Plutonium

Environmental Measurements Laboratory
U.S. Department of Energy

Pu-01-RC

PLUTONIUM IN AIR FILTERS

Contact Person(s) : Anna Berne

APPLICATION

This procedure is applicable to all types of air filters. However, if the filter is made of an organic polymer, it is advisable to first decompose the filter in a muffle furnace at 450°C overnight. Samples are then digested in concentrated HNO_3, after which the remaining residue and filter material are treated with HF.

SPECIAL APPARATUS

1. Muffle furnace.

2. Pyrex long stem fluted funnel with an inside diameter of 9.5 cm.

SPECIAL REAGENT

1. Plutonium-236 tracer - a standard solution containing ~0.15 Bq g^{-1} in a dispensing bottle.

SAMPLE PREPARATION

1. Place the air filter in a 400-mL (or appropriate size) beaker.

2. Add a known amount (~ 0.05 Bq) of ^{236}Pu tracer.

3. Add 150 mL of concentrated HNO_3 and 50 mL of concentrated HCl (or appropriate amounts based on air filter size), allow sample to react for about 1 h. Place the sample on a hot plate and cover with a watch glass. Heat the sample overnight on a low setting. Continue heating until the volume is ~100 mL.

4. Remove the sample from the hot plate, add 100 mL of water and let the sample cool to room temperature. Filter by gravity using a conical funnel with an 18.5 cm Whatman No. 42 filter paper. Wash with 50-75 mL of 1:1 HNO_3. Collect the filtrate in a 250-mL beaker, evaporate the filtrate to near dryness, add ~35 mL 1:1 HNO_3, and save. Cover the beaker with parafilm to prevent changes in the concentration of 1:1 HNO_3.

5. Transfer the filter and residue to the original beaker and wet ash with 150-200 mL concentrated HNO_3. Allow refluxing to occur on low heat. **Do not boil.** Evaporate to ~25-30 mL.

6. Transfer the filtered residue to a platinum dish or Teflon beaker. Add 10 mL of HF, 5 mL of HNO_3, and evaporate to near dryness. Add 10 mL of HNO_3 and 10 mL of HF, then evaporate to near dryness again. Repeat the addition of HNO_3 and HF two more times. Wash the sides of the vessel three times with 1:1 HNO_3, and evaporate to dryness after each addition.

7. Add 20 mL of 1:1 HNO_3 to sample.

8. Filter the sample into the beaker containing the filtrate from Step 4 by gravity filtration using a conical funnel with a 18.5 cm Whatman No. 40 or No. 42 filter paper. Wash with 1:1 HNO_3 (~ 20 mL).

9. Adjust sample volume to 100 mL with 1:1 HNO_3.

10. Proceed to **Plutonium Purification - Ion Exchange Technique**, Procedure Pu-11-RC.

Pu-02-RC

PLUTONIUM IN SOIL SAMPLES

(see Volume II)

Pu-03-RC

PLUTONIUM IN SOIL RESIDUE - TOTAL DISSOLUTION METHOD

Contact Person(s) : Anna Berne

APPLICATION

This procedure is applicable to acid leached soils as well as unleached soils (Krey and Bogen, 1987).

The silica in the sample is removed by the formation of SiF_4 using HF. The remaining residue is fused with KF to decompose the complex silicates, followed by the addition of H_2SO_4 to distill the remaining SiF_4. This procedure is followed by a pyrosulfate fusion, and finally, coprecipitation with $BaSO_4$.

SPECIAL REAGENTS

1. Potassium fluoride.

2. Sodium sulfate.

3. Hydroxylamine hydrochloride.

4. 10% $BaCl_2$ solution - 10 g of $BaCl_2$ 100 mL^{-1} of water.

SAMPLE PREPARATION

1. Using 100 mL of concentrated HNO_3, wet ash the sample and filter the remaining sample from the leaching of the soil (Steps 2-6 of **Sample Preparation**, Procedure Pu-02-RC).

2. Evaporate the sample on a hot plate making sure the sample is completely dry.

3. Transfer the mixture to a tared plastic zip lock bag. Quantitatively remove the soil residue from the beaker. Reweigh the bag to determine the mass of the residue.

4. Break apart large aggregates by kneading the residue in the bag so that the mixture is homogeneous.

5. Weigh out 50 g of the soil residue and transfer to a 250-mL platinum crucible.

6. Add a known amount (~ 0.05 Bq or appropriate amount) of ^{236}Pu tracer.

7. Add 5 mL of 1:1 HNO_3 to wet the soil residue. Very slowly add 10 mL of concentrated HF, it may be necessary to add the HF dropwise. If excessive frothing occurs, wet the sample with 1:1 HNO_3 from a wash bottle.

8. Add an additional 10 mL of HF carefully. Heat the sample at low heat on a hot plate until no liquid remains.

9. When the sample is dry, add another 10 mL of HF. Heat the sample on a hot plate until the sample is dry.

10. Repeat Step 9 until at least a total of 100 mL of HF has been added and the sample no longer fumes. During the heating of the sample, the soil residue may move up the walls of the platinum crucible. If this occurs wash the walls with HF.

11. To insure the absence of HF, heat the crucible with a Meker burner until the crucible glows red.

12. Cool the sample and add 20 g of $KF{\cdot}2H_2O$. Heat the sample on a hot plate, mixing with a Teflon stirrer.

13. Continue heating the sample until all the water has evaporated (so that no splattering occurs in the next step). It may be necessary to raise the temperature of the hot plate to assure drying.

14. Place the sample in a muffle furnace and heat at 950°C for 30 min.

15. Remove the crucible from the furnace and cool to room temperature.

16. Slowly add 35 mL of concentrated H_2SO_4 to the crucible on a hot plate at low heat. Heat the sample until the evolution of a small amount of SO_3 vapors occurs. After this step, continue heating for 5 min.

17. Cool and add 20 g of anhydrous Na_2SO_4.

18. Place the crucible on a clay triangle mounted on a ring stand and heat the crucible gently with a Meker burner, minimizing bumping and frothing of the sample. Gradually raise the amount of heat while watching for frothing, until the molten mass is dissolved (a clear red color), at this point the temperature is ~ 700ºC. Remove the Meker burner and cool the crucible to room temperature. (**Caution:** This fusion is to be performed in a hood because SO_3 fumes are emitted.)

19. Flex the walls of the crucible to break apart the fused cake. Transfer the fused material to a 1500-mL beaker and add 200 mL of water to the crucible. Heat the crucible on a hot plate to dissolve any material in the crucible. Transfer the water wash to the 1500-mL beaker containing the fused material.

20. Perform three additional 200-mL washes of the crucible, each time transferring the solution to the 1500-mL beaker.

21. Add 100 mL of concentrated HCl, 1 g of $NH_2OH{\cdot}HCl$ to the 1500-mL beaker, which contains the fused material and water, and heat the solution to dissolve the fused material. Cover with a watch glass.

22. Continue to heat the solution until the boiling point is reached. Using a pipette add 5 mL of 10% $BaCl_2$ solution and continue boiling the sample for 5 min.

23. Cool to room temperature and filter the solution mixture by gravity through a 15-cm Whatman No. 42 filter paper using a conical funnel. Wash the beaker well with four 50-mL portions of H_2O. Transfer each H_2O wash to the funnel containing the filtered precipitate. Discard the filtrate.

24. Transfer the filter paper containing the $BaSO_4$ precipitate to a small platinum crucible (40 mL). Heat the crucible with a Meker burner to decompose the filter paper.

25. Add 5 g of anhydrous Na_2CO_3 to the crucible and mix the Na_2CO_3 with the $BaSO_4$ precipitate. Place the crucible in a muffle furnace at 850°C for 30 min.

26. Remove the crucible from the furnace, cool to room temperature, add 5 mL of water, and heat gently on a hot plate.

27. Transfer the salts and wash the solution into a 40-mL centrifuge tube using a minimum amount of H_2O to effect the transfer.

28. Centrifuge the sample and discard the supernatant.

29. Dissolve the precipitate in the centrifuge tube with 5 mL of 6M HCl. Transfer the solution mixture to a 100-mL Teflon beaker using 6M HCl. Also, wash the sides of the platinum crucible with 6M HCl to remove any remaining residue and transfer to the Teflon beaker.

30. Add 5 mL of concentrated HF, place the beaker on a hot plate and evaporate the sample to near dryness.

31. Cool, add 5 mL of 1:1 HNO_3, and 5 mL of concentrated HF, heat on a hot plate and evaporate to near dryness.

32. Add 15 mL of 1:1 HNO_3 and evaporate to near dryness.

33. Repeat Step 32, three to five times, to remove any traces of HF.

34. Add 20 mL of 1:1 HNO_3 and heat gently for a few minutes. Remove the beaker from heat, filter mixture under reduced pressure using a 25-mm Millipore filter with a 0.45-µm pore size or by gravity filtration through a 15-cm Whatman No. 42 filter paper using a conical funnel.

35. Wash the Teflon beaker with 15 mL of 1:1 HNO_3 and transfer the wash to the filtered precipitate. Discard the precipitate. Proceed to Plutonium Purification - Ion Exchange Technique (see Procedure *Pu-11-RC*).

REFERENCE

Krey, P. W. and D. C. Bogen
"Determination of Acid Leachable and Total Plutonium in Large Samples"
J. of Radioanalytical and Nuclear Chemistry, 115, 335-355, December (1987)

Pu-04-RC

PLUTONIUM IN TISSUE

(*see Volume II*)

Pu-05-RC

PLUTONIUM IN TISSUE - SOLVENT EXTRACTION

(*see Volume II*)

Pu-06-RC

PLUTONIUM IN URINE

(*see Volume II*)

Pu-07-RC

PLUTONIUM IN LARGE URINE SAMPLES

(*see Volume II*)

Pu-08-RC

PLUTONIUM IN VEGETATION AND TISSUE - NITRIC/HYDROCHLORIC ACID METHOD

(*see Volume II*)

Pu-09-RC

PLUTONIUM IN VEGETATION AND TISSUE - NITRIC/SULFURIC ACID METHOD

(*see Volume II*)

Pu-10-RC

PLUTONIUM IN WATER

Contact Person(s) : Anna Berne

APPLICATION

This procedure is used for all types of water samples (i.e., sea water, lake water, tap water, etc.). If the sample contains suspended particulates, they must be removed by filtration. Large volume samples are analyzed after evaporation in an acidic medium.

The sample is heated in HNO_3 and then in 3:1 HNO_3:HCl. The volume is then reduced to near dryness and finally the volume of the sample is adjusted with 1:1 HNO_3. The sample is then ready to be purified by ion exchange separation (see Procedure Pu-11-RC).

SPECIAL REAGENT

1. ^{236}Pu tracer - a standard solution containing 0.2 Bq g^{-1} in a dispensing bottle. The purity of the tracer is measured by α spectrometry.

SAMPLE PREPARATION

1. Transfer 100-1000 mL of a H_2O sample to a beaker.

2. To the sample add ~ 0.05 Bq (or appropriate amount) of ^{236}Pu tracer.

3. Add an equal amount of concentrated HNO_3, cover the beaker with a watch glass and place on a hot plate. Reflux the solution for 4-8 h.

4. Replace the watch glass with a ribbed watch glass and evaporate the solution to near dryness. When the volume is reduced to ~ 100 mL, allow the solution to cool to room temperature and transfer to an appropriate size beaker.

5. Continue evaporating the sample to near dryness. Cool, add 75 mL of concentrated HNO_3 and 25 mL of concentrated HCl. Cover with a watch glass. Allow to react for 30 min. Then place the sample on a hot plate and bring to a boil. After the solution has boiled for 30 min, reduce heat and continue heating overnight. Do not allow the sample to evaporate to dryness.

6. Remove the sample from the hot plate and add 100 mL of H_2O to the sample. Allow the sample to cool to room temperature and filter under reduced pressure using a Buchner funnel with a Whatman No. 42 filter paper.

7. Wash with 50 mL of 1:1 HNO_3 and then 50 mL of H_2O.

8. Transfer the filtrate to a 250-mL beaker, cover the beaker with a ribbed watch glass.

9. (**Note**: If the filter paper contains a moderate amount of precipitate, it must be treated with HF.) Transfer the filter paper containing the residue from the HNO_3/HCl digestion to a platinum dish. Place the platinum dish in a muffle furnace and heat at 100°C, raise the temperature by increments of 100°C every hour until a final temperature of 450°C is reached. Continue heating at this temperature overnight. Turn off the muffle furnace and let the sample in the platinum dish cool sufficiently to remove it from the furnace. Add 15 mL of 1:1 HNO_3 and 15 mL of concentrated HF. Heat the sample to near dryness.

10. Repeat Step 9 two times.

11. Add 20 mL of 1:1 HNO_3 to the sample and heat on a hot plate under a low setting until near dryness (to remove traces of HF).

12. Repeat Step 11 two times.

13. Add 20 mL of 1:1 HNO_3 to sample.

14. Using a conical funnel, filter the sample by gravity through an 18.5 cm Whatman No. 42 filter paper into a beaker containing the filtrate from Step 9. Wash well with 1:1 HNO_3.

15. Reduce the volume of the solution to near dryness on a hot plate.

16. Adjust the volume to 100 mL by the addition of 1:1 HNO_3.

17. Proceed to Plutonium Purification - Ion Exchange Technique, Procedure *Pu-11-RC*.

Pu-11-RC

PLUTONIUM PURIFICATION - ION EXCHANGE TECHNIQUE

Contact Person(s) : Anna Berne

APPLICATION

This procedure has been applied to the leachates derived from the plutonium sample preparation methods described in this Manual. Ion exchange chromatography is used to remove the large amounts of impurities contained in these leachates.

SPECIAL APPARATUS

Ion exchange columns - see Specifications 7.5 and 7.6.

SPECIAL REAGENTS

1. 1:1 HNO_3 - 500 mL HNO_3 diluted to 1 L.

2. Hydroxylamine hydrochloride - $NH_2OH{\cdot}HCl$

3. 0.3M hydroxylamine hydrochloride-0.5M HNO_3 - 20.85 g of $NH_2OH{\cdot}HCl$ diluted to 1 L with 0.5M HNO_3.

4. Anion exchange resin - Bio-Rad AG 1-X8 (100-200 mesh, Cl^- form), see Specification 7.4.

ION EXCHANGE SEPARATION

1. Cool the sample in an ice bath, add 1 g of $NH_2OH \cdot HCl$, stir, and let stand in an ice bath for 15 min. Remove the sample from the ice bath and heat to boiling on a hot plate with medium heat for 1-3 min. Cool the sample to room temperature.

2. Prepare the ion exchange resin column (see **Note**).

3. Pass the sample through the resin bed at a flow of ~ 1 mL min^{-1}. Wash the beaker and the column with 30 mL 1:1 HNO_3 three times. Allow the liquid to flow until the level reaches the top of the resin bed prior to each wash. Reserve the sample and wash the effluent for ^{241}Am determination (or until yield has been determined as satisfactory).

4. Elute the plutonium with 10 mL of 0.5M HNO_3 twice - then with 100 mL of 0.3M hydroxylamine hydrochloride - 0.5M HNO_3 into a 250-mL beaker. Discard the resin.

5. Slowly add 25-30 mL HNO_3 until effervescence begins, then place on a hot plate and evaporate the eluate to dryness.

6. Dissolve the residue in 30 mL of 1:1 HNO_3 and cool in an ice bath. Add 500-600 mg of $NH_2OH \cdot HCl$ and repeat Steps 1-3 using a small column (see Specification 7.6) for all samples.

7. Wash the resin with 100 mL of HCl (two 10-mL portions followed by two 40 mL portions). Wash the resin with two 10-mL portions, followed by one 40-mL portion 1:1 HNO_3. Save the effluent until yield determination.

8. Repeat Steps 4 and 5. Discard the resin.

9. Convert the residue to the Cl^- form by adding 5 mL of HCl and evaporating to dryness three times at a low temperature.

10. See Procedure G-03 for microprecipitation for α spectrometry.

Note: Preparation of Columns

1. When preparing a large soil sample use a large column (Specification 7.5), otherwise use the column described in Specification 7.6.

2. Position a plug of glass wool in the base of the column so that no resin will drain out.

3. Add sufficient resin to form a resin bed of 10 cm in length. Wash the column with sufficient 1:1 HNO_3 to remove the Cl^- ion from the resin. Test the effluent with a dilute silver nitrate solution.

Pu-12-RC

PLUTONIUM AND/OR AMERICIUM IN SOIL OR SEDIMENTS

Contact Person(s) : Anna Berne

APPLICATION

This procedure is applicable to soils which contain plutonium and americium deposited from worldwide fallout and some nuclear activities. A total dissolution technique is required for some soil samples for plutonium determination.

Plutonium and americium isotopes are leached and equilibrated with ^{236}Pu and ^{243}Am tracers with nitric and hydrochloric acids from soil samples of up to 100 g in size. Plutonium is isolated and purified by ion exchange. Americium is collected with a calcium oxalate precipitation, isolated and purified by ion exchange. After source preparation by microprecipitation, the plutonium isotopes and americium are determined by alpha spectrometry.

SPECIAL APPARATUS

1. For microprecipitation, see Procedure G-03.

2. Ion-exchange columns - see Specification 7.5.

SPECIAL REAGENTS

1. Americium-243 tracer solution, ~ 0.15 Bq g^{-1} in a dispensing bottle.

2. Plutonium-236 (^{242}Pu can also be used) tracer solution, ~0.20 Bq g^{-1} in a dispensing bottle.

3. Bio-Rad AG 1-X8 resin (100-200 mesh) - see Specification 7.4.

4. Bio-Rad AG 1-X4 resin (100-200 mesh) - see Specification 7.4.

5. TEVA resin 2 mL ion extraction columns (Aliquat 336, methyltricapryl-ammonium chloride, Henkel Corporation, Tucson, AZ 85745-1273, on Amberchrom resin) or equivalent or can be prepared from TEVA resin, Eichrom Industries, 8205 Cass Ave. Suite 107, Darien, IL 60561) - place a plug of glass wool in the bottom of a 2 mL plastic transfer pipette (see Specification 7.7). Add slurried TEVA resin (0.5 g). Place additional glass wool on the top of the resin.

6. 2M ammonium thiocyanate in 0.1M formic acid solution - dissolve 152 g of NH_4SCN in ASTM Type 2 water, add 4.25 mL formic acid, and dilute to 1 L.

7. 1M ammonium thiocyanate in 0.1M formic acid - dissolve 76 g of NH_4SCN in ASTM Type 2 water, add 4.25 mL formic acid, and dilute to 1 L.

8. Calcium carrier solution, 100 mg mL^{-1} - dissolve 25 g $CaCO_3$ in a minimal amount of concentrated HNO_3, and dilute to 100 mL.

9. Iron carrier, 100 mg mL^{-1} - slowly heat 100 g of iron powder in 500 mL of HCl until reaction ceases. Carefully and slowly add 100 mL of HNO_3 while stirring. Cool and dilute to 1 L.

10. Oxalate wash solution - dissolve 10 g of oxalic acid ($H_2C_2O_4 \cdot 2H_2O$) to make 1 L of solution (~ 1% solution).

11. Hydroxylamine hydrochloride, $NH_2OH \cdot HCl$ - solid.

SAMPLE PREPARATION

1. Weigh 1-100 g of soil into an appropriate sized beaker. Add weighed amounts of ^{243}Am and ^{236}Pu tracers.

2. Slowly add 100 mL (**Note**: volumes are based on 100 g sample and should be adjusted if sample size is smaller) of HNO_3 to the beaker. Control the foaming with the addition of a few drops of n-octyl alcohol. Stir sample with a glass stir rod to mix sample and acid. When the reaction subsides, add 30 mL of HCl, and stir. Allow the mixture to react at room temperature, rinse and remove stir rod, cover with a watch glass, then reflux on a low temperature hot plate overnight. Remove from hot plate and cool.

3. Dilute the solution in the beaker with water to 1:1 HNO_3 and filter the solution with vacuum through 9 or 11 cm Whatman No. 42 filter paper on a Büchner funnel into a 1 L flask. Wash with 1:1 HNO_3. Retain the filtrate in a 2-L beaker, evaporate the filtrate until salting out begins to occur. Return the residue and filter to the original beaker using HNO_3 to complete the transfer.

4. Add HNO_3 to the beaker to bring the volume added to 100 mL. Stir with a glass rod to mix sample and acid. Cover with a watch glass and heat until filter is wet ashed. Remove from the hotplate and cool. Add 30 mL of HCl to the beaker, cover with the watch glass, and heat on a low temperature hot plate for about 3 h with occasional stirring. Remove the beaker from the hot plate, and cool.

5. Repeat Step 3; dilute, filter and wash. Combine the filtrates. Return the residue and filter to the original beaker.

6. Repeat Step 4; wet ash filter and leach sample.

7. Repeat Step 3; dilute, filter and wash. Combine the three filtrates in a beaker. Discard the residue and filter paper.

8. Heat the filtrate with repeated 50-mL additions of HNO_3, covering the sample with a watch glass and letting the sample reflux until all organic matter is decomposed. Evaporate the solution to incipient dryness. Redissolve in 50-200 mL of 1:1 HNO_3.

If the solution is not clear, proceed to Step 9, otherwise go to **Plutonium Determination.**

9. If any siliceous matter is present, filter into a flask by gravity through a Whatman No. 42 filter paper. Wash the residue with 1:1 HNO_3, and reserve the filtrate.

10. Transfer the filter paper with the residue to the original beaker and wet ash the paper with 100 mL of HNO_3. Repeat wet ashing two or three times, then transfer the residue in the beaker into a 250-mL Teflon beaker, using 1:1 HNO_3. Evaporate to dryness.

11. Add 5-25 mL of HF and 5-25 mL of HNO_3 to the beaker and evaporate on a medium temperature hot plate. Repeat the addition of the HF/HNO_3 and the evaporation process two or three times. Rinse the walls of the Teflon beaker with 1:1 HNO_3 and evaporate, and repeat. Evaporate to dryness. Dissolve with 1:1 HNO_3 and evaporate to dryness.

12. Dissolve the residue in 1:1 HNO_3 and filter by gravity through a Whatman No. 42 filter paper. Add the filtrate to the solution from Step 9. Discard the filter and any residue. Heat the combined solution to incipient dryness. Redissolve in 50-200 mL 1:1 HNO_3.

PLUTONIUM DETERMINATION

Proceed to Plutonium Purification Ion Exchange Technique Procedure *Pu-11-RC*. Save the column effluents for **Americium Determination**.

AMERICIUM DETERMINATION

1. Evaporate the americium effluents to incipient dryness. Redissolve in a minimum amount of 1:1 HNO_3, dilute with four volumes of water.

2. Add 5 mL of calcium carrier solution (500 mg of calcium) and 50 g L^{-1} of oxalic acid to the sample while stirring with a magnetic stirrer. (**Note**: The total volume of the

sample solution can be estimated using the markings on the beaker, and the amount of oxalic acid to be added is calculated using that volume.)

3. Adjust the pH of the solution to 2.0-2.5 with NH_4OH using pH paper as an indicator and continue to stir for 30 min. Remove the magnetic stir bar.

4. Cool the sample and let it stand until precipitate settles and solution clears (for more than 6 h or overnight). Check for completeness of precipitation using a drop of saturated $H_2C_2O_4$ solution. Aspirate (or decant), using a disposable transfer pipette and suction, as much liquid as possible without disturbing the precipitate. Transfer the precipitate to a 250-mL centrifuge bottle using oxalate wash solution (see **Note 1**). Balance the bottles on a double pan balance and centrifuge for 10 min at 2000 rpm. Decant and discard the supernate.

5. Break up the precipitate with a stirring rod and wash the precipitate with the oxalate wash solution. Centrifuge, decant and discard the wash. Repeat wash. Redissolve the precipitate in a minimal amount (50-70 mL) of concentrated HCl (the final precipitate should be redissolved in ~200 mL of HNO_3, then proceed to Step 8 below). (**Note**: Dissolution is easier if the centrifuge bottle is placed in a hot water bath and stirred with a glass rod).

6. Transfer the dissolved precipitate to the original 600-mL beaker. Add enough water to make ~ 1M solution. Add 50 g L^{-1} of oxalic acid.

7. Repeat Steps 3 through 6 until supernate is colorless.

8. Transfer the dissolved precipitate to the original beaker and heat to destroy the oxalate ion. Evaporate to near dryness. Dissolve in a minimum of 1:1 HNO_3. Transfer to centrifuge bottle using water to complete the transfer.

9. Add enough water to make ~1M HNO_3. Warm the solution in a 90˚ hot water bath and add 0.2 mL iron carrier solution (20 mg iron).

10. With the centrifuge bottle in the hot water bath adjacent to a hood, adjust the pH of the solution to 8-9 with NH_4OH while stirring with a glass rod. Allow the solution to digest in a hot water bath for 20 min.

11. Cool in a cold water bath, rinse, and remove the glass rod. Balance the bottles on a double pan balance and centrifuge for 40 min at 2000 rpm.

12. Decant (or aspirate) and discard the supernate. Add 10 mL concentrated HCl to dissolve the $Fe(OH)_3$ pellet. Add four drops 30% H_2O_2 to oxidize any Mn present, followed by 100 mL of water and heat in the water bath for 30 min to get rid of excess H_2O_2.

13. Repeat Steps 10 to 12 three times. Reprecipitate, centrifuge and redissolve. The final precipitate should be redissolved in HNO_3.

14. Transfer to a 250-mL beaker, evaporate to dryness, add 20 mL HNO_3, and evaporate to dryness again.

15. Dissolve the wet-ashed residue in 40 mL 1:1 HNO_3. Cool in an ice-water bath. Add 0.6-1.0 g $NH_2OH \cdot HCl$, dissolve, and let react for 15 min. Cover with a watch glass. Heat on a low temperature hot plate to decompose unreacted $NH_2OH \cdot HCl$, then bring to gentle boil for 1-2 min. Cool and pass the solution through a 1:1 HNO_3 ion-exchange column (see **Note 2**). Collect the effluent in a 400-mL beaker. Wash the column with 150 mL of 1:1 HNO_3 and collect in the beaker.

16. Evaporate the sample in the 400-mL beaker to dryness. Convert to HCl by adding 20-30 mL of HCl at a time, heat to almost dryness, and repeat the HCl addition and evaporation at least three times. Evaporate again and dissolve the final residue in 30 mL of HCl. Pass this solution through a 12N HCl ion exchange column (see **Note 3**). Collect the effluent in a 250-mL beaker. Wash the column with 100 mL of HCl and collect in the 250-mL beaker.

17. Evaporate to dryness. Dissolve the residue in 10 mL 2M NH_4SCN in 0.1M formic acid.

18. Prepare a TEVA column. Equilibrate the resin by adding 3-4 mL 2M NH_4SCN in 0.1M formic acid. Drain to the top of the resin.

19. Transfer the sample to the column. Drain to the top of the resin.

20. Wash the column with 10 mL 1M NH_4SCN in 0.1M formic acid. Discard wash.

21. Elute the americium with 15 mL 2M HCl into a clean 100-mL beaker.

22. Add approximately 10 mL aqua regia to the sample. Gently decompose the thiocyanate solution under a heat lamp. Allow the solution to develop a purple color which will slowly disappear.

23. Heat the sample on a hot plate to near dryness. Dissolve the residue in 3 to 4 mL HNO_3. Evaporate to dryness. Redissolve in HNO_3 and evaporate two more times.

24. Convert to HCl by addition of 3-4 mL HCL. Evaporate to dryness. Redissolve in HCl and evaporate two more times. Proceed to microprecipitation.

Notes:

1. If a centrifuge is not available, centrifugation can be replaced by filtering and wet ashing filter paper and precipitate in HNO_3.

2. Preparation of 1:1 HNO_3 column. Position a plug of glass wool at the base of an 11-mm o.d. column. Transfer with ASTM Type 2 water, 15 mL of wet settled Bio-Rad AG 1-X8 resin (100-200 mesh) to the column and allow it to settle. Place a second plug of glass wool on top of the resin, and with the stopcock open allow the water level to reach the top of the upper plug. Pass 150 mL (or enough so that the effluent tests free of Cl^- ion) of 1:1 HNO_3 through the resin bed in three 50-mL portions, allowing the level of each to reach the top of the upper glass wool plug.

3. Preparation of HCl column. Position a plug of glass wool at the base of an 11-mm o.d. column. Transfer with ASTM Type 2 water, 10 mL of wet settled Bio-Rad AG 1-X4 resin (100-200 mesh) to the column and allow it to settle. Place a second plug of glass wool on top of the resin and with the stopcock open allow the water level to reach the top of the upper plug. Pass two 50-mL volumes of HCl through the resin bed and allow each to reach the top of the upper glass wool plug.

MICROPRECIPITATION

See Microprecipitation Source Preparation for Alpha Spectrometry, Procedure G-03.

AMERICIUM
LOWER LIMIT OF DETECTION (LLD)

Counter Efficiency	(%)	25
Counter Background	(cps)	15×10^{-6}
Yield	(%)	50
Blank	(cps)	-
LLD (400 min)	(mBq)	1
LLD (1000 min)	(mBq)	0.5
LLD (5000 min)	(mBq)	0.3

PLUTONIUM
LOWER LIMIT OF DETECTION (LLD)

Counter Efficiency	(%)	25
Counter Background	(cps)	2×10^{-5}
Yield	(%)	75
Blank	(cps)	-
LLD (400 min)	(mBq)	1
LLD (1000 min)	(mBq)	0.5
LLD (5000 min)	(mBq)	0.2