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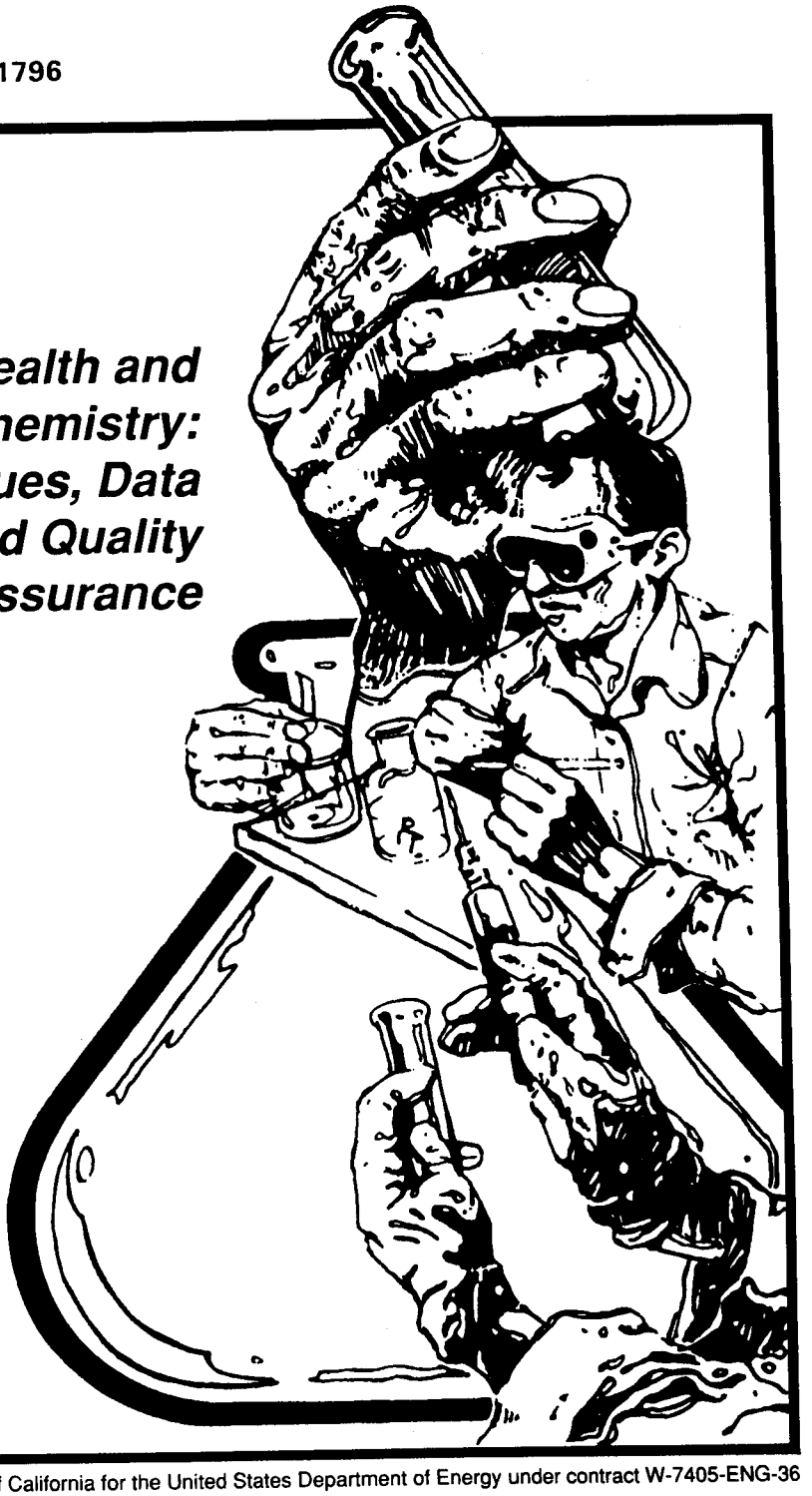

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LA-10300-M, Vol. IV
Manual

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Health and Environmental Chemistry: Analytical Techniques, Data Mangement, and Quality Assurance

Edited by Margaret A. Gautier

Los Alamos
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Los Alamos, New Mexico 87545

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PREFACE

The Health and Safety (HS) and Environmental Management (EM) Divisions are engaged in work to identify hazards to the workers and to the environment at the Los Alamos National Laboratory. Decisions that must be made in these areas depend upon the availability of sound analytical data. Procedures for the measurement of the presence and concentration of substances hazardous to human health as well as an evaluation of the quality of the environment are essential to satisfactory decision making.

The analytical method evaluates the exposure of workers and the environment to toxic substances. An important tool for evaluating the accuracy of the analytical method is the blind QC sample inserted into each analytical batch. This section of the manual is a collection of QC sample preparation methods used by the Quality Assurance Section to monitor the routine analytical performance of EM-9. These written methods are part of the overall quality control documentation effort to support the health, safety, and environment programs of the Laboratory.

The QC preparation procedures were developed by the staff of the Quality Assurance Section and are presented in a simple and concise manner. We hope that any competent analyst will be able to use these written procedures.

ACKNOWLEDGMENTS

The encouragement, leadership, and support offered by Group EM-9 management are appreciated. The information provided by the organic analysis section during the development of the organic QC preparation procedures is gratefully acknowledged. The general contributions of the following individuals in developing, evaluating, or writing the methods contained herin are formally acknowledged:

Margaret A. Gautier, Quality Assurance and Data Management Section Leader
Nancy L. Koski
William D. Moss

The section is prepared in loose-leaf form to facilitate revisions of the procedures and the inclusion of additional procedures. Requests for clarification or for help in resolving difficulties encountered while using the procedures are welcome.

Margaret A. Gautier
Editor

Quality Assurance
QC Sample Preparation Techniques

Edited by

Margaret A. Gautier

ABSTRACT

This section of the manual has been prepared to document the quality control methodology used by the Los Alamos National Laboratory's Environmental Chemistry Group (EM-9). It is part of the overall quality control effort to support the health, safety, and environmental programs at the Laboratory. The section provides organic, inorganic, and radiochemistry quality control sample preparation procedures used to QC the routine analytical work performed by EM-9. The QC preparation procedures were developed by staff within the Quality Assurance Section and have been used over several years to test their validity. QC samples prepared using these methods will fall within the calibration ranges of EM-9 analytical procedures. The QC methods utilize equipment and skills available in modern analytical chemistry laboratories. Precision and accuracy statements are provided for each procedure and analytical data is provided where data are available. Each procedure is presented in a simple and concise manner and includes sections on apparatus, reagent preparation, calibration methods, and data calculation formulas.

Quality Control

BERYLLIUM AND LEAD FOR VEGETATION ASH – QC SAMPLE PREPARATION

Analyte: Beryllium, lead	**Method No.:** QCI100
Matrix: Soil	**Spike Range:** Be 10-20 μg/g Pb 20-100 μg/g
Procedure: Inductively coupled plasma mass spectrometry (ICPMS)	**Accuracy and Precision:** Be 100% ± 4.0% RSD Pb 100% ± 6.4% RSD
Effective Date: 03/22/89	**Authors:** Nancy L. Koski

SAFETY NOTE: **Before beginning this procedure, read all of the Material Safety Data Sheets for the standards listed in Sec. 6. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.4 for proper waste disposal practices.**

1. Principle of Method

1.1. Vegetation ashes were tested for use as a suitable matrix for QC samples for the analysis of Be and Pb. No vegetation ash samples were acceptable because of generally high Be and low Pb recovery from the spiked ash samples. Although it is desirable to match the matrix of the QC sample to that of the samples to be analyzed, it has not yet been possible to match the matrix for this determination.

1.2. A soil with a certified Pb concentration in the desired spike range and a low noncertified Be concentration was chosen from our library of reference materials. This material is overspiked with Be and used as a QC sample for the analysis of Be and Pb in vegetation ash.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. Propagated error for beryllium.

2.1.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the pipette and of the balance. A relative standard deviation (RSD) of 4.0% at the 1σ level has been calculated for the calibration, spiking, and weighing process based on the following function:

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = weighing error.

2.1.2. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.3. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2} \times 100 ,$$

where S_{D_T} = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette, and
R^2_{M3} = relative variance of balance.

2.2. Propagated error for lead.

2.2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the balance. An RSD of 6.4% has been calculated for the calibration and weighing process based on the following function.

$$D = M1 \times M2 \quad ,$$

where D = calculated result,
M1 = calibration error, and
M2 = weighing error.

2.2.2. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} \quad ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.2.3. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R_{M1}^2 + R_{M2}^2} \times 100 \quad ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard, and
R^2_{M2} = relative variance of balance.

2.3. Analysis by ICPMS of QC samples prepared during 1989 and 1990 using this procedure (N = 2) gave a mean recovery of 115% ± 2% for Be and 82% ± 4% for Pb at the 1σ level. The data are published in Source Materials 9.1 and 9.2.

3. Collection and Storage of Samples

3.1. Quality-control soil samples for the determination of Be and Pb are stored in Teflon beakers at room temperature pending analysis.

4. Apparatus

4.1. Beakers: 250-mL, Teflon.

4.2. Auto-pipette: 100-μL, Rainin or equivalent.

4.3. Pipette tips: disposable.

4.4. Balance: 200-g minimum capacity, 0.1-mg accuracy.

4.5. Spatula.

4.6. Aluminum foil.

4.7. Labels printed with QC sample numbers.

5. Reagents

5.1. No reagents are required.

6. Calibration and Standards

6.1. The soil selected as a matrix for Be and Pb QCs should have a certified Pb concentration (28.2 ± 1.8 μg/g) in the desired range and a noncertified Be concentration (1.5 μg/g) of less than 5 μg/g, such as National Institute of Standards and Technology (NIST) 1646, Estuarine Sediment.

6.2. Obtain a standard such as the 1000-μg/mL Be solution from Spex Industries, Inc., 3880 Park Avenue, Edison, NJ 08820 and dilute to 100 μg/mL to overspike the certified soil.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. With each set of certified soil QC samples overspiked with Be, submit 0.25 g of the same certified soil not overspiked with Be to determine the Be concentration in the certified soil matrix. This will later be subtracted by the analyst from the Be concentration of the overspiked QC sample.

7.2. Place a Teflon beaker on the balance. Tare the balance.

7.3. Accurately weigh 0.25 g of the selected soil into the beaker. Record the weight of the soil on the beaker for reference by the analyst.

7.4. Spike an aliquot of 100-μg/mL Be standard solution on the soil so that the concentration of the Be overspike, minus the Be concentration present in the reference material, falls into the desired spike range.

7.5. Accurately weigh an additional 0.25 g of the reference material to be used to determine the amount of Be present in the reference material and mark it as the nonspiked sample.

7.6. Because a paper label does not adhere well to a Teflon beaker, write the QC sample number on the beaker. Destroy the paper label associated with the sample number used.

7.7. Cover the beaker with aluminum foil and store at room temperature pending analysis.

7.8. The concentration of Pb in the QC sample is the certified Pb concentration listed for the reference material.

7.9. The concentration of Be in the QC sample is the concentration of the overspike only. The analyst determines the Be concentration in the unspiked certified soil and subtracts that concentration from the total Be concentration in the overspiked QC sample to obtain the concentration of the Be overspike.

7.10. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD databases on the VAX.

8. Calculations

8.1. Record the certified concentration and uncertainty of Pb in the reference material as the concentration of Pb in the QC sample (μg/g).

8.2. Record the concentration of Be in the QC sample as the concentration of the Be overspike only, not as the sum of the Be overspike and the noncertified Be concentration in the soil matrix material (μg/g). The total Be concentration equals the Be concentration in the sample overspike plus the Be concentration of the certified soil.

8.3. Calculate spike concentration as in the following example.

8.3.1. If 0.2505 g of NIST 1646, Estuarine Sediment, with a certified Pb concentration of 28.2 ± 1.8 μg/g and a noncertified Be concentration of 1.5 μg/g, is overspiked with 30 μL of a 100-μg/mL Be standard, the concentration of Pb in the QC sample will be 28.2 μg/g and the concentration of the Be overspike in the QC sample will be 12 μg/g. Multiply the result by the RSD calculated in Steps 2.1 and 2.2 to obtain the uncertainty.

$$\frac{(100\ \mu g/mL)\ (1\ mL/1000\ \mu L)\ (30\ \mu L)}{0.2505\ g} = 12\ \mu g/g$$

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Solid waste contaminated with hazardous chemicals above the level of concern is not generated during the routine preparation of QC samples for analysis for inorganic contaminants.

9.1.2. If a solid waste is generated that may be contaminated at a level of concern, the appropriate paperwork is completed on a case by case basis.

9.1.2.1. A Waste Profile Request Form (WPRF) is completed describing the solid waste and is sent to the waste management group for review.

9.1.2.2. If the Waste Management group determines that the solid waste is contaminated below the level of concern, the waste is disposed of as ordinary laboratory trash.

9.1.2.3. If the Waste Management group determines that the solid waste is contaminated above the level of concern, pickup of the contaminated waste is requested as described in Sec. 9.3.

9.2. Liquid waste.

9.2.1. Liquid waste containing hazardous contaminants, such as solutions of trace metals, is accumulated in a coated glass bottle in a secondary containment tray and is kept segregated from liquid waste contaminated with organic or radioactive substances or cyanide.

9.2.2. The bottle is labeled with a hazardous waste label and a label indicating its use for trace metal waste.

9.2.3. The bottle is opened only for the period of time necessary to add waste to it.

9.2.4. When the bottle is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management group.

9.2.5. A new bottle is labeled for further use.

9.3. Waste pickup.

9.3.1. Pickup by the Waste Management group of solid waste or a full bottle of liquid waste containing trace metals is requested using the current Chemical Waste Disposal Request form (CWDR). The current Waste

Profile Request Form (WPRF) which describes the waste is referenced on the CWDR.

9.3.2. The Waste Management group picks up the waste for disposal in accord with Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.3. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 1646, Estuarine Sediment" (National Institute of Standards and Technology, Washington, DC, June 1982).

10.4. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒). Where a section heading is marked, the entire section has been revised.

TCLP ELEMENTS FOR WATER AND SOIL – QC SAMPLE PREPARATION

Analyte: Ba, Cd, Cr, Pb, As, Se, Ag	**Method No.:** QCI110
Matrix: Water, soil	**Spike Range:** 0.010-30 mg/L, dependent on the analyte and analytical method (see Step 1.5)
Procedure: TCLP followed by ICPAES, FAA, or ETVAA	**Accuracy and Precision:** 100% ± 4.2% RSD for all metals
Effective Date: 07/07/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.3 for proper waste disposal practices.

1. Principle of Method

1.1. Acidified water is spiked with standard solutions of the metals extracted by the Toxicity Characteristic Leaching Procedure (TCLP).

1.2. The acidified water QC sample represents the solution resulting from the extraction of TCLP metals from a soil matrix.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure or those sections of the procedure that are applicable.

1.4. The methods of analysis for seven TCLP metals include electrothermal vaporization atomic absorption (ETVAA) spectroscopy, flame atomic absorption (FAA) spectroscopy, and inductively coupled plasma atomic emission spectrometry (ICPAES).

1.5. The QC spike range depends on the metal of interest and on the method of analysis. Refer to the following table for spike ranges.

SPIKE RANGES FOR TCLP METALS			
Analyte	FAA (mg/L)	ETVAA (mg/L)	ICPAES (mg/L)
Ag	0.25-1.0	-	0.3-6
As	-	0.01-0.06	1.5-30
Ba	2.0-8.0	-	0.2-5
Cd	0.5-1.5	0.001-0.005	0.2-3
Cr	0.5-1.5	0.01-0.06	0.3-6
Pb	0.5-1.5	0.01-0.06	2-30
Se	-	0.01-0.06	2-30

1.6. Mercury is included as a TCLP metal but is prepared separately. The Hg standard solution must be stored in a glass container with $Cr_2O_7^{=}$ added to stabilize the Hg. Refer to QC procedure QCI200 for the preparation and storage of Hg QC samples.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with use of the pipette and the volumetric flask. A relative standard deviation (RSD) of 4.2% at the 1σ level has been calculated for the error of the standard and for the spiking and dilution process based on the following function:

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated result,
M1 = standard error,
M2 = spiking error, and
M3 = dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3}} \times 100 \quad ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette, and
R^2_{M3} = relative variance of volumetric flask.

2.1.3. All variables contributing to the error associated with the preparation of these QC samples were considered. The largest errors were used in the error propagation calculations.

2.2. Analytical results from QC samples prepared between 1989 and 1991 using this procedure and analyzed by ICPAES, FAA, or ETVAA are tabulated below. The data are published in Source Materials 10.1 and 10.2.

SPIKE RECOVERY DATA FOR TCLP METALS

	1989 mean ± SD (%)	N[a]	1990 mean ± SD (%)	N[a]	1991 mean ± SD (%)	N[a]
Water						
Ag	102 ± 19	(31)	99 ± 15	(65)	99 ± 9	(167)
As	101 ± 17	(32)	97 ± 18	(67)	102 ± 10	(119)
Ba	98 ± 18	(33)	105 ± 16	(73)	101 ± 12	(159)
Cd	98 ± 7	(30)	99 ± 8	(72)	102 ± 9	(171)
Cr	98 ± 14	(27)	104 ± 13	(60)	102 ± 7	(179)
Pb	95 ± 14	(41)	105 ± 32	(70)	102 ± 7	(199)
Se	98 ± 10	(32)	100 ± 8	(74)	100 ± 10	(134)
Soil						
Ag	102 ± 4	(5)	94 ± 30	(5)	—	
As	98 ± 7	(5)	100 ± 12	(6)	—	
Ba	104 ± 17	(9)	71 ± 40	(7)	—	
Cd	80 ± 30	(8)	99 ± 11	(8)	—	
Cr	97 ± 24	(7)	99 ± 14	(7)	—	
Pb	97 ± 9	(12)	102 ± 15	(6)	—	
Se	99 ± 15	(5)	95 ± 12	(6)	—	

[a] N = number of samples.

3. Collection and Storage of Samples

3.1. QC samples are prepared in a volumetric flask and are aliquoted into high-density polyethylene bottles for storage at room temperature pending analysis.

3.2. If any of the metal standard solutions contain HCl, they may not be added to a solution containing Ag because the Ag will be lost as insoluble AgCl. Prepare the Ag QC sample separately. The other metals may be prepared together in one QC sample.

3.3. Silver standards must be protected from light to prevent reduction and subsequent loss of Ag. Store the Ag standards at room temperature in an amber high-density polyethylene bottle or in an uncolored high-density polyethylene bottle completely wrapped in aluminum foil.

4. Apparatus

4.1. Flasks: 500-mL, volumetric, glass, class A.

4.2. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.3. Pipette tips: disposable.

4.4. Pipettes: 2-, 3-, 4-, 5-, 10-, and 15-mL, glass, volumetric, class A.

4.5. Graduated cylinder: 25-mL.

4.6. Bottles: 125-mL, high-density polyethylene.

4.7. Aluminum foil.

4.8. Bottles: 125-mL, amber, high-density polyethylene.

4.9. Labels printed with QC sample numbers.

5. Reagents

5.1. Nitric acid (16 M, low-temperature redistilled).

5.2. Water. Double-deionized, >10-MΩ/cm resistivity, specified in document ASTM-D1193 as ASTM Type I water and referred to in this procedure as double-deionized water.

6. Calibration and Standards

6.1. Spectroscopy-grade metal standards (100-10,000 μg/mL) such as those from Spex Industries, Inc., 3880 Park Avenue, Edison, NJ 08820.

6.2. If a more dilute standard is needed to reach the desired spike range, dilute a concentrated standard, taking care to match the type and concentration of acid in the dilution to those in the concentrated standard.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the number of samples to be analyzed. Periodically omit one or more metals from the acidified standard to serve as a blank QC for those metals.

7.2. This procedure describes the preparation of five 100-mL aliquots of a QC sample. QC samples may be prepared in larger or smaller quantities than described in this procedure. Adjust apparatus size as necessary.

7.3. Place approximately 400 mL of double-deionized water into a 500-mL volumetric flask.

7.4. Add 15 mL of 16 M low-temperature redistilled nitric acid to the water to produce a final acid concentration of 3% by volume.

7.5. Use a new disposable pipette tip to spike each metal standard solution into the QC sample to avoid contamination of the standards. Spike an aliquot of each TCLP metal standard solution into the acidified water in the flask to reach the desired spike range for that metal.

NOTE: If any of the metal standard solutions used to make the QC sample contain HCl, do not include Ag in the QC sample because it will be lost as insoluble AgCl. Prepare a separate QC sample for Ag as described in Step 7.9.

7.6. Dilute the QC sample to volume with double-deionized water. Mix by inversion.

7.7. Divide the QC sample evenly among five 125-mL high-density polyethylene bottles. If Ag has been included in the sample, use amber high-density polyethylene bottles or wrap uncolored bottles completely in aluminum foil.

7.8. Label each bottle with a QC sample number and store at room temperature pending analysis. The same QC number may be used for all aliquots of a QC sample from the same 500-mL volumetric flask.

7.9. If any of the metal standard solutions used to make the QC sample contain HCl, prepare a separate Ag QC sample as follows.

7.9.1. Place approximately 400 mL of double-deionized water into a 500-mL volumetric flask.

7.9.2. Add 15 mL of 16 M low-temperature redistilled nitric acid to produce a final acid concentration of 3% by volume.

7.9.3. Spike an aliquot of Ag standard solution into the acidified water in the flask to reach the desired spike range.

7.9.4. Dilute the QC sample to volume with double-deionized water. Mix by inversion.

7.9.5. To prevent the loss of Ag, divide the QC sample evenly among five 125-mL amber high-density polyethylene bottles or among uncolored high-density polyethylene bottles completely wrapped in aluminum foil.

7.9.6. Label each bottle with a QC sample number and store at room temperature pending analysis. The same QC sample number may be used for all aliquots of a QC sample from the same 500-mL volumetric flask.

7.10. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the concentration of each metal in the QC sample as μg/L or mg/L.

8.2. Calculate the spike concentration for each metal in the QC sample as in the following example.

8.2.1. If 250 μL of a 100-μg/mL Se standard is used to spike 400 mL of acidified water and the solution is diluted to 500 mL, the concentration of Se in the sample is 50 μg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(100\ \mu g/mL)\ (1\ mL/1000\ \mu L)\ (250 \mu L)}{(500\ mL)\ (1\ L/1000\ mL)} = 50\ \mu g/L$$

9. Proper Waste Disposal Practices

9.1. Solid Waste

9.1.1. Solid waste contaminated with hazardous chemicals above the level of concern is not generated during the routine preparation of QC samples for analysis for inorganic contaminants.

9.1.2. If a solid waste is generated that may be contaminated at a level of concern, the appropriate paperwork is completed on a case-by-case basis.

9.1.2.1. A Waste Profile Request Form (WPRF) describing the solid waste is completed and sent to the Waste Management Group for review.

9.1.2.2. If the Waste Management Group determines that the solid waste is contaminated below the level of concern, the waste is disposed of as ordinary laboratory trash.

9.1.2.3. If the Waste Management Group determines that the solid waste is contaminated above the level of concern, pickup of the contaminated waste is requested as described in Sec. 9.3.

9.2. Liquid Waste

9.2.1. Liquid waste containing hazardous contaminants, such as solutions of trace metals, is accumulated in a coated glass bottle in a secondary containment tray and is kept segregated from liquid waste contaminated with organic or radioactive substances or cyanide.

9.2.2. The bottle is labeled with a hazardous waste label and a label indicating its use for trace-metal waste.

9.2.3. The bottle is opened only for the period of time necessary to add waste to it.

9.2.4. When the bottle is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.5. A new bottle is labeled for further use.

9.3. Waste Pickup

9.3.1. Pickup by the Waste Management Group of solid waste or a full bottle of liquid waste containing trace metals is requested using the current Chemical Waste Disposal Request form (CWDR). The current Waste

Profile Request Form (WPRF) that describes the waste is referenced on the CWDR.

9.3.2. The Waste Management Group picks up the waste for disposal in accord with Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.3. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒▒). Where a section heading is marked, the entire section has been revised.

OIL AND GREASE IN WATER – QC SAMPLE PREPARATION

Analyte: Oil and grease	**Method No.:** QCI120
Matrix: Water	**Spike Range:** 20-100 mg/L
Procedure: Gravimetry	**Accuracy and Precision:** 100% ± 0.8%
Effective Date: 02/02/90	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.2 for proper waste disposal practices.

1. Principle of Method

1.1. Distilled water is spiked with vegetable oil to prepare a QC sample for oil and grease in water.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the balance. Since the analyst takes the sample to dryness, it is not necessary to dilute the sample to exactly one liter and the assumption is made that L = 1. A relative standard deviation (RSD) of 0.8% at the 1σ level has been calculated for the weighing process based on the following function:

$$D = \frac{M1}{L} ,$$

where D = calculated result,
M1 = weighing error, and
L = 1.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R^2_{M1} / L} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of balance, and
L = 1.

2.2. Analysis by gravimetry of QC samples (N = 13) prepared during 1990 using this procedure gave a mean recovery of 91% ± 8%. The data are published in Source Material 10.1.

3. Collection and Storage of Samples

3.1. QC samples are prepared in 1-L jars with Teflon-lined lids and are refrigerated pending analysis.

4. Apparatus

4.1. Glass jars: 1-L, with Teflon-lined screw-cap lids.

4.2. Weighing dish: small, polyethylene.

4.3. Sample dropper: glass or polyethylene.

4.4 Balance: 100-g minimum capacity, 0.1-mg accuracy.

4.5. Labels printed with QC sample numbers.

5. Reagents

5.1. Freon. Used to transfer the oil into the water.

6. Calibration and Standards

6.1. Spike QC samples with a pure vegetable oil, such as one used in cooking.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically include 3 mL of Freon in 1 L of distilled water in a jar with a Teflon-lined lid to be used as a blank.

7.2. Place a small polyethylene weighing dish on the balance. Tare the balance.

7.3. Using a sample dropper, add vegetable oil to the weighing dish to reach the desired spike range and record the weight.

7.4. Fill a 1-L glass jar nearly full of distilled water. The analyst will use the entire sample, assuming the volume to be 1 L.

7.5. Using a clean sample dropper and less than 3 mL of Freon, quantitatively transfer the oil from the weighing dish into the water in the 1-L jar.

7.6. Place the Teflon-lined lid on the jar and label it with a QC sample number.

7.7. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter the information into the CVS and CVD databases on the VAX.

8. Calculations

8.1. Record the concentration of oil and grease in the water as mg/L.

8.2. Calculate the spike concentration as in the following example.

8.2.1. If 50 mg of vegetable oil is quantitatively transferred into 1 L of distilled water, the oil and grease concentration in the water is 50 mg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

9. Proper Waste Disposal Practices

9.1. The hazardous components of an oil and grease sample are organic compounds. Dispose of waste as waste contaminated with organic compounds.

9.2. Solid waste.

9.2.1. Accumulate solid waste, such as paper wipes, glass or plastic pipettes, empty vials, and incorrectly spiked QC samples contaminated with organic compounds in a covered metal can lined with a plastic bag. Keep this segregated from PCB-contaminated solid waste and solid waste that is not contaminated with organic compounds.

9.2.2. Label the can with a hazardous waste label and a label indicating its use for waste contaminated with organic compounds.

9.2.3. Open the can only for the period of time necessary to add waste to it.

9.2.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as waste contaminated with organic compounds.

9.2.5. Place a new plastic bag into the waste can for continued use.

9.2.6. Accumulate the sealed bags in a larger container lined with a plastic bag and approved for the storage and transport of waste contaminated with organic compounds.

9.2.7. Send a reference to the current Waste Profile Request form (WPRF) describing the waste along with the sealed bag.

9.2.8. Label the larger container with a hazardous waste label and a label indicating its use for waste contaminated with organic compounds.

9.2.9. The larger container has a sealing cover. Open the can only for the period of time necessary to add waste to it.

9.2.10. When the plastic bag liner is full, seal it with tape and keep in the sealed container pending pickup by the Waste Management group.

9.3. Liquid waste.

9.3.1. Accumulate liquid waste containing organic compounds, such as rinses from syringes, out-of-date stock solutions, excess QC samples, and incorrectly spiked samples, in a covered metal can in a

secondary containment tray and is kept segregated from PCB-contaminated liquid waste.

9.3.2. Label the can with a hazardous waste label and a label indicating its use for waste containing organic compounds.

9.3.3. Open the can only for the period of time necessary to add waste to it.

9.3.4. When the can is full, cap it and keep it in the secondary containment trap pending pickup by the Waste Management group.

9.3.5. Label a new can for further use.

9.4. Waste pickup.

9.4.1. Request pickup by the Waste Management group of a full container or can of waste contaminated with organic compounds using the current Chemical Waste Disposal Request form. The current Waste Profile Request Form (WPRF) that describes the waste is referenced on the Chemical Waste Disposal Request.

9.4.2. The Waste Management group picks up the waste for disposal in accord with Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS(1991).

10.2 "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒). Where a section heading is marked, the entire section has been revised.

TRACE ELEMENTS ON FILTER MEDIA — QC SAMPLE PREPARATION

Analyte: Metals	**Method No.:** QCI140
Matrix: Filters	**Spike Range:** 0.01-4000 μg/filter dependent on analyte and analytical method
Procedure: ICPMS, ICPAES, ETVAA, or FAA	**Accuracy and Precision:** 100% ± 3.6% RSD
Effective Date: 01/01/84	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.9 for proper waste disposal practices.

1. Principle of Method

1.1. One or more standard solutions of single metals are spiked onto filter papers, preferably of the same type used to collect the samples.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

1.3. The methods of analysis for these filters are inductively coupled plasma mass spectrometry (ICPMS), inductively coupled atomic emission spectrometry (ICPAES), flame atomic absorption (FAA) spectroscopy, or electrothermal vaporization atomic absorption (ETVAA) spectroscopy.

1.4. The QC spike range depends on the metal of interest and the method of analysis. Refer to Table I for spike ranges.

TABLE I. SPIKE RANGES FOR TRACE ELEMENTS ON FILTER MEDIA

Analyte	FAA µg/filter	ETVAA µg/filter	ICPAES µg/filter	ICPMS µg/filter
Al	10-30		40-4000	
As		0.1-1.0		
Au				0.2-1.0
Ba			10-100	
Be	1-10		10-100	
Bi				0.05-0.5
Ca	10-30		100-500	
Cd		0.01-0.1	4-400	
Co			2-50	
Cr	5-20		10-100	
Cu	5-20		6-600	0.05-2
Fe	5-20		10-100	
Li	2-10		10-100	
Mn	5-20			
Mo			10-100	
Ni	5-20		10-100	0.2-1.0
Pb	5-20	0.2-1.0	10-100	0.05-0.2
Sn		0.2-1.0		
Sr				1-5
Ta	5-15			1-5
Th				0.05-0.5
Tl		0.1-0.6	10-100	0.01-0.05
U				1-5
Zn			2-30	

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with use of the pipette. A relative standard deviation (RSD) of 3.6% at the 1σ level has been calculated for the error of the standard and for the spiking process based on the following function:

$$D = M1 \times M2 \quad ,$$

where D = calculated result,
M1 = standard error, and
M2 = spiking error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} \quad ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R^2_{M1} + R^2_{M2}} \times 100 \quad ,$$

where S_{D_T} = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette, and
100 = factor to convert to percentage.

2.1.3. If two pipettes ($P_1 + P_2$) are used to add the analyte to the filter paper, use the following equation to calculate the pipetting error.

$$R_{M2} = \frac{\sqrt{\sigma_1^2 + \sigma_2^2}}{P_1 + P_2} \quad ,$$

where σ_1 = standard deviation of first pipette,
σ_2 = standard deviation of second pipette,

P_1 = mean of first pipette, and
P_2 = mean of second pipette.

Square R_{M2} and substitute this value in the equation in Step 2.1.2 to calculate the percent RSD for the use of two pipettes.

2.2. Analytical results from QC samples prepared between 1990 and 1991 using this procedure and analyzed by ICPMS, ICPAES, ETVAA, and FAA are tabulated below. The data are published in Source Materials 10.1 through 10.7.

SPIKE RECOVERY DATA FOR TRACE ELEMENTS IN FILTERS

Element	1990 mean ± SD (%)	N[a]	1991 mean ± SD (%)	N[a]
Al	98 ± 0	(2)	—	—
As	—	—	97 ± 2	(3)
Ba	122 ± 29	(7)	101 ± 0	(2)
Be	99 ± 9	(200)	102 ± 10	(170)
Ca	118 ± 25	(6)	—	—
Cd	104 ± 8	(19)	98 ± 9	(18)
Cr	101 ± 24	(30)	101 ± 8	(15)
Cu	113 ± 26	(13)	95 ± 14	(6)
Fe	127 ± 73	(23)	99 ± 5	(12)
Li	—	—	100 ± 0	(2)
Mn	97 ± 6	(6)	89 ± 0	(2)
Mo	—	—	88 ± 27	(8)
Ni	106 ± 12	(14)	98 ± 7	(12)
Pb	102 ± 14	(138)	104 ± 24	(78)
Sn	25 ± 0	(2)	—	—
Ta	—	—	62 ± 38	(5)
Th	—	—	79 ± 21	(15)
Tl	96 ± 78	(60)	220 ±380	(22)
U	104 ± 11	(51)	98 ± 15	(20)

[a]N = number of samples

3. Collection and Storage of Samples

3.1. QC samples are dried after spiking and stored in cellophane envelopes at room temperature pending analysis.

4. Apparatus

4.1. Balance: 100-g minimum capacity, 0.1-mg accuracy.

4.2. Spatula.

4.3. Platinum crucibles: approximately 50-mL capacity, with platinum covers. Each crucible should have a unique identifying number stamped or etched into it.

4.4. Desiccator.

4.5. Beaker: 50- mL, glass.

4.6. Flasks: 100- and 500-mL, volumetric, class A, glass.

4.7. Pipette: 10- mL, volumetric, class A, glass.

4.8. Graduated cylinder: 25-mL.

4.9. Filter paper: usually 1¼ -in.-diam, cellulose acetate or paper filters, such as Whatman No. 41 or 42.

4.10. Auto-pipette: 100-μL, Rainin or equivalent.

4.11. Pipette tips: disposable.

4.12. Microsyringes: 10- and 25-μL capacity, gas tight.

4.13. Envelopes: cellophane.

4.14. Filter containers: plastic.

4.15. Filter support: clean polyethylene bottle caps or beakers or similar support, slightly smaller in diameter than the filters to be spiked.

4.16. Heat lamp.

5. Reagents

5.1. Hydrochloric acid (low-temperature redistilled).

5.2. Nitric acid (low-temperature redistilled).

6. Calibration and Standards

6.1. Use spectroscopy-grade metal standards (100-10,000 μg/mL) such as those from Spex Industries, Inc., 3880 Park Avenue, Edison, NJ 08820.

6.2. If a more dilute standard is needed to reach the desired spike range, dilute a concentrated standard, taking care to match the type and concentration of acid in the dilution to those in the concentrated standard.

6.3. Solutions of the analyte of interest in the concentration range 100-10,000 μg/mL may be prepared from analytical-grade compounds for use as standards if suitable standard solutions are not readily available.

6.4. Prepare a standard for the determination of total uranium and $^{235}U/^{238}U$ isotopic ratios through ICPMS by dissolving a certified natural U_3O_8 standard, such as National Institute for Standards and Technology (NIST) Standard Reference Material (SRM) 950b, in 7.8 M HNO_3. The uranium concentration of the standard should be 1 mg/mL. The $^{235}U/^{238}U$ isotopic ratio of natural uranium is 0.0072.

6.4.1. Use low-temperature redistilled HNO_3 and double-deionized water to prepare the uranium standard solutions. Prepare 7.8 M HNO_3 by diluting 25 mL of concentrated HNO_3 to 50 mL with double-deionized water. Prepare 1.6 M HNO_3 by diluting 1 mL of concentrated HNO_3 to 10 mL with double-deionized water. Prepare 1% HNO_3 by diluting 5 mL of concentrated HNO_3 to 500 mL with double-deionized water.

6.4.2. Weigh 1 g of certified natural U_3O_8 into a platinum crucible. Cover the crucible with the platinum lid.

6.4.3. Heat the U_3O_8 in a muffle furnace at 900°C for 1 h.

6.4.4. Cool the U_3O_8 in a desiccator overnight.

6.4.5. Accurately weigh 590 ± 1 mg of the U_3O_8 into a 50-mL beaker.

6.4.6. With gentle heating, dissolve the U_3O_8 in 20 mL of 7.8 M low-temperature redistilled HNO_3.

6.4.7. Evaporate the solution to near dryness.

6.4.8. Redissolve the residue in 10 mL of 1.6 M HNO_3.

6.4.9. Quantitatively transfer the uranium solution into a 500-mL volumetric flask with 1% HNO_3 and dilute the solution to volume with 1% HNO_3.

6.4.10. Calculate the concentration of the resultant natural uranium standard as in the example in Step 8.1.

6.5. Make a uranium standard with a concentration of 100 µg/mL by diluting the 1 mg/mL uranium standard. Use double-deionized water and low-temperature redistilled HNO_3.

6.5.1. Place approximately 70 mL of double-deionized water into a 100-mL volumetric flask.

6.5.2. Add 1 mL of concentrated HNO_3 to the flask to achieve a final acid concentration of 1%.

6.5.3. Add 10 mL of the 1 mg/mL natural uranium standard to the volumetric flask using a volumetric pipette.

6.5.4. Dilute the solution to volume with double-deionized water.

6.5.5. Calculate the concentration of the resultant natural uranium standard. The $^{235}U/^{238}U$ isotopic ratio in the standard is 0.0072.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the number of samples to be analyzed. Periodically include a nonspiked filter to be used as a blank.

7.2. Select a standard solution containing the analyte of interest in a concentration such that a minimum amount of the standard will be needed to reach the desired spike range.

7.3. An auto-pipette may be used for spike volumes within the acceptable range of the auto-pipette. Microsyringes should be used for smaller spike volumes.

7.4. The volume of standard solution added to the filter at one time should never be large enough to allow contact of the solution with the filter support, which could cause loss of solution to the support. To add larger volumes of standard, it may be necessary to add the standard in more than one aliquot, allowing the filter to dry after each addition.

7.5. Place a filter paper on a bottle cap or beaker or similar so such that most of the filter is within the rim of the support.

7.6. Spike the standard solution onto the filter to reach the desired spike range.

7.7. If more than one analyte will be spiked on the filter, dry the filter after addition of each standard solution.

7.8. Dry the filters under a heat lamp or allow them to air dry.

7.9. Place the dried filters in cellophane envelopes, segregating the different analyte combinations and concentrations.

7.10. Label each envelope with a QC sample number.

7.11. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter the information into the CVS and CVD databases on the VAX.

7.12. National Institute for Occupational Safety and Health (NIOSH) Proficiency Analytical Testing (PAT) intercomparison samples.

7.12.1. Prepare filters using the metals designated by NIOSH as being included in the current set of intercomparison samples. Follow Steps 7.5 to 7.11 of this QC sample preparation method. Refer to Table II for spike ranges.

Table II. SPIKE RANGES FOR NIOSH PAT FILTERS

Cr	5-100 μg/filter
Pb	15-100 μg/filter
Cd	5-25 μg/filter
Zn	50-200 μg/filter

7.12.2. Obtain a NIOSH-PAT filter sample from a previous intercomparison round and assign it a QC sample number as a fourth QC sample.

7.12.3. Record the sample identification, concentration, and uncertainty supplied by NIOSH for the filter in the QC Laboratory notebook and enter them into the CVS and CVD databases on the VAX.

7.12.4. Place the four QC samples into plastic filter containers or clean cellophane envelopes, label with QC sample numbers, and distribute with the NIOSH-PAT intercomparison samples.

8. Calculations

8.1. Calculate the concentration of the uranium standard solution using the following atomic weights and example.

8.1.1. The atomic weight of U is 238.029 and the atomic weight of O is 16. You may also use the molecular weight of U_3O_8 recorded on the bottle label for the NIST SRM U_3O_8.

8.1.2. The purity of the NIST U_3O_8 is recorded on the certificate for the standard reference material used.

8.1.3. If 589.6 mg of 99.968% pure U_3O_8 which has been heated in the muffle furnace is dissolved in acid and diluted to 500 mL, the uranium concentration in the standard will be 1.0 g/L.

$$\frac{\left(\frac{714.087 \text{ g U}}{842.0822 \text{ g } U_3O_8}\right)(0.99968)(0.5896 \text{ g})}{(500 \text{ mL})(1 \text{ L}/1000 \text{ mL})} = 1.0 \text{ g/L}$$

8.1.4. The $^{235}U/^{238}U$ isotopic ratio in natural uranium is 0.0072 ± 0.0007.

8.2. Record sample concentrations as mg/filter, μg/filter, or ng/filter.

8.3. Calculate the spike concentration for each metal in the QC sample as in the following example.

8.3.1. If 30 μL of a 100-μg/mL beryllium standard is spiked onto a cellulose acetate filter, the concentration of Be in the QC sample is 3 μg/filter. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(100 \text{ μg/mL})(1 \text{ mL}/1000 \text{ μL})(30 \text{ μL})}{\text{filter}} = 3 \text{ μg/filter}$$

9. Proper Waste Disposal Practices

9.1. Solid Waste

9.1.1. Solid waste contaminated with hazardous chemicals above the level of concern is not generated during the routine preparation of QC samples for analysis for inorganic contaminants.

9.1.2. If a solid waste is generated that may be contaminated at a level of concern, the appropriate paperwork is completed on a case-by-case basis.

9.1.2.1. A Waste Profile Request Form (WPRF) describing the solid waste is completed and sent to the Waste Management Group for review.

9.1.2.2. If the Waste Management Group determines that the solid waste is contaminated below the level of concern, the waste is disposed of as ordinary laboratory trash.

9.1.2.3. If the Waste Management Group determines that the solid waste is contaminated above the level of concern, pickup of the contaminated waste is requested as described in Sec. 9.3.

9.2. Liquid Waste

9.2.1. Liquid waste containing hazardous contaminants, such as solutions of trace metals, is accumulated in a coated glass bottle in a secondary containment tray and kept segregated from liquid waste contaminated with organic or radioactive substances or cyanide.

9.2.2. The bottle is labeled with a hazardous waste label and a label indicating its use for trace-metal waste.

9.2.3. The bottle is opened only for the period of time necessary to add waste to it.

9.2.4. When the bottle is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.5. A new bottle is labeled for further use.

9.3. Waste Pickup

9.3.1. Pickup by the Waste Management Group of solid waste or a full bottle of liquid waste containing trace metals is requested using the current Chemical Waste Disposal Request form (CWDR). The current Waste Profile Request Form (WPRF) that describes the waste is referenced on the CWDR.

9.3.2. The Waste Management Group picks up the waste for disposal in accord with Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, and D. R. Perrin, "Quality Assurance for Health and Environmental Chemistry: 1984," Los Alamos National Laboratory report LA-10508-MS(1985).

10.2. M. A. Gautier, E. S. Gladney, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1985," Los Alamos National Laboratory report LA-10813-MS(1986).

10.3. M. A. Gautier, E. S. Gladney, W. D. Moss, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1986," Los Alamos National Laboratory report LA-11114-MS(1987).

10.4. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1987," Los Alamos National Laboratory report LA-11454-MS(1988).

10.5. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1988," Los Alamos National Laboratory report LA-11637-MS(1989).

10.6. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.7. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS(1991).

10.8. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 950b, Uranium Oxide (U_3O_8)" (National Institute of Standards and Technology, Washington, DC, March 1978).

10.9. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒). Where a section heading is marked, the entire section has been revised.

TRACE ELEMENTS IN SOLVENT — QC SAMPLE PREPARATION

Analytes: Ag, As, Ba, Cd, Cr, Hg, Ni, Pb, Se, Tl, Zn	**Method:** QCI150
Matrix: Kerosene, xylenes, methyl isobutyl ketone, and d-limonene	**Spike Range:** 300-1300 μg/L, dependent on the analytical method (see Table I)
Procedure: Analysis by ICPMS, FAA, ETVAA, or CVAA	**Accuracy and Precision:** 100% ± 5.1% RSD
Effective Date: 04/01/87	**Authors:** Nancy Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.4 for proper waste disposal practices.

1. Principle of Method

1.1. Obtain or prepare trace elements in oil standards.

1.2. Prepare working standards in base oil at approximately 100 μg/g for Ag, As, Ba, Cd, Cr, Ni, Pb, Se, Tl, and Zn, and 5 μg/g for Hg.

1.3. Spike an aliquot of each element into a polypropylene centrifuge tube. The analyst will dilute the sample to volume with the desired solvent before analysis.

1.4. The methods of analysis are inductively coupled plasma mass spectrometry (ICPMS), flame atomic absorption (FAA) spectroscopy, electrothermal vaporization atomic absorption (ETVAA) spectroscopy, or cold vapor atomic absorption (CVAA) spectrometry.

1.5. The QC spike range depends on the analytical method. Refer to Table I for the spike ranges.

TABLE I. SPIKE RANGES FOR METALS IN SOLVENT			
Element	Method	Spike range (μg/L)	Typical solvent
Ag, As, Ba, Cd, Cr, Ni, Pb, Se, Tl, Zn	ICPMS	300-800	kerosene or xylenes
Ag, As, Ba, Cd, Cr, Ni, Pb, Se, Tl, Zn	ETVAA	300-800	methyl isobutyl ketone or xylenes
Ag, As, Ba, Cd, Cr, Ni, Pb, Se, Tl, Zn	FAA	700-1300	d-limonene or xylenes
Hg	CVAA	2-4	1% nitric acid

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with the use of the analytical balance and the volumetric flask. A relative standard deviation (RSD) of 5.1% at the 1σ level has been calculated for the error of the standard and for the weighing and dilution process based on the following function:

$$D = \frac{M_1 \times M_2}{M_3} ,$$

where D = calculated result,
M_1 = standard error,
M_2 = weighing error, and
M_3 = dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3}} \times 100 \quad ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of volumetric flask, and
100 = factor to convert to percentage.

2.1.3. All variables contributing to the error associated with the preparation of these QC samples were considered. The largest errors were used in the error propogation calculations.

2.2. Results of analysis by ICPMS, FAA, ETVAA, and CVAA of QC samples prepared between 1990 and 1991 using these procedures are summarized below.

SPIKE RECOVERY DATA FOR TRACE ELEMENTS IN SOLVENTS

Analyte	1990 mean ± SD (%)	N[a]	1991 mean ± SD (%)	N[a]
Ag	112 ± 22	(6)	88	(1)
As	83 ± 35	(10)	96	(1)
Ba	95 ± 9	(8)	86	(1)
Cd	98 ± 8	(9)	88	(1)
Cr	102 ± 6	(9)	—	—
Hg	103 ± 29	(11)	103	(1)
Ni	—	—	—	—
Pb	106 ± 12	(10)	76	(1)
Se	68 ± 12	(5)	106	(1)
Tl	—	—	—	—
Zn	—	—	—	—

[a]N = number of samples

3. Collection and Storage of Samples

3.1. The QC samples are prepared on the day of analysis up to the point of dilution with solvents. The dilution is performed by the analyst just before analysis because all elements are unstable at low concentrations.

4. Apparatus

4.1. Flasks: 10- and 25-mL, volumetric, class A, glass.

4.2. Centrifuge tubes: polypropylene, 50-mL, graduated, with screw caps.

4.3. Pasteur pipettes: polyethylene, disposable.

4.4. Auto-pipette: 1000-μL, Rainin or equivalent.

4.5. Pipette tips: disposable.

4.6. Bottles: 60-mL, high-density polyethylene.

4.7. Bottles: 60-mL, amber high-density polyethylene.

4.8. Analytical balance: 100-g minimum capacity, 0.1-mg accuracy.

4.9. Labels printed with QC sample numbers.

5. Reagents

5.1. Conostan 75 Base Oil or equivalent: Conostan, Inc., P.O. Box 1267, Ponca City, OK 74603.

5.2. Conostan stabilizer or equivalent: Conostan, Inc., P.O. Box 1267, Ponca City, OK 74603.

5.3. 2-ethylhexanoic acid (reagent-grade).

5.4. Mixed xylenes (reagent-grade).

5.5. Thallium (I) acetylacetonate: ROC/RIC, P.O. Box 8390, Orange, CA 92664.

6. Calibration and Standards

6.1. Obtain organometallic single-element standards in base oil in concentrations of 100 μg/g for As, Se, and Hg and 5000 μg/g for Ba, Cd, Cr, Pb, Ni, Ag, and Zn.

6.2. Prepare organometallic single-element standards with a concentration of 100 μg/L from the 5000-μg/g standards.

6.2.1. Add 500 mg of 5000-μg/g organometallic standard to a clean, tared, 60-mL high-density polyethylene bottle. Record the mass added. **DO NOT** tare the balance.

6.2.2. Add 150-200 mg of commercial stabilizer solution to achieve 0.6-0.8% by weight of stabilizer after dilution. Record the mass added. **DO NOT** tare the balance.

6.2.3. Add the manufacturer's 75 Base Oil to bring the total mass to 25.0 g. Cap and mix the standard by inversion.

6.2.4. Repeat Steps 6.2.1 to 6.2.3 for each of the remaining 5000-μg/g standards.

6.2.5. Label each bottle with information identifying the standard.

6.2.6. Prepare the silver standard in an amber polyethylene bottle or an uncolored polyethylene bottle completely wrapped in aluminum foil to protect the silver from photodegradation.

6.3. Prepare a Hg organometallic standard with a concentration of 5 μg/g from the 100-μg/g standard immediately before analysis. A new 5-μg/g standard should be prepared each time it is used because the Hg standard is unstable at low concentrations.

6.3.1. Add 250 mg of 100-μg/g Hg organometallic standard to a clean, tared 60-mL high-density polyethylene bottle. Record the mass added. **DO NOT** tare the balance.

6.3.2. Add 30-40 mg of commercial stabilizer solution to achieve 0.6-0.8% by weight of stabilizer after dilution. Record the mass added. **DO NOT** tare the balance.

6.3.3. Add the manufacturer's 75 Base Oil to bring the total mass to 5 g. Cap and mix the standard by inversion.

6.3.4. Label the bottle with information identifying the standard.

6.4. Prepare an organothallium standard at 1000 μg/mL.

6.4.1. Weigh 37 mg of thallium acetylacetonate into a 25-mL volumetric flask. Record the mass added.

6.4.2. Add 250 μL of 2-ethylhexanoic acid as a stabilizer for the thallium.

6.4.3. Dilute the solution to volume with mixed xylenes. Mix by inversion.

6.5. Prepare a 100-μg/mL organothallium standard.

6.5.1. Pipet 1 mL of 1000-μg/mL thallium acetylacetonate standard into a 10-mL volumetric flask.

6.5.2. Dilute the standard to volume with mixed xylenes. Mix by inversion.

6.6. Check the concentration of the 100-μg/g As, Se, and Hg Conostan standards periodically. Discard and replace a standard when degradation is noted.

6.7. Conostan states that its 5000-μg/g standards are stable for more than 10 years.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed on the day requested by the analyst because all of the metals are relatively unstable at the low concentrations made for QCs. Periodically omit one or more of the metal analytes from the QC sample as a blank for that metal.

7.2. Determine which analysis method will be used. Select the spike range for the method from Table I.

7.3. Preparation of QC samples containing all metals except mercury.

7.3.1. Add most standards gravimetrically. Weigh an aliquot of a 100-μg/g standard into a 50-mL graduated polypropylene centrifuge tube to reach the desired spike range determined from Table I.

7.3.2. Record the element and mass added.

7.3.3. Retare the balance.

7.3.4. Repeat Steps 7.3.1 to 7.3.3 for each desired element except thallium.

7.3.5. Add the thallium standard volumetrically. Using an automatic pipette, add an aliquot of 100-μg/mL thallium standard to reach the desired spike range determined from Table I.

7.3.6. Screw the cap onto the centrifuge tube and submit the QC sample for analysis.

7.4. Preparation of a mercury QC sample.

NOTE: A new 5-μg/g Hg standard should be prepared each time it is used because the Hg standard is unstable at low concentrations.

7.4.1. Weigh an aliquot of the 5-μg/g Hg standard into a 50-mL graduated polypropylene centifuge tube to reach the desired spike range determined from Table I.

7.4.2. Record the element and mass added.

7.4.3. Screw the cap onto the centrifuge tube and submit the QC sample for analysis.

7.5. Just before analysis the analyst will dilute the Hg QC sample to 25.0 mL and all other metal QC samples to 50.0 mL with the solvent of choice for the analysis method. Refer to Table I for typical solvents.

7.6. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record sample concentrations as μg/L or mg/L.

8.2. Calculate spike concentrations after final dilution by the analyst as in the following example.

8.2.1. If 0.2672 g of 100 μg/g As standard is diluted to 50 mL with xylenes the concentration of As in the sample is 534 μg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(0.2672\ \text{g})\ (100\ \mu\text{g/g})}{(50\ \text{mL})\ (1\ \text{L}/1000\ \text{mL})} = 534\ \mu\text{g/L}$$

9. Proper Waste Disposal Practices

9.1. Solid Waste

9.1.1. Solid waste contaminated with hazardous chemicals above the level of concern is not generated during the routine preparation of QC samples for analysis for inorganic contaminants.

9.1.2. If a solid waste is generated that may be contaminated at a level of concern, the appropriate paperwork is completed on a case-by-case basis.

9.1.2.1. A Waste Profile Request Form (WPRF) describing the solid waste is completed and sent to the Waste Management Group for review.

9.1.2.2. If the Waste Management Group determines that the solid waste is contaminated below the level of concern, the waste is disposed of as ordinary laboratory trash.

9.1.2.3. If the Waste Management Group determines that the solid waste is contaminated above the level of concern, pickup of the contaminated waste is requested as described in Sec. 9.3.

9.2. Liquid Waste

9.2.1. Liquid waste containing hazardous contaminants, such as solutions of trace metals, is accumulated in a coated glass bottle in a secondary containment tray and kept segregated from liquid waste contaminated with organic or radioactive substances or cyanide.

9.2.2. The bottle is labeled with a hazardous waste label and a label indicating its use for trace-metal waste.

9.2.3. The bottle is opened only for the period of time necessary to add waste to it.

9.2.4. When the bottle is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.5. A new bottle is labeled for further use.

9.3. Waste Pickup

9.3.1. Pickup by the Waste Management Group of solid waste or a full bottle of liquid waste containing trace metals is requested using the current Chemical Waste Disposal Request form (CWDR). The current Waste Profile Request Form (WPRF) that describes the waste is referenced on the CWDR.

9.3.2. The Waste Management Group picks up the waste for disposal in accord with Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1988," Los Alamos National Laboratory report LA-11637-MS (1989).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.3. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.4. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒▒). Where a section heading is marked, the entire section has been revised

TRACE ELEMENTS IN NITRIC ACID — QC SAMPLE PREPARATION

Analyte: Metals	**Method No.:** QCI170
Matrix: Water, soil	**Accuracy and Precision:** 100% ± 4.3% RSD
Procedure: ICPMS, ICPAES, FAA, ETVAA, or CVAA, depending on metal and concentration range	**Spike Range:** 1 μg/L-90 mg/L
Effective Date: 04/01/88	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Section 5. Read Section 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.5 for proper waste disposal practices.

1. Principle of Method

1.1. Acidified water is spiked with standard solutions of metals.

1.2. An acidified water QC sample represents the solution resulting from the extraction of the metals from a soil matrix and is analyzed from that point using the same analytical procedure applied to the unknown samples.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed using the same analytical procedure or applicable sections of the procedure.

1.4. Methods of analysis depend on the metal and its concentration. They include inductively coupled plasma mass spectrometry (ICPMS), inductively coupled plasma atomic emission spectrometry (ICPAES), flame atomic absorption (FAA) spectroscopy, electrothermal vaporization atomic absorption (ETVAA) spectroscopy, and cold vapor atomic absorption (CVAA) spectroscopy.

1.5. The QC spike range depends on the metal of interest and the method of analysis. Refer to the following table for spike ranges.

SPIKE RANGES FOR TRACE ELEMENTS IN NITRIC ACID					
Analyte	ICPMS mg/L	ICPES mg/L	FAA mg/L	ETVAA mg/L	CVAA mg/L
Ag	0.005-0.05	0.3-6	0.25-1.0		
Al	0.1-0.5	1-20	5-10		
As	0.005-0.05	1.5-30		0.01-0.06	
Au		0.2-5			
B	0.01-0.09	0.2-5	0.1-6	0.1-6.0	
Ba	0.005-0.05	0.2-5	2.0-8.0		
Be	0.005-0.05	0.2-5	0.5-1.5	0.01-0.04	
Ca		0.6-13	5-10		
Cd	0.005-0.05	0.2-3	0.5-1.5	0.001-0.005	
Ce		1.5-30			
Co	0.025-0.05	0.2-5	0.5-1.5		
Cr	0.005-0.05	0.3-6	0.5-1.5	0.01-0.06	
Cu	0.005-0.2	0.2-3	0.5-1.5		
Fe	0.1-0.25	0.2-3	0.5-1.5	0.01-0.04	
Hg					0.001-0.004
K		9-90	1-5		
Li	0.05-0.2	0.2-3	0.2-1		
Mg		2-30	1-5		
Mn	0.005-0.05	0.05-1	0.5-1.5		
Mo	0.005-0.05	0.2-5	3-8		
Na		1-20	3-7.5		
Ni	0.03-0.1	0.4-7	0.5-1.5		

SPIKE RANGES FOR TRACE ELEMENTS IN NITRIC ACID (cont)					
Analyte	ICPMS mg/L	ICPES mg/L	FAA mg/L	ETVAA mg/L	CVAA mg/L
Pb	0.005-0.02	2-30	0.5-1.5	0.01-0.06	
Re		5-30			
S		6-60			
Sb	0.005-0.05	2-30	0.5-1.5	0.01-0.05	
Sc		0.05-1			
Se	0.05-0.1	2-30		0.01-0.06	
Si		0.5-10			
Sn		2-30	0.1-1	0.01-0.06	
Sr	0.1-0.5	0.02-0.5			
Ta	0.1-0.5	0.1-2.0	0.5-1.5		
Th	0.005-0.05				
Tl	0.005-0.02	2-30	0.5-1.5	0.01-0.06	
U	0.005-0.05				
U-235/238	0.05-0.1				
V	0.03-0.1	0.2-5		0.01-0.06	
Y		0.05-1			
Zn	0.01-0.2	0.2-3	0.5-1.5		

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and the error associated with the use of the pipette and the volumetric flask. A relative standard deviation (RSD) of 4.3% at the 1σ level has been calculated for the spiking and dilution process based on the following functions.

2.1.1. One dilution of the standard.

2.1.1.1. Use the following function for standards requiring a single dilution.

$$U = \frac{M_1 \times M_2}{M_3} ,$$

where U = uncertainty,
M_1 = standard error,
M_2 = spiking error, and
M_3 = dilution error.

2.1.1.2. Calculate relative variance for each variable using the following equation:

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.1.3. Calculate percent relative standard deviation using the following equation:

$$S_{D_T} = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of volumetric flask, and
100 = factor to convert to percentage.

2.1.2. Two dilutions of the standard.

2.1.2.1. Use the following function for standards requiring a second dilution.

$$U = \frac{M_1 \times M_2}{M_3} \times \frac{M_4}{M_5} ,$$

where U = uncertainty of standard,
M_1 = standard error,
M_2 = first spiking error,
M_3 = first dilution error,
M_4 = second spiking error, and
M_5 = second dilution error.

2.1.2.2. Calculate relative variance for each variable using the following equation:

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where $R^2{}_M$ = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2.3. Calculate relative standard deviation using the following equation:

$$S_{D_T} = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2 + R_{M4}^2 + R_{M5}^2} \times 100 ,$$

where SD_T = relative standard deviation (% error),
$R^2{}_{M1}$ = relative variance of standard,
$R^2{}_{M2}$ = relative variance of first pipette,
$R^2{}_{M3}$ = relative variance of first volumetric flask,
$R^2{}_{M4}$ = relative variance of second pipette,
$R^2{}_{M5}$ = relative variance of second volumetric flask, and
100 = factor to convert to percentage.

2.2. All variables contributing to the error associated with the preparation of these QC samples were considered. The largest errors were used in the error propagation calculations.

2.3. Analytical results from QC samples prepared between 1990 and 1991 using this procedure and analyzed by ICPMS, ICPAES, FAA, ETVAA, or CVAA are tabulated below. The data are published in Source Materials 10.1 through 10.3.

SPIKE RECOVERY DATA FOR TRACE ELEMENTS IN NITRIC ACID

Analyte	1990 Mean ± SD (%)	N[a]	1991 Mean ± SD (%)	N[a]
Ag	99 ± 15	(65)	99 ± 9	(167)
Al	109 ± 26	(126)	104 ± 16	(42)
As	97 ± 18	(67)	102 ± 10	(119)
Au	84 ± 0	(1)	218 ± 0	(1)
B	98 ± 16	(77)	102 ± 5	(30)
Ba	105 ± 16	(73)	101 ± 12	(159)
Be	102 ± 18	(102)	96 ± 13	(82)
Ca	114 ± 16	(14)	113 ± 9	(14)
Cd	99 ± 8	(72)	102 ± 9	(171)
Ce	—	—	—	—
Co	109 ± 37	(39)	106 ± 8	(37)
Cr	104 ± 13	(60)	102 ± 7	(179)
Cu	107 ± 36	(95)	101 ± 7	(82)
Fe	101 ± 40	(40)	103 ± 7	(54)
Hg	99 ± 13	(167)	98 ± 12	(83)
K	99 ± 7	(13)	102 ± 7	(14)
Li	99 ± 11	(10)	130 ± 8	(5)
Mg	103 ± 6	(31)	105 ± 7	(19)
Mn	106 ± 29	(60)	104 ± 22	(37)
Mo	101 ± 18	(61)	103 ± 10	(34)
Na	107 ± 10	(21)	109 ± 15	(23)
Ni	104 ± 26	(128)	101 ± 10	(88)
Pb	105 ± 32	(70)	102 ± 7	(199)
Re	—	—	—	—
S	115 ± 13	(15)	113 ± 14	(16)
Sb	101 ± 16	(111)	98 ± 10	(49)
Sc	—	—	—	—
Se	100 ± 8	(74)	100 ± 10	(134)
Si	100 ± 4	(71)	100 ± 4	(48)
Sr	98 ± 5	(61)	100 ± 9	(12)
Ta	—	—	—	—
Th	—	—	104 ± 9	(21)
Tl	105 ± 23	(100)	102 ± 9	(59)
U	98 ± 11	(262)	100 ± 11	(160)
U-235/238	100 ± 6	(119)	100 ± 5	(85)
V	102 ± 20	(40)	103 ± 11	(25)
Y	—	—	—	—
Zn	104 ± 39	(79)	103 ± 11	(57)

[a]N = number of samples.

3. Collection and Storage of Samples

3.1. Mercury standard samples must be stored in glass containers with $Cr_2O_7^=$ added as a stabilizer. Refer to QC Method No. QCI200 for the preparation and storage of Hg standard samples.

3.2. If the QC sample contains Ag, it must be protected from light to prevent reduction and subsequent loss of Ag. Store the sample at room temperature in an amber high-density polyethylene bottle or in an uncolored high-density polyethylene bottle completely wrapped in aluminum foil.

3.3. If any of the metal standard solutions contains HCl, it may not be added to a solution containing Ag because the Ag will be lost as insoluble AgCl.

3.4. QC samples are prepared in a volumetric flask and are aliquoted into high-density polyethylene bottles for storage at room temperature pending analysis.

4. Apparatus

4.1. Balance: 100-g minimum capacity, 0.1-mg accuracy.

4.2. Spatula.

4.3. Platinum crucibles: approximately 50-mL capacity, with platinum covers. Each crucible should have a unique identifying number stamped or etched into it.

4.4. Desiccator.

4.5. Beaker: 50-mL, glass.

4.6. Volumetric flasks: 10-, 50-, 100-, and 500-mL, class A, glass.

4.7. Graduated cylinders: 10- and 25-mL.

4.8. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.9. Pipette tips: disposable.

4.10. Volumetric pipettes: 2-, 3-, 4-, 5-, 6-, 7-, 8-, 9-, and 10-mL, class A, glass.

4.11 Bottles: 125- and 250-mL, high-density polyethylene.

4.12. Aluminum foil.

4.13. Bottles: 125-mL, amber, high-density polyethylene.

4.14. Labels printed with QC sample numbers.

5. Reagents

5.1. Nitric acid (16 M, low-temperature redistilled).

5.2. Water. Double-deionized, 10-MΩ/cm or greater resistivity, specified in document ASTM-D1193 as ASTM Type I water and referred to in this procedure as double-deionized water.

5.3. Potassium dichromate (reagent-grade).

6. Calibration and Standards

6.1. Spectroscopy-grade metal standards (100-10,000 μg/mL). Spex Industries, Inc., 3880 Park Avenue, Edison, NJ 08820, or equivalent.

6.2. If a more dilute standard is needed to reach the desired spike range, dilute a concentrated standard, taking care to match the kind and concentration of acid in the dilution to those in the concentrated standard.

6.3. Prepare a standard for the determination of Cr in the 6+ oxidation state by dissolving reagent-grade $K_2Cr_2O_7$ in double-deionized water. The Cr^{6+} concentration of the standard should be 1000 μg/mL.

6.3.1. Tare a 50-mL volumetric flask on the balance.

6.3.2. Accurately weigh 141.5 ± 0.2 mg of analytical-grade $K_2Cr_2O_7$ into the flask.

6.3.3. Dissolve the salt and dilute the standard to volume with double-deionized water.

6.3.4. Calculate the Cr^{6+} concentration of the standard as in the example in Step 8.1.

6.4. Prepare a standard for the determination of total uranium and $^{235}U/^{238}U$ isotopic ratios through ICPMS by dissolving a certified natural U_3O_8 standard, such as National Institute for Standards and Technology (NIST) Standard Reference Material (SRM) 950b, in 7.8 M HNO_3. The uranium concentration of the standard should be 1 mg/mL. The $^{235}U/^{238}U$ isotopic ratio of natural uranium is 0.0072.

6.4.1. Use low-temperature redistilled HNO_3 and double-deionized water to prepare the uranium standard solutions. Prepare 7.8 M HNO_3 by diluting 25 mL of concentrated HNO_3 to 50 mL with double-deionized water. Prepare 1.6 M HNO_3 by diluting 1 mL of concentrated HNO_3 to 10 mL with double-deionized water. Prepare 1% HNO_3 by diluting 5 mL of concentrated HNO_3 to 500 mL with double-deionized water.

6.4.2. Weigh 1 g of a certified natural U_3O_8 into a platinum crucible. Cover the crucible with the platinum lid.

6.4.3. Heat the U_3O_8 in a muffle furnace at 900°C for 1 h.

6.4.4. Cool the U_3O_8 in a desiccator overnight.

6.4.5. Accurately weigh 590 ± 1 mg of the U_3O_8 into a 50-mL beaker.

6.4.6. With gentle heating, dissolve the U_3O_8 in 20 mL of 7.8 M low-temperature redistilled HNO_3.

6.4.7. Evaporate the solution to near dryness.

6.4.8. Redissolve the residue in 10 mL of 1.6 M HNO_3.

6.4.9. Quantitatively transfer the uranium solution into a 500-mL volumetric flask with 1% HNO_3 and dilute the solution to volume with 1% HNO_3.

6.4.10. Calculate the concentration of the resultant natural uranium standard as in the example in Step 8.2.

6.5. Prepare a uranium standard with a concentration of 100 μg/mL by diluting the 1-mg/mL uranium standard. Use double-deionized water and low-temperature redistilled HNO_3.

6.5.1. Place approximately 70 mL of double-deionized water into a 100-mL volumetric flask.

6.5.2. Add 1 mL of concentrated HNO_3 to the flask to achieve a final acid concentration of 1%.

6.5.3. Add 10 mL of the 1-mg/mL natural uranium standard to the volumetric flask using a volumetric pipette.

6.5.4. Dilute the solution to volume with double-deionized water.

6.5.5. Calculate the concentration of the resultant natural uranium standard. The $^{235}U/^{238}U$ isotopic ratio in the standard is 0.0072.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the number of samples to be analyzed. Periodically omit one or more metals from the acidified standard as a blank QC for those metals.

7.2. This procedure describes the preparation of five 100-mL aliquots of multielement standard, five 100-mL aliquots of silver standard, and two 250-mL aliquots of natural uranium standard. QC samples may be prepared in larger or smaller quantities than described in this procedure, adjusting apparatus size as necessary.

7.3. Place approximately 400 mL of double-deionized water in a 500-mL volumetric flask.

7.4. Add 15 mL of 16 M low-temperature redistilled nitric acid to the water to produce a final acid concentration of 3% by volume.

7.5. Use a new disposable pipette tip to spike each metal standard solution into the QC sample to avoid contamination of the standards. Spike an aliquot of each metal standard solution into the acidified water in the flask to reach the desired spike range for that metal.

NOTE: If any of the metal standard solutions used to prepare the QC sample contains HCl, do not include Ag in the sample because it will be lost as insoluble AgCl. Prepare a separate QC sample for Ag.

7.6. Dilute the QC sample to volume with double-deionized water. Mix by inversion.

7.7. Divide the QC sample evenly among five 125-mL high-density polyethylene bottles. If Ag has been included in the sample, use amber 125-mL high-density polyethylene bottles or uncolored bottles completely wrapped in aluminum foil.

7.8. Label each bottle with a QC sample number and store at room temperature pending analysis. The same QC number may be used for all aliquots of a QC sample from the same 500-mL volumetric flask.

7.9. Preparing a separate Ag QC sample.

7.9.1. Place approximately 400 mL of double-deionized water into a 500-mL volumetric flask.

7.9.2. Add 15 mL of 16 M low-temperature redistilled nitric acid to produce a final acid concentration of 3% by volume.

7.9.3. Spike an aliquot of Ag standard solution into the acidified water in the flask to reach the desired spike range.

7.9.4. Dilute the QC sample to volume with double-deionized water. Mix by inversion.

7.9.5. Divide the QC sample evenly between five 100-mL, amber, high-density polyethylene bottles or five 100-mL, uncolored, high-density polyethylene bottles. The uncolored bottles must be completely wrapped in aluminum foil to prevent loss of Ag.

7.9.6. Label each bottle with a QC sample number and store at room temperature pending analysis. The same QC number may be used for all aliquots of a QC sample from the same 500-mL volumetric flask.

7.10. For the determination of total uranium and $^{235}U/^{238}U$ isotopic ratios through ICPMS, prepare a QC sample from the natural uranium standard made from a certified natural U_3O_8 material.

7.10.1. Place approximately 400 mL of double-deionized water into a 500-mL volumetric flask.

7.10.2. Add 15 mL of low-temperature redistilled nitric acid to produce a final acid concentration of 3% by volume.

7.10.3. Spike an aliquot of 100-μg/mL natural uranium standard into the acidified water in the flask to reach the desired spike range for total uranium.

7.10.4. Dilute the QC sample to volume with double-deionized water. Mix by inversion.

7.10.5. Divide the QC sample evenly between two 250-mL high-density polyethylene bottles.

7.10.6. Label each bottle with a QC number and store at room temperature pending analysis. The same QC number may be used for both aliquots of a QC sample from the same 500-mL volumetric flask.

7.11. Record QC standard information, sample number, sample description, spike amount, concentration, isotopic ratio, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Calculate the concentration of the Cr^{6+} standard solution using the following atomic weights and example.

8.1.1. Atomic weight of K = 39.1
Atomic weight of Cr = 52.0
Atomic weight of O = 16.0

8.1.2. If 141.5 mg of $K_2Cr_2O_7$ is dissolved in 50 mL of double-deoionized water, the Cr^{6+} concentration in the standard is 1000 μg/mL.

$$\frac{\left(\frac{104\ g\ Cr^{6+}}{294.2\ g\ K_2Cr_2O_7}\right)(141.5\ mg\ K_2Cr_2O_7)\left(\frac{1000\ \mu g}{1\ mg}\right)}{50\ mL} = 1000\ \mu g\ \frac{Cr^{6+}}{mL}$$

8.2. Calculate the concentration of the uranium standard solution using the following atomic weights and example.

8.2.1. Atomic weight of U = 238.029
Atomic weight of O = 16
You may also use the molecular weight of U_3O_8 recorded on the bottle label for NIST SRM U_3O_8.

8.2.2. The purity of the NIST U_8O_8 is recorded on the certificate for the standard reference material used.

8.2.3. If 589.6 mg of 99.968% pure U_3O_8, which has been heated in the muffle furnace, is dissolved in acid and diluted to 500 mL, the uranium concentration in the standard will be 1.0 g/L.

$$\frac{\left(\frac{714.087\ g\ U}{842.0822\ g\ U_3O_8}\right)(0.99968)\ (0.5896\ g)}{(500\ mL)\ (1\ L/1000\ mL)} = 1.0\ g/L$$

8.2.4. The $^{235}U/^{238}U$ isotopic ratio in natural uranium is 0.0072 ± 0.0007.

8.3. Record the concentration of each metal in the QC sample as μg/L or mg/L.

8.4. Calculate spike concentrations for each metal in the QC sample as in the following example:

8.4.1. If 250 μL of a 100-μg/mL Se standard is used to spike 400 mL of acidified water and the solution is diluted to 500 mL, the concentration of Se in the sample is 50 μg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(100\ \mu g/mL)\left(\frac{1\ mL}{1000\ \mu L}\right)(250\ \mu L)}{(500\ mL)\ (1\ L/1000\ mL)} = 50\ \mu g/L$$

8.4.2. If 450 μL of 1000-μg/mL Ag standard is used to spike 400 mL of acidified water and the solution is diluted to 500 mL, the concentration of Ag in the sample is 900 μg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(1000\ \mu g/mL)\left(\frac{1\ mL}{1000\ \mu L}\right)(450\ \mu L)}{(500\ mL)\ (1\ L/1000\ mL)} = 900\ \mu g/L$$

8.4.3. If 400 μL of 100-μg/mL natural uranium standard is used to spike 400 mL of acidified water and the solution is diluted to 500 mL, the concentration of U in the sample is 80 μg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(100\ \mu g/mL)\left(\frac{1\ mL}{1000\ \mu L}\right)(400\mu L)}{(500\ mL)\ (1\ L/1000\ mL)} = 80\ \mu g/L$$

The $^{235}U/^{238}U$ isotopic ratio in natural uranium is 0.0072 ± 0.0007.

9. Proper Waste Disposal Practices

9.1. Solid Waste

9.1.1. Solid waste contaminated with hazardous chemicals above the level of concern is not generated during the routine preparation of QC samples for analysis for inorganic contaminants.

9.1.2. If a solid waste is generated that may be contaminated at a level of concern, the appropriate paperwork is completed on a case-by-case basis.

9.1.2.1. A Waste Profile Request Form (WPRF) describing the solid waste is completed and sent to the Waste Management group for review.

9.1.2.2. If the Waste Management group determines that the solid waste is contaminated below the level of concern, the waste is disposed of as ordinary laboratory trash.

9.1.2.3. If the Waste Management group determines that the solid waste is contaminated above the level of concern, pickup of the contaminated waste is requested as described in Sec. 9.3.

9.2. Liquid Waste

9.2.1. Liquid waste containing hazardous contaminants, such as solutions of trace metals, is accumulated in a coated glass bottle in a secondary containment tray and is kept segregated from liquid waste contaminated with organic or radioactive substances or cyanide.

9.2.2. The bottle is labeled with a hazardous waste label and a label indicating its use for trace-metal waste.

9.2.3. The bottle is opened only for the period of time necessary to add waste to it.

9.2.4. When the bottle is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.5. A new bottle is labeled for further use.

9.3. Waste Pickup

9.3.1. Pickup by the Waste Management Group of solid waste or a full bottle of liquid waste containing trace metals is requested using the current Chemical Waste Disposal Request form (CWDR). The current Waste Profile Request Form (WPRF) that describes the waste is referenced on the CWDR.

9.3.2. The Waste Management Group picks up the waste for disposal in accord with Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1988," Los Alamos National Laboratory report LA-11637-MS (1989).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.3. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.4. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 950b, Uranium Oxide (U_3O_8)" (National Institute of Standards and Technology, Washington, DC, March 1978).

10.5. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒▒). Where a section heading is marked, the entire section has been revised.

TRACE ELEMENTS IN HYDROCHLORIC ACID — QC SAMPLE PREPARATION

Analyte: Metals	**Method No.:** QCI180
Matrix: Water, soil	**Spike Range:** Dependent on element and analytical method (see Step 1.5)
Procedure: ICPAES, FAA, or ETVAA, depending on metal and concentration range	**Accuracy and Precision:** 100% ± 5% RSD
Effective Date: 01/17/91	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.4 for proper waste disposal practices.

1. Principle of Method

1.1. Acidified water is spiked with standard solutions of metals. Tin and titanium are more stable in hydrochloric acid than in nitric acid.

1.2. The acidified water QC sample represents the solution resulting from the extraction of the metals from a soil matrix.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed from the extraction step in the same analytical procedure used for unknown samples.

1.4. The method of analysis depends on the metal and its concentration. The methods include inductively coupled plasma atomic emission spectrometry (ICPAES), flame atomic absorption (FAA) spectroscopy, and electrothermal vaporization atomic absorption (ETVAA) spectroscopy. QC samples of trace elements in hydrochloric acid are not run by inductively coupled plasma mass spectrometry (ICPMS) because the hydrochloric acid interferes.

1.5. The QC spike range depends on the metal of interest and on the method of analysis. Refer to the following table for spike ranges.

SPIKE RANGES FOR TRACE ELEMENTS IN HYDROCHLORIC ACID			
Element	ICPAES (mg/L)	FAA (mg/L)	ETVAA (mg/L)
Sn	2-30	0.1-1	0.010-0.060
Ti	0.1-2	2-5	0.005-0.050

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with the use of the pipette and of the volumetric flask. A relative standard deviation (RSD) of 5.0% at the 1σ level has been calculated for standard preparation and for the spiking and dilution process based on the following function:

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated result,
M1 = standard error,
M2 = spiking error, and
M3 = dilution error.

2.1.1. Calculate relative variance for each variable using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3}} \times 100 \quad ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of volumetric flask, and
100 = factor to convert to percentage.

2.1.3. All variables contributing to the error associated with the preparation of these QC samples were considered. The largest errors were used in the error propagation calculations.

2.2. Analytical results from QC samples prepared between 1990 and 1991 using this procedure and analyzed by ICPAES, FAA, and ETVAA are tabulated below. The data are published in Source Materials 10.1 through 10.3.

SPIKE RECOVERY DATA FOR TRACE ELEMENTS IN HCL

Analyte	1990 Mean ± SD (%)	N[a]	1991 Mean ± SD (%)	N[a]
Sn	104 ± 7	(8)	91 ± 11	(13)
Ti	109 ± 34	(45)	105 ± 17	(22)

[a]N = number of samples.

3. Collection and Storage of Samples

3.1. QC samples are prepared in a volumetric flask and are aliquoted into high-density polyethylene bottles for storage at room temperature pending analysis.

4. Apparatus

4.1. Flasks: 200-mL, glass, class A volumetric.

4.2. Graduated cylinder: 100-mL.

4.3. Auto-pipette: 100- and 1000-µL, Rainin or equivalent.

4.4. Pipette tips: disposable.

4.5. Pipettes: 2-, 3-, 4-, 5-, and 6-mL, glass, class A volumetric.

4.6. Bottles: 125-mL, high-density polyethylene.

4.7. Labels printed with QC sample numbers.

5. Reagents

5.1. Hydrochloric acid, 12 M, ultrapure, suitable for trace-metal analysis.

5.2. Water. Double-deionized, >10 MΩ/cm resistivity, specified in document ASTM-D1193 as ASTM Type I water and referred to in this procedure as double-deionized water.

6. Calibration and Standards

6.1. Spectroscopy-grade metal standards (100-10,000 µg/mL). Spex Industries, Inc., 3880 Park Avenue, Edison, NJ 08820, or equivalent.

6.2. If a more dilute standard is needed to reach the desired spike range, dilute a concentrated standard, taking care to match the type and concentration of acid in the dilution to those in the concentrated standard.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the number of samples to be analyzed. Periodically omit one or more metals from the acidified sample as a blank QC for those metals.

7.2. This procedure describes the preparation of two 100-mL aliquots of a QC sample. QC samples may be prepared in larger or smaller quantities than described in this procedure, adjusting apparatus size and reagent volume as necessary.

7.3. Place approximately 100 mL of double-deionized water into a 200-mL volumetric flask.

7.4. Add 40 mL of ultrapure hydrochloric acid to the water in the flask.

7.5. Use a new disposable pipette tip to spike each metal standard solution into the QC sample to avoid contamination of the standards. Spike an aliquot of each metal standard solution into the acidified water in the flask to reach the desired spike range for that metal.

7.6. Dilute the QC sample to volume with double-deionized water. Mix by inversion.

7.7. Divide the QC sample evenly between two 125-mL high-density polyethylene bottles.

7.8. Label each bottle with a QC sample number and store at room temperature pending analysis. The same QC number may be used for both aliquots of a QC sample from the same 200-mL volumetric flask.

8. Calculations

8.1. Record the concentration of each metal in the QC sample as μg/L or mg/L.

8.2. Calculate the spike concentration for each metal in the QC sample as in the following example.

8.2.1. If 3.0 mL of a 1000-μg/mL Sn standard is used to spike 180 mL of acidified water and the solution is diluted to 200 mL, the concentration of Sn in the sample is 15 mg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(1000\ \mu g/mL)\ (3.0\ mL)\ (1\ mg/1000\mu g)}{(200\ mL)\ (1\ L/1000\ mL)} = 15\ mg/L$$

9. Proper Waste Disposal Practices

9.1. Solid Waste

9.1.1. Solid waste contaminated with hazardous chemicals above the level of concern is not generated during the routine preparation of QC samples for analysis for inorganic contaminants.

9.1.2. If a solid waste is generated that may be contaminated at a level of concern, the appropriate paperwork is completed on a case-by-case basis.

9.1.2.1. A Waste Profile Request Form (WPRF) describing the solid waste is completed and sent to the Waste Management Group for review.

9.1.2.2. If the Waste Management Group determines that the solid waste is contaminated below the level of concern, the waste is disposed of as ordinary laboratory trash.

9.1.2.3. If the Waste Management Group determines that the solid waste is contaminated above the level of concern, pickup of the contaminated waste is requested as described in Sec. 9.3.

9.2. Liquid Waste

9.2.1. Liquid waste containing hazardous contaminants, such as solutions of trace metals, is accumulated in a coated glass bottle in a secondary containment tray and kept segregated from liquid waste contaminated with organic or radioactive substances or cyanide.

9.2.2. The bottle is labeled with a hazardous waste label and a label indicating its use for trace-metal waste.

9.2.3. The bottle is opened only for the period of time necessary to add waste to it.

9.2.4. When the bottle is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.5. A new bottle is labeled for further use.

9.3. Waste Pickup

9.3.1. Pickup by the Waste Management Group of solid waste or a full bottle of liquid waste containing trace metals is requested using the current Chemical Waste Disposal Request form (CWDR). The current Waste Profile Request Form (WPRF) that describes the waste is referenced on the CWDR.

9.3.2. The Waste Management Group picks up the waste for disposal in accord with Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1988," Los Alamos National Laboratory report LA-11637-MS (1989).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.3. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.4 "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒). Where a section heading is marked, the entire section has been revised.

TRACE ELEMENTS IN WATER — QC SAMPLE PREPARATION

Analytes: Boron, bromide, calcium, chloride, fluoride, lithium, magnesium, phosphorus, potassium, silicon, sodium, sulfate	**Method No.:** QCI190
Matrix: Water	**Spike Range:** Dependent on the element (see Table I)
Procedure: FIA, ACOLOR, ISE, or IC	**Accuracy and Precision:** 100% ± 3% RSD
Effective Date: 10/01/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.3 for proper waste disposal practices.

1. Principle of Method

1.1. Prepare individual standard solutions of bromide, chloride, fluoride, and sulfate salts in double-deionized water.

1.2. Obtain commercially prepared single-element solutions of boron, calcium, lithium, magnesium, phosphorus, potassium, silicon, and sodium.

1.3. Spike the chosen volume of each standard separately or in various combinations into double-deionized or distilled water as specified in Sec. 7.

1.4. Methods of analysis depend on the analyte and its concentration. They include flow injection analysis (FIA), ion-selective electrode (ISE), ion chromatography (IC), and colorimetry (ACOLOR).

1.5. The QC spike range depends on the analyte of interest and the analytical method. Refer to Table I for spike ranges.

TABLE I. SPIKE RANGES FOR TRACE ELEMENTS IN WATER

Analyte	Spike range (mg/L)	Analysis method
chloride (Cl)	5.0-50	FIA/IC
bromide (Br)	5.0-50	FIA/IC
fluoride (F)	0.1-2.0	ISE
sulfate (SO_4)	5.0-30	FIA/IC
phosphorus (P)	0.04-1.0	ACOLOR
boron (B)	0.1-6.0	FIA
silicon (Si)	3-50	FIA
silica (SiO_2)	5-100	FIA
sodium (Na)	5-30	IC
potassium (K)	3-20	IC
calcium (Ca)	5-30	IC
magnesium (Mg)	3-20	IC
lithium (Li)	1-10	IC

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with the use of the pipette and volumetric flask. A relative standard deviation (RSD) of 3% at the 1σ level has been calculated for the error of the standard and for the spiking and dilution process based on the following function:

$$D = \frac{M_1 \times M_2}{M_3} ,$$

where D = calculated result,
M_1 = standard error,
M_2 = spiking error, and
M_3 = dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2),
$\overline{X}$ = mean value of each variable (M), and
100 = factor to convert to percent.

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M2} = relative variance of standard,
R^2_{M2} = relative variance of pipette, and
R^2_{M3} = relative variance of volumetric flask.

2.1.3. If a second pipette is used to obtain the desired analyte concentration, use the following equation.

$$R_{M_2} = \frac{\sqrt{\sigma_1^2 + \sigma_2^2}}{P_1 + P_2} ,$$

where σ_1 = standard deviation of first pipette error,
σ_2 = standard deviation of second pipette error,
P_1 = mean of first pipette, and
P_2 = mean of second pipette.

2.2. Analytical results from QC samples prepared between 1990 and 1991 using this procedure and analyzed by FIA, ISE, ACOLOR, and IC are tabulated below. The data are published in Source Materials 10.1 and 10.2.

SPIKE RECOVERY DATA FOR TRACE ELEMENTS IN WATER

Analyte	1990 mean ± SD (%)	N[a]	1991 mean ± SD (%)	N[a]
Na	107 ± 10	(21)	109 ± 15	(23)
SO_4	115 ± 13	(15)	113 ± 14	(16)
Si	100 ± 3	(4)	103 ± 0	(1)
K	99 ± 7	(13)	102 ± 7	(14)
P	120 ± 120	(9)	120 ± 60	(14)
Mg	103 ± 6	(31)	105 ± 7	(19)
Li	99 ± 11	(10)	130 ± 8	(5)
F	105 ± 10	(20)	113 ± 16	(13)
Cl	116 ± 19	(18)	119 ± 23	(13)
Ca	114 ± 16	(14)	113 ± 9	(14)
Br	120 ± 34	(10)	100 ± 12	(4)
B	98 ± 16	(77)	102 ± 5	(30)

[a]N = number of samples.

3. Collection and Storage of Samples

3.1. QC samples for boron, fluoride, and silicon are prepared in polypropylene flasks to avoid interactions with borosilicate glass. All other samples may be made in glass flasks.

3.2. Spiked QC samples are aliquoted and stored in high-density polyethylene bottles at room temperature pending analysis.

4. Apparatus

4.1. Flasks: 50-, 250-, and 500-mL, class A volumetric, glass.

4.2. Flasks: 50-, 100-, and 500-mL, volumetric, polypropylene, Nalgene or equivalent.

4.3. Spatula.

4.4. Pipettes: 1- and 2-mL, class A volumetric, glass.

4.5. Auto-pipette: 100-μL and 1000-μL, Rainin or equivalent.

4.6. Pipette tips: disposable.

4.7. Bottles: 60-, 125-, and 250-mL, high-density polyethylene.

4.8. Balance: 100-g minimum capacity, 0.1-mg accuracy.

4.9. Labels printed with QC sample numbers.

5. Reagents

5.1. Water. Double-deionized, >10 MΩ/cm resistivity, specified in document ASTM-D1193 as ASTM Type I water and referred to in this procedure as double-deionized water.

5.2. Distilled water. Used to dilute silicon QC samples. Deionized water is not used because deionization may result in high levels of silicon when the resin begins to degrade, even though the resistivity of the water remains acceptable.

5.3. Potassium bromide (anhydrous, reagent-grade).

5.4. Sodium chloride (reagent-grade).

5.5. Sodium fluoride (reagent-grade).

5.6. Sodium sulfate (anhydrous, reagent-grade).

6. Calibration and Standards

6.1. Spectroscopy-grade metal standards (1000-10,000 μg/mL). Spex Industries, Inc., 3880 Park Avenue, Edison, NJ 08820, or equivalent.

6.2. If a more dilute standard is needed to reach the desired spike range, dilute a concentrated standard, taking care to match the kind and concentration of acid in the dilution to those in the concentrated standard. Use distilled water to make dilutions of a silicon standard.

6.3. Standard solutions for bromide, chloride, fluoride, and sulfate are made from salts.

6.4. Thoroughly rinse all glassware before preparing standards. The presence of nitric acid may oxidize bromide ion to bromine; it will also interfere with analysis by ion chromatography by giving a broad nitrate ion peak, which may obscure the differentation and quantitation of other anions present.

6.5. Prepare a standard solution of bromide ion at approximately 20 mg/mL using solid anhydrous potassium bromide.

6.5.1. Accurately weigh 1.5 ± 0.03 g of solid anhydrous potassium bromide into a 50-mL volumetric flask.

6.5.2. Add approximately 30 mL of double-deionized water and swirl the flask to dissolve the salt.

6.5.3. Dilute the solution to volume with double-deionized water and mix by inversion.

6.5.4. Transfer the solution to a 60-mL high-density polyethylene bottle and label the bottle with information identifying the standard.

6.5.5. Record information on standard preparation in the QC Laboratory notebook.

6.6. Prepare a standard solution of chloride ion at approximately 20 mg/mL using sodium chloride.

6.6.1. Dry 2 ± 0.1 g of solid sodium chloride at approximately 140°C for 30 minutes before using it to prepare the standard chloride solution.

6.6.2. Accurately weigh 1.65 ± 0.04 g of dried sodium chloride into a 50-mL volumetric flask.

6.6.3. Add approximately 30 mL of double-deionized water and swirl the flask to dissolve the salt.

6.6.4. Dilute the solution to volume with double-deionized water and mix by inversion.

6.6.5. Transfer the solution to a 60-mL high-density polyethylene bottle and label the bottle with information identifying the standard.

6.6.6. Record information on standard preparation in the QC Laboratory notebook.

6.7. Prepare a standard solution of fluoride ion at approximately 1000 μg/mL using solid sodium fluoride.

6.7.1. Make the fluoride standard in a polypropylene (Nalgene) volumetric flask to avoid reaction of the fluoride with borosilicate glass.

6.7.2. Accurately weigh 0.11 ± 0.005 g of solid anhydrous sodium fluoride into a 50-mL polypropylene volumetric flask.

6.7.3. Add approximately 30 mL of double-deionized water and swirl the flask to dissolve the salt.

6.7.4. Dilute the solution to volume with double-deionized water and mix by inversion.

6.7.5. Transfer the solution to a 60-mL high-density polyethylene bottle and label the bottle with information identifying the standard.

6.7.6. Record information on standard preparation in the QC Laboratory notebook.

6.8. Prepare a standard solution of sulfate ion at approximately 20 mg/mL using solid anhydrous sodium sulfate.

6.8.1. Accurately weigh 1.48 ± 0.03 g of solid anhydrous sodium sulfate into a 50-mL volumetric flask.

6.8.2. Add approximately 30 mL of double-deionized water and swirl the flask to dissolve the salt.

6.8.3. Dilute the solution to volume with double-deionized water and mix by inversion.

6.8.4. Transfer the solution to a 60-mL high-density polyethylene bottle and label the bottle with information identifying the standard.

6.8.5. Record information on standard preparation in the QC Laboratory notebook.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the number of samples to be analyzed. Periodically omit one or more analytes from the QC sample as a blank QC for those analytes.

7.2. QC samples may be prepared in larger or smaller quantities than described in this procedure, adjusting apparatus size as necessary.

7.3. This step describes the preparation of three approximately 150-mL aliquots of a standard containing bromide, chloride, fluoride, and sulfate anions.

7.3.1. Prepare QC samples containing fluoride in polypropylene (Nalgene) volumetric flasks to avoid loss of fluoride ion due to reaction with borosilicate glass.

7.3.2. Place approximately 400 mL of double-deionized water into a 500-mL polypropylene volumetric flask.

7.3.3. Spike aliquots of bromide, chloride, fluoride, and sulfate standards made from salts of the anions into the water to reach the desired spike range listed in Table I.

7.3.4. Dilute the QC sample to volume with double-deionized water. Mix by inversion.

7.3.5. Divide the QC sample evenly among three 250-mL high-density polyethylene bottles.

7.3.6. Label each bottle with a QC sample number and store at room temperature pending analysis. The same QC number may be used for all aliquots of a QC sample from the same 500-mL volumetric flask.

7.4. This step describes the preparation of four 25-mL aliquots of boron standard.

7.4.1. Prepare QC samples containing boron in polypropylene (Nalgene) volumetric flasks to prevent contamination by leaching of borosilicate glass surfaces.

7.4.2. Place approximately 80 mL of double-deionized water into a 100-mL polypropylene volumetric flask.

7.4.3. Add an aliquot of boron standard solution to the water to reach the desired spike range listed in Table I.

7.4.4. Dilute the QC sample to volume with double-deionized water. Mix by inversion.

7.4.5. Divide the QC sample evenly among four 60-mL high-density polyethylene bottles.

7.4.6. Label each bottle with a QC sample number and store at room temperature pending analysis. The same QC number may be used for all aliquots of a QC sample from the same 100-mL volumetric flask.

7.5. This step describes the preparation of five 100-mL aliquots of silicon standard.

7.5.1. Prepare QC samples containing silicon in polypropylene (Nalgene) volumetric flasks to prevent contamination by leaching of borosilicate glass surfaces.

7.5.2. Use distilled water to prepare silicon QC samples. Degrading ion-exchange resin used to make double-deionized water may contaminate the sample with silicon.

7.5.3. Place approximately 400 mL of distilled water into a 500-mL polypropylene volumetric flask.

7.5.4. Add an aliquot of silicon standard to the water to reach the desired spike range for silicon listed in Table I.

7.5.5. Dilute the QC sample to volume with distilled water. Mix by inversion.

7.5.6. Divide the QC sample evenly among five 125-mL high-density polyethylene bottles.

7.5.7. Label each bottle with a QC sample number and store at room temperature pending analysis. The same QC number may be used for all five aliquots of a QC sample from the same 500-mL volumetric flask.

7.6. This step describes the preparation of five 100-mL aliquots of phosphorus standard.

7.6.1. Place approximately 400 mL of double-deionized water into a 500-mL volumetric flask.

7.6.2. Add an aliquot of phosphorus standard solution to the water to reach the desired spike range listed in Table I.

7.6.3. Dilute the QC sample to volume with double-deionized water. Mix by inversion.

7.6.4. Divide the QC sample evenly among five 125-mL high-density polyethylene bottles.

7.6.5. Label each bottle with a QC sample number and store at room temperature pending analysis. The same QC number may be used for all aliquots of a QC sample from the same 500-mL volumetric flask.

7.7. This step describes the preparation of five 50-mL aliquots of a standard containing calcium, magnesium, potassium, sodium, and lithium.

7.7.1. Place approximately 200 mL of double-deionized water into a 250-mL volumetric flask.

7.7.2. Add aliquots of the Ca, Mg, K, Na, and Li standard solutions to the water to reach the desired spike range listed in Table I.

7.7.3. Dilute the QC sample to volume with double-deionized water. Mix by inversion.

7.7.4. Divide the QC sample evenly among five 60-mL high-density polyethylene bottles.

7.7.5. Label each bottle with a QC sample number and store at room temperature pending analysis. The same QC number may be used for all aliquots of a QC sample from the same 250-mL volumetric flask.

7.8. For each QC sample prepared, record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD data bases on the VAX. Record the concentration of the silicon QC sample in terms of both Si and SiO_2.

8. Calculations

8.1. Calculate the concentration of a bromide standard solution made from anhydrous potassium bromide using the following atomic weights and example.

8.1.1. Atomic weight of K = 39.1
Atomic weight of Br = 79.9

8.1.2. If 1.5000 g of KBr is dissolved in 50 mL of double-deionized water, the bromide concentration in the standard is 20 mg/mL.

$$\frac{\left(\frac{79.9\ \text{g Br}}{119.0\ \text{g KBr}}\right)(1.5000\ \text{g})\ (1000\ \text{mg/g})}{50\ \text{mL}} = 20\ \text{mg/mL}$$

8.2. Calculate the concentration of a chloride standard solution made from dried sodium chloride using the following atomic weights and example.

8.2.1. Atomic weight of Na = 23.0
Atomic weight of Cl = 35.5

8.2.2. If 1.6500 g of NaCl is dissolved in 50 mL of double-deionized water, the chloride concentration in the standard is 20 mg/L.

$$\frac{\left(\frac{35.5\ \text{g Cl}}{58.5\ \text{g NaCl}}\right)(1.6500\ \text{g})\ (1000\ \text{mg/g})}{50\ \text{mL}} = 20\ \text{mg/L}$$

8.3. Calculate the concentration of a fluoride standard solution made from sodium fluoride using the following atomic weights and example.

8.3.1. Atomic weight of Na = 23.0
Atomic weight of F = 19.0

8.3.2. If 0.1110 g of NaF is dissolved in 50 mL of double-deionized water, the fluoride concentration in the standard is 1004 μg/mL.

$$\frac{\left(\frac{19.0\ \text{g F}}{42.0\ \text{g NaF}}\right)(0.1110\ \text{g})\ (1{,}000{,}000\ \mu\text{g/g})}{50\ \text{mL}} = 1004\ \mu\text{g/mL}$$

8.4. Calculate the concentration of a sulfate standard solution made from anhydrous sodium sulfate using the following atomic weights and example.

8.4.1. Atomic weight of Na = 23.0
Atomic weight of S = 32.1
Atomic weight of O = 16.0

8.4.2. If 1.4750 g of Na_2SO_4 is dissolved in 50 mL of double-deionized water, the sulfate concentration in the standard is 20 mg/mL.

$$\frac{\left(\frac{96.1\ \text{g SO}_4}{142.1\ \text{g Na}_2\text{SO}_4}\right)(1.4750\ \text{g})\ (1000\ \text{mg/g})}{50\ \text{mL}} = 20\ \text{mg/mL}$$

8.5. Record the concentration of each analyte in a QC sample as μg/L or mg/L.

8.6. Calculate spike concentrations for each analyte in the QC sample as in the following examples.

8.6.1. If 750 μL of a 20-mg/mL Br standard is spiked into 400 mL of double-deionized water and the solution is diluted to 500 mL, the concentration

of Br in the QC sample is 30 mg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(20\ \text{mg/mL})\ (1\ \text{mL}/1000\ \mu\text{L})\ (750\ \mu\text{L})}{(500\ \text{mL})\ (1\ \text{L}/1000\ \text{mL})} = 30\ \text{mg/L}$$

8.6.2. If 300 μL of a 1000-μg/mL boron standard is spiked into 80 mL of double-deionized water and the solution is diluted to 100 mL, the concentration of boron in the QC sample is 3 mg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(1000\ \mu\text{g/mL})\left(\frac{1\ \text{mL}}{1000\ \mu\text{L}}\right)(300\mu\text{L})\left(\frac{1\ \text{mg}}{1000\ \mu\text{g}}\right)}{(100\ \text{mL})\ (1\ \text{L}/1000\ \text{mL})} = 3\ \text{mg/L}$$

8.6.3. If 900 μL of a 10,000-μg/mL Si standard is spiked into 400 mL of distilled water and the solution is diluted to 500 mL, the concentration of Si in the QC sample is 18 mg/L. The concentration of a Si QC sample is recorded in terms of both Si and SiO_2.

$$\frac{(10{,}000\ \mu\text{g/mL})\left(\frac{1\ \text{mL}}{1000\ \mu\text{L}}\right)(900\ \mu\text{L})\left(\frac{1\ \text{mg}}{1000\ \mu\text{g}}\right)}{(500\ \text{mL})\ (1\ \text{L}/1000\ \text{mL})} = 18\ \text{mg/L}$$

The concentration of the QC sample in terms of SiO_2 is calculated from the concentration in terms of Si.

A QC sample with a concentration of 18 mg/L Si has a concentration of 38 mg/L SiO_2.

$$(18\ \text{mg Si/L})\left(\frac{60.1\ \text{g}\ SiO_2}{28.1\ \text{g Si}}\right) = 38\ \text{mg}\ SiO_2/\text{L}$$

Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

8.6.4. If 250 μL of a 1000-μg/mL P standard is spiked into 400 mL of double-deionized water and the solution is diluted to 500 mL, the concentration of P in the QC sample is 500 μg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(1000\ \mu g/mL)\left(\frac{mL}{1000\ \mu L}\right)(250\ \mu L)}{(500\ mL)\ (1\ L/1000\ mL)} = 500\ \mu g/L$$

8.6.5. If 150 μL of a 10,000-μg/mL Mg standard is spiked into 200 mL of double-deionized water and the solution is diluted to 250 mL, the concentration of Mg in the QC sample is 6 mg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(10{,}000\ \mu g/mL)\left(\frac{1\ mL}{1000\ \mu L}\right)(150\ \mu L)\left(\frac{1\ mg}{1000\ \mu g}\right)}{(250\ mL)\ (1\ L/1000\ mL)} = 6\ mg/L$$

9. Proper Waste Disposal Practices

9.1. Solid Waste

9.1.1. Solid waste contaminated with hazardous chemicals above the level of concern is not generated during the routine preparation of QC samples for analysis for inorganic contaminants.

9.1.2. If a solid waste is generated that may be contaminated at a level of concern, the appropriate paperwork is completed on a case by case basis.

9.1.2.1. A Waste Profile Request Form (WPRF) describing the solid waste is completed and sent to the Waste Management Group for review.

9.1.2.2. If the Waste Management Group determines that the solid waste is contaminated below the level of concern, the waste is disposed of as ordinary laboratory trash.

9.1.2.3. If the Waste Management Group determines that the solid waste is contaminated above the level of concern, pickup of the contaminated waste is requested as described in Sec. 9.3.

9.2. Liquid Waste

9.2.1. Liquid waste containing hazardous contaminants, such as solutions of trace metals, is accumulated in a coated glass bottle in a secondary containment tray and kept segregated from liquid waste contaminated with organic or radioactive substances or cyanide.

9.2.2. The bottle is labeled with a hazardous waste label and a label indicating its use for trace-metal waste.

9.2.3. The bottle is opened only for the period of time necessary to add waste to it.

9.2.4. When the bottle is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.5. A new bottle is labeled for further use.

9.3. Waste Pickup

9.3.1. Pickup by the Waste Management Group of solid waste or a full bottle of liquid waste containing trace metals is requested using the current Chemical Waste Disposal Request form (CWDR). The current Waste Profile Request Form (WPRF) that describes the waste is referenced on the CWDR.

9.3.2. The Waste Management Group picks up the waste for disposal in accord with Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.3. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒). Where a section heading is marked, the entire section has been revised.

TRACE ELEMENTS FOR WATER AND SOIL (MERCURY BY TCLP) — QC SAMPLE PREPARATION

Analyte: Hg	**Method No.:** QCI200
Matrix: Water	**Spike Range:** 1-4 μg/L
Procedure: Toxicity Characteristic Leaching Procedure (TCLP) followed by cold vapor atomic absorption (CVAA) spectroscopy	**Accuracy and Precision:** 100% ± 5.9% RSD
Effective Date: 09/29/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.5 for proper waste disposal practices.

1. Principle of Method

1.1. Acidified water containing $Cr_2O_7^{=}$ as a stabilizer is spiked with a Hg standard.

1.2. The acidified water QC represents the solution resulting from the extraction of Hg from a soil matrix.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure or those sections of the procedure that are applicable.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the volumetric flask and of the pipette. A relative standard deviation (RSD) of 5.9% at the 1σ level has been calculated for the calibration, spiking, and dilution process based on the following function:

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of volumetric flask, and
100 = factor to convert to percentage.

2.2. Analytical results from QC samples prepared during 1989 and 1990 using this procedure and analyzed by cold vapor atomic absorption spectroscopy (N = 295) gave a mean recovery of 93% ± 9% at the 1σ level. The data are summarized below and are published in Source Materials 10.2 and 10.3.

MERCURY SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION

	Spike range µg/L	Mean ± SD (%)	Number of samples
1989	2.0-2.9	88 ± 7	91
	3.0-4.0	100 ± 3	78
1990	1.0-1.9	73 ± 20	6
	2.0-2.9	103 ± 1	24
	3-4.0	103 ± 2	96

3. Collection and Storage of Samples

3.1. Low-level Hg solutions must be stabilized to maintain their certified concentration longer than a few hours. Studies of the preservation of dilute Hg solutions (see Step 9.1) show that a dilute Hg solution will maintain its concentration for as long as five months if it is preserved with 0.01% $Cr_2O_7^{=}$ and 5% nitric acid and stored in a glass container.

3.2. QC samples are stored in glass bottles at room temperature pending analysis.

3.3. If QC samples for Hg are not used within five months of the date they were made, they are discarded.

4. Apparatus

4.1. Volumetric flasks: 5-, 25-, and 250-mL.

4.2. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.3. Pipette tips: disposable.

4.4. Graduated cylinder: 25-mL.

4.5. Bottles: 4-oz., glass, with Teflon-lined screw caps.

4.6. Balance: 100-g minimum capacity, 0.1-mg accuracy.

4.7. Spatula.

4.8. Weighing dish: small, polyethylene.

4.9. Labels printed with QC sample numbers.

5. Reagents

5.1. Nitric acid (16 M, ultrapure) suitable for trace metal analysis.

5.2. Water. In this procedure, water referred to as "reagent water" is understood to mean reagent water conforming to ASTM Types I, II, or III and free of interferences with the analytical method.

5.3. Potassium dichromate (analytical reagent-grade).

6. Calibration and Standards

6.1. Spectroscopy-grade Hg standard solution, 10-μg/mL concentration preferred. More concentrated standards, such as those supplied by Spex Industries, Inc., 3880 Park Avenue, Edison, NJ 08820, may be diluted to a Hg concentration of 10 μg/mL.

6.2. A spectroscopy-grade 10-μg/mL Hg standard solution is spiked into acidified water containing $K_2Cr_2O_7$ as a stabilizer for the Hg.

7. Procedure

7.1. Prepare a stabilizing solution for the Hg with a $Cr_2O_7^{=}$ concentration of 50 mg/mL.

7.1.1. Tare a small polyethylene weighing dish on the balance.

7.1.2. Accurately weigh 341 ± 2 mg of analytical-grade $K_2Cr_2O_7$ into the weighing dish.

7.1.3. Use reagent water to quantitatively rinse the $K_2Cr_2O_7$ into a 5-mL volumetric flask and to dilute the solution to volume. Mix by inversion.

7.1.4. Calculate the $Cr_2O_7^{=}$ concentration of the stabilizing solution as in the example in Step 8.1.

7.2. Prepare a 10-μg/mL Hg standard solution from a 1000-μg/mL standard solution.

7.2.1. Place 15 mL of reagent water into a 25-mL volumetric flask.

7.2.2. Add 2.5 mL of ultrapure nitric acid to the water in the flask to produce a final acid concentration of 10% by volume.

7.2.3. Add 50 μL of the stabilizing solution with a $Cr_2O_7^{=}$ concentration of 50 mg/mL to the acidified water to produce a $Cr_2O_7^{=}$ concentration of 0.01% by weight.

CAUTION: The nitric acid and $Cr_2O_7^{=}$ stabilizing solution must be added to the water before the mercury spike to prevent the immediate loss of Hg.

7.2.4. Add 250 μL of a 1000-μg/mL Hg standard solution to the water in the flask.

7.2.5. Dilute to volume with reagent water. Mix by inversion.

7.3. Prepare QC samples to equal at least 10% of the number of samples to be analyzed. Periodically include a sample of 5% HNO_3 and 0.01% $Cr_2O_7^=$ as a blank.

7.4. This procedure describes the preparation of two approximately 100-mL aliquots of a QC sample. QC solutions may be prepared in larger or smaller quantities than described in this procedure. Adjust apparatus size as necessary.

7.5. Place approximately 200 mL of reagent water into a 250-mL volumetric flask.

7.6. Add 12.5 mL of ultrapure nitric acid to the water to produce a final acid concentration of 5% by volume.

7.7. Add 500 μL of the stabilizing solution with a $Cr_2O_7^=$ concentration of 50 mg/mL to the acidified water to produce a $Cr_2O_7^=$ concentration of 0.01% by weight.

NOTE: The nitric acid and $Cr_2O_7^=$ stabilizing solution must be added to the water before the mercury spike to prevent the immediate loss of Hg.

7.8. Spike an aliquot of the 10-μg/mL Hg standard into the acidified water to reach the desired spike range.

7.9. Dilute the QC sample to volume with reagent water. Mix by inversion.

7.10. Divide the QC sample evenly between two 4-oz. glass bottles.

7.11. Label each bottle with a QC sample number and store at room temperature pending analysis. The same QC number may be used for both aliquots of a QC sample from the same 250-mL volumetric flask.

7.12. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Calculate the $Cr_2O_7^=$ concentration of the stabilizing solution using the following atomic weights and example.

8.1.1. Atomic weights: K = 39.1, Cr = 52.0, and O = 16.0.

8.1.2. If 341.0 mg of $K_2Cr_2O_7$ is dissolved in 5 mL of reagent water, the $Cr_2O_7^=$ concentration in the stabilizing solution is 50 mg/mL.

$$\frac{\left(\frac{216.0 \text{ g } Cr_2O_7^{=}}{294.2 \text{ g } K_2Cr_2O_7}\right)(341.0 \text{ mg } K_2Cr_2O_7)}{5 \text{ mL}} = 50 \text{ mg } Cr_2O_7^{=}/\text{mL}$$

8.2. Calculate Hg spike concentrations as in the following example.

8.2.1. If 50 μL of a 10-μg/mL Hg standard is diluted to 250 mL in acidified water containing $Cr_2O_7^{=}$ stabilizing solution, the Hg concentration in the sample is 2 μg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(10 \ \mu\text{g/mL})\ (1 \text{ mL}/1000 \ \mu\text{L})\ (50 \ \mu\text{L})}{(250 \text{ mL})\ (1 \text{ L}/1000 \text{ mL})} = 2 \ \mu\text{g/L}$$

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Solid waste contaminated with hazardous chemicals above the level of concern is not normally generated during the routine preparation of QC samples for analysis for inorganic contaminants.

9.1.2. If a solid waste is generated that may be contaminated at a level of concern, complete the appropriate paperwork on a case-by-case basis.

9.1.2.1. Complete a Waste Profile Request Form (WPRF) describing the solid waste and send to the Waste Management group for review.

9.1.2.2. If the Waste Management group determines that the solid waste is contaminated below the level of concern, dispose of the waste as ordinary laboratory trash.

9.1.2.3. If the Waste Management group determines that the solid waste is contaminated above the level of concern, request pickup of the contaminated waste as described in Sec. 9.3.

9.2. Liquid waste.

9.2.1. Accumulate liquid waste containing hazardous contaminants, such as solutions of trace metals, in a coated glass bottle in a secondary containment tray. Keep it segregated from liquid waste contaminated with organic or radioactive substances or cyanide.

9.2.2. Label the bottle with a hazardous waste label and a label indicating its use for trace metal waste.

9.2.3. Open the bottle only for the period of time necessary to add waste to it.

9.2.4. When the bottle is full, cap it and keep it in the secondary containment tray pending pickup by the Waste Management group.

9.2.5. Label a new bottle for further use.

9.3. Waste pickup.

9.3.1. Request pickup by the Waste Management group of solid waste or a full bottle of liquid waste containing trace metals using the current Chemical Waste Disposal Request form (CWDR). The current Waste Profile Request Form (WPRF) which describes the waste is referenced on the CWDR.

9.3.2. The Waste Management group picks up the waste for disposal in accord with Laboratory policy.

10. Source Materials

10.1. C. Feldman, "Preservation of Dilute Mercury Solutions," *Anal. Chem.* **46**, 99-102 (1974).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.3. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.4. SPEX Industries, Inc., "Certificate of Analysis for Metals" (SPEX Industries, Inc., Edison, NJ 08820, October 1990).

10.5. "Hazardous and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.6 ASTM, *1992 Annual Book of ASTM Standards*, Vol. 11.01, specification D1193-91, Standard Specification for Reagent Water, pp. 45-47.

Revisions or additions to the procedure are marked (▒▒▒). Where a section heading is marked, the entire section has been revised.

TRACE ELEMENTS IN WATER (CN) – QC SAMPLE PREPARATION

Analyte: CN	**Method No.:** QCI210
Matrix: Water	**Spike Range:** 0.1-0.5 mg/L
Procedure: Automated colorimetry	**Accuracy and Precision:** 100% ± 12% RSD
Effective Date: 02/02/90	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.3 for proper waste disposal practices.

1. Principle of Method

1.1. Slightly alkaline blank water is spiked with an Environmental Protection Agency (EPA) cyanide QC sample.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the volumetric flask and of the pipette. A relative standard deviation (RSD) of 12% at the 1σ level has been calculated for the calibration, spiking, and dilution process based on the following function:

$$D = \frac{M1 \times M2}{M3},$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of volumetric flask, and
100 = factor to convert to percentage.

2.2. Analytical results from QC samples prepared during 1990 using this procedure and analyzed by automated colorimetry (N = 11) gave a mean recovery of 83% ± 13% at the 1σ level. The data are published in Source Material 10.2.

3. Collection and Storage of Samples

3.1. QC samples are prepared in a volumetric flask and are aliquoted into high-density polyethylene bottles and refrigerated pending analysis.

CAUTION: QC samples must be prepared in an alkaline matrix because cyanide in an acid matrix will release hydrogen cyanide gas which is extremely hazardous.

4. Apparatus

4.1. Flasks: 250-mL, volumetric, glass, class A.

4.2. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.3. Pipette tips: disposable.

4.4. Pipette: 2-mL, volumetric, class A.

4.5. Bottles: 60-mL, high-density polyethylene.

4.6. Labels printed with QC sample numbers.

4.7. Glass vials: with Teflon-lined caps.

5. Reagents

5.1. Sodium hydroxide (50% or approximately 10 M).

5.2. Water. In this procedure, water referred to as "reagent water" is understood to mean water conforming to ASTM Types I, II, or III and free of interferences with the analytical method.

6. Calibration and Standards

6.1. An Environmental Protection Agency-Environmental Monitoring Systems Laboratory (EPA-EMSL) Cyanide Quality Control sample is used as a stock solution.

6.2. EPA records the concentration of the standard after dilution according to EPA instructions. The concentration of the original stock solution must be back-calculated before it is used, taking the EPA dilution instructions into consideration.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the number of samples to be analyzed. Periodically include an unspiked water sample made alkaline with 0.02 M NaOH as a blank.

7.2. This procedure describes the preparation of five 50-mL aliquots of QC samples. QC samples may be prepared in larger or smaller quantities than described in this procedure. Adjust apparatus size as necessary.

7.3. Back-calculate the concentration of the undiluted EPA cyanide stock solution. See Step 8.1.

7.4. Place approximately 200 mL of reagent water in a 250-mL volumetric flask.

7.5. Add 500 μL of 50% NaOH to the water to produce a final NaOH concentration of 0.02 M.

CAUTION: Cyanide compounds must not be added to an acidic solution because hydrogen cyanide gas is released and is very hazardous. Work with cyanide solutions in an approved fume hood.

7.6. Spike an aliquot of undiluted EPA cyanide sample into the water in the flask to reach the desired spike range.

7.7. Dilute the QC sample to volume with reagent water. Mix by inversion.

7.8. Divide the QC sample evenly among five 60-mL high-density polyethylene bottles.

7.9. Label each bottle with a QC sample number and refrigerate pending analysis. Use the same QC number for all five aliquots of the QC sample.

7.10. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD data bases on the VAX.

7.11. Store the unused portion of the EPA cyanide sample in a glass vial with a Teflon-lined cap and refrigerate.

8. Calculations

8.1. Back-calculate the concentration of the undiluted EPA cyanide sample as in the following example.

8.1.1. If the concentration of an EPA cyanide sample is 0.50 mg/L after dilution of a 10-mL aliquot to 1 L according to EPA instructions, the concentration of the undiluted cyanide sample is calculated as follows:

$$(0.50\ \text{mg/L})\ (1\ \text{L}/10\ \text{mL}) = 0.05\ \text{mg/mL}$$

8.1.2 The concentration of 0.05 mg/mL is then used to determine the aliquot used to spike the alkaline water sample.

8.2. Record the concentration of CN in the QC sample as mg/L.

8.3. Calculate CN concentrations as in the following example.

8.3.1. If 2 mL of a 0.05-mg/mL CN standard is used to spike 250 mL of alkaline water, the concentration of CN in the sample will be 0.40 mg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(0.05\ \text{mg/mL})\ (2\ \text{mL})}{(250\ \text{mL})\ (1\ \text{L}/1000\ \text{mL})} = 0.40\ \text{mg/L}\ .$$

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Solid waste contaminated with hazardous chemicals above the level of concern is not normally generated during the routine preparation of QC samples for analysis for inorganic contaminants.

9.1.2. If a solid waste is generated that may be contaminated at a level of concern, complete the appropriate paperwork on a case-by-case basis.

9.1.2.1. Complete a Waste Profile Request Form (WPRF) describing the solid waste and send to the Waste Management group for review.

9.1.2.2. If the Waste Management group determines that the solid waste is contaminated below the level of concern, dispose of the waste as ordinary laboratory trash.

9.1.2.3. If the Waste Management group determines that the solid waste is contaminated above the level of concern, request pickup of the contaminated waste as described in Step 9.3.

9.2. Liquid waste.

9.2.1. Accumulate liquid waste contaminated with cyanide in a glass bottle in a secondary containment tray and keep it in a basic solution segregated from any other liquid waste.

9.2.2. Label the bottle with a hazardous waste label and a label indicating the presence of cyanide. If the bottle is too small to accommodate the hazardous waste label, apply the label to the secondary container.

9.2.3. Open the bottle only for the period of time necessary to add waste to it and keep it in a functioning fume hood whenever it is open.

9.2.4. When the waste is ready for disposal, keep the bottle in the secondary containment tray pending pickup by the Waste Management group.

9.3. Waste pickup.

9.3.1. Request pickup by the Waste Management group of a bottle of cyanide-contaminated waste using the current Chemical Waste Disposal Request (CWDR) form. The current Waste Profile Request Form (WPRF) which describes the waste is referenced on the CWDR.

9.3.2. The Waste Management group picks up the waste for disposal in accord with Laboratory policy.

10. Source Materials

10.1. US EPA, "Instructions for Cyanide Quality Control Sample," (US Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Cincinnati, Ohio 45268, February 1990).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.3. "Hazardous and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.4. ASTM, *1992 Annual Book of ASTM Standards*, Vol 11.01, Specification D1193-91, Standard Specification for Reagent Water, pp. 45-47.

Revisions or additions to the procedure are marked (▒▒▒). Where a section heading is marked, the entire section has been revised.

TRACE ELEMENTS IN WATER (NO_2-N) — QC SAMPLE PREPARATION

Analyte: NO_2-N	**Method No.:** QCI220
Matrix: Water	**Spike Range:** 0.1-1.0 mg/L
Procedure: Flow Injection Colorimetry	**Accuracy and Precision:** 100% ± 5.8% RSD
Effective Date: 10/22/90	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.2 for proper waste disposal practices.

1. Principle of Method

1.1. Sodium nitrite is weighed into a volumetric flask.

1.2. Immediately before use, the weighed salt is dissolved in reagent water and the solution is used to spike reagent water for use as a QC sample.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with use of the pipette, balance, and volumetric flask. A relative standard deviation (RSD) of 5.8% at the 1σ level has been calculated for the error of the standard and for the spiking, weighing, and dilution process based on the following function:

$$D = \frac{M1 \times M2 \times M4}{M3 \times M5},$$

where D = calculated result,
M1 = standard error,
M2 = weighing error,
M3 = dilution error,

M4 = pipette error, and
M5 = second dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2}$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2 + R_{M4}^2 + R_{M5}^2} \times 100$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of balance,
R^2_{M3} = relative variance of volumetric flask,
R^2_{M4} = relative variance of pipette,
R^2_{M5} = relative variance of second volumetric flask, and
100 = factor to convert to percentage.

2.2. Analytical results from QC samples prepared during 1990 using this procedure and analyzed by flow injection colorimetry (N = 4) gave a mean recovery of 96% ± 15% at the 1σ level. The data are published in Source Material 10.1.

3. Collection and Storage of Samples

3.1. Aliquots of crystalline sodium nitrite to be diluted and used to prepare QC samples are stored at room temperature pending analysis.

3.2. A water solution of sodium nitrite cannot be stored as a QC sample because nitrite in solution is readily oxidized to nitrate by the oxygen in the air.

4. Apparatus

4.1. Balance: 100-g minimum capacity, 0.1-mg accuracy.

4.2. Flasks: 10- and 100-mL, volumetric.

4.3. Spatula.

4.4. Auto-pipette: 100-μL, Rainin or equivalent.

4.5. Pipette tips: disposable.

4.6. Labels printed with QC sample numbers.

5. Reagents

5.1. Sodium nitrite (analytical reagent-grade, crystalline).

5.2. Water. In this procedure, water referred to as "reagent water" is understood to mean reagent water conforming to ASTM Type I, II, or III and free of interferences with the analytical method.

6. Calibration and Standards

6.1. Dissolve 10-100 mg of analytical-grade crystalline sodium nitrite in 10 mL of reagent water immediately before use.

6.2. Spike 50 μL of the sodium nitrite solution into 100 mL of reagent water to prepare the QC sample.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed.

7.2. Accurately weigh between 10 and 100 mg of sodium nitrite into a 10-mL volumetric flask.

7.3. Stopper the flask and label it with a QC sample number. Store the sample at room temperature pending final dilution by the analyst.

7.4. The sodium nitrite should be dissolved and diluted as close to time of analysis as possible to prevent loss of the QC sample through oxidation of nitrite to nitrate.

7.5. Place a label on the flask with instructions to the analyst for completion of the QC sample as described below.

7.5.1. Dissolve the sodium nitrite in reagent water in the 10-mL volumetric flask. Dilute the solution to volume with reagent water. Mix by inversion.

7.5.2. Place approximately 90 mL of reagent water in a 100-mL volumetric flask.

7.5.3. Spike 50 μL of the sodium nitrite solution from the 10-mL flask into the water in the 100-mL flask.

7.5.4 Dilute the QC sample in the 100-mL flask to volume with reagent water. Mix by inversion.

7.5.5. Analyze the QC sample immediately after preparation, using the same QC sample number that is on the 10-mL volumetric flask.

7.6. Calculate the concentration of the QC sample as the concentration after final dilution in the 100-mL flask by the analyst.

7.7. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD databases on the VAX.

8. Calculations

8.1. Record the concentration of NO_2-N in the QC sample as mg/L.

8.2. Calculate spike concentrations as in the following example.

8.2.1. Atomic weights: Na = 23, N = 14, and O = 16. The ratio of the atomic weight of N in $NaNO_2$ to the molecular weight of $NaNO_2$ is 14:69.

8.2.2. If 55.8 mg of sodium nitrite is dissolved in 10 mL of reagent water, the NO_2-N concentration in the resulting stock solution will be 1.13 mg/mL.

$$\frac{(55.8 \text{ mg } NaNO_2)\ (14 \text{ mg N}/69 \text{ mg } NaNO_2)}{10 \text{ mL}} = 1.13 \text{ mg N/mL}$$

8.2.3. If 50 μL of the $NaNO_2$ stock solution with a NO_2-N concentration of 1.13 mg/mL is used to spike 100 mL of reagent water, the final concentration of NO_2-N in the QC sample will be 0.57 mg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(1.13 \text{ mg N/mL})\ (1 \text{ mL}/1000\ \mu\text{L})\ (50\ \mu\text{L})}{(100 \text{ mL})\ (1 \text{ L}/1000 \text{ mL})} = 0.57 \text{ mg/L}$$

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Solid waste contaminated with hazardous chemicals above the level of concern is not normally generated during the routine preparation of QC samples for analysis for inorganic contaminants.

9.1.2. If a solid waste is generated that may be contaminated at a level of concern, complete the appropriate paperwork on a case-by-case basis.

9.1.2.1. Complete a Waste Profile Request Form (WPRF) describing the solid waste and send to the Waste Management group for review.

9.1.2.2. If the Waste Management group determines that the solid waste is contaminated below the level of concern, dispose of the waste as ordinary laboratory trash.

9.1.2.3. If the Waste Management group determines that the solid waste is contaminated above the level of concern, request pickup of the contaminated waste as described in Sec. 9.3.

9.2. Liquid waste.

9.2.1. Accumulate liquid waste containing hazardous contaminants, such as solutions of trace metals, in a coated glass bottle in a secondary containment tray and keep it segregated from liquid waste contaminated with organic or radioactive substances or cyanide.

9.2.2. Label the bottle with a hazardous waste label and a label indicating its use for trace metal waste.

9.2.3. Open the bottle only for the period of time necessary to add waste to it.

9.2.4. When the bottle is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management group.

9.2.5. Label a new bottle for further use.

9.3. Waste pickup.

9.3.1. Request pickup by the Waste Management group of solid waste or a full bottle of liquid waste containing trace metals using the current Chemical Waste Disposal Request form (CWDR). The current Waste Profile Request Form (WPRF) which describes the waste is referenced on the CWDR.

9.3.2. The Waste Management group picks up the waste for disposal in accord with Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.2. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.3. ASTM *1992 Annual Book of ASTM Standards*, Vol. 11.01 Specification D1193-91, Standard Specification for Reagent Water, pp. 45-47.

Revisions or additions to the procedure are marked (▒). Where a section heading is marked, the entire section has been revised.

ORGANIC COMPOUNDS ON TUBES AND BADGES — QC SAMPLE PREPARATION

Analyte: Organic compounds	**Method No.:** QCO130
Matrix: Charcoal tubes and organic vapor monitor badges	**Spike Range:** charcoal tube: 30-300 μg/tube organic vapor monitor: 30-100 μg/sample NIOSH-PAT study: 0.2-2 mg/tube
Procedure: Gas chromatography with mass spectrometry (GCMS)	**Accuracy and Precision:** 100% ± 8.2% RSD
Effective Date: 03/05/84	**Author:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.8 for proper waste disposal practices.

1. Principle of Method

1.1. Prepare a stock solution of organic compounds of interest from Table I in a solvent compatible with the analytical extraction method.

1.2. The solvent most commonly used is carbon disulfide. Methylene chloride is also used frequently.

1.3. Spike the stock directly into a charcoal tube or onto the filter paper inside an organic vapor monitor (OVM) badge.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with use of the microsyringe, balance, and volumetric flask. A relative standard deviation (RSD) of 8.2% has been calculated for the error of the standard and for spiking, weighing, and dilution, based on the following:

$$D = \frac{M1 \times M2 \times M4}{M3},$$

where D = calculated result,
M1 = standard error,
M2 = weighing error,
M3 = dilution error, and
M4 = spiking error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where $R^2{}_M$ = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R_{M1}^2 + R_{M2}^2 \times R_{M3}^2 \times R_{M4}^2} \times 100 ,$$

where SD_T = relative standard deviation (% error),
$R^2{}_{M1}$ = relative variance of standard,
$R^2{}_{M2}$ = relative variance of balance,
$R^2{}_{M3}$ = relative variance of volumetric flask,
$R^2{}_{M4}$ = relative variance of microsyringe, and
100 = factor to convert to percentage.

2.2. Analysis by GCMS of QC samples prepared between 1984 and 1989 using this procedure are tabulated below. The data are published in Source Materials 10.1 to 10.7.

CHARCOAL TUBE AND BADGE SPIKE RECOVERY DATA

Compound	Year	Mean ± SD (%)	(N)
Benzene	1984	106 ± 2	4
	1985	133 ± 1	3
	1986	86 ± 3	3
	1987	61 ± 8	3
	1988	83 ± 6	4
	1989	118 ± 4	3
Carbon tetrachloride	1984	98 ± 9	4
	1985	103 ± 4	3
	1986	97 ± 2	5
	1987	110 ± 11	7
	1988	100 ± 6	8
	1989	90 ± 12	12
Cellosolve acetate	1986	95 ± 5	3
	1987	102 ± 4	3
	1988	102 ± 2	4
Chloroform	1984	116 ± 9	8
	1985	101 ± 4	3
	1986	95 ± 3	3
	1987	88 ± 5	4
	1988	99 ± 12	4
	1989	109 ± 2	4
1,2-Dichloroethane	1984	91 ± 12	4
	1985	99 ± 3	6
	1986	130	2
	1987	95 ± 5	7
	1988	99 ± 2	7
	1989	94 ± 6	11
p-Dioxane	1985	94 ± 4	6
	1986	101 ± 5	3
	1988	100 ± 1	4
Methyl chloroform	1984	106 ± 7	8
Methyl ethyl ketone	1984	93 ± 8	3
Trichloroethane	1989	89 ± 4	4

CHARCOAL TUBE AND BADGE SPIKE RECOVERY DATA (cont)			
Compound	Year	Mean ± SD (%)	(N)
Trichloroethylene	1984	90 ± 8	4
	1985	102 ± 4	3
	1986	106	2
	1987	94 ± 6	3
	1988	100 ± 1	4
	1989	90 ± 2	4
Toluene	1984	101 ± 1	3
	1985	105 ± 2	3
	1986	94 ± 6	3
	1987	98 ± 6	8
	1988	86 ± 1	4
	1989	96 ± 3	3
o-Xylene	1984	101 ± 1	3
	1985	103 ± 6	9
	1986	95 ± 5	6
	1987	101 ± 13	7
	1988	89 ± 4	8
	1989	96 ± 4	4

3. Collection and Storage of Samples

3.1. Charcoal tubes are received heat-sealed at both ends with plastic caps to seal the ends after the tubes are opened. Organic vapor monitor badges (OVM) are received in sealed cans. The OVM badges include a closure cap with center and side ports, a charcoal sorbent disc, and a spacer plate above the disc.

3.2. Spiked tubes and badges are stored in a freezer pending analysis.

4. Apparatus

4.1. Volumetric flasks: 1- and 2-mL, class A, glass.

4.2. Pipettes: Pasteur, disposable, glass.

4.3. Pipette bulb: rubber, to fit Pasteur pipette.

4.4. Syringes: gas-tight, 10-, 25-, 50-, and 100-μL.

4.5. Microspatulas.

4.6. Sorbent sample tubes: charcoal, 100-mg primary bed, 50-mg backup bed.

4.7. Sorbent sample tubes: charcoal, 400-mg primary bed, 200-mg backup bed.

4.8. Organic vapor monitors: 3M OVM #3500 or equivalent. 3M, St. Paul, MN 55144-1000.

4.9. Paper filters: 25 - 28 mm diameter, Whatman #2, #41, #42, or equivalent.

4.10. Teflon tape.

4.11. Pliers: to break off the tip of the charcoal sample tube.

4.12. Analytical balance: 100-g minimum capacity, 0.1-mg accuracy.

4.13. Laboratory oven: capable of sustaining 100-150°C.

4.14. Labels printed with QC sample numbers.

5. Reagents

5.1. Carbon disulfide (reagent-grade).

5.2. Methylene chloride (reagent-grade).

5.3. 1,2-dichloroethane (reagent-grade).

6. Calibration and Standards

6.1. Prepare a stock solution of neat, standard-grade analytes, chosen primarily from the list of target compounds in Table I. Use Chem Service or equivalent chemicals (Chem Service, Inc., Box 3108, West Chester, PA 19381). Add solid analytes first, then liquid analytes after the addition of solvent.

6.2. Solvent choice for the stock solution matrix depends on the analytes included in the solution and on the analytical extraction method.

6.2.1. Carbon disulfide is the solvent most commonly used.

6.2.2. A stock solution containing carbon disulfide or methylene chloride as an analyte should use 1,2-dichloroethane as a solvent because methylene chloride and carbon disulfide co-elute.

6.2.3. A stock solution containing ethanol as an analyte should use methylene chloride as a solvent because ethanol forms a colloidal suspension in carbon disulfide.

6.3. The stock solution may be prepared in a 1.0- or 2.0-mL volumetric flask. A solution containing multiple analytes is more conveniently prepared in a 2.0-mL volumetric flask.

6.3.1. The usual concentration range of a stock solution is between 5 and 20 mg/mL. A more concentrated stock solution may be required to prepare a QC sample with a higher spike range. The concentration of the stock solution should be such that a spike volume equal to or less than 15 μL onto a small charcoal tube or OVM badge and equal to or less than 30 μL onto a large charcoal tube will bring all analytes into the desired spike range.

6.3.2. Charcoal-tube QC samples prepared to accompany National Institute for Occupational Safety and Health Proficiency Analytical Testing (NIOSH PAT) intercomparison samples are spiked in the range 0.2-2 mg/tube. A stock solution with analytes in the concentration range of 15-150 mg/mL will reach the desired spike range on a small charcoal tube QC sample when a spike volume of 15 μL or less is used.

6.3.3. Add a solid analyte to a clean, tared 1.0- or 2.0-mL volumetric flask to reach the desired stock concentration range. Record the mass of the analyte added and tare the balance. Repeat for each solid analyte. See Step 8.1.

6.3.4. Add the solvent chosen for the stock solution matrix to the volumetric flask until it reaches approximately halfway to the graduation mark. Stopper the flask.

6.3.5. Swirl the flask to dissolve the solid analytes.

6.3.6. Calculate the volumes of the liquid analytes needed to reach the desired stock concentration range. See Step 8.2 to 8.3.

6.3.7. Place the stoppered flask on the analytical balance and tare the balance.

6.3.8. Add a liquid analyte, from a syringe just above the surface of the solvent, to reach the desired stock concentration range. Restopper the flask and record the mass and volume of the analyte added. Tare the balance. Repeat for each liquid analyte added.

6.3.9. Dilute the solution to volume with the solvent chosen in Step 6.3.4.

6.3.10. Mix by gently inverting three times.

NOTE: Vigorous shaking will cause the loss of volatile analytes.

6.3.11. Wrap Teflon tape around the stopper of the volumetric flask.

6.3.12. Label the flask with information identifying the stock solution and with the expiration date.

6.3.13. The stock solution is stable for approximately 2 weeks when stored at -20°C.

6.4. For QC samples containing multiple analytes, more than one stock solution may be prepared using different analytes. The combination of analytes in the QC sample may be varied by spiking with more than one stock solution.

7. Procedure

7.1. Cleaning glassware.

7.1.1. Wash volumetric flasks with detergent and rinse with hot tap water and then with methanol.

7.1.2. Store the glassware in a laboratory oven maintained at 100-150°C to minimize contamination.

7.2. Cleaning syringes.

7.2.1. Dedicate a group of syringes for use only with semivolatile compounds and a second group of syringes for use only with volatile compounds.

7.2.2. Fill the syringe through the needle with methanol. Discard the methanol into the waste container. Repeat for a total of 5-10 times.

7.2.3. Dry the syringe by pulling a vacuum on it for at least 10 min.

7.3. The spike range for volatile organic compounds on charcoal tubes or on OVM badges varies with the analyte spiked.

7.3.1. For most analytes the spike range is 30-100 μg/tube or 30-100 μg/badge.

7.3.2. For analytes with a shorter retention time than carbon disulfide during gas chromatography, the spike range is 100-300 μg/tube or 100-300 μg/badge. Acetone, methanol, ethanol, 2-propanol, and Freon are included in this category.

7.3.3. The spike range for analytes in QC samples prepared to accompany NIOSH PAT samples is 0.2-2 mg/tube.

7.4. Preparation of QC samples for volatile organic compounds on charcoal tubes.

7.4.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed.

7.4.2. Use the same size charcoal tube for the QC samples as was used to collect the samples to be analyzed.

7.4.3. The total spike volume onto small charcoal tubes should be equal to or less than 15 μL and onto large charcoal tubes should be equal to or less than 30 μL to avoid saturation of the primary charcoal bed.

7.4.4. Remove the stock solution from the freezer and allow it to come to room temperature. Do not allow the stock solution to sit at room temperature for longer than 1 h before spiking the QC sample.

7.4.5. Break off the tip of the entrance end of the charcoal tube using pliers. The entrance end is the end with the glass wool plug over the primary charcoal bed and an air space between the end of the tube and the charcoal bed.

7.4.6. Using a syringe, spike a volume of stock solution onto the primary charcoal bed to reach the desired spike range. Spike the solution beginning in the middle of the primary charcoal bed and drawing the syringe needle toward the glass wool plug at the top of bed as the spiking is completed. The entire volume of spike solution should be on the charcoal bed and not on the walls of the sample tube.

7.4.7. Cap both ends of the sample tube with the plastic tube caps and wrap Teflon tape around the broken end.

7.4.8. Label the tube with a QC sample number.

7.4.9. Return the stock solution to the freezer.

7.4.10. Store the QC samples in a freezer pending analysis.

7.4.11. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD databases on the VAX.

7.5. Preparation of QC samples for volatile organic compounds on organic vapor monitor (OVM) badges.

7.5.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed.

7.5.2. The total spike volume onto an OVM badge should be equal or less than 15 μL.

7.5.3. Remove the stock solution from the freezer and allow it to come to room temperature. Do not allow the stock solution to sit at room temperature for longer than 1 h before spiking the QC sample.

7.5.4. Remove the OVM badge from the can in which it was received.

7.5.5. Remove and discard the plastic ring and white film from the badge.

7.5.6. Place a 25-28-mm paper filter onto the spacer plate above the charcoal disc. Snap the closure cap onto the badge. Close the side port.

7.5.7. Using a syringe, spike a volume of the stock onto the filter paper through the center port to reach the desired spike range. Close the center port.

7.5.8. Return the badge to the can in which it was received and label the can with a QC number.

7.5.9. The badge should be kept in the refrigerator for at least 16-24 h before analysis to achieve complete transfer of the organic compounds from the filter paper onto the charcoal sorbent or in a freezer for longer periods of time pending analysis.

7.5.10. Return the stock solution to the freezer.

7.5.11. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD databases on the VAX.

8. Calculations

8.1. Solid analytes used in the stock solution are measured directly by mass.

8.2. Calculate the volume of neat liquid analyte to add to the stock solution as in the following example.

8.2.1. Read the density of the neat liquid analyte from Table I. The density of chloroform is 1.492 mg/μL.

8.2.2. The desired spike range for 1 mL of stock is 5-20 mg of neat analyte.

8.2.3. Divide the weight spike range by the density of the neat analyte to determine the desired volume spike range of neat analyte to use in 1 mL of stock solution.

$$\frac{5\ \text{mg}}{1.492\ \text{mg}/\mu\text{L}}\ \text{to}\ \frac{20\ \text{mg}}{1.492\ \text{mg}/\mu\text{L}} = 3.4\ \text{to}\ 13.4\ \mu\text{L}$$

For chloroform, 3.4-13.4 μL of the neat analyte should be diluted to 1 mL to reach the desired concentration range of 5-20 mg/mL in the stock solution.

8.3. Calculate the concentration of each neat analyte in the stock solution as in the following example.

8.3.1. If 5 μL of chloroform, weighing 7.5 mg, is diluted to 1 mL of stock solution, the concentration of chloroform in the stock solution is 7.5 mg/mL.

$$\frac{7.5\ \text{mg}}{1\ \text{mL}} = 7.5\ \text{mg/mL}$$

8.4. Calculate the concentration of each analyte on a charcoal tube QC sample as in the following example.

8.4.1. If 6 μL of a stock solution containing 7.5 mg/mL of chloroform is spiked onto a charcoal tube, the concentration of chloroform in the QC sample is 45 μg/sample. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(7.5\ \text{mg/mL})\left(\frac{1\ \text{mL}}{1000\ \mu\text{L}}\right)\left(\frac{1000\ \mu\text{g}}{1\ \text{mg}}\right)(6\ \mu\text{L})}{\text{sample}} = 45\ \mu\text{g/sample}$$

8.5. Calculate the concentration of each analyte on an OVM badge QC sample as in the following example.

8.5.1. If 9 μL of a stock solution containing 7.5 mg/mL of chloroform is spiked onto an OVM badge, the concentration of chloroform in the QC sample is 67.5 μg/sample. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(7.5\ \text{mg/mL})\left(\frac{1\ \text{mL}}{1000\ \mu\text{L}}\right)\left(\frac{1000\ \mu\text{g}}{1\ \text{mg}}\right)(9\ \mu\text{L})}{\text{sample}} = 67.5\ \mu\text{g/sample}$$

8.6. Record the analyte concentration on a charcoal tube QC sample or an OVM badge as μg/sample or mg/sample.

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Solid waste, such as paper wipes, glass or plastic pipettes, empty vials, and incorrectly spiked QC samples contaminated with organic compounds, is accumulated in a covered metal can lined with a plastic bag and is kept segregated from PCB-contaminated solid waste and solid waste that is not contaminated with organic compounds.

9.1.2. The can is labeled with a hazardous waste label and a label indicating its use for waste contaminated with organic compounds.

9.1.3. The can is opened only for the period of time necessary to add waste.

9.1.4. When the plastic bag is full, it is sealed with tape, removed from the can, identified as waste contaminated with organic compounds, and placed in a functioning chemical exhaust hood or in a closed secondary container pending pickup by the Waste Management Group.

9.1.5. A new plastic bag is put into the waste can for continued use.

9.2. Liquid waste.

9.2.1. Liquid waste containing organic compounds, such as rinses from syringes, excess QC samples, and incorrectly spiked QC samples, is accumulated in a covered metal can in a secondary containment tray.

9.2.1.1. Liquid waste containing organic compounds but no PCBs is kept segregated from PCB-contaminated liquid waste.

9.2.1.2. The can is labeled with a hazardous waste label and a label indicating its use for waste containing organic compounds.

9.2.1.3. The can is labeled with a hazardous waste label and a label indicating its use for waste containing organic compounds.

9.2.1.4. When the can is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.1.5. A new can is labeled for future use.

9.2.1. Two-mL vials containing out-of-date spiking solutions of organic compounds are accumulated in a glass jar in a secondary containment tray.

9.2.2.1. Solutions that are not contaminated with PCBs are kept segregated from those that are PCB-contaminated.

9.2.2.2. The jar is labeled with a hazardous waste label and a label indicating its use for waste containing organic compounds.

9.2.2.3. The jar is opened only for the period of time necessary to add vials.

9.2.2.4 When the jar is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.2.5. A new jar is labeled for future use.

9.3. Waste pickup.

9.3.1. Pickup by the Waste Management Group of a full bag, can, or jar of waste contaminated with organic compounds is requested using the current Chemical Waste Disposal Request form. The current Waste Profile Request Form (WRPF) that describes the waste is referenced on the Chemical Waste Disposal Request.

9.3.2. The Waste Management Group picks up the waste for disposal according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, and D. R. Perrin, "Quality Assurance for Health and Environmental Chemistry: 1984," Los Alamos National Laboratory report LA-10508-MS (1985).

10.2. M. A. Gautier, E. S. Gladney, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1985," Los Alamos National Laboratory report LA-10813-MS (1986).

10.3. M. A. Gautier, E. S. Gladney, W. D. Moss, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1986," Los Alamos National Laboratory report LA-11114-MS (1987).

10.4. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1987," Los Alamos National Laboratory report LA-11454-MS (1988).

10.5. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1988," Los Alamos National Laboratory report LA-11637-MS (1989).

10.6. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.7. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.8. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

TABLE I. ORGANIC COMPOUNDS

Compound	CAS Number	Density of liquids (mg/μL)
acetone	67641	0.791
benzene	71432	0.874
bromobenzene	108861	1.491
bromochloromethane	74975	1.991
bromodichloromethane	75274	1.980
bromoform	75252	2.894
bromomethane	74839	0.838
2-butanone (MEK)	78933	0.805
n-butylbenzene	104518	0.860
sec-butylbenzene	135988	0.863
tert-butylbenzene	98066	0.867
carbon disulfide	75150	1.266
carbon tetrachloride	56235	1.594
chlorobenzene	108907	1.107
chlorodibromomethane	124481	2.451
chloroethane	75003	0.891
chloroform	67663	1.492
1-chlorohexane	544105	0.879
2-chlorotoluene	95498	1.083
4-chlorotoluene	106434	1.070
1,2-dibromo-3-chloropropane	96128	2.093
1,2-dibromoethane	106934	2.180
dibromomethane	74953	2.477
1,2-dichlorobenzene	95501	1.306
1,3-dichlorobenzene	541731	1.288
1,4-dichlorobenzene	106467	1.241
1,1-dichloroethane	75343	1.176
1,2-dichloroethane	107062	1.256
1,1-dichloroethene	75354	1.213
cis-1,2-dichloroethene	156592	1.284
trans-1,2-dichloroethene	156605	1.257
1,2-dichloropropane	78875	1.156
1,3-dichloropropane	142289	1.190
2,2-dichloropropane	594207	1.082
1,1-dichloropropene	563586	1.169
cis-1,3-dichloropropene	10061015	1.224
trans-1,3-dichloropropene	10061026	1.224
ethylbenzene	100414	0.867
hexachlorobutadiene	87683	1.665

TABLE I. ORGANIC COMPOUNDS (cont)

Compound	CAS Number	Density of liquids (mg/μL)
2-hexanone	591786	0.812
isopropylbenzene	98828	0.864
4-isopropyltoluene	99876	0.860
methylene chloride	75092	1.325
4-methyl-2-pentanone (MIK)	108101	0.800
naphthalene	91203	solid
n-propylbenzene	103651	0.862
styrene	100425	0.909
1,1,1,2-tetrachloroethane	630206	1.598
1,1,2,2,-tetrachloroethane	79345	1.586
tetrachloroethene	127184	1.623
toluene	108883	0.867
1,2,3-trichlorobenzene	87616	solid
1,2,4-trichlorobenzene	120821	1.454
1,1,1-trichloroethane	71556	1.338
1,1,2-trichloroethane	79005	1.435
trichloroethene	79016	1.464
trichlorofluoromethane	75694	1.494
1,2,3-trichloropropane	96184	1.387
1,2,4-trimethylbenzene	95636	0.889
1,3,5-trimethylbenzene	108678	0.864
vinyl acetate	108054	0.934
o-xylene	95476	0.870
mixed xylenes (total)	1330207	0.860

POLYCHLORINATED BIPHENYLS IN OIL — QC SAMPLE PREPARATION

Analyte: Polychlorinated biphenyls (PCBs)	**Method No.:** QCO170
Matrix: Oil	**Spike Range:** 10-500 μg/g oil
Procedure: Analysis by gas chromatography with electron-capture detection (GCECD)	**Accuracy and Precision:** EPA standards - 100% ± 10.0% EM-9 standards - 100% ± 1.5%
Effective Date: 09/15/86	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.4 to 10.5 for proper waste disposal practices.

1. Principle of Method

1.1. Prepare stock solutions of Aroclor 1242, 1254, and 1260 in hexane, or obtain Environmental Protection Agency (EPA) Aroclor 1242, 1254, and 1260 standards. Refer to Table I for Aroclors and associated CAS numbers.

TABLE I

Compound	CAS Number
Aroclor 1242	53469219
Aroclor 1254	11097691
Aroclor 1260	11096825
Total PCBs	1336363

1.2. Spike the stock solution into a blank oil sample.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with the use of the microsyringe and of the balance. A relative standard deviation (RSD) of 10% for EPA standards and 1.5% for in-house standards has been calculated for the error of the standard and for spiking and weighing based on the following:

2.1.1. EPA standard.

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated result,
M1 = standard error,
M2 = spiking error, and
M3 = weighing error.

2.1.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2} \times 100 ,$$

where S_{D_T} = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of microsyringe,
R^2_{M3} = relative variance of balance, and
100 = factor to convert to percentage.

2.1.2. In-house standard.

$$U = \frac{M1 \times M2 \times M4}{M3 \times M5} ,$$

where U = uncertainty,
M1 = standard error,
M2 = weighing error,

M3 = dilution error,
M4 = second weighing error, and
M5 = spiking error.

2.1.2.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots . M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable.

2.1.2.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3} + R^2_{M4} + R^2_{M5}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of balance,
R^2_{M3} = relative variance of volumetric flask,
R^2_{M4} = relative variance of second balance,
R^2_{M5} = relative variance of microsyringe, and
100 = factor to convert to percentage.

2.2. Analytical results from QC samples prepared between 1990 and 1991 using this procedure and analyzed by gas chromatography with electron-capture detection are tabulated below. The data are published in Source Materials 10.1 to 10.3.

PCB IN OIL SPIKE RECOVERY DATA						
Compound	1990 Mean ± SD (%)		N	1991 Mean ± SD (%)		N
			Water			
Aroclor 1254	—			84 ± 34		(6)
Aroclor 1260	94		(1)	—		
Total Aroclor	94		(1)	84 ± 34		(6)
			Soil			
Aroclor 1242	106 ± 36		(19)	90 ± 21		(13)
Aroclor 1254	72		(2)	74 ± 28		(7)
Aroclor 1260	94 ± 19		(27)	111 ± 29		(16)
Total Aroclor	98 ± 27		(48)	96 ± 29		(36)
			Oil			
Aroclor 1242	96 ± 19		(56)	112 ± 20		(49)
Aroclor 1260	79 ± 14		(88)	96 ± 23		(78)
Total Aroclor	86 ± 18		(143)	102 ± 23		(127)

3. Collection and Storage of Samples

3.1. Spiked QC samples are stored at room temperature pending analysis. Samples prepared in 1986 and analyzed through 1991 have shown no decrease in analyte recovery.

4. Apparatus

4.1. Volumetric flasks: 5-mL, class A, glass.

4.2. Gas-tight syringes: 50-, 250-, 500-, and 2500 μL.

4.3. Bottles: 2- and 8-oz, glass with Teflon-lined lids.

4.4. Vials: 1.5-mL glass.

4.5. Caps: open-top to fit 1.5-mL vials.

4.6. Septa: Teflon/silicone discs to fit open-top caps.

4.7. Teflon tape.

4.8. Pipettes: Pasteur, disposable glass.

4.9. Pipette bulb: rubber, to fit Pasteur pipette.

4.10. Stirring rod: glass, 3- to 4-mm diameter.

4.11. Autosampler vials with Teflon-lined septa and crimp tops: 11- x 30-mm vials, and 11-mm silicone liner with Teflon face.

4.12. Portable crimper for sealing caps on autosampler vials.

4.13. Analytical balance: 100-g minimum capacity, 0.1-mg accuracy.

4.14. Analytical balance: 400-g minimum capacity, 1-mg accuracy.

4.15. Ultrasonic mixer.

4.16. Labels printed with QC sample numbers.

5. Reagents

5.1. Hexane (analytical-grade).

5.2. Blank vacuum pump oil. Any oil selected should be checked for interferences with the specific Aroclor chromatographic patterns.

6. Calibration and Standards

6.1. EPA Aroclor standards in isooctane or methanol are available in concentrations between 500 and 3000 μg/mL.

6.2. After opening the heat-sealed ampule containing an Aroclor standard, transfer the contents into a vial with a screw cap with Teflon/silicone septum. Wrap the cap with Teflon tape. Remove the original label and affix it to the vial or transfer the standards information to a label on the vial.

6.3. Solutions of PCBs may be prepared from pure PCBs in concentrations of 50-60 mg/mL.

6.4. Preparation of in-house standards in hexane.

6.4.1. Tare a 5-mL volumetric flask on a balance accurate to 0.1 mg. Add approximately 250-300 mg of a pure PCB to the bottom of the flask using a glass Pasteur pipette or a glass stirring rod. Record the mass added.

6.4.2. Dissolve the PCB and dilute it to volume with hexane. Mix by inversion.

6.4.3. Prepare a separate standard solution for each PCB desired. Do not mix Aroclors.

6.4.4. The standard concentration will be 50-60 mg/mL. See Step 8.1.

6.4.5. Transfer the standard to vials fitted with Teflon septa and caps. Wrap Teflon tape over and around the caps.

6.4.6. Label the vials with information identifying the standard.

6.5. Store the stock standards in a freezer.

7. Procedure

7.1. Discard used, PCB-contaminated flasks, bottles, and pipettes in the PCB waste container.

7.2. Cleaning of syringes.

7.2.1. Dedicate a group of syringes for use only with PCBs.

7.2.2. Fill the syringe with methanol through the needle. Discard the methanol into a waste container. Repeat for a total of 5-10 times.

7.2.3. Dry the syringe by pulling a vacuum on it for at least 10 min.

7.3. QC samples may be prepared in larger or smaller quantities than described in this procedure adjusting apparatus size as necessary. Preparation of 200 g of a PCB-in-oil QC sample from an in-house standard, or of 10 g of a PCB-in-oil QC sample from an EPA standard is described in this procedure.

7.4. QC sample preparation using in-house Aroclor standards.

7.4.1. Remove a vial of the desired in-house Aroclor standard from the freezer and allow it to reach room temperature.

7.4.2. Tare a clean 8-oz glass bottle on a balance accurate to 1 mg or better.

7.4.3. Add approximately 200 g of blank vacuum pump oil to the bottle. Record the mass of oil added.

7.4.4. Spike an aliquot of an in-house Aroclor standard into the oil using a syringe to reach the desired spike range. See Step 8.3.

7.4.5. Cap the bottle with a Teflon lid and label it with information identifying the sample.

7.4.6. Return the vial containing the in-house Aroclor standard to the freezer.

7.4.7. Prepare several PCB-in-oil QC samples using different Aroclors in hexane and different spike volumes to cover the entire spike range.

7.4.8. Mix the PCB-in-oil QC samples for at least 8 h in an ultrasonic mixer.

7.4.9. Store the samples at room temperature.

7.5. QC sample preparation using EPA aroclor standards.

7.5.1. Choose an EPA Aroclor standard with a concentration between 500 and 3000 μg/mL to reach the desired concentration range in 10 g of oil.

7.5.2. Remove a vial of the chosen Aroclor standard from the freezer and allow it to reach room temperature.

7.5.3. Tare a clean 2-oz glass bottle on a balance accurate to 1 mg or better.

7.5.4. Add approximately 10 g of blank vacuum pump oil to the bottle. Record the mass of oil added.

7.5.5. Using a syringe, spike an aliquot of EPA aroclor standard into the oil to reach the desired spike range. See Step 8.4.

7.5.6. Cap the bottle with a Teflon lid and label it with information identifying the sample.

7.5.7. Return the vial containing the Aroclor standard to the freezer.

7.5.8. Prepare several PCB-in-oil QC samples using different EPA Aroclors and different spike volumes to cover the entire spike range.

7.5.9. Mix the PCB-in-oil QC samples for at least 8 h in an ultrasonic mixer.

7.5.10. Store the samples at room temperature.

7.6. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD data bases on the VAX.

7.7. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed.

7.7.1. Aliquot 1-2 mL of a PCB-in-oil QC sample into an autosampler vial and seal the vial with a crimp-top cap.

7.7.2. Label the vial with a QC sample number.

8. Calculations

8.1. Calculate the concentration of the in-house Aroclor standard prepared from pure PCBs as in the following example.

8.1.1. If 275.0 mg of pure PCB is diluted to 5.0 mL with hexane, the concentration of the PCB in the standard is 55 mg/mL.

$$\frac{275.0 \text{ mg}}{5.0 \text{ mL}} = 55 \text{ mg/mL}$$

8.2. Calculate spike concentrations from an in-house Aroclor standard as in the following example.

8.2.1. If 150 μL of a 55 mg/mL in-house Aroclor standard is spiked into 198.0 g of blank oil, the PCB concentration in the QC sample is 42 μg/g. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(55 \text{ mg/mL}) \left(\frac{1 \text{ mL}}{1000 \ \mu\text{L}}\right) \left(\frac{1000 \ \mu\text{g}}{1 \text{ mg}}\right) (150 \ \mu\text{L})}{198.0 \text{ g}} = 42 \ \mu\text{g/g}$$

8.2.2. Record the Aroclor concentrations in the QC samples in μg/g.

8.3. Calculate spike concentrations from an EPA Aroclor standard as in the following example.

8.3.1. If 800 μL of a 3000 μg/mL EPA Aroclor standard is spiked into 10 g of blank oil, the PCB concentration in the QC sample is 240 μg/g. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(3000 \ \mu\text{g/mL}) \left(\frac{1 \text{ mL}}{1000 \ \mu\text{L}}\right) (800 \ \mu\text{L})}{10 \text{ g}} = 240 \ \mu\text{g/g}$$

9. Proper Waste Disposal Practices

9.1. Solid Waste.

9.1.1. PCB-contaminated solid waste, such as paper wipes, glass or plastic pipettes, empty vials, and incorrectly spiked QC samples, is accumulated in a covered metal can lined with a plastic bag and is kept segregated from non-PCB-contaminated solid waste.

9.1.2. The can is labeled with a hazardous waste label and a label indicating its use for PCB-contaminated waste.

9.1.3. The can is opened only for the period of time necessary to add waste to it.

9.1.4. When the plastic bag is full, it is sealed with tape, removed from the can, identified as PCB-contaminated waste, and placed in a functioning chemical exhaust hood pending pickup by the Waste Management Group.

9.1.5. A new plastic bag is put into the waste can for continued use.

9.2. Liquid Waste.

9.2.1. PCB-contaminated liquid waste, such as rinses from PCB-contaminated syringes, PCB standards, and incorrectly spiked QC samples, is accumulated in a covered polyethylene bottle in a secondary containment tray and is kept segregated from non-PCB-contaminated liquid waste.

9.2.2. The bottle is labeled with a hazardous waste label and a label indicating its use for PCB-contaminated waste.

9.2.3. The bottle is opened only for the period of time necessary to add waste to it.

9.2.4. When the bottle is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.5. A new bottle is labeled for further use.

9.3. Waste Pickup

9.3.1. Pickup by the Waste Management Group of a full bag or bottle of PCB-contaminated waste is requested using the current Chemical Waste Disposal Request form. The current Waste Profile Request Form

(WPRF) that describes the waste is referenced on the Chemical Waste Disposal Request.

9.3.2. The Waste Management Group picks up the waste for disposal in accordance with Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.3. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Environmental Chemistry: 1991," Los Alamos National Laboratory report LA-12436-MS (1992).

10.4. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.5. "Polychlorinated Biphenyls," Administrative Requirement 10-4, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

POLYCHLORINATED BIPHENYLS ON SOIL — QC SAMPLE PREPARATION

Analyte: Polychlorinated biphenyls (PCBs)	**Method No.:** QCO190
Matrix: Soil	**Spike Range:** 10-30 μg/g
Procedure: Analysis by gas chromatography with electron-capture detection (GCECD)	**Accuracy and Precision:** EPA standards - 100% ± 10.0% EM-9 standards - 100% ± 1.8%
Effective Date: 05/17/88	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.4 to 10.5 for proper waste disposal practices.

1. Principle of Method

1.1. Prepare stock solutions of PCB 1242, 1254, and 1260 in hexane, or obtain Environmental Protection Agency (EPA) Aroclor 1242, 1254, and 1260 standards. Refer to Table I for Aroclors and associated CAS numbers.

TABLE I

Compound	CAS Number
Aroclor 1242	53469219
Aroclor 1254	11097691
Aroclor 1260	11096825
Total PCBs	1336363

1.2. Spike the Aroclor standard directly onto a soil sample.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with use of the microsyringe, volumetric flask, and balance. A relative standard deviation (RSD) of 10.0% for EPA standards and 1.8% for in-house standards has been calculated for the error of the standard and for the spiking and weighing process based on the following:

2.1.1. EPA standard.

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated results,
M1 = standard error,
M2 = spiking error, and
M3 = weighing error.

2.1.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2} \times 100 ,$$

where S_{D_T} = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of microsyringe,
R^2_{M3} = relative variance of balance, and
100 = factor to convert to percentage.

2.1.2. In-house standard.

$$U = \frac{M1 \times M2 \times M4 \times M6}{M3 \times M5 \times M7} ,$$

where U = uncertainty,
M1 = standard error,
M2 = first weighing error,

M3 = first dilution error,
M4 = first spiking error,
M5 = second dilution error,
M6 = second spiking error, and
M7 = second weighing error.

2.1.2.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3} + R^2_{M4} + R^2_{M5} + R^2_{M6} + R^2_{M7}} \times 100 ,$$

where S_{DT} = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of first balance,
R^2_{M3} = relative variance of first volumetric flask,
R^2_{M4} = relative variance of first microsyringe,
R^2_{M5} = relative variance of second volumetric flask,
R^2_{M6} = relative variance of second microsyringe,
R^2_{M7} = relative variance of second balance, and
100 = factor to convert to percentage.

2.2. Analyses by GCECD of QC samples prepared between 1990 and 1991 using this procedure and analyzed by gas chromatography with electron-capture detection are tabulated below. The data are published in Source Materials 10.1 to 10.3.

SUMMARY OF IN-HOUSE QC DATA FOR PCBs (% recovery ± std dev)		
	1990 Mean ± SD N	1991 Mean ± SD N
	Water	
Aroclor 1254	—	84 ± 34 (6)
Aroclor 1260	94 (1)	—
Total Aroclor	94 (1)	84 ± 34 (6)
	Soil	
Aroclor 1242	106 ± 36 (19)	90 ± 20 (13)
Aroclor 1254	72 (2)	74 ± 28 (7)
Aroclor 1260	94 ± 19 (27)	111 ± 29 (16)
Total Aroclor	98 ± 27 (48)	96 ± 29 (36)
	Bulk	
Aroclor 1242	96 ± 19 (56)	112 ± 20 (49)
Aroclor 1260	—	96 ± 23 (78)
Total Aroclor	—	102 ± 23 (127)

3. Collection and Storage of Samples

3.1. The blank soil matrix and the spiked samples should be stored in glass bottles fitted with Teflon lids. Avoid using plastic containers and lids because phthalates may leach from plastics and contaminate the soil.

3.2. Spiked QC samples are stored at room temperature pending analysis. A study performed in 1989 - 1990 showed no appreciable difference in analyte recovery between samples stored at room temperature and those stored in the refrigerator.

4. Apparatus

4.1. Syringes: gas-tight, 100-, 250-, and 1000-μL.

4.2. Bottles: 1/2-oz, glass with Teflon-lined lids.

4.3. Vials: 1.5-mL, glass.

4.4. Caps: open-top to fit vials.

4.5. Septa: Teflon/silicone discs to fit open-top caps

4.6. Teflon tape.

4.7. Analytical balance: 100-g minimum capacity, 0.1-mg accuracy.

4.8. Pipettes: Pasteur, disposable glass.

4.9. Pipette bulb: rubber, to fit Pasteur pipette.

4.10. Stirring rod: glass, 3- to 4-mm diameter.

4.11. Volumetric flasks: 5-mL, class A, glass.

4.12. Labels printed with QC sample numbers.

5. Reagents

5.1. Hexane (analytical-grade).

5.2. Soil. A clean, dry 8-g aliquot is required for each sample. A suitable soil does not contain the analyte of interest and presents few interferences to the analytical method.

5.3. Blank soil heated to nominally 100°C for 5 h and stored in glass is used as a sample matrix.

6. Calibration and Standards

6.1. EPA Aroclor standards in isooctane or methanol are available in PCB concentrations between 500 and 3000 μg/mL.

6.2. After opening the heat-sealed ampules containing Aroclor standards, transfer the contents into vials with screw caps with Teflon/silicone septa. Wrap the caps with Teflon tape. Remove the original label and affix it to the vial or transfer the standards information to a label on the new vial.

6.3. Solutions of PCBs may be prepared from pure PCBs in concentrations of 50-60 mg/mL.

6.4. Preparation of in-house standards in hexane.

6.4.1. Tare a 5-mL volumetric flask on an analytical balance. Add approximately 250-300 mg of a pure PCB to the bottom of the flask using a glass Pasteur pipette or a glass stirring rod. Record the mass added.

6.4.2. Dissolve the PCB and dilute it to volume with hexane. Mix by inversion.

6.4.3. Prepare a separate standard solution for each PCB desired. Do not mix Aroclors.

6.4.4. The standard concentration will be 50-60 mg/mL. See Step 8.1.

6.4.5. Transfer the standard to vials fitted with Teflon septa and caps. Wrap Teflon tape over and around the caps.

6.4.6. Label the vials with information identifying the standard.

6.5. Prepare a 1:10 dilution of the in-house standard in hexane.

6.5.1. Add 4 mL of hexane to a clean 5-mL volumetric flask.

6.5.2. Using a syringe, add 500 μL of 50-60 mg/mL Aroclor in-house standard to the hexane in the flask.

6.5.3. Dilute the standard to volume with hexane. Mix by inversion.

6.5.4. The concentration of the dilution will be 5000-6000 μg/mL. See Step 8.2.

6.5.5. Transfer the standard to vials fitted with Teflon septa and caps. Wrap Teflon tape over and around the caps.

6.5.6. Label the vials with information identifying the standard.

6.6. Store stock standards in a freezer.

7. Procedure

7.1. Discard used, PCB-contaminated flasks, bottles, and pipettes in the PCB waste container.

7.2. Cleaning of syringes.

7.2.1. Dedicate a group of syringes for use only with PCBs.

7.2.2. Fill the syringe with methanol through the needle. Discard the methanol into a waste container. Repeat for a total of 5-10 times

7.2.3. Dry the syringe by pulling a vacuum on it for at least 10 min.

7.3. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed.

7.3.1. Accurately weigh 8 g of blank soil into a 1/2-oz glass bottle with a Teflon-lined lid. Record the mass of soil on the bottle with a felt-tipped pen for reference by the analyst.

7.3.2. Remove a vial containing a standard of the desired Aroclor in the concentration range 500-6000 μg/mL from the freezer and allow it to reach room temperature.

7.3.3. Spike a volume of the standard directly onto the soil to reach the desired spike range. Avoid wetting the bottle walls. The analyst will desorb the entire sample in the bottle. See Step 8.4.

7.3.4. Cap the bottle and label it with a QC sample number.

7.3.5. Return the vial containing the Aroclor standard to the freezer.

7.3.6. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD data bases on the VAX.

7.3.7. Store the QC samples at room temperature pending analysis.

8. Calculations

8.1. Calculate the concentration of the concentrated PCB standard prepared from pure PCBs as in the following example.

8.1.1. If 275.0 mg of pure PCB is diluted to 5 mL with hexane, the concentration of the PCB in the standard is 55 mg/mL.

$$\frac{275.0 \text{ mg}}{5 \text{ mL}} = 55 \text{ mg/mL}$$

8.2. Calculate the concentration of the 1:10 dilution of the concentrated PCB standard as in the following example.

8.2.1. If 500 μL of a PCB standard whose concentration is 55 mg/mL is diluted to 5 mL with hexane, the concentration of the dilute standard is 5500 μg/mL.

$$\frac{(55\ \text{mg/mL})\left(\frac{1\ \text{mL}}{1000\ \mu\text{L}}\right)(500\ \mu\text{L})\left(\frac{1000\ \mu\text{g}}{1\ \text{mg}}\right)}{5\ \text{mL}} = 5500\ \mu\text{g/mL}$$

8.3. Calculate spike concentrations as in the following example.

8.3.1. If 50 μL of a 5500 μg/mL PCB standard is spiked onto 8.0005 g of soil, the PCB concentration in the QC sample is 34 μg/g. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(5500\ \mu\text{g/mL})\left(\frac{1\ \text{mL}}{1000\ \mu\text{L}}\right)(50\ \mu\text{L})}{8.0005\ \text{g}} = 34\ \mu\text{g/g}$$

8.4. Record Aroclor concentrations in μg/g.

9. Proper Waste Disposal Practices

9.1. Solid Waste

9.1.1. PCB-contaminated solid waste, such as paper wipes, glass or plastic pipettes, empty vials, and incorrectly spiked QC samples, is accumulated in a covered metal can lined with a plastic bag and is kept segregated from non-PCB-contaminated solid waste.

9.1.2. The can is labeled with a hazardous waste label and a label indicating its use for PCB-contaminated waste.

9.1.3. The can is opened only for the period of time necessary to add waste to it.

9.1.4. When the plastic bag is full, it is sealed with tape, removed from the can, identified as PCB-contaminated waste, and placed in a functioning chemical exhaust hood pending pickup by the Waste Management Group.

9.1.5. A new plastic bag is put into the waste can for continued use.

9.2. Liquid Waste

9.2.1. PCB-contaminated liquid waste, such as rinses from PCB-contaminated syringes, PCB standards, and incorrectly spiked QC samples, is accumulated in a covered polyethylene bottle in a secondary

containment tray and is kept segregated from non-PCB-contaminated liquid waste.

9.2.2. The bottle is labeled with a hazardous waste label and a label indicating its use for PCB-contaminated waste.

9.2.3. The bottle is opened only for the period of time necessary to add waste to it.

9.2.4. When the bottle is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.5. A new bottle is labeled for further use.

9.3. Waste Pickup

9.3.1. Pickup by the Waste Management Group of a full bag or bottle of PCB-contaminated waste is requested using the current Chemical Waste Disposal Request form. The current Waste Profile Request Form (WPRF) that describes the waste is referenced on the Chemical Waste Disposal Request.

9.3.2. The Waste Management Group picks up the waste for disposal in accordance with Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.3. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Environmental Chemistry: 1991," Los Alamos National Laboratory report LA-12436-MS (1992).

10.4. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.5. "Polychlorinated Biphenyls," Administrative Requirement 10-4, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

POLYCHLORINATED BIPHENYLS ON SWIPES — QC SAMPLE PREPARATION

Analyte: Polychlorinated biphenyls (PCBs)	**Method No.:** QCO200
Matrix: Gauze Swipes	**Spike Range:** 5-20 μg/sample
Procedure: Analysis by gas chromatography with electron-capture detection (GCECD)	**Accuracy and Precision:** EPA standards - 100% ± 10.1% EM-9 standards - 100% ± 5.2%
Effective Date: 06/05/87	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.4 to 10.5 for proper waste disposal practices.

1. Principle of Method

1.1. Prepare stock solutions of PCB 1242, 1254, and 1260 in hexane, or obtain Environmental Protection Agency (EPA) Aroclor 1242, 1254, and 1260 standards. Refer to Table I for Aroclors and associated CAS numbers.

TABLE I.

Compound	CAS Number
Aroclor 1242	53469219
Aroclor 1254	11097691
Aroclor 1260	11096825
Total PCBs	1336363

1.2. Spike an Aroclor standard onto a blank 2- × 2-in. gauze in a 1/2-oz bottle. The analyst will desorb in this container.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with use of the microsyringe, balance, and volumetric flasks. A relative standard deviation (RSD) of 10.1% for EPA standards and 2.0% for in-house standards has been calculated for the error of the standard and for weighing, dilution, and spiking, based on the following:

2.1.1. EPA standard.

$$D = M1 \times M2 \quad ,$$

where D = calculated result,
M1 = standard error, and
M2 = spiking error.

2.1.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} \quad ,$$

where R^2_M = relative variance,
M = $M_1 \ldots . M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R_{M1}^2 + R_{M2}^2} \times 100 \quad ,$$

where S_{D_T} = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of microsyringe, and
100 = factor to convert to percentage.

2.1.2. In-house standard.

$$U = \frac{M1 \times M2 \times M4 \times M6}{M3 \times M5} ,$$

where U = uncertainty,
M1 = standard error,
M2 = weighing error
M3 = dilution error,
M4 = spiking error,
M5 = second dilution error, and
M6 = second spiking error.

2.1.2.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3} + R^2_{M4} + R^2_{M5} + R^2_{M6}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard
R^2_{M2} = relative variance of balance,
R^2_{M3} = relative variance of volumetric flask,
R^2_{M4} = relative variance of microsyringe,
R^2_{M5} = relative variance of second volumetric flask,
R^2_{M6} = relative variance of second microsyringe, and
100 = factor to convert to percentage

2.2. Analyses by GCECD of QC samples prepared between 1990 and 1991 using this procedure are tabulated below. The data are published in Source Materials 10.1 to 10.3.

PCB ON SWIPES SPIKE RECOVERY DATA		
	1990	1991
	Mean ± SD N	Mean ± SD N
Aroclor 1242	96 ± 30 (28)	105 ± 34 (25)
Aroclor 1254	98 ± 2 (9)	104 ± 30 (26)
Aroclor 1260	95 ± 19 (48)	104 ± 27 (31)
Aroclor Total	96 ± 24 (85)	104 ± 30 (82)

3. Collection and Storage of Samples

3.1. Spiked QC samples are stored at room temperature pending analysis. A study performed between 1989 and 1990 showed no appreciable difference in analyte recovery between samples stored at room temperature and those stored in the refrigerator.

3.2. There is no holding time requirement for the analysis of PCB on swipe samples.

4. Apparatus

4.1. Syringes: gas-tight, 25-, 50- and 100-μL.

4.2. Bottles: 1/2-oz, glass with Teflon-lined screw-on lids.

4.3. Gauze pads: 2 × 2 in.

4.4. Vials: 1.5-mL, glass.

4.5. Caps: open-top, to fit 1.5-mL vials.

4.6. Septa: Teflon/silicone discs to fit open-top caps.

4.7. Teflon tape.

4.8. Analytical balance: 100-g minimum capacity, 0.1-mg accuracy.

4.9. Pipettes: Pasteur, disposable glass.

4.10. Pipette bulb: rubber, to fit Pasteur pipette.

4.11. Stirring rod: glass, 3- to 4-mm diameter.

4.12. Volumetric flasks: 5-mL, class A, glass.

4.13. Labels printed with QC sample numbers.

5. Reagents

5.1. Hexane (analytical-grade).

6. Calibration and Standards

6.1. EPA Aroclor standards in isooctane or methanol are available in concentrations between 500 and 3000 μg/mL.

6.2. After opening the heat-sealed ampul containing an Aroclor standard; transfer the contents into vials fitted with screw caps with Teflon septa. Wrap the caps with Teflon tape. Remove the original label and affix it to the vial or transfer the standard information to a label on the vial.

6.3. Solutions of PCBs may be prepared from pure PCBs in concentrations of 50-60 mg/mL.

6.4. Preparation of in-house standards in hexane.

6.4.1. Tare a 5-mL volumetric flask on an analytical balance. Add approximately 250-300 mg of a pure PCB to the flask using a glass Pasteur pipette or a glass stirring rod. Record the mass added.

6.4.2. Dissolve the PCB and dilute it to volume with hexane. Mix by inversion.

6.4.3. Prepare a separate standard solution for each PCB desired. Do not mix Aroclors.

6.4.4. The standard concentration will be 50-60 mg/mL. See Step 8.1.

6.4.5. Transfer the standard to vials fitted with Teflon septa and caps. Wrap Teflon tape over and around the caps.

6.4.6. Label the vials with information identifying the standard.

6.5. Prepare a 1:100 dilution of the in-house standard in hexane.

6.5.1. Add 4 mL of hexane to a clean 5-mL volumetric flask.

6.5.2. Using a syringe, add 50 μL of a 50-60 mg/mL Aroclor in-house standard prepared in Step 6.4 to the hexane in the flask.

6.5.3. Dilute the standard to volume with hexane. Mix by inversion.

6.5.4. The concentration of the dilution will be 0.5-0.6 mg/mL or 500-600 μg/mL. See Step 8.2.

6.5.5. Transfer the standard to vials fitted with Teflon septa and caps. Wrap Teflon tape over and around the caps.

6.5.6. Label the vials with information identifying the standard.

6.6. Store the stock standards in a freezer.

7. Procedure

7.1. Discard used, PCB-contaminated flasks, bottles, and pipettes in the PCB waste container.

7.2. Cleaning of syringes.

7.2.1. Dedicate a group of syringes for use only with PCBs.

7.2.2. Fill the syringe with methanol through the needle. Discard the methanol into a waste container. Repeat for a total of 5-10 times.

7.2.3. Dry the syringe by pulling a vacuum on it for at least 10 min.

7.3. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed.

7.3.1. Remove a vial containing a standard of the desired Aroclor in the concentration range 500-1000 μg/mL from the freezer and allow it to reach room temperature.

7.3.2. Place a 2- × 2-in. blank gauze pad into a clean 1/2-oz bottle fitted with a Teflon-lined cap.

7.3.3. Spike an aliquot of PCB standard directly onto the gauze surface to reach the desired spike range. The analyst will desorb the sample in the bottle.

7.3.4. Cap the vial and label it with a QC number.

7.3.5. Return the vial containing the Aroclor standard to the freezer.

7.3.6. Store the QC samples at room temperature pending analysis.

7.4. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD databases on the VAX.

8. Calculations

8.1. Calculate the concentration of the concentrated PCB standard prepared from pure PCBs as in the following example.

8.1.1. If 275.0 mg of pure PCB is diluted to 5 mL with hexane, the concentration of the PCB in the standard is 55 mg/mL.

$$\frac{275.0 \text{ mg}}{5 \text{ mL}} = 55 \text{ mg/mL}$$

8.2. Calculate the concentration of the 1:100 dilution of the concentrated PCB standard as in the following example.

8.2.1. If 50 μL of a PCB standard whose concentration is 55 mg/mL is diluted to 5 mL with hexane, the concentration of the dilute standard is 550 μg/mL.

$$\frac{(55 \text{ mg/mL}) \left(\frac{1 \text{ mL}}{1000 \ \mu\text{L}}\right) (50 \ \mu\text{L}) \left(\frac{1000 \ \mu\text{g}}{1 \text{ mg}}\right)}{5 \text{ mL}} = 550 \ \mu\text{g/mL}$$

8.3. Calculate the concentration of PCB spiked onto a gauze sample as in the following example.

8.3.1. If 24 μL of a 550 μg/mL PCB standard is spiked onto a gauze square, the PCB concentration in the QC sample is 13 μg/sample. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(550 \ \mu\text{g/mL}) \left(\frac{1 \text{ mL}}{1000 \ \mu\text{L}}\right) (24 \ \mu\text{L})}{\text{sample}} = 13 \ \mu\text{g/sample}$$

8.4. Record Aroclor concentrations in μg/sample.

9. Proper Waste Disposal Practices

9.1. Solid Waste

9.1.1. PCB-contaminated solid waste, such as paper wipes, glass or plastic pipettes, empty vials, and incorrectly spiked QC samples, is accumulated in a covered metal can lined with a plastic bag and is kept segregated from non-PCB-contaminated solid waste.

9.1.2. The can is labeled with a hazardous waste label and a label indicating its use for PCB-contaminated waste.

9.1.3. The can is opened only for the period of time necessary to add waste to it.

9.1.4. When the plastic bag is full, it is sealed with tape, removed from the can, identified as PCB-contaminated waste, and placed in a functioning chemical exhaust hood pending pickup by the Waste Management Group.

9.1.5. A new plastic bag is put into the waste can for continued use.

9.2. Liquid Waste

9.2.1. PCB-contaminated liquid waste, such as rinses from PCB-contaminated syringes, PCB standards, and incorrectly spiked QC samples, is accumulated in a covered polyethylene bottle in a secondary containment tray and is kept segregated from non-PCB-contaminated liquid waste.

9.2.2. The bottle is labeled with a hazardous waste label and a label indicating its use for PCB-contaminated waste.

9.2.3. The bottle is opened only for the period of time necessary to add waste to it.

9.2.4. When the bottle is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.5. A new bottle is labeled for further use.

9.3. Waste Pickup

9.3.1. Pickup by the Waste Management Group of a full bag or bottle of PCB-contaminated waste is requested using the current Chemical Waste Disposal Request form. The current Waste Profile Request Form

(WPRF) that describes the waste is referenced on the Chemical Waste Disposal Request.

9.3.2. The Waste Management Group picks up the waste for disposal in accordance with Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.3. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Environmental Chemistry: 1991," Los Alamos National Laboratory report LA-12436-MS (1992).

10.4. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.5. "Polychlorinated Biphenyls," Administrative Requirement 10-4, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

SEMIVOLATILE ORGANIC COMPOUNDS ON SOIL — QC SAMPLE PREPARATION

Analyte: Semivolatile organic compounds	**Method No.** QCO210
Matrix: Soil	**Spike Range:** 0.67 - 6.67 mg/kg
Procedure: Gas chromatography and mass spectrometry	**Accuracy and Precision:** 100% ± 5.2% RSD
Effective Date: 01/22/90	**Authors:** Nancy L. Koski Margaret A. Gautier Carol R. Sutcliffe

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.3 for proper waste disposal practices.

1. Principle of Method

1.1. Prepare stock solutions in acetone of 4-10 semivolatile compounds from the list in Table I.

1.2. Spike the stocks onto 30 g of blank soil.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the volumetric flask, microsyringe, and balance. A relative standard deviation (RSD) of 5.2% has been calculated for the error of the standard and for spiking, weighing, and dilution, based on the following:

$$D = \frac{M1 \times M2 \times M4}{M3 \times M5},$$

where D = calculated result,
M1 = standard error,
M2 = weighing error,
M3 = dilution error,
M4 = spiking error, and
M5 = weighing error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3} + R^2_{M4} + R^2_{M5}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of balance,
R^2_{M3} = relative variance of volumetric flask,
R^2_{M4} = relative variance of microsyringe,
R^2_{M5} = relative variance of balance, and
100 = factor to convert to percentage.

2.2. Analysis by GCMS of QC samples prepared between 1990 and 1991 using this procedure are summarized and tabulated below. The data are published in Source Materials 10.1 and 10.2.

SEMIVOLATILE ORGANIC COMPOUNDS ON SOIL SPIKE RECOVERY DATA

Compound	1990 Mean ± SD	(n)	1991 Mean ± SD	(n)
Acenaphthene	—		62	(1)
Acenaphthylene	—		—	
Aniline	10 ± 4	(3)	—	
Anthracene	80 ± 28	(12)	85 ± 10	(8)
Azobenzene	—		—	
Benzo[a]anthracene	—		—	
Benzo[b]fluoranthene	98	(2)	78 ± 9	(3)
Benzo[k]fluoranthene	—		—	

SEMIVOLATILE ORGANIC COMPOUNDS ON SOIL
SPIKE RECOVERY DATA (cont.)

Compound	1990 Mean	± SD	(n)	1991 Mean	± SD	(n)
Benzoic acid	50	± 37	(8)	20	± 7	(3)
Benzo[g,h,i]perylene	—			—		
Benzo[a]pyrene	—			60		(1)
Benzyl alcohol	62	± 16	(4)	73	± 37	(8)
Bis(2-chloroethoxy)methane	—			—		
Bis(2-chloroethyl)ether	67	± 9	(5)	79	± 12	(4)
Bis(2-chloroisopropyl)ether	—			—		
Bis(2-ethylhexyl)phthalate	—			—		
4-Bromophenyl phenylether	—			—		
Butyl benzyl phthalate	—			—		
4-Chloroaniline	—			—		
4-Chloro-3-methylphenol	—			76	± 16	(5)
2-Chloronaphthalene	—			58	± 12	(6)
2-Chlorophenol	—			58	± 10	(3)
4-Chlorophenyl phenyl ether	—			—		
Chrysene	—			135	± 60	(4)
Di-n-butyl phthalate	—			67	± 4	(6)
Di-n-octyl phthalate	—			124	± 91	(3)
Dibenzo[a,h]anthracene	—			—		
Dibenzofuran	—			—		
1,2-Dichlorobenzene	—			94	± 11	(11)
1,3-Dichlorobenzene	—			—		
1,4-Dichlorobenzene	9		(1)	57	± 9	(4)
2,4-Dichlorophenol	66		(2)	62	± 14	(6)
Diethyl phthalate	111	± 33	(12)	69		(1)
2,4-Dimethylphenol	35		(2)	58	± 12	(5)
Dimethyl phthalate	78		(2)	—		
4,6-Dinitro-2-methylphenol	—			—		
2,4-Dinitrophenol	—			—		
2,4-Dinitrotoluene	—			88		(1)
2,6-Dinitrotoluene	—			68		(1)
Fluoranthene	—			93	± 12	(8)
Fluorene	—			—		
Hexachlorobenzene	54		(1)	—		
Hexachlorobutadiene	59	± 18	(12)	66	± 18	(8)
Hexachlorocyclopentadiene	36		(1)	64		(2)
Hexachloroethane	9		(1)	51	± 27	(4)
Indeno[1,2,3-cd]pyrene	—			—		
Isophorone	64	± 18	(11)	—		
2-Methylnaphthalene	—			—		

SEMIVOLATILE ORGANIC COMPOUNDS ON SOIL
SPIKE RECOVERY DATA (cont.)

Compound	1990 Mean	± SD	(n)	1991 Mean	± SD	(n)
2-Methylphenol	—			—		
4-Methylphenol	—			—		
Naphthalene	55	± 20	(3)	55	± 5	(3)
2-Nitroaniline	—			—		
3-Nitroaniline	—			23	± 11	(3)
4-Nitroaniline	39	± 23	(3)	31	± 13	(4)
Nitrobenzene	59		(2)	—		
2-Nitrophenol	—			—		
4-Nitrophenol	77	± 32	(5)	75	± 15	(11)
N-Nitrosodiphenylamine	—			77		(1)
N-Nitrosodi-n-propylamine	—			—		
Pentachlorophenol	—			—		
Phenanthrene	89		(2)	—		
Phenol	64	± 9	(6)	—		
Pyrene	—			—		
1,2,4-Trichlorobenzene	—			—		
2,4,5-Trichlorophenol	74	± 11	(3)	—		
2,4,6-Trichlorophenol	—			—		

3. Collection and Storage of Samples

3.1. The blank soil matrix and the spiked samples should be stored in glass bottles fitted with Teflon-lined lids. Avoid using plastic containers and lids because phthalates, which are semivolatile compounds, may leach from plastics and contaminate the sample.

3.2. Spiked QC samples are refrigerated pending analysis. The maximum holding time for the analysis of semivolatile compounds in soil is 14 days.

4. Apparatus

4.1. Volumetric flask: 5-mL, class A, glass.

4.2. Syringes: gas-tight, 25-, 50-, 100-, and 250-μL.

4.3. Spatulas.

4.4. Pipettes: Pasteur, glass.

4.5. Pipette bulb: rubber, to fit Pasteur pipette.

4.6. Vials: 1.5-mL, glass.

4.7. Caps: open-top, to fit vials.

4.8. Septa: Teflon/silicone discs to fit open-top caps.

4.9. Teflon tape.

4.10. Bottles: 2-oz, glass, with Teflon-lined caps.

4.11. Analytical balance: 200-g minimum capacity, 0.1-mg accuracy.

4.12. Laboratory oven: capable of sustaining 100-150°C.

4.13. Labels printed with QC sample numbers.

5. Reagents

5.1. Soil. A clean, dry 30-g aliquot is required for each sample. A suitable soil does not contain the analyte of interest and presents few interferences to the analytical method.

5.2. Blank soil, heated to nominally 100°C for 5 h and stored in glass, is used as a sample matrix.

5.3. Acetone (analytical-grade).

5.4. Methanol (analytical-grade).

5.5. Methylene chloride (analytical-grade).

6. Calibration and Standards

6.1. Prepare a stock solution of neat, standard-grade semivolatile analytes chosen from Table I. Use Chem Service or equivalent chemicals (Chem Service, Inc., Box 3108, West Chester, PA 19387). Use both acid and base/neutral semivolatile analytes because the two fractions are extracted separately before being recombined for analysis. Add solid analytes first, then liquid analytes after the addition of solvent.

6.1.1. Add 8-12 mg of a solid semivolatile analyte to a clean, tared 5.0-mL volumetric flask. Record the mass and tare the balance. Repeat for each solid analyte.

6.1.2. Add approximately 2 mL of acetone to the flask, stopper the flask, and swirl it to see if all analytes will dissolve. If not, methylene chloride and/or methanol may be added to effect total dissolution. For example,

chrysene requires methylene chloride and methanol and gentle heating to effect total dissolution.

6.1.3. Place the stoppered flask on the analytical balance and tare the balance.

6.1.4. Calculate the volumes of liquid analytes needed to reach a weight spike range of 8-12 mg in 5 mL of stock solution. See Step 8.2.

6.1.5. Add a liquid analyte from a syringe just above the surface of the acetone. Stopper the flask and record the mass and volume of the analyte added. Tare the balance. Repeat for each liquid analyte.

6.1.6. Dilute the solution to volume with acetone. Mix by inversion.

6.1.7. Discard the solution in the neck of the flask to minimize concentration changes due to loss of analytes from the surface of the solution.

6.1.8. The concentration of analytes in the stock solution will be 1.5-2.5 mg/mL.

6.1.9. Transfer the stock solution to vials fitted with Teflon septa and caps. Wrap Teflon tape over and around the caps.

6.1.10. Label the vials with information identifying the standard and with the expiration date.

6.1.11. The standard solution in acetone is stable for approximately three months stored at -20°C.

6.2. Prepare additional stock solution using different analytes from Table I. The number of analytes in the QC sample may be increased by using more than one stock solution to spike the sample.

7. Procedure

7.1. Cleaning glassware.

7.1.1. Wash volumetric flasks with detergent and rinse with hot tap water and then with methanol.

7.1.2. Store glassware in a laboratory oven maintained at 100-150°C to minimize contamination.

7.2. Cleaning syringes.

7.2.1. Dedicate a group of syringes for use only with semivolatile compounds.

7.2.2. Fill the syringe through the needle with methanol. Discard the methanol into a waste container. Repeat for a total of 5-10 times.

7.2.3. Dry the syringe by pulling a vacuum on it for approximately 10 min.

7.3. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed.

7.3.1. Remove a vial of semivolatile stock solution from the freezer and allow it to come to room temperature. Do not let the vial sit at room temperature for longer than 2 h before spiking the QC sample.

7.3.2. Accurately weigh 30 g of blank soil into a 2-oz glass bottle. Record the mass of soil on the bottle with a felt-tipped pen for reference by the analyst.

7.3.3. Using a gas-tight syringe, spike a volume of the standard onto the surface of the soil to reach the desired spike range. Blank QC samples may be prepared by spiking acetone onto the soil. The analyst will use the entire sample.

7.3.4. Cap the vial with a Teflon-lined lid and label it with a QC number.

7.3.5. Return the stock solution to the freezer.

7.3.6. Refrigerate the QC sample pending analysis. The maximum holding time for semivolatile organic compounds on soil QC samples is 14 days.

7.4. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD databases on the VAX.

8. Calculations

8.1. Use 8-12 mg of a solid analyte in 5.0 mL of stock solution.

8.2. Calculate the volume of neat liquid analyte needed to prepare a stock solution in the range of 8-12 mg/5 mL.

8.2.1. Read the density of the neat liquid analyte from Table I. The density of nitrobenzene is 1.196 mg/μL.

8.2.2. The desired weight spike range for 5 mL of stock solution is 8-12 mg of neat analyte.

8.2.3. Divide the weight spike range by the density of the neat analyte to determine the desired volume spike range of neat analyte to use in 5 mL of stock solution.

$$\frac{8\ \text{mg}}{1.196\ \text{mg}/\mu\text{L}}\ \text{to}\ \frac{12\ \text{mg}}{1.196\ \text{mg}/\mu\text{L}} = 6.7\ \text{to}\ 10.0\ \mu\text{L}$$

For nitrobenzene, between 6.7 and 10 μL of the neat analyte should be diluted to 5 mL to reach the desired concentration range of 1.5-2.5 mg/mL in the stock solution.

8.3. Calculate the concentration of each analyte in the stock solution as in the following examples.

8.3.1. If 9.6 mg of solid anthracene is dissolved in 5.0 mL of stock solution, the concentration of anthracene in the stock solution is 1.92 mg/mL.

$$\frac{9.6\ \text{mg}}{5.0\ \text{mL}} = 1.92\ \text{mg/mL}$$

8.3.2. If 9 μL of liquid nitrobenzene, weighing 10.8 mg, is diluted in 5 mL of stock solution, the concentration of nitrobenzene in the stock solution is 2.16 mg/mL.

$$\frac{10.8\ \text{mg}}{5\ \text{mL}} = 2.16\ \text{mg/mL}$$

8.4. Calculate the concentration of each analyte in the QC sample as in the following example.

8.4.1. If 60 μL of a stock standard solution containing 1.92 mg/mL of anthracene and 2.16 mg/mL of nitrobenzene is spiked onto 30.0018 g of blank soil, the concentration of anthracene in the QC sample will be 3.84 mg/kg. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(1.92\ \text{mg/mL})\ (60\ \mu\text{L})\left(\frac{1\ \text{mL}}{1000\ \mu\text{L}}\right)}{30.0018\ \text{g}\left(\frac{1\ \text{kg}}{1000\ \text{g}}\right)} = 3.84\ \text{mg/kg}$$

The concentration of nitrobenzene in the QC sample will be 4.32 mg/kg. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(2.16\ \text{mg/mL})\ (60\ \mu\text{L})\left(\frac{1\ \text{mL}}{1000\ \mu\text{L}}\right)}{30.0018\ \text{g}\left(\frac{1\ \text{kg}}{1000\ \text{g}}\right)} = 4.32\ \text{mg/kg}$$

8.5. Record the analyte concentrations in the QC sample in mg/kg or μg/g.

9. Proper Waste Disposal Practices

9.1. Solid Waste

9.1.1. Solid waste, such as paper wipes, glass or plastic pipettes, empty vials, and incorrectly spiked QC samples contaminated with organic compounds, is accumulated in a covered metal can lined with a plastic bag and is kept segregated from PCB-contaminated solid waste and solid waste that is not contaminated with organic compounds.

9.1.2. The can is labeled with a hazardous waste label and a label indicating its use for waste contaminated with organic compounds.

9.1.3. The can is opened only for the period of time necessary to add waste.

9.1.4. When the plastic bag is full, it is sealed with tape, removed from the can, identified as waste contaminated with organic compounds, and placed in a functioning chemical exhaust hood or in a closed secondary container pending pickup by the Waste Management Group.

9.1.5. A new plastic bag is put into the waste can for continued use.

9.2. Liquid Waste

9.2.1. Liquid waste containing organic compounds, such as rinses from syringes, excess QC samples, and incorrectly spiked QC samples, is accumulated in a covered metal can in a secondary containment tray.

9.2.1.1. Liquid waste containing organic compounds but no PCBs is kept segregated from PCB-contaminated liquid waste.

9.2.1.2. The can is labeled with a hazardous waste label and a label indicating its use for waste containing organic compounds.

9.2.1.3. When the can is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.1.4. A new can is labeled for future use.

9.2.2. Two-mL vials containing out-of-date spiking solutions of organic compounds are accumulated in a glass jar in a secondary containment tray.

9.2.2.1. Solutions that are not contaminated with PCBs are kept segregated from those that are PCB-contaminated.

9.2.2.2. The jar is labeled with a hazardous waste label and a label indicating its use for waste containing organic compounds.

9.2.2.3. The jar is opened only for the period of time necessary to add vials.

9.2.2.4 When the jar is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.2.5. A new jar is labeled for future use.

9.3. Waste Pickup

9.3.1. Pickup by the Waste Management Group of a full bag, can, or jar of waste contaminated with organic compounds is requested using the current Chemical Waste Disposal Request form. The current Waste Profile Request Form (WRPF) that describes the waste is referenced on the Chemical Waste Disposal Request.

9.3.2. The Waste Management Group picks up the waste for disposal according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Environmental Chemistry: 1991," Los Alamos National Laboratory report, LA-12436-MS (1992).

10.3. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

TABLE I. SEMIVOLATILE ORGANIC COMPOUNDS

Analyte	CAS No.	Category	Density of liquid, (mg/μL)
acenaphthene	83329	Base/Neutral	solid
acenaphthylene	208968	Base/Neutral	0.899
aniline	62533	Base/Neutral	1.022
anthracene	120127	Base/Neutral	solid
azobenzene	103333	Base/Neutral	1.090
benz[a]anthracene	56553	Base/Neutral	solid
benzo[b]fluoranthene	205992	Base/Neutral	solid
benzo[k]fluoranthene	207089	Base/Neutral	solid
benzoic acid	65850	Acid	solid
benzo[g,h,i]perylene	191242	Base/Neutral	solid
benzo[a]pyrene	50328	Base/Neutral	solid
benzyl alcohol	100516	Base/Neutral	1.045
bis(2-chloroethoxy)methane	111911	Base/Neutral	solid
bis(2-chloroethyl)ether	111444	Base/Neutral	1.220
bis(2-chloroisopropyl)ether	108601	Base/Neutral	solid
bis(2-ethylhexyl) phthalate	117817	Base/Neutral	0.981
4-bromophenyl phenyl ether	101553	Base/Neutral	1.423
butyl benzyl phthalate	85687	Base/Neutral	0.966
4-chloroaniline	106478	Base/Neutral	solid
4-chloro-3-methylphenol	59507	Acid	solid
2-chloronaphthalene	91587	Base/Neutral	solid
2-chlorophenol	95578	Acid	1.241
4-chlorophenyl phenyl ether	7005723	Base/Neutral	solid
chrysene	218019	Base/Neutral	solid
di-n-butyl phthalate	84742	Base/Neutral	1.043
di-n-octyl phthalate	117840	Base/Neutral	0.981
dibenz[a,h]anthracene	53703	Base/Neutral	solid
dibenzofuran	132649	Base/Neutral	solid
1,2-dichlorobenzene	95501	Base/Neutral	1.306
1,3-dichlorobenzene	541731	Base/Neutral	1.288
1,4-dichlorobenzene	106467	Base/Neutral	1.241
2,4-dichlorophenol	120832	Acid	solid
diethyl phthalate	84662	Base/Neutral	1.118
2,4-dimethylphenol	105679	Acid	1.027
dimethyl phthalate	131113	Base/Neutral	1.190
4,6-dinitro-2-methylphenol	534521	Acid	solid
2,4-dinitrophenol	51285	Acid	solid

TABLE I. SEMIVOLATILE ORGANIC COMPOUNDS (cont)

Analyte	CAS No.	Category	Density of liquid, (mg/μL)
2,4-dinitrotoluene	121142	Base/Neutral	solid
2,6-dinitrotoluene	606202	Base/Neutral	solid
fluoranthene	206440	Base/Neutral	solid
fluorene	86737	Base/Neutral	solid
hexachlorobenzene	118741	Base/Neutral	solid
hexachlorobutadiene	87683	Base/Neutral	1.665
hexachlorocyclopentadiene	77474	Base/Neutral	1.702
hexachloroethane	67721	Base/Neutral	2.091
indeno[1,2,3-cd]pyrene	193395	Base/Neutral	solid
isophorone	78591	Base/Neutral	0.923
2-methylnaphthalene	91576	Base/Neutral	1.000
2-methylphenol	95487	Acid	1.048
4-methylphenol	106445	Acid	1.034
naphthalene	91203	Base/Neutral	solid
2-nitroaniline	88744	Base/Neutral	solid
3-nitroaniline	99092	Base/Neutral	solid
4-nitroaniline	100016	Base/Neutral	solid
nitrobenzene	98953	Base/Neutral	1.196
2-nitrophenol	88755	Acid	solid
4-nitrophenol	100027	Acid	solid
N-nitrosodiphenylamine	86306	Base/Neutral	solid
N-nitrosodi-n-propylamine	621647	Base/Neutral	solid
pentachlorophenol	87865	Acid	1.978
phenanthrene	85018	Base/Neutral	solid
phenol	108952	Acid	solid
pyrene	129000	Base/Neutral	solid
1,2,4-trichlorobenzene	120821	Base/Neutral	1.454
2,4,5-trichlorophenol	95954	Acid	solid
2,4,6-trichlorophenol	88062	Acid	solid

SEMIVOLATILE ORGANIC COMPOUNDS IN WATER — QC SAMPLE PREPARATION

Analyte: Semivolatile organic compounds	**Method No.:** QCO220
Matrix: Water	**Spike Range:** 20-200 μg/L
Procedure: Gas chromatography and mass spectrometry (GCMS)	**Accuracy and Precision:** 100% ± 5.2% RSD
Effective Date: 01/01/90	**Authors:** Nancy L. Koski Margaret A. Gautier Carol Sutcliffe

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.3 for proper waste disposal practices.

1. Principle of Method

1.1. Prepare stock solutions of 4-10 semivolatile compounds in acetone from the list in Table I.

1.2. Spike the stocks into 1 L of distilled water.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with use of the microsyringe, balance, and volumetric flask. A relative standard deviation (RSD) of 5.2% has been calculated for the error of the standard and for spiking, weighing, and dilution based on the following:

$$D = \frac{M1 \times M2 \times M4}{M3 \times M5},$$

where D = calculated result,
M1 = standard error,
M2 = weighing error,
M3 = dilution error,
M4 = pipette error, and
M5 = second dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots . M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$\%S_{D_T} = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3} + R^2_{M4}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of balance,
R^2_{M3} = relative variance of volumetric flask,
R^2_{M4} = relative variance of microsyringe,
R^2_{M5} = relative variance of second volumetric flask, and
100 = factor to convert to percentage.

2.2. Analyses by GCMS of QC samples prepared between 1990 and 1991 using this procedure are tabulated below. The data are published in Source Materials 10.1 and 10.2.

SEMIVOLATILE ORGANIC COMPOUNDS IN WATER SPIKE RECOVERY DATA

Compound	1990 Mean ± SD	(n)	1991 Mean ± SD	(n)
Acenaphthene	—		84 ± 18	(4)
Acenaphthylene	—		—	
Aniline	84 ± 19	(4)	88	(1)
Anthracene	58 ± 15	(5)	—	
Azobenzene	—		—	
Benzo[a]anthracene	—		—	

SEMIVOLATILE ORGANIC COMPOUNDS IN WATER
SPIKE RECOVERY DATA (cont)

Compound	1990 Mean	± SD	(n)	1991 Mean	± SD	(n)
Benzo[b]fluoranthene	—			85		(2)
Benzo[k]fluoranthene	—			—		
Benzoic acid	54	± 35	(9)	57	± 43	(4)
Benzo[g,h,i]perylene	—			—		
Benzo[a]pyrene	—			40		(2)
Benzyl alcohol	53	± 29	(4)	79	± 22	(6)
Bis(2-chloroethoxy)methane	—			—		
Bis(2-chloroethyl)ether	63	± 7	(4)	60	± 19	(4)
Bis(2-chloroisopropyl)ether	—			—		
Bis(2-ethylhexyl)phthalate	—			—		
4-Bromophenyl phenyl ether		—			—	
Butyl benzyl phthalate	—			—		
4-Chloroaniline	—			—		
4-Chloro-3-methylphenol	—			67	± 29	(4)
2-Chloronaphthalene	—			60		(2)
2-Chlorophenol	—			60	± 15	(4)
4-Chlorophenyl phenyl ether	—			—		
Chrysene	—			16		(1)
Di-n-butyl phthalate	—			29		(2)
Di-n-octyl phthalate	—			66	± 15	(4)
Dibenzo[a,h]anthracene	—			—		
Dibenzofuran	—			—		
1,2-Dichlorobenzene	—			—		
1,3-Dichlorobenzene	—			—		
1,4-Dichlorobenzene	—			31		(2)
2,4-Dichlorophenol	58		(2)	67		(2)
Diethyl phthalate	46	± 56	(4)	15		(2)
2,4-Dimethylphenol	—			70	± 15	(6)
Dimethyl phthalate	—			—		
4,6-Dinitro-2-methylphenol	—			—		
2,4-Dinitrophenol	—			—		
2,4-Dinitrotoluene	—			—		
2,6-Dinitrotoluene	—			42	± 25	(4)
Fluoranthene	—			78	± 19	(6)
Fluorene	—			—		
Hexachlorobenzene	64	± 6	(3)	—		
Hexachlorobutadiene	47	± 6	(5)	50	± 6	(4)
Hexachlorocyclopentadiene	48		(2)	46	± 12	(4)
Hexachloroethane	48		(2)	—		
Indeno[1,2,3-cd]pyrene	—			—		

SEMIVOLATILE ORGANIC COMPOUNDS IN WATER
SPIKE RECOVERY DATA (cont)

Compound	1990 Mean ± SD	(n)	1991 Mean ± SD	(n)
Isophorone	69 ± 13	(5)	—	
2-Methylnaphthalene	—		—	
2-Methylphenol	—		—	
4-Methylphenol	—		—	
Naphthalene	—		58	(2)
2-Nitroaniline	—		—	
3-Nitroaniline	—		70	(2)
4-Nitroaniline	—		80 ± 17	(4)
Nitrobenzene	—		—	
2-Nitrophenol	—		—	
4-Nitrophenol	85 ± 23	(6)	62 ± 20	(8)
N-Nitrosodiphenylamine	—		42 ± 27	(4)
N-Nitrosodi-n-propylamine	—		—	
Pentachlorophenol	92 ± 27	(6)	—	
Phenanthrene	64	(2)	—	
Phenol	49 ± 14	(4)	66	(2)
Pyrene	—		—	
1,2,4-Trichlorobenzene	—		—	
2,4,5-Trichlorophenol	74 ± 17	(10)	—	
2,4,6-Trichlorophenol	—		—	

3. Collection and Storage of Samples

3.1. Distilled water is used as the blank matrix. Use water that has not come into contact with plastic tubing or plastic holding vessels because phthalates, which are semivolatile organic compounds, may leach from plastics and contaminate the sample.

3.2. Spiked QC samples are refrigerated pending analysis. The maximum holding time for semivolatiles in water is seven days.

4. Apparatus

4.1. Volumetric flasks: 5-mL, class A, glass.

4.2. Syringes: gas-tight, 25-, 50-, 100-, and 250-μL.

4.3. Spatulas.

4.4. Pipettes: Pasteur, glass.

4.5. Pipette bulb: rubber, to fit Pasteur pipette.

4.6. Vials: 1.5-mL, glass.

4.7. Caps: open-top, to fit vials.

4.8. Septa: Teflon/silicone discs to fit open-top caps.

4.9. Teflon tape.

4.10. Bottles: 1-L wide-mouth glass jars, amber, pre-cleaned, with Teflon-lined lids.

4.11. Analytical balance: 100-g minimum capacity, 0.1-mg accuracy.

4.12. Laboratory oven: capable of sustaining 100-150°C.

4.13. Labels: printed with QC sample numbers.

5. Reagents

5.1. Acetone (analytical-grade).

5.2. Methanol (analytical-grade).

5.3. Methylene chloride (analytical-grade).

6. Calibration and Standards

6.1. Prepare a stock solution of neat, standard-grade semivolatile analytes chosen from Table I. Use Chem Service or equivalent chemicals (Chem Service, Inc., Box 3108, West Chester, PA 19381). Use both acid and base/neutral semivolatile analytes because the two fractions are extracted separately before being recombined for analysis. Add solid analytes first, then liquid analytes after the addition of solvent.

6.1.1. Add 8-12 mg of a solid semivolatile analyte to a clean, tared 5.0-mL volumetric flask. Record the mass and tare the balance. Repeat for each solid analyte.

6.1.2. Add approximately 2 mL of acetone to the flask, stopper the flask, and swirl it to see if all analytes will dissolve. If not, methylene chloride and/or methanol may be added to effect total dissolution. For example, chrysene requires methylene chloride and methanol and gentle heating to effect total dissolution.

6.1.3. Place the stoppered flask on the analytical balance and tare the balance.

6.1.4. Calculate the volumes of liquid analytes needed to reach a weight spike range of 8-12 mg in 5 mL of stock solution. See Step 8.2.

6.1.5. Add a liquid analyte from a syringe just above the surface of the acetone. Stopper the flask and record the mass and volume of analyte added. Tare the balance. Repeat for each liquid analyte added.

6.1.6. Dilute the solution to volume with acetone. Mix by inversion.

6.1.7. Discard the solution in the neck of the flask to minimize concentration changes due to loss of analytes from the surface of the solution.

6.1.8. The concentration of analytes in the stock solution will be 1.5-2.5 mg/mL.

6.1.9. Transfer the stock solution to vials fitted with Teflon septa and caps. Wrap Teflon tape over and around the caps.

6.1.10. Label the vials with information identifying the standard and with the expiration date.

6.1.11. The stock solution in acetone is stable for approximately three months stored at -20°C.

6.2. Prepare additional stock solutions using different analytes from Table I. The number of analytes in the QC sample may be increased by using more than one solution to spike the sample.

7. Procedure

7.1. Cleaning glassware.

7.1.1. Wash volumetric flasks with detergent and rinse with hot tap water and then with methanol.

7.1.2. Store glassware in a laboratory oven maintained at 100-150°C to minimize contamination.

7.2. Cleaning syringes.

7.2.1. Dedicate a group of syringes for use only with semivolatile compounds.

7.2.2. Fill the syringe through the needle with methanol. Discard the methanol into a waste container. Repeat for a total of 5-10 times.

7.2.3. Dry the syringe by pulling a vacuum on it for approximately 10 min.

7.3. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed.

7.3.1. Remove a vial of semivolatile stock solution from the freezer and allow it to come to room temperature. Do not let the vial sit at room temperature for longer than 2 h before spiking the QC sample.

7.3.2. Fill a pre-cleaned 1-L amber bottle to the neck with distilled water. Use only distilled water that has not been stored in plastic or run through plastic tubing.

7.3.3. Using a syringe, spike a volume of the solution below the surface of the water to reach the desired spike range. Blank QC samples may be prepared by spiking acetone into the water. The analyst will use the entire sample assuming a 1-L volume.

7.3.4. Cap the bottle and label it with a QC sample number.

7.3.5. Return the stock solution to the freezer.

7.3.6. Refrigerate the QC sample pending analysis. The maximum holding time for semivolatile organic compounds in water QC samples is 7 days.

7.4. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD databases on the VAX.

8. Calculations

8.1. Use 8-12 mg of a solid analyte in 5.0 mL of stock solution.

8.2. Calculate the volume of a neat liquid analyte needed to prepare a stock solution in the range of 8-12 mg/5 mL.

8.2.1. Read the density of the neat liquid analyte from Table I. The density of nitrobenzene is 1.196 mg/μL.

8.2.2. The desired weight spike range for 5 mL of stock solution is 8-12 mg of neat analyte.

8.2.3. Divide the weight spike range by the density of the neat analyte to determine the desired volume spike range of neat analyte to use in 5 mL of stock solution.

$$\frac{8\ \text{mg}}{1.196\ \text{mg}/\mu\text{L}} \text{ to } \frac{12\ \text{mg}}{1.196\ \text{mg}/\mu\text{L}} = 6.7 \text{ to } 10.0\ \mu\text{L}$$

For nitrobenzene, between 6.7 and 10 μL of the neat analyte should be diluted to 5 mL to reach the desired concentration range of 1.5-2.5 mg/mL in the stock solution.

8.3. Calculate the concentration of each analyte in the stock solution as in the following examples.

8.3.1. If 9.6 mg of solid anthracene is dissolved in 5.0 mL of stock solution, the concentration of anthracene in the stock solution is 1.92 mg/mL.

$$\frac{9.6\ \text{mg}}{5.0\ \text{mL}} = 1.92\ \text{mg/mL}$$

8.3.2. If 9 μL of liquid nitrobenzene, weighing 10.8 mg, is diluted in 5 mL of stock solution, the concentration of nitrobenzene in the stock solution is 2.16 mg/mL.

$$\frac{10.8\ \text{mg}}{5\ \text{mL}} = 2.16\ \text{mg/mL}$$

8.4. Calculate the concentration of each analyte in the QC sample as in the following example.

8.4.1. If 60 μL of a stock standard solution containing 1.92 mg/mL of anthracene and 2.16 mg/mL of nitrobenzene is spiked into 1 L of distilled water, the concentration of anthracene in the QC sample will be 115 μg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(1.92\ \text{mg/mL})\ (60\ \mu\text{L})\left(\frac{1\ \text{mL}}{1000\ \mu\text{L}}\right)\left(\frac{1000\ \mu\text{g}}{1\ \text{mg}}\right)}{1\ \text{L}} = 115\ \mu\text{g/L}$$

The concentration of nitrobenzene in the QC sample will be 130 μg/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(2.16\ \text{mg/mL})\ (60\ \mu\text{L})\left(\frac{1\ \text{mL}}{1000\ \mu\text{L}}\right)\left(\frac{1000\ \mu\text{g}}{1\ \text{mg}}\right)}{1\ \text{L}} = 130\ \mu\text{g/L}$$

8.5. Record the analyte concentrations in the QC sample in μg/L.

9. Proper Waste Disposal Practices

9.1. Solid Waste

9.1.1. Solid waste, such as paper wipes, glass or plastic pipettes, empty vials, and incorrectly spiked QC samples contaminated with organic compounds, is accumulated in a covered metal can lined with a plastic bag and is kept segregated from PCB-contaminated solid waste and solid waste that is not contaminated with organic compounds.

9.1.2. The can is labeled with a hazardous waste label and a label indicating its use for waste contaminated with organic compounds.

9.1.3. The can is opened only for the period of time necessary to add waste.

9.1.4. When the plastic bag is full, it is sealed with tape, removed from the can, identified as waste contaminated with organic compounds, and placed in a functioning chemical exhaust hood or in a closed secondary container pending pickup by the Waste Management Group.

9.1.5. A new plastic bag is put into the waste can for continued use.

9.2. Liquid Waste

9.2.1. Liquid waste containing organic compounds, such as rinses from syringes, excess QC samples, and incorrectly spiked QC samples, is accumulated in a covered metal can in a secondary containment tray.

9.2.1.1. Liquid waste containing organic compounds but no PCBs is kept segregated from PCB-contaminated liquid waste.

9.2.1.2. The can is labeled with a hazardous waste label and a label indicating its use for waste containing organic compounds.

9.2.1.3. When the can is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.1.4. A new can is labeled for future use.

9.2.2. Two-mL vials containing out-of-date spiking solutions of organic compounds are accumulated in a glass jar in a secondary containment tray.

9.2.2.1. Solutions which are not contaminated with PCBs are kept segregated from those which are PCB-contaminated.

9.2.2.2. The jar is labeled with a hazardous waste label and a label indicating its use for waste containing organic compounds.

9.2.2.3. The jar is opened only for the period of time necessary to add vials.

9.2.2.4 When the jar is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.2.5. A new jar is labeled for future use.

9.3. Waste Pickup

9.3.1. Pickup by the Waste Management Group of a full bag, can, or jar of waste contaminated with organic compounds is requested using the current Chemical Waste Disposal Request form. The current Waste Profile Request Form (WRPF) that describes the waste is referenced on the Chemical Waste Disposal Request.

9.3.2. The Waste Management Group picks up the waste for disposal according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Environmental Chemistry: 1991," Los Alamos National Laboratory report, LA-12436-MS (1992).

10.3. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

TABLE I. SEMIVOLATILE ORGANIC COMPOUNDS

Analyte	CAS No.	Category	Density of liquid, (mg/μL)
acenaphthene	83329	Base/Neutral	solid
acenaphthylene	208968	Base/Neutral	0.899
aniline	62533	Base/Neutral	1.022
anthracene	120127	Base/Neutral	solid
azobenzene	103333	Base/Neutral	1.090
benz[a]anthracene	56553	Base/Neutral	solid
benzo[b]fluoranthene	205992	Base/Neutral	solid
benzo[k]fluoranthene	207089	Base/Neutral	solid
benzoic acid	65850	Acid	solid
benzo[g,h,i]perylene	191242	Base/Neutral	solid
benzo[a]pyrene	50328	Base/Neutral	solid
benzyl alcohol	100516	Base/Neutral	1.045
bis(2-chloroethoxy)methane	111911	Base/Neutral	solid
bis(2-chloroethyl)ether	111444	Base/Neutral	1.220
bis(2-chloroisopropyl)ether	108601	Base/Neutral	solid
bis(2-ethylhexyl) phthalate	117817	Base/Neutral	0.981
4-bromophenyl phenyl ether	101553	Base/Neutral	1.423
butyl benzyl phthalate	85687	Base/Neutral	0.966
4-chloroaniline	106478	Base/Neutral	solid
4-chloro-3-methylphenol	59507	Acid	solid
2-chloronaphthalene	91587	Base/Neutral	solid
2-chlorophenol	95578	Acid	1.241
4-chlorophenyl phenyl ether	7005723	Base/Neutral	solid
chrysene	218019	Base/Neutral	solid
di-n-butyl phthalate	84742	Base/Neutral	1.043
di-n-octyl phthalate	117840	Base/Neutral	0.981
dibenz[a,h]anthracene	53703	Base/Neutral	solid
dibenzofuran	132649	Base/Neutral	solid
1,2-dichlorobenzene	95501	Base/Neutral	1.306
1,3-dichlorobenzene	541731	Base/Neutral	1.288
1,4-dichlorobenzene	106467	Base/Neutral	1.241
2,4-dichlorophenol	120832	Acid	solid
diethyl phthalate	84662	Base/Neutral	1.118
2,4-dimethylphenol	105679	Acid	1.027
dimethyl phthalate	131113	Base/Neutral	1.190
4,6-dinitro-2-methylphenol	534521	Acid	solid
2,4-dinitrophenol	51285	Acid	solid

TABLE I. SEMIVOLATILE ORGANIC COMPOUNDS (cont)

Analyte	CAS No.	Category	Density of liquid, mg/μL
2,4-dinitrotoluene	121142	Base/Neutral	solid
2,6-dinitrotoluene	606202	Base/Neutral	solid
fluoranthene	206440	Base/Neutral	solid
fluorene	86737	Base/Neutral	solid
hexachlorobenzene	118741	Base/Neutral	solid
hexachlorobutadiene	87683	Base/Neutral	1.665
hexachlorocyclopentadiene	77474	Base/Neutral	1.702
hexachloroethane	67721	Base/Neutral	2.091
indeno[1,2,3-cd]pyrene	193395	Base/Neutral	solid
isophorone	78591	Base/Neutral	0.923
2-methylnaphthalene	91576	Base/Neutral	1.000
2-methylphenol	95487	Acid	1.048
4-methylphenol	106445	Acid	1.034
naphthalene	91203	Base/Neutral	solid
2-nitroaniline	88744	Base/Neutral	solid
3-nitroaniline	99092	Base/Neutral	solid
4-nitroaniline	100016	Base/Neutral	solid
nitrobenzene	98953	Base/Neutral	1.196
2-nitrophenol	88755	Acid	solid
4-nitrophenol	100027	Acid	solid
N-nitrosodiphenylamine	86306	Base/Neutral	solid
N-nitrosodi-n-propylamine	621647	Base/Neutral	solid
pentachlorophenol	87865	Acid	1.978
phenanthrene	85018	Base/Neutral	solid
phenol	108952	Acid	solid
pyrene	129000	Base/Neutral	solid
1,2,4-trichlorobenzene	120821	Base/Neutral	1.454
2,4,5-trichlorophenol	95954	Acid	solid
2,4,6-trichlorophenol	88062	Acid	solid

VOLATILE ORGANIC COMPOUNDS ON SOIL — QC SAMPLE PREPARATION

Analyte: Volatile organic compounds	**Method No.:** QCO250
Matrix: Ottawa sand	**Spike Range:** 100-200 μg/kg
Procedure: Analysis by purge and trap extraction, gas chromatography and mass spectrometry (PTGC)	**Accuracy and Precision:** 100% ± 2.0% RSD
Effective Date: 03/19/91	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.4 for proper waste disposal practices.

1. Principle of Method

1.1. Prepare a stock solution in methanol of 3 to 6 volatile organic compounds from the list in Table I.

1.2. Spike the stock solution into methanol.

1.3. The methanol stock dilution is spiked onto the soil matrix immediately before analysis.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with the use of the microsyringe, balance, and volumetric flask. A relative standard deviation (RSD) of 2.0% has been calculated for the error of the standard and for spiking, weighing, and dilution based on the following:

$$D1 = \frac{M1 \times M2}{M3} ,$$

$$D2 = \frac{\left(\frac{D1 \times M5}{(D1 \times M5) + M4}\right) \times M6}{M7} ,$$

$$D3 = \frac{(M3 \times M4)^2}{((M1 \times M2 \times M5) + (M3 \times M4))^2} ,$$

where D1 = first dilution calculated result,
D2 = final calculated result,
D3 = factor for percent relative standard deviation formula in section 2.1.2,
M1 = standard error,
M2 = weighing error,
M3 = dilution error,
M4 = first pipetting error,
M5 = second pipetting error,
M6 = third pipetting error, and
M7 = weighing error.

2.1.1. Calculate relative variance using the following equation:

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots . M_n$,
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation:

$$S_{D_T} = \sqrt{R^2_{M7} + R^2_{M6} + ((R^2_{M1} + R^2_{M2} + R^2_{M3} + R_{M4} + R_{M5}) \times D_3)} \times 100$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of balance,
R^2_{M3} = relative variance of volumetric flask,
R^2_{M4} = relative variance of first microsyringe,

R^2_{M5} = relative variance of second microsyringe,
R^2_{M6} = relative variance of third microsyringe,
R^2_{M7} = relative variance of balance,
D2 = factor above, and
100 = factor to convert to percentage.

2.2. Analytical results from samples prepared between 1990 and 1991 using this procedure and analyzed by purge-and-trap extraction, gas chromatography and mass spectrometry are summarized and tabulated below. The data are published in Source Materials 10.1 and 10.3.

VOLATILE ORGANIC COMPOUNDS ON SOIL RECOVERY DATA

Compound	1990 Mean ± SD	(n)	1991 Mean ± SD	(n)
Acetone	—		44 ± 17	(27)
Benzene	64 ± 30	(4)	34	(1)
Bromobenzene	—		92 ± 18	(14)
Bromochloromethane	52	(1)	—	
Bromodichloromethane	—		103 ± 25	(11)
Bromoform	—		95 ± 26	(9)
Bromomethane	—		—	
2-Butanone (MEK)	110 ± 72	(12)	52 ± 22	(13)
n-Butylbenzene	24	(1)	—	
sec-Butylbenzene	—		—	
tert-Butylbenzene	—		—	
Carbon disulfide	34 ± 14	(3)	37	(1)
Carbon tetrachloride	59 ± 34	(5)	102 ± 48	(11)
Chlorobenzene	53 ± 23	(9)	85 ± 18	(15)
Chlorodibromomethane	57 ± 29	(5)	97 ± 22	(17)
Chloroethane	—		—	
Chloroform	—		79 ± 28	(8)
2-Chlorotoluene	—		88 ± 36	(6)
4-Chlorotoluene	—		—	
1,2-Dibromo-3-chloropropane	—		—	
1,2-Dibromoethane	29	(2)	—	
Dibromomethane	—		82 ± 23	(8)
1,2-Dichlorobenzene	—		94 ± 11	(11)
1,3-Dichlorobenzene	—		—	
1,4-Dichlorobenzene	9	(1)	57 ± 9	(4)
1,1-Dichloroethane	56 ± 21	(8)	26 ± 16	(3)
1,2-Dichloroethane	66 ± 25	(10)	97 ± 42	(14)
1,1-Dichloroethene	—		—	

VOLATILE ORGANIC COMPOUNDS ON SOIL RECOVERY DATA (cont)

Compound	1990 Mean	± SD	(n)	1991 Mean	± SD	(n)
cis-1,2-Dichloroethene	—			—		
trans-1,2-Dichloroethene	—			—		
1,2-Dichloropropane	77		(1)	73	± 38	(7)
1,3-Dichloropropane	—			—		
2,2-Dichloropropane	—			—		
1,1-Dichloropropene	—			—		
cis-1,3-Dichloropropene	36	± 22	(5)	—		
trans-1,3-Dichloropropene	93	± 51	(5)	—		
Ethylbenzene	51	± 24	(11)	76	± 31	(9)
2-Hexanone	138	± 67	(8)	69	± 22	(9)
Isopropylbenzene	—			—		
p-Isopropyltoluene	—			—		
Methylene chloride	—			83	± 53	(27)
4-Methyl-2-pentanone (MIK)	—			94	± 20	(14)
n-Propylbenzene	32		(1)	67	± 22	(5)
Styrene	36		(2)	84	± 22	(14)
1,1,1,2-Tetrachloroethane	57		(1)	85	± 18	(5)
1,1,2,2-Tetrachloroethane	—			104	± 16	(11)
Tetrachloroethene	46	± 24	(12)	75	± 28	(15)
Toluene	53	± 28	(11)	71	± 24	(15)
1,1,1-Trichloroethane	48	± 29	(3)	98	± 45	(15)
1,1,2-Trichloroethane	67	± 32	(10)	86	± 21	(24)
Trichloroethene	42	± 28	(5)	71	± 26	(9)
Trichlorofluoromethane	—			—		
1,2,3-Trichloropropane	—			89	± 15	(5)
1,2,4-Trimethylbenzene	39		(1)	84	± 17	(8)
1,3,5-Trimethylbenzene	—			—		
Vinyl acetate	48	± 43	(5)	68		(2)
Mixed xylenes (total)	35		(1)	80	± 19	(5)

3. Collection and Storage of Samples

3.1. Ottawa sand is used as a blank soil matrix.

3.2. The QC spiking solution is refrigerated pending use. The maximum holding time for the analysis of volatile of organics on soil is 14 days.

4. Apparatus

4.1. Volumetric flasks: 5-mL, class A, glass.

4.2. Syringes: gas-tight, 10-, 25-, 50-, 100-, and 2500-μL.

4.3. Microspatula.

4.4. Pipettes: Pasteur, disposable, glass.

4.5. Pipette bulb: rubber, to fit Pasteur pipette.

4.6. Vials: 1.5-mL, glass, amber.

4.7. Caps: open-top, to fit vials.

4.8. Septa: Teflon/silicone discs to fit open-top caps.

4.9. Teflon tape.

4.10. Autosampler vials with Teflon-lined septa and crimp-tops: 11- x 30-mm vials and 11-mm silicone cap liner with Teflon face.

4.11. Portable crimper for sealing caps on autosampler vials.

4.12. Decapper for removing caps from autosampler vials.

4.13. Analytical balance, 100-g minimum capacity, 0.1-mg accuracy.

4.14. Laboratory oven: capable of sustaining 100-150°C.

4.15. Labels printed with QC sample numbers.

5. Reagents

5.1. Methanol (reagent-grade).

5.2. Ottawa sand: a clean, dry 5.0-g aliquot is required for each sample.

5.2.1. Standard quality Ottawa sand, graded to 20-30 mesh and used for preparing cement tensile-strength samples, meets ASTM C190 specifications.

5.2.2. Ottawa sand heated to nominally 500°C for a minimum of 2 h is used as a sample matrix.

6. Calibration and Standards

6.1. Prepare a stock solution of neat, standard-grade volatile analytes chosen from Table I. Use Chem Service or equivalent chemicals (Chem Service, Inc., Box 3108, West Chester, PA 19381). Add solid analytes first, then liquid analytes after the addition of solvent.

6.1.1. Add 8-12 mg of a solid volatile analyte to a clean, tared 5.0-mL volumetric flask. Record the mass and tare the balance. Repeat for each solid analyte.

6.1.2. Add approximately 3 mL of methanol to the flask, stopper the flask, and swirl it to dissolve the analytes.

6.1.3. Place the stoppered flask on the analytical balance and tare the balance.

6.1.4. Calculate the volumes of liquid analytes needed to reach a weight spike range of 8-12 mg in 5 mL of stock solution. See Step 8.1.

6.1.5. Add a liquid analyte from a gas-tight syringe just above the surface of the methanol. Stopper the flask and record the mass and volume of the analyte added. Tare the balance. Repeat for each liquid analyte added.

6.1.6. Dilute the solution to volume with methanol.

6.1.7. Mix by gently inverting three times.

NOTE: Vigorous shaking will cause the loss of volatile analytes.

6.1.8. Discard the solution in the neck of the flask to minimize concentration changes due to loss of volatile analytes from the surface of the solution.

6.1.9. The concentration of analytes in the stock solution will be between 1.5 - 2.5 mg/mL.

6.1.10. Transfer the stock solution to 1.5-mL amber vials fitted with Teflon septa and caps. Wrap Teflon tape over and around the caps.

6.1.11. Label the vials with information identifying the standard and with the expiration date.

6.1.12. The stock solution is stable for approximately three months when stored at -20°C.

6.2. Prepare additional stock solutions using different analytes from Table I. The number of analytes in the QC samples may be increased by using more than one stock solution to prepare the QC spiking solution.

7. Procedure

7.1. Cleaning glassware.

7.1.1. Wash volumetric flasks with detergent and rinse with hot tap water and then with methanol.

7.1.2. Store the glassware in a laboratory oven maintained at 100-150°C to minimize contamination.

7.2. Cleaning syringes.

7.2.1. Dedicate a group of syringes for use only with volatile analytes.

7.2.2. Fill the syringe through the needle with methanol. Discard the methanol into a waste container. Repeat for a total of 5-10 times.

7.2.3. Dry the syringe by pulling a vacuum on it for at least 10 min.

7.3. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed.

7.3.1. Remove one or more vials of volatile stock standard from the freezer and allow them to reach room temperature. Do not allow the vials to sit at room temperature for longer than 2 h before preparing the QC spiking solution.

7.3.2. Using a gas-tight syringe, add 1500 μL of methanol to an autosampler vial to be used as a matrix for the QC spiking solution.

7.3.3. Seal the vial with a septum and crimp the top.

7.3.4. Spike a volume of stock standard, through the septum and below the surface of the methanol, to reach a concentration range of 75-150 μg/1.5 mL for each analyte in the QC spiking solution.

7.3.5. More than one stock standard may be spiked into the methanol to increase the number of analytes in the QC sample.

7.3.6. Replace the punctured septum and cap from the autosampler vial with a new septum and cap.

7.3.7. Label the vial with a QC sample number.

7.3.8. Include directions for the analyst to spike 10 μL of the QC spiking solution onto 5.0 g of preheated Ottawa sand just before analysis.

7.3.9. Return the stock standard to the freezer.

7.4. Refrigerate the QC spiking solution pending use. The maximum holding time for a volatile QC spiking solution for soil is 14 days.

7.5. The analyst will spike 10 μL of the QC spiking solution onto an accurately weighed 5.0 g aliquot of preheated Ottawa sand just before analysis.

7.6. Calculate the analyte concentrations in the QC sample in μg/kg based on a 10 μL aliquot of the QC spiking solution on 5.0000 g of preheated Ottawa sand.

7.7. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD databases on the VAX.

8. Calculations

8.1. Calculate the volume of neat liquid analyte needed to prepare a stock solution in the range of 8-12 mg/5 mL.

8.1.1. Read the density of the neat liquid analyte from Table I. The density of toluene is 0.867 mg/μL.

8.1.2. The desired weight spike range for 5 mL of stock solution is 8-12 mg of neat analyte.

8.1.3. Divide the weight spike range by the density of the neat analyte to determine the desired volume spike range of neat analyte to use in 5 mL of stock solution.

$$\frac{8\ \text{mg}}{0.867\ \text{mg}/\mu\text{L}} \text{ to } \frac{12\ \text{mg}}{0.867\ \text{mg}/\mu\text{L}} = 9.2 \text{ to } 13.8\ \mu\text{L}$$

For toluene, between 9.2 and 13.8 μL of the neat analyte should be diluted to 5 mL to reach the desired concentration range of 1.5 - 2.5 mg/mL in the stock solution.

8.2. Calculate the concentration of each analyte in the stock solution as in the following example.

8.2.1. If 12 μL of toluene, weighing 10.4 mg, is diluted in 5 mL of stock solution, the concentration of toluene in the stock solution is 2.08 mg/mL.

$$\frac{10.4 \text{ mg}}{5 \text{ mL}} = 2.08 \text{ mg/mL}$$

8.3. Calculate the concentration of each analyte in the QC spiking solution as in the following example.

8.3.1. The final volume of the QC spiking solution is the total of 1500 μL of methanol plus the volumes of all the QC spiking solutions added to the methanol.

8.3.2. If 60 μL of a stock solution containing 2.08 mg/mL of toluene and 50 μL of a second QC stock solution are both added to 1500 μL of methanol in an autosampler vial, the concentration of toluene in the QC spiking solution is 0.0775 μg/μL.

$$\frac{(2.08 \text{ mg/mL}) \left(\frac{1 \text{ mL}}{1000 \ \mu\text{L}}\right) (60 \ \mu\text{L}) \left(\frac{1000 \ \mu\text{g}}{1 \text{ mg}}\right)}{1500 \ \mu\text{L} + 60 \ \mu\text{L} + 50 \ \mu\text{L}} = 0.0775 \ \frac{\mu\text{g}}{\mu\text{L}}$$

8.4. Calculate the concentration of each analyte in the QC sample as in the following example.

8.4.1. When the analyst spikes 10 μL of the QC spiking solution containing 0.0775 μg/μL toluene onto 5.0000 g of preheated Ottawa sand, the concentration of toluene in the resulting QC sample will be 155 μg/kg. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(0.0775 \ \mu\text{g}/\mu\text{L}) \ (10 \ \mu\text{L})}{(5.0000 \text{ g}) \ (\text{kg}/1000 \text{ g})} = 155 \ \mu\text{g/kg}$$

8.5. Record the analyte concentrations in the QC sample in μg/kg based on the analyst spiking a 10-μL aliquot of the QC spiking solution on 5.0000 g of preheated Ottawa sand.

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Solid waste, such as paper wipes, glass or plastic pipettes, empty vials, and incorrectly spiked QC samples contaminated with organic compounds, is accumulated in a covered metal can lined with a plastic bag and is kept segregated from PCB-contaminated solid waste and solid waste that is not contaminated with organic compounds.

9.1.2. The can is labeled with a hazardous waste label and a label indicating its use for waste contaminated with organic compounds.

9.1.3. The can is opened only for the period of time necessary to add waste.

9.1.4. When the plastic bag is full, it is sealed with tape, removed from the can, identified as waste contaminated with organic compounds, and placed in a functioning chemical exhaust hood or in a closed secondary container pending pickup by the Waste Management Group.

9.1.5. A new plastic bag is put into the waste can for continued use.

9.2. Liquid waste.

9.2.1. Liquid waste containing organic compounds, such as rinses from syringes, excess QC samples, and incorrectly spiked QC samples, is accumulated in a covered metal can in a secondary containment tray.

9.2.1.1. Liquid waste containing organic compounds but no PCBs is kept segregated from PCB-contaminated liquid waste.

9.2.1.2. The can is labeled with a hazardous waste label and a label indicating its use for waste containing organic compounds.

9.2.1.3. When the can is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.1.4. A new can is labeled for future use.

9.2.1. Two-mL vials containing out-of-date spiking solutions of organic compounds are accumulated in a glass jar in a secondary containment tray.

9.2.2.1. Solutions that are not contaminated with PCBs are kept segregated from those that are PCB-contaminated.

9.2.2.2. The jar is labeled with a hazardous waste label and a label indicating its use for waste containing organic compounds.

9.2.2.3. The jar is opened only for the period of time necessary to add vials.

9.2.2.4 When the jar is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.2.5. A new jar is labeled for future use.

9.3. Waste pickup.

9.3.1. Pickup by the Waste Management Group of a full bag, can, or jar of waste contaminated with organic compounds is requested using the current Chemical Waste Disposal Request form. The current Waste Profile Request Form (WRPF) that describes the waste is referenced on the Chemical Waste Disposal Request.

9.3.2. The Waste Management Group picks up the waste for disposal according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.3. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Enviromental Chemistry: 1991," Los Alamos National Laboratory report LA-12436-MS (1992).

10.4. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

TABLE I. VOLATILE ORGANIC COMPOUNDS

Compound	CAS Number	Density of liquids (mg/μL)
acetone	67641	0.791
benzene	71432	0.874
bromobenzene	108861	1.491
bromochloromethane	74975	1.991
bromodichloromethane	75274	1.980
bromoform	75252	2.894
bromomethane	74839	0.838
2-butanone (MEK)	78933	0.805
n-butylbenzene	104518	0.860
sec-butylbenzene	135988	0.863
tert-butylbenzene	98066	0.867
carbon disulfide	75150	1.266
carbon tetrachloride	56235	1.594
chlorobenzene	108907	1.107
chlorodibromomethane	124481	2.451
chloroethane	75003	0.891
chloroform	67663	1.492
2-chlorotoluene	95498	1.083
4-chlorotoluene	106434	1.070
1,2-dibromo-3-chloropropane	96128	2.093
1,2-dibromoethane	106934	2.180
dibromomethane	74953	2.477
1,2-dichlorobenzene	95501	1.306
1,3-dichlorobenzene	541731	1.288
1,4-dichlorobenzene	106467	1.241
1,1-dichloroethane	75343	1.176
1,2-dichloroethane	107062	1.256
1,1-dichloroethene	75354	1.213
cis-1,2-dichloroethene	156592	1.284
trans-1,2-dichloroethene	156605	1.257
1,2-dichloropropane	78875	1.156
1,3-dichloropropane	142289	1.190
2,2-dichloropropane	594207	1.082
1,1-dichloropropene	563586	1.169
cis-1,3-dichloropropene	10061015	1.224
trans-1,3-dichloropropene	10061026	1.224
ethylbenzene	100414	0.867
2-hexanone	591786	0.812

TABLE I. VOLATILE ORGANIC COMPOUNDS (cont)

Compound	CAS Number	Density of liquids (mg/μL)
isopropylbenzene	98828	0.864
4-isopropyltoluene	99876	0.860
methylene chloride	75092	1.325
4-methyl-2-pentanone (MIK)	108101	0.800
n-propylbenzene	103651	0.862
styrene	100425	0.909
1,1,1,2-tetrachloroethane	630206	1.598
1,1,2,2,-tetrachloroethane	79345	1.586
tetrachloroethene	127184	1.623
toluene	108883	0.867
1,1,1-trichloroethane	71556	1.338
1,1,2-trichloroethane	79005	1.435
trichloroethene	79016	1.464
trichlorofluoromethane	75694	1.494
1,2,3-trichloropropane	96184	1.387
1,2,4-trimethylbenzene	95636	0.889
1,3,5-trimethylbenzene	108678	0.864
vinyl acetate	108054	0.934
mixed xylenes (total)	1330207	0.860

VOLATILE ORGANIC COMPOUNDS IN WATER—QC SAMPLE PREPARATION

Analyte: Volatile organic compounds	**Method No.:** QCO260
Matrix: Water	**Range:** 30-150 μg/L
Procedure: Analysis by purge-and-trap extraction, gas chromatography, and mass spectrometry (PTGC)	**Accuracy and Precision:** 100% ± 5.3% RSD
Effective Date: 04/25/90	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Material 10.4 for proper waste disposal practices.

1. Principle of Method

1.1. Prepare a stock solution in methanol of 3-6 volatile organic compounds from the list in Table I.

1.2. Prepare a 1:20 dilution in methanol of the stock solution.

1.3. Spike the diluted standard into distilled water.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the error of the standard and on the error associated with the use of the microsyringe, balance, and volumetric flask. A relative standard deviation of 5.3% has been calculated for standard preparation and for weighing, spiking, and dilution based on the following:

$$D = \frac{M1 \times M2 \times M4 \times M6}{M3 \times M5 \times M7} ,$$

where D = calculated result,
M1 = standard error,
M2 = weighing error,
M3 = first dilution error,
M4 = first pipette error,
M5 = second dilution error,

M6 = second pipette error, and
M7 = third dilution error.

2.1.1. Calculate relative variance for each variable using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3} + R^2_{M4} + R^2_{M5} + R^2_{M6} + R^2_{M7}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of balance,
R^2_{M3} = relative variance of first volumetric flask,
R^2_{M4} = relative variance of first microsyringe,
R^2_{M5} = relative variance of second volumetric flask,
R^2_{M6} = relative variance of second microsyringe,
R^2_{M7} = relative variance of third volumetric flask, and
100 = factor to convert to percentage.

2.2. Analytical results from QC samples prepared between 1990 and 1991 using this procedure and analyzed by purge-and-trap extraction, gas chromatography, and mass spectrometry are summarized and tabulated below. The data are published in Source Materials 10.1 to 10.3.

VOLATILE ORGANIC COMPOUNDS IN WATER SPIKE RECOVERY DATA

Compound	1990 Mean	± SD	(n)	1991 Mean	± SD	(n)
Acetone	—			95	± 8	(17)
Benzene	94	± 30	(13)	88		(1)
Bromobenzene	—			98	± 6	(9)
Bromochloromethane	110		(10)	—		
Bromodichloromethane	—			131		(2)
Bromoform	61		(1)	102	± 8	(8)
Bromomethane	—			—		
2-Butanone (MEK)	90	± 58	(19)	56	± 20	(3)
n-Butylbenzene	—			—		
sec-Butylbenzene	—			—		
tert-Butylbenzene	—			—		
Carbon disulfide	98	± 62	(14)	75	± 27	(4)
Carbon tetrachloride	76	± 12	(8)	89	± 22	(3)
Chlorobenzene	85	± 18	(20)	85	± 5	(10)
Chlorodibromomethane	82	± 22	(10)	95	± 15	(9)
Chloroethane	—			—		
Chloroform	—			100	± 13	(3)
2-Chlorotoluene	—			78	± 12	(4)
4-Chlorotoluene	—			—		
1,2-Dibromo-3-chloropropane	—			—		
1,2-Dibromoethane	108		(1)	—		
Dibromomethane	—			106	± 11	(3)
1,2-Dichlorobenzene	—			74		(2)
1,3-Dichlorobenzene	—			—		
1,4-Dichlorobenzene	64		(2)	—		
1,1-Dichloroethane	104	± 27	(22)	85	± 10	(4)
1,2-Dichloroethane	102	± 14	(16)	120	± 36	(9)
1,1-Dichloroethene	—			—		
cis-1,2-Dichloroethene	—			—		
trans-1,2-Dichloroethene	—			—		
1,2-Dichloropropane	96	± 20	(3)	86	± 13	(10)
1,3-Dichloropropane	—			—		
2,2-Dichloropropane	—			—		
1,1-Dichloropropene	—			—		
cis-1,3-Dichloropropene	52	± 10	(13)	—		
trans-1,3-Dichloropropene	169	± 35	(13)	—		
Ethylbenzene	78	± 18	(22)	75	± 10	(10)
2-Hexanone	94	± 43	(23)	72	± 32	(8)

VOLATILE ORGANIC COMPOUNDS IN WATER
SPIKE RECOVERY DATA (cont)

Compound	1990 Mean	± SD	(n)	1991 Mean	± SD	(n)
Isopropylbenzene	—			—		
p-Isopropyltoluene	—			—		
Methylene chloride	—			80	± 23	(14)
4-Methyl-2-pentanone (MIK)	—			84	± 12	(5)
n-Propylbenzene	—			53	± 16	(4)
Styrene	77		(2)	67	± 19	(5)
1,1,1,2-Tetrachloroethane	97		(2)	87	± 5	(4)
1,1,2,2-Tetrachloroethane	—			—		
Tetrachloroethene	77	± 19	(19)	74	± 7	(9)
Toluene	80	± 19	(20)	75	± 4	(9)
1,1,1-Trichloroethane	85	± 24	(11)	86	± 16	(6)
1,1,2-Trichloroethane	94	± 10	(19)	95	± 8	(17)
Trichloroethene	76	± 9	(7)	82	± 5	(4)
Trichlorofluoromethane	—			—		
1,2,3-Trichloropropane	—			106	± 5	(4)
1,2,4-Trimethylbenzene	—			48	± 27	(3)
1,3,5-Trimethylbenzene	—			—		
Vinyl acetate	56	± 40	(8)	—		
Mixed xylenes	326		(1)	63	± 16	(4)

3. Collection and Storage of Samples

3.1. Distilled water purged with nitrogen gas is used as the blank matrix for spiking. Use water that has not come into contact with plastic tubing or plastic holding vessels because phthalates may leach from plastics and contaminate the sample.

3.2. Spiked QC samples are refrigerated pending analysis. The maximum holding time for volatile organic compounds in water QC samples is seven days. Preservation with HCl increases the holding time to 14 days.

4. Apparatus

4.1. Volumetric flasks: 5- and 50-mL, class A, glass.

4.2. Autopipette: 1000-μL, Rainin, or equivalent.

4.3. Pipette tips: disposable.

4.4. Syringes: gas-tight, 10-, 25-, 50-, 100-, and 500-μL.

4.5. Microspatula.

4.6. Pipettes: Pasteur, disposable, glass.

4.7. Pipette bulb: rubber, to fit Pasteur pipette.

4.8. Vials: 1.5-mL, glass, amber.

4.9. Caps: open-top, to fit vials.

4.10. Septa: Teflon/silicone discs to fit open-top caps.

4.11. Teflon tape.

4.12. Vials: 40-mL, zero headspace, with open-top screw caps with Teflon/silicone septa, pre-cleaned.

4.13. Analytical balance: 100-g minimum capacity, 0.1-mg accuracy.

4.14. Laboratory oven: capable of sustaining 100-150°C.

4.15. Bottle: gas-washing, with fritted dispersion disc, 500-mL minimum capacity.

4.16. Tubing and adapters: Teflon, to connect gas-washing bottle to nitrogen gas cylinder.

4.17. Labels printed with QC sample numbers.

5. Reagents

5.1. Methanol (analytical-grade).

5.2. Hydrochloric acid (concentrated, reagent-grade).

5.3. Nitrogen gas (high-purity) in a compressed gas cylinder.

6. Calibration and Standards

6.1. Prepare a stock solution of neat, standard-grade volatile analytes chosen from Table I. Use Chem Service or equivalent chemicals (Chem Service, Inc., Box 3108, West Chester, PA 19381). Add solid analytes first, then liquid analytes after the addition of solvent.

6.1.1. Add 8-12 mg of a solid volatile analyte to a clean, tared 5.0-mL volumetric flask. Record the mass and tare the balance. Repeat for each analyte.

6.1.2. Add approximately 3 mL of methanol to the flask, stopper the flask, and swirl it to dissolve the analytes.

6.1.3. Place the stoppered flask on the analytical balance and tare the balance.

6.1.4. Calculate the volumes of liquid analytes needed to reach a weight spike range of 8-12 mg in 5 mL of stock solution. See Step 8.1.

6.1.5. Add a liquid analyte from a syringe just above the surface of the methanol. Stopper the flask and record the mass and volume of the analyte added. Tare the balance. Repeat for each liquid analyte added.

6.1.6. Dilute the solution to volume with methanol.

6.1.7. Mix by gently inverting three times.

NOTE: Vigorous shaking will cause the loss of volatile analytes.

6.1.8. Discard the solution in the neck of the flask to minimize concentration changes due to loss of volatile analytes from the surface of the solution.

6.1.9. The concentration of analytes in the stock solution will be 1.5 - 2.5 mg/mL.

6.1.10. Transfer the stock solution to 1.5-mL amber vials fitted with Teflon septa and caps. Wrap Teflon tape over and around the caps.

6.1.11. Label the vials with information identifying the standard and with the expiration date.

6.1.12. The stock solution is stable for approximately three months when stored at -20°C.

6.2. Prepare additional stock solutions using different analytes from Table I. The number of analytes in the QC samples may be increased by combining the stocks in a 1:20 dilution.

6.3. Prepare a 1:20 dilution of the stock solution.

6.3.1. Add approximately 4 mL of methanol to a clean 5-mL volumetric flask.

6.3.2. Add 250 μL of a stock solution from a syringe just above the surface of the methanol. Repeat for each stock solution to be combined in the 1:20 dilution.

6.3.3. Dilute the solution to volume with methanol.

6.3.4. Mix by gently inverting three times.

NOTE: Vigorous shaking will cause the loss of volatile analytes.

6.3.5. Discard the solution in the neck of the flask to minimize concentration changes due to the loss of volatile analytes from the surface of the solution.

6.3.6. The concentration of analytes in the dilution will be 0.075-0.125 mg/mL.

6.3.7. Transfer the dilution to 1.5-mL amber vials fitted with Teflon septa and caps. Wrap Teflon tape over and around the cap.

6.3.8. Label the vials with information identifying the standard and with the expiration date.

6.3.9. The standard dilution is stable for approximately three months when stored at -20°C.

7. Procedure

7.1. Cleaning glassware.

7.1.1. Wash volumetric flasks with detergent and rinse with hot tap water and then with methanol.

7.1.2. Store the glassware in a laboratory oven maintained at 100-150°C to minimize contamination.

7.2. Cleaning syringes.

7.2.1. Dedicate a group of syringes for use only with volatile analytes.

7.2.2. Fill the syringe through the needle with methanol. Discard the methanol into a waste container. Repeat for a total of 5-10 times.

7.2.3. Dry the syringe by pulling a vacuum on it for at least 10 min.

7.3. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed.

7.3.1. Remove a vial of a 1:20 dilution of volatile stock standards from the freezer and allow it to reach room temperature. Do not let the vial sit at room temperature for longer than 2 h before spiking the QC sample.

7.3.2. Put 100-200 mL of distilled water into a gas-washing bottle fitted with a fritted gas-dispersion disc and connected to a nitrogen purge-gas tank. Use only distilled water that has not been stored in plastic or run through plastic tubing. Purge the water with nitrogen gas for at least 15 min to remove any volatile compounds dissolved in the water.

7.3.3. Add approximately 45 mL of the purged water to the 50-mL volumetric flask. Add 0.5 mL of concentrated HCl to the water as a preservative for the QC sample.

7.3.4. Spike a volume of a 1:20 dilution of a standard below the surface of the water to reach the desired spike range. Spike near the bottom of the flask using a syringe.

7.3.5 Dilute the solution to volume with purged water.

7.3.6. Mix by gently inverting three times.

NOTE: Vigorous shaking will cause the loss of volatile analytes.

7.3.7. Discard the solution in the neck of the flask to minimize concentration changes due to the loss of volatile analytes from the surface of the solution.

7.3.8. Pour the sample into a 40-mL glass, zero-headspace vial leaving zero headspace. Discard the excess sample.

7.3.9. Label the vial with a QC sample number.

7.3.10. Return the 1:20 dilution of the standard to the freezer.

7.3.11. Refrigerate the QC sample pending analysis. The maximum holding time for volatile organic compounds in water QC samples preserved with HCl is 14 days.

7.4. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and enter them into the CVS and CVD databases on the VAX.

8. Calculations

8.1. Calculate the volume of neat liquid analyte needed to prepare a stock solution in the range of 8-12 mg/5 mL.

8.1.1. Read the density of the neat liquid analyte from Table I. The density of toluene is 0.867 mg/μL.

8.1.2. The desired weight spike range for 5 mL of stock solution is 8-12 mg of neat analyte.

8.1.3. Divide the weight spike range by the density of the neat analyte to determine the desired volume spike range of neat analyte to use in 5 mL of stock solution.

$$\frac{8\ \text{mg}}{0.867\ \text{mg}/\mu\text{L}}\ \text{to}\ \frac{12\ \text{mg}}{0.867\ \text{mg}/\mu\text{L}} = 9.2\ \text{to}\ 13.8\ \mu\text{L}$$

For toluene, between 9.2 and 13.8 μL of the neat analyte should be diluted to 5 mL to reach the desired concentration range of 1.5 - 2.5 mg/mL in the stock solution.

8.2. Calculate the concentration of each analyte in the stock solution as in the following example.

8.2.1. If 12 μL of toluene, weighing 10.4 mg, is diluted to 5 mL of stock solution, the concentration of toluene in the stock solution is 2.08 mg/mL.

$$\frac{10.4\ \text{mg}}{5\ \text{mL}} = 2.08\ \text{mg/mL}$$

8.3. Calculate the concentration of each analyte in the 1:20 dilution of the stock solution as in the following example.

8.3.1. If 250 μL of a stock solution containing 2.08 mg/mL of toluene is diluted to 5 mL with methanol, the concentration of toluene in the 1:20 stock dilution is 0.104 μg/μL.

$$\frac{(2.08\ \text{mg/mL})\ (\frac{1\ \text{mL}}{1000\ \mu\text{L}})\ (\frac{1000\ \mu\text{g}}{1\ \text{mg}})\ (250\ \mu\text{L})}{(5\ \text{mL})\ (\frac{1000\ \mu\text{L}}{1\ \text{mL}})} = 0.104\ \mu\text{g}/\mu\text{L}$$

8.4. Calculate the concentration of each analyte in the QC water sample as in the following example.

8.4.1. If 30 μL of a 1:20 stock dilution containing 0.104 μg/μL of toluene is spiked into 50 mL of distilled water, the concentration of toluene in the sample is 62 μg/L. Multiply the result by the RSD calculated in Step 2.1. to obtain the uncertainty.

$$\frac{(0.104\ \mu\text{g}/\mu\text{L})\ (30\ \mu\text{L})}{(50\ \text{mL})\left(\frac{1\ \text{L}}{1000\ \text{mL}}\right)} = 62\ \mu\text{g/L}$$

8.5. Record the analyte concentration in the QC sample in μg/L.

9. Proper Waste Disposal Practices

9.1. Solid waste

9.1.1. Solid waste, such as paper wipes, glass or plastic pipettes, empty vials, and incorrectly spiked QC samples contaminated with organic compounds, is accumulated in a covered metal can lined with a plastic bag and is kept segregated from PCB-contaminated solid waste and solid waste that is not contaminated with organic compounds.

9.1.2. The can is labeled with a hazardous waste label and a label indicating its use for waste contaminated with organic compounds.

9.1.3. The can is opened only for the period of time necessary to add waste.

9.1.4. When the plastic bag is full, it is sealed with tape, removed from the can, identified as waste contaminated with organic compounds, and placed in a functioning chemical exhaust hood or in a closed secondary container pending pickup by the Waste Management Group.

9.1.5. A new plastic bag is put into the waste can for continued use.

9.2. Liquid waste

9.2.1. Liquid waste containing organic compounds, such as rinses from syringes, excess QC samples, and incorrectly spiked QC samples, is accumulated in a covered metal can in a secondary containment tray.

9.2.1.1. Liquid waste containing organic compounds but no PCBs is kept segregated from PCB-contaminated liquid waste.

9.2.1.2. The can is labeled with a hazardous waste label and a label indicating its use for waste containing organic compounds.

9.2.1.3. When the can is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.1.4. A new can is labeled for future use.

9.2.1. Two-mL vials containing out-of-date spiking solutions of organic compounds are accumulated in a glass jar in a secondary containment tray.

9.2.2.1. Solutions that are not contaminated with PCBs are kept segregated from those that are PCB-contaminated.

9.2.2.2. The jar is labeled with a hazardous waste label and a label indicating its use for waste containing organic compounds.

9.2.2.3. The jar is opened only for the period of time necessary to add vials.

9.2.2.4 When the jar is full, it is capped and kept in the secondary containment tray pending pickup by the Waste Management Group.

9.2.2.5. A new jar is labeled for future use.

9.3. Waste pickup

9.3.1. Pickup by the Waste Management Group of a full bag, can, or jar of waste contaminated with organic compounds is requested using the current Chemical Waste Disposal Request form. The current Waste Profile Request Form (WRPF) that describes the waste is referenced on the Chemical Waste Disposal Request.

9.3.2. The Waste Management Group picks up the waste for disposal according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.3. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Environmental Chemistry: 1991," Los Alamos National Laboratory report LA-12436-MS (1992).

10.4. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

TABLE I. VOLATILE ORGANIC COMPOUNDS

Compound	CAS Number	Density of liquids (mg/μL)
acetone	67641	0.791
benzene	71432	0.874
bromobenzene	108861	1.491
bromochloromethane	74975	1.991
bromodichloromethane	75274	1.980
bromoform	75252	2.894
bromomethane	74839	0.838
2-butanone (MEK)	78933	0.805
n-butylbenzene	104518	0.860
sec-butylbenzene	135988	0.863
tert-butylbenzene	98066	0.867
carbon disulfide	75150	1.266
carbon tetrachloride	56235	1.594
chlorobenzene	108907	1.107
chlorodibromomethane	124481	2.451
chloroethane	75003	0.891
chloroform	67663	1.492
2-chlorotoluene	95498	1.083
4-chlorotoluene	106434	1.070
1,2-dibromo-3-chloropropane	96128	2.093
1,2-dibromoethane	106934	2.180
dibromomethane	74953	2.477
1,2-dichlorobenzene	95501	1.306
1,3-dichlorobenzene	541731	1.288
1,4-dichlorobenzene	106467	1.241
1,1-dichloroethane	75343	1.176
1,2-dichloroethane	107062	1.256
1,1-dichloroethene	75354	1.213
cis-1,2-dichloroethene	156592	1.284
trans-1,2-dichloroethene	156605	1.257
1,2-dichloropropane	78875	1.156
1,3-dichloropropane	142289	1.190
2,2-dichloropropane	594207	1.082
1,1-dichloropropene	563586	1.169
cis-1,3-dichloropropene	10061015	1.224
trans-1,3-dichloropropene	10061026	1.224
ethylbenzene	100414	0.867
2-hexanone	591786	0.812

TABLE I. VOLATILE ORGANIC COMPOUNDS (cont)

Compound	CAS Number	Density of liquids (mg/μL)
isopropylbenzene	98828	0.864
4-isopropyltoluene	99876	0.860
methylene chloride	75092	1.325
4-methyl-2-pentanone (MIK)	108101	0.800
n-propylbenzene	103651	0.862
styrene	100425	0.909
1,1,1,2-tetrachloroethane	630206	1.598
1,1,2,2,-tetrachloroethane	79345	1.586
tetrachloroethene	127184	1.623
toluene	108883	0.867
1,1,1-trichloroethane	71556	1.338
1,1,2-trichloroethane	79005	1.435
trichloroethene	79016	1.464
trichlorofluoromethane	75694	1.494
1,2,3-trichloropropane	96184	1.387
1,2,4-trimethylbenzene	95636	0.889
1,3,5-trimethylbenzene	108678	0.864
vinyl acetate	108054	0.934
mixed xylenes (total)	1330207	0.860

AMERICIUM-241 IN URINE – QC SAMPLE PREPARATION

Analyte: Americium-241	**Method No.:** QCR100
Matrix: Urine	**Spike Range:** 0.0 to 1.0 pCi/sample
Procedure: Radiochemistry and alpha spectrometry	**Accuracy and Precision:** 100% ± 4% RSD
Effective Date: 01/01/75	**Authors:** Margaret A. Gautier William D. Moss Nancy L. Koski

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.11 to 10.13 for proper waste disposal practices.

1. Principle of Method

1.1. A pool of blank urine is prepared.

1.2. The blank urine is aliquoted and spiked with a known standard.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error from six sample plate preparations of the standard and on the error associated with use of the graduated cylinder and the pipette. A relative standard deviation (RSD) of 4.0% at the 1σ level has been calculated for the calibration, spiking, and aliquoting process based on the following:

$$D = \frac{M1 \times M2}{M3 + M2} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = aliquoting error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{\frac{\left[R^2_{M1} (M_3 + M_2)^2 + M_3^2 (R^2_{M2} + R^2_{M3})\right]}{(M_3 + M_2)^2}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
M_3 = volume of cylinder,
M_2 = volume of pipette,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of cylinder, and
100 = factor to convert to percentage.

2.2. Analysis by radiochemistry and alpha spectrometry of QC samples prepared between 1986 and 1989 using this procedure gave a mean recovery of 102% ± 3% RSD. The data are published in Source Materials 10.1 through 10.6 and are summarized below.

AMERICIUM-241 SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION AFTER CORRECTION FOR AMERICIUM-243 RECOVERY

Year	Spike level pCi/sample	Mean ± RSD (%)	Number of samples
1986	0.23	114 ± 3	3
	0.46	100 -	1
	0.55	100 -	1

AMERICIUM-241 SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION AFTER CORRECTION FOR AMERICIUM-243 RECOVERY (cont)

Year	Spike level pCi/sample	Mean ± RSD (%)	Number of samples
1987	0.23	104 - 1	
1987	0.54	112 ± 3	2
	0.90	109 ± 3	2
1988	0.24	104 -	1
	0.48	88 -	1
	0.58	97 -	1
	0.96	104 -	1
1989	0.24	92 ± -	1
	0.48	96 ± -	1
	0.96	102 ± -	2

3. Collection and Storage of Samples

3.1. Urine is collected as needed from group personnel who have no known contact with radiation work and is used to form a pool of blank urine.

3.2. Spiked QC samples are refrigerated at a nominal temperature of 4°C pending analysis.

4. Apparatus

4.1. Beakers: 4- or 5-L, polyethylene.

4.2. Graduated cylinders: 500-mL.

4.3. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.4. Pipette tips: disposable.

4.5. Specimen containers: 16-oz jars with screw caps. VWR Scientific Catalog No. 16193-810 or equivalent.

4.6. Labels printed with QC sample numbers.

4.7. Heat lamp.

4.8. Filter paper: 2.9-cm, Whatman No. 41 or equivalent.

4.9. Discs: stainless steel, 2.22-cm-diam.

4.10. Alpha phosphor on Mylar: The phosphor is the standard silver-activated zinc sulfide. William B. Johnson & Associates, Inc., Research Park, Montville, NJ 07045.

4.11. Rings and discs: plastic rings and discs, nominal 1-in.-diam, molded of nylon. Control Molding Corp., 84 Granite Ave., Staten Island, NY 10303.

4.12. Mylar film: 1.5-in.-wide rolls, 0.0005 in. thick. Manufactured by E. I. duPont de Nemours, retailed by Brownell, Inc., 85 Tenth Ave., New York, NY 10001.

5. Reagents

5.1. None required.

6. Calibration and Standards

6.1. QC samples are spiked with an ^{241}Am standard prepared in 4 N HNO_3. The solution is counted on a zinc sulfide scintillation system calibrated with National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 4904L-F, an ^{241}Am alpha-particle standard, using count room procedure CR340.

6.2. The ^{241}Am standard should be at a concentration of approximately 1.0 pCi/mL.

6.3. The ^{241}Am standard was calibrated against a NIST-certified ^{241}Am alpha-particle solution standard, such as NIST SRM 4322. Recalibrate the standard solution yearly by direct plating and alpha scintillation counting. Record all data in the QC laboratory notebook.

6.4. Direct plating procedure.

6.4.1. Pipette a 200- to 300-μL aliquot of the standard to be calibrated on each of six 2.22-cm stainless-steel discs and evaporate to dryness under a heat lamp.

6.4.2. Place a 2.9-cm filter paper on a molded nylon disc. The filter paper assures a tightly sealed package when assembled.

6.4.3. Place the stainless-steel disc containing the sample on the filter paper. Be sure they are centered.

6.4.4. Place a 0.875-in. phosphor paper over the stainless-steel disc (dull side against the disc).

6.4.5. Place a piece of Mylar film over all the layers.

6.4.6. Press a nylon ring down over the layers and around the nylon disc to form a tightly sealed package. Cut off the excess Mylar with scissors. Label the back of the assembly with an identification number.

6.4.7. Submit the samples to the count room for alpha scintillation counting and request a 60,000-s (overnight) per sample count.

6.4.8. The count room data are corrected for background and reported in disintegrations per minute (dis/min). Divide the dis/min results by 2.22 to convert to pCi. Calculate the mean and standard deviation of the six plates, and assign the value to the standard.

7. Procedure

7.1. Working with bodily fluids is discussed in detail in SOP-4, "Handling Human Biological Fluids," found in Appendix III of the EM-9 Safety Manual.

NOTE: Wear safety glasses, gloves, and a lab coat. Each day prepare a 1:100 dilution of household bleach in water in a spray bottle. Use this solution to thoroughly clean the work area after sample preparation is completed. Remove gloves, discard in the biohazard waste container, and wash hands with soap and water.

7.2. Prepare a pool from blank urine sufficient in volume to allow 400 mL for each QC sample to be prepared.

7.3. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Include blank QCs prepared from the same urine pool as the spiked samples.

7.4. Record QC standard information, sample spike amount, concentration, and uncertainty in the QC Laboratory notebook.

7.5. Label bottles with QC sample numbers.

7.6. Aliquot 400 mL of blank urine into each sample bottle.

7.7. Spike the blank urine with aliquots of standard as determined in Step 7.3.

7.8. Screw caps on bottles and refrigerate at a nominal temperature of 4°C pending analysis.

7.9. Enter the spike concentrations of each QC into the CVS and CVD data bases on the VAX. This procedure is done annually because the same concentration levels are used throughout the year.

8. Calculations

8.1. Record spike concentrations as pCi/sample of urine.

8.2. Calculate the spike concentration for a 500-μL aliquot of ^{241}Am standard using the following equation:

$$\text{pCi/sample per spike amount} = (1.0\ \text{pCi/mL}) \times 0.500\ \text{mL} = 0.50\ \text{pCi/sample}$$

8.3. Spike concentrations calculated in Step 8.2 are tabulated below.

Spike amount (μL)	Concentration (pCi/sample)	Uncertainty (±4.0%)
250	0.25	0.01
500	0.50	0.02
600	0.60	0.02
900	0.90	0.04

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Radiochemically contaminated waste.

9.1.1.1. Radiochemically contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, is accumulated in a covered metal can lined with a plastic bag and is kept segregated from nonradioactive solid waste.

9.1.1.2. The can is labeled with a hazardous waste label and a label indicating its use for radiochemical-contaminated waste.

9.1.1.3. The can is opened only for the time necessary to add waste.

9.1.1.4. When the plastic bag is full, it is sealed with tape, removed from the can, identified as radiochemically contaminated waste, and placed in a functioning chemical exhaust hood pending pickup by the Waste Management group.

9.1.1.5. A new plastic bag is put into the waste can for continued use.

9.1.2. Biohazardous waste.

9.1.2.1. Bottles used to collect blank urine for a quality control sample matrix are disposed of as uncontaminated laboratory trash after they have been rinsed with water in a laboratory sink.

9.1.2.2. Solid waste, such as paper wipes, plastic pipettes, and rubber gloves, contaminated with biohazardous material (primarily urine) is accumulated in a plastic bag labeled for the collection of biohazardous waste and is kept segregated from waste with no biohazard contamination.

9.1.2.3. Full bags of biohazardous waste are transferred to a cardboard box labeled with a biohazard label. The cardboard box is lined with a plastic bag bearing a biohazard label.

9.1.2.4. When the cardboard box if full, the liner bag and the box are sealed.

9.1.2.5. A new biohazard waste box lined with a plastic bag is prepared for further use.

9.2. Liquid waste.

9.2.1. Liquid waste contaminated with radionuclides is disposed of in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. Radioactively contaminated waste.

9.3.1.1. The surface exposure rate of the sealed bag of radiochemically contaminated solid waste is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) record form.

9.3.1.2. Pickup by the Waste Management group of a full bag of radiochemically contaminated solid waste is requested using the current Chemical Waste Disposal Request (CWDR) form. The

current Waste Profile Request form (WPRF) that describes the waste is referenced on both disposal request forms.

9.3.1.3. The Waste Management group picks up the waste for disposal according to Laboratory policy.

9.3.2. Biohazardous waste.

9.3.2.1. Pickup of a full box of biohazardous waste is requested through the Waste Management group using the current Chemical Waste Disposal Request (CWDR) form. The current Waste Profile Request form (WPRF) that describes the waste is referenced on the CWDR.

9.3.2.2. The Waste Management group arranges for pickup and disposal of the waste by the company currently under contract with the Laboratory for that purpose.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, and D. R. Perrin, "Quality Assurance for Health and Environmental Chemistry: 1984," Los Alamos National Laboratory report LA-10508-MS (1985).

10.2. M. A. Gautier, E. S. Gladney, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1985," Los Alamos National Laboratory report LA-10813-MS (1986).

10.3. M. A. Gautier, E. S. Gladney, W. D. Moss, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1986," Los Alamos National Laboratory report LA-11114-MS (1987).

10.4. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1987," Los Alamos National Laboratory report LA-11454-MS (1988).

10.5. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1988," Los Alamos National Laboratory report LA-11637-MS (1989).

10.6. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.7. *EML Procedures Manual*, 25th ed., H. L. Volchok and G. de Planque, Eds. (Environmental Measurements Laboratory, New York, August 1982).

10.8. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4322, Americium-241 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, November 1986).

10.9. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4904L-F, Alpha-Particle Standard," (National Institute of Standards and Technology, Washington, DC, April 1982).

10.10. M. Phillips, "Handling Human Biological Fluids," Health and Environmental Chemistry SOP-4, September 1988, Los Alamos National Laboratory document.

10.11. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.12. "Hazardous and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.13. "Managing Infectious Waste," Administrative Requirement 10-7, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒▒). Where a section heading is marked, the entire section has been revised.

CESIUM-137 ON SOIL — QC SAMPLE PREPARATION

Analyte: Cesium-137	**Method No.:** QCR110
Matrix: Sea sand	**Spike Range:** 5 to 60 pCi/g
Procedure: Gamma-ray spectrometry	**Accuracy and Precision:** 100% ± 5.4% RSD
Effective Date: 04/12/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.4 and 10.5 for proper waste disposal practices.

1. Principle of Method

1.1. Sea sand is spiked with a ^{137}Cs standard. The sand is used in place of soil as the QC matrix for ^{137}Cs analysis by gamma-ray spectrometry.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

1.3. Because the method of analysis is nondestructive, the QC samples can be reused provided the ^{137}Cs concentration is decayed periodically.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the pipette and of the balance. A relative standard deviation (RSD) of 5.4% at the 1σ level has been calculated for the calibration, spiking, and weighing process based on the following function:

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = weighing error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of balance, and
100 = factor to convert to percentage.

2.2. Analysis by gamma-ray spectrometry of QC samples prepared during 1989 and 1990 using this procedure gave a mean recovery of 99% ± 26% at the 1σ level. The data are published in Source Material 9.1 and are summarized below.

CESIUM-137 SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION

	Spike range pCi/g	Mean ± RSD (%)	Number of samples
1989	0 to 15	83 ± 42	5
	15 to 30	106 ± 21	7
	30 to 45	87 ± 8	3
	45 to 70	114 ± 5	5
1990	0 to 15	91 ± 14	3
	15 to 30	114 ± 48	2

3. Collection and Storage of Samples

3.1. Spiked QC samples are stored at room temperature pending analysis.

4. Apparatus

4.1. Bottles: 500-mL, high-density polyethylene.

4.2. Balance: 200-g minimum capacity, 0.1-g accuracy.

4.3. Vortex mixer.

4.4. Auto-pipette: 1000-μL, Rainin or equivalent.

4.5. Pipette tips: disposable.

4.6. Pipettes: 2- to 15-mL, class A volumetric.

4.7. Heat lamp.

4.8. Labels printed with QC sample numbers.

5. Reagents

5.1. Sand. A clean and dry 100-g aliquot is required for each sample.

5.2. A suitable sand does not contain the analyte of interest and presents few interferences to the analytical method.

NOTE: Surface soils have been contaminated with radionuclides as a result of the use and testing of nuclear weapons. A suitable blank soil for this procedure would have been collected before World War II or would be collected from a deep well or other excavation where radioactive contamination is not likely to have occurred. Before use, a representative aliquot of soil is analyzed to determine if it will provide a suitably blank matrix.

6. Calibration and Standards

6.1. QC samples are spiked with a ^{137}Cs standard prepared in 1 M HCl at a concentration of approximately 350 pCi/mL.

6.2. The ^{137}Cs standard was obtained from Amersham International, White Lion Road, Amersham, Buckinghamshire, England HP7 9LL.

6.3. The ^{137}Cs standard was calibrated against a National Institute Standards and Technology (NIST)-certified ^{137}Cs Standard Reference Material (SRM) solution, such as NIST SRM 4233-B, containing 19 μg of high-purity cesium chloride per

gram in 1 M HCl. Recalibrate the standards yearly using gamma spectrometry. Record all data in the QC Laboratory notebook.

6.4. Decay the ^{137}Cs standards each year.

6.4.1. The half-life of ^{137}Cs is 30 years.

6.4.2. The following equation describes the rate of physical decay of radionuclides:

$$A_t = A_o e^{-\lambda t} ,$$

where A_t = the activity at desired time t,
A_o = the activity at t = 0,
e = the base of natural logarithms, and
λ = (ln 2)/$T_{1/2}$ or the decay constant, where $T_{1/2}$ is the half-life.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically include a 100-g aliquot of the unspiked sand as a blank.

7.2. Accurately weigh 100 g of sand into a 500-mL high-density polyethylene bottle. Record the weight of the sand on the bottle for reference by the analyst.

7.3. Spike an aliquot of ^{137}Cs standard onto the sand to reach a ^{137}Cs concentration in the desired spike range. Be careful to wet only the sand and not the container walls.

7.4. Dry the spiked sand under a heat lamp. Periodically mix the sand with a vortex mixer until all large clumps are broken up and the sand is dry.

7.5. Cap the bottle and label it with a QC sample number.

7.6. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the concentration of ^{137}Cs on the sand as pCi/g.

8.2. Calculate spike concentrations as in the following example.

8.2.1. If 12 mL of a 350-pCi/mL ^{137}Cs standard is used to spike 100.0 g of sand, the ^{137}Cs concentration in the sand will be 42 pCi/g. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(350\ \text{pCi/mL})\ (12\ \text{mL})}{100.0\ \text{g}} = 42\ \text{pCi/g}$$

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.1.2. Label the can to indicate its use for radioactively contaminated waste.

9.1.3. Open the can only for the period of time necessary to add waste.

9.1.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.1.5. Put a new plastic bag into the waste can for continued use.

9.1.6. Place the sealed bag into a cardboard box lined with a plastic bag and keep the box inside a metal compactable waste container. Reference to the current Waste Profile Request form (WPRF) describing the waste accompanies the sealed bag.

9.1.7. Label the metal container to indicate its use for radioactively contaminated waste.

9.1.8. When the cardboard box is full, seal the plastic bag liner and the box. Check the outside of the box for radioactive contamination and record the information in the Radioactive Waste Disposal logbook.

9.1.9. The radioactive solid waste coordinator marks the cardboard box as radioactively contaminated compactable waste and references the current WPRF numbers describing the waste in the box on the outside of the box and in the Radioactive Waste Disposal logbook.

9.1.10. Place a new lined cardboard box into the metal container for continued use.

9.1.11. Place the sealed box in a dumpster for compactable mixed waste.

9.2. Liquid waste.

9.2.1. Dispose of radioactively contaminated liquid waste in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. When the dumpster containing solid waste is full, the surface exposure rate of the dumpster is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.2. Reference all the WPRF numbers associated with the waste in the dumpster on the RSWD.

9.3.3. Request pickup by the Waste Management group of a full dumpster. The RSWD and all the associated WPRFs accompany the dumpster.

9.3.4. The Waste Management group disposes of the waste according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. Amersham Corporation, "Certificate of Measurement of Caesium-137 Radioactive Reference Solution" (Amersham Corporation, Arlington Heights, Illinois, October 1973).

10.3. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4233-B, Cesium-137 Burn-Up Standard" (National Institute of Standards and Technology, Washington, DC, November 1979).

10.4. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.5 "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒). Where a section heading is marked, the entire section has been revised.

CESIUM-137 ON VEGETATION ASH – QC SAMPLE PREPARATION

Analyte: Cesium-137	**Method No.:** QCR120
Matrix: Vegetation ash	**Spike Range:** 5 to 60 pCi/g
Procedure: Gamma-ray spectrometry	**Accuracy and Precision:** 100% ± 5.4% RSD
Effective Date: 04/19/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.4 and 10.5 for proper waste disposal practices.

1. Principle of Method

1.1. Blank vegetation ash is spiked with a ^{137}Cs standard.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

1.3. Because the method of analysis is nondestructive, the QC sample can be reused provided the ^{137}Cs concentration is decayed periodically.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the pipette and of the balance. A relative standard deviation (RSD) of 5.4% at the 1σ level has been calculated for the calibration, spiking, and weighing process based on the following function:

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = weighing error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of balance, and
100 = factor to convert to percentage.

2.2. Analysis by gamma-ray spectrometry of QC samples prepared during 1989 and 1990 using this procedure gave a mean recovery of 102% ± 24% at the 1σ level (N = 7). The data are published in Source Material 9.1.

3. Collection and Storage of Samples

3.1. Spiked QC samples are stored at room temperature pending analysis.

4. Apparatus

4.1. Bottles: 500-mL, high-density polyethylene.

4.2. Balance: 200-g minimum capacity, 0.1-g accuracy.

4.3. Spatula.

4.4. Auto-pipette: 1000-μL, Rainin or equivalent.

4.5. Pipette tips: disposable.

4.6. Pipettes: 2- to 15-mL, class A volumetric.

4.7. Heat lamps.

4.8. Vortex mixer.

4.9. Labels printed with QC sample numbers.

5. Reagents

5.1. Blank vegetation ash is used as the sample matrix. A 100-g aliquot is required for each sample.

5.2. A suitable vegetation ash does not contain the analyte of interest and presents few interferences to the analytical method. Commercially available dry pet or farm stock foods may provide a suitable matrix.

6. Calibration and Standards

6.1. QC samples are spiked with a ^{137}Cs standard prepared in 1 M HCl at a concentration of approximately 350 pCi/mL.

6.2. The ^{137}Cs standard may be obtained from Amersham International, White Lion Road, Amersham, Buckinghamshire, England HP7 9LL.

6.3. The ^{137}Cs standard was calibrated against a National Institute Standards and Technology (NIST)-certified ^{137}Cs Standard Reference Material (SRM) solution, such as NIST SRM 4233-B, containing 19 μg of high-purity cesium chloride per gram in 1 M HCl. Recalibrate the standards yearly using gamma spectrometry. Record all data in the QC Laboratory notebook.

6.3.1. The half-life of ^{137}Cs is 30 years.

6.3.2. The following equation describes the rate of physical decay of radionuclides:

$$A_t = A_o e^{-\lambda t} \quad ,$$

where A_t = the activity at desired time t,
A_o = the activity at t = 0,
e = the base of natural logarithms, and
λ = (ln 2)/$T_{1/2}$ or the decay constant, where $T_{1/2}$ is the half-life.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically include a 100-g aliquot of the unspiked vegetation ash as a blank.

7.2. Accurately weigh 100 g of blank vegetation ash into a 500-mL high-density polyethylene bottle. Record the weight of ash on the bottle for reference by the analyst.

7.3. Pipette an aliquot of ^{137}Cs standard onto the ash to reach a ^{137}Cs concentration in the desired spike range. Be careful to wet only the ash and not the container walls.

7.4. Dry the spiked sample under a heat lamp, periodically mixing it with a vortex mixer, until completely dry and homogeneous.

7.5. Cap the bottle and label it with a QC sample number.

7.6. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the concentration of ^{137}Cs in the vegetation ash sample as pCi/g.

8.2. Calculate spike concentrations as in the following example.

8.2.1. If 2.0 mL of a 350-pCi/mL ^{137}Cs standard is spiked on 100.0 g of blank vegetation ash, the concentration of ^{137}Cs in the ash will be 7 pCi/g. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(350\ \text{pCi/mL})\ (2.0\ \text{mL})}{100.0\ \text{g}} = 7\ \text{pCi/g}$$

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.1.2. Label the can to indicate its use for radioactively contaminated waste.

9.1.3. Open the can only for the period of time necessary to add waste.

9.1.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.1.5. Put a new plastic bag into the waste can for continued use.

9.1.6. Place the sealed bag into a cardboard box lined with a plastic bag and keep the box inside a metal compactable waste container. Reference to the current Waste Profile Request form (WPRF) describing the waste accompanies the sealed bag.

9.1.7. Label the metal container to indicate its use for radioactively contaminated waste.

9.1.8. When the cardboard box is full, seal the plastic bag liner and the box. Check the outside of the box for radioactive contamination and record the information in the Radioactive Waste Disposal logbook.

9.1.9. The radioactive solid waste coordinator marks the cardboard box as radioactively contaminated compactable waste and references the current WPRF numbers describing the waste in the box on the outside of the box and in the Radioactive Waste Disposal logbook.

9.1.10. Place a new lined cardboard box into the metal container for continued use.

9.1.11. Place the sealed box in a dumpster for compactable mixed waste.

9.2. Liquid waste.

9.2.1. Dispose of radioactively contaminated liquid waste in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. When the dumpster containing solid waste is full, the surface exposure rate of the dumpster is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.2. Reference all the WPRF numbers associated with the waste in the dumpster on the RSWD.

9.3.3. Request pickup by the Waste Management group of a full dumpster. The RSWD and all the associated WPRFs accompany the dumpster.

9.3.4. The Waste Management group disposes of the waste according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. Amersham Corporation, "Certificate of Measurement of Caesium-137 Radioactive Reference Solution" (Amersham Corporation, Arlington Heights, Illinois, October 1973).

10.3. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4233-B, Cesium-137 Burn-Up Standard" (National Institute of Standards and Technology, Washington, DC, November 1979).

10.4. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.5. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒). Where a section heading is marked, the entire section has been revised.

CESIUM-137 IN WATER – QC SAMPLE PREPARATION

Analyte: Cesium-137	**Method No.:** QCR130
Matrix: Water	**Spike Range:** 500 to 2000 pCi/L
Procedure: Gamma-ray spectrometry	**Accuracy and Precision:** 100% ± 5.4% RSD
Effective Date: 04/17/90	**Author:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.4 and 10.5 for proper waste disposal practices.

1. Principle of Method

1.1. Water is spiked with a ^{137}Cs standard.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

1.3. Because the method of analysis is nondestructive, the QC sample can be reused provided the ^{137}Cs concentration is decayed periodically.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the pipette and of the volumetric flask. A relative standard deviation (RSD) of 5.4% at the 1σ level has been calculated for the calibration, spiking, and dilution process based on the following function:

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2} \times 100 ,$$

where S_{D_T} = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of volumetric flask, and
100 = factor to convert to percentage.

2.2. Analysis by gamma-ray spectrometry of QC samples prepared during 1989 and 1990 using this procedure gave a mean recovery of 91% ± 14% at the 1σ level. The data are published in Source Material 10.1 and are summarized below.

CESIUM-137 SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION

	Spike range pCi/L	Mean ± SD (%)	Number of samples
1989	100 to 400	91 ± 9	3
	500 to 900	75 ± 7	3
1990	100 to 500	88 ± 10	6
	500 to 1200	99 ± 16	8

3. Collection and Storage of Samples

3.1. Spiked QC samples are stored at room temperature pending analysis.

4. Apparatus

4.1. Volumetric flask: 500-mL, glass, class A.

4.2. Bottles: 500-mL, high-density polyethylene.

4.3. Auto-pipette: 1000-μL, Rainin or equivalent.

4.4. Pipette tips: disposable.

4.5. Pipettes: 1-, 2-, and 3-mL, class A volumetric.

4.6. Labels printed with QC sample numbers.

5. Reagents

5.1. Nitric acid (concentrated, reagent-grade).

6. Calibration and Standards

6.1. QC samples are spiked with a ^{137}Cs standard prepared in 1 M HCl at a concentration of approximately 350 pCi/mL.

6.2. The ^{137}Cs standard may be obtained from Amersham International, White Lion Road, Amersham, Buckinghamshire, England HP7 9LL.

6.3. The ^{137}Cs standard was calibrated against a National Institute of Standards and Technology (NIST)-certified ^{137}Cs Standard Reference Material (SRM) solution, such as NIST SRM 4233-B, containing 19 μg of high-purity cesium chloride per gram in 1 M HCl. Recalibrate the standards yearly using gamma spectrometry. Record all data in the QC Laboratory notebook.

6.4. Decay the ^{137}Cs standards each year.

6.4.1. The half-life of ^{137}Cs is 30 years.

6.4.2. The following equation describes the rate of physical decay of radionuclides:

$$A_t = A_0 e^{-\lambda t} \quad ,$$

where A_t = the activity at desired time t,
A_o = the activity at t = 0,
e = the base of natural logarithms, and
λ = (ln 2)/$T_{1/2}$ or the decay constant where $T_{1/2}$ is the half-life.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically include a 500-mL aliquot of the unspiked acidified water as a blank.

7.2. Measure approximately 400 mL of distilled water into a 500-mL volumetric flask.

7.3. Add 1.25 mL of concentrated HNO_3 to the water in the flask to reach a pH of approximately 1.

7.4. Spike an aliquot of ^{137}Cs standard into the flask to reach a ^{137}Cs concentration in the desired spike range.

7.5. Fill the volumetric flask to volume with distilled water and mix by inverting.

7.6. Transfer the solution to a 500-mL high-density polyethylene bottle, and label it with a QC sample number.

7.7. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the concentration of ^{137}Cs in the solution as nCi/L or pCi/L.

8.2. Calculate spike concentrations as in the following example.

8.2.1. If 1 mL of a 350-pCi/mL ^{137}Cs standard is used to spike 500 mL of acidified water, the concentration of ^{137}Cs in the acidified water will be 700 pCi/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(350\ \text{pCi/mL})\ (1\ \text{mL})}{(500\ \text{mL})\ (1\ \text{L}/1000\ \text{mL})} = 700\ \text{pCi/L}\ .$$

9. Proper Waste Disposal Practices

9.1. Solid Waste

9.1.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.1.2. Label the can to indicate its use for radioactively contaminated waste.

9.1.3. Open the can only for the period of time necessary to add waste.

9.1.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.1.5. Put a new plastic bag into the waste can for continued use.

9.1.6. Place the sealed bag into a cardboard box lined with a plastic bag and keep the box inside a metal compactable waste container. Reference to the current Waste Profile Request form (WPRF) describing the waste accompanies the sealed bag.

9.1.7. Label the metal container to indicate its use for radioactively contaminated waste.

9.1.8. When the cardboard box is full, seal the plastic bag liner and the box. Check the outside of the box for radioactive contamination and record the information in the Radioactive Waste Disposal logbook.

9.1.9. The radioactive solid waste coordinator marks the cardboard box as radioactively contaminated compactable waste and references the current WPRF numbers describing the waste in the box on the outside of the box and in the Radioactive Waste Disposal logbook.

9.1.10. Place a new lined cardboard box into the metal container for continued use.

9.1.11. Place the sealed box in a dumpster for compactable mixed waste.

9.2. Liquid Waste

9.2.1. Dispose of radioactively contaminated liquid waste in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste Pickup

9.3.1. When the dumpster containing solid waste is full, the surface exposure rate of the dumpster is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.2. Reference all the WPRF numbers associated with the waste in the dumpster on the RSWD.

9.3.3. Request pickup by the Waste Management group of a full dumpster. The RSWD and all the associated WPRFs accompany the dumpster.

9.3.4. The Waste Management group disposes of the waste according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. Amersham Corporation, "Certificate of Measurement of Caesium-137 Radioactive Reference Solution" (Amersham Corporation, Arlington Heights, Illinois, October 1973).

10.3. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4233-B, Cesium-137 Burn-Up Standard" (National Institute of Standards and Technology, Washington, DC, November 1979).

10.4. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.5. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒). Where a section heading is marked, the entire section has been revised.

GROSS ALPHA AND GROSS BETA ON SOIL — QC SAMPLE PREPARATION

Analyte: Gross alpha and gross beta	**Method No.:** QCR140
Matrix: Sand	**Spike Range:** 2.5-10 pCi/g
Procedure: Proportional counting	**Accuracy and Precision:** Gross alpha 100% ± 4.5% RSD Gross beta 100% ± 3.2% RSD
Effective Date: 06/01/90	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.2 and 10.3 for proper waste disposal practices.

1. Principle of Method

1.1. Blank sand spiked with ^{241}Am and ^{90}Sr standards is used in place of soil as the QC matrix for gross alpha and gross beta analysis by proportional counting.

1.2. Gross beta is contributed equally by ^{90}Sr and the ^{90}Y radioactive decay products that are in equilibrium in the ^{90}Sr standard solution.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the pipette and of the balance. A relative standard deviation (RSD) of 4.5% at the 1σ level for gross alpha and 3.2% at the 1σ level for gross beta has been calculated for the calibration, spiking, and weighing process based on the following function:

$$D = \frac{M1 \times M2}{M3},$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = weighing error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of balance, and
100 = factor to convert to percentage.

2.2. Recovery data for proportional counting of QC samples prepared during 1990 using this procedure are published in Source Material 10.1.

3. Collection and Storage of Samples

3.1. Store QC samples in glass beakers covered with plastic wrap at room temperature pending analysis.

4. Apparatus

4.1. Beakers: 100-mL, glass.

4.2. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.3. Pipette tips: disposable.

4.4. Balance: 200-g minimum capacity, 0.1-mg accuracy.

4.5. Spatula.

4.6. Plastic wrap.

4.7. Heat lamp.

4.8. Labels printed with QC sample numbers.

4.9. Filter paper: 2.9-cm, Whatman No. 41 or equivalent.

4.10. Discs: stainless-steel, 2.22-cm-diam.

4.11. Alpha phosphor on Mylar: The phosphor is the standard silver-activated zinc sulfide. William B. Johnson & Associates, Inc., Research Park, Montville, NJ 07045.

4.12. Rings and discs: plastic rings and discs, nominal 1-in.-diam, molded of nylon. Control Molding Corp., 84 Granite Ave., Staten Island, NY 10303.

4.13. Mylar film: 1.5-in.-wide rolls, 0.0005 in. thick. Manufactured by E. I. duPont de Nemours, retailed by Brownell, Inc., 85 Tenth Ave., New York, NY 10001.

5. Reagents

5.1. Sand. A clean, dry 2-g aliquot is required for each sample.

5.2. A suitable sand does not contain the analyte of interest and presents few interferences to the analytical method.

NOTE: Surface soils have been contaminated with radionuclides as a result of the use and testing of nuclear weapons. A suitable blank soil for this procedure would have been collected before World War II or would be collected from a deep well or other excavation where radioactive contamination is not likely to have occurred. Before use, a representative aliquot of soil is analyzed to determine if it will provide a suitably blank matrix.

6. Calibration and Standards

6.1. When certified primary standards are available, dilute them to make standards at the desired concentration.

6.2. Calibrate standard solutions that are not made from certified primary standards against an appropriate certified standard.

6.3. Gross alpha.

6.3.1. Spike QC samples with an ^{241}Am standard prepared in 4 N HNO_3 at a concentration of approximately 180 pCi/mL. Count the solution on a zinc sulfide scintillation system calibrated with National Institute of Standards and Technology (NIST) Standard Reference Material (SRM)

4904L-F, an ^{241}Am alpha-particle standard, using count room procedure CR340.

6.3.2. Calibrate the ^{241}Am standard against an NIST-certified ^{241}Am alpha-particle solution standard, such as NIST SRM 4322. Calibrate the standard solution annually by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.3.3. Direct plating procedure.

6.3.3.1. Pipette a 200- to 300-μL aliquot of the standard to be calibrated on each of six 2.22-cm stainless-steel discs, and evaporate to dryness under a heat lamp.

6.3.3.2. Place a 2.9-cm filter paper on a molded nylon disc. The filter paper assures the package is tightly sealed when assembled.

6.3.3.3. Place the stainless-steel disc containing the sample on the filter paper, making sure to center it.

6.3.3.4. Place a 0.875-in. phosphor paper over the stainless-steel disc (dull side against the disc).

6.3.3.5. Place a piece of Mylar film over all the layers.

6.3.3.6. Press a nylon ring down over the layers and around the nylon disc to form a tightly sealed package. Cut off the excess Mylar with scissors. Label the back of the assembly with an identification number.

6.3.3.7. Submit the samples to the count room for alpha scintillation counting and request a 60,000-s (overnight) count for each sample.

6.3.3.8. The count room data is corrected for background and reported in disintegrations per minute (dpm). Divide the dpm results by 2.22 to convert to pCi. Calculate the mean and standard deviation of the six plates and assign the value to the standard.

6.4. Gross beta.

6.4.1. Spike QC samples with a ^{90}Sr standard in 2 M HCl at a concentration of approximately 180 pCi/mL.

6.4.2. Calibrate the ^{90}Sr standard against an Environmental Protection Agency (EPA)-certified ^{90}Sr standard solution in equilibrium with its daughter ^{90}Y.

6.4.3. Decay the ^{90}Sr standards each year.

6.4.3.1. The half-life of ^{90}Sr is 28.5 years.

6.4.3.2. The following equation describes the rate of physical decay of radionuclides:

$$A_t = A_o e^{-\lambda t} ,$$

where A_t = the activity at time t,
A_o = the activity at t = 0,
e = the base of natural logarithms, and
λ = $(\ln 2)/T_{1/2}$, the decay constant, where $T_{1/2}$ the half-life.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically include a 2-g aliquot of the deep-well sand for use as a blank, omitting the ^{241}Am, the ^{90}Sr, or both.

7.2. Accurately weigh a 2-g aliquot of clean, dry sand into a 100-mL beaker. Record the weight of the sand on the beaker with a felt-tipped pen for reference by the analyst.

7.3. Spike an aliquot of ^{241}Am standard onto the sand in the beaker to reach the desired spike range for gross alpha.

7.4. The gross beta count is contributed equally by ^{90}Sr and the ^{90}Y radioactive decay products, which are in equilibrium in the ^{90}Sr standard solution.

7.4.1. The concentration of the ^{90}Sr standard solution is expressed in terms of ^{90}Sr although half the gross beta count is contributed by ^{90}Sr and the other half by ^{90}Y.

7.4.2. Divide the spike range in half and use the concentration of the ^{90}Sr standard solution to determine the amount of standard solution to use to reach the desired spike range of 1.25-5.0 pCi/g.

7.4.3. Spike the determined amount of ^{90}Sr standard onto the sand in the beaker.

7.4.4. The gross beta count will be within the spike range of 2.5-10.0 pCi/g because ^{90}Y in the standard solution contributes a gross beta count equal to that contributed by the ^{90}Sr. The beta concentration in the QC

sample based on the amount of ^{90}Sr added must be multiplied by 2 to determine the total gross beta concentration.

7.5. Label the beaker with a QC sample number.

7.6. Air dry the QC sample or dry it under a heat lamp to remove excess moisture.

7.7. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Determine background contributions to gross alpha and gross beta counts and add them to the spiked alpha and beta concentrations.

8.2. Record the gross alpha and gross beta concentrations on the sand as pCi/g.

8.3. Calculate the gross alpha spike concentration as in the following example.

8.3.1 If 50 μL of a 180-pCi/mL ^{241}Am standard is spiked onto 2.0000 g of sand, the gross alpha concentration in the sand is 4.5 pCi/g. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(180\ \text{pCi/mL})\ (1\ \text{mL}/1000\ \mu\text{L})\ (50\ \mu\text{L})}{2.0000\ \text{g}} = 4.5\ \text{pCi/g} \quad .$$

8.3.2 Add the alpha background to the alpha concentration from the ^{241}Am spike to obtain the total gross alpha concentration in the QC sample.

$$4.5\ \text{pCi/g} + 4.9\ \text{pCi/g} = 9.4\ \text{pCi/g} \quad .$$

8.4. Calculate the gross beta concentration as in the following example.

8.4.1 If 35 μL of a 174-pCi/mL ^{90}Sr standard is spiked onto 2.000 g of sand, the beta concentration contributed by ^{90}Sr in the sample is 3.0 pCi/g.

$$\frac{(174\ \text{pCi/mL})\ (1\ \text{mL}/1000\ \mu\text{L})\ (35\ \mu\text{L})}{2.0000\ \text{g}} = 3.0\ \text{pCi/g} \quad .$$

8.4.2. Multiply the beta concentration contributed by ^{90}Sr by 2 to determine the combined gross beta concentration contributed by ^{90}Sr and ^{90}Y.

$$(3.0\ pCi/g)\ (2) = 6.0\ pCi/g \quad .$$

8.4.3. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

8.4.4. Add the beta background to the beta concentration contributed by the combined ^{90}Sr and ^{90}Y to obtain the total gross beta concentration in the QC sample.

$$6.0\ pCi/g + 2.4\ pCi/g = 8.4\ pCi/g \quad .$$

9. Proper Waste Disposal Practices

9.1. Liquid waste.

9.1. Dispose of radioactively contaminated liquid waste in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.2. Solid waste.

9.2.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.2.2. Label the can to indicate its use for radioactively contaminated waste.

9.2.3. Open the can only for the period of time necessary to add waste.

9.2.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.2.5. Put a new plastic bag into the waste can for continued use.

9.2.6. Place the sealed bag into a cardboard box lined with a plastic bag and labelled to indicate its use for radioactively contaminated waste.

9.2.7. When the cardboard box is full, seal the plastic bag liner and the box.

9.2.8. Mark the box as radioactively contaminated compactable waste and reference the current Waste Profile Request form (WPRF) describing the waste in the box on the outside of the box.

9.2.9. Line a new box with a plastic bag and label it for continued use.

9.2.10. Complete the current Radioactive Solid Waste Record (RSWD) form describing the full, sealed box of waste. Reference the current WPRF number on the RSWD.

9.2.11. The outside of the full box is surveyed for radioactive contamination by a radiation protection technician. Record the results of the survey on the RSWD.

9.2.12. Submit the completed RSWD and a copy of the WPRF to the Waste Management group for approval.

9.3. Waste pickup.

9.3.1. After the RSWD describing the full box of solid waste has been approved by the Waste Management group, request pickup of the box by the contractor approved for the transportation of radioactively contaminated waste. A written job order may be required to request pickup of waste.

9.3.2. The RSWD and a copy of the associated WPRF accompany the full box.

9.3.3. The waste is disposed of according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.2. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.3. "Hazardous and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒▒). Where a section heading is marked, the entire section has been revised.

GROSS ALPHA AND GROSS BETA IN WATER — QC SAMPLE PREPARATION

Analyte: Gross alpha and gross beta	**Method No.:** QCR150
Matrix: Water	**Spike Range:** 5-20 pCi/L
Procedure: Proportional counting	**Accuracy and Precision:** Gross alpha 100% ± 3.4% RSD Gross beta 100% ± 3.2% RSD
Effective Date: 06/01/90	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.2 and 10.3 for proper waste disposal practices.

1. Principle of Method

1.1. A QC sample for the determination of gross alpha and gross beta in water is prepared by spiking 0.5 M HCl with ^{241}Am and ^{90}Sr standards.

1.2. Gross beta is contributed equally by ^{90}Sr and by the ^{90}Y radioactive decay products that are in equilibrium in the ^{90}Sr standard solution.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the volumetric flask and of the pipette. A relative standard deviation (RSD) of 3.4% at the 1σ level for gross alpha and 3.2% at the 1σ level for gross beta has been calculated for the calibration, spiking, and dilution process based on the following function:

$$D = \frac{M1 \times M2}{M3},$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of volumetric flask, and
100 = factor to convert to percentage.

2.2. Recovery data for proportional counting of QC samples prepared during 1990 using this procedure are published in Source Material 10.1.

3. Collection and Storage of Samples

3.1. QC samples are prepared in an acid medium in a volumetric flask and stored in the flask at room temperature pending analysis.

4. Apparatus

4.1. Volumetric flasks: 1-L, glass, class A.

4.2. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.3. Pipette tips: disposable.

4.4. Labels printed with QC sample numbers.

4.5. Heat lamp.

4.6. Filter paper: 2.9-cm, Whatman No. 41 or equivalent.

4.7. Discs: stainless-steel, 2.22-cm-diam.

4.8. Alpha phosphor on Mylar: The phosphor is the standard silver-activated zinc sulfide. William B. Johnson & Associates, Inc., Research Park, Montville, NJ 07045.

4.9. Rings and discs: plastic rings and discs, nominal 1-in.-diam, molded of nylon. Control Molding Corp., 84 Granite Ave., Staten Island, NY 10303.

4.10. Mylar film: 1.5-in.-wide rolls, 0.0005 in. thick. Manufactured by E. I. duPont de Numours, retailed by Brownell, Inc., 85 Tenth Ave., New York, NY 10001.

5. Reagents

5.1. Hydrochloric acid (concentrated, reagent-grade).

6. Calibration and Standards

6.1. When they are available, dilute certified primary standards to make standards at the desired concentration.

6.2. Calibrate standard solutions which are not made from certified primary standards against an appropriate certified standard.

6.3. Gross alpha.

6.3.1. Spike QC samples with an ^{241}Am standard prepared in 4 N HNO_3 at a concentration of approximately 18 pCi/mL. Count the solution on a zinc sulfide scintillation system calibrated with National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 4904L-F, an ^{241}Am alpha-particle standard, using count room procedure CR340.

6.3.2. Calibrate the ^{241}Am standard against a NIST-certified ^{241}Am alpha-particle solution standard, such as NIST SRM 4322. Calibrate the standard solution annually by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.3.3. Direct plating procedure.

6.3.3.1. Pipette a 200- to 300-μL aliquot of the standard to be calibrated on each of six 2.22-cm stainless-steel discs, and evaporate to dryness under a heat lamp.

6.3.3.2. Place a 2.9-cm filter paper on a molded nylon disc. The filter paper assures the package is tightly sealed when assembled.

6.3.3.3. Place the stainless-steel disc containing the sample on the filter paper, making sure it is centered.

6.3.3.4. Place a 0.875-in. phosphor paper over the stainless-steel disc (dull side against the disc).

6.3.3.5. Place a piece of Mylar film over all the layers.

6.3.3.6. Press a nylon ring down over the layers and around the nylon disc to form a tightly sealed package. Cut off the excess Mylar with scissors. Label the back of the assembly with an identification number.

6.3.3.7. Submit the samples to the count room for alpha scintillation counting and request a 60,000-s (overnight) count for each sample.

6.3.3.8. The count room data is corrected for background and reported in disintegrations per minute (dpm). Divide the dpm results by 2.22 to convert to pCi. Calculate the mean and standard deviation of the six plates and assign the value to the standard.

6.4. Gross beta.

6.4.1. Spike QC samples with a ^{90}Sr standard in 2 M HCl at a concentration of approximately 170 pCi/mL.

6.4.2. Calibrate the ^{90}Sr standard against an Environmental Protection Agency (EPA)-certified ^{90}Sr standard solution in equilibrium with its daughter ^{90}Y.

6.4.3. Decay the ^{90}Sr standards each year.

6.4.3.1. The half-life of ^{90}Sr is 28.5 years.

6.4.3.2. The following equation describes the rate of radioactive decay of radionuclides:

$$A_t = A_o e^{-\lambda t} ,$$

where A_t = the activity at time t,
A_o = the activity at t = 0,
e = the base of natural logarithms, and
λ = $(\ln 2)/T_{1/2}$, the decay constant, where $T_{1/2}$ is the half-life.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically omit the ^{241}Am, the ^{90}Sr, or both from the QC sample for use as a blank.

7.2. Add approximately 800 mL of distilled water to a 1-L volumetric flask.

7.3. Add 43 mL of 12 M hydrochloric acid to the water in the flask to produce a final acid concentration of 0.5 M.

7.4. Spike an aliquot of ^{241}Am standard into the water in the flask to reach the desired spike range for gross alpha.

7.5. The gross beta count is contributed equally by ^{90}Sr and by the ^{90}Y radioactive decay products that are in equilibrium in the ^{90}Sr standard solution.

7.5.1. The concentration of the ^{90}Sr standard solution is expressed in terms of ^{90}Sr although half the gross beta count is contributed by ^{90}Sr and the other half by ^{90}Y.

7.5.2. Divide the spike range in half and use the concentration of the ^{90}Sr standard solution to determine the amount of standard solution to use to reach the desired spike range of 2.5-10.0 pCi/L.

7.5.3. Spike the determined amount of ^{90}Sr standard into the water in the flask.

7.5.4. The gross beta count will be within the spike range of 5-20 pCi/L because ^{90}Y in the standard solution contributes a gross beta count equal to that contributed by the ^{90}Sr. The beta concentration in the QC sample based on the amount of ^{90}Sr added must be multiplied by 2 to determine the total gross beta concentration.

7.6. Dilute the QC sample to volume with distilled water. Mix by inversion. Label the flask with a QC sample number and store at room temperature pending analysis.

7.7. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the concentration of gross alpha and gross beta in the QC sample as pCi/L.

8.2. Calculate the gross alpha spike concentration as in the following example.

8.2.1. If 300 μL of a 17.73-pCi/mL ^{241}Am standard is used to prepare a 1-L QC sample, the gross alpha concentration in the sample is 5.3 pCi/L.

$$\frac{(17.73\ \text{pCi/mL})\ (1\ \text{mL}/1000\ \mu\text{L})\ (300\ \mu\text{L})}{1\ \text{L}} = 5.3\ \text{pCi/L} \quad .$$

8.2.2. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

8.3. Calculate the gross beta spike concentration as in the following example.

8.3.1. If 25 μL of a 174-pCi/mL ^{90}Sr standard is used to prepare a 1-L QC sample, the beta concentration contributed by ^{90}Sr in the sample is 4.4 pCi/L.

$$\frac{(174\ \text{pCi/mL})\ (1\ \text{mL}/1000\ \mu\text{L})\ (25\mu\text{L})}{1\ \text{L}} = 4.4\ \text{pCi/L} \quad .$$

8.3.2. Multiply the beta concentration contributed by ^{90}Sr by 2 to determine the total gross beta concentration contributed by ^{90}Sr and ^{90}Y.

$$(4.4\ \text{pCi/L})\ (2) = 8.8\ \text{pCi/L} \quad .$$

8.3.3. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

9. Proper Waste Disposal Practices

9.1. Liquid waste.

9.1. Dispose of radioactively contaminated liquid waste in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.2. Solid waste.

9.2.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.2.2. Label the can to indicate its use for radioactively contaminated waste.

9.2.3. Open the can only for the period of time necessary to add waste.

9.2.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.2.5. Put a new plastic bag into the waste can for continued use.

9.2.6. Place the sealed bag into a cardboard box lined with a plastic bag and labeled to indicate its use for radioactively contaminated waste.

9.2.7. When the cardboard box is full, seal the plastic bag liner and the box.

9.2.8. Mark the box as radioactively contaminated compactable waste and reference the current Waste Profile Request form (WPRF) describing the waste in the box on the outside of the box.

9.2.9. Line a new box with a plastic bag and label it for continued use.

9.2.10. Complete the current Radioactive Solid Waste Record (RSWD) form describing the full, sealed box of waste. Reference the current WPRF number on the RSWD.

9.2.11. The outside of the full box is surveyed for radioactive contamination by a radiation protection technician. Record the results of the survey on the RSWD.

9.2.12. Submit the completed RSWD and a copy of the WPRF to the Waste Management group for approval.

9.3. Waste pickup.

9.3.1. After the RSWD describing the full box of solid waste has been approved by the Waste Management group, request pickup of the box by the contractor approved for the transportation of radioactively contaminated waste. A written job order may be required to request pickup of waste.

9.3.2. The RSWD and a copy of the associated WPRF accompany the full box.

9.3.3. The waste is disposed of according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.2. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.3. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒▒). Where a section heading is marked, the entire section has been revised.

PLUTONIUM AND AMERICIUM ON SOIL – QC SAMPLE PREPARATION

Analyte: Plutonium-238, Plutonium-239, and Americium-241	**Method No.:** QCR160
Matrix: Soil	**Spike Range:** 0.1 to 1 pCi/g
Procedure: Radiochemistry and alpha spectrometry	**Accuracy and Precision:** ^{238}Pu 100% ± 3.4% RSD ^{239}Pu 100% ± 3.1% RSD ^{241}Am 100% ± 4.5% RSD
Effective Date: 04/12/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.7 and 10.8 for proper waste disposal practices.

1. Principle of Method

1.1. Blank sand is spiked with ^{238}Pu, ^{239}Pu, and ^{241}Am standards.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error from six sample plate preparations of the standard and on the error associated with use of the pipette and of the balance. Relative standard deviations (RSDs) of 3.4% for ^{238}Pu, 3.1% for ^{239}Pu, and 4.5% for ^{241}Am at the 1σ level have been calculated for the calibration, spiking, and weighing process based on the following function:

$$D = \frac{M1 \times M2}{M3},$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = weighing error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of balance, and
100 = factor to convert to percentage.

2.2. Analysis by radiochemistry and alpha spectrometry of QC samples prepared during 1989 using this procedure gave a mean recovery of 89% ± 4% for ^{238}Pu and 99% ± 6% for ^{239}Pu at the 1σ level. The data are published in Source Material 10.1 and are summarized below.

PLUTONIUM SPIKE RECOVERY DURING 1989 AS A FUNCTION OF CONCENTRATION

	Spike range pCi/g	Mean ± SD (%)	Number of samples
^{238}Pu	0.05 to 0.1	87 ± 4	5
	0.1 to 0.3	90 ± 4	7
	0.3 to 0.6	89 ± 5	4
^{239}Pu	0.1 to 0.2	102 ± 7	7
	0.2 to 0.3	94 ± 4	6
	0.3 to 0.5	100 ± 1	3

3. Collection and Storage of Samples

3.1. Spiked QC samples are stored at room temperature pending analysis.

4. Apparatus

4.1. Bottles: 500-mL, high-density polyethylene.

4.2. Balance: 200-g capacity, 0.1-mg accuracy.

4.3. Auto-pipette: 100-μL, Rainin or equivalent.

4.4. Pipette tips: disposable.

4.5. Spatula.

4.6. Heat lamp.

4.7. Labels printed with QC sample numbers.

4.8. Filter paper: 2.9-cm, Whatman No. 41 or equivalent.

4.9. Discs: stainless-steel, 2.22-cm-diam.

4.10. Alpha phosphor on Mylar: The phosphor is the standard silver-activated zinc sulfide. William B. Johnson & Associates, Inc., Research Park, Montville, NJ 07045.

4.11. Rings and discs: plastic rings and discs, nominal 1-in.-diam, molded of nylon. Control Molding Corp., 84 Granite Ave., Staten Island, NY 10303.

4.12. Mylar film: 1.5-in.-wide rolls, 0.0005 in. thick. Manufactured by E. I. duPont de Nemours, retailed by Brownell, Inc., 85 Tenth Ave., New York, NY 10001.

5. Reagents

5.1. Sand. A clean and dry 10-g aliquot is required for each sample.

5.2. A suitable sand does not contain the analyte of interest and presents few interferences to the analytical method.

NOTE: Surface soils have been contaminated with radionuclides as a result of the use and testing of nuclear weapons. A suitable blank soil for this procedure would have been collected before World War II or would be collected from a deep well or other excavation where radioactive contamination is not likely to have occurred. Before use, a representative aliquot of soil is analyzed to determine if it will provide a suitably blank matrix.

6. Calibration and Standards

6.1. QC samples are spiked with ^{238}Pu and ^{239}Pu standards prepared in 2 N HNO_3 and with an ^{241}Am standard prepared in 4 N HNO_3. The plutonium standards are heated at 80°C for 2 h to convert Pu in the polymeric form to the ionic 4+ state.

6.2. The radionuclide standards should have concentrations of approximately 180 pCi/mL.

6.3. When they are available, certified primary standards should be diluted to make standards at the desired concentration.

6.4. Standard solutions which are not made from certified primary standards should be calibrated against an appropriate certified standard.

6.5. The ^{238}Pu standard should be calibrated against a NIST-certified ^{238}Pu alpha-particle solution standard, such as NIST SRM 4323. The standard solution should be recalibrated yearly by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.6. The ^{239}Pu standard should be calibrated against a NIST-certified ^{239}Pu alpha-particle solution standard, such as NIST SRM 4331. The standard solution should be recalibrated yearly by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.7. The ^{241}Am standard should be calibrated against a NIST-certified ^{241}Am alpha-particle solution standard, such as NIST SRM 4322. The standard solution should be recalibrated yearly by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.8. All solutions are counted on a zinc sulfide scintillation system calibrated with National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 4904L-F, an ^{241}Am alpha-particle standard, using count room procedure CR340.

6.9. Direct plating procedure.

6.9.1. Pipette a 200- to 300-μL aliquot of the standard to be calibrated on each of six 2.22-cm stainless-steel discs and evaporate to dryness under a heat lamp.

6.9.2. Place a 2.9-cm filter paper on a molded nylon disc. The filter paper assures a tightly sealed package when assembled.

6.9.3. Place the stainless-steel disc containing the sample on the filter paper, making sure it is centered.

6.9.4. Place a 0.875-in. phosphor paper over the stainless-steel disc (dull side against the disc).

6.9.5. Place a piece of Mylar film over all the layers.

6.9.6. Press a nylon ring down over the layers and around the nylon disc to form a tightly sealed package. Cut off the excess Mylar with scissors. Label the back of the assembly with an identification number.

6.9.7. Submit the samples to the count room for alpha scintillation counting and request a 60,000-s (overnight) per sample count.

6.9.8. The count room data is corrected for background and reported in disintegrations per minute (dpm). Divide the dpm results by 2.22 to convert to pCi. Calculate the mean and standard deviation of the six plates and assign the value to the standard.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically analyze a 10-g aliquot of the unspiked sand as a blank.

7.2. Accurately weigh 10 g of clean, dry sand into a 500-mL high-density polyethylene bottle. Record the weight of sand on the polyethylene bottle with a felt-tipped pen for reference by the analyst.

7.3. Spike aliquots of the Pu and Am standard solutions onto the sand to reach the desired spike range.

7.4. Air-dry the samples or dry under a heat lamp until excess liquid is gone.

7.5. Cap the sample bottles and label with a QC sample number. The entire contents of the bottle must be analyzed.

7.6. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the concentration of ^{238}Pu, ^{239}Pu, and ^{241}Am on the sand as pCi/g.

8.2. Calculate spike concentrations as in the following example.

8.2.1. If 20 μL of a 175-pCi/mL ^{238}Pu standard is used to spike 10.0000 g of sand, the ^{238}Pu concentration in the sand is 0.35 pCi/g. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{\left(175\ \frac{pCi}{mL}\right)\left(\frac{mL}{1000\ \mu L}\right)(20\ \mu L)}{10.0000\ g} = 0.35\ \frac{pCi}{g}$$

8.3. Spike concentrations for ^{239}Pu and ^{241}Am are calculated like spike concentrations for ^{238}Pu using the equation in Step 8.2.1.

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.1.2. Label the can to indicate its use for radioactively contaminated waste.

9.1.3. Open the can only for the period of time necessary to add waste.

9.1.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.1.5. Put a new plastic bag into the waste can for continued use.

9.1.6. Place the sealed bag into a cardboard box lined with a plastic bag and keep the box inside a metal compactable waste container. Reference to the current Waste Profile Request form (WPRF) describing the waste accompanies the sealed bag.

9.1.7. Label the metal container to indicate its use for radioactively contaminated waste.

9.1.8. When the cardboard box is full, seal the plastic bag liner and the box. Check the outside of the box for radioactive contamination and record the information in the Radioactive Waste Disposal logbook.

9.1.9. The radioactive solid waste coordinator marks the cardboard box as radioactively contaminated compactable waste and references the current WPRF numbers describing the waste in the box on the outside of the box and in the Radioactive Waste Disposal logbook.

9.1.10. Place a new lined cardboard box into the metal container for continued use.

9.1.11. Place the sealed box in a dumpster for compactable mixed waste.

9.2. Liquid waste.

9.2.1. Dispose of liquid waste that is radioactively contaminated in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. When the dumpster containing solid waste is full, the surface exposure rate of the dumpster is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.2. Reference all the WPRF numbers associated with the waste in the dumpster on the RSWD.

9.3.3. Request pickup by the Waste Management group of a full dumpster. The RSWD and all the associated WPRFs accompany the dumpster.

9.3.4. The Waste Management group disposes of the waste according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. *EML Procedures Manual*, 25th ed., H. L. Volchok and G. de Planque, Eds. (Environmental Measurements Laboratory, New York, August 1982).

10.3. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4323, Plutonium-238 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, November 1986).

10.4. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4322, Americium-241 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, November 1986).

10.5. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4331, Plutonium-239 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, March 1975).

10.6. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4904L-F, Alpha-Particle Standard" (National Institute of Standards and Technology, Washington, DC, April 1982).

10.7. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.8. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒). Where a section heading is marked, the entire section has been revised.

URANIUM-238 IN URINE — QC SAMPLE PREPARATION

Analyte: Uranium-238	**Method No.:** QCR300
Matrix: Urine	**Spike Range:** 0.0 to 34.0 μg/L
Procedure: Delayed neutron activation	**Accuracy and Precision:** 100% ± 2.8% RSD
Effective Date: 01/01/75 to 08/01/91	**Authors:** Margaret A. Gautier William D. Moss Nancy L. Koski

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment.

1. Principle of Method

1.1. A pool of blank urine is prepared.

1.2. The blank urine is aliquoted and spiked with a known standard.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error from six sample plate preparations of the standard and on the error associated with use of the graduated cylinder and of the pipette. A relative standard deviation (RSD) of 2.8% has been calculated for the calibration, spiking, and aliquoting process based on the following function:

$$U = \frac{M1 \times M2}{M3 + M2} ,$$

where U = uncertainty,
M1 = calibration error,
M2 = spiking error, and
M3 = aliquoting error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{\frac{\left[R_{M1}^2 (M_3 + M_2)^2 + M_3^2 (R_{M2}^2 + R_{M3}^2)\right]}{(M_3 + M_2)^2}} \times 100 ,$$

where S_{D_T} = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
M_3 = volume of cylinder,
M_2 = volume of pipette,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of cylinder, and
100 = factor to convert to percentage.

2.2. Analysis by delayed neutron activation of QC samples prepared between 1984 and 1989 using this procedure gave a mean recovery of 98% ± 16 % RSD. The large standard deviation associated with the ^{238}U analysis in urine is related to the measurement of depleted uranium and its corresponding low production of delayed neutron counts. The ^{238}U standard used for the bioassay program is only 0.18% ^{235}U. The data are published in **Source Material** 9.1 through 9.6 and are summarized below.

URANIUM-238 SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION

	Spike Level (μg/L)	Mean ± RSD (%)	Number of Samples
1984	8.48	101 ± 30	16
	12.72	102 ± 23	14
	16.96	103 ± 26	15
	25.44	99 ± 30	7
	33.92	100 ± 5	15
	42.40	97 ± 5	5
	50.88	93 ± 6	6
1985	8.48	94 ± 35	14
	12.72	103 ± 21	9
	16.96	91 ± 17	13
	21.2	97 ± 7	7
	25.44	92 ± 16	14
	33.92	98 ± 7	12
1986	8.48	85 ± 19	8
	12.72	101 ± 24	11
	16.96	92 ± 14	12
	21.20	103 ± 12	11
	25.44	94 ± 14	15
	33.92	99 ± 10	14
1987	8.48	114 ± 20	12
	12.72	92 ± 21	9
	16.96	100 ± 17	14
	21.20	103 ± 21	12
	25.44	108 ± 19	14
	33.92	99 ± 12	14
1988	8.48	99 ± 27	13
	12.72	115 ± 19	8
	16.96	93 ± 16	10
	21.20	105 ± 10	8
	25.44	98 ± 11	13
	33.92	99 ± 15	11
1989	8.48	95 ± 16	13
	12.72	98 ± 18	11
	16.96	97 ± 10	8
	21.20	90 ± 18	12
	25.44	96 ± 6	10
	33.92	92 ± 8	18

3. Collection and Storage of Samples

3.1. Urine is collected as needed from group personnel who have no known contact with radiation work and is used to form a pool of blank urine.

3.2. Spiked QC samples are refrigerated at a nominal temperature of 4°C pending analysis.

4. Apparatus

4.1. Beakers: 4- or 5-L, polyethylene.

4.2. Graduated cylinders: 50-mL.

4.3. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.4. Pipette tips: disposable.

4.5. Specimen containers: 120-mL with screw caps, sterile inner surface, catalog no. C8845-3, Baxter Health Care Corp., Tempe, AZ 85282, or equivalent.

4.6. Labels printed with QC sample numbers.

4.7. Heat lamp.

4.8. Filter paper: 2.9-cm, Whatman No. 41 or equivalent.

4.9. Discs: stainless steel, 2.22-cm-diam.

4.10. Alpha phosphor on Mylar: The phosphor is the standard silver-activated zinc sulfide. William B. Johnson & Associates, Inc., Research Park, Montville, NJ 07045.

4.11. Rings and discs: plastic rings and discs, nominal 1-in.-diam, molded of nylon. Control Molding Corp., 84 Granite Ave., Staten Island, NY 10303.

4.12. Mylar film: 1.5-in.-wide rolls, 0.0005-in.-thick. Manufactured by E. I. duPont de Nemours, retailed by Brownell, Inc., 85 Tenth Ave., New York, NY 10001.

5. Reagents

5.1. None required.

RADIONUCLIDES ON VEGETATION ASH — QC SAMPLE PREPARATION

Analyte: Plutonium-238, Plutonium-239, Americium-241, and Strontium-90	**Method No:** QCR170
Matrix: Vegetation ash	**Spike Range:** 1 to 10 pCi/g
Procedure: Radiochemistry and alpha spectrometry or gas-flow proportional counting	**Accuracy and Precision:** ^{238}Pu 100% ± 3.5% RSD ^{239}Pu 100% ± 3.2% RSD ^{241}Am 100% ± 4.5% RSD ^{90}Sr 100% ± 3.2% RSD
Effective Date: 06/15/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.7 and 10.8 for proper waste disposal practices.

1. Principle of Method

1.1. Blank vegetation ash is spiked with ^{238}Pu, ^{239}Pu, ^{241}Am, and ^{90}Sr standards.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the pipette and of the balance. Relative standard deviations (RSDs) of 3.5% for ^{238}Pu, 3.2% for ^{239}Pu, 4.5% for ^{241}Am, and 3.2% for ^{90}Sr at the 1σ level, has been calculated for the calibration, spiking, and weighing process based on the following function:

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = weighing error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} \quad ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2} \times 100 \quad ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of balance, and
100 = factor to convert to percentage.

2.2. Analysis by radiochemistry and alpha spectrometry of QC samples prepared during 1989 using this procedure gave a mean recovery of 90% ± 7% for ^{239}Pu, 98% ± 9% for ^{238}Pu, and 90% ± 9% for ^{241}Am. The data are published in Source Material 10.1 and are summarized below.

SPIKE RECOVERY DURING 1989 AS A FUNCTION OF CONCENTRATION

	Spike range pCi/g	Mean ± SD (%)	Number of samples
^{238}Pu	0.5 to 2.0	88 ± 3	3
	2.0 to 4.5	91 ± 10	3
^{239}Pu	0.5 to 1.5	94 ± 8	4
	1.5 to 2.0	107 ± 4	2
^{241}Am	1.0 to 2.0	93 ± 7	3
	2.0 to 4.0	85 ± 12	2

3. Collection and Storage of Samples

3.1. Spiked QC samples are covered with plastic wrap and stored at room temperature pending analysis.

4. Apparatus

4.1. Beakers: 600-mL, glass.

4.2. Balance: 300-g capacity, 0.1-mg accuracy.

4.3. Auto-pipette: 100-μL, Rainin or equivalent.

4.4. Pipette tips: disposable.

4.5. Plastic wrap.

4.6. Labels printed with QC sample numbers.

4.7. Filter paper: 2.9-cm, Whatman No. 41 or equivalent.

4.8. Discs: stainless-steel, 2.22-cm-diam.

4.9 Alpha phosphor on Mylar: The phosphor is the standard silver-activated zinc sulfide. William B. Johnson & Associates, Inc., Research Park, Montville, NJ 07045.

4.10. Rings and discs: plastic rings and discs, nominal 1-in.-diam, molded of nylon. Control Molding Corp., 84 Granite Ave., Staten Island, NY 10303.

4.11. Mylar film: 1.5-in.-wide rolls, 0.0005 in. thick. Manufactured by E. I. duPont de Nemours, retailed by Brownell, Inc., 85 Tenth Ave., New York, NY 10001.

5. Reagents

5.1. Vegetation ash. A 1-g aliquot is required for each sample.

5.2. A suitable vegetation ash does not contain the analyte of interest and presents few interferences to the analytical method. Commercially available dry pet or farm stock foods may provide a suitable vegetation ash. Previously analyzed vegetation ash samples that did not contain Pu, Am, or Sr may also be used as a QC matrix.

6. Calibration and Standards

6.1. QC samples are spiked with either ^{238}Pu and ^{239}Pu standards prepared in 2 N HNO_3, an ^{241}Am standard prepared in 4 N HNO_3, or a ^{90}Sr standard prepared in 2 M HCl. The plutonium standards are heated at 80°C for 2 h to convert Pu in the polymeric form to the ionic 4+ state.

6.2. The radionuclide standards should have concentrations of approximately 180 pCi/mL.

6.3. When they are available, certified primary standards should be diluted to make standards at the desired concentration.

6.4. Standard solutions which are not made from certified primary standards should be calibrated against an appropriate certified standard.

6.5. The ^{238}Pu standard should be calibrated against a NIST-certified ^{238}Pu alpha-particle solution standard, such as NIST SRM 4323. The standard solution should be recalibrated yearly by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.6. The ^{239}Pu standard should be calibrated against a NIST-certified ^{239}Pu alpha-particle solution standard, such as NIST SRM 4331. The standard solution should be recalibrated yearly by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.7. The ^{241}Am standard should be calibrated against a NIST-certified ^{241}Am alpha-particle solution standard, such as NIST SRM 4322. The standard solution should be recalibrated yearly by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook

6.8. The Pu and Am solutions are counted on a zinc sulfide scintillation system calibrated with National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 4904L-F, and ^{241}Am alpha-particle standard, using count room procedure CR340.

6.9. Direct plating procedure.

6.9.1. Pipette a 200- to 300-μL aliquot of the standard to be calibrated on each of six 2.22-cm stainless-steel discs, and evaporate to dryness under a heat lamp.

6.9.2. Place a 2.9-cm filter paper on a molded nylon disc. The filter paper assures a tightly sealed package when assembled.

6.9.3. Place the stainless-steel disc containing the sample on the filter paper, making sure it is centered.

6.9.4. Place a 0.875-in. phosphor paper over the stainless steel disc (dull side against the disc).

6.9.5. Place a piece of Mylar film over all the layers.

6.9.6. Press a nylon ring down over the layers and around the nylon disc to form a tightly sealed package. Cut off the excess Mylar with scissors. Label the back of the assembly with an identification number.

6.9.7. Submit the samples to the count room for alpha scintillation counting and request a 60,000-s (overnight) per sample count.

6.9.8. The count room data is corrected for background and reported in disintegrations per minute (dpm). Divide the dpm results by 2.22 to convert to pCi. Calculate the mean and standard deviation of the six plates and assign the value to the standard.

6.10. The ^{90}Sr standard should be calibrated against a NIST or an Environmental Protection Agency (EPA)-certified ^{90}Sr standard solution in equilibrium with its daughter ^{90}Y.

6.11. Decay the ^{90}Sr standards each year

6.11.1. The half-life of ^{90}Sr is 28.5 years.

6.11.2. The following equation describes the rate of physical decay of radionuclides:

$$A_t = A_o e^{-\lambda t} ,$$

where A_t = the activity at time t,
A_o = the activity at t = 0,
e = the base of natural logarithms, and
λ = $(\ln 2)/T_{1/2}$, the decay constant, $T_{1/2}$ where is the half-life.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically analyze a 1-g aliquot of the unspiked vegetation ash as a blank.

7.2. Accurately weigh 1 g of vegetation ash into a 600-mL beaker. Record the weight of the ash on the beaker with a felt-tipped pen for reference by the analyst.

7.3. Spike aliquots of the Pu, Am, or Sr standard solutions onto the vegetation ash to reach the desired spike range.

7.4. Cover the beaker with plastic wrap and label it with a QC sample number. The entire contents of the beaker must be analyzed.

7.5. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the concentration of ^{238}Pu, ^{239}Pu, ^{241}Am, and ^{90}Sr on the vegetation ash as pCi/g.

8.2. Calculate spike concentrations as in the following example.

8.2.1. If 15 μL of a 175-pCi/mL ^{238}Pu standard is used to spike 1.0000 g of vegetation ash, the ^{238}Pu concentration in the ash is 2.6 pCi/g. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{\left(175\ \frac{pCi}{mL}\right)\left(\frac{mL}{1000\ \mu L}\right)(15\ \mu L)}{1.0000\ g} = 2.6\ \frac{pCi}{g}$$

8.3. Spike concentrations for ^{239}Pu, ^{241}Am, and ^{90}Sr are calculated like spike concentrations for ^{238}Pu using the equation in Step 8.2.1.

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.1.2. Label the can to indicate its use for radioactively contaminated waste.

9.1.3. Open the can only for the period of time necessary to add waste.

9.1.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.1.5. Put a new plastic bag into the waste can for continued use.

9.1.6. Place the sealed bag into a cardboard box lined with a plastic bag and keep the box inside a metal compactable waste container. Reference to the current Waste Profile Request form (WPRF) describing the waste accompanies the sealed bag.

9.1.7. Label the metal container to indicate its use for radioactively contaminated waste.

9.1.8. When the cardboard box is full, seal the plastic bag liner and the box. Check the outside of the box for radioactive contamination and record the information in the Radioactive Waste Disposal logbook.

9.1.9. The radioactive solid waste coordinator marks the cardboard box as radioactively contaminated compactable waste and references the current WPRF numbers describing the waste in the box on the outside of the box and in the Radioactive Waste Disposal logbook.

9.1.10. Place a new lined cardboard box into the metal container for continued use.

9.1.11. Place the sealed box in a dumpster for compactable mixed waste.

9.2. Liquid waste.

9.2.1. Dispose of liquid waste that is radioactively contaminated in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. When the dumpster containing solid waste is full, the surface exposure rate of the dumpster is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.2. Reference all the WPRF numbers associated with the waste in the dumpster on the RSWD.

9.3.3. Request pickup by the Waste Management group of a full dumpster. The RSWD and all the associated WPRFs accompany the dumpster.

9.3.4. The Waste Management group disposes of the waste according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. *EML Procedures Manual*, 25th ed., H.L. Volchok and G. de Planque, Eds. (Environmental Measurements Laboratory, New York, August 1982).

10.3. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4323, Plutonium-238 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, November 1986).

10.4. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4331, Plutonium-239 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, March 1975).

10.5. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4322, Americium-241 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, November 1986).

10.6. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4904L-F, Alpha-Particle Standard" (National Institute of Standards and Technology, Washington, DC, April 1982).

10.7. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.8. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒). Where a section heading is marked, the entire section has been revised.

PLUTONIUM AND AMERICIUM IN WATER — QC SAMPLE PREPARATION

Analyte: Plutonium-238, Plutonium-239, and Americium-241	**Method No.:** QCR180
Matrix: Water	**Spike Range:** 0.1 to 5 pCi/L
Procedure: Radiochemistry and alpha spectrometry	**Accuracy and Precision:** ^{238}Pu 100% ± 5.2% RSD ^{239}Pu 100% ± 1.7% RSD ^{241}Am 100% ± 1.9% RSD
Effective Date: 08/02/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.7 and 10.8 for proper waste disposal practices.

1. Principle of Method

1.1. Acidified blank water is spiked with ^{238}Pu, ^{239}Pu, and ^{241}Am standards.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error from six sample plate preparations of the standard and on the error associated with use of the volumetric flask and of the pipette. Relative standard deviations (RSDs) of 5.2% for ^{238}Pu, 1.7% for ^{239}Pu, and 1.9% for ^{241}Am, at the 1σ level have been calculated for the calibration, spiking, and dilution process based on the following function:

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where $R^2{}_M$ = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2} \times 100 ,$$

where SD_T = relative standard deviation (% error),
$R^2{}_{M1}$ = relative variance of standard,
$R^2{}_{M2}$ = relative variance of pipette,
$R^2{}_{M3}$ = relative variance of volumetric flask, and
100 = factor to convert to percentage.

2.2. Analysis by radiochemistry and alpha spectrometry of QC samples prepared during 1989 using this procedure gave a mean recovery of 89% ± 6% for ^{238}Pu and 94% ± 11% for ^{239}Pu at the 1σ level. The data are published in Source Material 10.1 and are summarized below.

PLUTONIUM SPIKE RECOVERY DURING 1989
AS A FUNCTION OF CONCENTRATION

	Spike range pCi/g	Mean ± SD (%)	Number of samples
^{238}Pu	0.2 to 0.5	81 ± 15	3
	0.5 to 1.2	91 ± 6	7
^{239}Pu	0.2 to 0.5	86 ± 11	6
	0.5 to 1.0	99 ± 9	9

3. Collection and Storage of Samples

3.1. QC samples are prepared in an acid medium in a volumetric flask and are stored in the flask at room temperature pending analysis.

4. Apparatus

4.1. Volumetric flasks: 500-mL, glass, class A.

4.2. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.3. Pipette tips: disposable.

4.4. Pipette: 2-mL, class A volumetric.

4.5. Labels printed with QC sample numbers.

4.6. Heat lamp.

4.7. Filter paper: 2.9-cm, Whatman No. 41 or equivalent.

4.8. Discs: stainless-steel, 2.22-cm-diam.

4.9. Alpha phosphor on Mylar: The phosphor is the standard silver-activated zinc sulfide. William B. Johnson & Associates, Inc., Research Park, Montville, NJ 07045.

4.10. Rings and discs: plastic rings and discs, nominal 1-in.-diam, molded of nylon. Control Molding Corp., 84 Granite Ave., Staten Island, NY 10303.

4.11. Mylar film: 1.5-in.-wide rolls, 0.0005 in. thick. Manufactured by E. I. duPont de Nemours, retailed by Brownell, Inc., 85 Tenth Ave., New York, NY 10001.

5. Reagents

5.1. Hydrochloric acid (concentrated, reagent-grade).

6. Calibration and Standards

6.1. QC samples are spiked with ^{238}Pu and ^{239}Pu standards prepared in 2 N HNO_3 and with an ^{241}Am standard prepared in 4 N HNO_3. The plutonium standards are heated at a nominal temperature of 80°C for 2 h to convert Pu in the polymeric form to the ionic 4+ state. All solutions are counted on a zinc sulfide scintillation system calibrated with National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 4904L-F, an ^{241}Am alpha-particle standard, using count room procedure CR340.

6.2. The ^{238}Pu and ^{239}Pu standards should be at a concentration of approximately 2 pCi/mL. The ^{241}Am standard should be at a concentration of approximately 6 pCi/mL.

6.3. The ^{238}Pu standard was calibrated against a NIST-certified ^{238}Pu alpha-particle solution standard, such as NIST SRM 4323. Recalibrate the standard solution yearly by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.4. The ^{239}Pu standard was calibrated against a NIST-certified ^{239}Pu alpha-particle solution standard, such as NIST SRM 4331. Recalibrate the standard solution yearly by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.5. The ^{241}Am standard was calibrated against a NIST-certified ^{241}Am alpha-particle solution standard, such as NIST SRM 4322. Recalibrate the standard solution yearly by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.6. Direct plating procedure.

6.6.1. Pipette a 200- to 300-μL aliquot of the standard to be calibrated on each of six 2.22-cm stainless-steel discs and evaporate to dryness under a heat lamp.

6.6.2. Place a 2.9-cm filter paper on a molded nylon disc. The filter paper assures a tightly sealed package when assembled.

6.6.3. Place the stainless-steel disc containing the sample on the filter paper, making sure it is centered.

6.6.4. Place a 0.875-in. phosphor paper over the stainless steel disc (dull side against the disc).

6.6.5. Place a piece of Mylar film over all the layers.

6.6.6. Press a nylon ring down over the layers and around the nylon disc to form a tightly sealed package. Cut off the excess Mylar with scissors. Label the back of the assembly with an identification number.

6.6.7. Submit the samples to the count room for alpha scintillation counting and request a 60,000-s (overnight) per sample count.

6.6.8. The count room data is corrected for background and reported in disintegrations per minute (dpm). Divide the dpm results by 2.22 to convert to pCi. Calculate the mean and standard deviation of the six plates and assign the value to the standard.

6.7. Alternatively, an Environmental Protection Agency (EPA) Plutonium in Water cross check sample may be used as a QC sample. After dilution and distribution of a cross check study, EM-9 transfers the remainder of the stock solution to a glass container for storage to prevent the loss of the radionuclides to the walls of the EPA polyethylene container.

6.7.1. The EPA records the concentration of the standard after dilution according to EPA instructions.

6.7.2. The concentration of the original stock solution must be back-calculated before it is used, taking the EPA dilution instructions into consideration.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically include an unspiked water sample acidified to 3 N with HCl as a blank.

7.2. Place approximately 300 mL of distilled water into a 500-mL volumetric flask.

7.3. Add 125 mL of 12 M HCl to produce a final acid concentration of 3 M.

7.4. Spike aliquots of the Pu and Am standard solutions into the water to reach the desired spike range.

7.5. Dilute the QC sample to volume using distilled water. Mix by inversion. Label the flask with a QC sample number and store at room temperature pending analysis.

7.6. EPA cross check standard.

7.6.1. The EPA standard contains only plutonium.

7.6.2. Back-calculate the concentration of the original stock before an aliquot is taken. See Step 8.3.

7.6.3. EPA recommends mixing the standard for 10 to 15 min before an aliquot is taken.

7.7. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the concentration of ^{238}Pu, ^{239}Pu, and ^{241}Am in the water as pCi/L.

8.2. Calculate spike concentrations as in the following example.

8.2.1. If 300 μL of a 2-pCi/mL ^{238}Pu standard is used to spike 450 mL of acidified water and the solution is diluted to 500 mL, the ^{238}Pu concentration in the acidified water is 1.2 pCi/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(2\ \text{pCi/mL})\ (1\ \text{mL}/1000\ \mu\text{L})\ (300\ \mu\text{L})}{(500\ \text{mL})\ (1\ \text{L}/1000\ \text{mL})} = 1.2\ \text{pCi/L} \quad .$$

8.2.2. Spike concentrations for ^{239}Pu and ^{241}Am are calculated like spike concentrations for ^{238}Pu using the equation in Step 8.2.1.

8.3. If an EPA cross check Plutonium in Water sample will be used to make a QC standard for plutonium in water, the undiluted concentration of the cross check sample must first be calculated.

8.3.1. If the concentration of the EPA sample is 10.6 ± 1.1 pCi/L after dilution of a 500-mL aliquot to 4 L, the concentration of the undiluted sample is calculated as follows:

$$(10.6\ \text{pCi/L})\ (4\ \text{L}/500\ \text{mL})\ (1000\ \text{mL/L}) = 84.8\ \text{pCi/L} \quad .$$

The concentration of 84.8 pCi/L is then used to determine the aliquot used to spike the acidified water sample.

8.3.2. If 4 mL of the 84.8-pCi/L EPA sample is spiked into 400 mL of acidified water and is diluted to 500 mL with distilled water, the plutonium concentration is 0.68 pCi/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(84.8\ \text{pCi/L})\ (1\ \text{L}/1000\ \text{mL})\ (4\ \text{mL})}{(500\ \text{mL})\ (1\ \text{L}/1000\ \text{mL})} = 0.68\ \text{pCi/L} \quad .$$

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.1.2. Label the can to indicate its use for radioactively contaminated waste.

9.1.3. Open the can only for the period of time necessary to add waste.

9.1.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.1.5. Put a new plastic bag into the waste can for continued use.

9.1.6. Place the sealed bag into a cardboard box lined with a plastic bag and keep the box inside a metal compactable waste container. Reference to the current Waste Profile Request form (WPRF) describing the waste accompanies the sealed bag.

9.1.7. Label the metal container to indicate its use for radioactively contaminated waste.

9.1.8. When the cardboard box is full, seal the plastic bag liner and the box. Check the outside of the box for radioactive contamination and record the information in the Radioactive Waste Disposal logbook.

9.1.9. The radioactive solid waste coordinator marks the cardboard box as radioactively contaminated compactable waste and references the current WPRF numbers describing the waste in the box on the outside of the box and in the Radioactive Waste Disposal logbook.

9.1.10. Place a new lined cardboard box into the metal container for continued use.

9.1.11. Place the sealed box in a dumpster for compactable mixed waste.

9.2. Liquid waste.

9.2.1. Dispose of radioactively contaminated liquid waste in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. When the dumpster containing solid waste is full, the surface exposure rate of the dumpster is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.2. Reference all the WPRF numbers associated with the waste in the dumpster on the RSWD.

9.3.3. Request pickup by the Waste Management group of a full dumpster. The RSWD and all the associated WPRFs accompany the dumpster.

9.3.4. The Waste Management group disposes of the waste according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. *EML Procedures Manual*, 25th ed., H. L. Volchok and G. de Planque, Eds. (Environmental Measurements Laboratory, New York, August 1982).

10.3. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4323, Plutonium-238 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, November 1986).

10.4. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4331, Plutonium-239 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, March 1975).

10.5. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4322, Americium-241 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, November 1986).

10.6. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4904L-F, Alpha-Particle Standard" (National Institute of Standards and Technology, Washington, DC, April 1982).

10.7. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.8. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒). Where a section heading is marked, the entire section has been revised.

PLUTONIUM-238 IN URINE — QC SAMPLE PREPARATION

Analyte: Plutonium-238	**Method No.:** QCR190
Matrix: Urine	**Spike Range:** 0.0-0.40 pCi/sample
Procedure: Radiochemistry and alpha spectrometry	**Accuracy and Precision:** 100% ± 3.7% RSD
Effective Date: 01/01/75	**Authors:** Margaret A. Gautier William D. Moss Nancy L. Koski

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.11 to 10.13 for proper waste disposal.

1. Principle of Method

1.1. A pool of blank urine is prepared.

1.2. The blank urine is aliquoted and spiked with a known standard.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error from six sample plate preparations of the standard and on the error associated with the use of the graduated cylinder and the pipette. A relative standard deviation (RSD) of 3.7% at the 1σ level has been calculated for the calibration, spiking, and aliquoting process based on the following:

$$D = \frac{M1 \times M2}{M3 + M2} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = aliquoting error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{\frac{\left[R^2_{M1}\,(M_3 + M_2)^2 + M_3^2\,(R^2_{M2} + R^2_{M3})\right]}{(M_3 + M_2)^2}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
M_3 = volume of cylinder,
M_2 = volume of pipette,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of cylinder, and
100 = factor to convert to percentage.

2.2. Analysis by radiochemistry and alpha spectrometry of QC samples prepared between 1984 and 1989 using this procedure gave a mean recovery of 99% ± 14% RSD. The data are published in Source Materials 10.1 through 10.6 and are summarized below.

PLUTONIUM-238 SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION AFTER CORRECTION FOR PLUTONIUM-242 RECOVERY

Year	Spike level pCi/sample	Mean ± RSD (%)	Number of samples
1984	0.06	82 ± 20	13
	0.12	93 ± 12	15
	0.20	89 ± 11	14
	0.40	89 ± 9	16

PLUTONIUM-238 SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION AFTER CORRECTION FOR PLUTONIUM-242 RECOVERY (cont)

Year	Spike level pCi/sample	Mean ± RSD (%)	Number of samples
1985	0.06	98 ± 20	9
	0.12	97 ± 17	18
	0.20	100 ± 14	11
	0.40	103 ± 7	11
1986	0.06	97 ± 21	13
	0.12	104 ± 11	14
	0.20	103 ± 11	11
	0.40	110 ± 8	11
1987	0.06	102 ± 16	12
	0.12	95 ± 11	11
	0.19	105 ± 5	12
	0.39	96 ± 10	12
1988	0.06	102 ± 22	13
	0.12	97 ± 7	13
	0.19	102 ± 10	14
	0.39	100 ± 8	13
1989	0.06	102 ± 24	18
	0.12	99 ± 10	16
	0.19	114 ± 18	15
	0.39	100 ± 15	12

3. Collection and Storage of Samples

3.1. Urine is collected as needed from group personnel who have no known contact with radiation work and is used to form a pool of blank urine.

3.2. Spiked QC samples are refrigerated at a nominal temperature of 4°C pending analysis.

4. Apparatus

4.1. Beakers: 4- or 5-L, polyethylene.

4.2. Graduated cylinders: 500-mL.

4.3. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.4. Pipette tips: disposable.

4.5. Specimen containers: 16-oz jars with screw caps. VWR Scientific Catalog No. 16193-810 or equivalent.

4.6. Labels printed with QC sample numbers.

4.7. Heat lamp.

4.8. Filter paper: 2.9-cm, Whatman No. 41 or equivalent.

4.9. Discs: stainless steel, 2.22-cm diam.

4.10. Alpha phosphor on Mylar: The phosphor is the standard silver-activated zinc sulfide. William B. Johnson & Associates, Inc., Research Park, Montville, NJ 07045.

4.11. Rings and discs: plastic rings and discs, nominal 1-in. diam, molded of nylon. Control Molding Corp., 84 Granite Ave., Staten Island, NY 10303.

4.12. Mylar film: 1.5-in.-wide rolls, 0.0005 in. thick. Manufactured by E. I. duPont de Nemours, retailed by Brownell, Inc., 85 Tenth Ave., New York, NY 10001.

5. Reagents

5.1. None required.

6. Calibration and Standards

6.1. QC samples are spiked with a ^{238}Pu standard prepared in 2 N HNO_3 and heated at a nominal temperature of 80°C for 2 h to convert Pu in the polymeric form to the ionic 4+ state. The solution is counted on a zinc sulfide scintillation system calibrated with National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 4904L-F, an ^{241}Am alpha-particle standard, using count room procedure CR340.

6.2. The ^{238}Pu standard should be at a concentration of approximately 0.8 pCi/mL.

6.3. The ^{238}Pu standard was calibrated against a NIST-certified ^{238}Pu alpha-particle solution standard, such as SRM 4323. Recalibrate the standard solution yearly by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.4. Direct plating procedure.

6.4.1. Pipette a 200- to 300-μL aliquot of the standard to be calibrated on each of six 2.22-cm stainless-steel discs, and evaporate to dryness under a heat lamp.

6.4.2. Place a 2.9-cm filter paper on a molded nylon disc. The filter paper assures a tightly sealed package when assembled.

6.4.3. Place the stainless-steel disc containing the sample on the filter paper. Be sure they are centered.

6.4.4. Place a 0.875-in. phosphor paper over the stainless-steel disc (dull side against the disc).

6.4.5. Place a piece of Mylar film over all the layers.

6.4.6. Press a nylon ring down over the layers and around the nylon disc to form a tightly sealed package. Cut off the excess Mylar with scissors. Label the back of the assembly with an identification number.

6.4.7. Submit the samples to the count room for alpha scintillation counting and request a 60,000-s (overnight) per sample count.

6.4.8. The count room data are corrected for background and reported in disintegrations per minute (dis/min). Divide the dis/min results by 2.22 to convert to pCi. Calculate the mean and standard deviation of the six plates and assign the value to the standard.

7. Procedure

7.1. Working with bodily fluids is discussed in detail in SOP-4, "Handling Human Biological Fluids," found in Appendix III of the EM-9 Safety Manual.

NOTE: Wear safety glasses, gloves, and a lab coat. Each day prepare a 1:100 dilution of household bleach in water in a spray bottle. Use this solution to thoroughly clean the work area after sample preparation is completed. Remove gloves, discard in the biohazard waste container, and wash hands with soap and water.

7.2. Prepare a pool from blank urine sufficient in volume to allow 400 mL for each QC sample to be prepared.

7.3. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Include blank QCs prepared from the same urine pool as the spiked samples.

7.4. Record QC standard information, sample spike amount, concentration, and uncertainty in the QC Laboratory notebook.

7.5. Label bottles with QC sample numbers.

7.6. Aliquot 400 mL of blank urine into each sample bottle.

7.7. Spike the blank urine with aliquots of standard as determined in Step 7.3.

7.8. Screw caps on bottles and refrigerate at a nominal temperature of 4°C pending analysis.

7.9. Enter the spike concentrations of each QC into the CVS and CVD data bases on the VAX. This procedure is done annually because the same concentration levels are used throughout the year.

8. Calculations

8.1. Record spike concentrations as pCi/sample of urine.

8.2. Calculate the spike concentration for a 150 μL aliquot of ^{238}Pu standard using the following equation.

$$\text{pCi/sample per spike amount} = (0.8\ \text{pCi/mL}) \times 0.150\ \text{mL} = 0.12\ \text{pCi/sample}$$

8.3. Spike concentrations calculated in Step 8.2 are tabulated below.

Spike amount (μL)	Concentration (pCi/sample)	Uncertainty (± 3.7%)
75	0.06	0.002
150	0.12	0.004
250	0.20	0.007
500	0.40	0.015

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Radiochemically contaminated waste.

9.1.1.1. Radiochemically contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, is accumulated in a covered metal can lined with a plastic bag and kept segregated from nonradioactive solid waste.

9.1.1.2. The can is labeled with a hazardous waste label and a label indicating its use for radiochemically contaminated waste.

9.1.1.3. The can is opened only for the time necessary to add waste.

9.1.1.4. When the plastic bag is full, it is sealed with tape, removed from the can, identified as radiochemically contaminated waste, and placed in a functioning chemical exhaust hood pending pickup by the Waste Management group.

9.1.1.5. A new plastic bag is put into the waste can for continued use.

9.1.2. Biohazardous waste.

9.1.2.1. Bottles used to collect blank urine for a quality control sample matrix are disposed of as uncontaminated laboratory trash after they have been rinsed with water in a laboratory sink.

9.1.2.2. Solid waste, such as paper wipes, plastic pipettes, and rubber gloves, contaminated with biohazardous material (primarily urine) is accumulated in a plastic bag labeled for the collection of biohazardous waste and is kept segregated from waste with no biohazard contamination.

9.1.2.3. Full bags of biohazardous waste are transferred to a cardboard box labeled with a biohazard label. The cardboard box is lined with a plastic bag bearing a biohazard label.

9.1.2.4. When the cardboard box is full, the liner bag and the box are sealed.

9.1.2.5. A new biohazard waste box lined with a plastic bag is prepared for further use.

9.2. Liquid waste.

9.2.1. Liquid waste contaminated with radionuclides is disposed of in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. Radioactively contaminated waste.

9.3.1.1. The surface exposure rate of the sealed bag of radiochemically contaminated solid waste is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.1.2. Pickup by the Waste Management group of a full bag of radiochemically contaminated solid waste is requested using the current Chemical Waste Disposal Request (CWDR) form. The current Waste Profile Request form (WPRF) that describes the waste is referenced on both disposal request forms.

9.3.1.3. The Waste Management group picks up the waste for disposal according to Laboratory policy.

9.3.2. Biohazardous waste.

9.3.2.1. Pickup of a full box of biohazardous waste is requested through the Waste Management group using the current Chemical Waste Disposal Request (CWDR) form. The current Waste Profile Request form (WPRF) that describes the waste is referenced on the CWDR.

9.3.2.2. The Waste Management group arranges for pick up and disposal of the waste by the company currently under contract with the Laboratory for that purpose.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, and D. R. Perrin, "Quality Assurance for Health and Environmental Chemistry: 1984," Los Alamos National Laboratory report LA-10508-MS (1985).

10.2. M. A. Gautier, E. S. Gladney, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1985," Los Alamos National Laboratory report LA-10813-MS (1986).

10.3. M. A. Gautier, E. S. Gladney, W. D. Moss, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1986," Los Alamos National Laboratory report LA-11114-MS (1987).

10.4. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1987," Los Alamos National Laboratory report LA-11454-MS (1988).

10.5. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1988," Los Alamos National Laboratory report LA-11637-MS (1989).

10.6. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.7. *EML Procedures Manual*, 25th ed., H. L. Volchok and G. de Planque, Eds. (Environmental Measurements Laboratory, New York, August 1982).

10.8. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4323, Plutonium-238 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, November 1986).

10.9. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4904L-F, Alpha-Particle Standard" (National Institute of Standards and Technology, Washington, DC, April 1982).

10.10. M. Phillips, "Handling Human Biological Fluids," Health and Environmental Chemistry SOP-4, September 1988, Los Alamos National Laboratory document.

10.11. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.12. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.13. "Managing Infectious Waste," Administrative Requirement 10-7, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒). Where a section heading is marked, the entire section has been revised.

PLUTONIUM-239 IN URINE — QC SAMPLE PREPARATION

Analyte: Plutonium-239	**Method No.:** QCR200
Matrix: Urine	**Spike Range:** 0.0-0.46 pCi/sample
Procedure: Radiochemistry and alpha spectrometry	**Accuracy and Precision:** 100% ± 3.3% RSD
Effective Date: 01/01/75	**Authors:** Margaret A. Gautier William D. Moss Nancy L. Koski

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.11 to 10.13 for proper waste disposal.

1. Principle of Method

1.1. A pool of blank urine is prepared.

1.2. The blank urine is aliquoted and spiked with a known standard.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error from six sample plate preparations of the standard and on the error associated with use of the graduated cylinder and the pipette. A relative standard deviation (RSD) of 3.3% at the 1σ level has been calculated for the calibration, spiking, and aliquoting process based on the following:

$$D = \frac{M1 \times M2}{M3 + M2} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = aliquoting error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{\frac{\left[R^2_{M1}\,(M_3 + M_2)^2 + M_3^2\,(R^2_{M2} + R^2_{M3})\right]}{(M_3 + M_2)^2}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
M_3 = volume of cylinder,
M_2 = volume of pipette,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of cylinder, and
100 = factor to convert to percentage.

2.2. Analysis by radiochemistry and alpha spectrometry of QC samples prepared between 1984 and 1989 using this procedure gave a mean recovery of 101% ± 12% RSD. The data are published in Source Materials 10.1 to 10.6 and are summarized below.

PLUTONIUM-239 SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION AFTER CORRECTION FOR PLUTONIUM-242 RECOVERY

Year	Spike level pCi/sample	Mean ± RSD (%)	Number of samples
1984	0.06	96 ± 24	13
	0.15	99 ± 12	14
	0.23	99 ± 7	13
	0.46	96 ± 4	15
	1.47	89 ± 15	15

PLUTONIUM-239 SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION AFTER CORRECTION FOR PLUTONIUM-242 RECOVERY (cont)

Year	Spike level pCi/sample	Mean ± RSD (%)	Number of samples
1985	0.06	102 ± 20	13
	0.15	108 ± 11	11
	0.23	98 ± 13	8
	0.45	110 ± 10	6
	1.50	96 ± 7	10
1986	0.06	108 ± 15	15
	0.15	107 ± 14	14
	0.23	100 ± 11	14
	0.46	100 ± 11	14
1987	0.06	105 ± 13	15
	0.15	105 ± 8	14
	0.22	102 ± 10	13
	0.44	103 ± 11	11
1988	0.06	99 ± 22	16
	0.15	96 ± 6	13
	0.22	99 ± 12	15
	0.44	103 ± 8	12
1989	0.06	98 ± 26	15
	0.15	99 ± 12	13
	0.22	101 ± 11	17
	0.44	100 ± 8	17

3. Collection and Storage of Samples

3.1. Urine is collected as needed from group personnel who have no known contact with radiation work and is used to form a pool of blank urine.

3.2. Spiked QC samples are refrigerated at a nominal temperature of 4°C pending analysis.

4. Apparatus

4.1. Beakers: 4- or 5-L, polyethylene.

4.2. Graduated cylinders: 500-mL.

4.3. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.4. Pipette tips: disposable.

4.5. Specimen containers: 16-oz jars with screw caps. VWR Scientific Catalog No. 16193-810 or equivalent.

4.6. Labels printed with QC sample numbers.

4.7. Heat lamp.

4.8. Filter paper: 2.9-cm, Whatman No. 41 or equivalent.

4.9. Discs: stainless steel, 2.22-cm diam.

4.10. Alpha phosphor on Mylar: The phosphor is the standard silver-activated zinc sulfide. William B. Johnson & Associates, Inc., Research Park, Montville, NJ 07045.

4.11. Rings and discs: plastic rings and discs, nominal 1-in. diam, molded of nylon. Control Molding Corp., 84 Granite Ave., Staten Island, NY 10303.

4.12. Mylar film: 1.5-in.-wide rolls, 0.0005 in. thick. Manufactured by E. I. duPont de Nemours, retailed by Brownell, Inc., 85 Tenth Ave., New York, NY 10001.

5. Reagents

5.1. None required.

6. Calibration and Standards

6.1. QC samples are spiked with a ^{239}Pu standard prepared in 2 N HNO_3 and heated at a nominal temperature of 80°C for 2 h to convert Pu in the polymeric form to the ionic 4+ state. The solution is counted on a zinc sulfide scintillation system calibrated with National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 4904L-F, an ^{241}Am alpha-particle standard, using count room procedure CR340.

6.2. The ^{239}Pu standard should be at a concentration of approximately 1.5 pCi/mL.

6.3. The ^{239}Pu standard was calibrated against a NIST-certified ^{239}Pu alpha-particle solution standard, such as SRM 4331. Recalibrate the standard solution yearly by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.4. Direct plating procedure.

6.4.1. Pipette a 200- to 300-μL aliquot of the standard to be calibrated on each of six 2.22-cm stainless-steel discs, and evaporate to dryness under a heat lamp.

6.4.2. Place a 2.9-cm filter paper on a molded nylon disc. The filter paper assures a tightly sealed package when assembled.

6.4.3. Place the stainless-steel disc containing the sample on the filter paper, making sure it is centered.

6.4.4. Place a 0.875-in. phosphor paper over the stainless-steel disc (dull side against the disc).

6.4.5. Place a piece of Mylar film over all the layers.

6.4.6. Press a nylon ring down over the layers and around the nylon disc to form a tightly sealed package. Cut off the excess Mylar with scissors. Label the back of the assembly with an identification number.

6.4.7. Submit the samples to the count room for alpha scintillation counting and request a 60,000-s (overnight) per sample count.

6.4.8. The count room data are corrected for background and reported in disintegrations per minute (dis/min). Divide the dis/min results by 2.22 to convert to pCi. Calculate the mean and standard deviation of the six plates and assign the value to the standard.

7. Procedure

7.1. Working with bodily fluids is discussed in detail in SOP-4, "Handling Human Biological Fluids," found in Appendix III of the EM-9 Safety Manual.

NOTE: Wear safety glasses, gloves, and a lab coat. Each day prepare a 1:100 dilution of household bleach in water in a spray bottle. Use this solution to thoroughly clean the work area after sample preparation is completed. Remove gloves, discard in the biohazard waste container, and wash hands with soap and water.

7.2. Prepare a pool from blank urine sufficient in volume to allow 400 mL for each QC sample to be prepared.

7.3. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Include blank QCs prepared from the same urine pool as the spiked samples.

7.4. Record QC standard information, sample spike amount, concentration, and uncertainty in the QC Laboratory notebook.

7.5. Label bottles with QC sample numbers.

7.6. Aliquot 400 mL of blank urine into each sample bottle.

7.7. Spike the blank urine with aliquots of standard as determined in Step 7.3.

7.8. Screw caps on bottles and refrigerate at a nominal temperature of 4°C pending analysis.

7.9. Enter the spike concentrations of each QC into the CVS and CVD data bases on the VAX. This procedure is done annually because the same concentration levels are used throughout the year.

8. Calculations

8.1. Record spike concentrations as pCi/sample of urine.

8.2. Calculate the spike concentration for a 300 μL aliquot of ^{239}Pu standard using the following equation:

$$x = (1.52 \text{ pCi/mL}) \times 0.300 \text{ mL} = 0.46 \text{ pCi/sample} ,$$

where x = pCi/sample per spike amount.

8.3. Spike concentrations calculated in Step 8.2 are tabulated below.

Spike amount (μL)	Concentration (pCi/sample)	Uncertainty (± 3.3%)
40	0.06	0.002
100	0.15	0.005
150	0.23	0.008
300	0.46	0.015

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Radiochemically contaminated waste.

9.1.1.1. Radiochemically contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, is accumulated in a covered metal can lined with a plastic bag and kept segregated from nonradioactive solid waste.

9.1.1.2. The can is labeled with a hazardous waste label and a label indicating its use for radiochemically contaminated waste.

9.1.1.3. The can is opened only for the time necessary to add waste.

9.1.1.4. When the plastic bag is full, it is sealed with tape, removed from the can, identified as radiochemically contaminated waste, and placed in a functioning chemical exhaust hood pending pickup by the Waste Management group.

9.1.1.5. A new plastic bag is put into the waste can for continued use.

9.1.2. Biohazardous waste.

9.1.2.1. Bottles used to collect blank urine for a quality control sample matrix are disposed of as uncontaminated laboratory trash after they have been rinsed with water in a laboratory sink.

9.1.2.2. Solid waste, such as paper wipes, plastic pipettes, and rubber gloves, contaminated with biohazardous material (primarily urine) is accumulated in a plastic bag labeled for the collection of biohazardous waste and is kept segregated from waste with no biohazard contamination.

9.1.2.3. Full bags of biohazardous waste are transferred to a cardboard box labeled with a biohazard label. The cardboard box is lined with a plastic bag bearing a biohazard label.

9.1.2.4. When the cardboard box is full, the liner bag and the box are sealed.

9.1.2.5. A new biohazard waste box lined with a plastic bag is prepared for further use.

9.2. Liquid waste.

9.2.1. Liquid waste contaminated with radionuclides is disposed of in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. Radioactively contaminated waste.

9.3.1.1. The surface exposure rate of the sealed bag of radiochemically contaminated solid waste is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.1.2. Pickup by the Waste Management group of a full bag of radiochemically contaminated solid waste is requested using the current Chemical Waste Disposal Request (CWDR) form. The current Waste Profile Request form (WPRF) that describes the waste is referenced on both disposal request forms.

9.3.1.3. The Waste Management group picks up the waste for disposal according to Laboratory policy.

9.3.2. Biohazardous waste.

9.3.2.1. Pickup of a full box of biohazardous waste is requested through the Waste Management group using the current Chemical Waste Disposal Request (CWDR) form. The current Waste Profile Request form (WPRF) that describes the waste is referenced on the CWDR.

9.3.2.2. The Waste Management group arranges for pickup and disposal of the waste by the company currently under contract with the Laboratory for that purpose.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, and D. R. Perrin, "Quality Assurance for Health and Environmental Chemistry: 1984," Los Alamos National Laboratory report LA-10508-MS (1985).

10.2. M. A. Gautier, E. S. Gladney, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1985," Los Alamos National Laboratory report LA-10813-MS (1986).

10.3. M. A. Gautier, E. S. Gladney, W. D. Moss, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1986," Los Alamos National Laboratory report LA-11114-MS (1987).

10.4. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1987," Los Alamos National Laboratory report LA-11454-MS (1988).

10.5. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1988," Los Alamos National Laboratory report LA-11637-MS (1989).

10.6. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.7. *EML Procedures Manual*, 25th ed., H. L. Volchok and G. de Planque, Eds. (Environmental Measurements Laboratory, New York, August 1982).

10.8. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4331, Plutonium-239 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, March 1975).

10.9. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4904L-F, Alpha-Particle Standard" (National Institute of Standards and Technology, Washington, DC, April 1982).

10.10. M. Phillips, "Handling Human Biological Fluids," Health and Environmental Chemistry SOP-4, September 1988, Los Alamos National Laboratory document.

10.11. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.12. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.13. "Managing Infectious Waste," Administrative Requirement 10-7, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒). Where a section heading is marked, the entire section has been revised.

RADIONUCLIDES ON FILTER MEDIA — QC SAMPLE PREPARATION

Analyte: Plutonium-238, Plutonium-239, and Americium-241	**Method No.:** QCR210
Matrix: Filters	**Spike Range:** 0.45-2.25 pCi/filter
Procedure: Radiochemistry and alpha spectrometry	**Accuracy and Precision:** ^{238}Pu 100% ± 4.3% RSD ^{239}Pu 100% ± 1.9% RSD ^{241}Am 100% ± 1.9% RSD
Effective Date: 01/21/85	**Authors:** Margaret A. Gautier Nancy L. Koski

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.11 and 10.12 for proper waste disposal practices.

1. Principle of Method

1.1. Blank sand is placed on a Microsorban filter and is spiked with ^{238}Pu, ^{239}Pu, and ^{241}Am standards. The standards are spiked onto sand because the filters are hydrophobic.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error from six sample plate preparations of the standard and on the error associated with use of the pipette. A relative standard deviation (RSD) of 4.3% for ^{238}Pu, 1.9% for ^{239}Pu, and 1.9% for ^{241}Am at the 1σ level has been calculated for the calibration, spiking, and weighing process based on the following function:

$$D = M1 \times M2 \quad ,$$

where D = calculated result,
M1 = calibration error, and
M2 = spiking error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R^2_{M1} + R^2_{M2}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette, and
100 = factor to convert to percentage.

2.2. Analysis by radiochemistry and alpha spectrometry of QC samples prepared during 1989 and 1990 using this procedure gave a mean recovery of 90% ± 13% for ^{238}Pu (N = 9), 105% ± 14% for ^{239}Pu (N = 12), and 83% ± 3% for ^{241}Am (N = 12) at the 1σ level. The data are published in Source Materials 10.1 to 10.5.

3. Collection and Storage of Samples

3.1. Microsorban filters are obtained from the sample submitter.

3.2. QC samples are prepared in a platinum crucible with a platinum cover and are kept at room temperature pending analysis.

4. Apparatus

4.1. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.2. Pipette tips: disposable.

4.3. Balance: 100-g minimum capacity, 0.1-mg accuracy.

4.4. Spatula.

4.5. Platinum crucibles: approximately 50-mL capacity with platinum covers. Each crucible should have a unique identifying number stamped or etched into it.

4.6. Heat lamp.

4.7. Microsorban filters: 8-cm-diam polyethylene filter obtained from the sample submitter.

4.8. Labels printed with QC sample numbers.

4.9. Filter paper: 2.9-cm, Whatman No. 41 or equivalent.

4.10. Discs: stainless-steel, 2.22-cm-diam.

4.11. Alpha phosphor on Mylar: The phosphor is the standard silver-activated zinc sulfide, William B. Johnson & Associates, Inc., Research Park, Montville, NJ 07045.

4.12. Rings and discs: plastic rings and discs, nominal 1-in.-diam, molded of nylon. Control Molding Corp., 84 Granite Ave., Staten Island, NY 10303.

4.13. Mylar film: 1.5-in.-wide rolls, 0.0005 in. thick. Manufactured by E. I. duPont de Nemours, retailed by Brownell, Inc., 85 Tenth Ave., New York, NY 10001.

5. Reagents

5.1. Sand. A clean, dry 0.1-g aliquot is required for each sample.

5.2. A suitable sand does not contain the analyte of interest and presents few interferences to the analytical method.

NOTE: Surface soils have been contaminated with radionuclides as a result of the use and testing of nuclear weapons. A suitable blank soil for this procedure would have been collected before World War II or would be collected from a deep well or other excavation where radioactive contamination is not likely to have occurred. Before use, a representative aliquot of soil is analyzed to determine if it will provide a suitably blank matrix.

6. Calibration and Standards

6.1. Spike QC samples with ^{238}Pu and ^{239}Pu standards prepared in 2 N HNO_3 and with an ^{241}Am standard prepared in 4 N HNO_3. Heat the plutonium standards at a nominal temperature of 80°C for 2 h to convert Pu in the polymeric form to the ionic +4 state. Count all solutions on a zinc sulfide scintillation system calibrated with National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 4904L-F, an ^{241}Am alpha-particle standard.

6.2. The ^{239}Pu standard should be at a concentration of approximately 2 pCi/mL. The ^{238}Pu and ^{241}Am standards should be at a concentration of approximately 17 pCi/mL.

6.3. When they are available, dilute certified primary standards to make standards at the desired concentration.

6.4. Calibrate standard solutions that are not made from certified primary standards against an appropriate certified standard.

6.5. Calibrate the ^{238}Pu standard against an NIST-certified ^{238}Pu alpha-particle solution standard, such as NIST SRM 4323. Calibrate the standard solution annually by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.6. Calibrate the ^{239}Pu standard against an NIST-certified ^{239}Pu alpha-particle solution standard such as NIST SRM 4331. Calibrate the standard solution annually by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.7. Calibrate the ^{241}Am standard against an NIST-certified ^{241}Am alpha-particle solution standard such as NIST SRM 4322. Calibrate the standard solution annually by direct plating and alpha scintillation counting. Record all data in the QC Laboratory notebook.

6.8. Count all solutions on a zinc sulfide scintillation system calibrated with National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 4904L-F, an ^{241}Am alpha-particle standard, using count room procedure CR340.

6.9. Direct plating procedure.

6.9.1. Pipette a 200- to 300-μL aliquot of the standard to be calibrated on to each of six 2.22-cm stainless steel discs, and evaporate to dryness under a heat lamp.

6.9.2. Place a 2.9-cm filter paper on a molded nylon disc. The filter paper assures the package is tightly sealed when assembled.

6.9.3. Place the stainless-steel disc containing the sample on the filter paper, making sure it is centered.

6.9.4. Place a 0.875-in. phosphor paper over the stainless steel disc (dull side against the disc).

6.9.5. Place a piece of Mylar film over all the layers.

6.9.6. Press a nylon ring down over the layers and around the nylon disc to form a tightly sealed package. Cut off the excess Mylar with scissors. Label the back of the assembly with an identification number.

6.9.7. Submit the samples to the count room for alpha scintillation counting and request a 60;000-s (overnight) count for each sample.

6.9.8. The count room data is corrected for background and reported in disintegrations per minute (dpm). Divide the dpm results by 2.22 to convert to pCi. Calculate the mean and standard deviation of the six plates and assign the value to the standard.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically submit an unspiked filter as a blank.

7.2. Cut a Microsorban filter in half and fold it so that it fits into a 50-mL platinum crucible.

7.3. Place the crucible with the filter on the balance and tare the balance.

7.4. Weigh 0.1 g of clean, dry sand onto the filter. Because the filter is hydrophobic, the sand is used to absorb the Pu and Am standard solutions.

7.5. Spike aliquots of the Pu and Am standard solutions onto the sand to reach the desired spike range.

7.6. Dry the spiked sand and filter under a heat lamp.

7.7. A paper label should not be affixed to the platinum crucible because it will be destroyed during the ashing procedure.

7.8. Assign a QC sample number to the QC sample and note the unique identifying number that is etched or stamped onto the platinum crucible body. Make a list of the QC sample numbers and the corresponding crucible number.

7.9. When the sample is dry, cover the crucible with a platinum cover and keep it at room temperature pending analysis.

7.10. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the concentration of ^{238}Pu, ^{239}Pu, and ^{241}Am on the filter as pCi/filter.

8.2. Calculate the spike concentrations as in the following example.

8.2.1. The weight of the sand placed on top of the filter does not enter into the calculation.

8.2.2. If 80 μL of a 16.42-pCi/mL ^{238}Pu standard is spiked onto sand on the Microsorban filter, the ^{238}Pu concentration in the filter will be 1.3 pCi/filter. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(16.42\ \text{pCi/mL})\ (1\ \text{mL}/1000\ \mu\text{L})\ (80\ \mu\text{L})}{\text{filter}} = 1.3\ \text{pCi/filter} \quad .$$

8.3. Spike concentrations for ^{239}Pu and ^{241}Am are calculated in the same way as spike concentrations for ^{238}Pu, using the equation in Step 8.2.2.

9. Proper Waste Disposal Practices

9.1. Liquid waste.

9.1. Dispose of radioactively contaminated liquid waste in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.2. Solid waste.

9.2.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.2.2. Label the can to indicate its use for radioactively contaminated waste.

9.2.3. Open the can only for the time necessary to add waste.

9.2.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.2.5. Put a new plastic bag into the waste can for continued use.

9.2.6. Place the sealed bag into a cardboard box lined with a plastic bag and labeled to indicate its use for radioactively contaminated waste.

9.2.7. When the cardboard box is full, seal the plastic bag liner and the box.

9.2.8. Mark the box as radioactively contaminated compactable waste and reference the current Waste Profile Request form (WPRF) describing the waste in the box on the outside of the box.

9.2.9. Line a new box with a plastic bag and label it for continued use.

9.2.10. Complete the current Radioactive Solid Waste Record (RSWD) form describing the full, sealed box of waste. Reference the current WPRF number on the RSWD.

9.2.11. The outside of the full box is surveyed for radioactive contamination by a radiation protection technician. Record the results of the survey on the RSWD.

9.2.12. Submit the completed RSWD and a copy of the WPRF to the Waste Management group for approval.

9.3. Waste pickup.

9.3.1. After the RSWD describing the full box of solid waste has been approved by the Waste Management group, request pickup of the box by the contractor approved for the transportation of radioactively contaminated waste. A written job order may be required to request pickup of waste.

9.3.2. The RSWD and a copy of the associated WPRF accompany the full box.

9.3.3. The waste is disposed of according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1985," Los Alamos National Laboratory report LA-10813-MS (1986).

10.2. M. A. Gautier, E. S. Gladney, W. D. Moss, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1986," Los Alamos National Laboratory report LA-11114-MS (1987).

10.3. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1987," Los Alamos National Laboratory report LA-11454-MS (1988).

10.4. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1988," Los Alamos National Laboratory report LA-11637-MS (1989).

10.5. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.6. *EML Procedures Manual*, 25th ed., H.L. Volchok and G. de Planque, Eds. (Environmental Measurements Laboratory, New York, August 1982).

10.7. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4323, Plutonium-238 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, November 1986).

10.8. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4322, Americium-241 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, November 1986).

10.9. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4331, Plutonium-239 Alpha-Particle Solution Standard" (National Institute of Standards and Technology, Washington, DC, March 1975).

10.10. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4904L-F, Alpha-Particle Standard" (National Institute of Standards and Technology, Washington, DC, April 1982).

10.11. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.12. "Hazardous and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒▒). Where a section heading is marked, the entire section has been revised.

RADIUM-226 IN WATER — QC SAMPLE PREPARATION

Analyte: Radium-226	**Method No.:** QCR220
Matrix: Water	**Spike Range:** 0.5-5.0 pCi/L
Procedure: Proportional counting	**Accuracy and Precision:** 100% ± 2.1% RSD
Effective Date: 02/05/90	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.3 and 10.4 for proper waste disposal practices.

1. Principle of Method

1.1. A QC sample for the determination of ^{226}Ra in water is prepared by spiking 0.12 M HCl with a ^{226}Ra standard.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the pipette and of the volumetric flask. A relative standard deviation (RSD) of 2.1% at the 1σ level has been calculated for the calibration, spiking, and dilution process based on the following function:

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3}} \times 100 ,$$

where S_{D_T} = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of volumetric flask, and
100 = factor to convert to percentage.

2.2. Analysis by proportional counting of QC samples (N = 4) prepared during 1990 using this procedure gave a mean recovery of 81% ± 22% at the 1σ level. The data are published in Source Material 10.1.

3. Collection and Storage of Samples

3.1. QC samples are stored in volumetric flasks at room temperature pending analysis.

4. Apparatus

4.1. Volumetric flasks: 1-L.

4.2. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.3. Pipette tips: disposable.

4.4. Labels printed with QC sample numbers.

5. Reagents

5.1. Hydrochloric acid (concentrated, reagent-grade).

6. Calibration and Standards

6.1. QC samples are spiked with a ^{226}Ra standard in 0.1 M HNO_3 at a concentration of approximately 10 pCi/mL.

6.2. A certified ^{226}Ra standard, such as National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 4958 (gamma-ray standard), is diluted to produce a standard with a ^{226}Ra concentration of approximately 10 pCi/mL.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically include 1 L of 0.12 M HCl as a blank.

7.2. Add approximately 900 mL of distilled water to a 1-L volumetric flask.

7.3. Add 10 mL of 12 M hydrochloric acid to the water in the flask to produce 0.12 M acid.

7.4. Add an aliquot of ^{226}Ra standard to the water in the flask to reach the desired spike range.

7.5. Dilute the QC sample to volume with distilled water. Mix by inversion. Label the flask with a QC sample number and store at room temperature pending analysis.

7.6. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the concentration of ^{226}Ra in the QC sample as pCi/L.

8.2. Calculate spike concentrations as in the following example.

8.2.1. If 220 μL of a 10-pCi/mL ^{226}Ra standard is used to prepare a 1-L QC sample, the ^{226}Ra concentration in the sample will be 2.2 pCi/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(10\ \text{pCi/mL})\ (1\ \text{mL}/1000\ \mu\text{L})\ (220\ \mu\text{L})}{1\ \text{L}} = 2.2\ \text{pCi/L}\ .$$

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.1.2. Label the can to indicate its use for radioactively contaminated waste.

9.1.3. Open the can only for the time necessary to add waste.

9.1.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.1.5. Put a new plastic bag into the waste can for continued use.

9.1.6. Place the sealed bag into a cardboard box lined with a plastic bag and keep the box inside a metal compactable waste container. Reference to the current Waste Profile Request form (WPRF) describing the waste accompanies the sealed bag.

9.1.7. Label the metal container to indicate its use for radioactively contaminated waste.

9.1.8. When the cardboard box is full, seal the plastic bag liner and the box. Check the outside of the box for radioactive contamination and record the information in the Radioactive Waste Disposal logbook.

9.1.9. The radioactive solid waste coordinator marks the cardboard box as radioactively contaminated compactable waste and references the current WPRF numbers describing the waste in the box on the outside of the box and in the Radioactive Waste Disposal logbook.

9.1.10. Place a new lined cardboard box into the metal container for continued use.

9.1.11. Place the sealed box in a dumpster for compactable mixed waste.

9.2. Liquid waste.

9.2.1. Dispose of radioactively contaminated liquid waste in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. When the dumpster containing solid waste is full, the surface exposure rate of the dumpster is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.2. Reference all the WPRF numbers associated with the waste in the dumpster on the RSWD.

9.3.3. Request pickup by the Waste Management group of a full dumpster. The RSWD and all the associated WPRFs accompany the dumpster.

9.3.4. The Waste Management group disposes of the waste according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.2. National Institute of Standards and Technology, "Certificate of Analysis for Radium-226" (National Institute of Standards and Technology, Washington, DC, March 1968).

10.3. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.4. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒). Where a section heading is marked, the entire section has been revised.

STRONTIUM-90 ON SOIL — QC SAMPLE PREPARATION

Analyte: Strontium-90	**Method No.:** QCR230
Matrix: Sand	**Spike Range:** 5-45 pCi/g
Procedure: Proportional counting	**Accuracy and Precision:** 100% ± 3.0% RSD
Effective Date: 04/07/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.3 and 10.4 for proper waste disposal practices.

1. Principle of Method

1.1. Blank sand is spiked with a ^{90}Sr standard. The sand is used in place of soil as the QC matrix for ^{90}Sr analysis by proportional counting.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the pipette and of the balance. A relative standard deviation (RSD) of 3.0% at the 1σ level has been calculated for the calibration, spiking, and weighing process based on the following function:

$$D = \frac{M1 \times M2}{M3},$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = weighing error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3}} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of balance, and
100 = factor to convert to percentage.

2.2. Analysis by proportional counting of QC samples (N = 10) prepared during 1989 and 1990 using this procedure gave a mean recovery of 76% ± 9% at the 1σ level. The data are published in Source Material 10.1.

3. Collection and Storage of Samples

3.1. QC samples are stored in Teflon beakers covered with aluminum foil at room temperature pending analysis.

4. Apparatus

4.1. Beakers: 250-mL, Teflon.

4.2. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.3. Pipette tips: disposable.

4.4. Balance: 200-g capacity, 0.1-mg accuracy.

4.5. Spatula.

4.6. Aluminum foil.

4.7. Labels printed with QC sample numbers.

5. Reagents

5.1. Sand. A clean and dry 2-g aliquot is required for each sample.

5.2. A suitable sand does not contain the analyte of interest and presents few interferences to the analytical method.

NOTE: Surface soils have been contaminated with radionuclides as a result of the use and testing of nuclear weapons. A suitable blank soil for this procedure would have been collected before World War II or would be collected from a deep well or other excavation where radioactive contamination is not likely to have occurred. Before use, a representative aliquot of soil is analyzed to determine if it will provide a suitably blank matrix.

6. Calibration and Standards

6.1. When they are available, certified primary standards should be diluted to make a standard at the desired concentration.

6.2. Standard solutions which are not made from certified primary standards should be calibrated against an appropriate certified standard.

6.3. QC samples are spiked with a ^{90}Sr standard prepared in 2 M HCl at a concentration of approximately 870 pCi/mL.

6.4 The ^{90}Sr standard should be calibrated against an Environmental Protection Agency (EPA)-certified ^{90}Sr standard solution in equilibrium with its daughter ^{90}Y.

6.5. Decay the ^{90}Sr standards each year.

6.5.1. The half-life of ^{90}Sr is 28.5 years.

6.5.2. The following equation describes the rate of physical decay of radionuclides:

$$A_t = A_o e^{-\lambda t} ,$$

where A_t = the activity at desired time t,
A_o = the activity at t = 0,
e = the base of natural logarithms, and

λ = (ln 2)/$T_{1/2}$, the decay constant, where $T_{1/2}$ is the half-life.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically include a 2-g aliquot of the unspiked deep-well sand as a blank.

7.2. Accurately weigh a 2-g aliquot of clean, dry sand into a 250-mL Teflon beaker. Record the weight of the sand on the beaker with a felt-tipped pen for reference by the analyst.

7.3. Spike an aliquot of ^{90}Sr standard onto the sand to reach the desired spike range.

7.4. Because a paper label does not adhere well to a Teflon beaker, write the QC number assigned to the sample on the beaker with a pen and destroy the paper label associated with the sample number used. Cover the top of the beaker with aluminum foil and store it at room temperature pending analysis.

7.5. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the concentration of ^{90}Sr on the sand as pCi/g.

8.2. Calculate spike concentrations as in the following example.

8.2.1. If 15 μL of a 871-pCi/mL ^{90}Sr standard is used to spike 2.0000 g of sand, the ^{90}Sr concentration in the sand will be 6.5 pCi/g. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{\left(871\ \frac{\text{pCi}}{\text{mL}}\right)\left(\frac{1\ \text{mL}}{1000\ \mu\text{L}}\right)(15\ \mu\text{L})}{2.0000\ \text{g}} = 6.5\ \frac{\text{pCi}}{\text{g}}$$

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC

samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.1.2. Label the can to indicate its use for radioactively contaminated waste.

9.1.3. Open the can only for the time necessary to add waste.

9.1.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.1.5. Put a new plastic bag into the waste can for continued use.

9.1.6. Place the sealed bag into a cardboard box lined with a plastic bag and keep the box inside a metal compactable waste container. Reference to the current Waste Profile Request form (WPRF) describing the waste accompanies the sealed bag.

9.1.7. Label the metal container to indicate its use for radioactively contaminated waste.

9.1.8. When the cardboard box is full, seal the plastic bag liner and the box. Check the outside of the box for radioactive contamination and record the information in the Radioactive Waste Disposal logbook.

9.1.9. The radioactive solid waste coordinator marks the cardboard box as radioactively contaminated compactable waste and references the current WPRF numbers describing the waste in the box on the outside of the box and in the Radioactive Waste Disposal logbook.

9.1.10. Place a new lined cardboard box into the metal container for continued use.

9.1.11. Place the sealed box in a dumpster for compactable mixed waste.

9.2. Liquid waste.

9.2.1. Dispose of radioactively contaminated liquid waste in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. When the dumpster containing solid waste is full, the surface exposure rate of the dumpster is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.2. Reference all the WPRF numbers associated with the waste in the dumpster on the RSWD.

9.3.3. Request pickup by the Waste Management group of a full dumpster. The RSWD and all the associated WPRFs accompany the dumpster.

9.3.4. The Waste Management group disposes of the waste according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.2. US Environmental Protection Agency, "Calibration Certificate for Strontium-90" (Environmental Monitoring Systems Laboratory, Quality Assurance Division, Las Vegas, Nevada, June 1987).

10.3. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.4. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, in Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒). Where a section heading is marked, the entire section has been revised

STRONTIUM-90 IN WATER – QC SAMPLE PREPARATION

Analyte: Strontium-90	**Method No.:** QCR240
Matrix: Water	**Spike Range:** 200-400 pCi/L
Procedure: Proportional counting	**Accuracy and Precision:** 100% ± 3.2 % RSD
Effective Date: 11/28/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.3 and 10.4 for proper waste disposal.

1. Principle of Method

1.1. A QC sample for the determination of ^{90}Sr in water is prepared by spiking 0.08 M HCl with a ^{90}Sr standard.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the pipette and the volumetric flask. A relative standard deviation (RSD) of 3.2% at the 1σ level has been calculated for the calibration, spiking, and dilution process based on the following:

$$D = \frac{M1 \times M2}{M3} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R^2_{M1} + R^2_{M2} + R^2_{M3}} \times 100 ,$$

where S_{D_T} = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of volumetric flask, and
100 = factor to convert to percentage.

2.2. Analysis by proportional counting of QC samples (N = 4) prepared during 1990 using this procedure gave a mean recovery of 94% ± 10% at the 1σ level. The data are published in Source Material 10.1.

3. Collection and Storage of Samples

3.1. QC samples are stored in volumetric flasks at room temperature pending analysis.

4. Apparatus

4.1. Volumetric flasks: 500-mL, class A, glass.

4.2. Auto-pipette: 1000-μL, Rainin or equivalent.

4.3. Pipette tips: disposable.

4.4. Beakers: 600-mL, glass.

4.5. Plastic wrap.

4.6. Labels printed with QC sample numbers.

5. Reagents

5.1. Hydrochloric acid (concentrated, reagent-grade).

6. Calibration and Standards

6.1. When they are available, certified primary standards should be diluted to make a standard at the desired concentration.

6.2. Standard solutions that are not made from certified primary standards should be calibrated against an appropriate certified standard.

6.3. QC samples are spiked with a ^{90}Sr standard in 2 M HCl at a concentration of approximately 870 pCi/mL.

6.4. The ^{90}Sr standard should be calibrated against an Environmental Protection Agency (EPA)-certified ^{90}Sr standard solution in equilibrium with its daughter ^{90}Y.

6.5. Decay the ^{90}Sr standards each year.

6.5.1. The half-life of ^{90}Sr is 28.5 years.

6.5.2. The following equation describes the rate of radioactive decay of radionuclides:

$$A_t = A_o e^{-\lambda t} ,$$

where A_t = the activity at time t,
A_o = the activity at t = 0,
e = the base of natural logarithms,
λ = the decay constant = $(\ln 2)/T_{1/2}$, where $T_{1/2}$ is the half-life.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically include 500 mL of 0.08 M HCl as a blank.

7.2. Add approximately 400 mL of distilled water to a 500-mL volumetric flask.

10.4. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒▒). Where a section heading is marked, the entire section has been revised.

TRITIUM AND PERCENT MOISTURE ON SOIL — QC SAMPLE PREPARATION

Analyte: Tritium and percent moisture	**Method No.:** QCR250
Matrix: Sand	**Spike Range:** Tritium 80-400,000 pCi/L or 2-10,000 pCi/sample percent moisture 2-5% H_2O
Procedure: Liquid scintillation counting and gravimetry	**Accuracy and Precision:** Tritium 100% ± 2.6% RSD percent moisture 100% ± 9% RSD
Effective Date: 04/07/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.4 and 10.5 for proper waste disposal practices.

1. Principle of Method

1.1. Blank sand is aliquoted and spiked with a tritiated water standard.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. Propagated error for tritium.

2.1.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the pipette and of the volumetric flask. A relative standard deviation (RSD) of 2.6% at the 1σ level has been calculated for the calibration and spiking process based on the following function:

$$D = M1 \times M2 \quad ,$$

where D = calculated result,
M1 = calibration error, and
M2 = spiking error.

2.1.2. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.3. Calculate percent relative standard deviation using the following equation.

$$SD_T = \sqrt{R_{M1}^2 + R_{M2}^2} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette, and
100 = factor to convert to percentage.

2.2. Propagated error for percent moisture.

2.2.1. The propagated error on QC preparation is based on the error associated with use of the analytical balance. An RSD of 9.0% has been calculated for the weighing process based on the following function:

$$D = \frac{M3}{M1 + M2} ,$$

where D = calculated result,
M3 = weighing error of water,
M1 = weighing error of first soil aliquot, and
M2 = weighing error of second soil aliquot.

2.2.2. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.2.3. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \frac{\sqrt{(R^2_{M3} + R^2_{M2})M_2^2 + (R^2_{M3} + R^2_{M1})M_1^2 + 2R^2_{M3}\, M_1\, M_2}}{(M_1 + M_2)} \times 100 ,$$

where SD_T = relative standard deviation (% error),
R^2_{M3} = relative variance of weight of water,
R^2_{M2} = relative variance of weight of second soil aliquot,
M_2 = weight of second soil aliquot,
R^2_{M1} = relative variance of weight of first soil aliquot,
M_1 = weight of first soil aliquot, and
100 = factor to convert to percentage.

2.3. Analysis by liquid scintillation counting of QC samples (N = 23) prepared during 1989 and 1990 using this procedure gave a mean recovery of 93% ± 6% RSD for tritium. Analysis by gravimetry of QC samples (N = 9) for percent moisture gave a mean recovery of 99% ± 5% RSD. The data are published in Source Material 10.1 and are summarized below.

TRITIUM SPIKE RECOVERY DURING 1989
AS A FUNCTION OF CONCENTRATION

Spike range nCi/L	Mean ± SD (%)	Number of samples
1 to 50	95 ± 5	13
50 to 100	93 ± 4	5
100 to 200	84 ± 5	3

3. Collection and Storage of Samples

3.1. QC samples are covered with plastic wrap and frozen pending analysis.

4. Apparatus

4.1. Beakers: 600-mL and 1-L.

4.2. Pipettes: 10-, 15-, 20-, and 25-mL, class A volumetric.

4.3. Desiccator.

4.4. Drying oven: capable of reaching at least 110°C.

4.5. Spatula.

4.6. Plastic wrap.

4.7. Labels printed with QC sample numbers.

4.8. Balance: 1-kg minimum capacity, 0.1-g accuracy.

5. Reagents

5.1. Sand. A clean and dry 500-g aliquot is required for each sample.

5.2. A suitable sand does not contain the analyte of interest and presents few interferences to the analytical method.

6. Calibration and Standards

6.1. QC samples are spiked with tritium standards that contain ^{3}H in the concentration range 80 to 400,000 pCi/L.

6.2. A 25-mL aliquot of a ^{3}H standard in the concentration range 80 to 400,000 pCi/L spiked onto 500.0 g of clean, dry sand will produce a QC sample containing ^{3}H in the concentration range 2 to 10,000 pCi/sample and a total moisture content of approximately 5% by weight. The volume of water spiked onto the sand may vary between 10 and 25 mL but must be at least 10 mL.

6.3. Decay the ^{3}H standards each year.

6.3.1. The half-life of ^{3}H is 12.35 years.

6.3.2. The following equation describes the rate of physical decay of radionuclides:

$$A_t = A_0 e^{-\lambda t} ,$$

where A_t = the activity at desired time t,
A_o = the activity at t = 0,
e = the base of natural logarithms, and
λ = $(\ln 2)/T_{1/2}$ or the decay constant, where $T_{1/2}$ is the half-life.

6.4. The ^3H standard was calibrated against a National Institute of Standards and Technology (NIST)-certified ^3H Standard Reference Material (SRM), such as NIST SRM 4927B, a solution of tritiated water. Recalibrate the standard solution yearly by liquid scintillation counting.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically add an aliquot of distilled water to a soil sample and include it as a blank for ^3H analysis.

7.2. Dry approximately 600 g of soil in a 1-L beaker at 105-110°C overnight.

7.3. Cool the soil in a desiccator.

7.4. Place a 600-mL beaker on a balance with a capacity of at least 1000 g. Tare the balance.

7.5. Add 300 g of soil to the beaker. Record the weight and tare the balance.

7.6. Pipette 10 to 25 mL of tritium standard onto the center of the soil. Record the weight and tare the balance.

7.7. Add 200 g of soil to the beaker to cover the wet soil, to bring the total soil weight to 500 ± 1 g and to achieve 2 to 5% H_2O. Record the final soil weight on the beaker with a felt-tipped pen for reference by the analyst.

7.8. Label the beaker with a QC sample number.

7.9. Cover the beaker with plastic wrap and freeze pending analysis.

7.10. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the weight percent of extractable (unbound) water (H_2O^-) using the total sample weight (e.g., 500 g of soil plus 25 g of added H_2O).

8.2. Record the concentration of tritium as pCi/L. The tritium is measured as pCi/L and not as pCi/g because the determination measures the tritium concentration in the water in the sample and not the tritium concentration on the soil. Tritium concentration can be related to sample weight through the percent of H_2O^- in the sample.

8.3. Calculate the spike concentrations as in the following example.

8.3.1. If 25 mL of a 200,000-pCi/L tritium standard is used to spike 500.0 g of soil, the concentration of tritium in the sample is 200,000 pCi/L (5000 pCi/25 mL). Multiply the result by the RSD calculated in Step 2.1.1 to obtain the uncertainty.

$$(200{,}000 \text{ pCi/L})\ (1 \text{ L}/1000 \text{ mL})\ (25 \text{ mL}) = 5{,}000 \text{ pCi/sample} \quad .$$

The percent moisture is approximately 5% H_2O^-. Multiply the result by the RSD calculated in Step 2.2.1 to obtain the uncertainty.

$$\frac{(25 \text{ mL } H_2O)\ (1 \text{ g/mL}) \times 100}{500.0 \text{ g soil} + 25.0 \text{ g } H_2O} = 4.8\% \ H_2O \quad .$$

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.1.2. Label the can to indicate its use for radioactively contaminated waste.

9.1.3. Open the can only for the time necessary to add waste.

9.1.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.1.5. Put a new plastic bag into the waste can for continued use.

9.1.6. Place the sealed bag into a cardboard box lined with a plastic bag and keep the box inside a metal compactable waste container. Reference to the current Waste Profile Request form (WPRF) describing the waste accompanies the sealed bag.

9.1.7. Label the metal container to indicate its use for radioactively contaminated waste.

9.1.8. When the cardboard box is full, seal the plastic bag liner and the box. Check the outside of the box for radioactive contamination and record the information in the Radioactive Waste Disposal logbook.

9.1.9. The radioactive solid waste coordinator marks the cardboard box as radioactively contaminated compactable waste and references the current WPRF numbers describing the waste in the box on the outside of the box and in the Radioactive Waste Disposal logbook.

9.1.10. Place a new lined cardboard box into the metal container for continued use.

9.1.11. Place the sealed box in a dumpster for compactable mixed waste.

9.2. Liquid waste.

9.2.1. Dispose of radioactively contaminated liquid waste in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. When the dumpster containing solid waste is full, the surface exposure rate of the dumpster is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.2. Reference all the WPRF numbers associated with the waste in the dumpster on the RSWD.

9.3.3. Request pickup by the Waste Management group of a full dumpster. The RSWD and all the associated WPRFs accompany the dumpster.

9.3.4. The Waste Management group disposes of the waste according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory LA-11995-MS (1990).

10.2. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4927-B, Hydrogen-3" (National Institute of Standards and Technology, Washington, DC, January 1979).

10.3. M. P. Unterweger, B. M. Coursey, F. J. Schima, and W. B. Mann, "Preparation and Calibration of the 1978 National Bureau of Standards Tritiated-water Standards," *International Journal of Applied Radiation and Isotopes* **31**, 611-614 (1980).

10.4. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*," Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.5. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒▒). Where a section heading is marked, the entire section has been revised.

TRITIUM IN URINE – QC SAMPLE PREPARATION

Analyte: Tritium	**Method No.:** QCR260
Matrix: Urine	**Spike Range:** 0.0-10.0 μCi/L
Procedure: Liquid scintillation counting	**Accuracy and Precision:** 100% ± 3.7% RSD
Effective Date: 01/01/75	**Authors:** Margaret A. Gautier William D. Moss Nancy L. Koski

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.10 to 10.12 for proper waste disposal.

1. Principle of Method

1.1. A pool of blank urine is prepared.

1.2. The blank urine is aliquoted and spiked with a known standard.

1.3. QC samples are submitted for analysis with each routine analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error from six preparations of the standard and on the error associated with the use of the graduated cylinder and the pipette. A relative standard deviation (RSD) of 3.7% at the 1σ level has been calculated for the calibration, spiking, and aliquoting process based on the following:

$$D = \frac{M1 \times M2}{M3 + M2} ,$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = aliquoting error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where $R^2{}_M$ = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate the percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{\frac{\left[R_{M1}^2 (M_3 + M_2)^2 + M_3^2 (R_{M2}^2 + R_{M3}^2)\right]}{(M_3 + M_2)^2}} \times 100 ,$$

where S_{D_T} = relative standard deviation (% error),
$R^2{}_{M1}$ = relative variance of standard,
M_3 = volume of cylinder,
M_2 = volume of pipette,
$R^2{}_{M2}$ = relative variance of pipette,
$R^2{}_{M3}$ = relative variance of cylinder, and
100 = factor to convert to percentage.

2.2. Analysis by liquid scintillation counting of QC samples prepared between 1984 and 1989 using this procedure gave a mean recovery of 98% ± 6% RSD. The data are published in Source Materials 10.1 through 10.6 and are summarized below.

TRITIUM SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION

Year	Spike level (μCi/L)	Mean ± RSD (%)	Number of samples
1984	3.38	95 ± 9	15
	6.76	91 ± 3	12
	10.13	92 ± 3	10
	13.51	92 ± 4	7
	16.89	92 ± 2	5
1985	3.29	98 ± 4	22
	6.57	95 ± 3	19

TRITIUM SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION (cont)

Year	Spike level (μCi/L)	Mean ± RSD (%)	Number of samples
1985	9.86	96 ± 5	15
	13.14	96 ± 2	12
	16.29	98 ± 2	9
1986	3.31	94 ± 3	22
	7.42	93 ± 4	22
	9.27	94 ± 3	18
	12.98	95 ± 3	16
	18.54	94 ± 4	18
1987	3.62	98 ± 6	34
	5.43	98 ± 7	38
	7.24	98 ± 5	39
	10.87	98 ± 5	27
1988	0.13	84 ± 23	16
	0.52	103 ± 10	41
	0.87	105 ± 8	41
	5.07	111 ± 8	41
	10.14	111 ± 7	39
1989	0.47	99 ± 14	27
	0.78	104 ± 14	27
	4.65	105 ± 14	28
	9.30	107 ± 13	26
	34.24	105 ± 14	25

3. Collection and Storage of Samples

3.1. Urine is collected as needed from group personnel who have no known contact with radiation work and is used to form a pool of blank urine.

3.2. Spiked QC samples are refrigerated at a nominal temperature of 4°C pending analysis.

4. Apparatus

4.1. Beakers: 4- or 5-L, polyethylene.

4.2. Graduated cylinders: 50-mL.

4.3. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.4. Pipette tips: disposable.

4.5. Specimen containers: 120-mL with screw caps, sterile inner surface. Catalog no. C8845-3, Baxter Health Care Corp., Tempe, AZ 85282, or equivalent.

4.6. Labels printed with QC sample numbers.

5. Reagents

5.1. None required.

6. Calibration and Standards

6.1. QC samples are spiked with tritium standards. The standards should be at a concentration of approximately 40 and 750 μCi/L.

6.2. Decay the tritium standard in January and July.

6.2.1. The half-life of tritium is 12.43 years.

6.2.2. The following equation describes the rate of physical decay of radionuclides:

$$A_t = A_o e^{-\lambda t} ,$$

where A_t = the activity at time t,
A_o = the activity at t = 0,
e = the base of natural logarithms,
λ = the decay constant = $(\ln 2)/T_{1/2}$, where $T_{1/2}$ is the half-life.

6.3. The ^3H standard was calibrated against a National Institute of Standards and Technology (NIST)-certified ^3H solution of tritiated water, such as NIST Standard Reference Material (SRM) 4927B. Recalibrate the standard solution yearly by liquid scintillation counting. Record all data in the QC Laboratory notebook.

7. Procedure

7.1. Working with bodily fluids is discussed in detail in SOP-4, "Handling Human Biological Fluids," found in Appendix III of the EM-9 Safety Manual.

NOTE: Wear safety glasses, gloves, and a lab coat. Each day prepare a 1:100 dilution of household bleach in water in a spray bottle. Use this solution to thoroughly clean the work area after sample preparation is completed. Remove gloves, discard in the biohazard waste container, and wash hands with soap and water.

7.2. Prepare a pool of blank urine sufficient in volume to allow 50 mL for each QC sample to be prepared.

7.3. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Include blank QCs prepared from the same urine pool as the spiked samples. Tritium QC samples may be divided into two aliquots because only a 200-μL sample size is required by the analyst.

7.4. Record QC standard information, sample spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook.

7.5. Label bottles with QC sample numbers.

7.6. Aliquot 50 mL of blank urine into each sample bottle.

7.7. Spike the blank urine with aliquots of standard as determined in Step 7.3.

7.8. Screw caps on bottles and refrigerate at a nominal temperature of 4°C pending analysis.

7.9. Enter the spike concentration of each QC into the CVS and CVD data bases on the VAX. This procedure is done annually because the same concentration levels are used throughout the year.

8. Calculations

8.1. Calculate spike concentrations for a 150-μL aliquot of ^{3}H standard and report concentration as μCi/L using the following equation.

$$\frac{(1.67\ \mu\text{Ci/mL})\ (\text{mL}/1000\ \mu\text{L})\ (150\ \mu\text{L})}{50\ \text{mL}\ (1\ \text{L}/1000\ \text{mL})} = 5.01\ \mu\text{Ci/L}\ .$$

8.2. Spike concentrations calculated in Step 8.1 are tabulated below.

Spike amount (μL)	Concentration (pCi/sample)	Uncertainty (±3.7%)
0	0.00	0.00
100	3.34	0.12
150	5.01	0.17
200	6.68	0.25
250	8.35	0.31
300	10.02	0.37
400	13.36	0.49

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Radiochemically contaminated waste.

9.1.1.1. Radiochemically contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, is accumulated in a covered metal can lined with a plastic bag and kept segregated from nonradioactive solid waste.

9.1.1.2. The can is labeled with a hazardous waste label and a label indicating its use for radiochemically contaminated waste.

9.1.1.3. The can is opened only for the time necessary to add waste.

9.1.1.4. When the plastic bag is full, it is sealed with tape, removed from the can, identified as radiochemically contaminated waste, and placed in a functioning chemical exhaust hood pending pickup by the Waste Management group.

9.1.1.5. A new plastic bag is put into the waste can for continued use.

9.1.2. Biohazardous waste.

9.1.2.1. Bottles used to collect blank urine for a quality control sample matrix are disposed of as uncontaminated laboratory trash after they have been rinsed with water in a laboratory sink.

9.1.2.2. Solid waste, such as paper wipes, plastic pipettes, and rubber gloves, contaminated with biohazardous material (primarily urine) is accumulated in a plastic bag labeled for the collection of biohazardous waste and is kept segregated from waste with no biohazard contamination.

9.1.2.3. Full bags of biohazardous waste are transferred to a cardboard box labeled with a biohazard label. The cardboard box is lined with a plastic bag bearing a biohazard label.

9.1.2.4. When the cardboard box is full, the liner bag and the box are sealed.

9.1.2.5. A new biohazard waste box lined with a plastic bag is prepared for further use.

9.2. Liquid waste.

9.2.1. Liquid waste contaminated with radionuclides is disposed of in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. Radioactively contaminated waste.

9.3.1.1. The surface exposure rate of the sealed bag of radiochemically contaminated solid waste is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.1.2. Pickup by the Waste Management group of a full bag of radiochemically contaminated solid waste is requested using the current Chemical Waste Disposal Request (CWDR) form. The current Waste Profile Request form (WPRF) that describes the waste is referenced on both disposal request forms.

9.3.1.3. The Waste Management group picks up the waste for disposal according to Laboratory policy.

9.3.2. Biohazardous waste.

9.3.2.1. Pickup of a full box of biohazardous waste is requested through the Waste Management Group using the current Chemical Waste Disposal Request (CWDR) form. The current Waste Profile Request form (WPRF) that describes the waste is referenced on the CWDR.

9.3.2.2. The Waste Management group arranges for pickup and disposal of the waste by the company currently under contract with the Laboratory for that purpose.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, and D. R. Perrin, "Quality Assurance for Health and Environmental Chemistry: 1984," Los Alamos National Laboratory report LA-10508-MS (1985).

10.2. M. A. Gautier, E. S. Gladney, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1985," Los Alamos National Laboratory report LA-10813-MS (1986).

10.3. M. A. Gautier, E. S. Gladney, W. D. Moss, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1986," Los Alamos National Laboratory report LA-11114-MS (1987).

10.4. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1987," Los Alamos National Laboratory report LA-11454-MS (1988).

10.5. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1988," Los Alamos National Laboratory report LA-11637-MS (1989).

10.6. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

10.7. M. P. Unterweger, B. M. Coursey, F. J. Schima, and W. B. Mann, "Preparation and Calibration of the 1978 National Bureau of Standards Tritiated-water Standards," *International Journal of Applied Radiation and Isotopes* **31**, 611-614 (1980).

10.8. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4927-B, Hydrogen-3" (National Institute of Standards and Technology, Washington, DC, January 1979).

10.9. M. Phillips, "Handling Human Biological Fluids," Health and Environmental Chemistry SOP-4, September 1988, Los Alamos National Laboratory document.

10.10. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.11. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.12. "Managing Infectious Waste," Administrative Requirement 10-7, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒). Where a section heading is marked, the entire section has been revised.

TRITIUM IN WATER — QC SAMPLE PREPARATION

Analyte: Tritium	**Method No.:** QCR270
Matrix: Water	**Spike Range:** 10-20 nCi/L
Procedure: Liquid scintillation counting	**Accuracy and Precision:** 100% ± 3.7% RSD
Effective Date: 03/29/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.5 and 10.6 for proper waste disposal practices.

1. Principle of Method

1.1. A QC sample for the determination of tritium in water is prepared by diluting a tritium standard to 10 mL with distilled water.

1.2. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standard and on the error associated with use of the pipette and of the volumetric flask. A relative standard deviation (RSD) of 3.7% at the 1σ level has been calculated for the calibration, spiking, and dilution process based on the following function:

$$D = \frac{M1 \times M2}{M3},$$

where D = calculated result,
M1 = calibration error,
M2 = spiking error, and
M3 = dilution error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{R_{M1}^2 + R_{M2}^2 + R_{M3}^2} \times 100 ,$$

where S_{D_T} = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of volumetric flask, and
100 = factor to convert to percentage.

2.2. Analysis by liquid scintillation counting of QC samples prepared during 1989 and 1990 using this procedure (N = 35) gave a mean recovery of 92% ± 7% RSD. The data are published in Source Materials 10.1 and 10.2.

3. Collection and Storage of Samples

3.1. QC samples are transferred to glass scintillation vials and are stored at room temperature pending analysis.

4. Apparatus

4.1. Volumetric flasks: 10-mL, Class A.

4.2. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.3. Pipette tips: disposable.

4.4. Graduated cylinder: 10-mL.

4.5. Scintillation vials: glass.

4.6. Parafilm.

4.7. Labels printed with QC sample numbers.

5. Reagents

5.1. None required.

6. Calibration and Standards

6.1. When a certified primary standard is available, dilute it to make a standard at the desired concentration.

6.2. Calibrate standard solutions that are not made from certified primary standards against an appropriate certified standard.

6.3. Prepare QC samples by diluting a tritium standard to 10 mL with distilled water. The standard should be at a concentration of approximately 300 pCi/mL.

6.4. Calibrate the ^3H standard against a National Institute of Standards and Technology (NIST)-certified ^3H Standard Reference Material, such as NIST SRM 4927B, a solution of tritiated water. Recalibrate the standard solution annually by liquid scintillation counting and decay the ^3H concentration midway through the calibration period.

6.5. Decay the ^3H standards each year.

6.5.1. The half-life of ^3H is 12.35 years.

6.5.2. The following equation describes the rate of physical decay of radionuclides:

$$A_t = A_o e^{-\lambda t} ,$$

where A_t = the activity at time t,
A_o = the activity at t = 0,
e = the base of natural logarithms, and
λ = (ln 2)/$T_{1/2}$, the decay constant, where $T_{1/2}$ is the half-life.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically include 10 mL of distilled water as a blank.

7.2. Preparation of QC blanks.

7.2.1. Prepare and seal QC blank samples before any tritium solutions are opened to avoid contamination of the blanks by tritiated water vapor.

7.2.2. Using a graduated cylinder, measure 10 mL of distilled water into a glass scintillation vial.

7.2.3. Cap the vial and wrap the cap with Parafilm.

7.2.4. Label the vial with a QC sample number.

7.2.5. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

7.3. Preparation of QC spiked samples.

7.3.1. Pour approximately 3 mL of distilled water into a 10-mL volumetric flask.

7.3.2. Pipette an aliquot of the tritium standard solution into the flask to reach the desired spike range.

7.3.3. Dilute the standard to volume with distilled water. Mix by inverting.

7.3.4. Transfer the standard from the flask to a glass scintillation vial.

7.3.5. Cap the vial and wrap the cap with Parafilm.

7.3.6. Label the vial with a QC sample number.

7.3.7. Record QC standard information, sample number, sample description, spike amount, concentration, uncertainty, and all calculations in the QC Laboratory notebook and in the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the concentration of ^{3}H in the water as pCi/L or nCi/L.

8.2. Calculate spike concentrations as in the following example.

8.2.1. If 400 μL of a 300 pCi/L tritium standard is used to spike a 10-mL QC sample, the concentration of tritium in the sample is 12 nCi/L. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{\left(300\ \frac{pCi}{mL}\right)\left(\frac{mL}{1000\ \mu L}\right)(400\ \mu L)\left(\frac{nCi}{1000\ pCi}\right)}{(10.0\ mL)\left(\frac{L}{1000\ mL}\right)} = 12\frac{nCi}{L}$$

9. Proper Waste Disposal Practices

9.1. Liquid waste.

9.1. Dispose of radioactively contaminated liquid waste in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.2. Solid waste.

9.2.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.2.2. Label the can to indicate its use for radioactively contaminated waste.

9.2.3. Open the can only for the time necessary to add waste.

9.2.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.2.5. Put a new plastic bag into the waste can for continued use.

9.2.6. Place the sealed bag into a cardboard box lined with a plastic bag and labelled to indicate its use for radioactively contaminated waste.

9.2.7. When the cardboard box is full, seal the plastic bag liner and the box.

9.2.8. Mark the box as radioactively contaminated compactable waste and reference the current Waste Profile Request form (WPRF) describing the waste in the box on the outside of the box.

9.2.9. Line a new box with a plastic bag and label it for continued use.

9.2.10. Complete the current Radioactive Solid Waste Record (RSWD) form describing the full, sealed box of waste. Reference the current WPRF number on the RSWD.

9.2.11. The outside of the full box is surveyed for radioactive contamination by a radiation protection technician. Record the results of the survey on the RSWD.

9.2.12. Submit the completed RSWD and a copy of the WPRF to the Waste Management group for approval.

9.3. Waste pickup.

9.3.1. After the RSWD describing the full box of solid waste has been approved by the Waste Management group, request pickup of the box by the contractor approved for the transportation of radioactively contaminated waste. A written job order may be required to request pickup of waste.

9.3.2. The RSWD and a copy of the associated WPRF accompany the full box.

9.3.3. The waste is disposed of according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11996-MS (1990).

10.2. M. A. Gautier, E. S. Gladney, N.L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

10.3. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4927-B, Hydrogen-3" (National Institute of Standards and Technology, Washington, DC, January 1979).

10.4. M. P. Unterweger, B. M. Coursey, F. J. Schima, and W. B. Mann, "Preparation and Calibration of the 1978 National Bureau of Standards Tritiated-water Standards," *International Journal of Applied Radiation and Isotopes* **31**, 611-614 (1980).

10.5 "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.6. "Hazardous and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (▒▒▒▒). Where a section heading is marked, the entire section has been revised.

URANIUM IN SOIL — QC SAMPLE PREPARATION

Analyte: Uranium	**Method No.:** QCR280
Matrix: Soil	**Spike Range:** 10-500 μg/g
Procedure: Delayed neutron activation	**Accuracy and Precision:** 100% ± 3.3% RSD
Effective Date: 08/09/89	**Authors:** Nancy L. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the standards listed in Sec. 6. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 9 of this procedure and Source Materials 10.5 and 10.6 for proper waste disposal practices.

1. Principle of Method

1.1. Two soils with certified uranium contents are combined in varying proportions to make QC samples with uranium concentrations in the desired spike range.

1.2. No soils tested had a sufficiently low and consistent uranium background to allow use as a blank sample or as a soil sample matrix.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error of the standards and on the error associated with use of the analytical balance. A relative standard deviation (RSD) of 3.3% at the 1σ level has been calculated for the calibration and spiking process based on the following function:

$$D = \frac{M1\ M3 + M2\ M4}{M3 + M4},$$

where D = calculated result,
M1 = calibration error of standard one,
M2 = calibration error of standard two,
M3 = weighing error of standard one, and
M4 = weighing error of standard two.

2.2. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where $R^2{}_M$ = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.3. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{A + B} \times 100 ,$$

where

$$A = \frac{(R_{M3}^2 + R_{M4}^2)\,(M_2 - M_1)^2\, M_3^2\, M_4^2}{(M_3 + M_4)^2\,(M_2\, M_4 + M_1\, M_3)^2} ,$$

$$B = \frac{R_{M2}^2\, M_2^2\, M_4^2 + R_{M1}^2\, M_1^2\, M_3^2}{(M_2\, M_4 + M_1\, M_3)^2} ,$$

SD_T = relative standard deviation (% error),
$R^2{}_{M3}$ = relative variance of standard weight one,
$R^2{}_{M4}$ = relative variance of standard weight two,
M_2 = calibration error of standard two,
M_1 = calibration error of standard one,
M_3 = weighing error of standard one,
M_4 = weighing error of standard two,
$R^2{}_{M2}$ = relative variance of standard two,
$R^2{}_{M1}$ = relative variance of standard one, and
100 = factor to convert to percentage.

2.4. Analysis by delayed neutron activation of QC samples prepared during 1989 and 1990 using this procedure gave a mean recovery of 94% ± 2% RSD. The data are published in Source Materials 10.1 and 10.2 and are summarized below.

URANIUM SPIKE RECOVERY DURING 1989 AND 1990 AS A FUNCTION OF CONCENTRATION		
Spike range, $\mu g/g$	Mean ± RSD (%)	Number of samples
10 to 50	95 ± 4	4
50 to 100	95 ± 1	4
150 to 200	94 ± 1	3
200 to 250	94 ± 1	5

3. Collection and Storage of Samples

3.1. QC samples are stored in polyethylene reactor rabbits at room temperature pending analysis.

4. Apparatus

4.1. Reactor rabbits: polyethylene, to contain soil samples during irradiation in the reactor.

4.2. Commercial canned dust-chaser spray.

4.3. Spatulas.

4.4. Analytical balance: 100-g capacity, 0.1-mg accuracy.

4.5. Labels printed with QC sample numbers.

5. Reagents

5.1. None required.

6. Calibration and Standards

6.1. The total weight of soil in each reactor rabbit is 1.0000 g.

6.2. Select two soils with consensus or certified uranium concentrations, one near the lower limit and the other near the upper limit of the desired concentration range. Two such soils are Canadian Certified Reference Materials Project (CCRMP) SO-3, Calcareous C Uranium Soil, with a certified uranium concentration of 1.11 ± 0.02 $\mu g/g$, and CCRMP SY-2, Syenite, with a certified uranium concentration of 284 ± 9 $\mu g/g$.

6.3. One certified soil may be used alone or combined with a second certified soil to make a QC sample with a uranium concentration in the desired spike range.

7. Procedure

7.1. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Periodically include an unspiked sample as a blank.

7.2. Place a polyethylene rabbit on the balance. Tare the balance.

7.3. To prepare a QC sample with the same uranium concentration as the certified soil, accurately weigh 1.0000 g of the certified soil into the reactor rabbit. Clean the rabbit threads before recording the soil weight.

NOTE: The threads on the polyethylene reactor rabbit must be free of any soil residue before closing to prevent the rabbit from opening during irradiation. Use a commercial canned dust-chaser spray to blow the rabbit threads free of any soil before determining the final weight of soil added and capping the rabbit.

7.4. To prepare a QC sample with a uranium concentration different from the concentration in either of the two certified soils, determine a suitable ratio in which to mix the two certified soils to obtain a uranium concentration in the desired spike range and a total sample weight of 1.0000 g.

NOTE: When mixing two soils, weigh out at least 30 mg (0.03 g) of each soil to remain within the calculated RSD in Step 2.1.

7.4.1. Weigh an aliquot of the first soil into the reactor rabbit. Clean the rabbit threads. Record the soil weight and tare the balance.

7.4.2. Weigh an aliquot of the second soil into the reactor rabbit. Clean the rabbit threads before recording the soil weight.

7.5. Cap the reactor rabbit, making certain no residual soil remains on the threads.

7.7. Mix the two soils in the QC sample by shaking the rabbit.

7.8. Do not place a sample label on the rabbit because it will interfere with the free movement of the rabbit in and out of the reactor. Use a pen to write a QC number on the reactor rabbit. Destroy the associated printed sample label. Record the total sample weight on the rabbit for reference by the analyst.

7.9. Record QC standard information, sample number, sample description, certified soil weights, uranium concentrations, uncertainty, and all calculations in the QC Laboratory notebook and enter into the CVS and CVD data bases on the VAX.

8. Calculations

8.1. Record the final concentration of uranium in the soil as $\mu g/g$.

8.2. Calculate final uranium concentration as in the following example.

8.2.1. If 0.7951 g of CCRMP SO-3, Calcareous C Uranium Soil, with a uranium concentration of 1.11 ± 0.02 $\mu g/g$, and 0.2646 g of CCRMP SY-2, Syenite, with a uranium concentration of 284 ± 9 $\mu g/g$, are combined in a reactor rabbit, the QC sample will have a final weight of 1.0597 g and a uranium concentration of 72 $\mu g/g$. Multiply the result by the RSD calculated in Step 2.1 to obtain the uncertainty.

$$\frac{(0.7951\ g)\ (1.11\ \mu g/g)\ +\ (0.2646\ g)\ (284\ \mu g/g)}{0.7951\ g\ +\ 0.2646\ g} = 72\ \mu g/g\ .$$

9. Proper Waste Disposal Practices

9.1. Solid waste.

9.1.1. Accumulate radioactively contaminated solid waste, such as paper wipes, glass or plastic pipettes, plastic cups, and incorrectly spiked QC samples, in a covered metal can lined with a plastic bag and keep it segregated from nonradioactive solid waste.

9.1.2. Label the can to indicate its use for radioactively contaminated waste.

9.1.3. Open the can only for the time necessary to add waste.

9.1.4. When the plastic bag is full, seal it with tape, remove it from the can, and identify it as radioactively contaminated waste.

9.1.5. Put a new plastic bag into the waste can for continued use.

9.1.6. Place the sealed bag into a cardboard box lined with a plastic bag and keep the box inside a metal compactable waste container. Reference to the current Waste Profile Request form (WPRF) describing the waste accompanies the sealed bag.

9.1.7. Label the metal container to indicate its use for radioactively contaminated waste.

9.1.8. When the cardboard box is full, seal the plastic bag liner and the box. Check the outside of the box for radioactive contamination and record the information in the Radioactive Waste Disposal logbook.

9.1.9. The radioactive solid waste coordinator marks the cardboard box as radioactively contaminated compactable waste and references the current WPRF numbers describing the waste in the box on the outside of the box and in the Radioactive Waste Disposal logbook.

9.1.10. Place a new lined cardboard box into the metal container for continued use.

9.1.11. Place the sealed box in a dumpster for compactable mixed waste.

9.2. Liquid waste.

9.2.1. Dispose of radioactively contaminated liquid waste in a sink specifically approved for the disposal of aqueous radioactive waste and inorganic acids and bases.

9.3. Waste pickup.

9.3.1. When the dumpster containing solid waste is full, the surface exposure rate of the dumpster is determined by a radiation protection technician and is recorded on the Radioactive Solid Waste Disposal (RSWD) Record form.

9.3.2. Reference all the WPRF numbers associated with the waste in the dumpster on the RSWD.

9.3.3. Request pickup by the Waste Management group of a full dumpster. The RSWD and all the associated WPRFs accompany the dumpster.

9.3.4. The Waste Management group disposes of the waste according to Laboratory policy.

10. Source Materials

10.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 7721989," Los Alamos National Laboratory LA-11995-MS (1990).

10.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory LA-12208-MS (1991).

10.3. Canada Centre for Mineral and Energy Technology, "Certificate of Analysis for Reference Soil Sample SO-3" (Canada Centre for Mineral and Energy Technology, Ottowa, Ontario, Canada, 1985).

10.4. Canada Centre for Mineral and Energy Technology, "Certificate of Analysis for Reference Soil Sample SY-2" (Canada Centre for Mineral and Energy Technology, Ottowa, Ontario, Canada, December 1979).

10.5. "Low-Level Radioactive Solid Waste," Administrative Requirement 10-2, in *Environment, Safety and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

10.6. "Chemical, Hazardous, and Mixed Waste," Administrative Requirement 10-3, in *Environment, Safety, and Health Manual*, Los Alamos National Laboratory Manual, Chapter 1 (most recent edition).

Revisions or additions to the procedure are marked (). Where a section heading is marked, the entire section has been revised.

URANIUM-235 SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION (cont)

	Spike Level pCi/L	Mean ± RSD (%)	Number of Samples
1985	6.8	98 ± 10	14
	10.2	100 ± 7	10
	17.0	103 ± 3	3
	20.4	103 ± 4	9
	25.5	106 ± 9	5
1986	7.74	101 ± 12	9
	10.32	97 ± 7	14
	12.90	103 ± 9	9
	15.48	97 ± 6	9
	20.64	103 ± 7	11
	25.8	99 ± 7	12
1987	4.58	108 ± 9	13
	9.16	109 ± 7	11
	13.74	116 ± 8	11
	18.32	112 ± 7	9
	22.90	109 ± 11	13
1988	5.14	84 ± 8	7
	7.71	87 ± 10	10
	10.28	90 ± 10	12
	20.56	93 ± 10	14
	25.70	89 ± 4	11
1989	5.14	95 ± 14	15
	10.28	94 ± 15	6
	15.42	98 ± 14	18
	20.56	95 ± 9	13
	25.70	99 ± 20	11

3. Collection and Storage of Samples

3.1. Urine is collected as needed from group personnel who have no known contact with radiation work and is used to form a pool of blank urine.

3.2. Spiked QC samples are refrigerated at a nominal temperature of 4°C pending analysis.

4. Apparatus

4.1. Beakers: 4- or 5-L, polyethylene.

4.2. Graduated cylinders: 50-mL.

4.3. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.4. Pipette tips: disposable.

4.5. Specimen containers: 120-mL with screw caps, sterile inner surface, catalog no. C8845-3, Baxter Health Care Corp., Tempe, AZ 85282, or equivalent.

4.6. Labels printed with QC sample numbers.

4.7. Heat lamp.

4.8. Filter paper: 2.9-cm, Whatman No. 41 or equivalent.

4.9. Discs: stainless steel, 2.22-cm-diam.

4.10. Alpha phosphor on Mylar: The phosphor is the standard silver-activated zinc sulfide. William B. Johnson & Associates, Inc., Research Park, Montville, NJ 07045.

4.11. Rings and discs: plastic rings and discs, nominal 1-in.-diam, molded of nylon. Control Molding Corp., 84 Granite Ave., Staten Island, NY 10303.

4.12. Mylar film: 1.5-in.-wide rolls, 0.0005-in.-thick. Manufactured by E. I. duPont de Nemours, retailed by Brownell, Inc., 85 Tenth Ave., New York, NY 10001.

5. Reagents

5.1. None required.

6. Calibration and Standards

6.1. QC samples are spiked with an enriched uranium standard prepared in 2 N HNO_3. The standard should be at a concentration of approximately 2 pCi/mL. The solution is counted on a zinc sulfide scintillation system calibrated with National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 4904L-F, an ^{241}Am alpha-particle standard, using count room procedure CR340.

6.2. The enriched uranium stock standard (93.276% ^{235}U by weight) was prepared from a certified NIST standard, such as NIST SRM U-930. SRM U-930 is composed of the following additional isotopes by weight percent: ^{234}U, 1.0759; ^{236}U, 0.2034; and ^{238}U, 5.445. The ^{235}U to ^{238}U ratio for this standard is 17.349.

6.3. Recalibrate the ^{235}U standard solution yearly by direct plating and alpha scintillation counting.

6.4. Direct plating procedure.

6.4.1. Pipette a 500-μL aliquot of the standard to be calibrated on each of six 2.22-cm stainless steel discs, and evaporate to dryness under a heat lamp.

6.4.2. Place a 2.9-cm filter paper on a molded nylon disc. The filter paper assures a tightly sealed package when assembled.

6.4.3. Place the stainless steel disc containing the sample on the filter paper. Be sure they are centered.

6.4.4. Place a .875-in. phosphor paper over the stainless steel disc (dull side against the disc).

6.4.5. Place a piece of Mylar film over all the layers.

6.4.6. Press a nylon ring down over the layers and around the nylon disc to form a tightly sealed package. Cut off the excess Mylar with scissors. Label the back of the assembly with an identification number.

6.4.7. Submit the samples to the count room for alpha scintillation counting and request a 60,000-s (overnight) per sample count.

6.4.8. The count room data are corrected for background and reported in disintegrations per minute (dpm). Divide the dpm results by 2.22 to convert to pCi. Calculate the mean and standard deviation of the six plates and assign the value to the standard.

7. Procedure

7.1. Working with bodily fluids is discussed in detail in SOP-4, "Handling Human Biological Fluids," found in Appendix III of the EM-9 Safety Manual.

NOTE: Wear safety glasses, gloves, and a lab coat. Each day prepare a 1:100 dilution of household bleach in water in a spray bottle. Use this solution to thoroughly clean the work area after sample preparation is completed. Remove gloves, discard in the biohazard waste container, and wash hands with soap and water.

7.2. Prepare a pool from blank urine sufficient in volume to allow 50 mL for each QC sample to be prepared.

7.3. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Include blank QCs prepared from the same urine pool as the spiked samples.

7.4. Record QC standard information, sample spike amount, concentration, and uncertainty in the QC Laboratory notebook.

7.5. Label bottles with QC sample numbers.

7.6. Aliquot 50 mL of blank urine into each sample bottle.

7.7. Spike the blank urine with aliquots of standard as determined in Step 7.3.

7.8. Screw caps on bottles and refrigerate at a nominal temperature of 4°C pending analysis.

7.9. Enter the spike concentrations of each QC into the CVS and CVD data bases on the VAX. This procedure is done annually because the same concentration levels are used throughout the year.

8. Calculations

8.1. Record spike concentrations as pCi/L of urine. Calculate the spike concentration for a 150-μL aliquot of a 2.58-pCi/mL ^{235}U standard using the following equation.

$$\frac{(2.58\ \text{pCi/mL})\ (\text{mL}/1000\ \mu\text{L})\ (150\ \mu\text{L})}{50\ \text{mL}\ (\text{L}/1000\ \text{mL})} = 7.74\ \text{pCi/L} \quad .$$

8.2. Spike concentrations calculated in Step 8.1 are tabulated below.

Spike Amount (μL)	Concentration (pCi/L)	Uncertainty (±3.5%)
100	5.16	0.17
150	7.74	0.25
200	10.32	0.33
300	15.48	0.50
400	20.64	0.66

9. Source Materials

9.1. M. A. Gautier, E. S. Gladney, and D. R. Perrin, "Quality Assurance for Health and Environmental Chemistry: 1984," Los Alamos National Laboratory report LA-10508-MS (1985).

9.2. M. A. Gautier, E. S. Gladney, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1985," Los Alamos National Laboratory report LA-10813-MS (1986).

9.3. M. A. Gautier, E. S. Gladney, W. D. Moss, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1986," Los Alamos National Laboratory report LA-11114-MS (1987).

9.4. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1987," Los Alamos National Laboratory report LA-11454-MS (1988).

9.5. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1988," Los Alamos National Laboratory report LA-11637-MS (1989).

9.6. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

9.7. *EML Procedures Manual*, 25th ed., H. L. Volchok and G. de Planque, Eds. (Environmental Measurements Laboratory, New York, August 1982).

9.8. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material U-930, Uranium Isotopic Standard" (National Institute of Standards and Technology, Washington, DC, February 1966).

9.9. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4904L-F, Alpha-Particle Standard" (National Institute of Standards and Technology, Washington, DC, April 1982).

9.10. M. Phillips, "Handling Human Biological Fluids," Health and Environmental Chemistry SOP-4, September 1988, Los Alamos National Laboratory document.

URANIUM-238 IN URINE — QC SAMPLE PREPARATION

Analyte: Uranium-238	**Method No.:** QCR300
Matrix: Urine	**Spike Range:** 0.0 to 34.0 μg/L
Procedure: Delayed neutron activation	**Accuracy and Precision:** 100% ± 2.8% RSD
Effective Date: 01/01/75 to 08/01/91	**Authors:** Margaret A. Gautier William D. Moss Nancy L. Koski

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 5. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment.

1. Principle of Method

1.1. A pool of blank urine is prepared.

1.2. The blank urine is aliquoted and spiked with a known standard.

1.3. QC samples are submitted for analysis with each analytical batch and are analyzed along with the unknown samples using the same analytical procedure.

2. Accuracy and Precision

2.1. The propagated error on QC preparation is based on the calibration error from six sample plate preparations of the standard and on the error associated with use of the graduated cylinder and of the pipette. A relative standard deviation (RSD) of 2.8% has been calculated for the calibration, spiking, and aliquoting process based on the following function:

$$U = \frac{M1 \times M2}{M3 + M2} ,$$

where U = uncertainty,
M1 = calibration error,
M2 = spiking error, and
M3 = aliquoting error.

2.1.1. Calculate relative variance using the following equation.

$$R_M^2 = \frac{\sigma^2}{\overline{X}^2} ,$$

where R^2_M = relative variance,
M = $M_1 \ldots M_n$ (variable),
σ^2 = variance (std dev^2), and
$\overline{X}$ = mean value of each variable (M).

2.1.2. Calculate percent relative standard deviation using the following equation.

$$S_{D_T} = \sqrt{\frac{\left[R^2_{M1}\,(M_3 + M_2)^2 + M_3^{\,2}\,(R^2_{M2} + R^2_{M3})\right]}{(M_3 + M_2)^2}} \times 100 ,$$

where S_{D_T} = relative standard deviation (% error),
R^2_{M1} = relative variance of standard,
M_3 = volume of cylinder,
M_2 = volume of pipette,
R^2_{M2} = relative variance of pipette,
R^2_{M3} = relative variance of cylinder, and
100 = factor to convert to percentage.

2.2. Analysis by delayed neutron activation of QC samples prepared between 1984 and 1989 using this procedure gave a mean recovery of 98% ± 16 % RSD. The large standard deviation associated with the ^{238}U analysis in urine is related to the measurement of depleted uranium and its corresponding low production of delayed neutron counts. The ^{238}U standard used for the bioassay program is only 0.18% ^{235}U. The data are published in **Source Material** 9.1 through 9.6 and are summarized below.

URANIUM-238 SPIKE RECOVERY AS A FUNCTION OF CONCENTRATION

	Spike Level (μg/L)	Mean ± RSD (%)	Number of Samples
1984	8.48	101 ± 30	16
	12.72	102 ± 23	14
	16.96	103 ± 26	15
	25.44	99 ± 30	7
	33.92	100 ± 5	15
	42.40	97 ± 5	5
	50.88	93 ± 6	6
1985	8.48	94 ± 35	14
	12.72	103 ± 21	9
	16.96	91 ± 17	13
	21.2	97 ± 7	7
	25.44	92 ± 16	14
	33.92	98 ± 7	12
1986	8.48	85 ± 19	8
	12.72	101 ± 24	11
	16.96	92 ± 14	12
	21.20	103 ± 12	11
	25.44	94 ± 14	15
	33.92	99 ± 10	14
1987	8.48	114 ± 20	12
	12.72	92 ± 21	9
	16.96	100 ± 17	14
	21.20	103 ± 21	12
	25.44	108 ± 19	14
	33.92	99 ± 12	14
1988	8.48	99 ± 27	13
	12.72	115 ± 19	8
	16.96	93 ± 16	10
	21.20	105 ± 10	8
	25.44	98 ± 11	13
	33.92	99 ± 15	11
1989	8.48	95 ± 16	13
	12.72	98 ± 18	11
	16.96	97 ± 10	8
	21.20	90 ± 18	12
	25.44	96 ± 6	10
	33.92	92 ± 8	18

3. Collection and Storage of Samples

3.1. Urine is collected as needed from group personnel who have no known contact with radiation work and is used to form a pool of blank urine.

3.2. Spiked QC samples are refrigerated at a nominal temperature of 4°C pending analysis.

4. Apparatus

4.1. Beakers: 4- or 5-L, polyethylene.

4.2. Graduated cylinders: 50-mL.

4.3. Auto-pipette: 100- and 1000-μL, Rainin or equivalent.

4.4. Pipette tips: disposable.

4.5. Specimen containers: 120-mL with screw caps, sterile inner surface, catalog no. C8845-3, Baxter Health Care Corp., Tempe, AZ 85282, or equivalent.

4.6. Labels printed with QC sample numbers.

4.7. Heat lamp.

4.8. Filter paper: 2.9-cm, Whatman No. 41 or equivalent.

4.9. Discs: stainless steel, 2.22-cm-diam.

4.10. Alpha phosphor on Mylar: The phosphor is the standard silver-activated zinc sulfide. William B. Johnson & Associates, Inc., Research Park, Montville, NJ 07045.

4.11. Rings and discs: plastic rings and discs, nominal 1-in.-diam, molded of nylon. Control Molding Corp., 84 Granite Ave., Staten Island, NY 10303.

4.12. Mylar film: 1.5-in.-wide rolls, 0.0005-in.-thick. Manufactured by E. I. duPont de Nemours, retailed by Brownell, Inc., 85 Tenth Ave., New York, NY 10001.

5. Reagents

5.1. None required.

6. Calibration and Standards

6.1. QC samples are spiked with a natural uranium standard prepared in 2 N HNO_3. The standard should be at a concentration of approximately 1 μg/mL. The solution is counted on a zinc sulfide scintillation system calibrated with National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 4904L-F, an ^{241}Am alpha-particle standard, using count room procedure CR340.

6.2. The natural uranium stock standard is prepared from a certified NIST standard, such as NIST SRM 950a, U_3O_8, and contains 0.72% ^{235}U. Record all data in the QC Laboratory notebook.

6.2.1. Ignite approximately 1 g of U_3O_8 for 1 h at 900°C.

6.2.2. Quantitatively transfer 0.5896 g of the U_3O_8 to a 50-mL beaker with 20 mL of 1:1 solution of HNO_3.

6.2.3. Dissolve the U_3O_8 in the acid and evaporate to near dryness.

6.2.4. Dissolve the residue (nitrate) in 10 mL of a 1% HNO_3 solution and transfer quantitatively to a 500-mL volumetric flask. Bring to volume with 1% HNO_3 and thoroughly shake. This solution contains 1000 mg of U/L.

6.3. Intermediate uranium standard.

6.3.1. Dilute 1 mL of the stock solution to 1000 mL with 1% HNO_3.

6.3.2. The solution contains 1 mg U/L (1 μg U/mL).

6.4. Recalibrate the ^{238}U standard solution yearly by direct plating and alpha scintillation counting. Natural uranium (0.715 weight percent ^{235}U) has an alpha activity of 1.5 disintegrations per minute (dpm) per μg. Record all data in the QC Laboratory notebook.

6.5. Direct plating procedure.

6.5.1. Pipette a 500-μL aliquot of the standard to be calibrated on each of six 2.22-cm stainless steel discs, and evaporate to dryness under a heat lamp.

6.5.2. Place a 2.9-cm filter paper on a molded nylon disc. The filter paper assures a tightly sealed package when assembled.

6.5.3. Place the stainless steel disc containing the sample on the filter paper. Be sure they are centered.

6.5.4. Place a .875-in. phosphor paper over the stainless steel disc (dull side against the disc).

6.5.5. Place a piece of Mylar film over all the layers.

6.5.6. Press a nylon ring down over the layers and around the nylon disc to form a tightly sealed package. Cut off the excess Mylar with scissors. Label the back of the assembly with an identification number.

6.5.7. Submit the samples to the count room for alpha scintillation counting and request a 60,000-s (overnight) per sample count.

6.5.8. The count room data is corrected for background and reported in dpm. Divide the dpm results by 2.22 to convert to pCi. Calculate the mean and standard deviation of the six plates and assign the value to the standard.

7. Procedure

7.1. Working with bodily fluids is discussed in detail in SOP-4, "Handling Human Biological Fluids," found in Appendix III of the EM-9 Safety Manual.

NOTE: Wear safety glasses, gloves, and a lab coat. Each day prepare a 1:100 dilution of household bleach in water in a spray bottle. Use this solution to thoroughly clean the work area after sample preparation is completed. Remove gloves, discard in the biohazard waste container, and wash hands with soap and water.

7.2. Prepare a pool from blank urine sufficient in volume to allow 50 mL for each QC sample to be prepared.

7.3. Prepare QC samples to equal at least 10% of the total number of samples to be analyzed. Include blank QCs prepared from the same urine pool as the spiked samples.

7.4. Record QC standard information, sample spike amount, concentration, and uncertainty in the QC Laboratory notebook.

7.5. Label bottles with QC sample numbers.

7.6. Aliquot 50 mL of blank urine into each sample bottle.

7.7. Spike the blank urine with aliquots of standard as determined in Step 7.3.

7.8. Screw caps on bottles and refrigerate at a nominal temperature of 4°C pending analysis.

7.9. Enter the spike concentrations of each QC into the CVS and CVD data bases on the VAX. This procedure is done annually because the same concentration levels are used throughout the year.

8. Calculations

8.1. On 2 July 1982, HSE-1 (now HS-1) notified HSE-9 (now EM-9) that uranium with a 0.17% enrichment of ^{235}U was being used in the shops. Our standard value was recalculated to that same isotopic ratio using a 4.24 conversion factor in the following equation. *This conversion factor can be used only for delayed neutron activation.*

$$4.24 = \frac{0.72\%}{0.17\%} ,$$

where 4.24 = conversion factor,
0.72 = enrichment of NIST natural uranium, and
0.17 = enrichment of uranium used in Los Alamos shops.

8.2. Record spike concentrations as μg/L of urine. Calculate the spike concentration for a 200-μL aliquot of a 1-mg/L uranium standard using the following equation.

$$\frac{(1\ \mu g/mL)\ (mL/1000\ \mu L)\ (200\ \mu L)}{50\ mL\ (L/1000\ mL)} = 4\ \mu g/L \times 4.24 = 16.96\ \mu g/L \quad .$$

8.3. Spike concentrations calculated in Step 8.2 are tabulated below.

Spike Amount (μL)	Concentration (μg/L)	Uncertainty (±2.8%)
0	0.00	0.00
100	8.48	0.24
150	12.72	0.36
200	16.96	0.47
250	21.20	0.59
300	25.44	0.71
400	33.92	0.95

9. Source Materials

9.1. M. A. Gautier, E. S. Gladney, and D. R. Perrin, "Quality Assurance for Health and Environmental Chemistry: 1984," Los Alamos National Laboratory report LA-10508-MS (1985).

9.2. M. A. Gautier, E. S. Gladney, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1985," Los Alamos National Laboratory report LA-10813-MS (1986).

9.3. M. A. Gautier, E. S. Gladney, W. D. Moss, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1986," Los Alamos National Laboratory report LA-11114-MS (1987).

9.4. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1987," Los Alamos National Laboratory report LA-11454-MS (1988).

9.5. M. A. Gautier, E. S. Gladney, M. B. Phillips, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1988," Los Alamos National Laboratory report LA-11637-MS (1989).

9.6. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

9.7. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 950a, Uranium Oxide" (National Institute of Standards and Technology, Washington, DC, October 1969).

9.8. National Institute of Standards and Technology, "Certificate of Analysis for Standard Reference Material 4904L-F, Alpha-Particle Standard" (National Institute of Standards and Technology, Washington, DC, April 1982).

9.9. M. Phillips, "Handling Human Biological Fluids," Health and Environmental Chemistry SOP-4, September 1988, Los Alamos National Laboratory document.

PREPARATION OF A VEGETATION ASH MATRIX

Analyte: None	**Method No.:** QCM200
Procedure: Ashing of vegetation	
Effective Date: 11/29/90	**Authors:** Bennie A. Martinez Nancy K. Koski Margaret A. Gautier

SAFETY NOTE: Before beginning this procedure, read all of the Material Safety Data Sheets for the chemicals listed in Sec. 3. Read Sec. 4.3 of the EM-9 Safety Manual for information on personal protective clothing and equipment. Read Sec. 5 of this procedure for proper waste disposal practices.

1. Principle of Method

1.1. Vegetable matter is ashed slowly to create a vegetation ash suitable for use as a blank matrix for QC samples.

2. Apparatus

2.1. Beakers: 1-L, glass.

2.2. Aluminum foil.

2.3. Balance: 1-kg minimum capacity, 0.1-g accuracy.

2.4. Muffle furnace capable of reaching and sustaining a temperature of at least 550°C.

2.5. Engraver.

2.6. Bottle: 1-gallon capacity or smaller.

2.7. Sieve: No. 18

2.8. Ball mill and polyethylene container, with lid, for ball mill.

2.9. Steel balls: 5/8- to 3/4-in. diameter.

2.10. Vinyl tape.

2.11. Spatula.

2.12. Insulated gloves.

3. Reagents

3.1. Chicken Scratch or an equivalent vegetation feed may provide a suitable ash.

3.2. A suitable vegetation ash does not contain the analyte of interest and presents few interferences to the analytical method.

4. Procedure

4.1. The muffle furnace should be equipped with a safety switch which shuts the furnace off in case the temperature exceeds its setting. It should also be vented or located where fumes will not contaminate working spaces.

4.2. Weigh approximately 500 g of chicken feed into a 1-L beaker.

4.3. Dry the feed in a muffle furnace at 100°C for about 68 h.

4.4. Cover the beaker with aluminum foil perforated with 1/4- to 1/2-in. holes.

4.5. Ash the feed at 150°C for approximately 8 h, then raise the temperature and ashing time as follows:

200°C for approximately 16 h,
250°C for approximately 8 h,
300°C for approximately 16 h,
400°C for approximately 8 h,
450°C for approximately 16 h,
500°C for approximately 8 h, and
550°C for approximately 7 d.

4.6. Turn off the furnace and allow the sample to cool for about 16 h.

4.7. Remove the beaker from the furnace and transfer the ash to the ball mill container. Fill the container approximately half full and add about 95 steel balls. Cap the container and seal it with tape.

4.8. Place the container on the ball mill and grind the ash for about 1 h.

4.9. Allow the ash to settle before opening the container.

4.10. Sift the ash through a No. 18 sieve. Using a spatula, transfer the ash that passes through the sieve to a storage bottle and place the rest back into the ball mill

container. Repeat the grinding and sifting until all the ash has been ground and sifted.

4.11. Re-ash the matrix to constant weight.

4.11.1. Fill a 1-L beaker, engraved with an identifying number, approximately half full with sifted ash. Cover the beaker with aluminum foil perforated with 1/4- to 1/2-in. holes.

4.11.2. Weigh the covered beaker and ash. Record the weight in a Laboratory notebook.

4.11.3. Ash the feed in a muffle furnace at 250°C for approximately 75 min, then raise the temperature and ashing time as follows:

300°C for approximately 1 h,
350°C for approximately 1 h,
400°C for approximately 90 min,
450°C for approximately 90 min,
500°C for approximately 90 min, and
550°C for approximately 3 d.

4.11.4. Turn off the furnace and allow the ash to cool for approximately 16 h.

4.11.5. Weigh the covered beaker and ash. Record the weight and compare it with the previously recorded weight. If there has been no significant change and the ash is gray, no further ashing is necessary.

4.11.6. If there has been a significant change in weight or if the ash is not gray in color, reash the feed at 550°C for about 36 h. Continue weighing and re-ashing until the ash is gray and at a constant weight.

5. Proper Waste Disposal Practices

5.1. No waste is produced.

6. Source Materials

6.1. M. A. Gautier, E. S. Gladney, N. L. Koski, M. B. Phillips, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1989," Los Alamos National Laboratory report LA-11995-MS (1990).

6.2. M. A. Gautier, E. S. Gladney, N. L. Koski, E. A. Jones, and B. T. O'Malley, "Quality Assurance for Health and Environmental Chemistry: 1990," Los Alamos National Laboratory report LA-12208-MS (1991).

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Laboratory Safety

HSE-9 SAFETY MANUAL

COMPILED AND EDITED BY

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ISSUED MAY, 1987

REVISED JANUARY, 1990

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Peggy Gautier, HSE-9 Safety Chairman	4-12-90

HSE-9 SAFETY MANUAL

ISSUED MAY, 1987

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HSE-9 SAFETY TRAINING RECORD

If you have any questions regarding this material contact your Section Leader or a member of the HSE-9 Safety Committee.

I have read and understood the material presented in the HSE-9 Safety Manual in fulfillment of employment requirements regarding safety.

Return to the Group Office for filing by_____

Signed_____

Date_____

HSE-9 is required by HSE-Division to establish and maintain a record system on all aspects of their safety program. These records are subject to HSE-Division Audit.

NEW EMPLOYEE SAFETY CHECKLIST

Health and Environmental Chemistry, Group HSE-9

The supervisor will give a copy of this form to the safety chairman after reviewing it with the new employee.

_____1. Safety is the responsibility of each employee. If the employee has any questions concerning the safety of any aspect of a job assignment, the question must be brought to the attention of the employee's supervisor or the HSE-9 Group Leader.

_____2. Standard Operating Procedures (SOPs) that are applicable to the employees job assignment will be reviewed by the employee on an annual basis. An employee sign-off sheet will be initiated by the supervisor to document that the employee has read and understood the SOP. The sign-off sheets will be filed by the group safety coordinator.

_____3. Copies of the site emergency plan and instructions for actions during an emergency (e.g., fire, explosion, etc.) are in the HSE-9 Safety Manual. A sign-off sheet will be maintained to document that the employee has read and understood the most current site emergency plan.

_____4. It is Laboratory policy that all employees (drivers and passengers) wear seat belts while using government vehicles.

_____5. Employees who use government vehicles are responsible for notifying the HSE-9 Group Office of any problems (mechanical, electrical, etc.) with the vehicles.

_____6. Any accident involving an injury to the employee shall be reported to the group office, section leader, and to the group safety chairman. A formal report can be made after receiving medical treatment at HSE-2. An accident involving a potential injury shall be reported to the supervisor and the safety coordinator. Information concerning potential injuries may be useful in preventing injuries to other employees.

_____7. Any accident involving a government vehicle shall be reported to the group office. Local police must be notified immediately if a government vehicle accident involves a personal injury occurring off site.

_____8. Employees will attend group health and safety meetings.

I have read and understood the safety policies of the Health and Environmental Chemistry Group.

_____

Signature Date

PREFACE

Operational and building safety is of primary importance to HSE-9. This manual has been prepared to assist the employees in understanding safety hazards and in learning the proper response to emergency situations.

This manual includes sections on safety policy, the safety committee, emergency plans, emergency procedures, personal safety, and chemical safety. Topics such as emergency alarms, fire, evacuation, personnel training, building diagrams, chemical and radiation protection, and work philosophy are some of the subjects discussed in depth. Supplemental information is presented in the appendixes.

All who work in HSE-9 want accidents and injuries to be eliminated or kept to an absolute minimum and want methodical and safe response to unexpected incidents. Therefore, it is the responsibility of all members of HSE-9 to become familiar with the safety manual and to use safe working habits.

This document is the first edition of a formal safety manual of good health and safety practices for HSE-9 personnel. It will be reviewed annually and updated as necessary.

Copies of the Safety Manual distributed externally to the Los Alamos National Laboratory will not contain Appendixes I - VI. The enclosed document is to be used for informative purposes only. The Los Alamos National Laboratory, the University of California, and the Department of Energy expressly disclaim any liability for accident, injury or loss of property associated with the use by other agencies or organizations of this Los Alamos National Laboratory document in either its original or modified form.

ACKNOWLEDGMENTS

Thanks are due Stuart D. Nielsen and John Miglio for their past contributions to the Chemical Safety section of this manual. The general format of this manual was modeled after that used by MST Division for the TA-55 facility.

Peggy Gautier
Chairman, Safety Committee
Manual Editor

HSE-9 EMERGENCY TELEPHONE NUMBERS

1. For all emergencies call 911

   A. Sound local alarm.

   B. Give the following information on all 911 calls and stay on the line until the operator breaks the connection.

      Name_____

      Nature of emergency_____

      Location of TA_____ Bldg_____ Rm_____

      Your telephone no_____

   C. Notify your supervisor_____

   D. Remain in the area, if possible, to direct responding emergency units.

   E. Use fire extinguisher if you can do so without endangering yourself.

   F. Unless a life-threatening situation exists, do not move injured personnel.

   G. Report to the assembly area to be counted if evacuation is ordered.

2. Additional telephone numbers.

HSE Division Office		7-4218
HSE-1 Group Office		7-5296
HSE-2 Physician		7-7251
HSE-5 Group Office		7-5231
HSE-7 Group Office		7-4301
HSE-9 Group Office	Craig Leasure	7-3269
HSE-9 Section Leaders		
Radiochemistry	Daryl Knab	7-7094
Inorganic	Michael Bell	7-6011
Organic	Chris Leibman	7-5889
QA and Data Management	Peggy Gautier	7-6235
Tissue	Jim McInroy	7-4709

Protective Force Duty Officer 7-4437

Laboratory Emergency Duty Officer 7-6211

Health Division Assistance for Radiological, Chemical or
Industrial Accidents (8 am - 5 pm) 7-7878

Health Division Assistance (nonduty hours) 7-7080

HSE Division On-Call Emergency Coordinator (after hours)
Pager Number 104-2631
From a non-Lab Phone . . . . . 665-0351 then 2631

PAN AM Assistance 7-6191

3. Fire alarm--high pitched whooping sound.

4. Basement alarms TA-59.

   A. Ringing Bell. Immediate assistance required in room B4.

   B. High pitched sound and light by room 105 flashing. Immediate assistance required in room B8-K or B8-M.

   C. Lights activated on panel between room 105 and room 107. Alarm system for Tissue Section freezers.

5. Bomb Threat--Use checklist on page xv.

6. Evacuation--Shut down equipment, close doors, report to muster area.

7. HSE-9 After Hours Emergency On-Call List

HSE-9 Group Office . . . . . Group Leader	Craig Leasure	662-7685
Radiochemistry Acting Section Ldr. . . . . Radiochemistry Analysis, Bioassay Samples Environmental Samples, Radiocounting	Daryl Knab	672-9540
QA & Data Management Section Ldr . . . . . QA & Data Management, Bioassay Samples	Peggy Gautier	662-5205
Inorganic Section Leader . . . . . Inorganic Analysis, Inorganic Samples Waste Treatment Samples	Mike Bell	662-3673
Organic Section Leader . . . . . Organic Analysis, PCB Analysis Organic Samples, Solvents Analysis	Chris Leibman	988-5372

BOMB THREAT CALL CHECKLIST

INSTRUCTIONS: IF RECEIVED BY TELEPHONE, BE CALM AND LISTEN CAREFULLY, DO NOT INTERRUPT OR ANTAGONIZE THE CALLER. ATTEMPT TO KEEP THE CALLER ON THE LINE WHILE SIGNALING ANOTHER PERSON THAT YOU HAVE A BOMB THREAT. THE OTHER PERSON SHOULD DIAL 911 AND ASK THAT THE EMERGENCY OPERATIONS CENTER BE NOTIFIED THAT A BOMB THREAT HAS BEEN RECEIVED AND INSTRUCT THEM AS TO WHAT TELEPHONE LINE THE THREAT CAME IN ON. AFTER NOTIFICATION, THE OTHER PERSON SHOULD TRY TO JOIN YOU ON THE THREAT LINE AND TAKE NOTES OF THE CONVERSATION AS THE CALLER IS TALKING.

Time and date received: _____

How received: _____ Time caller hung up: _____

Exact words of the caller: _____

_____

_____

QUESTIONS TO ASK:
1. When is the bomb going to explode? _____
2. Where is the bomb right now? _____
3. What kind of bomb is it? _____
4. What does it look like? _____
5. Why did you place the bomb? _____
6. Where are you calling from? _____

Describe the callers voice _____

MALE____FEMALE____YOUNG____OLD____ACCENT_____TONE OF VOICE _____

Background noise: _____

Is the voice familiar?_____ Who does it sound like? _____

Other voice characteristics: _____

Remarks: _____

_____

_____

Your name, address and telephone number: _____

_____

When 911 is called regarding a bomb threat, there are several services that are alerted to the fact that a threat has been received against the laboratory. Emergency management is immediately notified by the Protective Force and a phone trace is initiated by the Police Department. If the threat is a short telephone conversation and you are unable to keep the caller on line, it is still possible to receive a trace; however, if the call originated from other than a local exchange, a trace can be accomplished only if the caller stays on the line for a longer time.

Trying to develop a rapport with the telephone caller and being sympathetic to his cause seems to work best. The caller is looking for recognition and fulfilling that need can help to keep the conversation going. It is impossible to suggest what will work with each caller. If you do develop rapport with the individual, you may be surprised at the amount of information that you can obtain. One of the last questions that needs to be asked (and one of the most important) is the callers name, address, and the phone that they are using. Believe it or not, many bomb threat callers have given the above information simply because they were asked to furnish it.

REMEMBER, REMAIN CALM AND OBTAIN AS MUCH INFORMATION AS POSSIBLE!

SOURCE MATERIALS

Administrative Manual, Section 900 Health and Safety, Los Alamos National Laboratory (November 1985).

Administrative Manual, Section 700 Employee Responsibilities, Los Alamos National Laboratory (May 1983).

L. A. Gritzo, "Transmittal of Laboratory Standard Operating Procedures (SOPs) to External Agencies," Los Alamos National Laboratory memorandum M-DO-1177 (June 14, 1988).

D. W. Apts and K. L. Blankenship, *Back Facts*, 2nd ed. (FPR, Inc., Ashland, Kentucky, 1981).

W. Braker, A. L. Mossman, and D. S. Siegel, *Effects of Exposure to Toxic Gases-First Aid and Medical Treatment*, 2nd ed. (Matheson Publications, Lyndhurst, New Jersey, 1979).

Cardiopulmonary Resuscitation (The American National Red Cross, 1981).

"Designation of Hazardous Substances," 40 CFR 116.

Electrical Safety Training Course Manual, Group HSE-3, Los Alamos National Laboratory, (1986).

"Emergency Planning, Preparedness, and Response for Operations," DOE Order 5500.2 (August 1981).

"Environmental Protection, Safety, and Health Protection Program for DOE Operations," Department of Energy Order 5480.1A (August 1981).

Environmental, Safety, and Health Bulletin, DOE/ED-0003, Issue No. 14 (September 1985).

Guide to Safe Handling of Compressed Gases (Matheson Publications, Lyndhurst, New Jersey, 1982).

Handbook of Fire Protection, 15th Ed., National Fire Protection Association (NFPA) (1982).

"Hazard Communication Standard," 29 CFR 1910.1200.

Health and Safety Manual, Los Alamos National Laboratory (March 1986).

"Health Safety and Environment Division Emergency Operations Plan," HSE Division internal document, Los Alamos National Laboratory (June 1986).

"Los Alamos National Laboratory Emergency Response Plan;" (September 1985).

Matheson Gas Data Book, 6th ed. (Matheson Publications, Lyndhurst, New Jersey, 1980).

NIOSH Pocket Guide to Chemical Hazards (U.S. Government Printing Office, Washington D.C., September 1985).

NIOSH/OSHA Occupational Health Guidelines for Chemical Hazards (US Department of Health and Social Services and US Department of Labor 1981).

"Occupational Safety and Health Standards," 29 CFR 1910 (revised June 1981).

Safety in Academic Chemistry Laboratories, 4th ed. (American Chemical Society Committee on Chemical Safety, Washington, D.C., 1985).

G. J. Shugar, R. A. Shugar, and L. W. Bauman, *Chemical Technicians' Ready Reference Handbook*, 2nd ed. (McGraw-Hill Book Company, New York, 1981), Chap. 1.

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Health, Safety, and Environment Policies

Introduction

The Laboratory's health, safety, and environment policies, as stated below, are implemented through the administrative requirements found in this Manual. The Health, Safety, and Environment Council, whose members are chosen from upper-level management, recommends policies to the Director and oversees policy implementation.

1. Overall Policy

The Laboratory will provide the highest possible level of protection to its employees, the public, government property, and the environment from harm that could arise from Laboratory operations. To accomplish this objective, (1) line management will be responsible for conducting only those operations and activities that can be controlled safely; (2) the Health, Safety, and Environment Division will maintain a comprehensive health, safety, and environment program to assist line management and to provide an overview of health, safety, and environment activities; and (3) Laboratory employees will be required to observe the health, safety, and environment procedures and requirements specified by their supervisors.

2. Management Responsibility

The primary responsibility for employee health and safety on the job as well as for environmental protection from Laboratory operations rests with line management; this responsibility will be given first priority before Laboratory operations are approved or carried out. Supervisors are expected to recognize and anticipate potential hazards, to inform employees of risks associated with their work, to specify protective measures, and to ensure that their employees receive appropriate training. Supervisors also will establish and maintain a system to ensure that appropriate consideration is given to significant changes made in operations, procedures, materials, or equipment that could affect the safety of an activity, including environmental impact.

3. Employee Responsibility

Employees are often in the best position to evaluate health and safety risks that might result in harm to themselves and their coworkers. Therefore, Laboratory employees are responsible for observing applicable health, safety, and environment procedures; for using prescribed personal protective equipment; for promptly reporting accidents, injuries, and unsafe conditions; and for participating in required medical and biological monitoring programs.

4. Health, Safety, and Environment Division Responsibility

The Health, Safety, and Environment (HSE) Division will initiate and promote a comprehensive health, safety, and environment program, which will include such special fields as radiation protection, occupational medicine, industrial safety, industrial hygiene, nuclear criticality safety, waste management, and environmental protection and preservation. HSE Division will address special requirements or needed emphasis within these programs, such as maintenance of toxic and radiation levels as low as reasonably achievable; transportation, handling, and disposal of hazardous materials; specific health, safety, and environment training; and emergency response. An appraisal program will be maintained by HSE Division to ensure that the Laboratory's health, safety, and environment policies are appropriately interpreted and seek to minimize risk, and to assist Laboratory organizations in implementing programs consistent with those policies. Laboratory activities will be monitored and any activity or situation that is inconsistent with good health, safety, and environment practices will be brought to the responsible manager's attention.

5. Training

Laboratory employees will be provided with health, safety, and environment training to help them safely perform their assigned tasks. This training will emphasize the Laboratory's health, safety, and environment procedures--particularly recognition and control of potential hazards--to enable employees to better evaluate risks associated with their work.

Supervisors will instruct their employees in applicable health, safety, and environment procedures relevant to specific job requirements and will maintain records to reflect the current status of training received by each employee, including certification and retraining.

6. Transportation of Hazardous Materials

Off-site and on-site shipments of radioactive and other hazardous materials and waste will be made in accordance with applicable regulations. These shipments will be made in a manner that protects the health and safety of employees and the public and minimizes the potential for release of such materials to the environment.

7. Siting Activities or Facilities in Hazardous Areas

The Laboratory will generally avoid siting unrelated or nonhazardous activities in potentially or known hazardous areas. If a short-term (up to 6 months) activity or operation is planned for such a hazardous area, a special work permit must be prepared by the division proposing the activity and approved by the division leader responsible for the area and the Health, Safety, and Environment Division Leader. If the activity is planned for a period of 6 months or more, it also must be approved by the Health, Safety, and Environment Council.

8. Protection of Certain Classes of Employees

Employment and activities of certain employees will be restricted in accordance with applicable federal and state regulations.

People with Disabilities. The Laboratory makes reasonable accommodations for disabled employees or applicants, unless the accommodation will impose undue hardship on Laboratory operations (see *Administrative Policies and Procedures Manual*, Sec. AM 115).

The Occupational Medicine Group (HSE-2) determines work restrictions made necessary by the employee's or prospective employee's disability. HSE-2 also conducts a medical evaluation and recommends appropriate accommodatio to permit an employee to continue working when that person becomes or is believed to have become disabled.

Women of Childbearing Age. The Laboratory recognizes that women of childbearing age engaged in certain occupations may require special consideration. To enable the Laboratory to provide the necessary protection, a female employee working in a radiation area or with toxic materials must report a pregnancy or planned pregnancy to the Occupational Medicine (HSE-2) Group Leader. The need for temporary restrictions or change of work assignment then will be determined on a case-by-case basis and discussed with the appropriate supervisor to protect both the employee and the fetus. Such temporary restrictions or transfers will not result in an economic penalty or a change in status for the employee.

9. Safety of Contract Personnel and Visitors

A safe work environment will be provided for contract personnel and visitors. This includes certain health protection services provided by HSE Division. The Laboratory's health, safety, and environment rules will be enforced for everyone visiting or working at the Laboratory. Laboratory employees who visit or work at another installation will strictly observe that facility's health, safety, and environment regulations.

1.2. HSE Division Safety Policy Statement

HSE DIVISION SAFETY POLICY AND PROGRAM

REVISED - DECEMBER 1988

APPROVED BY:

JOHN M. PUCKETT

DIVISION LEADER

The HSE Division shall conduct its activities in such a manner that the health and safety of all its employees, guests and the public are protected, and that the probability of damage to property and the environment are reduced to the lowest level reasonably achievable.

The Division Leader (DL), each Group Leader (GL) and Program Office Manager (PL) shall develop and implement a program that addresses the health, safety and environment risks (referred to hereafter as "Safety" Program) associated with that group's activities and is in conformance with the Laboratory's Health and Safety Manual and DOE requirements.

DLs, GLs and PLs are responsible for their programs and shall make sure that each employee is aware of and meets his safety responsibilities.

GLs and PLs shall evaluate their individual programs on an annual basis and report their findings to the HSE Division Leader by January 30 of each year.

For the purpose of this document, "Group" shall be understood to mean all organizational units within the Division, including the Division Office.

The entire HSE Division Health, Safety and Environment Policy and Program is reproduced in full in Appendix II.

1.3. HSE-9 Safety Policy Statement

Los Alamos

Los Alamos National Laboratory
Los Alamos, New Mexico 87545

memorandum

TO: HSE-9 Personnel

DATE: January 12, 1990

FROM: Craig Leasure, HSE-9 Group Leader CSL

MAIL STOP/TELEPHONE: K484/3269

SYMBOL: HSE-9/86-345

SUBJECT: HSE-9 SAFETY POLICY

Health and Environmental Chemistry management recognizes that chemical operations are potentially hazardous to its employees. HSE-9 will conduct its operations in compliance with the general health and safety policy of the Laboratory. Particular emphasis is placed on assuring that all hazardous operations are conducted to provide for the protection of HSE-9 employees or the operation will not be conducted.

Line management of HSE-9 will be responsible for the establishment, implementation, and maintenance of an effective health, safety, and environment program for all facilities and activities under their control. HSE-9 management advises that any person performing or expecting to be performing operations he feels unsafe shall cease those operations until he is convinced they are safe. All HSE-9 employees shall keep the risk of their activities at the lowest levels achievable.

To assist HSE-9 employees in conducting their operations safely, the following general safety rules apply.

- Any operation deemed hazardous enough to require assistance in case of an emergency will be conducted only when another individual is nearby.
- With the exception of office work, working alone after hours is categorically forbidden.
- All hazardous work, including work with hazardous chemicals, should be evaluated to determine if an SOP/SWP is required.
- All operations will be conducted to verify that the least flammable materials are used in the operation and that all general fire protection rules are followed.
- All new procedures shall be reviewed by the section leader, the group office, and appropriate Health Division personnel to verify that hazards have been defined before the project begins.

A conscientious and continuing safety training program will improve safety performance in today's work environment. HSE-9 supports this philosophy by implementing a safety training program and a laboratory safety manual for its employees. Periodic safety meetings and safety inspections are conducted to inform HSE-9 personnel of safety considerations.

We will continue to make HSE-9 a safe working environment by informing employees of work-related risks, specifying the necessary protective measures, helping employees to recognize potential hazards, and assuring our employees are properly trained. Employees who do not follow safety procedures may be subject to appropriate disciplinary action up to and including dismissal.

1.4. HSE-9 Safety Committee Charter

The HSE-9 Safety Committee provides assistance to group management in the areas of health and safety. The primary objective of this committee is the promotion of individual and corporate safety in the HSE-9 working areas. The committee coordinates safety activities, facilitates communication, and deals with group-wide problems.

1.4.1. Membership

Safety committee members are appointed by the group leader for staggered two-year terms to ensure the continuity of the safety committee. In addition to the chairman of the committee, members include one representative from each section.

1.4.2. Support Services

The safety committee can invite to its meetings support personnel from health physics (HSE-1, -10, -11), occupational medicine (HSE-2), safety (HSE-3), industrial hygiene (HSE-5), waste management (HSE-7), maintenance and operations (ENG-4), and other groups as needed. These persons will not be voting members of the committee.

1.4.3. Organization

A chairman is appointed by the group leader for a term of two years and the chair rotates through the sections. The committee elects other officers (e.g., secretary) from the membership for a term of one year. Committee members may be assigned specific tasks, such as training, inspection scheduling and coordination, as determined by the committee.

1.4.4. Meetings

The safety committee will meet as required to pursue the safety program, but not less than quarterly.

1.4.5. Goals

The major goals of the safety committee are to create a safer working environment for HSE-9 and to increase safety awareness of HSE-9 employees both on and off the job.

1.4.6. Activities

The safety committee recommends safety-related actions to the group leader. Specific activities of the committee include the following.

- Attend committee meetings or arrange for a substitute.

- Prepare and distribute minutes of meetings to HSE-DO, HSE-3, and the HSE-9 group office.

- Verify compliance with policies and review any prior recommendations at each meeting to determine progress and/or status of the recommendations.

- Arrange for electrical and safety inspections to identify hazards of physical facilities, equipment, materials, and procedures.

- Maintain a file of inspection reports and follow-up actions.

- Arrange programs for a minimum of four safety meetings per year for HSE-9 personnel. Document the topic of each meeting and attendance. Line managers must put their title after their name. File these records in the HSE-9 group office and send copies to HSE-DO.

- Review the HSE-9 Safety Manual annually and issue revisions as needed.

- Review annually the Emergency Response Plans for the areas where HSE-9 members are working.

- Receive reports of HSE-9 accidents, incidents, and unusual occurrences and make such records available to authorized personnel. Encourage and participate in the preparation of reports of unusual occurrences.

- Determine training needs and provide comprehensive and job-specific training to each employee.

- Promote safety awareness and encourage safety-related communication on all levels.

- Assess risks related to HSE-9 activities and determine if Standard Operating Procedures (SOPs) or Special Work Permits (SWPs) may be needed.

- Review all SOPs initially, annually, and whenever major changes are made to processes, equipment, or facilities. Maintain written records of such reviews and send written notification to HSE's SOP Office when the review is completed.

- Assist the HSE-9 group office on other safety-related issues.

1.4.7. Inspection Teams

A safety inspection team consists of at least one staff member and two technicians and can be accompanied by others, including a representative from HSE-3.

The team leader writes a report of the inspection, emphasizing repeat items, and gives the report to the safety committee chairman within one week. Line managers present during safety inspections must be noted by name and title in the written report.

The schedule of inspections for an entire calendar year is set up by the safety committee chairman at the beginning of the year. At least one electrical and two general safety inspections will be conducted each year.

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2. EMERGENCY PLAN

2.1. Introduction

Emergencies are situations that endanger or adversely affect people, property, or the environment, occur rapidly and unexpectedly, and require immediate action. Emergencies of concern to HSE-9 may occur within our Group or may originate elsewhere in the Laboratory.

The keys to successful emergency response lie in anticipation, advance planning, and adequate preparation, along with the use of generalized guidelines for specific types of possible emergencies. The remainder of this section discusses the emergency organization, the Emergency Response Team (ERT), the emergency alarm system, the emergency equipment at TA-59 and TA-50, personnel accountability, and the test evacuation. Section 3, Emergency Response, discusses general guidelines for emergency response to major anticipated emergencies.

2.2. General Objectives

This emergency plan is to provide protective measures for possible emergencies and to minimize the consequences of any accident in HSE-9. The plan is to

- provide a timely and organized response,
- minimize injury to personnel,
- minimize damage to laboratory property, and
- restore normal operations as quickly as possible.

2.3. Possible Emergencies

Possible emergencies occurring in HSE-9 are as follows:

- utility failures,
- occupational accidents,
- medical illness or injury.
- vehicle accident,
- fires and explosions,
- hazardous materials release,
- natural disasters, such as heavy snowfall or forest fire,
- radiological incidents, and
- terrorist attack.

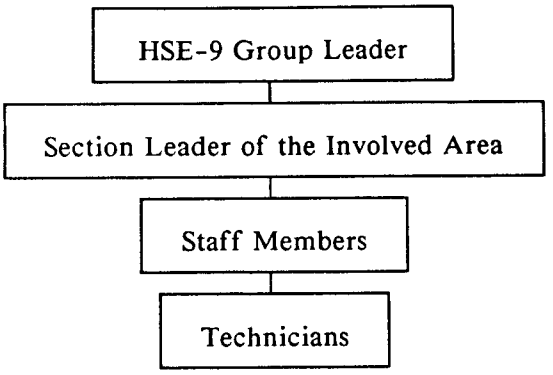


Fig. 2.1. Emergency chain of command at HSE-9.

2.4. Emergency Organization

2.4.1. Chain of Command

The HSE-9 Group Leader or his designee is the Emergency Coordinator and will respond to all emergency situations involving HSE-9. The responsibility for directing emergency response during emergencies is automatically delegated to individuals on the scene according to the chain of command shown in Fig. 2.1.

When emergencies arise, the first person on the scene is to coordinate all emergency response activities. This responsibility can only be transferred upon arrival of a person of higher authority in the emergency chain of command. In all emergencies close cooperation and communication is mandatory.

2.5. Emergency Response Team

2.5.1. Definition

The Emergency Response Team (ERT) is composed of members of HSE-9 who have been trained and certified in first aid and CPR and are knowledgeable about the operations in HSE-9. An Emergency Response Team Leader will be appointed by the Group Leader. The Emergency Response Team Leader's duties are to ensure that the team is trained and certified on skills, techniques, equipment, and procedures to effectively respond to any potential emergency situations in HSE-9 and to see that team certifications remain current. The Team Leader provides current ERT membership listings to the HSE-9 Group Leader.

2.5.2. Training

The HSE-9 Emergency Response Team receives the following training.

Topic	Frequency of Training
Red Cross First Aid	Every 3 years
Red Cross CPR	Every year
Chemical Spill Response	Every year

The HSE-9 Safety Committee will sponsor other safety-related courses upon request.

2.5.3. Team Response

Emergency team members are expected to respond independently to situations requiring CPR and first aid. The ERTs duties are as follows:

- Perform emergency first-aid and CPR.
- Take immediate actions necessary for safe shutdown of operations in the area.
- Assist in describing and evaluating the emergency situation.

2.6. Emergency Alarms

2.6.1. Individual Alarms at TA-59

Alarm	Sound
B4 Alarm	Ringing Bell
B8-K and B8-M Alarm	High Pitch with Flashing Light by Room 105
Freezer Alarm	Lights Activated on Panel by Room 105

Action upon sounding of an alarm is outlined in Emergency Response, Section 3.

2.6.2. Activation of Alarms

- Fire Alarm. Primary fire detection is provided by a sensor system on the wet pipe sprinkler that alerts Fire Department Headquarters. For manual activation of the alarms, use any fire-alarm box shown in Figs. 2.2.-2.6. Fires in HSE-9 laboratories are serious because of the potential damage from the fire itself and the reactive nature of the chemicals in use.

- Evacuation Alarm. Evacuation of building OH-1 for other than fire is announced over the PA system.

- B4 Basement Alarm. The alarm system is activated by pulling ring chains in the following locations:

  (1) adjacent to the door to the furnace room;

  (2) near the emergency shower in room B-4;

  (3) at the concrete pillar across from the basement woodworking shop; and

  (4) at the concrete pillar across from the dumbwaiter located in the northeast end of the basement. See Fig. 2.3.

  These areas are located in the east end of the basement. Once activated, the alarm will continue to ring until the reset button located adjacent to room 107, on the 1st floor, east wing is pushed.

- B8-K and B8-M Alarm. A flashing light, accompanied by a high pitched alarm, is activated by pushing the RED switch in rooms B8-K and B8-M. The system is deactivated and reset by pushing the BLACK button on the bottom of the switch box in these rooms. These labs are located in the west end of the basement. See Fig 2.3.

- Freezer Alarms. Lights are activated on the electrical panel located on the wall between rooms 105 and 107 on the 1st floor, east wing. This is the alarm system for the tissue section freezers.

2.6.3. Familiarization of Personnel with Alarm Sounds

The alarm system will be demonstrated annually. A Safety Committee representative ensures that the alarm drill is conducted.

2.7. Emergency Equipment and Its Location

2.7.1. Safety Showers

Safety showers are provided throughout the hallways and in several laboratories in the west wing of OH-1 at TA-59 and in the laboratory at TA-50. A shower is directly across from room A137 at TA-35. Exact locations are shown in Figs. 2.2.-2.6.

2.7.2. Eyewash Stations

Eyewash stations are located in all chemical laboratories in the east and west wings of OH-1, and in the laboratory at TA-50. Exact locations are shown in Figs. 2.2.-2.6.

2.7.3. Fire Extinguishers

Fire extinguishers are located in the hallways of OH-1 and TA-35 and in the laboratory at TA-50. Exact locations are shown in Figs. 2.2.-2.6.

2.7.4. First Aid Kits

First aid kits are located at TA-50 and in the east and west wings of OH-1 at TA-35 and TA-59. Locations are shown in Figs. 2.2.-2.6.

2.7.5. Chemical Spill Clean-Up Kits

Clean-up kits for acids, caustics, and solvents are located in the east and west hallways and basement of OH-1, in the main laboratory and in the basement near room 118 at TA-50, and in room A133 at TA-35. Each kit contains an instruction manual on how to clean up spills.

2.8. Accounting for Personnel in HSE-9

The attendance list of personnel will be used for personnel accountability. The group secretary at TA-59 will bring the attendance list to all building evacuations and drills. All employees should inform their section leader each time they leave the building. HSE-9 personnel located at TA-50 and TA-35 will follow the emergency procedures for those areas.

2.9. Building Evacuation

2.9.1. Test Evacuation

A test evacuation will be held annually to familiarize HSE-9 personnel with evacuation procedures. All practice evacuations will have advance notice.

2.9.2. General Evacuation Guidelines

- Employee participation in drills is mandatory as proper procedures are shown and any flaws in the evacuation plan become apparent.
- Close and lock safes, security file cabinets, etc. However, if you are away from the office, exit immediately; do not return to your office to do this task.
- Adjust operations to a safe stand-down status. The stand-down procedure should be outlined in the operating instructions for each system.
- Use the closest exit and leave the work area as rapidly as possible. Know the location of a second exit in case your primary exit is blocked. (Occupants of rooms 101-104, in the east wing of OH-1, should exit through the east door and ignore that it is wired to an alarm.)

- Proceed without socializing to the assembly area. See Fig. 3.1. in Section 3. Do not loiter. Escort visitors and contract personnel out of the building. Evacuation of OH-1 and TA50 should be completed in less than two minutes.

- Do not take along coffee or drinks. Spills in crowded hallways lead to more problems.

- Do not run. A fast walk covers as much ground as a jog or slow run, and you will have better control of your footing.

- Do not re-enter the building once you have exited. Proceed to the assembly point. Do not go through a building as a short cut.

- Muster in your proper area. See Fig 3.1. in Section 3. Take roll by name using a group roster. Move upwind if directed to do so.

- Stay in the assembly area after you have been accounted for. Further information and instructions will be given from this area.

- Stay calm and help those around you to stay calm. Do not spread rumors.

- Do not light up a cigarette. There may be a broken gas main or flammable vapors in the area.

- Watch out for emergency vehicles. They have right-of-way.

- Do not return to your work area until advised to do so by the person in charge.

2.10. Review of Group Emergency Plan

The emergency plan is reviewed and updated yearly by the HSE-9 Safety Committee.

2.11. HSE-9 Responsibilities within the HSE-DO Emergency Plan

HSE-9 responds to emergencies requiring chemical analysis, such as spills or potential chemical or radionuclide exposures, and has the following responsibilities:

- providing organic, inorganic, stable element, and radiochemical analyses in support of environmental monitoring, environmental compliance, industrial hygiene, health physics, and waste management,

- developing new, rapid-response analysis techniques that provide immediate estimates of the magnitude of an emergency,

- providing expert advice on sampling techniques,

- advising the HSE Division Leader on Health and Environmental Chemistry Group readiness to handle emergency situations,
- training personnel on rapid analysis techniques, and
- maintaining complete records on all aspects of analytical work.

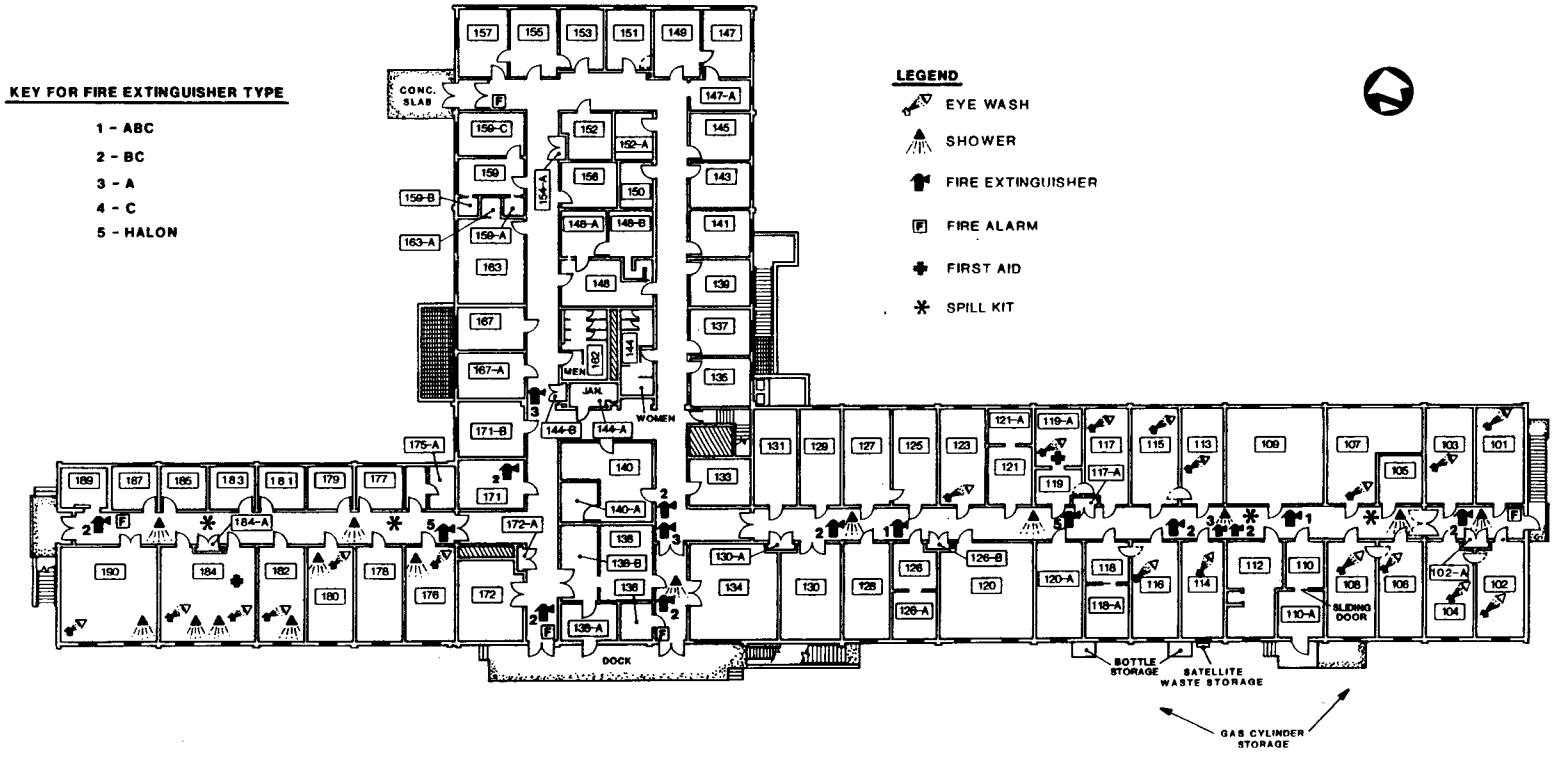


Fig. 2.2. TA-59, Bldg. OH-1, 1st Floor

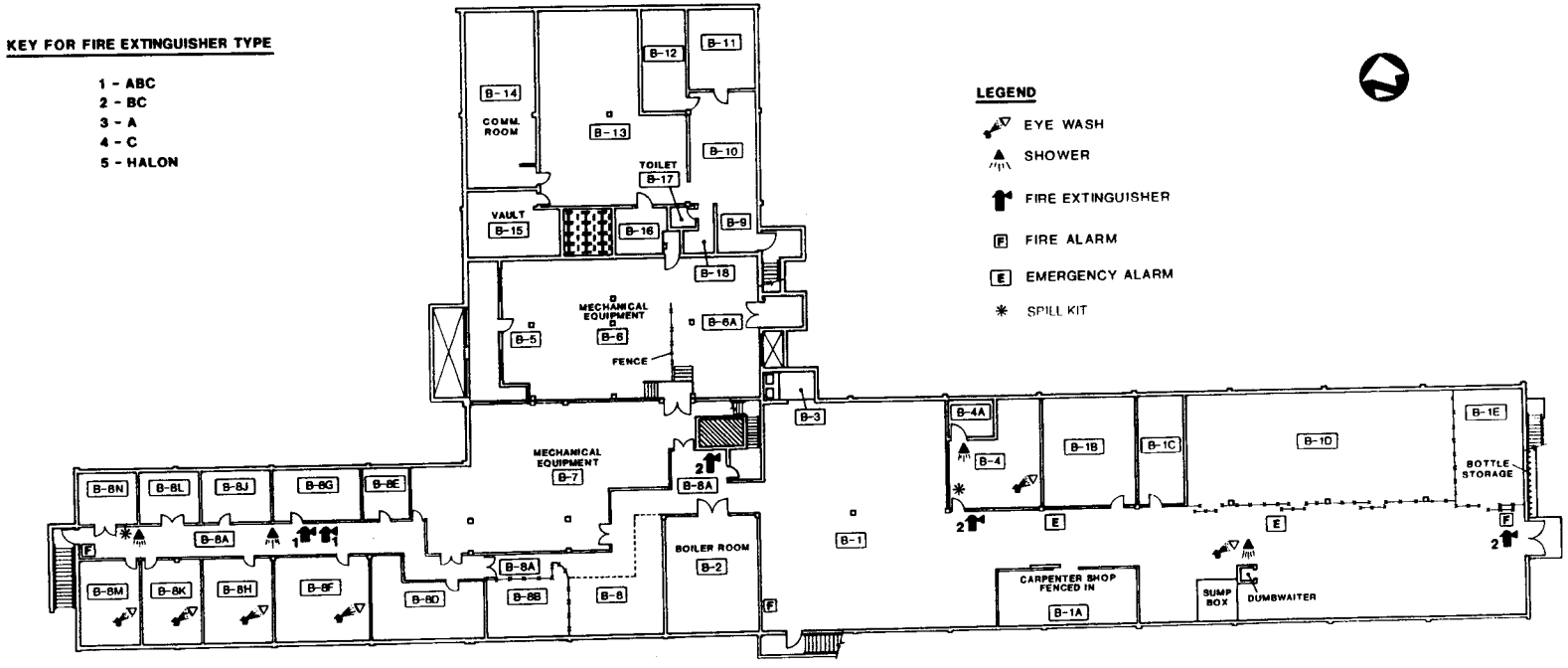


Fig. 2.3. TA-59, Bldg. OH-1, Basement

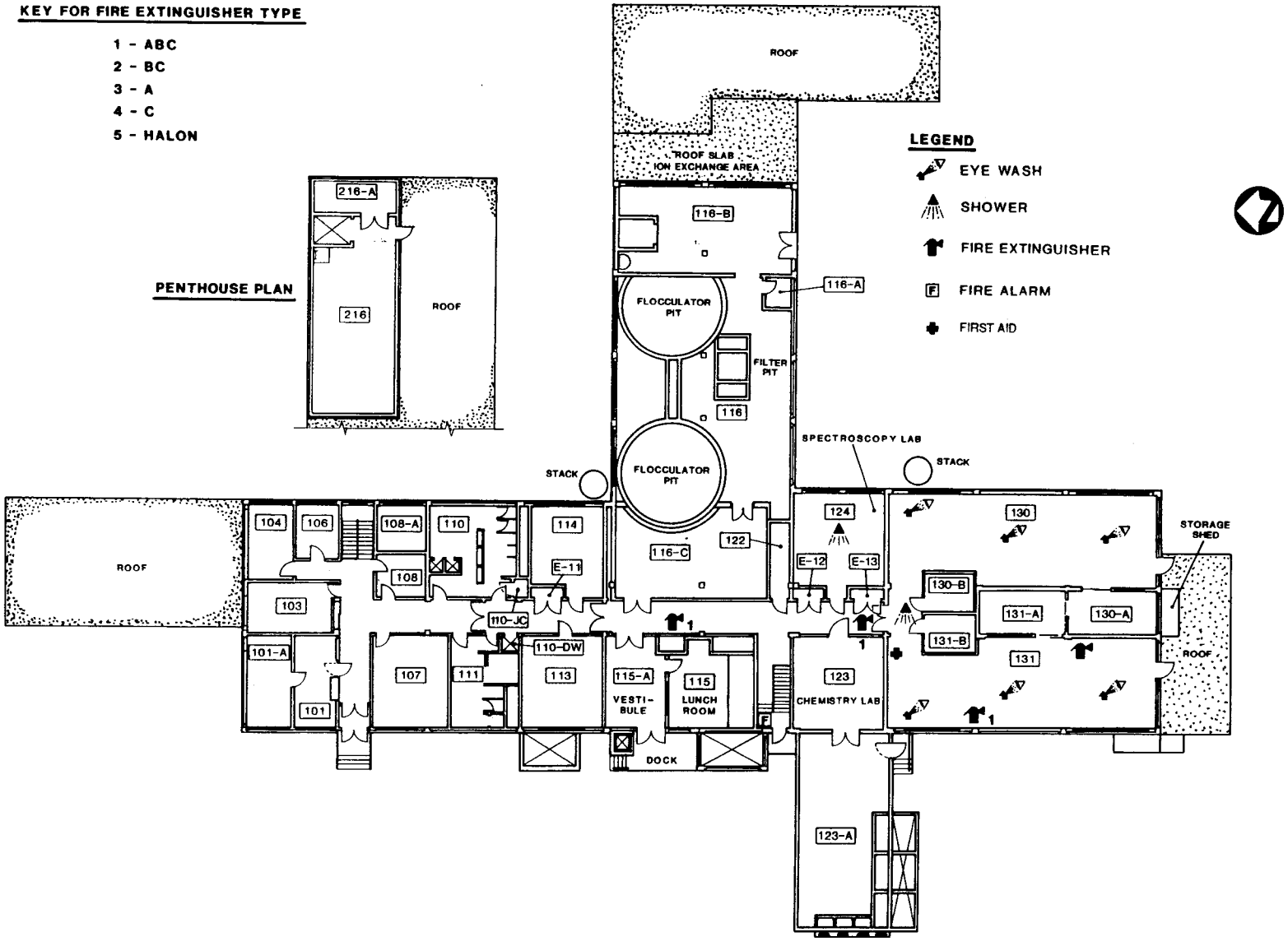


Fig. 2.4. TA-50, Bldg. LD-1, 1st Floor

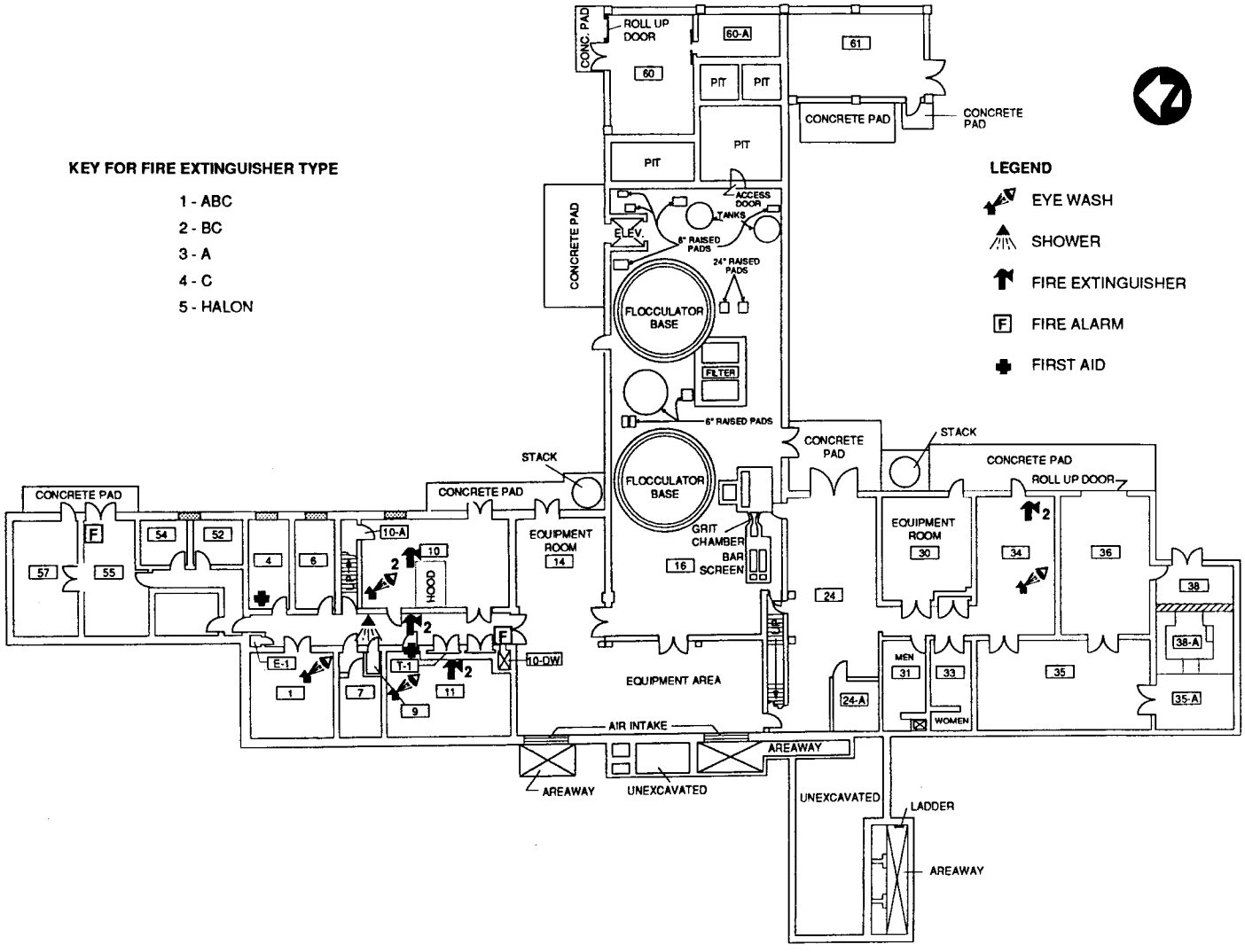
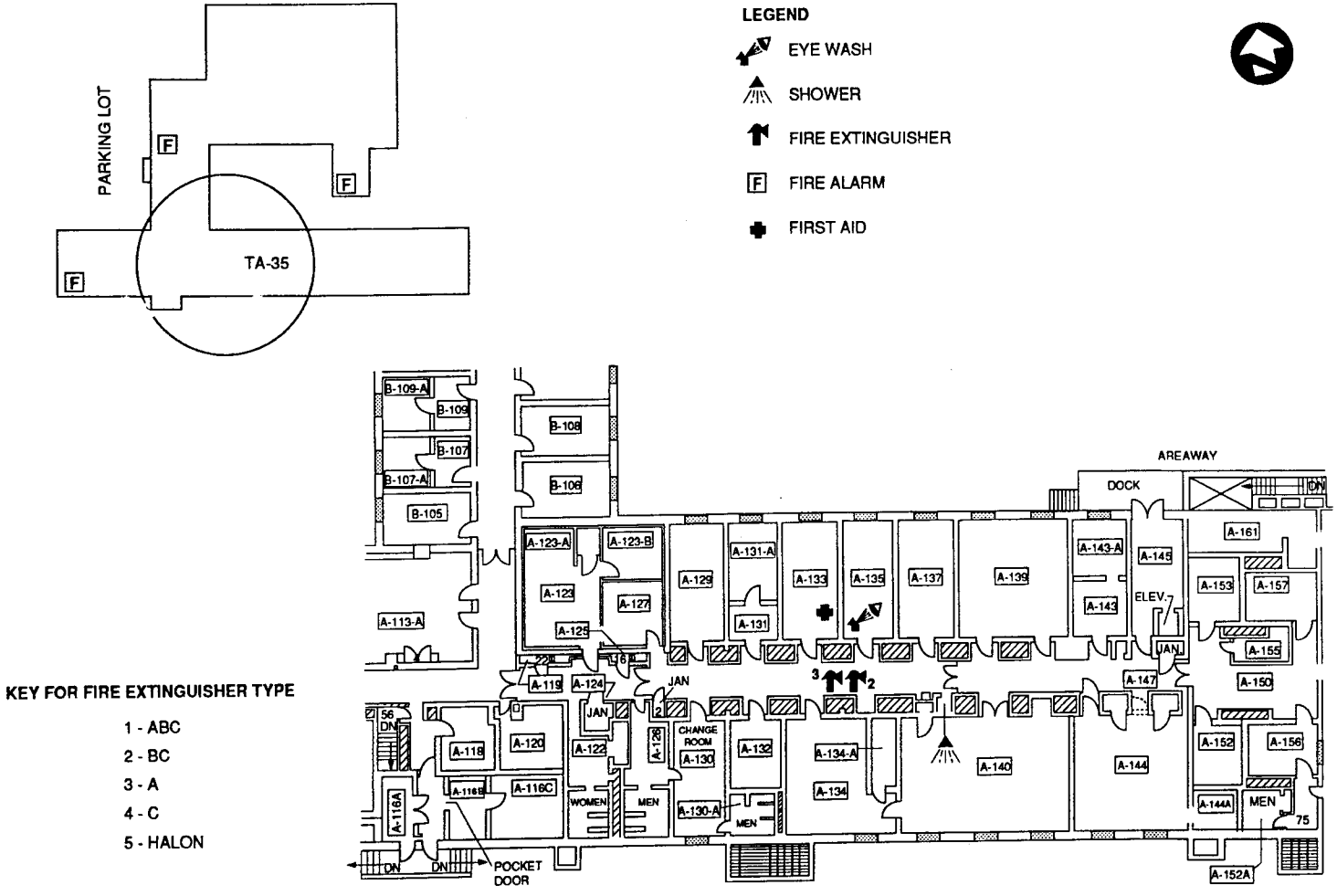


Fig. 2.5. TA-50, Bldg. LD-1, Basement



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3. EMERGENCY RESPONSE

3.1. Introduction

The Emergency Coordinator should immediately assume command and maintain order by establishing a command post near the center of the action, safely removed from any real danger, and directing the emergency response. In HSE-9, the Group Leader or his designee is the Emergency Coordinator. Further responsibility is shown in the chain of command shown in Fig. 2.1. of Section 2.

Other supervisory personnel, both operational and support (i.e., HSE-1, HSE-3, HSE-5, etc.) will work closely with the person in charge to provide advice and to assure that all decisions are carried out. The person in charge will coordinate all activities, will know what has already been done and what needs to be done, and will make any final decisions.

3.2. Emergency Site Plans for TA-59, TA-50, TA-35, and TA-2 Omega Site

Group HSE-9 is located at TA-59, TA-50, and TA-35. The emergency site plans for each of these technical areas are reproduced in full in Appendix I of this manual. The technical area site plans are designed to provide maximum feasible protection of Laboratory personnel, the general public, and property should an emergency occur at these sites.

A few HSE-9 employees perform irradiation work at the Omega West Reactor. The emergency procedures for that site are included in Appendix I for HSE-9 personnel who work at that facility.

3.3. Basic Guidelines for Emergency Response in HSE-9

3.3.1. Be familiar with the work area, the types of work in progress, and the location of potentially hazardous materials. Know the emergency plans and the emergency equipment.

3.3.2. When an emergency arises, remain calm and think about actions to take. While rapid and effective action is frequently imperative, hasty action before consideration can compound difficulties.

3.3.3. The first concern is for personnel. Personnel will be evacuated to an area where known hazards are low. Obtain medical assistance for any injured personnel.

3.3.4. Re-entry should not be attempted until the situation has been carefully evaluated.

3.3.5. In extreme cases, entry into an area may be necessary to remove injured personnel from life-threatening conditions. If any possibility of neck or back injury exists, move the victim *only* in life-threatening situations.

3.3.6. All emergencies should be reported to appropriate HSE-9 supervision. After normal working hours, all emergencies should be reported to the

Group Leader. For general assistance involving all types of emergency situations, call the Laboratory emergency number 911. Indicate to which entrance to the building (main, west, south, east) the response team should report. The HSE Division emergency response number is 7-7878.

3.3.7. As public dissemination of information about an emergency can cause unnecessary alarm, please refer all inquiries to the Public Information Office at 7-7000.

3.4. Fire Alarm Response

3.4.1. Immediate Response

Locate and extinguish all fires as rapidly as possible before they get out of control. If a person finds a fire in their area, he or she should immediately alert the Fire Department by activating a fire alarm and/or by calling 911. The person calling should give the Fire Department the vital information (your name, where, what, how big) and stay on the line until the Fire Department breaks the connection.

When a fire alarm sounds, all personnel in the alarmed area should evacuate the building, report to the assigned muster area, and await further instructions.

When the Fire Department arrives following a fire alarm, they automatically assume responsibility for the fire fighting. Supervisory personnel will work closely with the Fire Department to advise them of facility considerations.

3.4.2. General Fire Fighting Procedures

Fire-alarm pull boxes are located at EXIT doors at TA-50, TA-35, and in OH-1 at all EXIT doors with the exception of the south EXIT through room 110 in OH-1. See Figs. 2.2. through 2.6.

If a fire is found, evaluate it. An attempt to extinguish the fire may be made only if it can be done safely. Select the proper fire extinquisher for the type of fire being controlled (see 3.4.3.). If you cannot put out the fire with a single extinguisher, immediately summon the fire department.

As a general rule, cool the area immediately surrounding the fire with a fire extinguisher to prevent the flames from spreading. The base of the blaze should then be extinguished. Remove flammable materials from around the fire and unplug any electrical equipment in the area.

Abandon the fire if it cannot be safely extinguished because of chemical or other considerations and leave the fire fighting to professional fire fighters.

If your clothing should catch fire, smother the flames promptly with a laboratory coat or lie down on the floor and roll. If you are close to a safety shower, use the shower. *Do not run.* Running will fan the flames.

If you use a fire extinguisher, call ENG-10 at 7-2295 to recharge the fire extinguisher. *Do not rehang a used fire extinguisher.*

3.4.3. Types of Fire Extinguishers

Fire extinguishers are located in all corridors of OH-1 and in the laboratory at TA-50. Most fire extinguishers are Type BC for flammable liquids and/or electrical fire. Each extinguisher is labeled on the canister for easy identification. Locate the fire extinguishers near your work area and note which type they are.

The fire extinguishers used in HSE-9 are as follows:

CLASS	WHERE USED
A	Ordinary combustibles, such as paper, wood, upholstery, drapes.
B	Flammable liquids, such as fuel oil, gasoline, paint, grease, solvents.
C	Electrical equipment, wiring, fuse boxes, conductors.
D	Metals, such as magnesium, aluminum, zinc, lithium, sodium, potassium, plutonium, or metal hydrides.

3.5. Evacuation Alarm Response

3.5.1. Equipment Shutdown for Evacuation

- Do not remain to shut down equipment when immediate building evacuation has been called for.
- Shut down equipment such as the ICPMS, ICPAES, and AAS.
- Shut off all gases (e.g., nitrous oxide torch of FAA) except inert purging gases.
- Shut down extraction apparatus and turn off hot plates.
- Power down computer-controlled robots.
- Turn off all high voltage equipment, computers, counting systems, and other equipment that might cause injury to fire fighters and emergency personnel or might be damaged by power failure or unattended operation.
- Shut down analyses using toxic or radioactive materials.

- Shut office and laboratory doors.

3.5.2. Building Evacuation

Immediately evacuate the area using the most direct route to the assembly (muster) areas shown in Fig. 3.1.

All personnel should remain at their assembly area until dismissed by the Emergency Coordinator. The group secretary will provide the Emergency Coordinator with an attendance list to be used for personnel accountability.

3.5.3. Emergency Control Center

Following evacuation, a control center will be established. The Emergency Coordinator will direct all emergency response from this center. The HSE Division Office maintains an Emergency Operations Center (EOC) in building OH-3, room 313. An alternate location is also available in the OH-1 Meterology Laboratory. The Los Alamos National Laboratory Emergency Operations Center can also be used when circumstances warrant its use.

3.5.4. Emergency Response Team Action

During an emergency evacuation, Emergency Response Team members should proceed immediately to the assembly area to be counted and then to the emergency control center for additional instructions.

3.6. Emergency Situations

The principle areas of concern for HSE-9 are the chemical laboratories. Building OH-1 also contains the Laboratory's Emergency Operations Center, a potential target for sabotage and terrorism.

3.6.1. Vehicle Accident

In case of a vehicle accident call 911. If there is threat of fire, call 911 before attempting to extinguish the fire. For other emergency procedures, such as rescuing the personnel, notify HSE-DO at 7-7878.

3.6.2. Toxic Releases

In the event of serious release of toxic materials to the environment, immediately evacuate the area. Call 911 and HSE-DO at 7-7878. In the event of chemical exposure call HSE-5 at 7-5231. In the event of a chemical spill call HSE-7 at 7-4301. Place individuals at a sufficient number of safe locations to control entry into the area. Immediately notify the Emergency Coordinator, then your section supervisor.

The Emergency Response Team will provide first aid and call for medical assistance as needed. Place personnel near the main entrance to guide

emergency personnel to exact locations. The cleanup of hazardous materials requires special procedures and is directed by HSE-5 and HSE-7.

3.6.3. Fire and Explosion

If the Fire Department is summoned by pulling a fire alarm box, call the dispatcher at 911 to provide additional information on the location and nature of the alarm. Then call HSE-DO at 7-7878.

In the case of explosion, immediately evacuate the area and call 911. Depending on the severity of the situation, attempt to rescue trapped personnel. The Emergency Response Team will administer first aid to the injured. Re-entry into the area for rescue must be determined by the extent of structural damage and any associated fire.

Place personnel near the main parking lot entrance to guide fire fighters and ambulances to the location of the fire or any injuries.

3.6.4. Natural Disasters

The primary result of natural disasters, such as heavy winter snow storms or forest fires, would be structural damage to buildings and equipment. In these cases, personnel should be evacuated from the damaged structure and electrical power and equipment shut down to minimize the danger of fire. Call 911 and HSE-DO at 7-7878.

3.6.5. Occupational or Industrial Accidents

In the case of an accident injuring personnel, emergency first aid should be administered by the Emergency Response Team. If the injuries are extensive, an ambulance should be summoned by calling 911. Notify HSE-DO at 7-7878. Place personnel near the main parking lot entrance to guide the ambulance to the location of any injured personnel. All injuries to personnel must be reported to the Occupational Medicine Group (HSE-2) at 7-7251.

3.6.6. Radioactive Material Incidents

The possibility of radiological incidents arising from HSE-9 laboratory operations at TA-35 and TA-59 is small. There is a higher chance of this type of incident happening at TA-50.

If it is suspected that radioactive materials have been released into any area, immediately call 7-7137. Health physics personnel from HSE-1 will respond and they will determine the level of radiation. Operations personnel should determine the exact nature of the incident.

Personnel should be evacuated from the immediate area to a nearby designated area until they have been checked by health physics personnel for contamination. Minimize the spread of contamination by establishing exclusion zones around the area. Call HSE-1 at 7-5296 to monitor the area

and to determine the level and extent of the contamination. A plan for decontamination can then be formulated.

3.6.7. Utility Service Failures

Shut down all operations requiring mechanical ventilation. Shut down electrical equipment such as computers, radiological counting equipment, mass spectrometers, and any other equipment that might cause injury to personnel or be damaged by unattended operation or power surges.

3.6.8. Medical Illness

In the event of an illness or serious injury, the Emergency Response Team should immediately administer first aid. Summon additional help by calling 911. Station someone at the main entrance of the site to direct the ambulance when it arrives.

3.6.9. Terrorist Attack

Call 911. The Protective Force Duty Officer must be notified, the site buildings secured, and the staff notified of the situation. Stay away from windows. Physical security will be provided by the Protective Force. Notify HSE-DO at 7-7878.

3.6.10. Bomb Threat

Use the checklist on page one of the Laboratory Telephone Directory. The page is also reproduced in the front of this manual. Call 911 and HSE-DO at 7-7878.

3.7. Emergency Response after Work Hours

When emergencies occur after normal working hours, the primary concerns are (1) assuring that all persons are properly evacuated from potentially hazardous areas and (2) contacting the appropriate HSE-9 supervisory personnel to handle the situation.

3.8. HSE-9 Response within the HSE-Division Emergency Operations Plan

3.8.1. Responsibilities

In case of an emergency, the Health and Environmental Chemistry Group will

- advise on types of samples to be taken,
- analyze for chemicals in the environment, in the workplace, or in waste samples, and
- interpret results.

In the HSE-Division Emergency Operations Plan, HSE-9 is listed as potentially being called for analytical services for the following incidents:

- hazardous solid or liquid releases to the environment,
- radiation/radioactive material incidents,
- toxic material (nonradioactive) release to the atmosphere,
- toxic or potentially toxic material release (nonradioactive) in buildings,
- vehicle acidents and,
- handling of deceased persons.

3.8.2. Chemical Analyses

Chemical analysis of samples will be conducted as quickly as possible to provide data for management decisions. The time required to obtain data varies greatly, depending on the type of analyte and matrix and on the sensitivity and precision required. Immediate estimates are often acceptable, but are usually followed with more precise results. Identification of unknowns usually requires more effort and time than quantification of known compounds or elements.

3.8.3. Personnel Resources

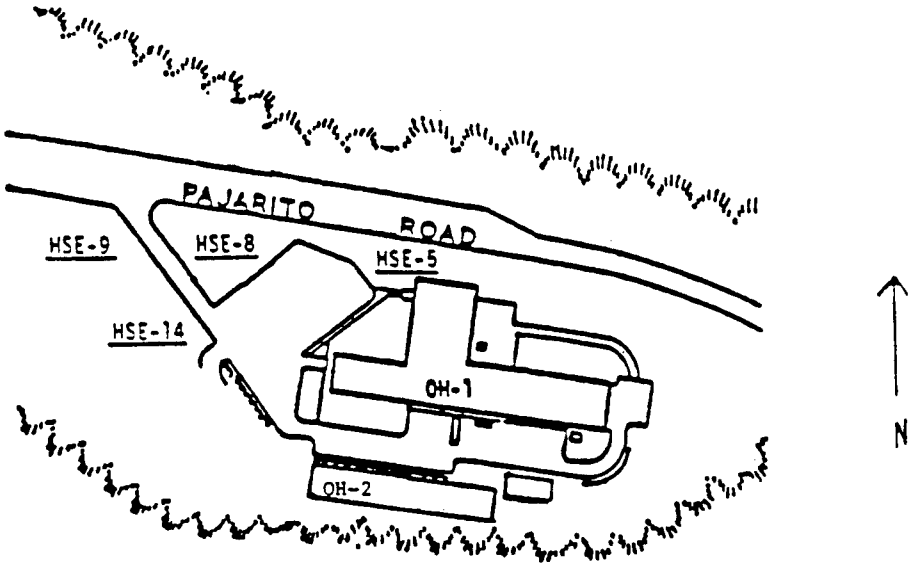
Group HSE-9 has chemistry staff members with expertise in the following areas:

- Organic Chemistry - 7 staff members
- Inorganic Chemistry - 6 staff members
- Radiochemistry - 6 staff members

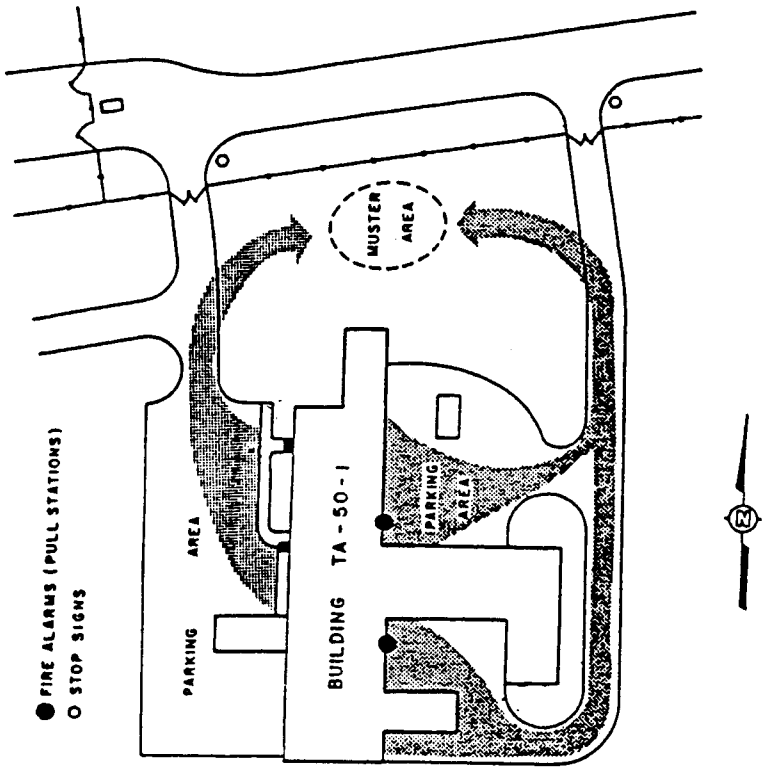
In addition 19 full-time and 5 part-time HSE-9 technicians have experience in these areas.

3.8.4. Equipment Resources

Table I. lists HSE-9 instruments available for emergency use.

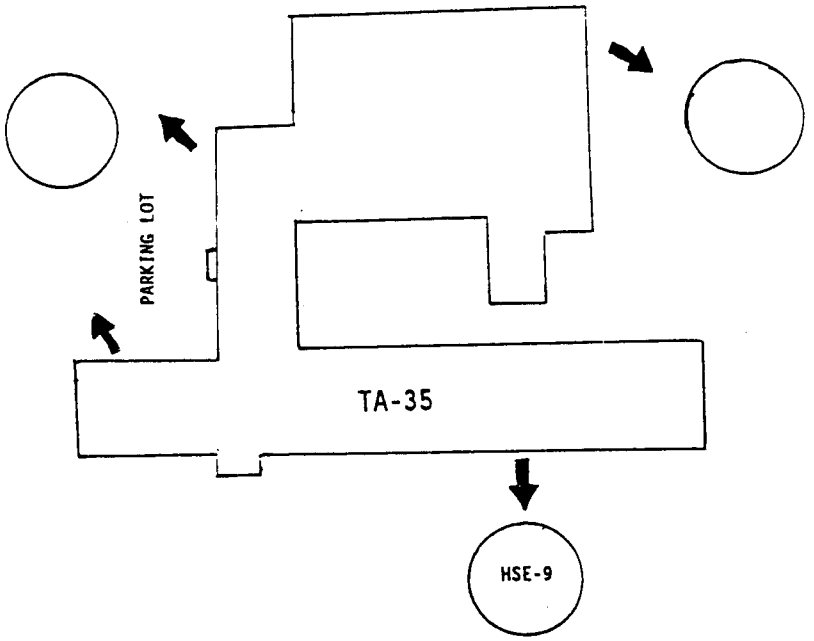


OH-1 Evacuation Assembly Area

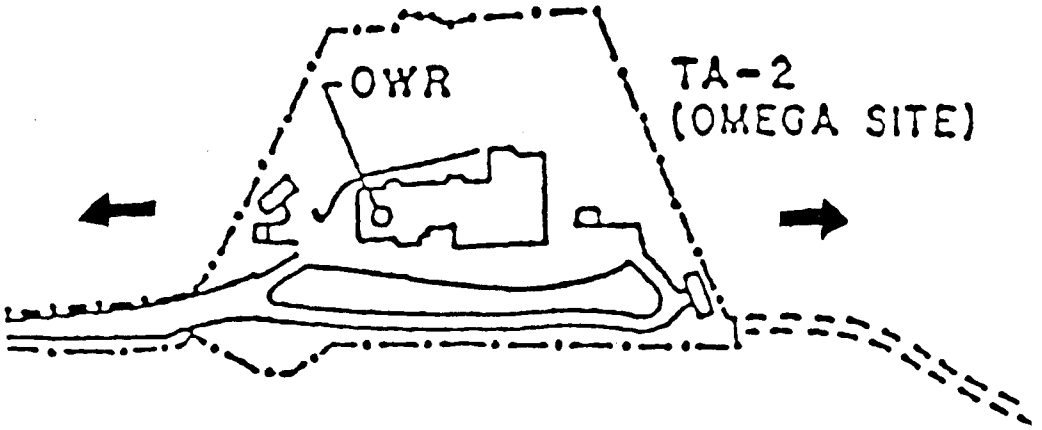


TA-50 Evacuation Assembly Area

Fig. 3.1. Building evacuation assembly areas.



TA-35 Evacuation Assembly Area



TA-2 Evacuation Assembly Area

Fig. 3.1. Building evacuation assembly areas (cont).

TABLE I. HSE-9 INSTRUMENTS AVAILABLE FOR EMERGENCY USE

Instrument	Number	Common Uses	Comments
Organic Analysis:			
• Gas chromatograph/ mass spectrometer (GC/MS)	5	Identification and quantification of unknown organics	Data system and extensive reference library
• Gas chromatographs	4	Quantitative analysis for most organics 500 amu or less in molecular weight	Various general and specific detectors
• Infrared spectrometers	2	Identification and quantitation of unknown organics	FTIR has a data system with reference library
• Ultraviolet/visible spectrometers	1	Identification and quantification of a variety of organic compounds	
• High performance liquid chromatograph	1	Quantitation of less volatile organics or derivatives	
Inorganic Analysis:			
• Atomic absorption spectrometers	4	Quantitative analysis for many elements	One element at a time; sample preparation required
• Omega West reactor	1	Neutron activation analysis for many elements	Used for uranium in urine, air, water, etc.
• Mercury analyzers	3	Mercury	
• Ion Chromatographs	3	Ions; acid or inorganic mists	
• Ultraviolet/visible spectrometers and colorimetric auto-analyzer	4	Quantitative analyses for selected aqueous ions	

TABLE I. (cont)

Instrument	Number	Common Uses	Comments
Inorganic Analysis (cont):			
• ICPMS	2	Quantitative analyses for many elements	Simultaneous multi-element
• ICPES	1	Quantitative analysis for many elements	Single element
Radiochemical Analysis:			
• Alpha spectrometer systems (112 detectors)	7	Pu, Am, U and Th in a variety of matrices	Considerable sample preparation required
• Liquid scintillation spectrometers	3	Tritium	
• Gamma spectrometers	6	Neutron activation analyses for a variety of elements	Facilities also available at Omega West Reactor
• Alpha, beta, and gamma counters	Many	Direct sample	

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4. PERSONNEL SAFETY

4.1. Introduction

It is the intent of Los Alamos National Laboratory, the Health Safety and Environment Division, and the Health and Environmental Chemistry Group (HSE-9) to provide a workplace that is reasonably free of hazards. This goal can be reached only through the cooperation of the Laboratory and its employees. The Laboratory shall control and limit known hazards to an acceptable level. Employees have the professional, moral, and legal responsibility to be as familiar as possible with the hazards of the workplace, to follow all rules and regulations that apply, and to bring to the attention of their supervisor any situation or condition they feel is hazardous to the health and safety of employees.

Many technical undertakings can be hazardous when performed without adequate knowledge or understanding. Accidents can be minimized by emphasizing responsibility, knowledge, and safety at both the individual and group levels.

Although it is not possible to define generalized safety practices in a research and service environment, all employees must be observant and identify new hazards. As these hazards are identified, they must be codified into new safety regulations. Work must be undertaken with forethought. We must become aware of our fellow workers, particularly those with less experience, to see that they develop safe working habits and are not injured through ignorance of the hazards involved with a particular operation.

Supervisors must be sure that employees use safe procedures and techniques. Administrators are ultimately responsible for safe operations in HSE-9. These supervisory and administrative responsibilities do not lessen the employee's obligation to work safely.

4.2. Responsibility for Safety

4.2.1. Individual Employee Responsibilities

In conducting their activities, employees must accept and exercise certain health and safety responsibilities. These responsibilities, taken from the Los Alamos Administrative Manual, Section 700, are listed below. Failure to comply with health and safety regulations may result in disciplinary action, up to and including termination.

- Conduct only those activities that your supervisors have approved, and use Laboratory facilities, equipment, and tools for the purposes for which they were designed.

- Adhere to any standard operating procedures associated with your work assignments.

- Observe health and safety requirements, instructions, signs, posters, and warning signals, as applicable to your work place.

- Follow any health and safety instructions from your supervisors.

- Know the emergency plans and procedures established for your work areas. (HSE-9 is located in Building OH-1 at TA-59, in Building 1 at TA-50, and in Building TSL-2 at TA-35).

- Become familiar with potential hazards associated with your operations and work areas.

- Use the personal protective equipment provided or recommended by your supervisor for the tasks or operations being performed.

- Report all unsafe conditions and potentially hazardous incidents, including but not limited to the following, as soon as possible:

  (1) Any malfunctioning equipment or devices. Post signs until such time as the equipment or devices are repaired or removed from service.

  (2) Any condition or act believed to be a threat to the health and safety of employees.

  (3) All injuries, no matter how small, and any incident that causes damage.

  (4) Any incident that could cause damage to property or facilities or that could result in bodily injury.

- Participate in bioassay and dosimetry monitoring programs when appropriate.

In addition, HSE-9 employees have the following responsibilities:

- Study, learn, and understand procedures and policies set forth in the HSE-9 Safety Manual, in section and group safety meetings, and in other pertinent Laboratory publications. The intent is to develop and practice a safety attitude and awareness. Ask your supervisors or co-workers if you have any questions regarding any aspect of safety in HSE-9.

- Wear laboratory coats when working in the laboratory.

- Safety glasses should be worn at all times when working in the laboratory and worn in other locations when warranted.

- Wear radiation dosimeters if deemed necessary.

- Learn the locations in your areas for safety-related items, such as fire extinguishers, fire pull-boxes, eye washes, and showers. Learn the procedures to follow in case of emergencies.

- Do not bring food, drinks, tobacco, or other materials for personal consumption into laboratory working areas. Food storage in refrigerators not specifically approved for food is prohibited.

- Know your physical limitations and do not exceed them. Ask for help in lifting or moving heavy objects.

- Good housekeeping practices contribute to a safe working environment. Laboratories must be kept neat and clean.

4.2.2. Supervisory Personnel Responsibilities

The implementation and management of health and safety functions are line responsibilities. Supervisory personnel must follow all responsibilities outlined in Section 4.2.1. and must ensure that the above individual responsibilities are discharged by personnel under their supervision. Supervisors have the following additional responsibilities.

- Set the level of awareness and safety-conscious behavior expected of their employees through supervisory interest, involvement, and attitude.

- Teach employees safety techniques and approved safety practices.

- Verify that standard operating procedures are written and kept current.

- Furnish job and safety information to HSE-DO concerning individuals and activities in their areas.

- Contact the approving authority whenever major changes in operational procedures, new techniques, alterations in the building, or new operations are anticipated.

- Eliminate unsafe operating conditions and practices.

- Conduct only those operations and activities that can be safely controlled.

- Verify that employees understand and implement policy, rules, regulations, and procedures.

- Terminate any activity in their area of responsibility that is unsafe or has excessive risk.

- Take time to actively listen to employees who are preoccupied because of personal problems, and when necessary, assign alternate work when the health and safety of employees is being jeopardized.

4.2.3. Group Leader Responsibilities

Although the details of implementing various functions of the health and safety program can be delegated, the group leader has the following basic responsibilities.

- Outline management's goals, objectives, and priorities regarding an effective health and safety program.
- Ensure that group operations have a complete and effective safety program.
- Demonstrate a positive attitude and a commitment to health and safety.
- Document the entire program with responsibilities, authority, and lines of communication clearly stated.
- Participate actively in the safety committee.
- Provide the appropriate level of comprehensive and job-specific training to each employee.
- Maintain a record system that includes records of safety training, inspections, safety meetings, equipment maintenance, SOPs, SWPs, and emergency drills.

4.2.4. External Support Organizations

Safety support services are available from the Health, Safety, and Environment Division. Group health and safety representatives are encouraged to use the following support services as needed. The telephone numbers for these organizations are listed, and the services they offer are briefly described below.

- The health physics staff (HSE-1, 7-5296) provides many services, including advice and monitoring for the safe handling of radioactive materials and the safe operation of radiation-generating equipment.
- The Occupational Medicine Group (HSE-2, 7-7251) provides first-aid treatment of minor injuries and illnesses and assistance in the diagnosis and control of occupational illness.
- The Safety Group (HSE-3, 7-4644) consults in safety matters, accident investigations, and health and safety training, and also assists in developing standard operating procedures.
- The Industrial Hygiene Group (HSE-5, 7-5231) monitors workplaces for toxic materials, recommends control measures, and advises on the use of personal protective equipment, issues concerning hazardous substances and agents, and standard operating procedures.

- The Criticality Safety Group (HSE-6, 7-4789) provides support regarding nuclear criticality safety.

- The Waste Management Group (HSE-7, 7-4301) provides advice, services, and facilities for the disposal of hazardous and radioactive materials.

- The Environmental Protection Group (HSE-8, 7-5021) provides climatological and meteorological data and observation, as requested, and sampling of environmental pollutants.

- Our group, Health and Environmental Chemistry (HSE-9, 7-3269), conducts an ongoing program of chemical measurements in support of both monitoring and research programs within HSE Division and, where appropriate, the Laboratory. These chemical measurements are monitored by a quality control program with National Bureau of Standards traceability where possible.

4.2.5. Accident Reporting

All accidents should be reported. Accidents causing injury should be reported to your section leader and group leader even if the injury appears insignificant at the time.

4.3. Personal Protective Equipment

A large variety of personal protective equipment is available to the employee, either from stock or by special order. All employees are to be familiar with what is available, and how, when, and why to use it.

4.3.1. Eye Protection

Eye protection is of exceptional importance. *Employees wearing contact lenses should inform their supervisors that they wear contacts.* This information could be very important in emergency situations. Workers must always wear suitable eye protection when they are working in a laboratory. All visitors in a laboratory where work is being performed must wear safety glasses regardless of the duration of their visit. Visitor safety glasses are available from dispensers located in hallways outside our laboratories.

The only exception to the above rules is that eye protection is not necessary when working at a desk when no laboratory work is being performed in the immediate vicinity. The term experimental work includes, but is not limited to, using hot plates and instruments that are operating unattended.

Contact lenses are strongly discouraged and are never to be considered as eye protection. They do not protect the eye from contact with flying dust or droplets; they can create visual problems by sudden displacement of the lenses; they tend to trap and concentrate fumes and droplets between the lens and the eye; they can be difficult to remove under stress conditions

when something gets into the eye and delay removal of the foreign material; and they retard or inhibit the natural eye fluids from flushing contaminants from the eye.

Eye glasses with side shields provide more protection than regular eye glasses. Various types of face shields are also available and should be worn when performing especially hazardous operations, such as mixing acids, or when the potential of an explosion is great. The use of a safety shield in conjunction with eye protection is mandatory whenever there is potential for an explosion.

Nonprescription safety glasses are available from stock. Only glasses meeting the specifications of the American National Standards Institute (ANSI) are acceptable at the Laboratory. Visitors must also wear glasses meeting ANSI standards. A recommended model is the UVEX Ultra-Spec 2000.

Prescription safety glasses will be purchased by the Laboratory with group leader approval. Fill out an Authorization and Invoice for Prescription Safety Glasses, have your group leader sign it, and present the form plus your prescription to the local approved source. These glasses are more impact resistant than those generally purchased by the public and meet the ANSI standard for safety glasses.

If your glasses need repair, have them repaired as soon as possible. Replace scratched lenses because they lose impact resistance.

4.3.2. Hand Protection

Heavy fiberglass gloves must be worn when handling hot objects. Plastic or rubber gloves must be worn when handling hazardous and radioactive chemicals. Penetration of chemicals through gloves is possible. Consult the reports from the Industrial Hygiene Group (HSE-5) glove permeation projects or ask the industrial hygienist assigned to HSE-9 before deciding which glove material is suitable for a given chemical.

4.3.3. Foot Protection

Bare feet, sandals, and open-toed shoes are not permitted while working in the laboratory. Although allowed, soft shoes, such as tennis or jogging shoes, are discouraged. A heavy object dropped on the foot can cause serious injury, and spilled acids can readily penetrate the fabric, causing injury. Regular street shoes are the minimum recommended footware. The Laboratory will provide safety shoes with built-in steel toe caps for employees when necessary. Group leader approval is necessary to obtain these shoes.

4.3.4. Protective Clothing

Laboratory coats or aprons must be worn and must be buttoned when working in the laboratory. While this will minimize the danger of damage

to clothing, it will also prevent contaminating clothing with toxic or otherwise harmful chemicals that may later cause injury either by contact or by contact and subsequent ingestion. TA-50 personnel will wear "Anti-C" laboratory coats. If acid is spilled on clothing, immediately remove the clothing and rinse the affected area for at least 15 minutes with large volumes of water.

4.3.5. Respiratory Protection

Respiratory protection is normally not needed in HSE-9. For emergencies or abnormal circumstances, contact the Industrial Hygiene Group at 7-5231.

4.3.6. Radiation Monitoring

Radiation Protection provides thermal luminescent dosimeters (TLDs) to employees who work in areas in which the potential for external radiation exposure is in excess of 10% of the prescribed annual standards for radiation workers. This standard is three rem per calendar quarter or five rem per year. Consult Administrative Requirements, section three of the Health and Safety Chapter of the Los Alamos Administrative Manual and Appendix III of this manual for more information. TLDs are required at TA-50 and at the Omega West Reactor (TA-2).

The TLD must be worn at all times while at work. They may be worn when leaving for lunch and should be taken home after work hours to avoid recording data while the employee is not present in the work place. Avoid exposing TLDs to television sets, microwaves, etc. The TLD must not be worn when visiting a doctor or dentist during work hours as a nonoccupational exposure will be recorded, particularly if x-rays are taken.

4.4. Substance Abuse

Work assignments in HSE-9 require concentration and judgment. All employees must be drug- and alcohol-free when at work. Your safety and the safety of those around you depend on it.

4.4.1. Policy

The Laboratory is committed to a safe work environment for its employees, to public safety, and to protection of its national security mission. *Substance abuse in the workplace or while on Laboratory business is prohibited and can result in disciplinary action, up to and including termination.*

Substance abuse is defined as the use of controlled substances that are identified in Schedules I through V of the Controlled Substances Act where the use is neither authorized by law nor a valid prescription, or the misuse of a legal substance, including but not limited to alcohol or prescription drugs, that may affect an individual's ability to perform his or her job in a safe and secure manner.

The unauthorized manufacture, distribution, dispensation, possession, or transfer of controlled substances on Laboratory premises also constitutes a violation of this policy; such violation may result in disciplinary action up to and including termination.

The unauthorized use or possession of alcohol or alcoholic beverages on Laboratory premises is prohibited.

Legal drugs, so long as these drugs do not adversely affect the employee's ability to perform required work in a safe and secure manner, may be used on the work site. When such legal drugs are to be used at the work site, *employees must inform their supervisors* either directly or through the Occupational Medicine Group.

4.4.2. Employee Assistance

The Laboratory strongly encourages employees who suffer any form of substance abuse, including alcohol or other drug-related problems, to voluntarily refer themselves for assistance. Laboratory management and all organizations at all levels will provide support for employee assistance, rehabilitation, and counseling.

4.4.3. Fitness for Duty

Supervisors will continue to observe employee performance and on-the-job conduct to detect behavior that could compromise the security, health, or safety of the employee or others. When that observation indicates cause to believe that substance abuse is a factor, *supervisors are to take appropriate management action as directed in the Laboratory policy.*

4.4.4. Convictions for Substance Abuse Violations

Employees are *required*, as a condition of employment, to notify their managers (Group Leaders or above) of any criminal drug statute conviction for a violation no later than five days after such a conviction.

4.4.5. Policy Violations

All employees are expected to cooperate fully with the Laboratory's objective of maintaining a Drug Free Workplace. Failure to do so is deemed to be a violation of Laboratory policy. Accordingly, in the event that an employee violates this policy, refuses to submit to testing as directed, refuses a formal offer of rehabilitation, fails to complete a prescribed rehabilitation program, falsifies any record relative to substance abuse, fails to report an arrest or conviction for substance-abuse violations as required by this policy, or is found to have submitted an adulterated or fraudulent urine sample when tested, such employee will be subject to disciplinary action up to and including termination.

4.5. Facilities and Equipment Management

Laboratory facilities and equipment must be designed, constructed, modified, operated, maintained, and disposed of in accordance with applicable safety standards.

4.5.1. Class A Facilities and Equipment

Laboratory Class A facilities and equipment comprise the land and all permanent or substantial objects affixed to it (for example, buildings, fences, elevators, heating systems, plumbing, and trees).

The Facilities Engineering (ENG) Division is responsible for the design, construction, maintenance, operation, and disposal of Class A facilities and equipment. In addition, the line management of each operating group shall conduct reviews, such as surveys, inspections, or design reviews, to verify that these facilities and equipment do not adversely affect the safety of their personnel or the continuity of their operations.

4.5.2. Class B Facilities and Equipment

Laboratory Class B facilities and equipment are generally movable items purchased by an operating group for use in a particular operation or experiment (for example, glove boxes and vacuum pumps) or for everyday staff use (for example, microwave ovens and refrigerators).

The line management of each operating group to which the systems are assigned is responsible for the design, construction, maintenance, operation, and disposal of Class B facilities and equipment. They are responsible for conducting reviews, such as surveys or inspections, to verify that these facilities and equipment do not adversely affect the safety of their personnel or the continuity of their operations.

4.5.3. Government Vehicles

An official vehicle is any vehicle used for official business, including government-owned vehicles, taxis, rental vehicles, and personal automobiles.

4.5.3.1. Seat Belts

Administrative Requirement 12-2 of the Laboratory Health and Safety Manual specifies the need for seat belt use at the Laboratory. *Laboratory employees must wear seat belts, if provided, when driving or riding in any motor vehicle on official business.* Laboratory drivers must not start a vehicle until all passengers have fastened their seat belts. Supervisors must ensure that their employees are aware of this requirement.

4.5.3.2. Two-Wheel Vehicles

You may use bicycles but not motorcycles for official purposes at the Laboratory. This includes official errands while an employee is on duty, such as, traveling from one Laboratory building to another for obtaining materials, attending meetings, and performing work.

4.5.3.3. Vehicle Accident

Employees must report any accident involving a General Services Administration (GSA)-owned vehicle to GSA by telephone within 24 hours. See AR 1-1 of the Laboratory Health and Safety Manual for additional information. Local police must be notified immediately if the accident involves a personal injury or if it occurs offsite.

4.5.3.4. Vehicle Inspection

HSE-9 policy requires that vehicles assigned to the group be inspected biannually. An HSE-9 employee is assigned this responsibility and the vehicle inspection form is shown on p. LS 4-17.

4.6. Risk Management

Risk management identifies and evaluates deficiencies and hazards that can have adverse consequences in relation to their probability of occurrence and the severity of consequences. Risks are determined to be acceptable or unacceptable. If unacceptable, controls are applied to eliminate or reduce the risk or to lessen the consequences of such risk. Line management must ensure that their administrative controls are adequate to prevent unplanned personnel exposure to unacceptable risks. Effectively applied controls can give maximum protection through proper safety measures.

- Physical controls are actual physical barriers between the hazard and the human. Engineering controls, such as electrical and mechanical interlocks and fume hoods, are types of physical controls designed either to reduce or eliminate the hazard or to lessen the consequences of an accident. Personal protective equipment is considered a physical control. Laboratory coats, safety glasses, and many other items that are available at the Laboratory must be evaluated and worn during each activity associated with unacceptable risks.

- Administrative controls consist mainly of rules and regulations. They include procedures, written policies, training requirements, certification or permit requirements, administrative requirements set forth in the Health and Safety Manual, equipment maintenance or area rules, warning lights, buzzers, signs, and posted regulations.

- At the Laboratory, procedural controls are common and include standard operating procedures (SOPs), special work permits (SWPs), and operating instructions (OIs). These documents outline specific procedures, precautionary measures, or step-by-step instructions for conducting a hazardous or potentially hazardous activity. For example, SOPs identify the hazards involved in a particular activity and the specific measures needed to control those hazards. They include established procedures for performing the operation safely and usually specify training requirements and an emergency plan.

4.7. Accountability

Detailed record keeping provides feedback to measure performance and effectiveness of the control system. HSE-9's record-keeping system includes the following: accident/incident reports and investigations; SOPs and SWPs; training records; safety inspections, surveys, and operational reviews; minutes of any meetings related to safety matters; memoranda establishing responsibility or authority for safety matters; equipment maintenance records; monitoring and dosimetry records; quality assurance documents, safety analysis reports (SARs), and Action Description Memoranda.

Safety inspections and operational reviews in HSE-9 contribute in identifying hazards and deficiencies, determining whether operations are proceeding as intended, and observing whether personnel are complying with policy, regulations, and procedures. Each inspection is documented, and the results or follow-ups are added to health and safety records. Subsequent inspections check the status of prior problems, follow-ups, and corrections.

Group safety meetings and safety inspection reports *must* reflect line managers presence and participation in resolving safety concerns and issues. Management attendance is documented by including their *names and "title"* on the attendance sheets.

4.8. Employee Safety Training

The group leader has established a health and safety training program that includes the periodic assessment of training needs, the development and implementation of training activities, and the documentation of training received by each employee. In HSE-9 training is an integral part of the health and safety program and assists employees in acquiring the knowledge required for safe operations. Safety meetings represent a source of valuable safety information, and employee attendance at the group safety meetings is mandatory.

Training methods include formal training sessions, special meetings, formal reading assignments, and the study of SOPs and safe working procedures. Informal on-the-job training by section leaders is also effective. Feedback mechanisms are an integral part of health and safety training and ensure that the employee understands and retains essential knowledge. Section leaders should encourage question-and-answer sessions or structured discussions for employees to help understanding and provide useful feedback.

The HSE-9 training data base is administered by the Quality Assurance and Data Management Section. Completion of training and attendance at safety meetings is documented in a computerized record-keeping system in accordance with Administrative Requirement 1-4 of the Laboratory Health and Safety Manual. This documentation includes a brief description of the program, the date of each session, the instructor's name if one is present, and the names of attendees.

4.8.1. Job-Specific Training

Job-specific training for HSE-9 personnel is required by the HSE-9 group leader and is provided by the HSE-9 section leaders. Training shall include the following:

- hazards associated with the job,
- use of personal protective equipment,
- emergency response,
- applicable standard operating procedures and special work permits,
- job skills related to safety,
- applicable portions of the Health and Safety Manual, and
- accident/incident reporting procedures.

To aid section leaders with job-specific training, the HSE-9 Training Program in conjunction with the HSE-9 Safety Committee has acquired or will locate and will present the following videotaped material:

- hazard communication,
- general laboratory safety,
- chemical safety,
- radiation safety,
- handling glassware safely,
- using compressed gas cylinders, and
- fire safety and using fire extinguishers.

These materials will be presented at group safety meetings, at which attendance is mandatory. In addition, HSE-9 has a VCR and color television located in room 112 in OH-1 for training use. Section leaders have the responsibility to see that their section members view these materials if they were unable to attend the group safety meeting when the material was initially presented. It is also the section leader's responsibility to arrange

with the Quality Assurance and Data Management Section for new/transferred employees to view these materials.

The HSE-9 training program will continue to develop new training courses as they are needed or requested. A summary of HSE-9 video-taped training materials is listed in Appendix IV.

4.8.2. Training New/Transferred Employees

HSE-9 provides an organized and documented safety program for new or transferred employees within the first two weeks of reassignment or hire. Employees receive information about the topics listed under Section 4.8.1. This orientation is the responsibility of the section leader supervising the new/transferred employee and this training should be documented in the employee's file.

4.8.3. Non-Job-Specific Training

HSE Division regularly offers non-job-specific safety courses, such as radiation protection, first aid, and safety management. Upon request, the Safety Group and other HSE groups provide safety films or personnel to give safety talks for either group safety meetings or training sessions. The first-line supervisor shall arrange for employees to attend the applicable HSE training courses.

HSE-9 management has outlined the following non-job-specific training as mandatory for HSE-9 personnel:

- Electrical Safety (HSE-3)
- Fire Extinguishment (ENG-4)
- Safety Orientation for Managers (HSE-3)
- Basic Radiation Safety (HSE-1)

The following non-job-specific training is recommended by HSE-9 management.

- Cardiopulmonary Resuscitation (HSE-2)
- Introduction to Basic First Aid (HSE-2)

The HSE-9 training program has acquired LABNET which offers a variety of courses.

4.8.4. Supervisory Training

Training for supervisors in both safety functions and supervisory skills important to safety is part of the HSE-9 Health and Safety Program. Supervisors receive training on how to perform the safety functions for which they are responsible, including hazard and deficiency identification, and risk assessment and control. To help management fulfill its training requirements, HSE Division provides a Safety Orientation for Managers class that the HSE-9 group leader requires section leaders to attend.

VEHICLE INSPECTION FORM

Vehicle No.:_____ Date:_____

Make: _____ Inspected by: _____

Model:_____ _____

	Operable or "O.K."	Needs Repair		Operable or "O.K."	Needs Repair
Brakes, foot	_____	_____	High beam	_____	_____
Brakes, park	_____	_____	Low beam	_____	_____
Wipers	_____	_____	Running lights	_____	_____
Washer	_____	_____	Park lights	_____	_____
Mirrors	_____	_____	Turn signals	_____	_____
Seatbelts	_____	_____	Tail lights	_____	_____
Sun visors	_____	_____	Brake lights	_____	_____
Door locks	_____	_____	Back-up lights	_____	_____
Horn	_____	_____	License lights	_____	_____
Tires	_____	_____	Dash lights	_____	_____
Heater & defroster	_____	_____	Emergency flasher	_____	_____
Tailgate (if pick-up)	_____	_____	Windshield	_____	_____

Housekeeping Poor_____ Good_____ Excellent_____

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5. GENERAL LABORATORY PROCEDURES

5.1. Introduction

Safe practice requires an open-minded attitude and a knowledgeable awareness of potential hazards. Safety is a collective responsibility and requires the full cooperation of everyone in the laboratory. Most operations can be done safely using common sense. General laboratory procedures are discussed in this section. For further information, consult *Chemical Technicians' Ready Reference Handbook* by G. J. Shugar, R. A. Shugar, and L. Bauman.

5.2. Safety Rules for Laboratories

A limited number of rules exist for the laboratory. The section leader will verify that these rules are rigidly and impartially enforced. Willful noncompliance with the rules will subject employees to appropriate disciplinary action up to and including termination.

- Eye protection is required at all times in the laboratories and where chemicals are stored and handled.
- Horseplay and pranks are especially dangerous and are prohibited.
- The hazards of chemicals used should be known (e.g., reactivity, flammability, corrosivity, stability, and toxicity). Material safety data sheets are obtained for each chemical and kept up to date.
- Eating and drinking in the laboratories are not allowed. Using laboratory glassware as food or beverage containers is prohibited.
- Smoking is not permitted in laboratory buildings at TA-35, TA-50, or TA-59.
- Appropriate clothing must be worn, including a protective apron or laboratory coat. Confine long hair and loose clothing. Sandals are not permitted.
- Pipetting by mouth is prohibited. Use a pipetting device, such as a pipetting bulb, or a syringe connected to the pipette by a piece of tubing.
- Areas where biological samples are handled should be washed down each day with disinfectant. Always wash hands with soap and water before leaving the laboratory.
- Never perform analytical work when alone. Others should be present in the laboratory area.
- Visitors are reqiuired to follow the same rules as laboratory workers.
- Containers of acids, alkalies, flammable or combustable substances, or corrosive chemicals should be encased in plastic or transported in carriers to protect them from breakage and to limit their spread in case of leaks.

- Aisles, hallways, and exits must be kept clear at all times. See Section 5.4.

5.3. Housekeeping

Avoid unnecessary hazards by keeping drawers and cabinets closed while working. Keep aisles free of obstructions, such as boxes, chairs, and waste receptacles. Avoid slipping hazards by picking up ice, glass beads, stoppers, glass rods, and other small items from the floor and by wiping up wet spots. Use recommended procedures in Section 6.15.4. for the proper disposal of chemical waste.

5.4. Corridors, Stairs, and Exitways

Furniture and equipment in corridors often result in a distorted exit path. In case of an emergency, many people may need to exit through such paths, possibly in total darkness, and would be faced with numerous projections and tripping hazards. During rescue operations, the fire department needs to be able to maneuver stretchers and other emergency equipment through the halls and up and down stairs.

A clear width of at least 48 inches along a fairly straight path is needed. Equipment in corridors must not block fire alarm boxes or extinguishers. Fire alarm boxes and extinguishers must be visible from either direction down the corridor and should be placed only along one side. The storage of combustible and flammable materials in corridors and stairways increases the potential danger to building occupants, as well as emergency personnel, and may not be stored or allowed to accumulate in such spaces.

Two exit paths from one's work station to the outside of the building should be checked frequently by every employee. These routes should be used during any scheduled fire drills to familiarize personnel with alternate paths should primary ones become blocked.

5.5. Bottle Disposal

Thoroughly rinse all chemical bottles before disposal. Large bottles that have been rinsed may be set outside the laboratory for janitorial pickup. Small rinsed bottles may be deposited into laboratory trash cans for disposal. A properly rinsed bottle minimizes the hazards associated with breaking a bottle during the disposal process. At TA-50, discard rinsed bottles in the barrel labeled noncompactible trash.

5.6. Scalpel Blade and Hypodermic Needle Disposal

Razor or scalpel blades must not be thrown in the trash without taking precautions to ensure that they cannot cut someone who is handling the waste. Wrap blades in heavy tape prior to disposal. When large numbers of blades are involved, place them in a sealed container such as a metal can.

Place hypodermic needles in a can for disposal. Keep syringes and hypodermic needles in a locked drawer or cabinet when not in use.

Scalpel blades and hypodermic needles that have been used on biological samples must be disposed of differently because of their potential infectivity. Do not bend,

break or otherwise manipulate these instruments as this increases the chance of accidental puncture. Place such instruments into rigid, puncture-proof containers and carefully seal container prior to disposal.

5.7. Drying Ovens

No combustible material may be stored in any drying oven. This includes the large drying ovens built into laboratory benches.

5.8. Refrigerators

Refrigerators constitute a unique hazard because explosions may occur when volatile or unstable chemicals are stored. Domestic refrigerators should not be used for chemical storage even when they have been modified to eliminate sources of open electrical contacts inside the storage cabinet. Even when modified, the motor and other electrical parts on the exterior can still ignite flammable vapors in the room. Only explosive-proof refrigerators may be used for chemical storage, and they must be wired directly to the power source.

Chemicals stored in refrigerators should be sealed and labeled with the name of the material, the date placed in the refrigerator, and the name of the person storing the material.

Food storage in refrigerators used for chemicals is absolutely prohibited.

5.9. Centrifuges

Users of centrifuges must be aware of the importance of balancing each time the centrifuge is used. Securely anchor bench top centrifuges. Important items to check on a centrifuge are the following:

- adequate wall thickness to shield against accidental flyaway objects,
- wheel brakes to prevent the centrifuge from "walking,"
- electrical grounding,
- a disconnect switch to shut off the rotor if the top is inadvertently opened, and
- location to reduce vibration of other equipment and so that supplies will not vibrate off shelves.

5.10. Fume Hoods

Laboratory fume hoods should be operating properly. Hoods serve to exhaust toxic, offensive, or flammable vapors from the laboratory and, with the sash closed, to provide a physical barrier between the worker and the chemical reaction. Exhaust rate is not a reliable single measure of hood performance. Air supply to the room is also important because drafts across the hood face decrease the hood's effectiveness. Extremely high exhaust rates will cause turbulence and degrade the hood's performance. Velometers should be used to survey hoods on a regular

schedule to ensure uniformity of air flow over the face of the hood and to detect any changes.

The HSE-9 ventilation hoods have sliding doors or panels to ensure adequate air velocity through the hoods. On the hoods with doors that can close off the hood opening, the doors should be closed enough to cover at least 3/4 of the hood opening except for periods when the operator is physically reaching through the opening to perform some manipulation. For those hoods with sliding panels, all panels must remain as installed no matter how inconvenient this may be.

All operations involving flammable, toxic, noxious, or otherwise hazardous solids, liquids, vapors, or gases must be conducted in a fume hood as much as possible. Perchloric acid digestions should only be carried out in the special perchloric acid hoods. The air slots at the back base of the working surface must not be blocked. Operations should be carried out as far into the hood as practical and at least 6 in. from the front edge of the hood. Keep your head outside the hood face. Equipment used within the hood should be raised on legs to avoid interference with the airflow, and the sash should be pulled as low as work permits.

The hood should not be used as a means for evaporating waste chemicals. Give waste chemicals to the Chemical Waste Coordinator for proper disposal. Chemicals should not be stored in hoods as they block air slots and crowd the available working area.

Hoods are evaluated annually by HSE-5, and the results are posted on the hood face. Hoods that have been evaluated and passed are marked with a sticker approving usage. Hoods without this sticker should not be used. Even with the sticker, adequate airflow should be verified by each operator before starting an operation that may emanate toxic or hazardous vapors or mists. If the airflow does not seem adequate, the operator should notify his supervisor and should not carry out any operations in the hood until adequate airflow is established.

5.11. Storage of Chemicals

Storage facilities must be stable and secure so tilting and collapse will not occur. Large containers of reagents should be stored on low shelves in a tray adequate to contain spills or leakage. Chemicals should be separated into compatible chemical families and then arranged in alphabetical order. See Table I. Examples of Incompatible Chemicals.

HSE-9 has observed that plastic bottles used to store metal standards in an acid matrix become brittle upon standing over an extended period of time. Normal handling of these bottles has caused fractures to develop that later resulted in leakage. Literature has reported that small plastic bottles of 30% hydrogen peroxide become brittle upon standing for one year or more. Normal handling has also caused these bottles to develop fractures that result in severe leakage. Since 30% hydrogen peroxide is such a strong oxidizing agent, leakage can create a very dangerous situation if any oxidizable materials are in the vicinity. Check all plastic bottles for fractures and then store the bottles in a beaker that could contain any leakage.

Keep only minimal quantities of flammable liquids in the laboratory. Store large quantities in approved safety containers or in fire-resistant solvent cabinets. Keep flammable liquids away from heat and direct sunlight. Many aldehydes, ethers, unsaturated hydrocarbons, and other compounds form peroxides on standing. Peroxide formation is accelerated by the presence of UV light and elevated temperature. Flammable liquids must be stored so that accidental contact with strong oxidizing agents such as permanganates or chlorates is avoided.

In the laboratory, keep chemicals not in current use in a chemical storage cabinet. HSE-9 keeps a current inventory of all chemicals and their locations.

5.12 Container Labels

Clearly label all reagent bottles and containers as to their contents. All chemicals received for laboratory use should be labeled with the date received. Prepared reagents must be labeled with their chemical composition, concentration, date of preparation, and name of the person who prepared the reagent. All unlabeled containers shall be discarded. Consult your supervisor about how and where to discard such reagents.

5.13 Compressed Gases

Compressed gases are most frequently used in standard cylinders of approximately 3000 cu ft. These gas cylinders are filled to high pressures, up to 2500 pounds per square inch on gauge (psig). The minimum-sized cylinder adequate to perform the job should be routinely used, particularly for hazardous gases such as the halogens and sulfur dioxide.

Cylinders, as received from the Liquid and Compressed Gas Processing Center (MAT-1), must be stored in the cages behind building OH-1 at TA-59 and at the south dock and lower east side of the building at TA-50. All cylinders must be capped and secured in place by a strap or chain located above center. Only those cylinders actually in use are allowed in the laboratory. Periodically inventory the gas cylinder storage areas and return those cylinders not in use to MAT-1.

Know the properties of the gas being handled. This includes factors such as toxicity and flammability. It is recommended that the section of the Matheson Gas Data Book pertaining to a particular gas be read by employees to alert them to any special precautions required by a gas. The following safety rules for use of compressed gases are suggested by the American Chemical Society, Committee on Chemical Safety:

- Handle cylinders of compressed gases as high-energy sources and potential explosives.
- When storing or transporting cylinders, make sure the protective cap is in place to protect the valve stem.
- When moving large cylinders, use a hand truck with the safety chain or strap in place. Never roll, slide, or drag the cylinder to move it even for a short distance.

- Do not expose cylinders to temperatures higher than approximately 50°C. Some rupture devices on cylinders will release at about 65°C.

- Be sure that the contents of the cylinder are properly identified. The contents of a cylinder are stamped on the valve and noted by a tag around the valve. Contents are also indicated by stenciling and a decal on the cylinder body. If all indicators do not agree, notify the Liquid and Compressed Gas Processing Center and return the cylinder to the gas plant for checking. The color of a cylinder is not a reliable indicator of the contents. Label any cylinder that you have ordered with your name.

- Never lubricate, modify, force, or tamper with a cylinder valve or any relief valve.

- Cylinders of toxic, flammable, or reactive gases should be used in fume hoods only. They should be stored in appropriately ventilated cabinets.

- Cylinders located in laboratories must be secured to a wall, bench, or other firm support by means of a chain or strap. The securing device must support the upper half of the cylinder to prevent tipping.

- Do not extinguish a flame involving a highly combustible gas until the source of gas has been shut off; otherwise, it can reignite, causing an explosion.

- The Compressed Gas Association has developed various cylinder valve outlet connectors for different families of gases to prevent the interchange of regulating and control equipment between gases that are not compatible. Use the proper regulator to control pressure and rate of flow. Do not use adaptors.

- Oil or grease on the high-pressure side of a cylinder of oxygen, chlorine, or oxidizing agent can lead to an explosion. Never lubricate a regulator. Use new regulators for oxygen cylinders or a regulator currently on an oxygen cylinder.

- Completely release the regulator pressure adjustment valve before opening the cylinder valve, and open the cylinder valve slowly. Never use a hammer or oversized wrench to force a valve that is stuck. Return all cylinders that leak or have stuck valves to the Liquid and Compressed Gas Processing Center.

- Always use a trap or check valve to prevent back-siphonage of chemicals into the cylinder.

- When the pressure in a cylinder is reduced to approximately 5 psig, attach an "empty" label and replace it with a fresh cylinder. Never bleed a cylinder completely empty. Leave a slight pressure to keep contaminants out. Place the empty cylinder in one of the outside storage areas for eventual return to the Liquid and Compressed Gas Processing Center.

- Always wear safety glasses when handling and using compressed gases.

5.14. Glassware

Borosilicate glassware is recommended for all laboratory glassware except for special experiments that use UV or other light sources. The only soft glass in the laboratory should be reagent bottles, measuring equipment, stirring rods, and tubing. The Laboratory maintains a well-equipped glass shop staffed with professional glass blowers. Difficult jobs should be referred to the glass shop in MEC-10, Specialized Fabrication.

Cracked or broken glassware must be discarded. All glassware for disposal must be placed in a plastic-lined cardboard box. Tape the box shut when full and place in the hallway for janitorial pickup. Glassware contaminated with radionuclides should be placed in the special radioactive contamination containers. A special waste container has been set up for PCB contaminated items. Never throw broken glassware in the regular trash as it can cause injury to janitorial personnel.

5.14.1. Preparing Glass Tubing

Simple operations, such as cutting and bending glass tubing, will be performed by competent personnel. Score the tubing with one quick firm stroke with a sharp file, rocking the file to extend the deep nick one-third around the circumference. Wetting the nick will help keep the fracture open. Wrap the tubing with a towel, turn the nick away from your body with the nick centered between your hands, and push on the tubing with your thumbs. Fire-polish all cut ends.

When inserting glass tubing through a perforated cork or rubber stopper, make sure the hole is sufficiently large and lubricate the glass and stopper with water or glycerine. Wrap both hands in a towel or wear leather gloves and insert the tubing with a slow, rotating, pushing motion. Keep the distance between the hands at a minimum.

5.14.2. Ground-Glass Stoppers

Frozen ground-glass stoppers can be hazardous to remove as the container could break during the process. Volumetric flasks can be especially hazardous due to their long, thin necks. In some cases the stopper can be removed using a special tool made expressly for this purpose, or it may be freed by placing it under hot running water for 30 seconds and/or gently tapping the stopper. It is better to discard the apparatus than to sustain an injury trying to remove a frozen stopper. If you have questions about a particular situation, consult your supervisor.

5.15. Electrical Safety

Under certain circumstances even electrical currents of very low amperage and voltage may result in fatal shock. Low-voltage dc circuits do not normally present a hazard to human life, although severe burns are possible. The length of contact with a live circuit affects the degree of damage.

- Only qualified persons should maintain electric or electronic equipment.

- Make no modifications that negate the built-in safety features of the equipment.
- Never use electric wires as supports, and never pull on live wires.
- Locate portable heaters away from combustible materials.
- Report evidence of any excessive heating of equipment to your supervisor.
- Cardiopulmonary resuscitation (CPR) often will revive the victims of high-voltage shock, including lightning strikes.

All laboratory personnel should know the location of circuit breakers and how to cut off all electrical service to their laboratory. With 60-cycle ac, the perception threshold of electrical shock is 1 mA. The effects of increasing current on an adult human are listed below.

Current (mA)	Effect
0-1	No sensation
1-3	Mild perception
3-10	Painful shock, muscular contraction
Over 10	Paralysis, inability to let go
Over 30	Asphyxiation, unconsciousness
80-240	Fibrillation
4000	Heart paralysis
Over 5000	Burning

When used improperly or with no regard for good safety practices, most electrical equipment used in the laboratory could cause fatalities. Ground fault interrupters should be installed in each circuit, and all electrical equipment must be properly grounded. Equipment with a three-pronged plug will provide a satisfactory ground. Equipment without a three-pronged plug can use a three-wire adapter plug. Connect the green grounding wire to the case of the instrument.

Electrical equipment that produces the slightest shock is not to be used until it has been repaired and properly grounded. Do not operate any electrical equipment when standing on a wet surface, when your hands are wet, or when your body is in contact with a metal surface. Do not operate any equipment that has a frayed cord. Frequently inspect all electrical equipment for faulty connections and other unsafe conditions. HSE-9 holds an annual electrical safety inspection.

5.16. Lifting and Carrying

Do not lift excessively heavy loads. If you are in doubt about your lifting capability, get help. Never carry a load that you cannot see over or around. Use a cart or hand truck to move heavy, bulky, or unwieldy items. Arrange to have Pan Am riggers do difficult operations. Supervisors should anticipate work requiring Pan Am help and make the necessary arrangements in advance.

When lifting a heavy object, correct posture is important to prevent injury. The following procedures should be followed when lifting is required:

- Set feet solidly, with one foot slightly ahead of the other, and far enough apart to give good balance and stability.
- Get as close to the load as possible.
- Bend legs about 90 degrees at the knees keeping the back as straight as possible. The back need not be vertical, but it should not be arched.
- Bend at the hips, not the middle of the back.
- Grip the object firmly.
- Straighten the legs to lift the object, bringing the back to the vertical position at the same time.
- If it becomes necessary to change your grip, first set the object down.
- The above procedure is reversed when setting an object down.

Refer to the HSE-9 Back Facts booklet, which is a program for the prevention of back injury and maintenance of a healthy back. Consult the Occupational Medicine Group (HSE-2) for more information on back care. The HSE division training program offers a back care clinic. This clinic teaches proper care of the spinal column and its supporting muscular system, back anatomy and physiology, body mechanics and posture, proper lifting and pulling techniques, stress management, and relaxation exercises. The clinic is conducted by a registered physical therapist. Call Occupational Medicine, 7-7251 for information

5.17 After-Hours Work

No one shall work alone in the HSE-9 laboratories. Laboratory work after normal working hours is only permitted when there are at least two people present. Office work is permitted at any time. The Group Leader, or his alternate, must approve all laboratory work outside normal working hours.

TABLE I. Examples of Incompatible Chemicals

Chemicals	Incompatible With
Acetic acid	Chromic acid, nitric acid, hydroxyl compounds, ethylene glycol, perchloric acid, peroxides, permanganates
Acetylene	Chlorine, bromine, copper, fluorine, silver, mercury
Acetone	Concentrated nitric and sulfuric acid mixtures
Alkali and alkaline earth metals (such as powdered aluminum or magnesium, calcium, lithium, sodium, potassium)	Water, carbon tetrachloride or other chlorinated hydrocarbons, carbon dioxide, halogens
Ammonia (anhydrous)	Mercury (in manometers, for example), chlorine, calcium hypochlorite, iodine, bromine, hydrofluoric acid (anhydrous)
Ammonium nitrate	Acids, powdered metals, flammable liquids, chlorates, nitrites, sulfur, finely divided organic combustible materials
Aniline	Nitric acid, hydrogen peroxide
Arsenical materials	Any reducing agent
Azides	Acids
Bromine	See chlorine
Calcium oxide	Water
Carbon (activated)	Calcium hypochlorite, all oxidizing agents
Carbon tetrachloride	Sodium
Chlorates	Ammonium salts, acids, powdered metals, sulfur, finely divided organic or combustible materials
Chromic acid and chromium trioxide	Acetic acid, naphthalene, camphor, glycerol, alcohol, flammable liquids in general
Chlorine	Ammonia, acetylene, butadiene, butane, methane, propane (or other petroleum gases), hydrogen, sodium carbide, benzene, finely divided metals, turpentine
Chlorine dioxide	Ammonia, methane, phosphine, hydrogen sulfide
Copper	Acetylene, hydrogen peroxide

TABLE I. (cont)

Chemicals	Incompatible With
Cumene hydroperoxide	Acids (organic or inorganic)
Cyanides	Acids
Flammable liquids	Ammonium nitrate, chromic acid, hydrogen peroxide, nitric acid, sodium peroxide, halogens
Fluorine	All other chemicals
Hydrocarbons (such as butane, propane, benzene)	Fluorine, chlorine, bromine, chromic acid, sodium peroxide
Hydrocyanic acid	Nitric acid, alkali
Hydrofluoric acid (anhydrous)	Ammonia (aqueous or anhydrous)
Hydrogen peroxide	Copper, chromium, iron, most metals or their salts, alcohols, acetone, organic materials, aniline, nitromethane, combustible materials
Hydrogen sulfide	Fuming nitric acid, oxidizing gases
Hypochlorites	Acids, activated carbon
Iodine	Acetylene, ammonia (aqueous or anhydrous), hydrogen
Mercury	Acetylene, fulminic acid, ammonia
Nitrates	Sulfuric acid
Nitric acid (concentrated)	Acetic acid, aniline, chromic acid, hydrocyanic acid, hydrogen sulfide, flammable liquids, flammable gases, copper, brass, and heavy metals
Nitrites	Acids
Nitroparaffins	Inorganic bases, amines
Oxalic acid	Silver, mercury
Oxygen	Oils, grease, hydrogen; flammable liquids, solids, or gases
Perchloric acid	Acetic anhydride, bismuth and its alloys, alcohol, paper, wood, grease, oils
Peroxides, organic	Acids (organic or mineral), avoid friction, store cold

TABLE I. (cont)

Chemicals	Incompatible With
Phosphorus (white)	Air, oxygen, alkalies, reducing agents
Potassium	Carbon tetrachloride, carbon dioxide, water
Potassium chlorate	Sulfuric and other acids
Potassium perchlorate (see also chlorates)	Sulfuric and other acids
Potassium permanganate	Glycerol, ethylene glycol, benzaldehyde, sulfuric acid
Selenides	Reducing agents
Silver	Acetylene, oxalic acid, tartartic acid, ammonium compounds, fulminic acid
Sodium	Carbon tetrachloride, carbon dioxide, water
Sodium nitrite	Ammonium nitrate and other ammonium salts
Sodium peroxide	Ethyl or methyl alcohol, glacial acetic acid, anhydride, benzaldehyde, carbon disulfide, glycerin, ethylene glycol, ethyl acetate, methyl acetate, furfural
Sulfides	Acids
Sulfuric acid	Potassium chlorate, potassium perchlorate, potassium permanganate (similar compounds of light metals such as sodium and lithium)
Tellurides	Reducing agents

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6. CHEMICAL SAFETY AND HAZARD COMMUNICATION

6.1. Introduction

Chemicals may be hazardous due to several mechanisms. Some chemicals are toxic, while others may be corrosive, flammable or radioactive. It is quite common for a substance to fall into more than one category; for example, p-dioxane is flammable, toxic, and carcinogenic. The information in this section does not cover all possible hazards. Consult the label on the container and the material safety data sheet as received from the supplier for information on the hazards of the contents. Further information on chemical hazards can be found in the OHSA handbooks located in the Group office, in the NIOSH Pocket Guide to Chemical Hazards given to all HSE-9 personnel, and by consulting the HSE-5 industrial hygiene group or your supervisor.

When handling chemicals, wear protective clothing to prevent accidental contact with the material. Depending on the quantities being handled, the type of material, and the operation, this may mean rubber gloves, goggles, face shields, and rubber aprons. Many chemicals should be handled in the hood or with some type of respiratory protection.

Disposal of chemical hazardous waste down sink and floor drains is prohibited by Laboratory policy (AR 10-3 of the Health and Safety Manual) without prior approval from the Waste Management Group (HSE-7). This policy *must be* followed to keep the Laboratory in compliance with Federal Environmental Protection Agency regulations and DOE orders.

6.2. Chemical Spills

Chemical-spill, clean-up kits for acids, caustics and solvents are located in the east and west hallways and basement of OH-1, in the main laboratory and in the basement of TA-50 near room 118, and in room A133 at TA-35. Locate these spill control products and become familiar with the Instruction Manual. The manual contains a list of hazardous chemicals for which the spill control products can be used. Materials used to clean up spills at TA-50 should be placed in the radioactive waste containers after thoroughly rinsing with water.

6.3. Organic Peroxides

As a class, organic peroxides are low-power explosives. They are hazardous because of their extreme sensitivity to shock, sparks, and other forms of accidental ignition. All organic peroxides are extremely flammable. Compounds of the following types are known to form peroxides:

- Aldehydes
- Ethers, especially cyclic ethers, and ethers derived from primary and secondary alcohols.
- Compounds containing benzylic hydrogen atoms.

- Compounds containing the allylic structure, including most alkenes.
- Ketones, especially cyclic ketones.
- Vinyl and vinylidene compounds (e.g., vinyl acetate and vinylidene chloride).

6.4. Acids and Alkalies

All acids and alkalies should be handled with care, especially when in concentrated form. Take extra precautions when opening containers sealed at sea level and in handling bulk acids and bases. Air tight containers sealed near sea level are mildly pressurized compared with Los Alamos atmospheric pressure and should be opened with care.

If acids or alkalies are splashed on the skin, immediately wash the affected area with large volumes of water. Under no circumstances should the acid or alkali be neutralized. Alkalies require prolonged washing to remove the chemical. Glacial acetic acid and sulfuric acid also require prolonged washing. Wash the affected area for at least 15 minutes. Gentle massage may help. Give particular attention to the crevices around the fingernails. Report to HSE-2, Occupational Medicine, for further treatment. If the eyes are involved, wash for at least 20-30 minutes under running water and report immediately to HSE-2. Know the location of the safety showers and eye wash stations in your work area. See Fig. 2.2-2.6.

Hydrofluoric acid is extremely hazardous and must always be handled with neoprene gloves. Hydrofluoric acid burns are deep, painful, and slow to heal. The fumes of this acid can be as hazardous as exposure to the liquid.

The amount of concentrated acid allowed in each laboratory is limited to one bottle in use plus the amount expected to be used in one week. Plastic-coated bottles are recommended. Acid bottles should be placed in an appropriate tray or container. Never carry bottles of acid between laboratories except in a safety container. Store oxidizing acids, such as nitric and perchloric, away from reducible materials such as organic solvents, paper or cardboard. Do not store acids and bases together. Whenever possible, conduct all operations involving acids in fume hoods. When pouring acids, use a tray capable of holding all of the liquid in the event of glassware failure.

6.5. Oxidizing Agents

Paper or cotton towels are frequently used to wipe up spills of nitric acid and/or hydrogen peroxide. Rinse these towels thoroughly with water before discarding in the trash because the contents of the trash container have been known to spontaneously ignite at a later time. Do not discard strong oxidizing agents in trash containers where easily oxidized waste is discarded. Perchloric acid spills should be wiped up with wet towels. These towels must also be thoroughly rinsed with water before disposal.

6.6. Perchloric Acid

Due to the extremely hazardous nature of perchloric acid and the potential for misuse, it should be used only by experienced personnel in procedures of demonstrated safety in a fume hood with wash-down provisions. At its boiling point it will react violently with organic and some inorganic materials. Perchloric acid fumes can form unstable perchlorates with dust and other organic matter that can ultimately ignite or explode. Old containers of perchloric acid are a hazard and should not be kept or opened if crystals have formed around the cap. The fume hood must be washed down after each use. Do not mix the acid with a strong dehydrating agent, such as acetic anhydride or concentrated sulfuric acid because anhydrous perchloric acid, which is very unstable, is produced. Spills should be diluted with water and wiped up with a towel. The towel must be thoroughly rinsed with water before disposal.

6.7. Organic Solvents

Most of the organic solvents commonly used are volatile and are harmful when relatively small amounts are inhaled. Use organic solvents in a hood and wear the proper type of gloves. Many solvents are readily absorbed through the skin. Some examples are aniline, nitrobenzene, phenylhydrazine, and dimethylsulfate. Although not very toxic itself, dimethylsulfoxide can penetrate the skin carrying with it dissolved materials that are toxic. A comprehensive list indicating the types of compounds known to be toxic is contained in "Threshold Limit Values for Chemical Substances and Physical Agents in the Workroom Environment," American Conference of Governmental Industrial Hygienists, Cincinnati, OH 45201. More information about the toxicity of chemicals can be obtained through the industrial hygienist assigned to your area and from the OSHA handbooks.

Inhalation of contaminated air can be hazardous. Some compounds can be absorbed through the lungs and subsequently transported to other parts of the body. All potentially toxic chemicals should be handled in a hood to minimize the potential for inhalation. Never directly smell a chemical as a means of identification.

The fire hazard of some organic solvents can be appreciable. Refer to Section 6.12. on flammable liquids. Many organic solvents are denser than air and tend to collect in low spots, such as the floor, or they can travel along bench tops. Beware of distant flames and hot ovens. Work in areas of adequate ventilation. Do not use highly volatile, flammable solvents in glove boxes.

Diethyl ether must be stored in a refrigerator designed or modified for the storage of flammable solvents. The ether must be inhibited the copper wire, if possible, and the container must be labeled with the opening date. Storage is limited to six months. After six months, give the container to the Chemical Waste Coordinator for disposal.

Inventories of solvents should be kept as low as possible. Check the HSE-9 chemical inventory database before ordering new solvents. Beware of plastic containers. Some containers are permeable to organic solvents and others may dissolve in the solvent after a specific period of time has passed.

6.8. Carcinogens, Mutagens, and Teratogens

Within the expansive array of toxic chemicals, chemical carcinogens, mutagens, and teratogens have been recognized as a significant subclass of hazardous materials. The toxicity of these materials is unique because of the length of time between exposure to the agent and the onset of symptoms. Because of this latent effect, it is difficult to establish which chemicals fall into the following categories:

- Carcinogens-substances capable of inducing cancer.
- Mutagens-substances causing permanent, transmissible, alterations in genetic information.
- Teratogens-agents interfering with normal prenatal development causing abnormalities in the fetus.

A list of known and suspected carcinogens is continually expanded or updated by OSHA. As substances are scientifically evaluated, they are placed in one of three categories. The entire Category I listing is available from regional offices of OSHA.

- Category I. Confirmed carcinogens whose classification is based on human data, on tests in two mammalian species, or on replicated tests in one species.
- Category II. Substances whose carcinogenicity is presumed based on evidence that is only suggestive or on testing that is positive in only one species and has not yet been replicated.
- Category III. Substances that have shown no evidence or at best only meager evidence of carcinogenic risk during testing.

A current list of carcinogens that includes the following information must be readily accessible to emergency and HSE personnel:

- chemical name,
- Chemical Abstract Service (CAS) number,
- quantity of carcinogen,
- location of use and storage,
- brief description of operation,
- Standard Operating Procedure (SOP) number, and
- names of employees who work with or handle the carcinogen.

HSE-9 must use the following three lists to determine whether materials in use or in storage in HSE-9 are carcinogens.

The current 29 CFR 1910 OSHA-regulated carcinogens listed in AR 6-1 (February, 1990) of the Laboratory Health and Safety Manual are the following:

Asbestos
Benzene
4-Nitrobiphenyl
alpha-Naphthylamine
Ethylene Oxide
Methyl Chloromethyl ether
3,3'-Dichlorobenzidine (and its salts)
bis-Chloromethyl Ether
beta-Naphthylamine
Benzidine
4-Aminodiphenyl
beta-Propriolactone
2-Acetylamino Fluorene
4-Dimethylamino Azobenzene
N-Nitroso Dimethylamine
Vinyl Chloride
Inorganic Arsenic
Coke Oven Emissions
Ethyleneimine
Formaldehyde
1,2-Dibromo-3-Chloropropane
Acetonitrile

The following confirmed human carcinogens are substances, or substances associated with industrial processes, recognized to have carcinogenic or cocarcinogenic potential:

Chromate (chromite ore processing)
Chromium (VI) certain water insoluble compounds
Coal tar pitch volatiles
Nickel, fume and dust (sulfide roasting)
Zinc chromates

The following suspected human carcinogens (confirmed animal carcinogens) are chemical substances or substances associated with industrial processes that are suspect of inducing cancer, based either on limited epidemiological evidence or demonstrated carcinogenesis in one or more animal species by appropriate methods:

Acrylamide
Antimony trioxide production
Arsenic trioxide production
Benzo[a]pyrene
Beryllium
1,3-Butadiene
Carbon tetrachloride
Chloroform
Chromates of Pb and Zn, as Cr
Chrysene
Dimethylcarbamoyl chloride
1,1-Dimethylhydrazine
Dimethyl sulfate
Ethylene dibromide (EDB)
Hexachlorobutadiene
Hexamethyl phosphoramide
Hydrazine
4,4'-Methylenebis (2-chloroaniline)
Methylene chloride
4,4'-Methylenedianiline
Methylhydrazine
Methyl iodide
2-Nitropropane
N-Phenyl-beta-naphthylamine
Phenylhydrazine
Propane sultone
Propylene imine
o-Tolidine (DMB)
o-Toluidine
p-Toluidine
Vinyl bromide
Vinylcyclohexane dioxide

6.9. Metals

Some metals are very hazardous and require special precautions. Many metals are toxic and some can burn or explode when in finely divided form. Some form

organo-metallic compounds that can explode under certain conditions. Check with your supervisor before beginning any new or unfamiliar procedure.

6.9.1. Mercury

Mercury can be absorbed through the skin. Soluble compounds will cause severe poisoning if ingested. The volatility of mercury at room temperature is sufficient to constitute an inhalation hazard. Mercury spills must be immediately and thoroughly cleaned up using an aspirator bulb or a vacuum device. Mercury spilled into floor cracks can be made nonvolatile by amalgamation with zinc dust. Contact the area industrial hygienist to determine the effectiveness of the cleanup.

6.9.2. Cadmium

Cadmium, as finely divided metal or as cadmium oxide, is highly toxic when inhaled or ingested. Silver soldering must be done in a well-ventilated area as cadmium oxide fumes may be present.

6.9.3. Beryllium

Beryllium and many of its compounds are especially toxic, and care must be exercised when working with them. Beryllium includes beryllium metal, beryllium oxide, alloys containing 4% or more beryllium, and beryllium compounds such as beryllium sulfate. An SOP and the approval of your immediate supervisor and HSE-5 is required before initiating any work with beryllium or its compounds. Low level beryllium standard solutions used by HSE-9 do not need this approval. Well-ventilated enclosures or glove boxes may be required. Respirators may also be necessary.

6.9.4. Reactivity with Water

Some metals, such as lithium, sodium, potassium, calcium, barium, and strontium, react violently with water-releasing hydrogen gas in the process. The heat of reaction is often enough to ignite the hydrogen causing an explosion. These metals are usually stored under mineral oil.

The examples discussed above do not constitute a complete list of all the hazards of working with metals but are descriptions of some of the more common hazards. Consult your supervisor, your assigned industrial hygienist, and the available literature before working with any new material or on a new or unfamiliar procedure.

6.10. Cyanides and Nitriles

Cyanides and Nitriles are among the most toxic and rapidly acting substances encountered in the chemical laboratory. A few breaths of hydrogen cyanide (HCN) can be fatal.

Because the toxic action of inhaling cyanides and nitriles is so rapid, first-aid treatment and an antidote (amyl nitrite) must be given immediately. Persons using

cyanide should have amyl nitrite ampules pinned to their lab coats and taped to the hood. The person administering first aid should break open an amyl nitrite ampule and hold it under the affected person's nose for about 15 seconds and continue at two-minute intervals until help arrives. Excess nitrite will reduce blood pressure. If the patient is not breathing, artificial respiration must also be administered.

Section leaders shall verify that personnel using cyanides are thoroughly familiar with proper disposal methods and with methods to convert the material to cyanates.

6.11. Hydrogen Sulfide

Hydrogen sulfide is extremely dangerous. Exposed persons quickly lose their ability to smell it, even when it is still present in the air. Exposure to low concentrations causes irritation of all parts of the respiratory system and eyes, followed by headache, dizziness, and nausea. Higher concentrations may cause immediate loss of consciousness followed by respiratory paralysis. All laboratory operations with hydrogen sulfide must be carried out in a fume hood.

6.12. Flammable Liquids

Flammable liquids are used in a variety of analytical procedures and are used to clean apparatus that contain water-insoluble materials. Flammable liquids should only be used in a hood. If the hood is operating properly, the airflow will be adequate to keep the concentration of vapor in the air below the flammability range. When using organic solvents, choose the least flammable solvent in situations where you have a choice. If possible, avoid using diethyl ether or acetone. Work away from flames, furnaces, hot plates, and other sources of ignition.

Store flammable liquids in approved containers. For quantities less than one liter, a glass bottle with a tight screw-cap seal is usually acceptable. Quantities greater than one liter should be transferred to a metal safety can with a spring-loaded lid and an antiflashback screen. Properly label the new container to show its contents. When transferring a large quantity of flammable liquid from one container to another, electrically ground and bond both containers as the friction flow of the liquid can build up enough static charge to ignite the vapors.

If a large amount of flammable material is spilled, prompt action is necessary to prevent fire:

- immediately shut down all flames and electrical equipment;
- close off the source of the flammable material as quickly as possible;
- vacate the area of all personnel not immediately needed to handle the emergency; and
- ventilate the area as effectively as possible.

Use extreme care in any clean-up operation. Almost anything done to clean up a spill has a tendency to increase the vapor concentration and, consequently, to increase the fire hazard. Spill clean-up kits are available near all work areas.

Spreading the absorbent from the kit on the spill will greatly reduce the hazards. In the event of a fire, dial 911, pull the fire alarms, and evacuate the building. See fire safety Section 3.5. and building evacuation Section 2.9. for more information.

6.13. Cryogenic Liquids and Solids

Cryogenic liquids and solids are used as very low temperature coolants and are hazardous because of their extreme cold and the properties of the vaporized gas. Cryogenic materials are normally gases but are converted to liquids or solids by very low temperatures and/or high pressures. Examples are liquid nitrogen, liquid air, and dry ice.

Contact with the cold liquid, solid, or vapor can cause frostbite, and contact with cold pipes or containers can cause the skin to stick. When working with these materials, wearing a face shield is mandatory. Heavy gloves must also be worn. When liquids vaporize, large volumes of gas are produced, and these gases can be toxic, explosive or corrosive. Relatively inert gases, such as nitrogen and carbon dioxide, as well as the so-called "inert gases," can become asphyxiants because of rapid displacement of air in an enclosed area.

Cryogenic liquids and solids must not be stored in sealed containers because excessive pressures can build up. In addition accidental pressurization from ice plugs forming on vents or entrapment of cryogenic fluids between valves without pressure relief must be avoided. Dewar flasks made of glass must be completely taped on the outside or plastic weave wrapped to minimize the danger from flying glass in the event of an implosion.

A special hazard of using cryogenic liquids is possible oxygen condensation from the atmosphere. Oxygen has a higher boiling point than does nitrogen. As liquid nitrogen evaporates, it becomes richer in oxygen until almost pure oxygen remains. If this oxygen comes in contact with a combustible material, an explosion may occur. Even metals have been known to explode when in contact with liquid oxygen, possibly because of oil film on the metal.

6.14. Radioactive Materials

"As low as is reasonably achievable" (ALARA) is an operating principle that encourages keeping exposure to toxic materials and radiation to the lowest reasonably achievable level. Using this principle is prudent because the harmful effect threshold for ionizing radiation and some toxic agents has not been demonstrated absolutely. In practice, ALARA means conducting activities at the Laboratory such that exposures to radiation and other toxic agents are reduced to the lowest practical levels, considering the possible means of exposure reduction and the cost.

The ALARA principle requires that

- employees who work with radiation-producing machines, radioactive materials, or toxic agents know about and apply reasonable exposure-reducing techniques BEFORE acting; and

- supervisory personnel fully support and train employees in ways to reduce exposures and hold the employees accountable for their actions.

As adopted by the Department of Energy and this Laboratory, the ALARA principle also applies to radioactive and toxic material releases to the environment, to collective doses, and to individual doses for which it was originally set forth.

Because of the nature of the work in HSE-9, criticality hazards do not exist. If the nature of the work changes in the future and the work involves several hundred grams of fissionable material, HSE-6 should be consulted to eliminate any possibility of a criticality accident. At that time a Standard Operating Procedure (SOP) must be written.

When exiting a laboratory in which above-background levels of radionuclides, including standards, are used or stored, shoes and clothing should be monitored. Monitoring will prevent the spread of contamination into other areas, including low-level laboratories. This is especially true at TA-50 where higher-level radioactive samples, such as liquid waste treatment sludge, are handled. Bench surfaces should be monitored daily by the occupant of the laboratory. All radioactive spills will be cleaned up immediately until the level of surface contamination is nondetectable above background.

An HSE-1 Health Protection Technician (HPT) is assigned to Building 1 at TA-50 to provide monitoring support for radioactive contamination control. Instruments are available to laboratory personnel for self-monitoring of the work area as well as their own person. HSE-1 monitors the laboratory area at TA-50 once a week to determine whether the floor and bench tops are free from removable radioactivity. The spilling of radioactive material in the laboratory at TA-50 when processing high-level samples or high-level standard radioisotope solutions is considered an emergency situation. Any radioactive spills must be reported to the supervisor and the HPT immediately. The HPT shall determine the extent of the contamination and establish safety precautions to be taken during the cleanup. TA-50 laboratory personnel who perform radiochemical analyses should periodically submit urine samples (according to the established HSE-1 schedule) to the bioassay section for the analysis of alpha emitters.

Surface contamination can be removed by a variety of methods. Covering the spot with tape, pressing down, and removing the tape is sometimes effective. Scrubbing with 6 M hydrochloric acid or 4 M nitric acid, depending on the surface, may be more effective in removing contamination. Organic solvents are sometimes effective. If a spot cannot be removed, report it to your supervisor and HSE-1.

In case of an accident involving radioactive material, the first concern is the health and safety of the people involved. If anyone requires emergency care or first aid, see that it is provided at once. In the event of a significant spill, the laboratory should be vacated immediately. Notify your supervisor and HSE-1 at 7-7878. It may be necessary to cordon the area prior to initiating decontamination procedures. When decontamination is complete, dispose of all cleaning materials in the contaminated waste bin. Monitor all clothing to avoid spreading the contamination. Any cuts obtained while working with radioactive material must be monitored for contamination. If the contaminant is plutonium or americium, an HSE-1 wound

monitoring system will be used. Call HSE-1 at 7-7878 to report potentially contaminated injuries and proceed to HSE-2, Occupational Medicine for medical treatment after monitoring.

See Appendix V., Personnel Radiation Exposure Control AR 3-1, for more information on radiation exposure standards and personnel exposure monitoring.

6.15. Hazardous Waste Management

6.15.1. Introduction

Chemical waste disposal is complex. It is governed by many federal and state laws from the time we as a generator declare it waste to the time it is treated or disposed of. The Laboratory operates under an extensive waste analysis plan. If there is not accurate and documented process knowledge, such as labels on bottles or lists of materials in a container, the material must be analyzed to identify hazardous constituents. The Laboratory must assume that the material is hazardous waste until analytical information is received proving otherwise.

The responsibility of hazardous waste management and for its minimization lies with the Group Leader. Group leaders must verify that the potentially hazardous waste materials in their work areas are identified and that the Industrial Hygiene Group (HSE-5) is contacted if workers could be exposed while handling, treating, or disposing of such materials.

Group leaders must verify that their operating personnel are trained in Waste Management and follow those policies and procedures appropriate to their work.

6.15.2. Definition of Hazardous Waste

Hazardous chemical waste is any chemical or mixture of chemicals that is intended for disposal and is corrosive to living tissue; is toxic, flammable, carcinogenic, teratogenic, mutagenic, or infectious; or in any way poses a present or potential hazard to human health or the environment.

Hazardous waste is defined as materials that are either characteristic wastes or those specifically listed. Characteristic wastes are classified as follows:

- Ignitable—flammable materials with a flash point of less than 60°C (e.g., acetone and ethanol).
- Corrosive—materials that dissolve metal or other materials, or can burn skin and tissue (e.g., nitric acid and sodium hydroxide).
- Reactive—unstable materials that undergo rapid or violent chemical reaction (e.g., explosives and lithium hydride).

Listed wastes are as follows:

- D List—also defined EP Toxic, an extractive procedure for As, Ba, Cd, Cr, Pb, Hg, Se, Ag, and 6 pesticides.
- F List—halogenated and nonhalogenated solvents and cyanide wastes from nonspecific sources.
- K List—86 chemicals including waste water treatment sludges and explosive processing sludges from specific process sources.
- P List—120 acutely hazardous materials such as phosgene.
- U List—250 toxic chemicals such as benzene and toluene.

6.15.3. Temporary Storage of Hazardous Waste

HSE-9 is a Satellite Storage area and may accumulate up to 55 gallons of waste or 1 quart of acutely hazardous waste (P and U lists). The storage area must be labeled "Hazardous Waste Satellite Storage." HSE-9 has a properly designated locked chemical storage cabinet located on the south side of building OH-1 at TA-59 near the lunch room. The HSE-9 Chemical Waste Coordinator is responsible for the storage cabinet and for regularly inspecting all containers for leakage that are placed in it. All containers must be labeled "hazardous waste" with the contents identified and the date noted on which accumulation began.

Chemically contaminated rags and wipes are regulated waste if the material involved is spent solvents, heavy metals, cyanides, and/or other toxic chemicals. If the chemical involved is only regulated under the Resource Conservation and Recovery Act (RCRA) because of a characteristic, such as ignitability, then the rags are exempted from regulation. Rags, chemwipes, Q-tips, etc., that come into contact with regulated chemicals must be segregated from sanitary waste.

HSE-9 personnel have the following responsibilities in their everyday work with chemicals:

- Identify and document waste as it is generated.
- Keep lists of chemicals when placed in storage bottles.
- Keep metals and solvents segregated.
- Use the proper storage container and storage area.
- Take steps to reduce the amount of waste generated.

6.15.4. Chemical Waste Coordinator

A Chemical Waste Coordinator must be identified for each waste-generating group and will serve as the primary contact to the Waste Management staff. The HSE-9 Chemical Waste Coordinator must verify the safe use of chemical waste collection areas or cabinets and the predisposal treatment of the wastes, as determined appropriate by the Waste Management Group. This person is also the focal point for hazardous waste and minimization issues.

6.15.5. Disposal of Chemicals and Hazardous Waste

The Waste Management Group, HSE-7, disposes of hazardous waste (toxic, carcinogenic, explosive). When HSE-9 personnel determine that chemical wastes are ready for disposal, the HSE-9 Chemical Waste Coordinator will complete a Chemical Waste Disposal Request and send the form to Waste Management. Upon receipt of the form, Waste Management determines the packaging, handling, treatment, transportation, and disposal requirements, based on the type and quantity of the waste involved. HSE-9 may be required to complete some pre-disposal treatment/chemical analysis of the wastes before Waste Management will take over the disposal operations. Details on their requirements are given in the Health and Safety Manual, Section 10.

All waste must be properly packaged, with incompatible chemicals kept separate, in Waste Management-approved, leakproof containers to ensure its safe handling, transportation, and disposal. The containers must be monitored for radioactive contamination and clearly labeled as to the contents.

Radioactive waste must be properly packaged, labeled, and recorded on the radioactive dumpster log before it is placed in the radioactive waste container for subsequent transfer to HSE-7. The key to the radioactive waste dumpster is kept in the HSE-9 group office. Refer to SOP 85-172 "Disposal of Solid Radioactive Wastes at TA-59" for detailed information.

The HSE-7 Generator Interface Section has the following responsibilities:

- provide engineering and technical support,
- provide training and education,
- maintain a technical data base,
- provide support for storage, transportation, and disposal efforts, and
- take the lead in minimization efforts.

6.15.6. Hazardous Waste Minimization

Generation of chemical waste must be kept as low as reasonably achievable (ALARA). Whenever possible, unopened containers of chemicals should be returned to Chemical Stock (VWR) rather than to the Chemical Waste Coordinator for disposal.

RCRA requires that we no longer generate hazardous waste and then treat it. We must make every effort to reduce the amount initially generated. Waste minimization is the practice that reduces, avoids or eliminates the generation of hazardous waste at the source. This includes the reduction of mass and toxicity. Waste minimization methods are as follows:

- Abatement—substitution of new primary processes and material that alter the wastes and amounts generated.
- General Housekeeping—methods of reducing waste that address the operation of the system. This includes inventory control, process control, waste stream segregation, material management, and spill leak control.
- Reuse—direct reuse of waste streams without altering the stream.
- Recycling—reclamation of value from waste streams by the application of unit processors.
- Treatment—operations on the generated waste stream that reduce the mass, volume, or toxicity of the stream.

6.15.7. Recyclable Materials

Where feasible, chemical wastes should be recycled or salvaged according to the appropriate Laboratory procedures. Those materials listed below are prime recycling candidates.

- Elemental mercury
- Gas cylinders
- Batteries
- Oil or solvents

6.16. Hazardous Material Transport On-Site

6.16.1. Introduction

Shipping and packaging hazardous materials are regulated by federal laws and by DOE orders. An On-Site Transportation Manual has been written that specifies the responsibilities, authorities, requirements, and procedures needed for controlled on-site movement of hazardous materials and wastes

at the Laboratory. The manual applies to any on-site transportation of hazardous materials, including those shipments using roads not accessible to the public.

A copy of this manual is available for use by HSE-9 personnel at TA-35, TA-50, and TA-59. The manual is divided into five sections: administrative, radioactive materials, hazardous materials, explosives, and waste. Specifications are given for packaging, marking, labeling, documenting, transportation methods, and quality assurance.

6.16.2. Policy

The Laboratory requires that on-site transfers of hazardous material be made in a manner that

- Protects the health and safety of employees and the public;
- Protects the material itself;
- Prevents the release of such materials into the environment; and
- Ensures compliance with DOE requirements and regulations.

6.16.3. HAZPACT Coordinators

HSE-3 formed a Hazardous Materials Packaging and Transportation (HAZPACT) Section to provide technical support and assistance to Laboratory personnel involved in packaging hazardous materials for transport on-site and off-site. These personnel and their specialties are listed below. Call them for assistance.

Hazardous Materials Packaging Specialists

Nate King	7-4127	Administrative Radioactive Materials Radioactive Waste
Karen Balo	7-7391	Chemical/Radioactive Waste
Shirley O'Rourke	7-5115	Hazardous Materials
Humberto Martinez	7-6792	Explosives

6.17. Chemical Hazard Communication

6.17.1. Introduction

Activities at Los Alamos National Laboratory may require the use of processes or materials that could endanger the health and safety of personnel if proper precautions are not observed. AR 1-9 and 6-1 set forth the requirements for (1) identifying and evaluating the hazards of chemical,

biological, and physical agents associated with Laboratory operations and for (2) disseminating hazard information to employees exposed to those hazards. The group leader has the overall responsibility to verify that the requirements are met.

6.17.2. Program Elements

The basic elements of the Hazard Communication Program are

- identification and labeling of hazardous materials and operations,
- development, as needed, and maintenance of material safety data sheets (MSDS) for each hazardous material used in the workplace, and
- training of employees in the use of hazardous materials and the existence of hazardous operations in their work area.

An MSDS is written, printed, or electronically transmitted data on the hazards and properties of a particular material, including instructions for safe use.

6.17.3. Evaluation of Operations

The HSE-9 group leader and supervisors must ensure that personnel exposures to hazards are kept as low as reasonably achievable (ALARA). HSE-9 must notify their HSE Division contact before initiating or making major changes in operations involving hazards. HSE Division will help determine the hazards and appropriate control measures for such operations and will conduct follow-up, periodic surveys.

6.17.4. Hazard Notification and Training

HSE-9 employees receive the appropriate information and education on chemicals associated with their work area. They receive such information and education from their section leader upon initial assignment to the group, whenever their assignment changes in a manner that affects their potential for exposure to chemicals, and whenever new chemicals are introduced to the work area. Employees are informed of the location and availability of reference information, such as the ARs, required hazard inventory lists, and MSDS sheets.

Employees must be taught to detect hazards in the workplace, interpret MSDS sheets and other written hazard information, and use specified measures (for example, special work procedures, emergency response procedures, and protective equipment) to control exposure to hazards.

Such training shall include a review of applicable SOPs, specific portions of the Health and Safety Manual, and job-specific education administered by supervisors with assistance from HSE Division. Other appropriate sources of supplemental information shall be included, such as published reports, literature, videotapes, and reference materials.

Contractor employees are informed of the chemical hazards to which they may be exposed in the HSE-9 work areas by the respective section leaders and are advised of the recommended protective measures to reduce or eliminate worker exposure.

6.17.5. Training Support

The Health, Safety, and Environment (HSE) Division provides chemical hazard training for Laboratory managers and first-line supervisors who shall use this information in training those employees exposed to chemicals in the workplace.

6.17.6. Chemical Inventory

HSE-9 performed an initial inventory of approximately 8,000 chemicals. An annual inventory is conducted to account for all chemicals present in the workplace, including reagents, mixtures, solvents, and cleaners. Section leaders are responsible for ensuring that the inventory is kept current. Records of all past inventories are maintained. Employees have access to current inventories at any time during their work hours.

HSE-9 chemical inventory records are available in the Quality Assurance and Data Management Section. These records are auditable and include the following information:

- the identity of each chemical as it is referenced on an appropriate MSDS;
- the Chemical Abstracts Services (CAS) Registry number;
- the location(s) in which the chemicals are present; and
- the estimated quantity of the chemical used annually and the amount currently on hand.

6.17.7. Material Safety Data Sheets

Copies of the required hazard information sheets (MSDS) are maintained for each chemical in the workplace. This information is accessible to employees working in the area. An MSDS will be on file for each manufacturer whose material is present in the workplace, even if more than one manufacturer is supplying the same hazardous material. MSDSs for TA-59 will be available in the Quality Assurance and Data Management Section. TA-35 and TA-50 will have MSDSs for the chemicals located in their areas.

Industrial Hygiene initially distributes the available hazard information sheets to HSE-9 based on the initial inventory. HSE-9 shall obtain any remaining or new MSDSs from the manufacturer. Copies of MSDSs obtained by HSE-9 must be sent to HSE-5 and the Laboratory's main library.

6.17.8. Labeling of Hazardous Materials

Containers of chemicals used in the workplace shall be clearly labeled, tagged, or marked with the (1) identity of the chemical contained within and (2) appropriate hazard warnings. Chemicals transferred from the original container into smaller containers are not required to be labeled except for appropriate identification so long as their subsequent use is limited to the employee who performs the transfer.

In certain situations, the required label information may be contained in operating instructions or other written materials in lieu of affixing labels to individual containers. In such cases, the alternative method is explained to employees working with the chemicals, and the written materials must be readily accessible to those employees.

Section leaders must make sure that labels are kept legible from the time the hazardous materials arrive at the workplace to the time the materials are transferred or disposed of.

6.17.9. Procurement

As specified in AR 1-9, chemicals shall be procured and received only by personnel knowledgeable of the hazards imposed by those chemicals. The requestor must make note in the "Special Handling Information" block on the purchase request that the order involves chemicals, even if the requestor does not consider the chemicals to be hazardous.

6.17.10. Packaging and Transport

Chemicals shall be properly packaged and transported in government vehicles, not in official taxis or private vehicles. Transport of chemicals must be made in accordance with Laboratory regulations.

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7. LABORATORY FIRST AID

7.1. Introduction

Generally, treatment of an ill or injured individual should only be undertaken by appropriately trained personnel. In the event of an accident, do whatever is necessary to verify that the injured person is not in danger of further injury. Obtain emergency assistance by dialing 911. If the person is not severely injured and can be easily moved, help can be obtained at HSE-2. There are several people within the group trained in cardiopulmonary resuscitation. If possible, call one of them when this type of immediate aid is needed. A list of CPR trained personnel is posted in the group office, on the bulletin board outside of room 112, and in the section leaders office at TA-50.

7.2. Bleeding Aid

Severe bleeding is best controlled by the application of direct pressure on the wound site. Apply pressure by placing the palm of your hand on a compress (a thick pad of cloth) directly over the open wound. If a compress cannot be found, use your bare hand. Do not disturb clots formed within the pad. If blood soaks through the pad without clotting, use additional pads and continue applying pressure. If there is no evidence of a fracture and the wound is in the arm, hand, or leg, elevation of the wound will help stop the bleeding. Obtain assistance from HSE-2 at 7-7251.

7.3. Breathing Aid

Cardiopulmonary Resuscitation (CPR). Learn it, use it, save a life. The following information is presented as a review for HSE-9 personnel certified in CPR. This information does not take the place of CPR training.

7.3.1. Check for Unresponsiveness

Tap or gently shake the victim and shout, "Are you O.K.?" If no response shout "HELP!" (Someone nearby may be able to assist).

7.3.2. Airway Step

Place one hand on the forehead and push firmly backward. Place the other hand under the neck near the base of the skull and lift gently. Tip the head until the chin points straight up. This should open the airway. Place your ear near the victim's mouth and nose. LOOK at the chest for breathing movements, LISTEN for breaths and FEEL for breathing against your cheek (3 to 5 seconds). If no breathing occurs proceed to the next step.

7.3.3. Quick Step

Give 2 full breaths, one on top of the other. To do this, keep the head tipped and pinch the nose. Open your mouth wide, take a deep breath, and place open mouth over the victim's open mouth making a good seal. Now, give the 2 full quick breaths without waiting in between.

7.3.4. Check Step

Check the victim's carotid pulse for heartbeat (5 to 10 seconds). To do this, keep the head tipped with the hand on the forehead. Place the fingertips of your other hand on the adam's apple, slide your fingers into the groove at the side of the neck nearest you. Tell someone to phone for emergency assistance (911). If there is no pulse, find the correct hand position and position your body to give compressions.

7.3.5. Hand Position for Chest Compressions

- With your middle and index fingers find the lower edge of the victim's rib cage on the side nearest you.
- Trace the edge of the ribs up to the notch where the ribs meet the breastbone.
- Place the middle finger on the notch with the index finger next to it. Put the heel of the other hand on the breastbone next to the fingers.
- Put your first hand on top of the hand on the breastbone. Keep the fingers off the chest.

7.3.6. Chest Compressions

Push straight down without bending your elbows and maintain proper hand position. Keep knees shoulder width apart. Shoulders should be directly over victim's breastbone. Keep hands along midline of body. Bend from the hip, not the knees. Keep fingers off of the chest. Push down about 1 1/2 to 2 inches. Push smoothly.

7.3.7. Push 15-Breathe 2

Give 15 compressions without stopping, at a rate of 80 to 100 per minute, counting out loud, "One and two and three and four and five and six and seven and eight and nine and ten and eleven and twelve and thirteen and fourteen and fifteen and." Push down as you say the number and come up as you say "and."

Next, quickly tilt the victim's head back to lift the jaw, and give 2 full breaths to the victim the same way you gave the first 2 breaths.

Keep repeating—15 compressions, 2 breaths, 15 compressions, 2 breaths, and so on. The complete cycle of 15 compressions and 2 breaths should take from 11 to 14 seconds.

7.3.8. Recheck Pulse

After doing four cycles (or about one minute) of continuous CPR, you should check to see if the victim has a pulse. Do this after giving the 2 breaths at the end of the fourth cycle of 15 compressions and 2 breaths. Tilt the victim's head back and check the carotid pulse for 5 seconds. If there

is no pulse, give 2 breaths and continue CPR (compressions and rescue breaths). Repeat these pulse checks every few minutes.

If you do find a pulse, check for breathing for 3 to 5 seconds. If breathing is present, keep the airway open and monitor breathing and pulse closely. This means that you should look, listen, and feel for breathing while you keep checking the pulse. If there is no breathing, perform rescue breathing and keep checking the pulse.

NOTE: Do not practice chest compressions on people as it could cause internal injuries.

7.4. Burn Aid

Burns are classified by the depth of skin damage. First-degree burns show redness or discoloration of the skin, accompanied by mild swelling and pain. Second-degree burns show a red or mottled appearance, accompanied by blisters and extensive pain. Third-degree burns show deep tissue destruction and a white or charred look with complete loss of all layers of skin. There is usually less pain with serious burns because of destruction of the nerve endings. All burns should be checked by medical personnel.

7.4.1. First-Degree Burns

Submerge the burned area in cold water if possible or apply an ice pack. Apply a dry dressing if necessary. Go to HSE-2 to ensure there is no hidden damage.

7.4.2. Second-Degree Burns

Submerge the burned area in cold water or apply cold water liberally until the pain subsides. Do not break the blisters or remove any tissue. Do not apply an antiseptic preparation, ointment, or spray. Gently blot dry and go to HSE-2 for medical help.

7.4.3. Third-Degree Burns

Summon professional medical help. These burns are best treated by professionals. Do not submerge large burned areas or use cold water as this may increase shock. Cold packs may be used when the burns are to the hands, feet, or face. Do not remove charred clothing. Do not apply ointments, grease or other materials. Keep burned hands or feet elevated. Have the victim with a burned face sit up or prop him up and keep him under constant observation, particularly for breathing problems.

7.5. Electric Shock

Do not touch the victim of a disabling electric shock until you are certain that their body is no longer part of a live circuit. This can be accomplished by opening a switch or circuit breaker. If the victim is breathing, call 911. If the victim is not breathing, begin artificial respiration at once. If no pulse can be detected, initiate

CPR. Have someone who is not administering first aid call for help, or if no one is present, trip the fire alarm, then administer aid as quickly as possible.

7.6. Eye Aid

If a chemical accidently gets into the eye, immediately flush out the eye with water. This is accomplished with the eyewash fountains in each laboratory. In an emergency, water from a beaker can be poured into the eye. Be sure that water gets under the eyelid to wash the entire eye. The victim may need help holding the eye open. Continue washing for at least 15 minutes, or until directed to stop by emergency personnel.

Eyewash stations are to be flushed weekly for at least three minutes to ensure proper functioning and to purge the lines of any accumulated material. Cards are posted at each eyewash station to record the initials of the person flushing the eyewash station and the date that this function was performed.

7.7. Fracture Aid

Do not attempt to treat a fracture unless emergency assistance will not be available for a significant period of time or if it is necessary to transport the victim without emergency assistance. Do not attempt to set a fracture or to push back a protruding bone. If it is necessary to splint a fracture, use anything available to immobilize the fracture and the joints on both sides of the fracture. Do not manipulate or move the fractured area. Do not have the victim test for fracture by having him move or walk. Call HSE-2 at 7-7251.

7.8. Poisoning Aid

7.8.1. Conscious Victim

If the person is conscious and not convulsing, dilute the poison by giving water. If the victim vomits, save a sample for analysis. Get emergency assistance as soon as possible.

7.8.2. Unconscious Victim

Maintain an open airway and give artificial respiration or CPR if necessary. Do not give fluids or induce vomiting. If the victim is vomiting while unconscious, turn the head so the material drains and is not aspirated into the lungs. Save a sample of vomitus for analysis.

7.8.3. Convulsing Victim

Do not attempt to restrain the victim. Move surrounding objects to prevent further injury. After convulsions have stopped, place the victim in the prone position with the head turned to allow fluids to drain. Loosen clothing and watch for airway obstruction. Give artificial respiration or CPR if necessary. Do not force anything between the teeth. Do not give fluids. Do not induce vomiting. If vomiting occurs, save a sample of the vomitus for analysis.

7.9. Shock Aid

Shock is common after an accident and can be fatal as it results in the depression of all bodily functions. The symptoms are pale or bluish skin, moist or clammy skin, rapid (often faint) pulse, increased breathing rate (which may be shallow), weakness, and vomiting. In late stages the victim is apathetic and unresponsive. The eyes are vacant with dilated pupils.

Have the victim lie down and cover him/her with a blanket to prevent chilling. Do not add heat. If there is no head injury, elevate the feet a few inches. If the victim experiences breathing difficulty, lower the feet. Obtain medical assistance.

7.10. Skin Aid

If a hazardous chemical, such as sulfuric acid, is splashed on the skin, it must be washed away immediately. wash under the emergency shower and remove any contaminated clothing. Shield the victim from onlookers. Wash for 15 minutes or until directed to stop by emergency personnel. Emergency showers are located at intervals in the hallway.

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A Department of Energy
Environmental Cleanup Program

Reference Set for Analysis and Assessment Focus Area

Volume 2

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44019 Birkeland, P. W., 1984. *Soils and Geomorphology*, Oxford University Press, New York, New York. (Birkeland 1984, 44019)

49726 Broxton, D. E., and S. L. Reneau, August 1995. "Stratigraphic Nomenclature of the Bandelier Tuff for the Environmental Restoration Project at Los Alamos National Laboratory," Los Alamos National Laboratory Report LA-13010-MS, Los Alamos, New Mexico. (Broxton and Reneau 1995, 49726)

50121 Broxton, D. E., G. H. Heiken, S. J. Chipera, and F. M. Byers, Jr., June 1995. "Stratigraphy, Petrography, and Mineralogy of Bandelier Tuff and Cerro Toledo Deposits," in "Earth Science Investigations for Environmental Restoration—Los Alamos National Laboratory Technical Area 21," Los Alamos National Laboratory Report LA-12934-MS, Los Alamos, New Mexico, pp. 33–63. (Broxton et al. 1995, 50121)

52227 Broxton, D. E., R. T. Ryti, S. L. Reneau, C. Duffy, and P. A. Longmire, October 1, 1995. "Natural Background Geochemistry of the Bandelier Tuff and Other Rock Units, Los Alamos, New Mexico," in "Natural Background Geochemistry and Statistical Analysis of Selected Soil Profiles, Sediments, and Bandelier Tuff, Los Alamos, New Mexico," Los Alamos National Laboratory Report LA-UR-95-3468, Los Alamos, New Mexico. (Broxton et al. 1995, 52227)

52227 Longmire, P. A., E. V. McDonald, R. T. Ryti, S. L. Reneau, and P. M. Watt, October 1, 1995. "Natural Background Geochemistry and Statistical Analysis of Selected Soil Profiles, Los Alamos, New Mexico," in "Natural Background Geochemistry and Statistical Analysis of Selected Soil Profiles, Sediments, and Bandelier Tuff, Los Alamos, New Mexico," Los Alamos National Laboratory Report LA-UR-95-3468, Los Alamos, New Mexico. (Longmire et al. 1995, 52227)

52227 Reneau, S. L., P. A. Longmire, K. Campbell, and E. V. McDonald, October 1, 1995. "Geochemistry of Background Sediment Samples at Technical Area 39," in "Natural Background Geochemistry and Statistical Analysis of Selected Soil Profiles, Sediments, and Bandelier Tuff, Los Alamos, New Mexico," Los Alamos National Laboratory Report LA-UR-95-3468, Los Alamos, New Mexico. (Reneau et al. 1995, 52227)

53953 Ryti, R. T., P. Longmire, and E. McDonald, March 29, 1996. "Application of LANL Background Data to ER Project Decision-Making, Part I: Inorganics in Soils, Sediments, and Tuff," Los Alamos National Laboratory Report LA-UR-96-1534, Los Alamos, New Mexico. (Ryti et al. 1996, 53953)

54709 Broxton, D. E., D. Vaniman, S. J. Chipera, E. C. Kluk, and R. G. Warren, December 1, 1995. "Stratigraphy, Mineralogy and Chemistry of Bedrock Tuffs at Pajarito Mesa," in "Geological Site Characterization for the Proposed Mixed Waste Disposal Facility," Los Alamos National Laboratory Report LA-13089-MS, Los Alamos, New Mexico, pp. 5–30. (Broxton et al. 1995, 54709)

54948 Broxton, D. E., R. T. Ryti, D. Carlson, R. G. Warren, E. Kluk, and S. Chipera, March 20, 1996. "Natural Background Geochemistry of the Bandelier Tuff at MDA P, Los Alamos National Laboratory," Los Alamos National Laboratory Report LA-UR-96-1151, Los Alamos, New Mexico. (Broxton et al. 1996, 54948)

55115 Longmire, P., S. Reneau, P. Watt, L. McFadden, J. Gardner, C. Duffy, and R. Ryti, May 1, 1996. "Natural Background Geochemistry, Geomorphology, and Pedogenesis of Selected Soil Profiles and Bandelier Tuff, Los Alamos, New Mexico," Los Alamos National Laboratory Report LA-12913-MS, Los Alamos, New Mexico. (Longmire et al. 1996, 55115)

55532 McDonald, E., R. T. Ryti, S. L. Reneau, D. Carlson, and J. Harris, September 1997. "Natural Background Geochemistry of Sediments, Los Alamos National Laboratory," (draft), Los Alamos National Laboratory report, Los Alamos, New Mexico. (McDonald et al. 1997, 55532)

56135 Incorrect citation in Yu et al. 1993, 56135; see 56895, p. 62.

56179 Gilbert, R. O., 1987. *Statistical Methods for Environmental Pollution Monitoring*, ISBN 0-442-23050-8, Van Nostrand Reinhold, New York, New York. (Gilbert 1987, 56179)