RESL TECHNICAL PROCEDURE

CHEM-TP- A.1

SEQUENTIAL DETERMINATION OF THE ACTINIDES IN SMALL ENVIRONMENTAL SAMPLES

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TITLE: CHEM-TP- A.1, SEQUENTIAL DETERMINATION OF THE ACTINIDES IN SMALL ENVIRONMENTAL SAMPLES

PURPOSE

The purpose of this procedure is to determine the actinides in a variety of samples.

APPLICABILITY

This procedure can be applied soil, water, filter papers, sludges, ores and various types of organic samples. It is also well suited for analyses of hazardous wastes. Samples that contain high concentrations of calcium can cause severe chemical interferences. No radiochemical interferences are known. Analysts performing this procedure must be familiar with the precautions associated with perchloric acid work (RESL-TP-IH.6).

RESPONSIBILITIES

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

DEFINITIONS

None.

PROCEDURE

1 **ABSTRACT** Soil and samples that contain silica are dissolved completely in potassium fluoride fusions. Water samples are evaporated almost to dryness and the residues are dissolved in pyrosulfate fusions. Samples that contain organic material are treated as small soil samples after the organic material has been oxidized. The actinides are separated from the bulk of the sample with barium sulfate. The barium sulfate is dissolved in alkaline EDTA and the actinides are precipitated as hydroxides. The actinides are separated from each other by coprecipitation with neodymium fluoride after adjusting their oxidation states. Each fraction is mounted on a 0.1-µm membrane filter and counted in an alpha spectrometer. This method gives yields of better than 90% and is considerably less time consuming and labor intensive than methods employing solvent extraction and ion exchange. The detection limits for the actinides based on 1-g and 50-mL samples are 0.03 pCi/g and 0.5 pCi/L, respectively.

2 **ES&H PRECAUTIONS**

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

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

2.3 Wear proper personnel protective equipment (gloves, safety glasses, etc.) in accordance with RESL-TP-IH.1.

2.4 Refer to RESL-TP-IH.6 when handling perchloric acid. Perchloric acid is a powerful oxidizing agent and can lead to the formation of explosive compounds under certain conditions.

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

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

3 APPARATUS

3.1 60-mL platinum dishes.

3.2 125-mL Erlenmeyer flasks.

3.3 50-mL polymethylpentene centrifuge tubes.

3.4 Centrifuge.

3.5 25-mm Gelman HT-200. (0.2 μm pores)

3.6 Gelman 25-mm filter funnel.

3.7 SGA filtering jar.

3.8 Alpha Spectrometer.

4 **REAGENTS** All reagents must be filtered through a 0.45 μm filter to eliminate insoluble material that will degrade the subsequent alpha spectra. All reagents should be stored in appropriately sized polypropylene (PP) bottles.

4.1 **Neodymium Perchlorate 0.5 and 5 mg/mL Nd** Dissolve 0.583 g of 99.9% Nd_2O_3 in 2 mL of 72% perchloric acid with heating. Cool the solution and dilute to 100 mL with water. Dilute 10 mL of this 5 mg/mL Nd solution with 100 mL of water containing 1 mL of perchloric acid.

4.2 **Ferrous Perchlorate 20%** Dissolve 1 g of ferrous perchlorate hexahydrate in 4.5 mL of water containing two drops of perchloric acid.

4.3 **Sodium Permanganate 0.5%** Dissolve 50 mg of sodium permanganate in 10 mL of water containing two drops of 72% perchloric acid.

4.4 **Barium Chloride 0.45%** Dissolve 2.224 g of barium chloride dihydrate in 500 mL of water.

4.5 **Sodium Hydroxide 0.25M** Dissolve 5g of sodium hydroxide in 500 mL of water in a 500-mL wash bottle.

4.6 **Ferrous Ammonium Sulfate 20%** Dissolve 10 g of ferrous ammonium sulfate in 46 mL of water and ten drops of sulfuric acid.

4.7 **0.1 M EDTA** Dissolve 14.6 g of EDTA acid in 485 mL of water containing 12.5 g of potassium hydroxide. Adjust the pH of the solution to 11.1.

4.8 **Tellurous Acid 5 mg/mL Te** Dissolve 625 mg of tellurium dioxide in 100 mL of 25% hydrochloric acid.

4.9 **Seeding Suspension** Add 223 mg of barium chloride dihydrate, 6 g of anhydrous sodium sulfate and 4 mL of sulfuric acid to a 250-mL Erlenmeyer flask. Heat the flask over a blast burner until the excess sulfuric acid has been driven off and a pyrosulfate fusion has been obtained. Cool the melt to room temperature and add 100 mL of water. Heat the flask on a bare hotplate until the pyrosulfate cake has dissolved. Transfer the barium sulfate suspension to a 125-mL PP bottle. Immediately before each use shake the bottle to suspend the barium sulfate.

4.10 **EDTA Cleaning Bath** Add 20 g of EDTA to a 1-L beaker containing 600 mL of water and 2 mL of 0.04% thymol blue. Heat the suspension and add 50% sodium hydroxide with swirling until the EDTA has dissolved and the full blue color of the indicator remains. Dilute the solution to 1 L with water.

4.11 **Fluoride Cleaning Bath** Add 20 g of boric acid to a 1-L beaker containing 750 mL of water and 250 mL of concentrated nitric acid.

5 WATER SAMPLES

5.1 Transfer up to 500 mL of water to a 1-L PTFE beaker.

5.2 Add three drops of 0.04% thymol blue (TB) and two 14 mesh carborundum boiling chips and 10 mL hydrofluoric acid.

5.3 If the solution is blue or yellow, add concentrated nitric acid to the red endpoint of the TB, then add an additional 5 mL of nitric acid.

5.4 If the solution is red, add 5 mL of concentrated nitric acid.

5.5 Add an accurately know amount of Am^{243}, Pu^{236} (or Pu^{242}) and U^{232} tracers at individual activities of 10 dpm.

Note: *If a Total Spectrometric Alpha determination is to be performed, omit adding the isotopic tracers.*

5.6 Evaporate the solution to near dryness, add 3 mL concentrated sulfuric acid, and heat to sulfuric fumes. Transfer to 125-mL Erlenmeyer flask.

5.7 Add 4.5 g of anhydrous potassium sulfate and 2.0 g of anhydrous sodium sulfate.

5.8 Swirl to mix and heat slowly over a blast burner until the salts dissolve.

5.9 Increase the temperature of the blast burner and continue to heat until the evolution of sulfuric acid fumes have slowed, the residue has dissolved and a clear pyrosulfate fusion is obtained.

5.10 Cool the pyrosulfate cake to room temperature and add 5 mL of concentrated hydrochloric acid and 35 mL of water.

5.11 Place the Erlenmeyer on a high temperature hot plate and heat the solution to boiling. Swirl the solution occasionally to aid in the dissolution of the cake.

5.12 Add ten drops of 20% ferrous ammonium sulfate and boil the solution for ten minutes to ensure complete reduction of the plutonium.

Note: ***If uranium is to be determined***, *replace the ten drops of 20% ferrous ammonium sulfate with five drops of 20% titanium trichloride. Continue to add titanium trichloride until the yellow iron color has been discharged and the solution exhibits a violet color. However do not use more than fifteen drops of the titanium reagent or an insoluble potassium titanium titanate will precipitate. On water samples that contain more iron than can be reduced directly by the limited amount of titanium, add 1 mL of 25% hydrazine in Step 5.12 to reduce most of the iron before the titanium reductant is used.*

5.13 Continue under "Barium Sulfate Precipitation" (Section 8).

6 SOIL SAMPLES

6.1 Add 0.5 mL of nitric acid to 1 g of soil in a 50-mL platinum dish.

Note: *Prewetting the soil with water may be necessary if the evolution of* CO_2 *is vigorous enough to cause some loss of the sample due to spraying.*

6.2 Heat the dish on a hot plate until the soil is almost dry.

6.3 Cool and add an accurately know amount of Am^{243}, Pu^{236} (or Pu^{242}) and U^{232} tracers at individual activities of . 10 dpm.

6.4 Heat the platinum dish on the hot plate until the soil is almost dry, taking care not to lose any tracer by splattering.

6.5 Cool, and add 2 g of anhydrous potassium fluoride and 1.3 g of potassium hydrogen fluoride.

6.6 Mix the potassium fluoride and soil thoroughly with a teflon stirring rod.

6.7 Place the dish on a ring stand and fuse over the full heat of a Fisher blast burner until a clear melt is obtained.

6.8 Cool the melt to room temperature and add 4 mL of sulfuric acid.

6.9 Heat the dish on the hot plate until the potassium fluoride cake has dissolved and the transposition has been completed.

Note: *When the dissolution becomes vigorous enough for the solution to froth over the sides of the dish, cool the dish in a bath of cold running water until the reaction slows, then reheat. When most of the cake has dissolved, the vigorous reactions will subside enough so that the dish can be placed on the full heat of the bare hotplate.*

6.10 After all of the potassium fluoride cake has transposed, add 2 g of anhydrous sodium sulfate, swirl to mix and heat over a small flame of the blast burner until the evolution of sulfuric acid fumes have slowed and a clear red pyrosulfate fusion is obtained.

6.11 Remove the dish from the heat and roll the melt high onto the sides of the dish while cooling.

6.12 Cool the dish to room temperature and gently flex the sides of the dish to fracture the cake and facilitate it's removal.

6.13 Add the pyrosulfate cake to a boiling solution of 35 mL of water and 5 mL of concentrated hydrochloric acid in a 125-mL Erlenmeyer flask.

6.14 Swirl the solution until the cake has dissolved.

6.15 Add 1 mL of 20% ferrous ammonium sulfate or 0.5 mL 25% hydrazine.

6.16 Boil the solution for fifteen minutes to hydrolyze condensed phosphates and ensure reduction of the plutonium.

Note: *Uranium cannot be included in the first barium sulfate precipitation when the pyrosulfate fusion has been carried out in a platinum dish. The dissolved platinum will be reduced to the metal during the titanium reduction and carry through the procedure. The uranium fraction must be treated separately as described under "Determination of Uranium from Soil Samples" (Section 9). However, if the soil or wet ashed organic sample is to be used for screening purposes, or if isotopic tracers have been omitted, uranium can be included in the first barium sulfate by using the Total Spectrometric Alpha portion (Section 17) of this procedure. Additional separations can be made on that "total" fraction as needed.*

6.17 Continue under "Barium Sulfate Precipitation." (Section 8)

7 ORGANIC MATERIAL

7.1 Add 1 g of organic material, 5 mL of sulfuric acid, and 5 mL of nitric acid to a 125-mL Erlenmeyer flask.

7.2 Heat the Erlenmeyer on a hot plate until the nitric acid has oxidized the easily oxidizable organic material.

7.3 Continue to heat until the excess nitric acid has been driven off and the sulfuric acid has charred the organic material.

7.4 While swirling the flask, add 1 mL of nitric acid dropwise to oxidize the charred organic material.

7.5 After the reaction subsides, continue to add 1-mL portions of nitric acid until the solution has either lightened in color or until the nitric acid has no more effect on oxidizing the organic material.

7.6 Continue to char the sample with sulfuric acid and to oxidize the charred material with nitric acid until the sample no longer chars when fumed with sulfuric acid. (Steps 7.3-7.6)

7.7 Add 1 mL of perchloric acid dropwise to the hot concentrated sulfuric acid to oxidize the remaining organic material.

Note: *CAUTION!! If the perchloric acid is added while the solution still contains carbon or while the solution is very dark in color a VERY vigorous reaction or EXPLOSION can result!! Never add perchloric acid to a sample without first pretreating the sample extensively with nitric acid. Use perchloric acid only as a last resort to oxidize the most difficult to oxidize organic material, and only after extensive treatment of repeatedly charring with sulfuric acid and then oxidizing the charred material with nitric acid!*

7.8 Evaporate the sulfuric acid to .5 mL and transfer the solution quantitatively to a 50-mL platinum dish.

7.9 Evaporate the solution to dryness and proceed with the soil procedure. (Section 6)

8 BARIUM SULFATE PRECIPITATION

8.1 If necessary, add enough water to bring the total volume in the flask to .35 mL.

8.2 While swirling the solution, add 1 mL of 0.45% barium chloride dropwise.

8.3 Place the Erlenmeyer on the hot plate and boil the solution for one minute.

8.4 Repeat the 1 mL dropwise addition of barium chloride and the one minute boiling time four more times. (Steps 8.2-8.4)

Note: *If the barium chloride solution is added at a faster rate then that specified or without swirling the solution during the addition, there will be a loss of the actinides on the barium sulfate.*

8.5 Transfer the solution and precipitate to a 50-mL conical polymethylpentene (PMP) centrifuge tube.

8.6 Centrifuge for five minutes while the solution is still hot to prevent calcium sulfate from precipitating.

8.7 **If titanium trichloride was used** as a reducing agent in Step to include uranium in the barium sulfate, discard the supernate.

8.8 **If the pyrosulfate fusion was performed in a platinum dish and uranium is to be determined, (Section 6)** decant the supernate back into the original Erlenmeyer.

8.9 Wash the precipitate with . 10 mL of water, directed in a forceful stream from a wash bottle.

8.10 Centrifuge, decant and discard the wash.

8.11 Treat the solution in the Erlenmeyer without delay as described under "Determination of Uranium from Soil Samples" (Section 9).

8.12 Continue under "EDTA Dissolution" with the barium sulfate precipitate. (Section 10).

9 DETERMINATION OF URANIUM FROM SOIL SAMPLES

9.1 Cool the filtrate in the Erlenmeyer to .50.C and add 200 uL of tellurous acid.

9.2 Add 0.5 mL of 25% hydrazine to the solution slowly and with swirling.

9.3 Let stand for at least five minutes to reduce most of the iron.

Note: *The solution should turn black from the precipitation of elemental tellurium once most of the iron has been reduced. If the solution stays yellow more hydrazine must be added.*

9.4 Boil the solution for at least five minutes to flocculate the tellurium.

9.5 Add 2 mL of tellurous acid, two drops of 1% Safranine-O indicator and heat the solution to boiling.

9.6 Add 1.5 mL of titanium trichloride dropwise until a dense black precipitate of elemental tellurium forms, the colorless luco form of the indicator is obtained, and about four drops excess has been added.

9.7 Boil the solution until the tellurium has flocculated completely.

9.8 Transfer the solution to a 50-mL PMP centrifuge tube and centrifuge for five minutes.

9.9 Add .50 mg of paper pulp directly onto the scum that will be riding on the surface of the supernate and recentrifuge for five minutes.

9.10 Decant the clear supernate into another 50-mL PMP centrifuge tube.

9.11 Discard the tellurium precipitate that contains the polonium.

9.12 Add one drop of Safranine-O to verify the reducing conditions of the solution.

9.13 If the indicator turns red, add additional drops of titanium trichloride until the luco form is restored.

9.14 While swirling the solution, add 1 mL of 0.45% barium chloride dropwise.

9.15 Place the Erlenmeyer on the hot plate and boil the solution for one minute.

9.16 Repeat the 1-mL dropwise addition of barium chloride and the one minute boiling time four more times.

9.17 Transfer the solution and precipitate to a 50-mL conical (PMP) centrifuge tube.

9.18 Centrifuge for five minutes, decant and discard the supernate.

9.19 Dislodge the impacted barium sulfate precipitate by flipping the centrifuge tube sharply in a downward motion.

9.20 Add 3 mL of water, 3 mL of 0.1 M potassium EDTA and heat in a water bath until the barium sulfate has dissolved completely.

Note: *It may be necessary to place the tube in an ultrasonic bath to disperse the precipitate to ensure complete dissolution.*

9.21 With rapid swirling, add one drop of 30% titanium trichloride followed by 2 mL of 10 M potassium hydroxide.

9.22 Heat in a hot water bath for 5 min to flocculate the titanous hydroxide precipitate.

9.23 Centrifuge for 5 min, decant and discard the supernate.

9.24 Wash the precipitate with 5 mL of 0.25M sodium hydroxide directed in a forceful stream from a wash bottle.

9.25 Centrifuge for five minutes, decant and discard the wash.

9.26 Flip the centrifuge tube sharply to dislodge the precipitate and add fifteen drops of hydrochloric acid, 4 mL of water and 200 µL of 0.5 mg/mL Nd carrier.

9.27 Heat the centrifuge tube in a bath of boiling water until the precipitate dissolves completely.

9.28 Add five drops of 20% titanium trichloride and heat in a boiling water bath for five minutes.

9.29 Swirl the solution and add 1 mL of hydrofluoric acid.

9.30 Immediately transfer the tube to a bath of cold water and let stand for fifteen minutes to maximize the yield of uranium.

9.31 Mount and count as described in "Mounting Fluoride Precipitates" (Section 15).

10 EDTA DISSOLUTION

10.1 Dislodge the impacted barium sulfate precipitate by flipping the centrifuge tube sharply in a downward motion.

10.2 Add 3 mL of water, 3 mL of 0.1 M potassium EDTA and heat in a water bath until the barium sulfate has dissolved completely.

Note: *It may be necessary to place the tube in an ultrasonic bath to disperse the precipitate to ensure complete dissolution.*

10.3 With rapid swirling, add one drop of 30% titanium trichloride followed by 2 mL of 10 M potassium hydroxide.

10.4 Heat in a hot water bath for 5 min to flocculate the titanous hydroxide precipitate.

10.5 Centrifuge for 5 min, decant and discard the supernate.

10.6 Wash the precipitate with 5 mL of 0.25M sodium hydroxide directed in a forceful stream from a wash bottle.

10.7 Centrifuge for five minutes, decant and discard the wash.

Note: *This hydroxide precipitate contains all of the actinides and will also contain uranium (from a water sample) if uranium was included in the barium sulfate precipitate with titanium trichloride.*

10.8 **If a "Total Spectrometric Alpha"** determination (except soils) is to be performed, continue with Section 14.

10.9 **If further separations of the actinides** from each other are to be performed, continue with Section 11 below.

11 SEPARATION OF URANIUM AND PLUTONIUM FROM AMERICIUM

11.1 Flip the centrifuge tube sharply to dislodge the precipitate and add fifteen drops of perchloric acid, 4 mL of water and 200 µL of 0.5 mg/mL Nd carrier. Add only 100 µL of 0.5 mg/mL Nd if the sample was a 0.5 to 1 g soil sample.

11.2 Heat the centrifuge tube in a bath of boiling water until the precipitate dissolves completely.

11.3 Add five drops of 0.5% sodium permanganate and continue to heat for another five minutes.

11.4 Swirl the solution and add 1 mL of hydrofluoric acid.

11.5 Immediately transfer the tube to a bath of cold water and let stand for fifteen minutes to maximize the yield of americium.

11.6 Wash both sides of a 25-mm 0.2 µm Gelman HT-200 membrane filter paper with 80% ethanol and place the filter right side up in a Gelman polysulfone filter holder equipped with a stainless steel support screen.

11.7 Tighten the funnel against the base as tight as possible without wrinkling the filter.

11.8 Place the filtering assembly in the top of a bell jar that is equipped with vacuum line from a water aspirator so that the solution can be filtered directly into either a PFA jar or a 125-mL Erlenmeyer flask.

11.9 If uranium (from a water sample) is to be determined, filter the solution directly into the original 125-mL Erlenmeyer flask; otherwise, filter the solution and collect the filtrate in a 60-mL PFA jar.

11.10 Wash the precipitate with 2 mL of water containing five drops of HF. (Collect the wash in the same container as the major fraction.)

11.11 Remove the collection container.

11.12 Wash the precipitate with 5 mL of 80% ethanol. Do not collect the ethanol washes in the collection container.

11.13 Remove the filter containing the americium from the filtering assembly and dry under a 250-watt infrared lamp at a distance of .4" for five minutes.

11.14 Count the americium fraction in a alpha spectrometer for a long enough period of time to obtain desired statistics.

    11.14.1 **If uranium and plutonium are to be determined,** continue under "Determination of Uranium and Plutonium from Water Samples" (Section 12).

11.14.2 **If only plutonium is to be determined,** continue under "Determination of Plutonium" (Section 13).

12 DETERMINATION OF URANIUM AND PLUTONIUM FROM WATER SAMPLES

12.1 Add 4.5 g of anhydrous potassium sulfate, 2.0 g of anhydrous sodium sulfate and 3 mL of concentrated sulfuric acid to the Erlenmeyer flask that contains the filtrate from the americium separation.

12.2 Swirl to mix and heat slowly over a blast burner until the salts dissolve.

12.3 Increase the temperature of the blast burner and continue to heat until the evolution of sulfuric acid fumes have slowed, the residue has dissolved and a clear pyrosulfate fusion is obtained.

12.4 Cool the pyrosulfate cake to room temperature and add 5 mL of concentrated hydrochloric acid and 35 mL of water.

12.5 Place the Erlenmeyer on a high temperature hot plate and heat the solution to boiling.

12.6 Add five drops of 20% titanium trichloride and boil the solution for ten minutes to ensure complete reduction of the uranium and plutonium.

12.7 If necessary, add enough water to bring the total volume in the flask to .35 mL.

12.8 While swirling the solution, add 1 mL of 0.45% barium chloride dropwise.

12.9 Place the Erlenmeyer on the hot plate and boil the solution for one minute.

12.10 Repeat the 1-mL dropwise addition of barium chloride and the one minute boiling time four more times (steps 12.8-12.10).

Note: *If the barium chloride solution is added at a faster rate then that specified or without swirling the solution during the addition, there will be a loss of the actinides on the barium sulfate.*

12.11 Transfer the solution and precipitate to a 50-mL conical polymethylpentene (PMP) centrifuge tube.

12.12 Centrifuge for five minutes, decant and discard the supernate.

12.13 Dislodge the impacted barium sulfate precipitate by flipping the centrifuge tube sharply in a downward motion.

12.14 Add 3 mL of water, 3 mL of 0.1 M potassium EDTA and heat in a water bath until the barium sulfate has dissolved completely.

Note: *It may be necessary to place the tube in an ultrasonic bath to disperse the precipitate to ensure complete dissolution.*

12.15 With rapid swirling, add one drop of 30% titanium trichloride followed by 2 mL of 10 M potassium hydroxide.

12.16 Heat in a hot water bath for five minutes to flocculate the titanous hydroxide precipitate.

12.17 Centrifuge for five minutes, decant and discard the supernate.

12.18 Wash the precipitate with 5 mL of 0.25 sodium hydroxide directed in a forceful stream from a wash bottle.

12.19 Centrifuge for five minutes, decant and discard the wash.

12.20 Flip the centrifuge tube sharply to dislodge the precipitate and add fifteen drops of hydrochloric acid, 4 mL of water and 200 µL of 0.5 mg/mL Nd carrier.

12.21 Heat the centrifuge tube in a bath of boiling water until the precipitate dissolves completely.

12.22 Add five drops of 20% titanium trichloride and heat in a boiling water bath for five minutes.

12.23 Swirl the solution and add 1 mL of hydrofluoric acid. Immediately transfer the tube to a bath of cold water and let stand for fifteen minutes to maximize the yield of uranium and plutonium.

12.24 Mount and count as described in "Mounting Fluoride Precipitates" (Section 15).

13 DETERMINATION OF PLUTONIUM

13.1 To the 60 mL PFA jar that contains the filtrate and wash from the plutonium-americium separation, add 1 mL of perchloric acid and evaporate the solution to 0.5 mL.

13.2 Add 4 mL water, transfer the solution to a 50-mL PMP centrifuge tube, add three drops of 20% ferrous perchlorate, and heat for three minutes in a boiling water bath to reduce the plutonium.

13.3 Add 200 µL of 0.5 mg/mL neodymium carrier solution.

13.4 Swirl the centrifuge tube to mix thoroughly, and add 1 mL of HF.

13.5 Immediately transfer the tube to a cold water bath.

13.6 Let the tube stand for fifteen minutes to ensure complete precipitation of the plutonium.

13.7 Mount and count as described in "Mounting Fluoride Precipitates" (Section 15).

14 TOTAL SPECTROMETRIC ALPHA (EXCEPT SOILS)

14.1 Flip the centrifuge tube sharply to dislodge the precipitate and add fifteen drops of hydrochloric acid, 4 mL of water and 200 µL of 0.5 mg/mL Nd carrier.

14.2 Heat the centrifuge tube in a bath of boiling water until the precipitate dissolves completely.

14.3 Add five drops of 20% titanium trichloride and heat in a boiling water bath for five minutes.

14.4 Swirl the solution and add 1 mL of hydrofluoric acid.

14.5 Immediately transfer the tube to a bath of cold water and let stand for fifteen minutes to maximize the yield of uranium and plutonium.

14.6 Mount and count as described in "Mounting Fluoride Precipitates." (Section 15)

14.7 After counting, the precipitate can be wet ashed if necessary (Section 16) and appropriate separations made.

15 MOUNTING FLUORIDE PRECIPITATES

15.1 Wash both sides of a 25-mm 0.2 µm Gelman HT-200 membrane filter paper with 80% ethanol and place the filter right side up in a Gelman polysulfone filter holder equipped with a stainless steel support screen.

15.2 Tighten the funnel against the base as tight as possible without wrinkling the filter and add 2 mL of carbon suspension.

15.3 Filter the fluoride precipitated solution through the filter paper.

15.4 Wash the precipitate with 2 mL of water containing five drops of HF, then with 5 mL of 80% ethanol.

15.5 Remove the filter from the filtering assembly and dry under a 250-watt infrared lamp at a distance of .4" for five minutes.

15.6 Mount and count the filter paper in a alpha spectrometer for a long enough time to obtain desired statistic.

Note: *All equipment that has be used with or contained barium sulfate and fluoride precipitates should be cleaned in the EDTA and fluoride cleaning baths respectively. These baths should be hot and equipment cleaned in the EDTA bath should be immersed in a bath containing 5% HCl to dissolve any hydrolyzed compounds.*

16 WET ASHING OF MOUNTED FILTER PAPERS

16.1 Place the HT-200 filter paper to be wet ashed in a 60-mL PFA jar and add 0.5 mL of nitric acid and 5 mL of perchloric acid.

16.2 Place the jar on a clay triangle on a high temperature hotplate and evaporate to 0.5 mL of perchloric acid without swirling the solution.

16.3 Add 4 mL of water to the jar while still hot and transfer the solution to a 50-mL PMP centrifuge tube.

16.4 Continue with Step for an uranium and plutonium separation from americium and thorium.

17 TOTAL SPECTROMETRIC ALPHA FOR SOILS

17.1 Add 1 g of soil to a 50-mL platinum dish.

17.2 Add 2 g of anhydrous potassium fluoride and 1.3 g of potassium hydrogen fluoride.

17.3 Mix the potassium fluoride and soil thoroughly with a teflon stirring rod.

17.4 Place the dish on a ring stand and fuse over the full heat of a Fisher blast burner until a clear melt is obtained.

17.5 Cool the melt to room temperature and add 5 mL of sulfuric acid.

17.6 Heat the dish on the hot plate until the potassium fluoride cake has dissolved and the transposition has been completed.

Note: *If the dissolution becomes vigorous enough for the solution to froth over the sides of the dish, cool the dish in a bath of cold running water until the reaction slows, then reheat. When most of the cake has dissolved, the vigorous reactions will subside enough so that the dish can be placed on the full heat of the bare hotplate.*

17.7 After the cake has dissolved completely, add 0.5 g of boric acid with swirling to the dish.

17.8 Transfer the transposed cake directly to a 125-mL Erlenmeyer flask containing 2 g of sodium sulfate.

17.9 Wash the platinum dish with water, heat the dish over a gentle flame to suspend the cake, and transfer the slurry to the flask.

17.10 Swirl the flask and heat slowly over a blast burner until fumes of sulfuric acid are being evolved strongly.

17.11 Increase the temperature of the blast burner and continue to heat until the evolution of sulfuric acid fumes have slowed, the residue has dissolved and a clear pyrosulfate fusion is obtained.

17.12 Heat the entire flask strongly while swirling the flux high onto the sides of the flask. The molten fusion should coat the entire inside of the flask (to retrieve any unfused sample that was splattered). The flask should be hot enough to keep the molten flux from solidifying on the sides.

17.13 Remove the flask from the heat and set on an insulated surface to cool.

17.14 Do not swirl the cake onto the sides of the flask while cooling.

Note: *Platinum is eliminated from the sample by performing the pyrosulfate fusion in glass instead of in the platinum dish. However, the pyrosulfate fusion in an Erlenmeyer flask solidifies much slower than in a platinum dish, this can cause a very insoluble crystalline precipitate to form in the subsequent dissolution if the pyrosulfate fusion is not heated long and hot enough. If this precipitate is present, the barium sulfate must be recrystallized (Step 17.30), or the resolution of the final fraction will be badly degraded.*

17.15 After the pyrosulfate cake has cooled to room temperature, add two 14 mesh carborundum boiling chips, 5 mL of concentrated hydrochloric acid, 35 mL of water and 0.5 mL of 25% hydrazine.

17.16 Place the Erlenmeyer flask on a high temperature hot plate and heat the solution to boiling.

17.17 Swirl the solution occasionally to aid in the dissolution of the cake.

17.18 Check the solution in a darkened room with a flashlight. If a very crystalline needle-like precipitate is present, the barium sulfate precipitate MUST be reprecipitated to ensure complete dissolution in EDTA.

17.19 Add five drops of 20% titanium trichloride and boil the solution for ten minutes to ensure complete reduction of the uranium and plutonium.

Note: *The yellow ferric iron color should be absent and the solution should have a violet color from the titanium reductant. If the solution is yellow or colorless, continue to add titanium trichloride dropwise until a permanent violet color is present.*

17.20 If necessary, add enough water to bring the total volume in the flask to 35 mL.

17.21 While swirling the solution, add 1 mL of 0.45% barium chloride dropwise.

17.22 Place the Erlenmeyer flask on the hot plate and boil the solution for one minute.

17.23 Repeat the 1-mL dropwise addition of barium chloride and the one minute boiling time four more times.

Note: *If the barium chloride solution is added at a faster rate then that specified or without swirling the solution during the addition, there will be a loss of the actinides on the barium sulfate.*

17.24 If the solution obtained from dissolving the pyrosulfate cake contained the crystalline precipitate described above, transfer the solution and precipitate to a 50-mL conical Pyrex centrifuge tube; otherwise transfer the solution and precipitate to a 50-mL conical PMP centrifuge tube.

17.25 Centrifuge for five minutes while the solution is still hot to prevent calcium sulfate from precipitating.

17.26 Wash the precipitate in the centrifuge tube with . 10 mL of water, directed in a forceful stream from a wash bottle.

17.27 Add five drops of hydrofluoric acid with swirling to dissolve any of the titanium titinate precipitate that might have formed.

17.28 Centrifuge, decant and discard the wash.

17.29 If the barium sulfate needs to be reprecipitated, continue with the next step, otherwise skip to Step .

17.30 Add 2 mL of sulfuric acid to the Pyrex centrifuge tube containing the barium sulfate to be reprecipitated.

17.31 Heat gently over a small flame of a blast burner until the precipitate has dissolved completely.

17.32 Cool, and add 20 mL of reprecipitating solution while swirling the centrifuge tube rapidly.

17.33 Centrifuge for five minutes, decant and discard the supernate.

17.34 Wash the precipitate with . 10 mL of water.

17.35 Transfer the solution and precipitate to a 50-mL conical polymethylpentene (PMP) centrifuge tube.

17.36 Centrifuge, decant and discard the wash.

17.37 Dislodge the impacted barium sulfate precipitate by flipping the centrifuge tube sharply in a downward motion.

17.38 Add 3 mL of water, 3 mL of 0.1 M potassium EDTA and heat in a boiling water bath until the barium sulfate has dissolved completely.

Note: *It may be necessary to place the tube in an ultrasonic bath to disperse the precipitate to ensure complete dissolution. Quadravalent titanium from the soil will precipitate at this point and is very flocculant. This precipitate should not be confused with undissolved barium sulfate.*

17.39 With rapid swirling, add one drop of 30% titanium trichloride followed by 2 mL of 10 M potassium hydroxide.

17.40 Heat in a hot water bath for five minutes to flocculate the titanous hydroxide precipitate.

17.41 Centrifuge for five minutes, decant and discard the supernate.

17.42 Wash the precipitate with 5 mL of 0.25 M sodium hydroxide directed in a forceful stream from a wash bottle.

17.43 Centrifuge for five minutes, decant and discard the wash.

Note: *This hydroxide precipitate contains uranium, thorium and all of the transuranium elements that were present in the sample.*

17.44 Flip the centrifuge tube sharply to dislodge the precipitate and add fifteen drops of hydrochloric acid, 4 mL of water and 100 µL of 0.5 mg/mL Nd carrier.

17.45 Heat the centrifuge tube in a bath of boiling water until the precipitate dissolves completely.

17.46 Add three drops of 20% titanium trichloride and heat in a boiling water bath for five minutes.

17.47 Swirl the solution and add 1 mL of hydrofluoric acid.

17.48 Immediately transfer the tube to a bath of cold water and let stand for fifteen minutes to maximize the yield of the actinides.

17.49 Mount the precipitate on a 25-mm Gelman HT-200 filter paper and count in an alpha spectrometer for a long enough period of time to obtain desired statistics.

17.50 After this "total-alpha" fraction has been counted in a alpha spectrometer, the filter paper containing the precipitate can be wet ashed and additional separations that are necessary can be performed.

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QUALITY RECORDS

None.