Environmental Restoration
University of California
Environmental Restoration Project, MS M992
Los Alamos, New Mexico 87545
505-667-0808/FAX 505-665-4747

U. S. Department of Energy
Los Alamos Area Office, MS A316
Environmental Restoration Program
Los Alamos, New Mexico 87544
505-667-7203/FAX 505-665-4504

Date: June 10, 1998
Refer to: EM/ER:98-191

JUN

Dr. Robert S. Dinwiddie
NMED-HRMB
P.O. Box 26110
Santa Fe, NM 87502

SUBJECT: RESPONSE TO REQUEST FOR SUPPLEMENTAL INFORMATION ON THE RFI REPORT FOR TA-0, PRS 0-030(g) (FORMER OU 1071, FU 1)

Dear Dr. Dinwiddie:

Enclosed is the Los Alamos National Laboratory's response to the New Mexico Environment Department Hazardous and Radioactive Materials Bureau's request for supplemental information (RSI) on the Resource Conservation and Recovery Act Facility Investigation Report for Technical Area 0, Potential Release Site 0-030(g). The RSI was received on May 14, 1998.

If you have any questions, please contact Terry Rust at (505) 665-8843 or Joe Mose at (505) 667-5808.

Sincerely,

James M. Ray for

Julie A. Canepa, Program Manager
LANL/ER Project

Sincerely,

T.J. Taylor

Theodore J. Taylor, Program Manager
DOE/LAAO

JC/TT/rfr

Enclosure Response to RSI on the RFI Report for TA-0 (Former OU 1071, FU 1)

7011

HSWA LANL 1/1071/0/0-030(g)

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RESPONSE TO REQUEST FOR SUPPLEMENTAL INFORMATION FOR THE RFI REPORT ON POTENTIAL RELEASE SITE 0-030(g)

INTRODUCTION

This document responds to a letter titled, "Request for Supplemental Information for RFI Report on SWMU 0-030(g), Dated May 12, 1998 for Los Alamos National Laboratory LA-UR-95-3263," from the New Mexico Environment Department (NMED) Hazardous and Radioactive Materials Bureau (HRMB) to the Los Alamos National Laboratory (LANL) Environmental Restoration (ER) Project. To facilitate review of this response, NMED's comments are included verbatim. The comments are divided into general and specific categories as presented in the letter. LANL's responses follow each NMED comment.

GENERAL COMMENTS

NMED Comment

1. *LANL shall develop a new Sampling and Analyses Plan (SAP) to define the nature, rate and extent of contamination beneath the former septic tank, vitrified clay pipe (VCP) and outfall area to the point where Canyons investigation begins. The contamination has not been delineated e.g. plutonium, americium and PCBs were found in the last sample (AAB3580) taken downstream from the outfall. Further investigation should be done to determine the lateral and vertical extent of contamination. Lead, chromium and nickel were found above background values beneath the outfall VCP which suggests a release(s) (e.g. AAA4375) to the environment. It does not appear to the RPMP that the extent was delineated beneath the VCP. As part of the new SAP(s) LANL shall perform storm water sampling in the drainage channel and Acid Canyon to the former TA-45 outfall.*

LANL Response

A SAP development team, schedule, budget, and contracts are currently being developed. A reconfirmation sampling scheme and extent sampling requirements will be developed with NMED's involvement. The SAP has been tentatively scheduled to begin at the start of FY 99 (Oct 1, 1998).

NMED Comment

2. *Include the SOP methodology for the kinetic phosphorescence analyses for total uranium with the detection limit/quantitation limit for this technique for different matrices (water, soil, tuff/rock). Clarify what methods were used for the digestion of the samples for all inorganic analyses (i.e. nitric or hydrofluoric acid).*

LANL Response

The analytical laboratory performing the kinetic phosphorescence analysis reported detection limits at 5 ng/g in soils and 1 ng/ml in water. Sample digestion was done using nitric acid per the SOP. Documentation of this analysis is included in Attachments 1 and 2.

NMED Comment

3. *Clarify if any samples used in determining nature, rate and extent of contamination as well as the confirmatory samples were composited. Table A-1, Appendix A, suggests that sample AAA1909 was composited as it was collected between 3.0 and 8.0 feet bgs (below ground surface).*

LANL Response

No samples taken at this site were composited and the referenced sample depth information was incorrectly reported. Actual sample depth was 2–2.5 ft. bgs, which was also identified as the surface below the oufall pipe. It is being corrected now and the NMED/HRMB has been notified of the change.

NMED Comment

4. *Clarify the matrix of samples, whether soil, sediment, fill or tuff and provide the information on grain size, total organic carbon content (estimated or determined) of the soil, sediment and fill samples. The report indicates that the septic tank was excavated in tuff and the clay pipe may also have been been located in the tuff and not in the soil.*

LANL Response

It appears that the report used the term "soil" to generically describe unconsolidated tuff that was abraded or crushed during excavation and seemed to be loose in the bottom of the excavations. This will be confirmed as part of the SAP (See Comment 1 above) implementation.

NMED Comment

5. *The report did not address if any cracks or fractures were observed in the tuff beneath the septic tank and clay pipe. Provide any pictures or videos taken at the time of excavation of the septic tank and VCP to determine the presence of cracks or fractures in the tuff.*

LANL Response

The former project leader of this site recalls that there were no visible fractures in the excavation under the septic tank. However, there was not a concerted effort to identify fractures small enough to be obscured by loose material that remained in the excavation after the tank and over-excavated material was removed. Videotape history of the entire remediation with additional video footage of the excavation is included as Attachment 5.

NMED Comment

6. *LANL shall provide rationale for performing limited suite analyses (e.g. mercury and lead only) at certain locations and performing a more complete suite (all detected COPCs) at other locations, when a variety of COPCs has been positively identified in the contents of the septic tank and outfall area (e.g. plutonium, chromium, PCBs etc.). A representative suite of analyses shall be completed especially on the confirmatory samples.*

LANL Response

Confirmatory sampling was done with EPA Region 6 input and approval. The sample suites were developed, with the determination that only lead and mercury were of significant concern to warrant the number of sample locations chosen, and agreed upon by EPA, DOE, and LANL. Documentation of this agreement is included as Attachment 3.

NMED Comment

7. *It is likely the waste in the septic tank was the source of PCBs detected during confirmatory sampling. In addition, a surface sample which contains PCBs slightly less than SAL (1ppp)* (sic) *indicates the need for subsurface characterization.*

LANL Response

This will be addressed in the SAP being developed.

NMED Comment

8. *LANL states in the executive summary and in the conclusions that "Moreover should further migration occur, the chemicals' concentrations will decrease even more because of surface runoff dilution." This approach to corrective action is not acceptable to RPMP.*

LANL Response

The wording for these sections was poorly chosen. The intent was to convey the fact that the concentrations present at the surface are extremely low to begin with and that the potential for migration and concentration is negligible. New text to address this issue is included as Attachment 4.

NMED Comment

9. *Once the nature and extent of the contamination has been defined, both Human Health and Ecotoxicological Screening Assessments should follow procedures described in the Risk Based Decision Tree.*

LANL Response

This will be addressed in a supplemental report that will be prepared prepared after the SAP is executed.

NMED Comment

10. *If available, please provide any "As-built" plans for the septic tank.*

LANL Response

LANL is searching records for plans, blueprints, and photographs of this tank. If located, they will be provided to NMED.

Attachment 1
to the
Request for Supplemental Information
for
PRS 0-030(g)

Quanterra Incorporated
13715 Rider Trail North
Earth City, Missouri 63045

314 298-8566 Telephone
314 298-8757 Fax

Quanterra
Environmental Services

REVISION

FAX: 505-665-4632

TO: Terry Rust
LANL
FROM: Bob White RBW
DATE: 5/28/98

Total Uranium by KPA:

Detection Limit

Soils	5 ng/g
Water	1 ng/mL

Attachment 2
to the
Request for Supplemental Information
for
PRS 0-030(g)

UNCONTROLLED COPY

Controlled Copy No. _____

OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: Analysis of Total Uranium by Laser-induced Phosphorimetry

(SUPERSEDES: SL13022)

Prepared by: Margaret Writer

Reviewed by: Donald L. Dill
Technical Specialist

Approved by: Margaret Writer
Quality Assurance Manager

Approved by: Donald L. Dill for JK
Environment, Health and Safety Coordinator

Approved by: [illegible]
Laboratory Director

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OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

Title: Analysis of Total Uranium by Laser -induced Phosphorimetry

1.0 SCOPE AND APPLICATION

1.1 This procedure provides detailed instructions for the preparation of liquid samples for determination of total uranium in water by laser induced phosphorimetry, following the procedure described in ASTM method D5174-91. This procedure also applies to solid samples which have been prepared according to STL-RC-0004.

1.2 This procedure provides instructions for the calibration of the Kinetic Phosphorescence Analyzer (KPA) and the analysis of liquid samples.

1.3 This procedure is suitable for measurement of uranium concentrations above 1.0 µg/L. For solid samples, the detection limit is 50 µg/kg ,0.05 µg/g) assuming 0.1 g. is taken to 5 ml. for analysis.

1.4 Responsibilities:

1.4.1 It is the responsibility of the Radiological Preparation Lab to order and schedule samples for preparation and analysis of samples that require total uranium by KPA.

1.4.2 It is the responsibility of the Radiological Group Leader to ensure that data is reviewed according to the Quanterra review procedures before submitting to Document Control.

1.4.3 It is the responsibility of the analyst to follow this procedure and to report any abnormal results to the Radiological Group Leader or designate.

2.0 SUMMARY OF METHOD

2.1 A water sample preserved with nitric acid to a pH of < 2 is thoroughly shaken to suspend any particulates. A 5 ml aliquot is wet ashed in a pre-leached 20 ml scintillation vial to remove any organics present. The wet ashed sample is diluted to 5.0 ml with 0.8N nitric acid. Solid samples are prepared according to SOP STL-RC-0004, and brought to 5.0 ml in the same manner.

2.2 A 1.0 ml sample aliquot is put into a plastic disposable 5.0 ml cuvette and 1.5 ml of Uraplex is added to the sample and mixed. The sample is then ready for analysis by the Kinetic Phosphorescence Analyzer (KPA).

3.0 DEFINITIONS

3.1 See Quanterra Quality Assurance Management Plan (QAMP) for glossary of common terms.

4.0 INTERFERENCES

4.1 Method interferences may be caused by organics, chlorides and reducing metals if samples were not properly wet ashed. After the sample is wet ashed and diluted to 5.0 ml with 0.8N nitric acid, **caution should be taken not to pipette any solids from the bottom of vial when taking aliquot for analysis.**

4.2 Care should be taken not to touch disposable cuvettes optical surfaces. Finger oils will increase sample background. See the Chemchek Instrument Manufacturer's Manual for a detailed description of proper cuvette handling.

5.0 SAFETY

5.1 Procedures shall be carried out in a manner that protects the health and safety of all Quanterra Associates.

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3 The following materials are known to be hazardous:

5.3.1 The following materials are known to be corrosive: nitric acid.

5.3.2 The following materials are known to be oxidizing agents: hydrogen peroxide, nitric acid.

5.4 THE LASER BEAM PRODUCES INVISIBLE ULTRAVIOLET RADIATION. MAKE SURE THAT THE LASER BEAM WILL NOT CONTACT SKIN, HIGHLY REFLECTIVE SURFACES, OR THE EYES.

5.5 Exposure to chemicals must be maintained as low as reasonably achievable, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6 The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit or by other means of mechanical ventilation.

5.7 All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra Associate. The situation must be reported immediately to a laboratory supervisor.

6.0 EQUIPMENT AND SUPPLIES

6.1 Kinetic Phosphorescence Analyzer (Chemchek KPA 10). Contains a laser dye cell with methanol solution of stilbene-420.

6.2 Volumetric Pipettes

6.3 Volumetric Flasks

6.4 Plastic disposable cuvettes (5.0 ml). A cuvette with a cap is required for the Reference Standard.

6.5 Repipetter 0 - 10 ml capability

6.6 Liquid scintillation vials leached prior to analysis by placing vials in 4N nitric acid for 48 hours. Decant the 4N nitric acid into the appropriate waste container. Rinse each vial with deionized water and air dry.

7.0 REAGENTS AND STANDARDS

7.1 Reagents

7.1.1 Uraplex, proprietary complexing agent for uranium (Chemchek Instrument, Inc.), or equivalent. Refrigerate Uraplex when not in use. Dilute with reagent water, and use diluted Uraplex within one month of dilution.

7.1.2 Hydrogen Peroxide (H_2O_2), 30%.

7.1.3 Nitric acid, concentrated (HNO_3), 16N, Tracepur.

7.1.4 4N Nitric acid (HNO_3): add 250 ml of concentrated nitric acid to 500 ml of reagent water and dilute to 1 liter with reagent water. Mix well.

7.1.6 0.8N Nitric acid (HNO_3): Add 5 ml of concentrated nitric acid to 50 ml of reagent water and dilute to 100 ml with reagent water. Mix well. **This solution must be prepared fresh daily.**

7.1.7 Uranium Standards: At least two separate manufacturers or lots of standards are required. One set of standards, at a minimum, must be traceable to EPA or NIST standards or otherwise certified by the manufacturer. The primary standards have an expiration date defined by the manufacturer. Intermediate solutions prepared from the stock will be disposed of after 6 months, or upon expiration of the primary, whichever comes first. Working standards prepared from the Intermediate will expire in 90 days from preparation, or upon the expiration of the parent, whichever comes first.

7.1.8 Ottawa sand, used for Method Blank and LCS for soil samples.

7.2 Prepared Reagents

7.2.1 Reagents are prepared from reagent grade chemicals, unless otherwise specified.

7.2.2 Reagent water is used throughout. Reagent water is monitored for interfering impurities by analyzing blank reagent water.

7.2.3 Replace lab prepared reagents annually, unless otherwise specified below. As a minimum, label all reagents with chemical name, concentration, date prepared, preparer's initials, and expiration date.

7.2.4 Uranium calibration standards are prepared in acid leached volumetric flasks (use procedure in section 6.6) to prevent contamination from natural uranium in glass. Standards are prepared for calibration curves in 0.8M nitric acid. A minimum of five standard levels are required for initial calibration.

7.2.5 Standards are prepared with a concentration range of 1.0 ng/ml to 200 ng/ml. If the criteria for acceptance of curves through this range are not possible, the range must be narrowed by lowering the concentration of the highest standard. Alternatively, separate calibration curves for a low range and a high range may be established.

7.2.6 One of the calibration standards described in section 7.2.5 is also used as the ICV and CCV (see calibration requirements, Section 10). A standard from a separate

manufacturer and lot is used to prepare an LCS solution. For waters, the LCS spiking mix is prepared at a level of 200 ng/ml, and 1.0 ml is pipetted into 4 ml of deionized water. For soil samples, 1 ml. of a mix made up at 1000 ng/ml is spiked on to 1 g. of Ottawa sand. Other concentrations which give a mid range spike may be used.

7.2.7 Reference Standard: A solution of uranium in URAPLEX containing 0.1-0.3 µg of uranium in a filled cell. The reference standard should give 20-70 counts per pulse. **Prepare Reference Standard daily.**

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 All samples must be preserved with nitric acid to a pH < 2.

8.2 Samples may be collected in plastic or glass bottles.

8.3 The maximum holding time for all samples is 6 months from date of collection.

9.0 QUALITY CONTROL

9.1 A calibration verification standard shall be run immediately following calibration (Initial Calibration Verification or ICV) and at a frequency of once every 10 samples thereafter (Continuing Calibration Verification or CCV). Acceptable limits for verification standards are 90-110% of true value. If the results for a verification standard are confirmed to be outside these limits, the analysis must be stopped, a new calibration must be performed, and all samples run after the last valid verification standard must by reanalyzed.

9.2 One Method Blank must be prepared and analyzed with each analytical batch. The concentration of analyte in the method blank may not exceed the reporting limit. If the result for a Method Blank is greater than the reporting limit, all associated samples must be reprepared and reanalyzed. An exception to this is that sample results which exceed the Method Blank concentration by a factor greater than or equal to ten (10) may be reported.

9.3 One Laboratory Control Sample (LCS) must be prepared and analyzed with each analytical batch. The acceptable limit for the recovery of the spike in the LCS is determined by laboratory generated control tables, which will give upper and lower limits of acceptability.

If the result for an LCS is outside established control limits, the all associated samples must be reprepared and reanalyzed.

9.4 A duplicate sample must be analyzed with each analytical batch The suggested control limits for the Relative Percent Difference (RPD) for all duplicate analyses are 20% for water samples and 40% for solid samples which have concentrations ≥ 5 times the Reporting Limit. For samples < 5 times the Reporting Limit, the duplicate should be within the error of the sample.

9.5 Matrix spike/matrix spike duplicates will be analyzed on a project-specific basis. The suggested limits for the MS/MSD are those determined for the LCS by control tables.

9.6 Samples associated with method blanks or laboratory control samples which fail the criteria of this section must be prepared and re-analyzed with an acceptable blank and LCS. For matrix spike, matrix spike duplicate, or sample duplicate analyses, data will be reported with appropriate flags when the results fall outside the suggested control limits. No reanalyses will be performed for out-of-control matrix spikes, matrix spike duplicates, or sample duplicates unless specifically requested by a Project Manager.

10.0 CALIBRATION AND STANDARDIZATION

10.1 Prepare a reference cell by adding 1.0 ml of 100-300 ng/ml natural uranium and 1.5 ml of Uraplex to the reference cuvette. Cap, mix well and wipe the outside of the reference cuvette with a lint-free tissue. Place the reference cell in the reference cell holder on the KPA.

10.2 Prepare a background sample by combining 1.0 ml of 0.8N nitric acid and 1.5 ml of Uraplex in a cuvette. Cap, mix well and wipe the outside of the reference cuvette with a lint-free tissue. Load cell for analytical measurement.

10.3 With the reference and background sample cuvettes in place, select calibration mode. Once in calibration mode select "measure background" (F3). Do not open the door of the instrument while it is in operation, as irreversible damage may be caused to the instrument. Do not look directly at the laser, as damage to the eye may result. **If high and low curves are used, a separate background must be run for each curve.**

10.4 Instrument Calibration

10.4.1 Enter operator ID into computer file before beginning sequence. Initiate analysis run log for analytical sequence (Figure 2).

10.4.2 **Clear previous calibrations.**

10.4.3 After the background is complete select "continue calibration" (F1) followed by "edit calibration"(F2). Enter all standard concentrations the curve.

10.4.4 To start the measurement of standards select "Standard" (F1) and position cursor to the standard to be analyzed.

10.4.5 Starting with the lowest concentration standard pipette 1.0 ml of standard solution and 1.5 ml of Uraplex into a 5 ml cuvette. Mix and place the standard in sample holder and with the standard in place select "enter" to start measurement of standard. The low standard should give approximately 10 counts per pulse. Significantly higher or lower counts indicate either that the laser dye solution needs changing or that the laser plasma may need service. Contact supervisor for maintenance instructions.

10.4.6 Continue with a minimum of four additional standard concentrations to complete the calibration curve.

10.5 Acceptable laser measurements will have the following characteristics:

10.5.1 The lifetime for each measurement must be greater than 100 µs.

10.5.2 R^2 for each sample measurement must be >0.96.

10.5.3 R^2 for each calibration standard measurement must be >0.99.

10.5.4 The concentration of each standard calculated from the resultant curve must be within 10% of true value.

10.6 The curve may be set up as linear through the origin, linear with an intercept other than zero, or quadratic. Only the highest and lowest points may be dropped from the calibration curve, and the lowest point may be dropped only from a high curve (when more than one curve is used).

11.0 PROCEDURE

11.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and a corrective action described.

11.3 Analysis/Preparation of Sample for Uranium Analysis

11.3.1 Thoroughly shake water samples to suspend any particulates and pipette 5.0 ml into a leached liquid scintillation vial. If preparing a spiked sample, pipette 4.0 ml of deionized water (for a LCS) or 4.0 ml of sample (for a MS or MSD) into a scintillation vial and spike with the solution described in section 7.2.5. Document the sample preparation on the sample preparation sheet. (Figure 1).

11.3.2 For solid samples, prepare samples according to SOP STL-RC-0004. Treat aliquot of prepared sample equivalent to 0.1 g. in the same manner as a water sample. Prepare soil method blank from 1 g. of Ottawa sand.

11.3.3 Add 3 ml of 16N nitric acid and place on a hot plate at medium heat. Add 0.5 ml of hydrogen peroxide (30%) dropwise and wet ash to dryness. Repeat (approximately three (3) times) wet ashing until a white or translucent residue is all that remains.

NOTE: If sample residue does not appear white or translucent, place the scintillation vial with sample residue in a muffle furnace and heat the sample for 1 hr at 500 ± 25°C.

11.3.4 The sample may be held after muffling until scheduled for analysis. **Do not proceed to 11.3.5 until just before KPA analysis.**

11.3.5 Dissolve residue in 5.0 ml of 0.8N nitric acid, cap and mix.

11.3.6 Add 1.0 ml of sample to a sample cuvette and add 1.5 ml of Uraplex solution. Wipe the outside of the cuvette with a lint-free tissue. Place the cuvette in the instrument for analysis.

11.3.7 In analyze mode select F1 and use the following instructions:

11.3.7.1 Enter Sample ID.

11.3.7.2 Enter Sample Description.

11.3.7.3 Select Concentration Range (H,L).

11.3.7.4 Enter Final Volume after treating Aliquot.

11.3.7.5 Enter Sample Aliquot Volume or weight.

11.3.7.6 Check that sample cuvette is placed squarely into sample holder.

11.3.7.7 Press enter to start analysis.

11.3.7.8 Print Report (F2).

11.3.7.9 Select F1 and repeat steps 11.3.4 through 11.3.7.8 to analyze additional samples. Record all runs on the analysis run log (Figure 2).

11.3.7.10 Check sample printouts for lifetime and R^2 criteria of acceptability (10.5.1 and 10.5.2, except for samples with no detectable concentration). Dilution of the sample may be required to achieve an acceptable analysis.

11.3.5.11 Record results on the sample data sheet (Figure 3).

11.3.5.12 If the sample appearance or initial results indicate that a dilution is necessary, a smaller amount of sample may be used and an appropriate amount of 0.8M nitric acid added to bring the volume to 1.0 ml.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 The KPA instrument calculates the uranium concentration, giving the results in units of nanograms uranium per milliliter in the analyzed sample. These results can also be reported in units of activity (picocuries per liter), assuming a natural abundance of uranium isotopes exists in the sample.. The following formula is used for the conversion:

Activity (pCi/L) =(ng/ml Uranium) (7.06×10^{-4} pCi/ng) (1000 ml or g/L)

For soil samples:

$$\text{Concentration } (\mu g/g) = \frac{(\text{ng/ml Uranium}) \times (\text{ml final aliquot})}{\text{g soil in aliquot} \times 1000\ \text{ng}/\mu g}$$

Activity (pCi/g) = µg /g Uranium × 0.706 pCi/ µg

Other conversions can be made on the results following the requirements of a client. For reporting purposes, the error calculated by the instrument may also be converted according to the following formula into the total error for the procedure:

$$\text{Total Error (pCi / L or kg)} = 1.96 \times A_s \times \sqrt{\left(\frac{U_s}{A_s}\right)^2 + 0.0025}$$

where:

A_s = Activity in pCi/L or kg
U_s = The instrument error converted to pCi/L by the same activity conversion used for the sample activity

12.2 Spike Recovery (LCS)

$$\%\ \text{Recovery} = \frac{\text{ng found}}{\text{ng added}} \bullet 100$$

where:

ng found = ng of uranium determined from analysis
ng added = ng of uranium added to LCS

12.3 Spike Recovery (MS or MSD)

$$\%\ \text{Recovery} = \frac{\text{ng found - ng sample}}{\text{ng added}} \bullet 100$$

where:

ng found = ng of uranium determined from analysis
ng sample = ng of uranium in original sample
ng added = ng of uranium added to MS or MSD

12.4 Relative Percent Difference (RPD).

$$\text{RPD} = \frac{|\text{sample}_1 - \text{sample}_2|}{(\text{sample}_1 + \text{sample}_2)/2} \bullet 100$$

where:

sample 1 = ng of uranium in the MS or sample replicate 1
sample 2 = ng of uranium in the MSD or sample replicate 2

13.0 METHOD PERFORMANCE

Training Qualification:

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

14.0 POLLUTION PREVENTION

This procedure will be carried out in a manner consistent with all applicable federal, state and local regulations regarding pollution control and prevention. Specific controls due to the accidental release of hazardous materials can be found in the Quanterra Chemical Hygiene Plan and facility attachments.

15.0 WASTE MANAGEMENT

Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedure. The Health and Safety Director should be contacted if additional information is required.

16.0 REFERENCES

16.1 "Operations and Service Manual-Kinetic Phosphorescence Analyzer KPA-10", Chemchek Instruments, Inc., Richland, WA, 1987.

16.2 Quanterra Quality Assurance Manual, current revision.

16.3 American Society for Testing and Materials, Volume 11.01, Method D5174-91, "Standard Test Method for Trace Uranium in Water by Pulsed-Laser Phosphorimetry".

16.4 Associated SOPs

16.4.1 STL-QA-0002, "Standards Preparation".

16.4.2 STL-QA-0006, "Sample Receipt and Chain-of-Custody".

16.5.3 STL-QA-0013, "Personnel Training and Evaluation".

16.5.4 STL-QA-0004, "Automatic Pipetter Calibration".

16.5.5 STL-RC-0004, "Preparation of Soil, Sludge, and Filter Paper Samples for Radiochemical Analysis".

17.0 MISCELLANEOUS

17.1 Records Management/Documentation

17.1.1 Record all preparation data on a sample preparation data sheet (Figure 1) and analysis data on a sample analysis data sheet (Figure 2). Include all method blanks, LCSs, duplicates, and MS/MSDs.

17.1.2 All raw data, data run logs, copies of standard logs, and quality control charts are released to the Document Control Coordinator after review and approval.

Figure 1
Example Sample Preparation Data Sheet

Total Uranium by KPA – Preparation Sheet

St. Louis

Preparation Date: 7/17/96 QC Batch Number: 106556
Analysis Date: _____ Reviewed By: _____
Preparation Analyst: _____ Method Number: STL-RC-0110
Instrument Analyst: _____ Project Number: 519.06

Lab Identifier	Matrix	Aliquot	Final Volume	Dilution Factor	Notes or Anomalies About Sample
QCBLK106556-1	Water	ml	5 ml		
QCDUP106556-1	Water	ml	5 ml		
QCLCS106556-1	Water	ml	5 ml		
QCLCS106556-2	Water	ml	5 ml		
(11395-001)	Water	ml	5 ml		
(11395-002)	Water	ml	5 ml		
(11395-003)	Water	ml	5 ml		
(11395-004)	Water	ml	5 ml		
(11395-004DUP	Water	ml	5 ml		
(11395-004MS)	Water	ml	5 ml		

EXAMPLE PREPARATION SHEET

Comments: _____

Spike Standard ID	Volume (ml)	Conc. (ng/ml)
	1 ml	

Figure 2
Example Analysis Run Log

Quanterra Environmental Services

St. Louis Laboratory
13715 Rider Trail North
Earth City, MO 63045
(314) 298-8566

KPA Run Log

Batch Number : _____ Project Number: _____
Analyst : _____ Analysis Date: _____

Concentration	Standard ID
ng/ml	
ng/ml	
ng/ml	
ng/ml	
ng/ml	
ng/ml	
ng/ml check	

See calibration page ___

Zero point included
___ Yes
___ No

Concentration	Standard ID
ng/ml	
ng/ml	
ng/ml	
ng/ml	
ng/ml	
ng/ml	
ng/ml check	

	Sample ID	Matrix	Dil	Comments
1				
2				
3				
4				
5				
6				
7				
8				
9				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				

Reviewed By: _____ Date: _____

KPARL02.DOC Rev 2 (10/95)

Figure 3
Example Sample Data Sheet

Quanterra Environmental Services

St. Louis Laboratory
13715 Rider Trail North
Earth City, MO 63045-1205

KPA Results Data Sheet

Project: Batch:

SAMPLE	Aliquot (L)	Final Aliquot (mL)	INST CONC. (ng/mL)	INST ERROR (ng/mL)	RESULT CONC (µg/L)	RESULT ERROR (µg/L)	RESULT ACTIVITY (pCi/L)	RESULT ERROR (pCi/L)	KPA Flag

LCS True Value: MDC : 1 ng/ml = 1 µg/L = .706 pCi/L

LCS Spike Recovery: Duplicate RPD:

MS True Value:

MS Recovery :

Note: The coversion of µg of uranium to pCi of uranium is based on the DOT (49CFR173.434) conversion factor of 0.706 pCi/µg.

CALCULATED BY: _____ REVIEWED BY: _____

DATE: 7/17/96 DATE: _____

Attachment 3
to the
Request for Supplemental Information
for
PRS 0-030(g)

30 Aug 93

TELCON WITH STEVE SLATER, KELLY PITNER, AND BARBARA DRISCOLL – CONCERNING VERIFICATION SAMPLES AT SEPTIC TANK 0-030(g)

Data were faxed to Steve, Kelly, and Barbara on Friday 27 August. They all concurred that the area was clean enough to fill in the holes and restore the site. Barbara asked about background values of Hg & Pb which were not on the table. Aldrich said the data would be put on another table (analytical results + SALs + background #) and faxed to them. Assuming that the values were close to background and not near SALs there was no need to talk again. If any of them have a problem with the data they should let me know.

Attachment 4
to the
Request for Supplemental Information
for
PRS 0-030(g)

Request for Supplemental Information for PRS 0-030(g)

The full context from which the comment was derived is as follows (clarifying comments are in bold, key point is also presented in italics):
"Although these chemicals have the potential to migrate down the outfall channel, *they are not present at high enough concentrations to become a source of contamination to sensitive habitats in the canyons.* Moreover, should further migration occur, the chemicals concentration will decrease even more because of surface runoff dilution. **In addition, *removal of the septic tank, its contents, and surrounding soil eliminates any potential source of increased concentrations of COPECs at the outfall.*"**

Attachment 5
to the
Request for Supplemental Information
for
PRS 0-030(g)

This attachment is a video tape. One tape is being supplied to Dr. Robert S. Dinwiddie of NMED-HRMB and one tape is being maintained by LANL.