RESL TECHNICAL PROCEDURE

CHEM-TP-A.20

ACTINIDE SEPARATIONS FOR ALPHA SPECTROMETRY USING NEODYMIUM FLUORIDE COPRECIPITATION

Preparer:	Date 5-6-99	Independent Review:	Date 5/10/99
ES&H Review:	Date 5/10/99	Radiological Controls Review:	Date 5/11/99
QA Review:	Date 5/11/99	Supervisor Approval:	Date 5-10-99

TITLE: CHEM-TP- A.20, ACTINIDE SEPARATIONS FOR ALPHA SPECTROMETRY USING NEODYMIUM FLUORIDE COPRECIPITATION

PURPOSE

The purpose of this procedure is to provide a method in which air filters, soil, water, and vegetation samples can be analyzed for Th, U, Pu, and Am. This procedure supersedes ACB-TP-A.20 (Rev 1).

APPLICABILITY

This procedure is applicable to the analysis of soil, water, vegetation, or air filter samples for uranium, plutonium, americium, or thorium. However, this procedure will not work in the presence of neptunium because neptunium is incompletely separated from the other nuclides.

RESPONSIBILITIES

RESL staff responsible for implementing this procedure are:
Radiochemist(s)

DEFINITIONS

None.

PROCEDURE

1 **ABSTRACT** Air filters, 10-g soil, 500-mL water, and 5-g vegetation-ash samples are fused in potassium fluoride followed by a pyrosulfate fusion. The flux is dissolved in dilute hydrochloric acid and thorium, uranium, plutonium, and americium are coprecipitated on barium sulfate. These actinides can be directly coprecipitaed from water samples on Barium sulfate, if total decomposition is not necessary. The barium sulfate is dissolved and reprecipitated in the presence of diethylenetriamine-pentaacetic acid (DPTA) to separate the actinides from barium sulfate. The filtrate is wet ashed, fused in a pyrosulfate flux, and the fusion cake dissolved in dilute nitric acid. Thorium is separated from the other actinides by coprecipitation on ceric iodate. Uranium, plutonium, and americium are separated sequentially, one from the other, by oxidizing them to their highest oxidation states followed by a series of coprecipitations of the nuclides. The isolated nuclides are coprecipitated on neodymium fluoride, filtered, and determined by alpha spectrometry. The recoveries range from 85-95%. The resolution for full width at half maximum (fwhm) is between 40 and 70 kilo-electron volts (keV). Decontamination factors range between 10^3 and 10^4.

2 **ES&H PRECAUTIONS**

2.1 Apply this procedure only to low-level samples, *i.e.*, those containing 5-20 dpm of alpha tracer and a comparable or lesser activity of analyte nuclide(s), in order to prevent (1) cross-contamination of samples and (2) contamination of personnel, equipment, laboratory work areas, and alpha spectrometry systems.

2.2 Work on this procedure involving acid fume venting (*i.e.*, after Step 11.5) must be limited to three samples per lab when there are personnel on the roof.

2.3 Refer to CHEM-AP-11 for proper management of chemicals.

2.4 Follow laboratory safety rules addressed in RESL-TP-IH.2 and RESL-TP-IH.4.

2.5 Wear proper eye protection (*cf*. RESL-TP-IH.1). In the parts of this procedure where hydrofluoric acid is used, rubber gloves and safety glasses must be worn.

2.6 Refer to RESL-TP-IH.6 when handling perchloric acid. Perchloric acid is a powerful oxidizing agent. This procedure makes use of perchloric acid, so a perchloric acid fume hood must be used.

2.7 Refer to CHEM-TP-IH.9 when handling HF.

2.8 Dispose of all wastes into appropriate Satellite Accumulation Area containers (*cf*. RESL-AP-10).

2.9 Conduct all work in accordance with requirements of the applicable Radiation Work Permit(s) (*cf*. RESL-TP-HP.8).

3 EQUIPMENT

3.1 Perchloric acid fume hood.

3.2 Hotplate, 3600 W, 46 x 61 cm (Hevi-duty Equipment Co., Watertown, WI), Multiple Unit, Type 32.

3.3 Fiberglass mat, 1.6 mm thick, to cover hotplate.

3.4 Centrifuge (IEC Model K), Size 2 with 6-place rotor and cups for 1000-mL bottles (tops of bottles are cut off at the shoulder), 12-place rotor, trunnions, and cups for 100-mL centrifuge tubes.

3.5 Fisher blast burner, Catalog No. 03-910-5, Fisher Scientific.

3.6 Forty-seven millimeter Millipore filters, Catalog No. HAWP 04700, Millipore Corp., Bedford, MA 01730.

3.7 Vortex mixer Catalog No. 58815-178 VWR Scientific.

3.8 Twenty-five milliliter Tuffryn HT-200 membrane filter (Gelman Sciences, Ann Arbor, MI).

3.9 Polysulfone twist-lock funnel (Gelman Sciences, Ann Arbor, MI).

3.10 An alpha spectrometry system.

3.11 Heating "block" (aluminum hollow cylinder, 4 cm high, 6 cm o.d., i.d. large enough to fit the 100-mL centrifuge tubes).

3.12 Platinum dishes, 250 mL.

3.13 Centrifuge tubes, 100 mL.

NOTE: *Heavy duty 100-ml centrifuge tubes are no longer available. Thin-walled tubes are tested prior to use as follows: determine a speed of centrifugation that will not crush the centrifuge tubes; centrifuge twelve tubes full of water at higher and higher speeds until one of the tubes is crushed; use the speed just below the highest speed for any centrifuging done in the future with the surviving tubes.*

4 **REAGENTS** Store all solutions in polypropylene bottles.

4.1 Neodymium Chloride (10 mg of Nd/mL): Heat 1.17 g of neodymium oxide in 25 mL of 12M hydrochloric acid until the neodymium oxide is in solution. Cool and dilute the solution to 100 mL with water.

4.2 Neodymium Chloride (0.5 mg of Nd/mL): Dilute 5 mL of neodymium chloride (10 mg of Nd/mL) to 100 mL with water.

4.3 Neodymium Perchlorate (0.5 mg of Nd/mL): Heat 5 mL of neodymium chloride (10 mg of Nd/mL) to perchloric acid fumes in 5 mL of 12M perchloric acid. Cool and dilute the solution to 100 mL with water. Add 10 to 20 mg of solid potassium dichromate.

4.4 Tracer Solutions: Use .5 dpm of ^{229}Th, ^{242}Pu, and ^{243}Am and .10 dpm of ^{232}U tracer, as appropriate, in each sample (*cf.* CHEM-TP-CA.1).

4.5 Carbon Suspension: Fume ten 47-mm Millipore filters for ten minutes in 10 mL of 18M sulfuric acid. Cool and dilute the suspension to 500 mL.

4.6 Substrate Suspension: Dilute 1 mL of neodymium chloride (10 mg of Nd/mL), 20 mL of 12M hydrochloric acid, and 10 mL of carbon suspension to 400 mL with water. Add, while swirling, 10 mL of 29M hydrofluoric acid and dilute to 500 mL with water.

4.7 Ammonium Hydroxide-DTPA: Add 30 g of diethylenetriaminepentaacetic acid (DTPA) to 800 mL of water. Add 120 mL of 15M ammonium hydroxide to dissolve the DTPA and dilute the solution to 1000 mL.

4.8 Reprecipitating Solution: Heat 240 g of anhydrous potassium sulfate in 700 mL of water and 200 mL of 12M hydrochloric acid until it is dissolved. Cool the solution and dilute to 1000 mL.

4.9 Potassium and Sodium Sulfate: Dissolve 75 g each of potassium and sodium sulfate in 800 mL of water with heat. Cool and dilute the solution to 1000 mL.

4.10 Ammonium Iodate: Add 20 g of ammonium iodate and 20 mL of 16M nitric acid to 800 mL of water. Boil until the ammonium iodate dissolves. Cool the solution and dilute to 1000 mL.

4.11 Ammonium Fluoride: Dissolve 80 g of ammonium fluoride in 800 mL of water and dilute the solution to 1000 mL. **NOTE**: *Treat the portion needed each day, for use in the precipitation of the rare earths from americium, with enough solid ammonium persulfate to give a solution ~5% in ammonium persulfate.*

4.12 Zirconyl Perchlorate (10 mg of Zr/mL): Fume 2.9 g of zirconyl nitrate dihydrate (Fisher Scientific Company, thorium free) in 10 mL of 12M perchloric acid until the volume reaches 5 mL. Cool and dilute the solution to 100 mL with water.

4.13 Cerium Nitrate (10 mg of Ce/mL): Dissolve 3.1 g of cerous nitrate hexahydrate (Allied Chemical Company, thorium free) in 1% nitric acid and dilute to 100 mL with 1% nitric acid. If thorium free cerium nitrate is not available, utilize the following steps to purify the cerium nitrate: Dissolve 3.1 g of cerous nitrate hexahydrate in 60 mL of 2% nitric acid, add five drops of 25% sodium nitrite, 1 mL of zirconyl perchlorate (10 mg of Zr/mL), and 10 mL of ammonium iodate with swirling. After ten minutes, centrifuge for five minutes in a 100-mL round-bottomed glass centrifuge tube. Centrifuge the solution at a speed that will not crush the centrifuge tube and dilute the supernate (which contains the cerium) to 100 mL with water. A yellow color (free iodine) will form with time but the reagent will be good.

4.14 DPTA Wash Solution: Add 10 g of DTPA to a 2000-mL beaker containing 1000 mL of water and a few drops of a thymol blue indicator solution. Dissolve the DTPA by heating and slowly adding 10M NaOH, with stirring, to the blue endpoint of the indicator. Store the solution in the beaker covered with a watch glass. Rinse the appropriate glassware with hot wash solution to remove residual traces of barium sulfate.

4.15 Many other reagents are utilized in this procedure; however, they are made-up by dilution with water and are not listed individually.

5 SOIL AND VEGETATION ASH DECOMPOSITION

5.1 Place 10 g of soil, or 5 g of vegetation ash, 10 mL of water, 2 mL of 16M nitric acid, the appropriate tracers, 30 g of potassium fluoride, and 10 mL of 29M hydrofluoric acid in a 250-mL platinum dish. Swirl the dish after each addition. Heat to dryness on a covered hotplate.

NOTE: Omit the 30 g of potassium fluoride from the blank and add after the blank has been heated on the hotplate to dryness. This eliminates spattering from the platinum dish.

5.2 Heat the contents of the platinum dish to a clear fusion over a blast burner.

NOTE: *This is an appropriate stopping point.*

5.3 Continue the analysis as described in Step 8.1.

6 WATER SAMPLE DECOMPOSITION

6.1 If total decomposition is not necessary add 500 mL of water sample (assuming water sample is not greater than 5 % HCl) to a 1 L beaker. Add 20 g of Na_2SO_4 and 45 g of K_2SO_4. Add 150 mL of 12M hydrochloric acid , about 5 boiling chips and bring to a boil. Boil for 20 minutes. Continue the analysis as described in 9.3

6.2 If total decomposition is necessary, place 500 mL of sample in a 1000-mL beaker. Add 20 mL of 16M nitric acid, the appropriate tracers, cover with a watchglass, and evaporate to . 10 mL on a covered hotplate.

6.3 Add 20 mL more of 16M nitric acid and evaporate the sample to . 10 mL. Repeat this step as many times as is needed (three times should be enough) to completely destroy all hydrochloric acid or chlorides that might have been present in the original sample.

6.4 Transfer the solution to a 250-mL platinum dish using a minimum of water to complete the transfer.

6.5 Add 10 mL of 29M hydrofluoric acid to the solution and evaporate to dryness on a covered hotplate.

6.6 Add 30 g of anhydrous potassium fluoride and heat the mixture to a clear fusion over a blast burner.

NOTE: *This is an appropriate stopping point.*

6.7 Continue the analysis as described in Step 8.1.

7 AIR FILTER DECOMPOSITION

7.1 Place the filters in a 1000-mL beaker. Add 10 mL of 18M sulfuric acid, the appropriate tracers, and char the filters on a covered hotplate.

7.1 Cool the solution and add .5 mL each of 16M nitric acid and 12M perchloric acid. Reheat the sample on the covered hotplate to destroy the black char. Repeat if necessary. When the organic material has been completely destroyed, heat the solution to sulfuric acid fumes, cool, and transfer the solution to a 250-mL platinum dish using a minimum of water to complete the transfer.

7.2 Add 10 mL of 29M hydrofluoric acid and evaporate the solution to dryness on the covered hotplate.

7.3 Add 30 g of anhydrous potassium fluoride and heat the mixture to a fusion over the blast burner.

NOTE: *This is an appropriate stopping point.*

7.4 Continue the analysis as described in Step 8.1.

8 SAMPLE TRANSPOSITION

8.1 Place the fused sample on the covered hotplate and allow to cool to the temperature of the hotplate.

8.2 Add 35 mL of 18M sulfuric acid in successively smaller portions, 5-10 mL, as quickly as the frothing in the dish will allow.

8.3 Add 20 g of anhydrous sodium sulfate when the reaction has stopped and heat the mixture to a fusion over the blast burner. Cool the fusion cake to "touch".

NOTE: *This is an appropriate stopping point.*

9 COPRECIPITATION ON BARIUM SULFATE

9.1 Heat 500 mL of water and 150 mL of 12M hydrochloric acid to boiling in a 1000-mL beaker containing five silicon carbide boiling chips.

9.1 Transfer the fusion cake from the platinum dish (Step 8.3) to the boiling solution. Boil the solution for 20 minutes.

9.2 Add just enough solid stannous chloride to the boiling solution to reduce the yellow ferric iron and provide a colorless solution at the end of 20 minutes.

9.3 Add four drops of 1% safranine-O and ten drops of 20% titanium trichloride.

9.4 Add 100 g of anhydrous potassium sulfate to the boiling solution.

9.5 Bring the solution to a boil following dissolution of the potassium sulfate, add four 10-mL portions of 0.5% barium chloride dihydrate with a five-minute boiling period after each addition.

9.6 Transfer the hot mixture to a 1000-mL plastic centrifuge cup using 0.5% sulfuric acid in a squeeze bottle to rinse the 1000-mL glass beaker.

9.7 Balance the sample with either another sample or a tare solution in another 1000-mL plastic centrifuge cup. This can be done by weighing the two solutions to the same weight on a balance using 0.5% sulfuric acid to add weight to the lighter cup.

9.8 Centrifuge the mixture for ten minutes at 2,000 rpm. Let the mixture stand for five minutes and decant the supernate from the precipitate. Discard the supernate.

9.9 Transfer the precipitate to a 100-mL, round-bottomed, glass centrifuge tube using 0.5% sulfuric acid to rinse the centrifuge cup.

9.10 Dilute the sample to 60 mL with 0.5% sulfuric acid swirl, and centrifuge the mixture for five minutes at the predetermined centrifugation speed (Step 3.13).

9.11 Decant and discard the supernate.

NOTE: *This is an appropriate stopping point.*

10 PREPRECIPITATION OF THE BARIUM SULFATE

10.1 Add 4 mL of 18M sulfuric acid to the precipitate in the centrifuge tube and heat on the bare hotplate in an aluminum heating block. Continue heating until the barium sulfate has dissolved in the sulfuric acid and the hot solution has turned to a light brown.

10.2 Cool the sample to touch and add 60 mL of reprecipitating solution containing five drops of 20% titanium trichloride. Heat the sample in a bath of boiling water for ten minutes.

10.3 Centrifuge the hot solution at 2000 rpm for five minutes, decant, and discard the supernate.

10.4 Suspend the precipitate in 60 mL of 0.5% sulfuric acid with the aid of a vortex mixer, centrifuge again, decant, and discard the supernate.

11 BARIUM SULFATE DISSOLUTION

11.1 Suspend the precipitate in 40 mL of ammonium hydroxide-DTPA solution using the vortex mixer. Heat the mixture in a bath of boiling water until the precipitate has dissolved. Heat the solution for ten minutes more.

11.2 Add 5 mL of the 17M acetic acid and 2 mL of the potassium and sodium sulfate solution.

11.3 Heat the solution for five minutes in the bath of boiling water, cool in a bath of cold, running tap water, and centrifuge at 2000 rpm for five minutes.

11.4 Decant the supernate into a 1000-mL beaker that has been rinsed with hot DTPA wash solution to ensure the beaker is free of barium sulfate. Discard the precipitate.

NOTE: *This is an appropriate stopping point.*

11.5 Add 10 mL of 18M sulfuric acid, 15 mL of 16M nitric acid, 45 mL of 12M hydrochloric acid, 10 mL of 12M perchloric acid, and 0.5 mL of cerium nitrate.

NOTE: *Do not continue past this point if personnel are on the roof of CF-690.*

11.6 Cover the beaker with a watchglass, heat the solution to sulfuric acid fumes on a covered hotplate, remove the watchglass, and heat the solution to dryness on the bare hotplate. Cool the residue to room temperature.

12 COPRECIPITATION OF THORIUM AND OTHER PLUS FOUR ELEMENTS ON CERIC IODATE

12.1 Add 20 mL of 2% nitric acid to the residue in the beaker from Step 11.6, heat the mixture on a covered hotplate to dissolve the salts, and transfer the solution to a 50-mL round-bottomed glass centrifuge tube. Use a minimum of water to complete the transfer.

12.2 Add one drop of 0.3% manganous sulfate monohydrate to the solution, heat in a bath of boiling water, add 0.25 g of solid ammonium persulfate and swirl the sample. Continue heating until the yellow ceric ion color and the purple permanganate color are fully developed. Heat for ten minutes more and cool to room temperature in the bath of cold, running tap water.

12.3 Precipitate ceric iodate with the addition of 10 mL of ammonium iodate solution. Allow the mixture to stand at room temperature for five minutes and centrifuge at 2000 rpm for five minutes.

12.4 Decant the supernate, without rinsing, into the original 1000-mL beaker and save for uranium, plutonium, and americium determination. Save the ceric iodate precipitate for thorium determination.

13 URANIUM DETERMINATION

13.1 Add 5 mL of 12M hydrochloric acid and 1 mL of 18M sulfuric acid to the supernate from Step 12.4. Heat to sulfuric acid fumes on the covered hotplate. Heat to dryness on the bare hotplate and cool.

13.2 Add 20 mL of 2% hydrochloric acid, one drop of 25% potassium metabisulfite, and heat the solution on the bare hotplate to boiling. Remove the beaker immediately from the hotplate and transfer the contents to a 50-mL round-bottomed glass centrifuge tube using a minimum of water to complete the transfer.

13.3 Add 0.5 mL of neodymium chloride (10 mg of Nd/mL) to the hot solution with swirling. Cool the sample in a bath of cold, running tap water, add 3 mL of ammonium fluoride solution, allow to stand for ten minutes, and centrifuge at 2000 rpm for five minutes.

13.4 Add 1 mL of neodymium chloride (0.5 mg of Nd/mL) and swirl gently to avoid disturbing the precipitate in the bottom of the centrifuge tube. Allow the mixture to stand for ten minutes.

13.5 Centrifuge the mixture for five minutes, decant gently, without rinsing the glass tube, into a 50-mL round-bottomed polycarbonate centrifuge tube, and save the supernate for the uranium determination.

13.6 Add 2 mL of 12M perchloric acid and two drops of 1% potassium dichromate to the precipitate. Heat the mixture to perchloric acid fumes on the covered hotplate and save for plutonium determination. Do this without delay to minimize the contact time of fluoride with glass.

NOTE: *This is an appropriate stopping point.*

13.7 Add 2 mL of 12M hydrochloric acid, one drop of 0.1% safranine-O, and two drops of 20% titanium trichloride to the supernate from Step 13.5.

13.8 Add 0.1 mL of neodymium chloride (0.5 mL of Nd/mL) and 5 mL of 29M hydrofluoric acid in that order, with swirling, after each addition. Heat in a bath of boiling water for ten minutes and cool to room temperature.

13.9 Mount a 25-mm HT-200 membrane filter on a stainless steel support in a polysulfone twist-lock funnel. With vacuum applied, draw 0.2 mL of 80% ethanol through the filter. As the filter goes dry, add in order to the center of the filter: 5 mL of substrate suspension, another 5 mL of substrate suspension, the sample (after vigorous swirling), a hot 5-mL rinse of the centrifuge tube with 20% hydrofluoric acid-10% perchloric acid solution, a 5-mL water rinse of the filter, and a 2-mL 80% ethanol rinse of the filter.

13.10 Dry the filter for five minutes under a heat lamp at a distance of 12-16" and submit to alpha spectrometry for the determination of uranium.

NOTE: *This is an appropriate stopping point.*

13.11 Use this filtration procedure (Steps 13.9-13.10) for each nuclide fraction being determined.

14 PLUTONIUM DETERMINATION

14.1 Heat the perchloric acid solution in the glass, 50-mL, round-bottomed centrifuge tube from Step 13.6 to boiling in an aluminum heating block on the covered hotplate for 15 minutes, cool, and add 20 mL of 2% perchloric acid-0.01% potassium dichromate solution. Cool the solution in a bath of cold, running tap water.

14.2 Add, with swirling, 5 mL of 29M hydrofluoric acid containing two drops of 1% potassium dichromate. Allow the mixture to stand for ten minutes and centrifuge for five minutes at 2,000 rpm.

14.3 Add 1 mL of neodymium perchlorate (0.5 mg of Nd/mL) and swirl the solution gently to avoid disturbing the precipitate in the bottom of the centrifuge tube.

14.4 Allow the mixture to stand for another ten minutes and centrifuge again.

14.5 Decant the supernate gently, without rinsing the glass tube, into a 50-mL, round-bottomed, polycarbonate centrifuge tube and save for the plutonium determination.

14.6 Add 1 mL of 18M sulfuric acid and 2 mL of potassium and sodium sulfate solution to the precipitate from Step 14.5.

14.7 Heat the mixture to a clear melt over the blast burner, cool, and save for the americium determination. Complete the fusion without delay to minimize the contact time of fluoride with glass.

NOTE: *This is an appropriate stopping point.*

14.8 Treat the plutonium fraction from Step 14.5 with one drop of 30% hydrogen peroxide and heat in a bath of boiling water for ten minutes.

14.9 Add 0.1 mL of neodymium chloride (0.5 mg of Nd/mL) with swirling, heat the solution for ten minutes, and cool.

14.10 Filter the neodymium fluoride precipitate onto an HT-200 membrane filter as described in Steps 13.9-13.10.

15 AMERICIUM DETERMINATION

15.1 Add 20 mL of 2% nitric acid, one drop of 0.2% silver nitrate, and one drop of 0.3% manganous sulfate monohydrate to the salts in the 50-mL, round-bottomed, glass centrifuge tube from Step 14.7 .

15.2 Heat the mixture in the bath of boiling water until the salts are in solution. Add 0.25 g of solid ammonium persulfate with swirling and heat the solution in a bath of boiling water to the purple permanganate color. Continue heating the solution for ten minutes and cool in a bath of cold, running tap water.

15.3 Add 3 mL of ammonium fluoride solution, allow the sample to stand for ten minutes, and centrifuge for five minutes at 2,000 rpm.

15.4 Carefully decant the supernate, without rinsing the centrifuge tube, into a 50-mL, round-bottomed, polycarbonate centrifuge tube and discard the precipitate.

15.5 Add 2 mL of 16% manganous sulfate monohydrate solution to the supernate in the polycarbonate centrifuge tube and allow the solution to stand for ten minutes.

15.6 Add 0.1 mL of neodymium perchlorate (0.5 mg of Nd/mL) and 5 mL of 29M hydrofluoric acid (pretreated with two drops of 1% potassium dichromate); add the reagents above in that order and with swirling after each addition.

15.7 Allow the solution to stand at room temperature for ten minutes and filter the neodymium precipitate onto an HT-200 membrane filter as described in Step 13.11.

NOTE: *This is an appropriate stopping point.*

16 THORIUM DETERMINATION

16.1 Add 2 mL of 12M perchloric acid and 1 mL of 12M hydrochloric acid to the ceric iodate precipitate from Step 12.4 . Heat the sample in an aluminum heating block on the uncovered hotplate to perchloric acid fumes, cool, and add 20 mL of 2% perchloric acid solution.

16.2 Add two drops of 25% sodium nitrite and 0.5 mL of zirconyl perchlorate.

16.3 Precipitate zirconium iodate with the addition of 10 mL of ammonium iodate and allow the mixture to stand for five minutes.

16.4 Centrifuge the sample for five minutes at 2,000 rpm, decant, and discard the supernate.

16.5 Reprecipitate the sample starting with the addition of 2 mL of 12M perchloric acid and 1 mL of hydrochloric in Step 16.1.

16.6 Discard the supernate from the second centrifugation and add 2 mL of 12M perchloric acid, 1 mL of 12M hydrochloric acid, and 0.1 mL of 29M hydrofluoric acid to the precipitate.

16.7 Heat the sample to perchloric acid fumes in an aluminum heating block on the covered hotplate, cool, and transfer the solution to a 50-mL, round-bottomed, polycarbonate centrifuge tube with 20 mL of 2% perchloric acid.

16.8 Add 0.1 mL of neodymium chloride (0.5 mg of Nd/mL) and 5 mL of 29M hydrofluoric acid. Add the reagents above in that order and swirl after each addition.

16.9 Heat the mixture in the bath of boiling water for ten minutes, cool, and filter onto an HT-200 membrane filter as described in Steps 13.9-13.10.

NOTE: *This is an appropriate stopping point.*

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CHEM-TP-CA.1 Preparation of Standards, Tracers, and QC Samples
CHEM-AP-11
RESL-IH-1,2,4,6,9
RESL-AP-10
RESL-TP-HP.8

QUALITY RECORDS

None.