measurement of Radioactivity in Drinking Water, Environmental Monitoring and support Laboratory, Section 10, Method 906, "Tritium in Drinking Water", August, 1980

17.2 Standard Methods for the Examination of Water and Wastewater. 19th Edition, Method 7500-1B Mod "Precipitation Method", 1985

17.3 EPA 520/5-84-006, EERF Radiochemistry Procedures Manual, Method C-01-1, "Radiochemical Determination of Carbon-14 in Aqueous Samples", August 1984

17.4 Eichrom Technologies, Inc. Analytical Procedures, TCS01, "TC-99 in Soil", April, 2002

17.5 Eichrom Technologies, Inc. Analytical Procedures,TCW01, TC-99 in Water, April 2002

17.6 Eichrom Technologies, Inc. Analytical Procedures,OTW01, Pb-210 in Water, April 2001

17.7 Eichrom Technologies, Inc. Analytical Procedures,OTS01, Pb-210 in Soil, August, 2002

17.8 TestAmerica Quality Assurance Manual (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 revisions

17.11.1 ST-PM-0002, Sample Receipt and Custody

17.11.2 ST-QA-0002, Standard and Reagent Preparation

17.11.3 ST-QA-0014, Evaluation of Analytical Accuracy and Precision Through the Use of Control Charts

17.11.4 ST-QA-0036, Non-conformance Memorandum (NCM) Process

17.11.5 ST-RC-0030, The Determination of Tritium in Water (and Other Fluids), Soil and Silica Gels
17.11.6 ST-RC-0031, Tritium Determination by Cryogenic Distillation
17.11.7 ST-RC-0042, Iodine-129 in Water
17.11.8 ST-RC-0055, Determination of Iron-55, Nickel-59 and Nickel-63 by Liquid Scintillation Spectrometry
17.11.9 ST-RC-0056 , Carbon-14 by Liquid Scintillation Spectrometry
17.11.10 ST-RC-0125, Determination of Technetium-99 using Eichrom® Teva Resin
17.11.11 ST-RC-0211, Determination of Lead-210 by Liquid Scintillation Counting
17.11.12 ST-RC-0245 , The Determination of Pu-241 by Liquid Scintillation Counting
17.11.12 ST-RC-0247, Promethium-147 and Samarium-151 analysis Utilizing Lanthide Resin Separation Procedure

18.0 CLARIFICATIONS, MODIFICATIONS TO THE REFERENCE METHOD

18.1 EPA method 906 is a drinking water method, and as such formulated its' Tritium calculation to reflect counts per minute (CPM) rather than disintegrations per minute (dpm). In true drinking water samples, it is acceptable to use CPM since drinking water samples should have little effect on efficiencies. However, EPA method 906 is commonly referenced for wastewater samples, and even silica gels and soil matrices. For these matrices, there can be a significant difference in efficiency and thus using dpm provides a more accurate result.

18.2 Calculation is given in the QAM.

19.0 CHANGES TO PREVIOUS REVISION

19.1 Added Promethium back to the list of standards in section 7.3.11 and 7.3.10.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Ni-59 Quench Curve – LSC 3170

STD #: Rad07-0006
Amount: 0.2 mL
Activity: 3634.5 dpm/mL
Reference Date: 10/31/06

Vial #	UGAB mL	3N HNO3 mL	DI H2O mL	Ni-59 Spike mL	Yellow Food Coloring uL
1	16	0	4	0.2	0
2	16	1	3	0.2	0
3	16	2	2	0.2	0
4	16	3	1	0.2	0
5	16	3	1	0.2	1
6	16	3	1	0.2	2
7	16	3	1	0.2	3
8	16	3	1	0.2	4
9	16	3	1	0.2	5

Yellow food coloring solution: 200 uL to 600 uL with DI H2O.

Prepared by: Sarah Hurst
Date: 01/30/08
Counted: 6/4/08

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Ni-63 Quench Curve - LSC 3170

STD #: Rad07-0008
Amount: 0.1 mL
Activity: 18229 dpm/mL
Reference Date: 01/01/2000

Vial #	UGAB mL	3N HNO3 mL	DI H2O mL	Ni-63 Spike mL	Yellow Food Coloring uL
1	16	0	4	0.1	0
2	16	1	3	0.1	0
3	16	2	2	0.1	0
4	16	3	1	0.1	0
5	16	3	1	0.1	1
6	16	3	1	0.1	2
7	16	3	1	0.1	3
8	16	3	1	0.1	4
9	16	3	1	0.1	5

Yellow food coloring solution: 200 uL to 600 uL with DI H2O.

Prepared by: Sarah Hurst
Date: 01/29/08

Recounted 6/2/08 RR

2008 Pb-210 Quench Curve Information

2008 Pb-210 Quench Curve Data				
Vial #	Nitromethane (mL)	D.I. Water (mL)	Ultima Gold AB (mL)	Cleaned STD (mL)
1	0.25	2.75	16	2
2	0.1	3	16	2
3	0.05	3	16	2
4	0.025	3	16	2
5	0	3	16	2
6	0	2	17	2
7	0	1	18	2

STD: Rad08-0017
Activity: 2256.0 dpm/mL
Reference date: 1/16/2008

Due to the presence of Bi-210 and Po-210 found in equilibrium with the Pb-210 in the calibration standard, a clean up procedure was used to selectively remove the Pb-210 from the contaminants. 8mL of the Rad08-0017 standard and 0.5mL of lead carrier (15mg/mL) were diluted up to 20mL with 1M nitric acid. After a thorough mixing, 0.2mL was removed (added to 10mL of 1M nitric in another digi-tube) for the initial ICP measurement. The remaining solution's lead was then isolated using Eichrom lead resin and the STL-RC-211 procedure. 0.2mL of the elution was removed (and added to 10mL of 1M nitric acid in another digi-tube) for the final ICP measurement. 2mL of the eluted solution was used for each quench curve point and back calculations were performed.

Prepped by: _____

Date: 04-29-08

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Pm-147 Quench Curve - LSC 3170

Pm-147 Quench Curve 03/01/2007 LSC 3170

Std is 2mL of Rad06-0050 2159.12 dpm/mL on 09/01/2004

Decay corrected to 03/01/2007 = 1116.98 dpm/mL 2mL = 2233.966 dpm
Cocktail is Ultima Gold AB

Std #	mL.01M HNO_3	mL 06-0050	mL UGB	uL Nitromethane
1	10	2	10	0
2	10	2	10	10
3	10	2	10	25
4	10	2	10	50
5	10	2	10	75
6	10	2	10	100
7	10	2	10	125
8	10	2	10	175
9	10	2	10	200

Perpared by Kim Young 2/22/2007

Pm-147

Initial Activity:	2.16E+03	dpm	
Reference Date:	9/1/2004		
Current Date:	3/1/2007		(or date at which you wish to determine activity)
Elapsed Time:	911	days	
Half Life:	958.05	days	22993.2
Exponential Term:	0.51731329		
Corrected Activity:	1116.98286	dpm	503.1454344 pCi

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Pu-241 Quench Curve
LSC 2250

Pu-241 Std Rad05-0042 103.2606 pCi/mL or 229.2387 dpm/mL on 06/20/05
Counted 1/14/2006

Table: Quench Curve Data
Pu-241, Ultima Gold AB, H2O, Nnitromethane
Prep by Ivan Vania 1/12/2006

Vial #	UGAB mL	H2O mL	Pu-241 mL	Nitromethane uL
1	15.0	1.000	3.0	0
2	15.0	0.990	3.0	10
3	15.0	0.980	3.0	20
4	15.0	0.970	3.0	30
5	15.0	0.955	3.0	45
6	15.0	0.940	3.0	60
7	15.0	0.925	3.0	75
8	15.0	0.910	3.0	90
9	15.0	0.900	3.0	100

3.0 mL Pu-241 Rad05-0042 = 671.716476 dpm

Plutonium-241

Initial Activity:	230.15 dpm		
Reference Date:	6/20/2005		
Current Date:	1/14/2006		(or date at which you wish to determine activity)
Elapsed Time:	208 days		
Half Life:	5241.338 days	125792.1	
Exponential Term:	0.972868		
Corrected Activity:	223.9055 dpm	100.8583297 pCi	

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Pu-241 Quench Curve – LSC 3170

STD #: Rad07-0019
Amount: 0.2 mL
Activity: 5644.4 dpm/mL
Reference date: 2/20/07

Pu-241 Quench Curve 2008				
Vial #	Ultima Gold (mL)	Pu241 Spike	2M nitric acid (mL)	Nitromethane (microliters)
1	15	0.2	3.8	0
2	15	0.2	3.8	10
3	15	0.2	3.8	20
4	15	0.2	3.8	30
5	15	0.2	3.8	45
6	15	0.2	3.8	60
7	15	0.2	3.8	75
8	15	0.2	3.8	90
9	15	0.2	3.8	100

Prepared by: Gina Fowler
Date: 04/15/08
0.2 mL of standard was used, decay corrected activity per standard is 1067 dpm/std

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Tc-99 Quench Curve - LSC 2200

Tc-99 Std 00-0056 10260.2 pCi/mL or 22778 dpm/mL
Counted 0/06/06

Table: Quench Curve Data
Tc-99, TEVA column, Ultima Gold, Nitric acid, Nitromethane

Vial #	Ultima Gold mL	TEVA Column	2N HNO3 mL	Tc-99 Spike mL	Nitromethane uL
1	15.0	0.3	0.000	0.100	0
2	15.0	0.7	0.100	0.100	10
3	15.0	1.0	0.250	0.100	25
4	15.0	1.0	0.250	0.100	50
5	15.0	1.0	0.250	0.100	100
6	15.0	1.0	0.250	0.100	150
7	15.0	1.0	0.250	0.100	200
8	15.0	1.0	0.250	0.100	250
9	15.0	1.0	0.250	0.100	300
10	15.0	1.0	0.250	0.100	400

Curve developed using RC-125 Rev. 1 and RD-0302 Rev. 1

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

Tc-99 Quench Curve – LSC 3170

STD #: Rad00-0053
Amount: 0.1mL
Activity: 10260.2pCi/mL
Reference date: 04/01/2000

Tc Quench Curve 2008				
Vial #	Insta Gel (mL)	Teva Column	2M nitric acid (mL)	Nitromethane (microliters)
1	15	0.3	0	0
2	15	0.7	0.1	10
3	15	1	0.25	25
4	15	1	0.25	50
5	15	1	0.25	75
6	15	1	0.25	100
7	15	1	0.25	150
8	15	1	0.25	200
9	15	1	0.25	250

Prepared by: Gina Fowler
Date: 06/18/08

Counted: 6/19/08

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

I-129 Quench Curve 2008				
STD amount	2N NH4OH	Nitromethane (in microliters)	Ultima Gold AB	Vial #
.1mL	0mL	0	20mL	1
.1mL	1mL	0	19mL	2
.1mL	2mL	0	18mL	3
.1mL	4mL	50	16mL	4
.1mL	5mL	75	15mL	5
.1mL	5mL	100	15mL	6
.1mL	5mL	125	14mL	7
.1mL	5mL	150	14mL	8
.1mL	5mL	175	13.5mL	9

Standard ID: Rad03-0079
Reference date: 1/1/2000
Activity: 23398.8dpm/mL
Half life of STD: 1.57e07 years
Decay correction not needed

Date of Prep: 4/5/2007
Date Counted: 4/17/08

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

H3 Quench Curve – LSC 3170

Date: 4/3/08
STD used: Rad05-0027
Amount used: 1mL
Activity: 18738dpm/mL
Reference date: 09/03/1998

STD #	mL H20	mL Rad05-0027	mL UGLLT	uL Nitromethane
1	4	1	15	0
2	7	1	12	0
3	9	1	10	0
4	9	1	10	5
5	9	1	10	10
6	9	1	10	15
7	9	1	10	25
8	9	1	10	50
9	9	1	10	75
10	9	1	10	100

Prepared by: Gina Fowler
Date: ~~5/01/08~~ GF 5/5/08

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

Fe-55 Quench Curve LSC 3170

STD: Rad05-0075
Activity: 226064 dpm/mL
Reference Date: 08/08/05

Fe-55 Quench Curve Data -2008				
Vial #	Ultima Gold AB (mL)	4.0M HNO3 (mL)	Fe-55 spike (mL)	yellow dye (microliters)
1	17.0	0.0	0.2	0
2	16.0	1.0	0.2	0
3	15.0	2.0	0.2	0
4	15.0	3.0	0.2	0
5	15.0	4.0	0.2	0
6	15.0	4.8	0.2	0
7	15.0	4.8	0.2	1
8	15.0	4.8	0.2	2
9	15.0	4.8	0.2	3
10	15.0	4.8	0.2	4
11	15.0	4.8	0.2	5

Yellow Food Coloring Solution: 200uL to 600uL with DI H2O.

Prepared by: Sarah Hurst
Date: 03/06/08

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

C-14 Quench Curve – LSC 3170

Date: 04/24/08
STD used: Rad04-0025
Activity: 23319.5dpm/mL
Amount used: 0.1mL
Reference date: 08/07/2002

STD #	mL CarbosorbE	mL Permafluor E	uL Nitromethane
1	10	10	0
2	10	10	10
3	10	10	20
4	10	10	30
5	10	10	40
6	10	10	50
7	10	10	60
8	10	10	70
9	10	10	80
10	10	10	90

Prepared by: Sarah Hurst

TAL Reference Data Summary

Structured Analysis Code: A-J3-3W-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography-> LSC Prep
Method: Ni-59 & Ni-63 by Liquid Scint. Spec.
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6564							Spike List 6565						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4160	Nickel 59	5	pCi/g			0	C	Y			71	114	40	C	Y			70	130	40
4067	Nickel 63	5	pCi/g			0	C	Y			84	123	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: I-J3-3W-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography-> LSC Prep
Method: Ni-59 & Ni-63 by Liquid Scint. Spec.
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6564							Spike List 6565						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4160	Nickel 59	5	pCi/L			0	C	Y			74	124	40	C	Y			70	130	40
4067	Nickel 63	5	pCi/L			0	C	Y			75	125	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: I-G8-1L-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Distillation and Suspended in LSC Cocktail
Method: Tritium by LSC by DOE H3-04-RC MOD.
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6507							Spike List 6552						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4045	Tritium	500	pCi/L			0	C	Y			85	111	40	C	Y			72	132	40

TAL Reference Data Summary

Structured Analysis Code: I-K5-1L-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: As Received, Direct Addition of Sample
Method: Tritium by LSC by DOE H3-04-RC MOD.
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List			Detection Limits				Check List 6507							Spike List 6552						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4045	Tritium	500	pCi/L			0	C	Y			85	111	40	C	Y			72	132	40

TAL Reference Data Summary

Structured Analysis Code: A-G8-1L-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Distillation and Suspended in LSC Cocktail
Method: Tritium by LSC by DOE H3-04-RC MOD.
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6507							Spike List 6552						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4045	Tritium	2	pCi/g			0	C	Y			75	104	40	C	Y			62	129	40

TAL Reference Data Summary

Structured Analysis Code: A-K6-1L-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Distillation and Suspension in LSC Cocktail
Method: Tritium by LSC by DOE H3-04-RC MOD.
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6507							Spike List 6552						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4045	Tritium	2	pCi/g			0	C	Y			75	104	40	C	Y			62	129	40

TAL Reference Data Summary

Structured Analysis Code: I-G8-ZC-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Distillation and Suspended in LSC Cocktail
Method: TRITIUM (Distill) by EPA 906.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6507							Spike List 6552						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4045	Tritium	500	pCi/L			0	C	Y			85	111	40	C	Y			72	132	40

TAL Reference Data Summary

Structured Analysis Code: A-G8-ZC-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Distillation and Suspended in LSC Cocktail
Method: TRITIUM (Distill) by EPA 906.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6507							Spike List 6552						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4045	Tritium	1	pCi/g			0	C	Y			75	104	40	C	Y			62	129	40
4046	H3	1	pCi/g			0														

TAL Reference Data Summary

Structured Analysis Code: A-HJ-ZC-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Cryogenic Tritium by LSC
Method: TRITIUM (Distill) by EPA 906.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List			Detection Limits				Check List 6560							Spike List 6561						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4045	Tritium	2	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: I-IM-2Q-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: TC-99 by Extraction Chromatography Resin (EIChroM)
Method: TC-99 by LSC by DOE TC-02-RC Mod.
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6523							Spike List 6559						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4113	Technetium 99	3	pCi/L			0	C	Y			78	110	40	C	Y			44	149	40

TAL Reference Data Summary

Structured Analysis Code: A-IM-2Q-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: TC-99 by Extraction Chromatography Resin (EIChroM)
Method: TC-99 by LSC by DOE TC-02-RC Mod.
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6523							Spike List 6559						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4113	Technetium 99	1	pCi/g			0	C	Y			74	110	40	C	Y			71	110	40

TAL Reference Data Summary

Structured Analysis Code: A-KY-2Q-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Oil - Muffle/Ext Chromatography Resin
Method: TC-99 by LSC by DOE TC-02-RC Mod.
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List			Detection Limits				Check List 6523							Spike List 6559						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4113	Technetium 99	1	pCi/g			0	C	Y			74	110	40	C	Y			71	110	40

TAL Reference Data Summary

Structured Analysis Code: A-LA-2Q-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: KD LEACH -> LIQUID/LIQUID EXTRACTION (BY KNOLL)
Method: TC-99 by LSC by DOE TC-02-RC Mod.
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List			Detection Limits				Check List 6523							Spike List 6559						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4113	Technetium 99	10	pCi/L			0	C	Y			74	110	40	C	Y			71	110	40

TAL Reference Data Summary

Structured Analysis Code: A-LG-2Q-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Recd,TC-99 by Extraction Chromatography Resin (ElChr
Method: TC-99 by LSC by DOE TC-02-RC Mod.
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6523							Spike List 6559						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4113	Technetium 99	1	pCi/g			0	C	Y			74	110	40	C	Y			71	110	40

TAL Reference Data Summary

Structured Analysis Code: A-JA-3O-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography -> LSC PREP
Method: Plutonium-241 by Liquid Scintillation
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6540							Spike List 6541						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4089	Plutonium 241	5	pCi/g			0	C	Y			61	132	40	C	Y			32	146	40

TAL Reference Data Summary

Structured Analysis Code: I-JA-3O-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography -> LSC PREP
Method: Plutonium-241 by Liquid Scintillation
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List			Detection Limits				Check List 6540							Spike List 6541						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4089	Plutonium 241	5	pCi/L	.		0	C	Y			70	106	40	C	Y			28	157	40

TAL Reference Data Summary

Structured Analysis Code: A-LH-3W-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Recd,Ion Exchange Resin preconcentration and purifical
Method: Ni-59 & Ni-63 by Liquid Scint. Spec.
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6564							Spike List 6565						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4160	Nickel 59	5	pCi/g			0	C	Y			71	114	40	C	Y			70	130	40
4067	Nickel 63	5	pCi/g			0	C	Y			84	123	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: A-J5-3Y-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Purification as Lead Chloride
Method: Pb-210 by Liquid Scint. Counting
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6567							Spike List 6590						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4156	Lead 210	5	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: I-J5-3Y-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Purification as Lead Chloride
Method: Pb-210 by Liquid Scint. Counting
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6567							Spike List 6590						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4156	Lead 210	5	pCi/L			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: I-J3-3V-01-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography-> LSC Prep
Method: Iron-55 by Liquid Scint. Spectrometry
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List			Detection Limits				Check List 6562							Spike List 6563						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4158	Iron 55	5	pCi/L			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-G8-4E-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Distillation and Suspended in LSC Cocktail
Method: Carbon 14 by EERF C-01-1
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6579							Spike List 6580						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4007	Carbon 14	20	pCi/L			0	C	Y			38	112	40	C	Y			37	121	40

TAL Reference Data Summary

Structured Analysis Code: A-G8-4E-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Distillation and Suspended in LSC Cocktail
Method: Carbon 14 by EERF C-01-1
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6579							Spike List 6580						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4007	Carbon 14	5	pCi/g			0	C	Y			62	109	40	C	Y			78	110	40

TAL Reference Data Summary

Structured Analysis Code: A-K6-4E-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Distillation and Suspension in LSC Cocktail
Method: Carbon 14 by EERF C-01-1
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List			Detection Limits				Check List 6579							Spike List 6580						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4007	Carbon 14	5	pCi/g			0	C	Y			62	109	40	C	Y			78	110	40

TAL Reference Data Summary

Structured Analysis Code: I-G8-1L-DO-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Distillation and Suspended in LSC Cocktail
Method: Tritium by LSC by DOE H3-04-RC MOD.
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6878							Spike List 6879						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4045	Tritium	500	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: A-G8-1L-DO-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Distillation and Suspended in LSC Cocktail
Method: Tritium by LSC by DOE H3-04-RC MOD.
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List			Detection Limits				Check List 6878							Spike List 6879						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4045	Tritium	2	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: I-G8-ZC-DO-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Distillation and Suspended in LSC Cocktail
Method: TRITIUM (Distill) by EPA 906.0 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6878							Spike List 6879						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4045	Tritium	500	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: A-G8-ZC-DO-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Distillation and Suspended in LSC Cocktail
Method: TRITIUM (Distill) by EPA 906.0 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6878							Spike List 6879						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4045	Tritium	1	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25
4046	H3	1	pCi/g			0														

TAL Reference Data Summary

Structured Analysis Code: A-IM-2Q-DO-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: TC-99 by Extraction Chromatography Resin (EIChroM)
Method: TC-99 by LSC by DOE TC-02-RC Mod.
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6876							Spike List 6877						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4113	Technetium 99	1	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-IM-2Q-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: TC-99 by Extraction Chromatography Resin (EIChroM)
Method: TC-99 by LSC by DOE TC-02-RC Mod.
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6876							Spike List 6877						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4113	Technetium 99	3	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: I-J3-3W-DO-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Extraction Chromatography-> LSC Prep
Method: Ni-59 & Ni-63 by Liquid Scint. Spec.
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6874							Spike List 6875						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4160	Nickel 59	5	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25
4067	Nickel 63	5	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: A-J3-3W-DO-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Extraction Chromatography-> LSC Prep
Method: Ni-59 & Ni-63 by Liquid Scint. Spec.
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6874							Spike List 6875						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4160	Nickel 59	5	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25
4067	Nickel 63	5	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: A-G8-4E-DO-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Distillation and Suspended in LSC Cocktail
Method: Carbon 14 by EERF C-01-1
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6872							Spike List 6873						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4007	Carbon 14	5	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: I-G8-4E-DO-06

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Distillation and Suspended in LSC Cocktail
Method: Carbon 14 by EERF C-01-1
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6872							Spike List 6873						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4007	Carbon 14	20	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

Title: LOW BACKGROUND GAS FLOW PROPORTIONAL COUNTING (GFPC) SYSTEM ANALYSIS

Approvals (Signature/Date):

3-14-11 Rhonda Ridenhower — Date Radiochemistry Manager	3/16/11 Michael Ridenhower — Date Health & Safety Manager / Coordinator
3-16-11 Marti Ward — Date Quality Assurance Manager	3/16/11 Elaine Wild — Date Laboratory Director

This SOP was previously identified as SOP No. ST-RD-0403 Rev. 10

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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 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.

1.3 The SOP applies to GFPC analysis of liquid and solid matrices.

1.4 The laboratory target analytes supported by this method, the reporting limits, method detection limits and QC limits are maintained in the Information Management System (QuantIMS). A copy of the Structure and Analysis Code (SAC), which lists this information, is included in the appendix of this SOP.

2.0 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 (QAM) for a glossary of common terms and data qualifiers.

3.2 IQC - a computerized Quality Control Program where the counting results of Radioactive check sources 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.

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

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

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 **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.

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>, a <u>Laboratory Control Sample</u> (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.

9.1.3.1 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.

9.2 **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.

9.3 **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.

9.4 **Matrix Spike**

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.

9.5 **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.

9.6 **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 Voltage Plateau Determination

10.1.1 **Frequency**:

10.1.1.1 Performed as a part of the Intial Calibration.

10.1.2 **Voltage Plateau Determination on LB4110 Red**

10.1.2.1 Place the Am-241 sources in Drawers A and B.

10.1.2.2 Select the auto-sequence file "Plateau"

10.1.2.3 Highlight the detectors in Drawers A and B

10.1.2.4 Click on 'Run"

10.1.2.5 Type 'Alpha' for the sample ID for the detectors with the alpha source

10.1.2.6 Click 'Done'. The voltage plateau will begin automatically.

10.1.2.7 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.

10.1.2.8 Place the Sr^{90} beta source on the detector.

10.1.2.9 Click 'Done'. The voltage plateau will complete automatically.

10.1.2.10 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.

10.1.2.11 Repeat the steps above for drawers C and D.

10.1.3 **Criteria for Plateaus for LB4110 Red**

10.1.3.1 Voltage range used to determine the plateau is 300-1500V

10.1.3.2 Voltage increase per step is <50V per step

10.1.4 **Voltage Plateau Determination on Protean MPC 9604**

10.1.4.1 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.

10.1.5 **Criteria for Plateaus for Protean MPC 9604**

10.1.5.1 Plateaus are permanently set for this instrument from the manufactuer.

10.2 **Discriminator Settings**

10.2.1 **Frequency:**

10.2.1.1 Performed as a part of the Intial Calibration.

10.2.2 **Discrimator Settings on LB4110 Red**

10.2.2.1 From the unit menu, select 'Change ROI'

10.2.2.2 Place a beta source in each detector

10.2.2.3 Highligh a detector with a beta source

10.2.2.4 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%.

10.2.2.5 Select 'Count'. Accumulate at least 100,000 beta counts

10.2.2.6 Reduce the beta upper limit/alpha lower limit until there is 3.5% beta into alpha crosstalk.
10.2.2.7 Raise the alpha lower limit until there is 0.10% beta into alpha crosstalk.
10.2.2.8 Raise the beta upper limit until it is 5% less than the alpha lower limit.
10.2.2.9 Select 'Halt'.
10.2.2.10 Repeat steps above until all detectors have been set.
10.2.2.11 Select 'Close'. Discriminator settings are updated automatically.

10.2.3 **Discrimator Settings on Protean MPC 9604**

10.2.3.1 Collect a minimum of 10,000 counts for each of Am-241, Th-230 and Po-210 sources
10.2.3.2 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.

10.3 **Initial Calibration**:

10.3.1 **Frequency**:

10.3.1.1 The Gas Flow Proportional Counter (GFPC) is calibrated initially and each year thereafter. Recalibration may be required if indicated during the operation of the instrument.

10.3.2 The specific calibration source preparations can be found in the file containing the previous calibration.

10.3.3 All nuclide sources shall be NIST traceable.

10.3.4 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.

10.3.5 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.

10.3.5.1 For alpha and beta analysis, separate sets of calibration sources shall be prepared.

10.3.6 The mass attenuation is accomplished by utilization of a salt solution with comparable make up to the majority of samples seen in the laboratory.

10.3.6.1 Alternatively, the mass attenuation may be accomplished by using the same carrier solution used in a specific analysis.

10.3.7 Each standard shall be counted in every detector to be calibrated.

10.3.8 **Criteria for a Single or Dual Set Calibration**

10.3.8.1 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.

10.3.8.2 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.

10.3.8.3 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.

10.3.8.4 The percent difference of the measured efficiency and theoretical efficiency shall be calculated for all data points.

10.3.8.5 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.

10.3.8.6 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.

10.3.8.7 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.

10.4 **Independent Calibration Verification (ICV)**

10.4.1 **Frequency**:

10.4.1.1 Performed with every intial calibration

10.4.2 GFPC initial calibrations must be verified by a second source standard.

10.4.3 The ICV standard is NIST traceable.

10.4.4 The ICV is counted to accumulate at least 5,000 counts.

10.4.5 **ICV for Dual Set Calibrations**:

10.4.5.1 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.

10.4.5.2 Prepare a blank at the same time.

10.4.5.3 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.

10.4.5.3.1 Alternatively, verification source nuclides may consist of different nuclides than the calibration curve if it is customary to do so.

10.4.5.4 Count each secondary source and the blank in all detectors that were calibrated.

10.4.5.5 Calculate the results in terms of percentage recovery.

10.4.5.6 Calculate the mean results of all masses across each detector.

10.4.5.7 Criteria:

10.4.5.7.1 Individual points are within 30 percent of the true value

10.4.5.7.2 The mean result of all masses across all detectors is less than 10 percent.

10.4.5.7.3 If any detector fails the validation tests the Technical Director must be consulted to provide corrective action.

10.4.6 **ICV for Single Set Calibrations**:

10.4.6.1 Prepare at least one set of verification sources varying in expected mass within the calibration range of the curve.

10.4.6.2 Prepare a blank at the same time.

10.4.6.3 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.

10.4.6.3.1 Alternatively, verification source nuclides may consist of different nuclides than the calibration curve if it is customary to do so.

10.4.6.4 Count the secondary source and the blank in all detectors that were calibrated.

10.4.6.5 Calculate the results in terms of percentage recovery.

10.4.6.6 Calculate the mean results of all masses across each detector.

10.4.6.7 Criteria:

10.4.6.7.1 Individual points are within 30 percent of the true value

10.4.6.7.2 The mean result of all masses across all detectors is less than 10 percent.

10.4.6.7.3 If any detector fails the validation tests the Technical Director must be consulted to provide corrective action.

10.5 **Setting Performance Check Criteria After Calibration**

10.5.1 Twenty background check samples are counted and used to establish quality control limits for the daily background checks.

10.5.2 Twenty alpha/beta check sources are counted after calibration and used to establish quality control limits for the daily source checks.

10.5.3 The limits will be a running average of the four months post calibration.

10.5.3.1 The limits are to be documented.

10.5.3.2 The limits will be re-established monthly at the following frequency

10.5.3.2.1 1st month – take first five data points from the new month and fifteen data points from the initial calibration.

10.5.3.2.2 2nd month – take first five points from new month, five from prior month and ten from initial calibration.

10.5.3.2.3 3rd month – take first five points from new month, five points each from the previous two months and five from the initial calibration.

10.5.3.2.4 4th month – take first five data points from new month and five points each from the previous three months.

10.5.3.3 Limits are set.

10.6 **Alpha to Beta Crosstalk Determination**

10.6.1 The mean mass is determined for each data point used to calculate the mass attenuation curve.

10.6.1.1 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.

10.6.1.2 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.

10.6.1.3 The crosstalk curve is plotted as mean crosstalk values relative to the mean mass for the two sets of data.

10.6.1.3.1 In this manner the crosstalk factor can be determined for any given mass.

10.6.1.4 The equation of the curve shall be determined using polynomial functions.

10.6.1.5 The coefficient of determination (R^2) shall be calculated and displayed on the plot as well as the equation for the trendline.

10.7 **Beta to Alpha Crosstalk Determination**

10.7.1 Since beta to alpha crosstalk does not vary across mass, a mean beta to alpha crosstalk correction factor is calculated.

10.7.2 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.

10.7.3 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.

10.8 **Long Background**

10.8.1 **Frequency**:

10.8.1.1 Monthly or whenever instrument conditions have significantly changed since the previous background was performed (e.g. detector replaced, etc.)

10.8.1.2 Minimum count time: 1000 minutes.

10.8.2 Wash the planchet holder and clean the drawers with a 20% radiac wash or ethyl alcohol.

10.8.2.1 Do not spray cleaner directly onto the drawers. Spray cleaner on a Kimwipe, a cotton ball, or paper towel and wipe out the drawers.

10.8.3 Check that instrument settings are as specified in 11.1.

10.8.4 **Red Long Background Count Set Up**

10.8.4.1 Place the cursor on the red box in the upper left hand corner of the screen and right click on the mouse.

10.8.4.2 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.

10.8.4.3 Place clean empty planchets in instruments.

10.8.4.4 Place the cursor on the red box in the upper left hand corner of the screen and right click on the mouse.

10.8.4.5 Select 'create batch' from the instrument menu.

10.8.4.6 Select 'background'

10.8.4.7 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.

10.8.4.8 Select 'run'

10.8.4.9 Select 'done'

10.8.4.10 When backgrounds are complete, review the printouts for acceptance.

10.8.5 **Protean Long Background Count Set Up**

10.8.5.1 Double click on detector 0.

10.8.5.2 Select the 'source' bullet.

10.8.5.3 Select LB0 by clicking on the file list arrow. (L for long. B for background. 0 for detector).

10.8.5.4 Ensure count time is set to 1000 minutes.

10.8.5.5 Select 'start'.

10.8.5.6 Continue these steps with detectors 1-15.

10.8.5.7 Review the data for acceptance when the backgrounds are complete.

10.8.6 **Orange and Purple Long Background Count Set Up**

10.8.6.1 Select detector 0

10.8.6.2 Select 'source' bullet

10.8.6.3 Select 'monthly long background' by clicking on the file list arrow.

10.8.6.4 Ensure count time is set to 1000 minutes

10.8.6.5 Select 'start'

10.8.6.6 Continue these steps with detectors 1-23.

10.8.6.7 Review the data for acceptance when the backgrounds are complete.

10.8.7 **Long Background Criteria:**

10.8.7.1 The CPM for the alpha are <0.2 and the beta < 2.0, the detector may be used.

10.8.7.1.1 The data printout must include initials and date to indicate that has been reviewed.

10.8.7.1.2 If a detector is above this limit, discard planchet.

10.8.7.1.3 Clean the planchet holder with radiac wash, ethyl alcohol or a detergent spray cleaner and dry thoroughly.
10.8.7.1.4 Place a clean planchet in the holder and repeat steps for that detector (s) only.
10.8.7.1.5 Perform a new background.
10.8.7.1.5.1 Note: the detector is tagged out of service until a successful background has been achieved.

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 in the maintenance logbook.

11.2 Record date of Daily Background and Check Source Data in runlog logbook.

11.3 Maintenance
11.3.1.1 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.
11.3.1.2 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 right click 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 each file generated above individually, 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 QAM.

12.2 Result calculations are performed by TestAmerica St. Louis' Rad Capture software program. These calculations are found in the TestAmerica St. Louis 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 Clouseau Nonconformance Memorandum (NCM) process. The NCM process is described in SOP: ST-QA-0036. Below is a subset of the data assessment and QC excursion types within Clouseau; the text in underline is the exact "type" line in Clouseau. For a complete and current listing, please access the software program.

13.2 Method Blank

13.2.1 Acceptance Criteria:

13.2.1.1 No target analytes may be present in the method blank above the reporting limit.

13.2.1.2 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 – See Clouseau NCM for corrective action (e.g. reprep/reanalysis, narration). Note certain analytes are common laboratory contaminants which require special narrative comment. These compounds are so designated in Clouseau.

13.3 Laboratory Control Sample (LCS)

13.3.1 Acceptance Criteria:

13.3.1.1 All control analytes must be within 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) – See Clouseau NCM for corrective action (e.g. , reanalysis, narration).

13.3.2.2 LCS Spike Recovery excursion (low) – See Clouseau NCM for corrective action (e.g. , reanalysis, narration).

13.3.2.3 RPD/RER Duplicate excursion – See Clouseau NCM for corrective action (e.g. , reanalysis, narration).

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. See Clouseau NCM to determine if re-preparation re-analysis is required.

13.5 Sample result evaluation

13.5.1 Tracer/Carrier recovery must be within specified limits.

13.5.2 Tracer/Carrier recovery low– See Clouseau NCM for corrective action.

13.5.3 Tracer/Carrier recovery high– See Clouseau NCM for corrective action.

13.5.3.1 A sample tracer recovery outside QC limits may be accepted if the sample results are determined valid:

13.5.3.2 minimum number of tracer counts

13.5.3.3 level of uncertainty

13.5.3.4 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.

13.6 Insufficient Sample

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

14.0 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 QAM section 18.3.

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 QAM section 18.3.

14.4 Annually, the analyst must successfully demonstrate proficiency to continue to perform this analysis. See requirements in QAM section 18.3.

15.0 VALIDATION

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

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:
- 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-0036, "Non-Conformance Memorandum (NCM) Process"
- 17.11.4 ST-RC-0004, "Preparation of Soil, Sludge, Filter, Biota and Oil/Grease Samples for Radiochemical Analysis".
- 17.11.5 ST-RC-0020, "Determination of Gross Alpha/Beta Activity"
- 17.11.6 ST-RC-0021, "Gross Alpha Radition in Water using Copreciptation"
- 17.11.7 ST-RC-0036, "Determination of Chlorine-36 in Various Matrices by GFPC"
- 17.11.8 ST-RC-0040, 'Total Alpha Emitting Isotopes of Radium"
- 17.11.9 ST-RC-0041, "Radium 228 in Water"
- 17.11.10 ST-RC-0050, "Preparation of Strontium 89 and 90"
- 17.11.11 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 daughters in-grow, the 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's 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 Radium 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; Added instument Purple throughout section 10 and 11.
19.3 Rev. 11; Adjusted procedure steps throughout section 11.

TAL Reference Data Summary

Structured Analysis Code: **I-FR-ZA-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Evaporative Preparation, Total
Method: GROSS A/B BY GFPC SW846 9310 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6501							Spike List 6548						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	3	pCi/L			0	C	Y			62	134	40	C	Y			35	150	40
4171	Gross Beta	4	pCi/L			0	C	Y			58	133	40	C	Y			54	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-HZ-ZA-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Evaporative Preparation, Dissolved
Method: GROSS A/B BY GFPC SW846 9310 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6498							Spike List 6533						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5630	Gross Alpha, Dissolved	3	pCi/L			0	C	Y			80	140	40	C	Y			33	150	40
5632	Gross Beta, Dissolved	4	pCi/L			0	C	Y			77	123	40	C	Y			71	146	40

TAL Reference Data Summary

Structured Analysis Code: **I-H6-ZA-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Evaporative Preparation, Suspended
Method: GROSS A/B BY GFPC SW846 9310 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6499							Spike List 6534						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5629	Gross Alpha, Suspended	3	pCi/L			0	C	Y			73	136	40	C	Y			44	150	40
5631	Gross Beta, Suspended	4	pCi/L			0	C	Y			73	122	40	C	Y			66	147	40

TAL Reference Data Summary

Structured Analysis Code: **A-FS-ZA-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Thin Layer on Planchet
Method: GROSS A/B BY GFPC SW846 9310 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6501							Spike List 6548						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	10	pCi/g			0	C	Y			38	138	40	C	Y			43	123	40
4171	Gross Beta	10	pCi/g			0	C	Y			44	124	40	C	Y			55	125	40

TAL Reference Data Summary

Structured Analysis Code: **A-HI-ZA-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Total Dissolution
Method: GROSS A/B BY GFPC SW846 9310 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6527							Spike List 6528						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	10	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40
4171	Gross Beta	10	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-K2-ZA-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Thin Layer on Planchet
Method: GROSS A/B BY GFPC SW846 9310 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6501							Spike List 6548						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	10	pCi/g			0	C	Y			38	138	40	C	Y			43	123	40
4171	Gross Beta	10	pCi/g			0	C	Y			44	124	40	C	Y			55	125	40

TAL Reference Data Summary

Structured Analysis Code: **I-FR-Y9-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Evaporative Preparation, Total
Method: Gross Alpha/Beta EPA 900
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6501							Spike List 6548						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	3	pCi/L			0	C	Y			62	134	40	C	Y			35	150	40
4171	Gross Beta	4	pCi/L			0	C	Y			58	133	40	C	Y			54	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-HZ-Y9-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Evaporative Preparation, Dissolved
Method: Gross Alpha/Beta EPA 900
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6584							Spike List 6585						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5630	Gross Alpha, Dissolved	3	pCi/L			0	C	Y			63	130	40	C	Y			63	130	40
5632	Gross Beta, Dissolved	4	pCi/L			0	C	Y			80	120	40	C	Y			80	120	40

TAL Reference Data Summary

Structured Analysis Code: **I-H6-Y9-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Evaporative Preparation, Suspended
Method: Gross Alpha/Beta EPA 900
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6582							Spike List 6583						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5629	Gross Alpha, Suspended	3	pCi/L			0	C	Y			63	130	40	C	Y			63	130	40
5631	Gross Beta, Suspended	4	pCi/L			0	C	Y			80	120	40	C	Y			80	120	40

TAL Reference Data Summary

Structured Analysis Code: **I-J6-Y9-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: 48 HOUR - EVAPORATIVE PREPARATION
Method: Gross Alpha/Beta EPA 900
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6501							Spike List 6548						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	3	pCi/L			0	C	Y			62	134	40	C	Y			35	150	40
4171	Gross Beta	4	pCi/L			0	C	Y			58	133	40	C	Y			54	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-MH-Y9-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: 48 hour- Evaporative Prep Suspended
Method: Gross Alpha/Beta EPA 900
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6501							Spike List 6548						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	2	pCi/L			0	C	Y			62	134	40	C	Y			35	150	40
4171	Gross Beta	2	pCi/L			0	C	Y			58	133	40	C	Y			54	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-MI-Y9-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: 48 hour- Evaporative Prep Dissolved
Method: Gross Alpha/Beta EPA 900
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6501							Spike List 6548						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	2	pCi/L			0	C	Y			62	134	40	C	Y			35	150	40
4171	Gross Beta	2	pCi/L			0	C	Y			58	133	40	C	Y			54	150	40

TAL Reference Data Summary

Structured Analysis Code: **A-FS-Y9-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Thin Layer on Planchet
Method: Gross Alpha/Beta EPA 900
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6501							Spike List 6548						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	10	pCi/g			0	C	Y			38	138	40	C	Y			43	123	40
4171	Gross Beta	10	pCi/g			0	C	Y			44	124	40	C	Y			55	125	40

TAL Reference Data Summary

Structured Analysis Code: **A-HI-Y9-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Total Dissolution
Method: Gross Alpha/Beta EPA 900
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6527							Spike List 6528						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	10	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40
4171	Gross Beta	10	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-J6-3Z-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: 48 HOUR - EVAPORATIVE PREPARATION
Method: Gross A/B by NJ EPA 900 PWTA
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6501							Spike List 6548						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	2	pCi/L			0	C	Y			62	134	40	C	Y			35	150	40
4171	Gross Beta	2	pCi/L			0	C	Y			58	133	40	C	Y			54	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-FR-ZA-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Evaporative Preparation, Total
Method: GROSS A/B BY GFPC SW846 9310 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6880							Spike List 6881						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	3	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25
4171	Gross Beta	4	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-FS-ZA-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Thin Layer on Planchet
Method: GROSS A/B BY GFPC SW846 9310 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6880							Spike List 6881						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	10	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25
4171	Gross Beta	10	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-FR-Y9-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Evaporative Preparation, Total
Method: Gross Alpha/Beta EPA 900
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6880							Spike List 6881						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	3	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25
4171	Gross Beta	4	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-FS-Y9-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Thin Layer on Planchet
Method: Gross Alpha/Beta EPA 900
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6880							Spike List 6881						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4170	Gross Alpha	10	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25
4171	Gross Beta	10	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-G2-ZW-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth
Method: Radium 226 by SW846 9315 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/L			0	C	Y			68	136	40	C	Y			50	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-G3-ZW-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 14 day Ingrowth
Method: Radium 226 by SW846 9315 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/L			0	C	Y			68	136	40	C	Y			50	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-NR-ZW-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth, Dissolved
Method: Radium 226 by SW846 9315 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5656	Radium (226), Dissolved	1	pCi/L			0														

TAL Reference Data Summary

Structured Analysis Code: **A-G1-ZW-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 14day ingrowth
Method: Radium 226 by SW846 9315 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/g			0	C	Y			64	138	40	C	Y			72	140	40

TAL Reference Data Summary

Structured Analysis Code: **A-G5-ZW-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth
Method: Radium 226 by SW846 9315 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/g			0	C	Y			64	138	40	C	Y			72	140	40

TAL Reference Data Summary

Structured Analysis Code: **A-RC-ZW-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Ra-226, ASTM 3987-85/ SW846 9315
Method: Radium 226 by SW846 9315 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/L			0	C	Y			64	138	40	C	Y			72	140	40

TAL Reference Data Summary

Structured Analysis Code: **I-G2-Z3-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth
Method: Radium 226 by DOE RA-06-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/L			0	C	Y			68	136	40	C	Y			50	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-G3-Z3-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 14 day Ingrowth
Method: Radium 226 by DOE RA-06-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/L			0	C	Y			68	136	40	C	Y			50	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-G1-Z3-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 14day ingrowth
Method: Radium 226 by DOE RA-06-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/g			0	C	Y			64	138	40	C	Y			72	140	40

TAL Reference Data Summary

Structured Analysis Code: **A-G5-Z3-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth
Method: Radium 226 by DOE RA-06-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/g			0	C	Y			64	138	40	C	Y			72	140	40

TAL Reference Data Summary

Structured Analysis Code: **A-K4-Z3-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Digest, Precipitate, Seperation - 14 Day Ingro
Method: Radium 226 by DOE RA-06-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/g			0	C	Y			64	138	40	C	Y			72	140	40

TAL Reference Data Summary

Structured Analysis Code: **I-G2-ZY-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth
Method: Radium 226 by EPA 903.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/L			0	C	Y			68	136	40	C	Y			50	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-G3-ZY-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 14 day Ingrowth
Method: Radium 226 by EPA 903.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/L			0	C	Y			68	136	40	C	Y			50	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-P3-ZY-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 14 day Ingrowth, Dissolved
Method: Radium 226 by EPA 903.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6321							Spike List 6322						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5656	Radium (226), Dissolved	1	pCi/L			0	C	Y			45	150	40	C	Y			50	130	40

TAL Reference Data Summary

Structured Analysis Code: A-G1-ZY-01-06

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 14day ingrowth
Method: Radium 226 by EPA 903.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/g			0	C	Y			64	138	40	C	Y			72	140	40

TAL Reference Data Summary

Structured Analysis Code: **A-G5-ZY-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth
Method: Radium 226 by EPA 903.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/g			0	C	Y			64	138	40	C	Y			72	140	40

TAL Reference Data Summary

Structured Analysis Code: **A-S7-ZY-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As received, Digest, Precipitate, Separation 21 day ingrowtr
Method: Radium 226 by EPA 903.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6504							Spike List 6550						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/g			0	C	Y			64	138	40	C	Y			72	140	40

TAL Reference Data Summary

Structured Analysis Code: **I-G2-ZY-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth
Method: Radium 226 by EPA 903.0 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6882							Spike List 6883						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-G5-ZY-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth
Method: Radium 226 by EPA 903.0 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6882							Spike List 6883						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-G2-Z3-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth
Method: Radium 226 by DOE RA-06-RC MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6882							Spike List 6883						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-G5-Z3-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth
Method: Radium 226 by DOE RA-06-RC MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6882							Spike List 6883						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-G2-ZW-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth
Method: Radium 226 by SW846 9315 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6882							Spike List 6883						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-G5-ZW-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth
Method: Radium 226 by SW846 9315 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6882							Spike List 6883						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2257	Radium (226)	1	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-G2-Z5-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth
Method: Radium 228 by GFPC SW846 9320 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/L			0	C	Y			60	142	40	C	Y			64	150	40
4152	Ra-228	1	pCi/L			0														

TAL Reference Data Summary

Structured Analysis Code: **I-G3-Z5-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 14 day Ingrowth
Method: Radium 228 by GFPC SW846 9320 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/L			0	C	Y			60	142	40	C	Y			64	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-NR-Z5-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth, Dissolved
Method: Radium 228 by GFPC SW846 9320 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5657	Radium 228, Dissolved	1	pCi/L			0														

TAL Reference Data Summary

Structured Analysis Code: **A-G1-Z5-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 14day ingrowth
Method: Radium 228 by GFPC SW846 9320 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/g			0	C	Y			62	132	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **A-G5-Z5-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth
Method: Radium 228 by GFPC SW846 9320 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/g			0	C	Y			62	132	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **A-RD-Z5-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Ra-228, ASTM 3987-85/ SW846 9320
Method: Radium 228 by GFPC SW846 9320 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/L			0	C	Y			62	132	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-G2-Z6-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth
Method: Radium 228 by GFPC DOE RA-06-RC Mod
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/L			0	C	Y			60	142	40	C	Y			64	150	40
4152	Ra-228	1	pCi/L			0														

TAL Reference Data Summary

Structured Analysis Code: **I-G3-Z6-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 14 day Ingrowth
Method: Radium 228 by GFPC DOE RA-06-RC Mod
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/L			0	C	Y			60	142	40	C	Y			64	150	40

TAL Reference Data Summary

Structured Analysis Code: **A-G1-Z6-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 14day ingrowth
Method: Radium 228 by GFPC DOE RA-06-RC Mod
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/g			0	C	Y			62	132	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **A-G5-Z6-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth
Method: Radium 228 by GFPC DOE RA-06-RC Mod
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/g			0	C	Y			62	132	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **A-K4-Z6-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Digest, Precipitate, Seperation - 14 Day Ingro
Method: Radium 228 by GFPC DOE RA-06-RC Mod
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/g			0	C	Y			62	132	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-G2-Z4-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth
Method: Radium 228 by GFPC EPA 904 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/L			0	C	Y			60	142	40	C	Y			64	150	40
4152	Ra-228	1	pCi/L			0														

TAL Reference Data Summary

Structured Analysis Code: **I-G3-Z4-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 14 day Ingrowth
Method: Radium 228 by GFPC EPA 904 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/L			0	C	Y			60	142	40	C	Y			64	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-P3-Z4-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 14 day Ingrowth, Dissolved
Method: Radium 228 by GFPC EPA 904 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6323							Spike List 6324						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5657	Radium 228, Dissolved	1	pCi/L			0	C	Y			60	149	40	C	Y			62	150	40

TAL Reference Data Summary

Structured Analysis Code: **A-G1-Z4-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 14day ingrowth
Method: Radium 228 by GFPC EPA 904 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/g			0	C	Y			62	132	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **A-G5-Z4-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth
Method: Radium 228 by GFPC EPA 904 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/g			0	C	Y			62	132	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **A-S7-Z4-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As received, Digest, Precipitate, Separation 21 day ingrowth
Method: Radium 228 by GFPC EPA 904 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/g			0	C	Y			62	132	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-G2-Z4-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth
Method: Radium 228 by GFPC EPA 904 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6884							Spike List 6885						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25
4152	Ra-228	1	pCi/L			0														

TAL Reference Data Summary

Structured Analysis Code: **A-G5-Z4-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth
Method: Radium 228 by GFPC EPA 904 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6884							Spike List 6885						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-G5-Z6-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth
Method: Radium 228 by GFPC DOE RA-06-RC Mod
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/g			0	C	Y			62	132	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-G2-Z6-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth
Method: Radium 228 by GFPC DOE RA-06-RC Mod
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/L			0	C	Y			60	142	40	C	Y			64	150	40
4152	Ra-228	1	pCi/L			0														

TAL Reference Data Summary

Structured Analysis Code: **I-G2-Z5-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation - 21 day Ingrowth
Method: Radium 228 by GFPC SW846 9320 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/L			0	C	Y			60	142	40	C	Y			64	150	40
4152	Ra-228	1	pCi/L			0														

TAL Reference Data Summary

Structured Analysis Code: **A-G5-Z5-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation - 21day ingrowth
Method: Radium 228 by GFPC SW846 9320 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6505							Spike List 6551						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2259	Radium 228	1	pCi/g			0	C	Y			62	132	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-LP-2Z-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: Total Alpha Radium EPA 903.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6503							Spike List 6596						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2261	Radium (total)	1	pCi/L			0	C	Y			70	125	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-LQ-2Z-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Precipitate, Separation
Method: Total Alpha Radium EPA 903.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6503							Spike List 6596						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2261	Radium (total)	1	pCi/g			0	C	Y			52	114	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-L1-2Z-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Precipitate, Separation
Method: Total Alpha Radium EPA 903.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6503							Spike List 6596						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2261	Radium (total)	1	pCi/g			0	C	Y			52	114	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-LP-20-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: Total Radium by DOE RA-06-RC
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6890							Spike List 6891						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2261	Radium (total)	1	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-LQ-20-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Precipitate, Separation
Method: Total Radium by DOE RA-06-RC
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6890							Spike List 6891						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
2261	Radium (total)	1	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-FX-0K-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: SR-89 BY GFPC EPA 905.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6524							Spike List 6516						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4109	Strontium 89	3	pCi/L			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-FW-0K-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation
Method: SR-89 BY GFPC EPA 905.0 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6524							Spike List 6516						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4109	Strontium 89	3	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-FX-0J-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: SR-89 BY GFPC DOE SR-01-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6524							Spike List 6516						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4109	Strontium 89	3	pCi/L			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-FW-0J-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation
Method: SR-89 BY GFPC DOE SR-01-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6524							Spike List 6516						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4109	Strontium 89	3	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-K3-0J-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Digest, Precipitate, Seperation
Method: SR-89 BY GFPC DOE SR-01-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6524							Spike List 6516						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4109	Strontium 89	3	pCi/g			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-FX-0J-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: SR-89 BY GFPC DOE SR-01-RC MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6886							Spike List 6887						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4109	Strontium 89	3	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-FW-0J-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation
Method: SR-89 BY GFPC DOE SR-01-RC MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6886							Spike List 6887						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4109	Strontium 89	3	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-FX-0K-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: SR-89 BY GFPC EPA 905.0 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6886							Spike List 6887						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4109	Strontium 89	3	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-FW-0K-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation
Method: SR-89 BY GFPC EPA 905.0 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6886							Spike List 6887						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4109	Strontium 89	3	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-FX-ZO-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: SR-90 BY GFPC EPA-905 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6502							Spike List 6549						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4111	Strontium 90	3	pCi/L			0	C	Y			80	130	40	C	Y			19	150	40

TAL Reference Data Summary

Structured Analysis Code: **A-FW-ZO-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation
Method: SR-90 BY GFPC EPA-905 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6502							Spike List 6549						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4111	Strontium 90	3	pCi/g			0	C	Y			86	127	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-HL-ZO-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: PREP RC-0003 , RC-0004 , SEP RC-0050 , CT SL-13021
Method: SR-90 BY GFPC EPA-905 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6502							Spike List 6549						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4111	Strontium 90	3	pCi/g			0	C	Y			86	127	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: I-FX-ZK-01-06

Target Analyte List: 'All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: Strontium-90 by GFPC DOE SR-03-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6502							Spike List 6549						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4111	Strontium 90	3	pCi/L			0	C	Y			80	130	40	C	Y			19	150	40

TAL Reference Data Summary

Structured Analysis Code: **A-FW-ZK-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation
Method: Strontium-90 by GFPC DOE SR-03-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6502							Spike List 6549						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4111	Strontium 90	3	pCi/g			0	C	Y			86	127	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-HL-ZK-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: PREP RC-0003 , RC-0004 , SEP RC-0050 , CT SL-13021
Method: Strontium-90 by GFPC DOE SR-03-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6502							Spike List 6549						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4111	Strontium 90	3	pCi/g			0	C	Y			86	127	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-K3-ZK-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Digest, Precipitate, Seperation
Method: Strontium-90 by GFPC DOE SR-03-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6502							Spike List 6549						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4111	Strontium 90	3	pCi/g			0	C	Y			86	127	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-FX-ZK-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: Strontium-90 by GFPC DOE SR-03-RC MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6888							Spike List 6889						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4111	Strontium 90	3	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-FW-ZK-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation
Method: Strontium-90 by GFPC DOE SR-03-RC MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List			Detection Limits				Check List 6888							Spike List 6889						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4111	Strontium 90	3	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-FX-ZK-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: Strontium-90 by GFPC DOE SR-03-RC MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6888							Spike List 6889						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4111	Strontium 90	3	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-FW-ZK-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation
Method: Strontium-90 by GFPC DOE SR-03-RC MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6888							Spike List 6889						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4111	Strontium 90	3	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-FX-ZM-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: Total SR BY GFPC EPA-905 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6525							Spike List 6593						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4154	Strontium Total	3	pCi/L			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-FW-ZM-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation
Method: Total SR BY GFPC EPA-905 MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6525							Spike List 6593						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4154	Strontium Total	3	pCi/g			0	C	Y			72	110	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-FX-ZM-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: Total SR BY GFPC EPA-905 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6892							Spike List 6893						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4154	Strontium Total	3	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-FW-ZM-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation
Method: Total SR BY GFPC EPA-905 MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6892							Spike List 6893						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4154	Strontium Total	3	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-FX-ZJ-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: Total Strontium by GFPC DOE SR-03-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6525							Spike List 6593						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4154	Strontium Total	3	pCi/L			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-FW-ZJ-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation
Method: Total Strontium by GFPC DOE SR-03-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6525							Spike List 6593						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4154	Strontium Total	3	pCi/g			0	C	Y			72	110	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-K3-ZJ-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: As Received, Digest, Precipitate, Seperation
Method: Total Strontium by GFPC DOE SR-03-RC MOD
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6525							Spike List 6593						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4154	Strontium Total	10	pCi/sampl			0	C	Y			72	110	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **I-FX-ZJ-DO-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Precipitate, Separation
Method: Total Strontium by GFPC DOE SR-03-RC MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6892							Spike List 6893						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4154	Strontium Total	3	pCi/L			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **A-FW-ZJ-DO-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Digest, Precipitate, Separation
Method: Total Strontium by GFPC DOE SR-03-RC MOD
QC Program: DOE QSAS
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6892							Spike List 6893						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
4154	Strontium Total	3	pCi/g			0	C	Y			75	125	25	C	Y			60	140	25

TAL Reference Data Summary

Structured Analysis Code: **I-JE-3S-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Dry, Grind, Leach/Digestion, phos ppt. -> amm. ppt.
Method: Phosphorus-32/33 by GFPC
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6544							Spike List 6568						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5481	Phosphorus-32/33	5	pCi/L			0	C	Y			70	130	5	C	Y			50	150	5

TAL Reference Data Summary

Structured Analysis Code: **A-JE-3S-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Leach/Digestion, phos ppt. -> amm. ppt.
Method: Phosphorus-32/33 by GFPC
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6544							Spike List 6568						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5481	Phosphorus-32/33	5	pCi/g			0	C	Y			70	130	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-JG-3U-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Dry, Grind, Leach/digestion,separation -> BaSO4 ppt.
Method: Sulfur-32/35 by GFPC
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6546							Spike List 6570						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5485	Sulfur-32/35	5	pCi/L			0	C	Y			70	130	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **A-JG-3U-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Leach/digestion,separation -> BaSO4 ppt.
Method: Sulfur-32/35 by GFPC
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6546							Spike List 6570						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5485	Sulfur-32/35	5	pCi/g			0	C	Y			70	130	40	C	Y			50	150	40

TAL Reference Data Summary

Structured Analysis Code: **I-JF-3T-01-06**

Target Analyte List: All Analytes

Matrix: WATER
Extraction: Dry, Grind, Leach/digestion,separation -> AgCl ppt.
Method: Chlorine-36 by GFPC
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6545							Spike List 6569						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5483	Chlorine-36	5	pCi/L			0	C	Y			70	130	40	C	Y			70	130	40

TAL Reference Data Summary

Structured Analysis Code: **A-JF-3T-01-06**

Target Analyte List: All Analytes

Matrix: SOLID
Extraction: Dry, Grind, Leach/digestion,separation -> AgCl ppt.
Method: Chlorine-36 by GFPC
QC Program: STANDARD TEST SET
Location: TestAmerica St. Louis

Analyte List		Detection Limits					Check List 6545							Spike List 6569						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
5483	Chlorine-36	5	pCi/g			0	C	Y			68	120	40	C	Y			70	130	40

Data Validation Report

This report contains the results of the review and validation of the specified data package performed by Marcia Olive, Bhate Environmental Associates, Denver, Colorado.

Introduction

This data validation report covers samples taken from Holloman Air Force Base, New Mexico, on September 3, 2008. One aqueous sample was taken from the Background Study for analyses. All analyses were performed by TestAmerica Laboratories, St. Louis. The specific sample included in this validation was:

Sample ID	Matrix	Collection Date	Lab SDG	Analyses
TDS-MW01	Water	09/03/08	D8I040185	Carbon 14 (C-01-1), Isotopic Thorium (A-01-R), Isotopic Uranium (A-01-R), Lead-210 (STL-RC-0211), Radium-226 (903.0), Radium-228 (904.0)

This data was validated against the laboratory's QA/QC limits using the guidelines and practices published in the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (USEPA, October 2004), *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (USEPA, October 1999) and *Evaluation of Radiochemical Data Usability* (DOE, April 1997).

Sample Handling and Holding times

The sample was properly preserved and transferred under chain-of-custody to the laboratory for analysis and analyzed within the required holding times.

Blanks

Thorium-230 was detected in the method blank. Since the normalized absolute difference between the sample and method blank was less than 2.58, this radionuclide was qualified estimated "J".

Laboratory control sample (LCS)

The LCS for Lead-210 was above the upper control limit. Since the sample didn't yield a positive result for Pb-210, no qualification was required.

Matrix spike/matrix spike duplicates (MS/MSD)

The MSD and relative percent difference (RPD) for Carbon 14 were outside QC limits. The results of the MS analysis were from a sample from another lot therefore, precision and accuracy were measured by the LCS recovery, which was in control, and no qualification was required.

Project specific quality assurance/quality control

No field duplicate samples or other project specific samples were submitted as part of this sampling event.

Compound quantitation and reporting limits

The lead carrier recovery was above control limits. The yield was acceptable and the LCS above control limits but since the sample <MDC; no qualification was required.

Overall assessment of data

Overall, the data is suitable for the intended data usage. All analyses were performed, and the data met the required QC criteria except where noted. The data is 100% complete.

Summary of Qualified Data

Sample ID	Parameter	*Qualifier	Reason Code
TDS-MW01	Thorium 230	0.20 J	MB

* in pCi/L

Data Validation Report

This report contains the results of the review and validation of the specified data package performed by Marcia Olive, Bhate Environmental Associates, Denver, Colorado.

Introduction

This data validation report covers samples taken from Holloman Air Force Base, New Mexico, on September 3-4, 2008. Two aqueous samples and twelve soil samples were taken from the Background Study for analyses. All analyses were performed by TestAmerica Laboratories, St. Louis. The specific samples included in this validation were:

Sample ID	Matrix	Collection Date	Lab SDG	Analyses
MW3903	Water	9/03/08	D8I050142	Carbon 14 (C-01-1), Isotopic Thorium (A-01-R), Isotopic Uranium (A-01-R), Lead-210 (STL-RC-0211), Radium-226 (903.0), Radium-228 (904.0)
TDS-MW02		9/04/08		
BWBG-SB01-0.5	Soil	9/03/08		
BWBG-SB01-20		9/03/08		
BWBG-SB01-39		9/03/08		
BWBG-SB02-0.5		9/03/08		
BWBG-SB02-10		9/03/08		
BWBG-SB02-20		9/03/08		
BWBG-SB26-0.5		09/04/08		
BWBG-SB26-11		09/04/08		
BWBG-SB26-22		09/04/08		
BWBG-SB11-0.5		09/04/08		
BWBG-SB11-14		09/04/08		
BWBG-SB11-28		09/04/08		

This data was validated against the laboratory's QA/QC limits using the guidelines and practices published in the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (USEPA, October 2004), *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (USEPA, October 1999) and *Evaluation of Radiochemical Data Usability* (DOE, April 1997).

Sample Handling and Holding times

The samples were properly preserved and transferred under chain-of-custody to the laboratory for analysis and analyzed within the required holding times.

Blanks

No radionuclides were detected in the method blanks.

Laboratory control sample (LCS)

The LCS for Lead 210 was above QC limits associated with all soil samples as well as sample MW3903. No sample detection of lead was reported therefore, no qualification was required.

Matrix spike/matrix spike duplicates (MS/MSD)

The MSD percent recovery (%R) and relative percent difference (RPD) for carbon 14 were outside QC limits associated with the aqueous samples. The sample used for this analysis was from another batch and not this sample group. Therefore precision and accuracy were measured by the LCS recovery, which was in control, no qualification was required.

The MS %R was above control limits for lead 210, associated with the soil samples. The LCS recovery was reported above QC limits as well but all sample results were <MDC; therefore, no qualification was necessary.

Project specific quality assurance/quality control

No field duplicate sample was submitted as part of this sampling event.

The laboratory duplicate met the requirements of radiochemical data usability without qualification.

Compound quantitation and reporting limits

The lead carrier recovery was outside the lower control limit for TDS-MW02 and MW3903. The samples were re-extracted with a reduced aliquot to minimize matrix interference. Sample TDS-MW02 sill reported low carrier recovery and was re-extracted again with acceptable results.
The lead carrier recovery was outside the upper control limit for BWBG-SB02-10 and BWBG-SB26-0.5. Sample results are not qualified based on yield alone. Cross reference with the LCS recovery, which was also high, determined that the non detected results for Pb210 did not required qualification.
The barium carrier recovery for Radium 226 was below control limits for samples BWBG-SB01-0.5, BWBG-SB26-11, BWBG-SB26-22 and BWBG-SB11-0.5 and above QC limits for radium 228 in sample BWBG-SB02-20. A comparison with the LCS recoveries, which were in control, determined the data was accepted without qualification.
Finally, the lead 210 LCS carrier recovery for TDS-MW02 was above QC limits. The LCS spike recoveries were within control as well as the sample carrier recovery. Therefore, no qualification was required.

Overall assessment of data

In order to minimize matrix interference the TDS-MW02 sample was reduced which resulted in the lead 210 reporting limit not being met. Also, the Radium 228 reporting limit for BWBG-SB01-0.5, BWBG-SB26-11, BWBG-SB26-22, BWBG-SB11-0.5, BWBG-SB11-14 and BWBG-SB11-28 was not met due to reduced barium carrier recovery; yet in acceptable range. The minimum detectable activity (MDA) was met and no qualification of the sample data was required.

Overall, the data is suitable for the intended data usage. All analyses were performed, and the data met the required QC criteria. The data is 100% complete.

Data Validation Report

This report contains the results of the review and validation of the specified data package performed by Marcia Olive, Bhate Environmental Associates, Denver, Colorado.

Introduction

This data validation report covers samples taken from Holloman Air Force Base, New Mexico, on September 4-5, 2008. Five aqueous samples and twelve soil samples were taken from the Background Study for analyses. All analyses were performed by TestAmerica Laboratories, St. Louis. The specific samples included in this validation were:

Sample ID	Matrix	Collection Date	Lab SDG	Analyses
TDS-MW03	Water	09/05/08	D8I060139	Carbon 14 (C-01-1), Isotopic Thorium (A-01-R), Isotopic Uranium (A-01-R), Lead-210 (STL-RC-0211), Radium-226 (903.0), Radium-228 (904.0)
TDS-MW03-A		09/05/08		
MW38-01		09/04/08		
MW37-06		09/04/08		
MW24-01		09/05/08		
BWBG-SB04-0.5	Soil	09/04/08		
BWBG-SB04-23		09/04/08		
BWBG-SB04-45		09/04/08		
BWBG-SB03-0.5		09/05/08		
BWBG-SB03-15		09/05/08		
BWBG-SB03-30		09/05/08		
BWBG-SB05-0.5		09/05/08		
BWBG-SB05-18		09/05/08		
BWBG-SB05-35		09/05/08		
BWBG-SB07-0.5		09/05/08		
BWBG-SB07-18		09/05/08		
BWBG-SB07-35		09/05/08		

This data was validated against the laboratory's QA/QC limits using the guidelines and practices published in the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (USEPA, October 2004), *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (USEPA, October 1999) and *Evaluation of Radiochemical Data Usability* (DOE, April 1997).

Sample Handling and Holding times

The samples were properly preserved and transferred under chain-of-custody to the laboratory for analysis and analyzed within the required holding times.

Blanks

Thorium 228 and 230 were detected in the method blank associated with the soil samples. Those sample concentrations that resulted in the normalized absolute difference, between the sample and method blank, to be less than 2.58 were qualified estimated "J".

Laboratory control sample (LCS)

The LCS for Lead 210 was above the upper control limit associated with all aqueous samples as well as samples BWBG-SB03-30, BWBG-SB05-0.5, BWBG-SB05-18, BWBG-SB05-35 and BWBG-SB07-0.5 indicating a potential positive bias. Since the associated samples didn't yield a positive result for lead 210, no qualification was required.

Matrix spike/matrix spike duplicates (MS/MSD)

The MSD percent recovery (%R) and relative percent difference (RPD) for Carbon 14, associated with the aqueous samples, were outside QC limits. The sample used for the spike analysis that was outside control limits was from another batch; the sample used from this batch was in control. Therefore, no qualification was required.

The lead 210 MS %R was above control limits for samples BWBG-SB03-30, BWBG-SB05-0.5, BWBG-SB05-18, BWBG-SB05-35 and BWBG-SB07-0.5. Since this matrix spike sample was from another batch and not this sample set the results were cross referenced, for precision and accuracy, with the LCS and sample data. These samples reported deficiencies in both the LCS and MS recoveries above control limits. Since there were no reported concentrations of Pb210 in the samples, using professional judgment no qualification was required.

The thorium MSD recovery was outside QC limits. The entire batch was re-prepped/re-extracted with acceptable results.

Project specific quality assurance/quality control

One field duplicate sample was submitted as part of this sampling event. The RPD, according to the QAPP, between the sample (TDS-MW03) and duplicate for Radium 228 was exceeded. This radionuclide was qualified estimated "J" in both the sample and duplicate.

The laboratory duplicate met the requirements of radiochemical data usability without qualification.

Compound quantitation and reporting limits

The lead carrier recovery was outside the lower control limit for the aqueous samples. The samples were re-extracted with a reduced aliquot and results were acceptable.

The lead carrier recovery was outside the upper control limit for BWBG-SB04-23, BWBG-SB04-45, BWBG-SB03-0.5, BWBG-SB03-15, BWBG-SB07-18 and BWBG-SB07-35. Recovery of the LCS carrier was outside limits as well. Deficient carrier recovery does not warrant sample qualification alone. The associated method blank did not yield a detection for lead and the LCSD and RPD were within control limits. Therefore, data was accepted without qualification.

Overall assessment of data

Overall, the data is suitable for the intended data usage. All analyses were performed, and the data met the required QC criteria except where noted. The data is 100% complete.

Summary of Qualified Data

Sample ID	Parameter	*Qualifier	Reason Code
TDS-MW03	Radium 228	1.37 J	RPD Dup
TDS-MW03-A	Radium 228	0.79 J	RPD Dup
BWBG-SB04-45	Thorium 228 Thorium 230	0.39 J 0.44 J	MB
BWBG-SB03-0.5	Thorium 228 Thorium 230	0.094 J 0.26 J	MB
BWBG-SB03-30	Thorium 228 Thorium 230	0.15 J 0.36 J	MB
BWBG-SB05-0.5	Thorium 228	0.29 J	MB
BWBG-SB05-35	Thorium 228 Thorium 230	0.37 J 0.33 J	MB
BWBG-SB07-0.5	Thorium 228	0.35 J	MB
BWBG-SB07-18	Thorium 228 Thorium 230	0.44 J 0.48 J	MB

* in pCi/L

Data Validation Report

This report contains the results of the review and validation of the specified data package performed by Marcia Olive, Bhate Environmental Associates, Denver, Colorado.

Introduction

This data validation report covers samples taken from Holloman Air Force Base, New Mexico, on September 5-8, 2008. Seven aqueous samples and twenty seven soil samples were taken from the Background Study for analyses. All analyses were performed by TestAmerica Laboratories, St. Louis. The specific samples included in this validation were:

Sample ID	Matrix	Collection Date	Lab SDG	Analyses
MW30&33-02	Water	9/07/08	D8I090195	Carbon 14 (C-01-1), Isotopic Thorium (A-01-R), Isotopic Uranium (A-01-R), Lead-210 (STL-RC-0211), Radium-226 (903.0), Radium-228 (904.0)
MW30&33-01		9/07/08		
SS61-MW01		9/07/08		
MW-29-01		9/07/08		
MW04-01		9/08/08		
S1-MW5-A		9/08/08		
S1-MW5		9/08/08		
BWBG-SB12-0.5	Soil	9/05/08		
BWBG-SB12-10		9/05/08		
BWBG-SB12-20		9/05/08		
BWBG-SB18-0.5		9/06/08		
BWBG-SB18-18		9/06/08		
BWBG-SB18-35		9/06/08		
BWBG-SB20-0.5		9/06/08		
BWBG-SB20-20		9/06/08		
BWBG-SB20-40		9/06/08		
BWBG-SB20-40-A		9/06/08		
BWBG-SB19-0.5		9/06/08		
BWBG-SB19-0.5-A		9/06/08		
BWBG-SB19-25		9/07/08		

BWBG-SB19-49		9/07/08		
BWBG-SB25-0.5		9/06/08		
BWBG-SB25-15		9/06/08		
BWBG-SB25-15A		9/06/08		
BWBG-SB25-35		9/06/08		
BWBG-SB08-0.5		9/07/08		
BWBG-SB08-20		9/07/08		
BWBG-SB08-20-A		9/07/08		
BWBG-SB08-45		9/07/08		
BWBG-SB21-0.5		9/06/08		
BWBG-SB21-20		9/06/08		
BWBG-SB21-20-A		9/06/08		
BWBG-SB21-40		9/06/08		
BWBG-SB21-40-A		9/06/08		

This data was validated against the laboratory's QA/QC limits using the guidelines and practices published in the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (USEPA, October 2004), *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (USEPA, October 1999) and *Evaluation of Radiochemical Data Usability* (DOE, April 1997).

Sample Handling and Holding times

The samples were properly preserved and transferred under chain-of-custody to the laboratory for analysis and analyzed within the required holding times.

Blanks

Thorium 230, uranium 234 and uranium 238 were detected in the method blank, associated with samples BWBG-SB12-0.5, BWBG-SB12-10, BWBG-SB12-20, BWBG-SB18-0.5, BWBG-SB18-18, BWBG-SB18-35, BWBG-SB20-0.5, BWBG-SB20-20, BWBG-SB20-40, BWBG-SB20-40-A, BWBG-SB19-0.5, BWBG-SB19-0.5-A, BWBG-SB19-25 and BWBG-SB19-49. Also, Thorium 228 and 230 were detected in the method blank associated with BWBG-SB25-0.5, BWBG-SB25-15, BWBG-SB25-15A, BWBG-SB25-35, BWBG-SB08-0.5, BWBG-SB08-20, BWBG-SB08-20-A, BWBG-SB08-45, BWBG-SB21-0.5, BWBG-SB21-20, BWBG-SB21-20-A, BWBG-SB21-40 and BWBG-SB21-40-A. Those sample concentrations that resulted in the normalized absolute difference, between the sample and method blank, to be less than 2.58 were qualified estimated "J".

Laboratory control sample (LCS)

The LCS recovery for Lead 210, associate with MW30&33-02, SS61-MW01, MW04-01 and BWBG-SB20-40, was above QC limits. Since the sample concentrations did not yield positive results; no qualification of the data was required.

Matrix spike/matrix spike duplicates (MS/MSD)

The MS percent recovery (%R) for lead 210 was above QC limits associated with sample BWBG-SB20-40. The QC deficiencies of high LCS recovery and high MS recovery did not require the non detected result of the aforementioned sample to be qualified. The MS and/or MSD %Rs for lead 210 associated with all soil samples except BWBG-SB20-40 were below QC limits. See Compound quantitation and reporting limits section for qualification.

The carbon 14 MS/MSD %Rs and/or RPDs for the all water and multiple soil samples were outside control limits. The LCS recovery was in control therefore matrix effect was reserved for the sample used in the spike analysis and not the associated samples. No qualification was required.

It was incorrectly reported that Radium 226 &228 yielded RPDs outside control limits for batches 8254090 and 8254091.

Project specific quality assurance/quality control

Seven field duplicate samples were submitted as part of this sampling event. The RPD, according to the QAPP, between the samples (S1-MW5, BWBG-SB20-40) and the duplicates were exceeded for Thorium 230. The RPD between the sample (BWBG-SB19-0.5) and the duplicate was exceeded for Radium 226, Uranium 234, Thorium 230 and 232. These radionuclides were qualified estimated “J” in both samples.

Compound quantitation and reporting limits

Due to matrix interference, the lead carrier recoveries were above control limits for samples MW30&33-02, MW30&33-01, SS61-MW01, S1-MW5-A, BWBG-SB12-10, BWBG-SB18-18, BWBG-SB18-35, BWBG-SB20-0.5, BWBG-SB20-20, BWBG-SB20-40-A, BWBG-SB19-0.5, BWBG-SB19-0.5-A, BWBG-SB19-25, BWBG-SB19-49, BWBG-SB25-15, BWBG-SB25-15A, BWBG-SB08-0.5, BWBG-SB08-20-A, BWBG-SB21-20-A, BWBG-SB21-40 and BWBG-SB21-40-A. The aforementioned soil samples yielded QC deficiencies of low matrix spike recoveries as well as high carrier recoveries and lead was qualified either estimated non-detected, ”UJ” or estimated, “J”.
The lead 210 LCS carrier recovery associated with samples BWBG-SB12-0.5, BWBG-SB12-10, BWBG-SB12-20, BWBG-SB18-0.5, BWBG-SB18-18, BWBG-SB18-35, BWBG-SB20-0.5, BWBG-SB20-20, BWBG-SB20-40-A, BWBG-SB19-0.5, BWBG-SB19-0.5-A, BWBG-SB19-25, BWBG-SB19-49 was incorrectly reported in the case narrative as being above the QC limits. It was reported at 105% which was below the upper limit of 110%.
Additionally the LCS carrier recovery for lead 210 was above QC limits associated with samples MW30&33-01, MW-29-01, S1-MW5-A and S1-MW5. These samples required no qualification from high LCS and carrier recoveries.

Finally, the barium carrier recoveries for Radium 226 and 228 was outside control limits for samples BWBG-SB12-10, BWBG-SB12-20, BWBG-SB18-18, BWBG-SB20-40-A, BWBG-SB25-0.5, BWBG-SB21-20, BWBG-SB21-40 and BWBG-SB25-15. Samples are not qualified based on carrier recovery alone. Since the radionuclides in the LCS were acceptable data was accepted without qualification.

Overall assessment of data

In order to minimize matrix interference the aqueous sample sizes, with the exception of MW30&33-02, SS61-MW01 and MW04-01, were reduced which resulted in the lead 210 reporting limit not being met. The minimum detectable activity (MDA) was met and no qualification of the sample data was required.
The aqueous samples yielded tracer recoveries for thorium below the lower QC limit. The batch was re-prepped/re-extracted with satisfactory results.
Sample BWBG-SB20-20 was double traced with barium carrier and would not precipitate for Radium 226. The recovery for Radium 226 (57%) was within control limits therefore no qualification was necessary.

Overall, the data is suitable for the intended data usage. All analyses were performed, and the data met the required QC criteria with the following exceptions. The data is 100% complete.

Summary of Qualified Data

Sample ID	Parameter	*Qualifier	Reason Code
S1-MW5	Thorium 230	0.15 J	RPD Dup
S1-MW5-A	Thorium 230	0.103 J	RPD Dup
BWBG-SB12-0.5	Uranium 234 Uranium 238 Thorium 230	0.24 J 0.23 J 0.48 J	MB
BWBG-SB12-10	Uranium 234 Uranium 238 Thorium 230 Lead 210	0.39 J 0.24 J 0.38 J 0.07 UJ	MB/MS/Carrier
BWBG-SB12-20	Uranium 234 Uranium 238 Thorium 230	0.134 J 0.086 J 0.24 J	MB
BWBG-SB18-0.5	Uranium 234 Uranium 238	0.46 J 0.31 J	MB
BWBG-SB18-18	Uranium 234 Thorium 230 Lead 210	0.128 J 0.30 J 0.16 UJ	MB/MS/Carrier
BWBG-SB18-35	Uranium 234 Uranium 238 Thorium 230 Lead 210	0.098 J 0.140 J 0.30 J -0.07 UJ	MB/MS/Carrier
BWBG-SB20-0.5	Uranium 234 Uranium 238 Thorium 230 Lead 210	0.28 J 0.192 J 0.40 J 0.06 UJ	MB/MS/Carrier
BWBG-SB20-20	Uranium 234 Uranium 238 Thorium 230 Lead 210	0.22 J 0.080 J 0.24 J 0.35 UJ	MB/MS/Carrier
BWBG-SB20-40	Uranium 234 Uranium 238 Thorium 230	0.55 J 0.44 J 0.79 J	MB/RPD Dup

Summary of Qualified Data

Sample ID	Parameter	*Qualifier	Reason Code
BWBG-SB20-40-A	Uranium 234	0.35 J	MB/RPD Dup/MS/Carrier
	Uranium 238	0.33 J	
	Thorium 230	0.39 J	
	Lead 210	0.31 UJ	
BWBG-SB19-0.5	Radium 226	0.38 J	MB/RPD Dup/MS/Carrier
	Uranium 234	0.27 J	
	Uranium 238	0.32 J	
	Thorium 230	0.62 J	
	Thorium 232	0.088 J	
	Lead 210	0.07 UJ	
BWBG-SB19-0.5-A	Radium 226	0.70 J	MB/RPD Dup/MS/Carrier
	Uranium 234	0.45 J	
	Uranium 238	0.50 J	
	Thorium 230	1.37 J	
	Thorium 232	0.45 J	
	Lead 210	1.15 J	
BWBG-SB19-25	Uranium 234	0.51 J	MB/MS/Carrier
	Uranium 238	0.43 J	
	Thorium 230	0.55 J	
	Lead 210	0.04 UJ	
BWBG-SB19-49	Uranium 234	0.180 J	MB/MS/Carrier
	Uranium 238	0.225 J	
	Thorium 230	0.51 J	
	Lead 210	-0.04 UJ	
BWBG-SB25-0.5	Thorium 228	0.34 J	MB
	Thorium 230	0.39 J	
BWBG-SB25-15	Thorium 230	0.22 J	MB/MS/Carrier
	Lead 210	0.05 UJ	
BWBG-SB25-15A	Thorium 228	0.12 J	MB/MS/Carrier
	Thorium 230	0.16 J	
	Lead 210	-0.18 UJ	
BWBG-SB25-35	Thorium 228	0.35 J	MB
BWBG-SB08-0.5	Thorium 228	0.37 J	MB/MS/Carrier
	Lead 210	0.85 UJ	
BWBG-SB08-20-A	Lead 210	0.19 UJ	MS/Carrier
BWBG-SB08-45	Thorium 228	0.38 J	MB
	Thorium 230	0.32 J	
BWBG-SB21-0.5	Thorium 228	0.40 J	MB
BWBG-SB21-20	Thorium 228	0.22 J	MB
	Thorium 230	0.27 J	
BWBG-SB21-20-A	Thorium 228	0.22 J	MB/MS/Carrier
	Thorium 230	0.34 J	
	Lead 210	0.49 UJ	
BWBG-SB21-40	Thorium 228	0.29 J	MB/MS/Carrier
	Thorium 230	0.35 J	
	Lead 210	-0.23 UJ	
BWBG-SB21-40-A	Thorium 228	0.23 J	MB/MS/Carrier
	Thorium 230	0.38 J	
	Lead 210	-0.08 UJ	

*In pCi/L

Data Validation Report

This report contains the results of the review and validation of the specified data package performed by Marcia Olive, Bhate Environmental Associates, Denver, Colorado.

Introduction

This data validation report covers samples taken from Holloman Air Force Base, New Mexico, on September 7-9, 2008. Four aqueous samples and thirteen soil samples were taken from the Background Study for analyses. All analyses were performed by TestAmerica Laboratories, St. Louis. The specific samples included in this validation were:

Sample ID	Matrix	Collection Date	Lab SDG	Analyses
S1-MW2	Water	9/09/08	D8I100172	Carbon 14 (C-01-1), Isotopic Thorium (A-01-R), Isotopic Uranium (A-01-R), Lead-210 (STL-RC-0211), Radium-226 (903.0), Radium-228 (904.0)
S1-MW1-A		9/09/08		
S1-MW1		9/09/08		
MW-BG-04		09/08/08		
BWBG-SB06-0.5	Soil	9/07/08		
BWBG-SB06-25		9/07/08		
BWBG-SB06-50		9/07/08		
BWBG-SB09-0.5		9/08/08		
BWBG-SB09-30		9/08/08		
BWBG-SB09-30-A		9/08/08		
BWBG-SB09-59		9/08/08		
BWBG-SB15-0.5		9/08/08		
BWBG-SB15-25		9/08/08		
BWBG-SB15-50		9/08/08		
BWBG-SB17-0.5		9/08/08		
BWBG-SB17-25		9/08/08		
BWBG-SB17-50		9/08/08		

This data was validated against the laboratory's QA/QC limits using the guidelines and practices published in the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (USEPA, October 2004), *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (USEPA, October 1999) and *Evaluation of Radiochemical Data Usability* (DOE, April 1997).

Sample Handling and Holding times

The samples were properly preserved and transferred under chain-of-custody to the laboratory for analysis and analyzed within the required holding times.

Blanks

Uranium 234 was detected in the method blank, associated with the soil samples. Those samples where the normalized absolute difference, between the sample and method blank, was less than 2.58 were qualified estimated "J".

Laboratory control sample (LCS)

The LCS percent recoveries were within control limits for all analysis.

Matrix spike/matrix spike duplicates (MS/MSD)

The MS/MSD percent recoveries (%Rs) for lead 210 were outside QC limits associated withBWBG-SB06-0.5, BWBG-SB06-25, BWBG-SB06-50, BWBG-SB09-0.5, BWBG-SB09-30 and BWBG-SB09-30-A. The MS %R was outside control limits for carbon 14, associated with the above mentioned soil samples with the exception of the duplicate. The sample used for the analysis of the matrix spike was from another lot. Therefore precision and accuracy were determined by the LCS recoveries, which were in control, and no qualification was required.

Project specific quality assurance/quality control

Two field duplicate samples were submitted as part of this sampling event. The RPD, according to the QAPP, between the sample (S1-MW1) and duplicate for Radium 226 was exceeded. The RPD between the sample (BWBG-SB09-30) and the duplicate was exceeded for Thorium 228. These radionuclides were qualified estimated "J" in both samples.

The laboratory duplicate met the requirements of radiochemical data usability without qualification.

Compound quantitation and reporting limits

The lead carrier recovery was outside the upper control limit for samples BWBG-SB06-50, BWBG-SB09-0.5, BWBG-SB17-25, S1-MW2, S1-MW1-A and S1-MW1. Additionally, the lead 210 LCS carrier recovery, associated with the aqueous samples, was outside limits. Sample results should not be qualified based on yield alone. The recovery of lead 210 in the quality indicator sample (LCS) was acceptable therefore data was accepted without qualification.

The barium carrier recovery for Radium 228 was below control limits for samples BWBG-SB09-0.5 and BWBG-SB15-0.5. This may cause a high bias in the result. Since radium was not detected in either sample; no qualification was necessary.
The thorium tracer recoveries were below QC limits associated with the aqueous samples. The batch was re-extracted with acceptable results.
Uranium tracer recovery in sample MW-BG-04 was below QC limits. The batch was re-extracted with acceptable results.

Overall assessment of data

In order to minimize matrix interference the aqueous sample sizes were reduced which resulted in the lead 210 reporting limit not being met. Low barium recovery caused the Radium 228 reporting limit, for samples BWBG-SB15-0.5 and BWBG-SB17-0.5, not to be met. The minimum detectable activity (MDA) was met and no qualification of the sample data was required.

Overall, the data is suitable for the intended data usage. All analyses were performed, and the data met the required QC criteria with the following exceptions. The data is 100% complete.

Summary of Qualified Data

Sample ID	Parameter	*Qualifier	Reason Code
S1-MW1	Radium 226	0.65 J	RPD Dup
S1-MW1-A	Radium 226	0.45 J	RPD Dup
BWBG-SB06-50	Uranium 234	0.35 J	MB
BWBG-SB09-0.5	Uranium 234	0.22 J	MB
BWBG-SB09-30	Thorium 228	0.55 J	RPD Dup
BWBG-SB09-30-A	Thorium 228	1.06 J	RPD Dup
BWBG-SB09-59	Uranium 234	0.28 J	MB
BWBG-SB15-0.5	Uranium 234	0.19 J	MB
BWBG-SB15-25	Uranium 234	0.33 J	MB
BWBG-SB15-50	Uranium 234	0.28 J	MB
BWBG-SB17-50	Uranium 234	0.34 J	MB

*In pCi/L

Data Validation Report

This report contains the results of the review and validation of the specified data package performed by Marcia Olive, Bhate Environmental Associates, Denver, Colorado.

Introduction

This data validation report covers samples taken from Holloman Air Force Base, New Mexico, on September 9-10, 2008. Four aqueous samples and fifteen soil samples were taken from the Background Study for analyses. All analyses were performed by TestAmerica Laboratories, St. Louis. The specific samples included in this validation were:

Sample ID	Matrix	Collection Date	Lab SDG	Analyses
MW-23-01	Water	9/09/08	D8I110184	Carbon 14 (C-01-1), Isotopic Thorium (A-01-R), Isotopic Uranium (A-01-R), Lead-210 (STL-RC-0211), Radium-226 (903.0), Radium-228 (904.0)
MW-21-04		9/09/08		
S10-MW4		9/10/08		
MW-01		09/10/08		
BWBG-SB23-0.5	Soil	9/09/08		
BWBG-SB23-30		9/09/08		
BWBG-SB23-64		9/09/08		
BWBG-SB16-0.5		9/09/08		
BWBG-SB16-20		9/09/08		
BWBG-SB16-39		9/09/08		
BWBG-SB24-0.5		9/09/08		
BWBG-SB24-20		9/10/08		
BWBG-SB24-40		9/10/08		
BWBG-SB27-0.5		9/10/08		
BWBG-SB27-13		9/10/08		
BWBG-SB27-25		9/10/08		
BWBG-SB30-0.5		9/10/08		
BWBG-SB30-14		9/10/08		
BWBG-SB30-28		9/10/08		

This data was validated against the laboratory's QA/QC limits using the guidelines and practices published in the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (USEPA, October 2004), *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (USEPA, October 1999) and *Evaluation of Radiochemical Data Usability* (DOE, April 1997).

Sample Handling and Holding times

The samples were properly preserved and transferred under chain-of-custody to the laboratory for analysis and analyzed within the required holding times.

Blanks

Thorium 230 was detected in the method blank, associated with the soil samples. Those sample concentrations that resulted in the normalized absolute difference, between the sample and method blank, to be less than 2.58 were qualified estimated "J".

Laboratory control sample (LCS)

The LCS percent recoveries were within control limits for all analysis.

Matrix spike/matrix spike duplicates (MS/MSD)

The MS percent recovery (%R) and RPD for lead 210 were outside QC limits associated with samples BWBG-SB30-14 and BWBW-SB30-28. The sample used for the matrix spike analysis was from another batch and not indicative of this sample set. Therefore, accuracy and precision were measured by the LCS recovery, which was in control. Therefore sample results were not adversely affected and no qualification was necessary.

Project specific quality assurance/quality control

No field duplicate sample was submitted as part of this sampling event.

The laboratory duplicate met the requirements of radiochemical data usability without qualification.

Compound quantitation and reporting limits

The lead carrier recoveries were outside the upper control limit for BWBG-SB24-20, BWBG-SB27-13, BWBG-SB30-0.5, BWBG-SB27-25 and BWBG-SB30-28. Also, the lead 210 carrier recoveries for the LCSD associated with samples BWBG-SB30-14 and BWBG-SB30-28 and the LCS associated with the aqueous samples were above limits. The quality indicator (LCS) recoveries were within control limits. Therefore data was accepted without qualification.

The thorium tracer recoveries were below QC limits associated with the aqueous samples and sample BWBG-SB16-20. And the uranium tracer recovery associated with MW-21-04 was below QC limits. The batch was re-prepped/re-extracted with acceptable results.

Overall assessment of data

In order to minimize matrix interference the aqueous sample sizes were reduced which resulted in the lead 210 reporting limit not being met. The minimum detectable activity (MDA) was met and no qualification of the sample data was required.

Overall, the data is suitable for the intended data usage. All analyses were performed, and the data met the required QC criteria with the following exceptions. The data is 100% complete.

Summary of Qualified Data

Sample ID	Parameter	*Qualifier	Reason Code
BWBG-SB23-0.5	Thorium 230	0.17 J	MB
BWBG-SB23-64	Thorium 230	0.23 J	MB
BWBG-SB16-20	Thorium 230	0.66 J	MB
BWBG-SB24-0.5	Thorium 230	0.77 J	MB
BWBG-SB24-20	Thorium 230	0.50 J	MB
BWBG-SB24-40	Thorium 230	0.42 J	MB
BWBG-SB27-0.5	Thorium 230	0.63 J	MB
BWBG-SB27-13	Thorium 230	0.38 J	MB
BWBG-SB27-25	Thorium 230	0.32 J	MB
BWBG-SB30-28	Thorium 230	0.26 J	MB

*In pCi/L

Data Validation Report

This report contains the results of the review and validation of the specified data package performed by Marcia Olive, Bhate Environmental Associates, Denver, Colorado.

Introduction

This data validation report covers samples taken from Holloman Air Force Base, New Mexico, on September 10, 2008. One aqueous sample and six soil samples were taken from the Background Study for analyses. All analyses were performed by TestAmerica Laboratories, St. Louis. The specific samples included in this validation were:

Sample ID	Matrix	Collection Date	Lab SDG	Analyses
SS61-MW11	Water	09/10/08	D8I120256	Carbon 14 (C-01-1), Isotopic Thorium (A-01-R), Isotopic Uranium (A-01-R), Lead-210 (STL-RC-0211), Radium-226 (903.0), Radium-228 (904.0)
BWBG-SB29-0.5	Soil			
BWBG-SB29-15				
BWBG-SB29-30				
BWBG-SB31-0.5				
BWBG-SB31-15				
BWBG-SB31-30				

This data was validated against the laboratory's QA/QC limits using the guidelines and practices published in the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (USEPA, October 2004), *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (USEPA, October 1999) and *Evaluation of Radiochemical Data Usability* (DOE, April 1997).

Sample Handling and Holding times

The samples were properly preserved and transferred under chain-of-custody to the laboratory for analysis and analyzed within the required holding times.

Blanks

Thorium-230 was detected in the method blank associated with the soil samples. All sample concentrations resulted in the normalized absolute difference between the sample and method blank to be less than 2.58 and therefore were qualified estimated "J".

Laboratory control sample (LCS)

All LCS percent recoveries (%Rs) were within QC limits.

Matrix spike/matrix spike duplicates (MS/MSD)

The MS and relative percent difference (RPD) for lead 210 were outside QC limits associated with samples BWBG-SB29-0.5, BWBG-SB29-15, BWBG-SB29-30 and BWBG-SB31-0.5. Since the sample used for the MS analysis was from another lot, precision and accuracy were measured by the LCS recoveries, which were in control. No qualification was required; with the exception of BWBG-SB29-15 (See below).

Project specific quality assurance/quality control

No field duplicate sample was submitted as part of this sampling event.

The laboratory duplicate met the requirements of radiochemical data usability without qualification.

Compound quantitation and reporting limits

The barium carrier recoveries for both radium 226 and 228 were above upper control limits for sample BWBG-SB31-15. Sample results were not qualified based on yield alone. The recoveries of the target radionuclides in the LCS were within QC limits and the data was accepted without qualification.
The lead carrier recovery was above upper QC limits for sample SS61-MW11. Recovery of the LCS carrier recovery was also above QC limits. Since carriers are not usually recovered at levels greater than 100% contamination may be present. Since the method blank did not yield a detection for lead and the LCSD was within control limits. Using professional judgment, the non detected result in the sample required no qualification.
The lead carrier recovery was below QC limits for sample BWBG-SB29-15 and BWBG-SB31-30. The low recovery may indicate a potential high bias to the data although no detections occurred in either sample. Comparison with the LCS/LCSD recovery resulted in the LCSD carrier recovery above QC limits (associated with sample BWBG-SB29-15). Considering the MS was outside control limits as well, Pb210 was qualified estimated non detected, “UJ” in sample BWBG-SB29-15.

The tracer recovery for thorium, in several samples, was below QC limits. The entire batch was re-extracted and all recoveries were within QC limits. Therefore, no qualification was required.

Overall assessment of data

In order to minimize matrix interference the aqueous sample was reduced which resulted in the lead 210 reporting limit not being met. The minimum detectable activity (MDA) was met and no qualification of the sample data was required.

Overall, the data is suitable for the intended data usage. All analyses were performed, and the data met the required QC criteria except where noted. The data is 100% complete.

Summary of Qualified Data

Sample ID	Parameter	*Qualifier	Reason Code
BWBG-SB29-0.5	Thorium 230	0.52 J	MB
BWBG-SB29-15	Thorium 230 Lead 210	0.59 J UJ	MB/MS/carrier recov.
BWBG-SB29-30	Thorium 230	0.38 J	MB
BWBG-SB31-0.5	Thorium 230	0.26 J	MB
BWBG-SB31-15	Thorium 230	0.27 J	MB
BWBG-SB31-30	Thorium 230	0.82 J	MB

* in pCi/L

Data Validation Report

This report contains the results of the review and validation of the specified data package performed by Marcia Olive, Bhate Environmental Associates, Denver, Colorado.

Introduction

This data validation report covers samples taken from Holloman Air Force Base, New Mexico, on September 17-18, 2008. Three aqueous samples and eighteen soil samples were taken from the Background Study for analyses. All analyses were performed by TestAmerica Laboratories, St. Louis. The specific samples included in this validation were:

Sample ID	Matrix	Collection Date	Lab SDG	Analyses
TDS-MW04	Water	9/17/08	D8I190223	Carbon 14 (C-01-1), Isotopic Thorium (A-01-R), Isotopic Uranium (A-01-R), Lead-210 (STL-RC-0211), Radium-226 (903.0), Radium-228 (904.0)
MW24-03		9/18/08		
MW24-05		9/18/08		
BWBG-SB42-0.5	Soil	9/17/08		
BWBG-SB42-10				
BWBG-SB42-20				
BWBG-SB39-0.5				
BWBG-SB39-12				
BWBG-SB39-24				
BWBG-SB40-0.5				
BWBG-SB40-0.5-A				
BWBG-SB40-12				
BWBG-SB40-23				
BWBG-SB41-0.5				
BWBG-SB41-12				
BWBG-SB41-25				
BWBG-SB41-25-A				
BWBG-SB28-0.5				
BWBG-SB28-15				
BWBG-SB28-15-A				
BWBG-SB28-30				

This data was validated against the laboratory's QA/QC limits using the guidelines and practices published in the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (USEPA, October 2004), *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (USEPA, October 1999) and *Evaluation of Radiochemical Data Usability* (DOE, April 1997).

Sample Handling and Holding times

The samples were properly preserved and transferred under chain-of-custody to the laboratory for analysis and analyzed within the required holding times.

Blanks

Thorium 230 was detected in the method blank, associated with the soil samples. Radium 226 was detected in the method blank associated with samples BWBG-SB42-0.5, BWBG-SB42-10, BWBG-SB42-20, BWBG-SB39-0.5, BWBG-SB39-12, BWBG-SB39-

24, BWBG-SB40-0.5, BWBG-SB40-0.5-A, BWBG-SB40-12, BWBG-SB40-23, BWBG-SB41-0.5 and BWBG-SB41-12. Those sample concentrations that resulted in the normalized absolute difference, between the sample and method blank, to be less than 2.58 were qualified estimated "J".

Laboratory control sample (LCS)

The LCS percent recoveries were within control limits for all analysis.

Matrix spike/matrix spike duplicates (MS/MSD)

The MS percent recovery (%R) for carbon 14 was below QC limits associated with the soil samples with the exception of BWBG-SB41-25 and BWBG-SB28-30. The MSD and RPD were in control. Using professional judgment, since the LCS recovery was within limits, no qualification was necessary.

The lead 210 MSD %R and RPD were above QC limits associated with BWBG-SB41-12 and BWBG-SB41-25. These two samples did yield carrier recoveries above upper control limits. But the high bias for the MS and carrier did not warrant qualification of the sample data.

Project specific quality assurance/quality control

Three field duplicate samples were submitted as part of this sampling event. The RPD, according to the QAPP, between the sample (BWBG-SB40-0.5) and the duplicate was exceeded for Uranium 234/238 and Thorium 228/230/232. These radionuclides were qualified estimated "J" in both samples.

The laboratory duplicate met the requirements of radiochemical data usability without qualification.

Compound quantitation and reporting limits

The lead carrier recovery was outside the upper control limit for samples TDS-MW04, MW24-05, BWBG-SB42-0.5, BWBG-SB39-24, BWBG-SB40-0.5, BWBG-SB40-12, BWBG-SB40-23, BWBG-SB41-12, BWBG-SB41-25, BWBG-SB41-25-A, BWBG-SB28-0.5 and BWBG-SB28-30. Also, the lead 210 LCS carrier recovery for the aqueous samples was above limits. The lead 210 LCSD carrier recovery for BWBG-SB41-12 and BWBG-SB41-25 was above limits, as well. Carrier recovery alone does not necessitate qualification. Only samples BWBG-SB41-12 and BWBG-SB41-25 yielded multiple QC deficiencies but the high bias in both MS and carrier recovery did not merit qualification.

Overall assessment of data

In order to minimize matrix interference, the aqueous samples were analyzed with a reduced aliquot which resulted in the lead 210 reporting limit not being met. The minimum detectable activity (MDA) was met and no qualification of the sample data was required.

Overall, the data is suitable for the intended data usage. All analyses were performed, and the data met the required QC criteria with the following exceptions. The data is 100% complete.

Summary of Qualified Data

Sample ID	Parameter	*Qualifier	Reason Code
BWBG-SB42-10	Radium 226 Thorium 230	0.40 J 0.48 J	MB
BWBG-SB42-20	Radium 226	0.39 J	MB
BWBG-SB39-0.5	Radium 226	0.91 J	MB
BWBG-SB39-12	Radium 226 Thorium 230	0.33 J 0.45 J	MB
BWBG-SB39-24	Radium 226 Thorium 230	0.26 J 0.20 J	MB
BWBG-SB40-0.5	Radium 226 Uranium 234 Uranium 238 Thorium 228 Thorium 230 Thorium 232	0.37 J 0.28 J 0.138 J 0.16 J 0.35 J 0.124 J	MB/RPD Dup
BWBG-SB40-0.4-A	Radium 226 Uranium 234 Uranium 238 Thorium 228 Thorium 230 Thorium 232	0.52 J 0.52 J 0.34 J 0.37 J 0.60 J 0.44 J	MB/RPD Dup
BWBG-SB40-12	Radium 226 Thorium 230	0.29 J 0.46 J	MB
BWBG-SB40-23	Radium 226 Thorium 230	0.24 J 0.35 J	MB
BWBG-SB41-0.5	Radium 226 Thorium 230	0.16 J 0.39 J	MB
BWBG-SB41-12	Thorium 230	0.23 J	MB
BWBG-SB28-0.5	Thorium 230	0.42 J	MB
BWBG-SB28-15	Thorium 230	0.38 J	MB
BWBG-SB28-15-A	Thorium 230	0.60 J	MB

*In pCi/L

Data Validation Report

This report contains the results of the review and validation of the specified data package performed by Marcia Olive, Bhate Environmental Associates, Denver, Colorado.

Introduction

This data validation report covers samples taken from Holloman Air Force Base, New Mexico, on September 18-19, 2008. Four aqueous samples and twenty three soil samples were taken from the Background Study for analyses. All analyses were performed by TestAmerica Laboratories, St. Louis. The specific samples included:

Sample ID	Matrix	Collection Date	Lab SDG	Analyses
MW-13	Water	9/18/08	D8I200171	Carbon 14 (C-01-1), Isotopic Thorium (A-01-R), Isotopic Uranium (A-01-R), Lead-210 (STL-RC-0211), Radium-226 (903.0), Radium-228 (904.0)
MW-19-03		9/19/08		
MW-06		9/18/08		
MW22-03		9/18/08		
BWBG-SB38-0.5	Soil	9/18/08		
BWBG-SB38-7		9/18/08		
BWBG-SB38-15		9/18/08		
BWBG-SB36-0.5		9/19/08		
BWBG-SB36-0.5-A		9/19/08		
BWBG-SB36-7		9/19/08		
BWBG-SB36-15		9/19/08		
BWBG-SB37-0.5		9/19/08		
BWBG-SB37-8		9/19/08		
BWBG-SB37-16		9/19/08		
BWBG-SB33-0.5		9/18/08		
BWBG-SB33-0.5-A		9/18/08		
BWBG-SB33-12		9/18/08		
BWBG-SB33-25		9/18/08		
BWBG-SB34-0.5		9/18/08		
BWBG-SB34-15		9/18/08		
BWBG-SB34-28		9/18/08		
BWBG-SB32-0.5		9/18/08		

BWBG-SB32-17		9/18/08		
BWBG-SB32-35		9/18/08		
BWBG-SB35-0.5		9/18/08		
BWBG-SB35-20		9/18/08		
BWBG-SB35-40		9/18/08		

This data was validated against the laboratory's QA/QC limits using the guidelines and practices published in the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (USEPA, October 2004), *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (USEPA, October 1999) and *Evaluation of Radiochemical Data Usability* (DOE, April 1997).

Sample Handling and Holding times

The samples were properly preserved and transferred under chain-of-custody to the laboratory for analysis and analyzed within the required holding times.

Blanks

Thorium 230 was detected in the method blank, associated with samples BWBG-SB33-12, BWBG-SB33-25, BWBG-SB34-0.5, BWBG-SB34-15, BWBG-SB34-28, BWBG-SB32-0.5, BWBG-SB32-17, BWBG-SB32-35, BWBG-SB35-0.5, BWBG-SB35-20 and BWBG-SB35-40. Also, Thorium 228 was detected in the method blank associated with the above mentioned samples with the exception of BWBG-SB32-25. Finally uranium 234 was detected in the method blank associated with MW-19-03. Those sample concentrations that resulted in the normalized absolute difference, between the sample and method blank, to be less than 2.58 were qualified estimated "J".

Laboratory control sample (LCS)

The LCS percent recoveries were within control limits for all analysis.

Matrix spike/matrix spike duplicates (MS/MSD)

The MS percent recovery for carbon 14 was below QC limits associated with BWBG-SB32-17, BWBG-SB32-35, BWBG-SB35-0.5, BWBG-SB35-20 and BWBG-SB35-40. The Lead 210 MS %R and RPD were outside QC limits associated with BWBG-SB38-0.5 and BWBG-SB38-7, BWBG-SB38-15, BWBG-SB36-0.5, BWBG-SB36-0.5-A, BWBG-SB36-7, BWBG-SB36-15, BWBG-SB37-0.5, BWBG-SB37-8 and BWBG-SB37-16. The sample(s) used for this analysis were from another lot and not this sample batch; therefore precision and accuracy were measured by the LCS recovery, which was in control. Therefore, data was not adversely affected and required no qualification.

Project specific quality assurance/quality control

Two field duplicate samples were submitted as part of this sampling event. The RPD, according to the QAPP, between the sample (BWBG-SB36-0.5) and the duplicate was

exceeded for Uranium 234&238 and Radium 228. These radionuclides were qualified estimated “J” in both samples.

The laboratory duplicate(s) met the requirements of radiochemical data usability without qualification.

Compound quantitation and reporting limits

The lead yield for samples MW-13 and BWBG-SB38-0.5 were below QC limits which may indicate a high bias to the sample results. Since there was no detection for Pb210; no qualification was required.

The lead yield for all samples with the exception of MW-13 and BWBG-SB38-0.5 were above QC limits. Additionally, the lead 210 LCS or LCSD carrier recovery associated with most samples was above limits. Carriers are not usually expected to be recovered at levels greater than 100% but sample results are not qualified based on yield alone. Considering the LCS spike recoveries were within control limits the data was acceptable without qualification.

It was incorrectly reported that the LCS tracer recovery for Thorium 229 was above QC limits for samples BWBG-SB33-12, BWBG-SB33-25, BWBG-SB34-0.5, BWBG-SB34-15, BWBG-SB34-28, BWBG-B32-0.5, BWBG-SB32-17, BWBG-SB32-35, BWBG-SB35-0.5, BWBG-SB35-20 and BWBG-SB35-40. Thorium 229 is a tracer used in biological samples and the tracer recovery for the reported thorium isotopes were in control.

Finally, the barium carrier recovery was outside lower control limits for samples BWBG-SB38-0.5, BWBG-SB36-0.5 and BWBG-SB36-0.5-A. The yield was low but recoveries of target radionuclides in the LCS were acceptable. Therefore, data was accepted without qualification.

Overall assessment of data

In order to minimize matrix interference the aqueous sample size was reduced which resulted in the lead 210 reporting limit not being met. The Radium 228 reporting limit was not met for BWBG-SB38-0.5 and BWBG-SB36-0.5 due to reduced barium carrier recovery. The minimum detectable activity (MDA) was met and no qualification of the sample data was required.
The Uranium sample MW-19-03 required a dilution to quantify the data. The reporting limit for the re-extracted sample was not met. There is activity above the MDA which resulted in the three isotopes to be qualified estimated “J” for that samples.

Overall, the data is suitable for the intended data usage. All analyses were performed, and the data met the required QC criteria with the following exceptions. The data is 100% complete.

Summary of Qualified Data

Sample ID	Parameter	Qualifier (pCi/L)	Reason Code
MW-19-03	Uranium 234 Uranium 235/236 Uranium 238	183 J 3.9 J 99 J	RL not met
BWBG-SB36-0.5	Uranium 234 Uranium 238 Radium 228	1.03 J 0.58 J 3.6 J	RPD Dup
BWBG-SB36-0.5-A	Uranium 234 Uranium 238 Radium 228	2.11 J 1.0 J 0.98 J	RPD Dup
BWBG-SB33-25	Thorium 228 Thorium 230	0.49 J 0.62 J	MB
BWBG-SB34-15	Thorium 228 Thorium 230	0.51 J 0.43 J	MB
BWBG-SB34-28	Thorium 228	0.38 J	MB
BWBG-SB35-0.5	Thorium 228	0.32 J	MB
BWBG-SB35-40	Thorium 228 Thorium 230	0.23 J 0.50 J	MB

Data Validation Report

This report contains the results of the review and validation of the specified data package performed by Marcia Olive, Bhate Environmental Associates, Denver, Colorado.

Introduction

This data validation report covers samples taken from Holloman Air Force Base, New Mexico, on September 19-21, 2008. Two aqueous samples and thirteen soil samples were taken from the Background Study for analyses. All analyses were performed by TestAmerica Laboratories, St. Louis. The specific samples included in this validation were:

Sample ID	Matrix	Collection Date	Lab SDG	Analyses
MW41-03	Water	9/21/08	D8I230152	Carbon 14 (C-01-1), Isotopic Thorium (A-01-R), Isotopic Uranium (A-01-R), Lead-210 (STL-RC-0211), Radium-226 (903.0), Radium-228 (904.0)
MW58-03				
BWBG-SB22-0.5	Soil	9/19/08		
BWBG-SB22-30		9/19/08		
BWBG-SB22-60		9/19/08		
BWBG-SB10-0.5		9/20/08		
BWBG-SB10-25		9/20/08		
BWBG-SB10-50		9/20/08		
BWBG-SB13-0.5		9/20/08		
BWBG-SB13-22		9/20/08		
BWBG-SB13-50		9/20/08		
BWBG-SB14-0.5		9/20/08		
BWBG-SB14-27		9/20/08		
BWBG-SB14-55		9/20/08		
BWBG-SB14-55-A		9/20/08		

This data was validated against the laboratory's QA/QC limits using the guidelines and practices published in the *USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review* (EPA, October 2004), *USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review* (EPA, October 1999) and *Evaluation of Radiochemical Data Usability* (DOE, April 1997).

Sample Handling and Holding times

The samples were properly preserved and transferred under chain-of-custody to the laboratory for analysis and analyzed within the required holding times.

Blanks

Thorium 230 was detected in the method blank associated with the soil samples. Radium 228 was detected in the method blank associated with the aqueous samples. Only those sample concentrations where the normalized absolute difference between the sample and method blank was less than 2.58 were qualified estimated “J”.

Laboratory control sample (LCS)

The LCS percent recoveries were within control limits for all analysis.

Matrix spike/matrix spike duplicates (MS/MSD)

The MS percent recovery (%R) for carbon 14 was outside QC limits associated with the soil samples. The sample used in the quality control analysis was from another lot. Therefore precision and accuracy was gauged by the LCS recovery, which was in control, and no qualification was required.

Project specific quality assurance/quality control

One field duplicate sample was submitted as part of this sampling event. The RPD, according to the QAPP, between the sample (BWBG-SB14-55) and the duplicate was exceeded for Radium 226, Uranium 238 and Thorium 228/230. These radionuclides were qualified estimated “J” in both samples.

The laboratory duplicate met the requirements of radiochemical data usability without qualification.

Compound quantitation and reporting limits

The lead carrier recovery was outside the upper control limit for all samples with the exception of BWBG-SB22-0.5. The sample carrier yield was evaluated in reference to the chemical yield of the quality control samples. The LCS yields and recoveries were within control limits; data was accepted without qualification.

Overall assessment of data

In order to minimize matrix interference the sample size of the Lead 210 aqueous samples were reduced which resulted in the reporting limit not being met. The minimum detectable activity (MDA) was met and no qualification of the sample data was required.

Overall, the data is suitable for the intended data usage. All analyses were performed, and the data met the required QC criteria with the following exceptions. The data is 100% complete.

Summary of Qualified Data

Sample ID	Parameter	*Qualifier	Reason Code
MW41-03	Radium 228	1.06 J	MB
MW58-03	Radium 228	1.27 J	MB
BWBG-SB22-0.5	Thorium 230	0.24 J	MB
BWBG-SB22-30	Thorium 230	0.49 J	MB
BWBG-SB10-25	Thorium 230	0.16 J	MB
BWBG-SB14-27	Thorium 230	0.45 J	MB
BWBG-SB14-55	Radium 226 Uranium 238 Thorium 228 Thorium 230	0.78 J 0.74 J 1.01 J 0.74 J	RPD Dup
BWBG-SB14-55-A	Radium 226 Uranium 238 Thorium 228 Thorium 230	0.36 J 0.25 J 0.58 J 0.41 J	RPD Dup/MB

*In pCi/L

TestAmerica

THE LEADER IN ENVIRONMENTAL TESTING

To: Marcia Olive, Bhate Environmental Associates, Inc.

Re: Truncation of Carrier Yields

Date: April 15, 2011

Most Radiochemistry methods employ a radioactive tracer or stable carrier to determine the actual chemical recovery of the analysis. The tracer or carrier is chosen that exhibits similar chemistry (in terms of the method) such that it will follow the analyte of interest through the method directly. For method 903.0, barium is used as the stable carrier, as barium and radium have nearly identical chemical characteristics. A known amount of the barium carrier is added at the start of the analysis. A series of chemical separations and cleanups is performed, eventually leading to a final precipitation of barium sulfate. The radium follows along in the process and is co-precipitated with the barium sulfate.

During analysis, it is not expected to get back more barium carrier than added (>100%). Recoveries slightly over 100% may be observed simply due to uncertainties in the measurement and normal variance in the analysis process. However, much higher recoveries (>110%) are not typically observed unless the sample contains inherent concentrations of the carrier. Recoveries influenced/biased high by native concentrations of the carrier element result in low biased activity results. To reduce the effect of the low bias to activity results, carrier chemical recoveries (typically above 110%) may be truncated to 100%. While the “true” recovery may be something different (less than 100%), truncating to 100% helps to reduce the magnitude of the low bias to the result/uncertainty/MDC.

Terry Romanko

Technical Director

TestAmerica St. Louis

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