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APPENDIX 20

**TELEDYNE BROWN ENGINEERING
ENVIRONMENTAL SERVICES**

Procedure TBE-2008

Gamma Emitting Radioisotope Analysis

Rev. 0

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Approved by:

Keith O. Jeter, Operations Manager

Date _____

Martin R. Keller, Quality Assurance
Manager

Date _____

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DETERMINATION OF GAMMA EMITTING RADIOISOTOPES

1.0 SCOPE & APPLICABILITY

- 1.1 This procedure presents the methods for determining gamma emitting radioisotopes by high purity germanium detectors with high resolution spectrometry in specific media: air particulate filters, charcoal filters, milk, water, vegetation, soil/sediments, biological media, etc.
- 1.2 No chemical separation and purification procedures are required for gamma ray analysis. This is a nondestructive analysis, and after completion of the assay, the aliquot can be used for other analyses. However, to identify a specific target gamma emitter, chemical separation can be employed to isolate the desired gamma emitter(s) when other gamma emitters are present in high concentrations (see Section 1.4).
- 1.3 For water samples, a procedure is included to pre-concentrate the samples by evaporation for greater sensitivity. Advantages gained include:
- Permits the analysis of more than one liter of water in comparison to the restriction of one liter maximum for the standard Marinelli geometry
 - Filter geometry is significantly a more efficient geometry since the sample is concentrated directly in front of and close proximity to the detector
- 1.4 For in-plant samples, a radiochemical method that includes chemical separation and purification is presented for determining the Ce-141 and Ce-144 activities of in-plant samples that have high concentrations of other gamma emitters.

2.0 SUMMARY OF METHOD

- 2.1 GEOMETRIES. Each sample to be assayed is put into a standard geometry for gamma counting such as 1-liter wrap-around Marinelli containers, 300 mL or 150

mL bottles, charcoal cartridge or 2-inch filter paper source geometries.

Calibration and counting efficiencies of the gamma counting system for these geometries must have been determined with standard (known) radionuclide activity traceable to the National Institute of Standards and Technology.

To improve the sensitivity for measurement of gamma emitting radionuclides in water matrices, the sample can be evaporated from any known volume of one liter or greater and the residue collected in a standard filter geometry.

2.2 COUNTING. Samples are counted on large (>55 cc volume) germanium detectors connected to dedicated data acquisitions and data computation systems. All resultant spectra are stored electrically.

2.3 CALCULATION. The analysis of each sample consists of calculating the specific activities of all detected radionuclides or the detection limits from a standard list of nuclides. If water samples were pre-concentrated by evaporation, the specific activities or detection limits are divided by the volume of water represented in the filter geometry.

2.4 IN-PLANT SAMPLES FOR CE-141/CE-144. The radiochemical isolation of cerium reduces the gamma counting background and improves the sensitivity of analysis by a factor of 100 or more compared to direct gamma spectral analysis. This method is an adaptation of the Los Alamos Collected Radiochemical Procedures La-1721, 3rd edition, September 1967.

2.4.1 Solid samples are leached in acid, then filtered. Aqueous samples need no special preparation. Stable cerium carrier is added to the sample, then cerium is purified by precipitating first as fluoride, then as hydroxide, and finally as oxalate. The cerium oxalate is collected by vacuum filtration, dried and weighed to determine chemical yield. The precipitate is mounted on a nylon planchet and is covered with Mylar film.

2.4.2 The sample planchet is analyzed on a high resolution germanium detector. Ce-141 is inferred by its emission at 145 keV (48.4% gamma abundance). Ce-144 is inferred by its emission at 134 keV (10.8% gamma abundance). Results are calculated using a counting efficiency curve derived by analyzing multiple nuclide standards prepared in the same counting geometry.

3.0 DEFINITIONS

3.1 MSDS – Material Safety Data Sheet

3.2 NIST – National Institute of Standards and Technology

3.3 TBE-ES – Teledyne Brown Engineering – Environmental Services

4.0 HEALTH & SAFETY WARNINGS

4.1 At a minimum, personnel performing this procedure are required to wear the following protective equipment: laboratory coats, safety glasses, and disposable gloves.

4.2 Appropriate precautions, as specified in the Laboratory Radiation Protection Program (RPP) Manual, will be adhered to when handling radioactive material.

5.0 CAUTIONS – N/A

6.0 INTERFERENCES – N/A

7.0 PERSONNEL QUALIFICATIONS

Analysts performing this procedure must be trained, qualified, and certified in accordance with the TBE-ES Quality Control Manual IWL-032-365, Sections 2.2 through 2.9 inclusive.

Analysts in training may perform this procedure only under the direct supervision and observation of a senior technician certified to perform this procedure.

8.0 EQUIPMENT & SUPPLIES

8.1 Gamma-Ray Spectrometer consisting of high resolution germanium detectors connected to Nuclear Data acquisition and data computation systems. For each detector, 2048 channels (1 KeV per channel) or 4096 channels (0.5 KeV per channel) are assigned for pulse height analysis).

8.2 Standard sample container geometries, as appropriate:

- 1-Liter wrap around Marinelli containers
- 300 mL or 150 mL bottles
- 2-inch filter paper for air particulates
- Charcoal cartridges
- Nylon planchets (see Section 8.6)

8.3 Evaporation supplies for filter geometry

- Beakers: 1, 2, or 4-liter graduated
- Hot plate
- 2-inch stainless steel planchet
- Kimwipes, or equivalent
- Marking pen, to write on beaker and planchets
- Fiber sample trays
- Heat lamps, Heat hood
- Parafilm
- Paper envelope to store prepared planchets

- HNO_3 , concentrated, in a dropping bottle
- Distilled water in a wash bottle
- Laboratory aerosol, dispensable by drop

8.4 In-Plant Samples Ce-141/Ce-144: Preparation

8.4.1 Aqueous

- Centrifuge tubes, 50-mL disposable
- Pipets, 10-mL disposable and 1-mL Eppendorf or glass volumetric
- Rubber pipet bulb
- pH paper
- Hydrochloric acid (HCl) to adjust pH if needed
- Deionized water, in a wash bottle
- Cerium carrier solution, standardized

8.4.2 Resins and other solids

- Beakers, 150-mL
- Gravity filtration apparatus and glass fiber filters
- Balance, analytical
- Hood
- Spatula
- Heat lamps or drying oven
- Poly bottles
- Pipets, disposable (10-mL)
- Centrifuge tube, 50-mL plastic and centrifuge tube rack
- Hydrochloric acid (HCl), 6 M: for resins
- Nitric acid (HNO_3), 8 M: for non-resin solids

- Cerium carrier solution, standardized

8.5 In-Plant Samples Ce-141/Ce-144: Chemical Separation & Purification

- Glass rod
- Hot water bath: 250 or 400-mL beaker half full of water on a moderate hot plate
- Deionized water, in a wash bottle
- Hydrofluoric acid (HF), conc. **!! Extreme Hazard !! No Skin Contact !!**
- Boric Acid (H_3BO_3), saturated solution
- Nitric acid (HNO_3), concentrated
- Sulfuric acid (H_2SO_4), 2 M
- Ammonium oxalate, saturated

8.6 In-Plant Samples Ce-141/Ce-144: Mounting the Precipitate

- Filter paper, 2.8 cm No. 42 ashless disc
- Vacuum filtration apparatus
- Petri dishes, 4-way partitioned
- Hot air oven
- Dessicator
- Balance, analytical
- Spatula
- Gummed labels
- Nylon planchets, 2-inch with Mylar film and nylon ring
- Scissors or razor blade
- Deionized water
- Ethanol

9.0 PROCEDURE

9.1 Detection Capability

Gamma ray spectroscopy, using a germanium detector, provides a high resolution method of distinguishing many gamma emitting nuclides in a single sample.

Each of the most commonly observed nuclides listed in the ensuing Table 9.1 has at least one gamma ray with a unique energy. Consequently, each nuclide in the Table may be identified in the presence of any or all of the others. The Table 9.1 also lists the nominal detectable limits for three of the standard sample container geometries.

**Table 9.1A Gamma Spectroscopy Detection Sensitivities¹
 by High Resolution Germanium for Environmental Samples**

Nuclide	Milk and Water ² (pCi/L)	Animal, Fish, Soil Vegetation, etc. (pCi/g)	Filters (pCi/total filter)
Be-7	50	0.2	20
K-40	80	0.4	50
Mn-54	5	0.02	2
Co-58	5	0.02	2
Fe-59	10	0.04	3
Co-60	5	0.02	2
Zn-65	10	0.04	5
Zr-95-Nb-95	5	0.04	3
Ru-103	5	0.02	2
Ru-106	50	0.2	20
I-131	15	0.1	4
Cs-134	5	0.02	2
Cs-137	5	0.02	2
Ba-140/La-140	10	0.2	3
Ce-141	10	0.1	3
Ce-144	40	0.2	20
Ra-226	80	0.1	10
Th-228	10	0.02	10

¹The detection limits are referenced to the count time and are based on two standard deviations of the background statistics.

²For water samples that have been pre-concentrated by evaporation onto a planchet, divide the values by the volume of water represented in the filter geometry.

Table 9.1B Ce-141/Ce-144 Minimum Detectable Activity (MDA)

Matrix	MDA ¹	Sigma Level ²	Sample Volume	Chem Yield	Counting Interval (hour)	Counting Efficiency (cpm)	Backgd (cpm)
Ce-141 ³	7x10 ⁻⁶ μCi/mL	4.66	10 mL	0.80	6		
	4x10 ⁻⁵ μCi/g	4.66	2 g	0.80	6		
Ce-144 ⁴	3x10 ⁻⁵ μCi/mL	4.66	10 mL	0.80	6		
	2x10 ⁻⁴ μCi/g	4.66	2 g	0.80	6		

¹Assumes there is no delay between collection and counting

²Sigma multiplier will be 4.66 unless otherwise specified by the client

³ Half-life for Ce-141 is 32.5 days; therefore, delay in counting would significantly increase the MDA

⁴ Half-life for Ce-144 is 284 days; therefore, delay in counting would increase the MDA

9.2 Sample Selection

9.2.1 Using the sample receipt form with the TBE-ES sample number, locate the sample (or sample group) in the sample receiving and storage room. Sign for the samples on the Receiving Room Log and return with them to the environmental laboratory.

9.2.2 Begin filling out the Radiochemical Preparation Logbook, entering the customer name, the sample numbers in order, the desired analyses, sample type, collection dates, the sample preparation date and the initials of the analyst.

After processing all samples within the sample group, begin filling out the Radiochemical Work Sheet – Gamma Spectroscopy. Using the laboratory logbook as a guide, fill in the customer name, collection date, sample type, analyst's initials, preparation date and aliquot used. Write the sample number of each sample in numerical order, and indicate the desired analysis (gamma spec).

9.2.3 Make an entry in the Gamma Spec laboratory logbook showing customer name, sample number, sample type, collection dates and desired analyses.

9.3 Sample Preparation

A laboratory sample for this SOP is defined as the material collected for analysis. A test source is prepared from laboratory sample material for purpose of determining its radioactive constituents. This is accomplished by putting the laboratory sample in a geometry suitable for the counting instrument, in this case a standard geometry that is user-friendly to the gamma spectrometer. The geometries used for the test source should be identical to the geometry of the calibration source, to the extent possible.

Important considerations in preparing test sources for gamma-ray spectrometry are geometry (shape), size, and homogeneity (uniformity) of source.

9.3.1 MILK AND WATER: Load environmental water and milk samples into 1-liter Marinelli containers.

9.3.2 WATER, larger volumes: For water volume exceeding the capacity of the 1-liter Marinelli, evaporate and mount the residue on a 2-inch stainless steel planchet, as follows:

9.3.2.1 Mark the sample number with a laboratory marking pen onto a clean, graduated 1, 2 or 4 liter beaker.

9.3.2.2 Shake the sample container to distribute any particulate matter evenly. Decant 1 liter or more of sample into the beaker and record the sample volume (and customer name and sample identification number) on the beaker with a marking pen.

9.3.2.3 Add approximately 1 mL concentrated HNO₃ to the sample from a dropping bottle. Place the beaker on a hot plate under the hood in the Gamma Preparation Room and set the hot plate for approximately 200°F temperature.

9.3.2.4 Evaporate the sample until the volume is reduced to 1-5 ml. Take care to reduce hot plate temperature as the sample volume

decreases in order to avoid loss by spattering from the beaker.
Remove from hot plate.

- 9.3.2.5 Prepare a 2-inch stainless steel planchet for each water sample by first wiping it clean with a Kimwipe. Write sample number, customer name, and volume on the back of planchet with a marking pen.
- 9.3.2.6 Transfer the solution from each sample beaker to its correspondingly numbered planchet. Wash the beaker sparingly with deionized water using a wash bottle and collect the washings in the planchet.
- 9.3.2.7 Place the filled planchets in the fiber sample tray under heat lamps in the Light Hood. Add 1 drop of laboratory aerosol to each planchet. Evaporate to dryness. Remove and allow to cool.
- 9.3.2.8 Stretch parafilm over the planchet.
- 9.3.2.9 Insert the planchet into a new clean paper envelope on which the sample number, customer name, and volume have been inscribed.
- 9.3.3 AIR PARTICULATE FILTERS: Position two-inch diameter filter papers in front of the detector without change in geometry.
- 9.3.4 VEGETATION AND BIOLOGICAL MEDIA, e.g., food crops, fish, soils, etc.: Load into tared 300 or 150 mL plastic bottles or 1-liter Marinelli containers. Determine and record the net weight of the sample.
- 9.3.5 CHARCOAL CARTRIDGES: Position charcoal cartridges on the face only of the detector or on the face of the detector and up to four (depending on the number of charcoal cartridges in the weekly set) around

the cylindrical surface of the detector. If I-131 is observed, individually recount each cartridge, positioned on the face of the detector.

9.3.6 IN-PLANT SAMPLES FOR CE-141/CE-144: Refer to Appendix A for sample preparation and chemical separation and purification.

9.4 Calibration of Equipment For Gamma Ray Spectroscopy

The standard sample container geometries are the 1-liter Marinelli container, 300 mL and 150 mL polyethylene bottles, 2-inch diameter filter paper, a charcoal cartridge, and a 2” stainless steel planchet. Mixed gamma ray standards traceable to the National Institute of Science and Technology or best available are used to calibrate the various standard geometries.

Each standard is initially counted on each germanium detector and an efficiency versus energy curve is determined for each geometry for the energy range of approximately 50 KeV to 2 MeV.

On an on-going basis, once a week the check source standard is counted on each detector for energy, efficiency, calibration and resolution. A more detailed calibration procedure is described In PRO-042-44, “Calibration of Gamma Ray Spectrometers.”

9.5 Sample Counting

9.5.1 Verify that the samples contain the same sample numbers as on the accompanying Radiochemical Work Sheet—Gamma Spec.

9.5.2 Write counting sequence numbers on the work sheet following the order that the sample numbers appear on the sheet. Begin with the number 1 if starting a new sample counting group; otherwise, use the number which follows the last sequence number assigned.

- 9.5.3 Write the counting start date and the number of the gamma-ray spectrometer used on the Radiochemical Work Sheet.
- 9.5.4 Measure an aliquot of sample in a standard geometry (one that has been calibrated). Record the amount on the work sheet.
- 9.5.5 Place the standard geometry with the sample aliquot on a shielded Ge(Li) detector and gamma count for a period of time that will meet the required sensitivity of measurement.
- 9.5.6 After the counting period the Nuclear Data system performs a peak search and identification. Print the gamma spectrum and/or store the spectrum on the appropriate computer-compatible device.
- 9.5.7 Calculate the radioactivity of the gamma emitters present in the sample.
- 9.5.8 For greater counting efficiency, reduce the size of the test source, as in Step 9.3.2. This will allow a greater amount of laboratory sample to be counted in a more favorable geometry.

9.6 Calculation of the Sample Activity or of the MDA

- 9.6.1 The Nuclear Data system performs a peak search and identification of all photopeak energies. The photopeak regions of the spectrum are integrated and the area under the baseline continuum is subtracted to determine the true photopeak area. Isotopes are identified by their appropriate photopeaks, and ratios to each other when more than one gamma photon is emitted by an isotope in the sample.
- 9.6.2 Radionuclide concentrations, A , (or detection limit based on the background if no peak is observed) are calculated by for a library of isotopes in pCi:

$$A = \frac{C}{2.22} \times BEV, \text{ where:}$$

- C = net count rate, cpm, in the peak area above the baseline continuum
- B = the gamma ray abundance of the radionuclide being measured (gammas/disintegration)
- E = detector efficiency (counts/gamma) for the particular photopeak energy being considered
- V = volume (or mass) of sample aliquot used (liters or gram)
- 2.22 = conversion factor from dpm/pCi

9.6.3 The calculation includes the efficiency of the detector for each gamma ray energy and for the sample geometry, the percent abundance for the gamma ray, the sample size, length of count, and references the results to the collection date.

9.6.4 The results are typed by a line printer and then transcribed onto a computer compatible format and manual data entry or directly by tape into the Interim Report system.

10.0 DATA AND RECORDS MANAGEMENT

10.1 All laboratory data and ancillary information shall be documented in bound laboratory logbooks or appropriate worksheets in permanent ink. Appropriate supervisory personnel shall review logbook entries and worksheets as required by the TBE-ES Quality Assurance Program.

10.2 Corrections to recorded data in logbooks or on worksheets shall be noted by drawing through the incorrect data with a single line and recording the date of the correction and the initials of the person making the correction. The correct data will be recorded in an unambiguous location in the immediate proximity of the incorrect data.

11.0 QUALITY CONTROL & QUALITY ASSURANCE

- 11.1 Sample duplicates shall be run to meet client requirements or, at a minimum, as required by the TBE-ES laboratory QC program.
- 11.2 Analysis blank and spikes shall be run to meet client requirements or, at a minimum, as required by the TBE-ES laboratory QC program.
- 11.3 A matrix spike consisting of a sample spiked with an appropriate standard (NIST traceable when possible) shall be run to meet client requirements.
- 11.4 If any batch control sample fails laboratory established quality control criteria (IWL-032-365, Section 9.1.2) or fails to meet specific client contract requirements, the samples comprising the controlled batch shall be reanalyzed.
- 11.5 Alpha and beta counters used in this analysis shall be controlled as set out in PRO-032-27.
- 11.6 The Laboratory Operations Manager or designee will interpret control charts. Five percent of the plotted values are expected to fall outside the $+2\sigma$ precision band on statistics alone. If a check source falls outside the $\pm 3\sigma$ precision band, another reading of the check source shall be made. If the second reading also falls outside the $\pm 3\sigma$ band, the counter is judged to be out of control & shall be taken out of service.
- 11.7 A counter control chart may show trends without being out of control by the above criteria. The Laboratory Operations Manager or a qualified designee shall interpret these trends and take corrective action. It is good practice to investigate trends which approach a 2 sigma control line. The instrument maintenance log shall be used to document the occurrence and interpretation of trends, and any corrective action taken.
- 11.8 When a counter is out of control, the Laboratory Operations Manager or the person he/she designates will examine the check source for defects. Re-

calibration of the counter may be necessary. New calibrations should be compared to previous calibrations to identify major changes in counter operations. Corrective action shall be documented in the maintenance log for that instrument.

- 11.9 When a counter is out of control for a given analysis, it may not be used for that analysis. A label must be placed on the instrument indicating its status.

12.0 REFERENCES

- 12.1 EPA-600/4-80-032, Prescribed *Procedures for Measurement of Radioactivity in Drinking Water*, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio, August 1980. Procedures, except SM-19-7110B, are based on Method 901.1 "Gamma Emitting Radionuclides," augmented for non-aqueous matrices by TBE-ES technical personnel.

A1 In-Plant Sample Ce-141/Ce-144 Preparation and Chemical Separation and Purification

A1.1 Sample Preparation

This section describes how samples are aliquoted and prepared for chemical separations. Wear a laboratory coat, disposable gloves and safety glasses while carrying out the steps below.

A1.1.1 Aqueous Samples

- A1.1.1.1 Write the Teledyne sample number or login number on a 50 ml disposable plastic centrifuge tube using a laboratory marking pen. Also write the analysis on the tube.
- A1.1.1.2 Shake the sample container to mix, then withdraw 10 ml of sample using a disposable pipet and rubber bulb. Transfer the liquid to the labeled sample tube. Different aliquots may be used according to sample availability or desired detection limit. Write the measured aliquot in the laboratory data book.
- A1.1.1.3 Test the sample for acidity by dipping a stirring rod into it, then touching to pH paper. If the sample is not acidic, add several drops HCl from a dropping bottle, then stir and test again. Add deionized water from a wash bottle to bring the volume near 20 ml.
- A1.1.1.4 Add 1.00 ml of standardized cerium carrier solution (nominally 10 mg Ce/ml) to the sample using an Eppendorf pipet or a glass volumetric pipet. Proceed to Section 9.4 for chemical separation procedures.

A1.1.2 Resin Samples

- A1.1.2.1 Resin samples are leached in 6M HCl without heating, then are filtered. The filtrate is diluted to 100 ml, then measured aliquots are taken for various analyses.
- A1.1.2.2 Write the Teledyne sample number on a new 150 ml beaker. Obtain the tare weight of the beaker using the analytical balance. Record this figure in the laboratory notebook along with the client name, sample number and sample type.
- A1.1.2.3 Working in a hood, use a laboratory spatula to scoop a representative aliquot of the sample from its container into the labeled beaker. The size of the aliquot will vary according to sample availability, overall sample activity and desired detection limit.
- A1.1.2.4 Reweigh the sample beaker and record this gross weight in the laboratory notebook. Subtract the beaker tare weight and record the sample aliquot weight. Also note whether the sample is dry or wet.
- NOTE: If the sample is wet and client wants results on a dry basis, place the sample beaker under heat lamps or in a drying oven and dry it before taking the final weight. In rare cases the client wants results on both a wet and dry basis. In these cases take a final weight on both a wet and a dry basis.
- A1.1.2.5 Working in a hood, pour 6 M HCl from a beaker into the sample beaker, covering the sample with about 75 ml of liquid. Allow the sample to leach for 2-5 hours without heating.
- A1.1.2.6 Fold a glass fiber filter in quarters and place in a plastic funnel. Place the funnel in the mouth of a new poly bottle which has been marked at an appropriate volume level and which has been labeled

with the client name and sample number. Filter the sample into the bottle, washing the resin with 6 M HCl and with water. Discard the filter and resin into a radioactive solid waste container.

A1.1.2.7 Add deionized water to the bottle, filling to the volume mark. Screw on the cap and shake to mix.

A1.1.2.8 Use a disposable pipet to draw a measured aliquot (usually 10 ml) from the sample bottle, transferring it to a 50 ml plastic "C" tube labeled with the sample number and the analysis. Record the measured aliquot and the dilution information in the laboratory notebook.

A1.1.2.9 Using a calibrated fixed or adjustable volume pipet, add 1.00 ml standardized cerium carrier (nominally 10 mg Ce/ml) to the sample. Proceed to Section 9.4 for chemical separation.

A1.1.3 Other Solid Samples

Solid samples other than resin are leached in 8M HNO₃ with heating, then are filtered. The filtrate is diluted to an appropriate volume, then measured aliquots are taken for various analyses.

A1.1.3.1 Write the Teledyne sample number on a new 150 ml beaker. Obtain the tare weight of the beaker using the analytical balance. Record this figure in the laboratory notebook along with the client name, sample number and sample type.

A1.1.3.2 Working in a hood, use a laboratory spatula to scoop a representative aliquot of the sample from its container into the labeled beaker. The size of the aliquot will vary according to sample availability, overall sample activity and desired detection limit.

A1.1.3.3 Reweigh the sample beaker and record this gross weight in the laboratory notebook. Subtract the beaker tare weight and record the sample aliquot weight. Also note whether the sample is dry or wet.

NOTE: If the sample is wet and client wants results on a dry basis, place the sample beaker under heat lamps or in a drying oven and dry it before taking the final weight. In rare cases the client wants results on both a wet and dry basis. In these cases take a final weight on both a wet and a dry basis.

A1.1.3.4 Working in a hood, pour 8 M HNO₃ from a beaker into the sample beaker, covering the sample with about 50 ml of liquid. Place the beaker on a moderate hot plate (setting near 2) and allow the sample to leach for 2 hours or longer, adding deionized water as necessary. Remove the sample beaker and allow to cool.

A1.1.3.5 Fold a glass fiber filter in quarters and place in a plastic funnel. Place the funnel in the mouth of a new 150 ml poly bottle which has been marked at an appropriate volume level and which has been labeled with the client name and sample number. Filter the sample into the bottle, washing the solids 8 M HNO₃ and with water. Discard the filter and resin into a radioactive solid waste container.

A1.1.3.6 Add deionized water to the bottle, filling to the volume mark. Screw on the cap and shake to mix.

A1.1.3.7 Use a disposable pipet to draw a measured aliquot (usually 10 ml) from the sample bottle, transferring it to a 50 ml plastic centrifuge tube labeled with the sample number and the analysis. Record the measured aliquot and the dilution information in the laboratory notebook.

A1.1.3.8 Using a calibrated pipet, add 1.00 ml standardized cerium carrier (nominally 10 mg Ce/ml) to the sample. Proceed to Section 9.4 for chemical separations.

A1.2 Chemical Separation and Purification

This section describes the isolation of cerium by precipitating the fluoride, then the hydroxide, then the oxalate. Wear a laboratory coat, safety glasses and disposable gloves while carrying out the following steps. **Exercise extreme caution when working with HF.**

A1.2.1 Precipitation of CeF₃

A1.2.1.1 Add deionized water from a wash bottle to the sample tube to bring the liquid level to near 20 ml.

A1.2.1.2 Wearing a laboratory coat, disposable gloves and a face shield or safety glasses with side shields, and **exercising extreme caution**, add approximately 3 ml concentrated HF from its plastic squeeze container to the sample tube. Stir with a glass rod, but do not leave the rod in the sample tube. Using a deionized water wash bottle, rinse the rod while withdrawing it from the tube.

A1.2.1.3 Make a hot water bath by partly filling a 250 ml or 400 ml beaker with tap water and heating on a moderate hot plate (setting near 3). Place the sample tubes in the bath to precipitate CeF₃. Remove sample tubes from the beaker and place in a rack to cool.

A1.2.1.4 Equalize the liquid levels in the sample tubes and centrifuge for 10 minutes. Decant and discard the supernate into a radwaste beaker.

A1.2.1.5 Rinse the precipitate by adding approximately 5 ml deionized water to the sample tube from a wash bottle. Stir with a glass rod, then rinse the rod while withdrawing it from the tube. Equalize liquid

levels and centrifuge again for 10 minutes. Decant and discard the supernate into a radwaste beaker.

A1.2.1.6 Dissolve the CeF_3 precipitate by adding 1 ml saturated boric acid (H_3BO_3) solution, then 1 ml concentrated HNO_3 . Stir (and heat in a water bath if necessary) until the precipitate dissolves.

A1.2.2 Precipitation of $\text{Ce}(\text{OH})_3$

A1.2.2.1 Add deionized water from a wash bottle to the sample tube and bring the liquid level to near 20 ml. Stir and add concentrated NH_4OH from a dropping bottle until the solution is basic and particles of $\text{Ce}(\text{OH})_3$ form. Do not heat.

A1.2.2.2 Centrifuge the sample for 10 minutes immediately after the preceding step. Discard the supernate into a radwaste beaker.

A1.2.2.3 Add 5 ml 2M H_2SO_4 to the sample tube and stir with a glass rod to break up the precipitate. Add 5 ml deionized water and heat in a water bath for a half hour or longer to dissolve the precipitate. Some particles may remain undissolved.

A1.2.3 Precipitation of $\text{Ce}_2(\text{C}_2\text{O}_4)_3$

A1.2.3.1 With the sample tube still in the water bath from the previous step, add approximately 20 ml saturated ammonium oxalate from a beaker. Stir, then continue heating in the water bath for 20 minutes or longer to precipitate cerium oxalate.

A1.2.3.2 Remove the sample tube and place it in a rack to cool. Proceed to section 6.0 to mount this final precipitate for gravimetric yielding and gamma counting.

A1.3 Mounting the Precipitate

- A1.3.1 Prepare a 2.8 cm No. 42 ashless filter paper disc for each sample by mounting it on a vacuum filtration apparatus and rinsing with deionized water and ethanol.
- A1.3.2 Place the prepared discs in 4-way partitioned petri dishes which have been marked with sequence numbers (one number per partition, beginning with 1). Write corresponding sequence numbers beside each sample number entry in the laboratory data book. The sequence number indicates the correspondence between a filter and the sample which will be mounted on it.
- A1.3.3 Place petri dishes containing prepared filters in an approximately 100°C hot air oven for 10 minutes or longer to dry. Remove petri dishes and allow to cool in a desiccator.
- A1.3.4 Weigh the filter discs on the analytical balance using a clean spatula to handle them. Record this tare weight beside the corresponding sequence number and sample number in the laboratory data book. Take care to replace each filter after weighing in the numbered petri dish partition from which it came.
- A1.3.5 Using a laboratory spatula, take the tared filters in sequence number order and transfer to the vacuum mounting apparatus. Wet with deionized water. Using the laboratory data book to establish the correspondence between sequence number and sample number, filter each sample from its centrifuge tube onto its corresponding filter disc.
- A1.3.6 Rinse precipitate on filter with deionized water and then with ethanol. Transfer each filter from vacuum mounting apparatus back to the numbered petri dish partition from which it came. Place petri dish in a 105-120°C hot air oven for 10 minutes or longer.

- A1.3.7 Remove petri dish and allow to cool in desiccator. Weigh filters on the analytical balance within 5 minutes of removing from the desiccator. Write the weights beside the corresponding sequence numbers in the laboratory data book. Return each filter to its original partition in the petri dish.
- A1.3.8 Write a gummed label for each sample designated Ce-141, Ce-144, or both. All labels must contain the sample number and customer. Fix each label to the back of a nylon planchet.
- A1.3.9 Using the laboratory data book to establish the correspondence between sample number and sequence number, transfer each filter to its planchet and fix in place with a 2-inch piece of Mylar film and a nylon ring. Trim excess Mylar with scissors or a razor blade.
- A1.3.10 Subtract filter tare weight from final weight for each sample. Write the difference (mount weight) in the laboratory data book. Divide the mount weight by the corresponding carrier yield figure (written on the cerium carrier flask) to obtain chemical yield. Enter the cerium yield percentage in the laboratory data book.
- A1.3.11 Using the laboratory data book as a guide, begin filling out a Radiochemical Work Sheet for each sample. Enter sample number, company name, analysis (Ce-141, Ce-144, or both), collection date, aliquot used, sample type, chemical yield, and analyst's initials.
- A1.3.12 Submit finished planchets and work sheets to the Counting Room for radioassay.
- A1.3.13 Proceed to: Section 9.4.