

G-03

**MICROPRECIPITATION SOURCE PREPARATION  
FOR ALPHA SPECTROMETRY**

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Contact Person(s) : Isabel M. Fisenne

**APPLICATION**

Microprecipitates of Th, U, Pu, Am, and Cm, suitable for  $\alpha$ -spectrometry measurements, are prepared by coprecipitation with Nd as the F. [Adapted from Sill and Williams (1981) and Hindman (1983).]

**SPECIAL APPARATUS**

1. Ultrasonic bath.
2. Millipore 47 mm diameter Pyrex glass filtration chimney, fitted glass support and metal clamp.
3. Millipore 47 mm diameter filters, 0.45  $\mu\text{m}$  pore size.
4. Gelman 25 mm diameter polysulfone filtration chimney, stem support and stainless steel screen.
5. Gelman 25 mm Metrical filter, 0.1  $\mu\text{m}$  pore size.
6. Eppendorf 100  $\mu\text{L}$  pipette or equivalent.
7. 100  $\mu\text{L}$  disposable pipette tips.
8. 10 mL plastic pipette.

9. 10 mL plastic culture tubes.
10. Pipetting bulb.
11. 50 mL plastic graduated cylinder.
12. 10 mL plastic graduated cylinder.
13. 20 L plastic carboy with spigot.
14. 2 L vacuum filtration flask.
15. 250 mL vacuum filtration flask.

**Special Precautions** - Due to the use of HF in the preparation of the reagents and in the precipitation procedure, rubber gloves must be worn and plasticware must be used as noted above.

#### SPECIAL REAGENTS

1. Filtered deionized water - filter 20 L of deionized water through 0.45  $\mu\text{m}$  pore size Millipore filters. Store the filtered water in a 20-L capacity plastic carboy with a spigot (see **Note 1**).
2. 1N HCl - add 83 mL of concentrated HCl to 917 mL of filtered deionized water and store in a plastic bottle.
3. Neodymium carrier solution, 1000  $\mu\text{g mL}^{-1}$  (Spex Industries, Wayne, NJ), or equivalent (see **Note 2**).
4. Neodymium carrier solution, 0.5  $\text{mg mL}^{-1}$ . Dilute 10 mL of the 1000  $\mu\text{g mL}^{-1}$  Nd carrier solution to 20 mL with filtered deionized water.
5. 48% HF.

6. Neodymium fluoride substrate solution -  $10 \mu\text{g mL}^{-1}$  - pipette 5 mL of Nd carrier ( $1000 \mu\text{g mL}^{-1}$ ) into a 500-mL plastic bottle. Add 460 mL of 1N HCl to the plastic bottle. Cap the bottle and shake to mix. Measure 40 mL of 48% HF in a plastic graduated cylinder. Uncap the bottle and add the HF. Recap the bottle and shake to mix thoroughly.
7. 0.58 N HF - pour 980 mL of filtered deionized water into a 1 L plastic bottle. Measure 20 mL of 48% HF in a plastic graduated cylinder. Uncap the bottle and add the HF. Recap the bottle tightly and shake to mix.
8. Ethyl alcohol, 100%.
9. Ethyl alcohol, 80% - mix 800 mL of 100% ethyl alcohol and 200 mL of filtered deionized water. Store in a 1 L plastic bottle.
10. Titanium trichloride, 20% solution (see **Note 3**).

**Notes:**

1. Deionized water may contain a sufficient quantity of solid material to adversely effect the resolution of the final filtered sample.
2. Neodymium is preferred as a carrier for the determination of thorium. Cerium compounds tend to contain variable and measurable quantities of thorium.
3. Titanium trichloride is an extremely powerful reducing agent, which should be used in a well-ventilated hood.

PRECIPITATION OF Th, Pu, Am, AND Cm

1. The separated Th, Pu, Am or Cm solution for precipitation should be in a 1-2 mL volume of 1N HCl or 1N HNO<sub>3</sub> solution. (The conditions for the precipitation of U are noted separately.)

2. Transfer the solution to a 10-mL plastic culture tube. Wash the original sample vessel twice with 1-mL washes of the same concentration acid as the sample. Transfer the washings to the culture tube. Mix by gently shaking the tube.
3. Add 100  $\mu\text{L}$  of the 0.5  $\text{mg mL}^{-1}$  Nd carrier solution to the tube with an Eppendorf pipette. Gently shake the tube to mix the solution.
4. Add 10 drops (0.5 mL) of 48% HF to the tube and mix well by gentle shaking.
5. Place the tube in a cold-water ice bath for at least 30 min.
6. Insert the polysulfone filter stem in the 250-mL vacuum flask. Place the stainless steel screen on top of the fitted plastic filter stem.
7. Place a 25-mm Metrical filter on the stainless steel screen. **Caution** - place the less glossy side of the Metrical filter face up. The filters are usually shipped in the box in this manner, but the analyst should check each filter visually.
8. Wet the filter with 100% ethyl alcohol. Center the filter on the stainless steel screen support and apply a vacuum.
9. Lock the filter chimney firmly in place on the filter stem. Open the system to full vacuum.
10. Wash the filter with 100% ethyl alcohol, followed by a filtered deionized water wash.
11. Draw 5000  $\mu\text{L}$  (5 mL) of Nd substrate solution into a plastic pipette.
12. Add 5 mL of the Nd substrate solution down the side of the filter chimney. Apply a vacuum to the filter for at least 15 sec.
13. Repeat Steps 11 and 12 with an additional 5000  $\mu\text{L}$  of the substrate solution.
14. Place the sample to be filtered in a 150 mL beaker containing 25 mL of  $\text{H}_2\text{O}$ . Set the beaker in an ultrasonic unit containing about a 2.54 cm depth of  $\text{H}_2\text{O}$ .

15. Ultrasonicate the sample tube for about 1 min to suspend the  $\text{NdF}_3$  precipitate.
16. Pour the sample down the side of the filter chimney and apply a vacuum.
17. Add about 2 mL of 0.58N HF to the tube and ultrasonicate briefly. Pour the wash down the side of the filter chimney.
18. Repeat Step 17.
19. Add about 2 mL of filtered deionized water to the tube and ultrasonicate briefly. Pour the wash down the side of the filter chimney.
20. Repeat Step 19.
21. Add about 2 mL of 80% ethyl alcohol to the tube and ultrasonicate briefly. Pour the wash down the side of the filter chimney.
22. Repeat Step 21.
23. Wash any drops remaining on the sides of the chimney down toward the filter with 80% ethyl alcohol. **Caution** - Directing of a stream of liquid onto the filter will disturb the distribution of the precipitate on the filter and render the sample unsuitable for  $\alpha$ -spectrometry resolution.
24. Without turning off the vacuum, remove the filter chimney.
25. Reduce or turn off the vacuum to remove the filter. Discard the filtrate. (**Caution** - If the filtrate is to be retained, it should be placed in a plastic container to avoid dissolution of the glass vessel by dilute HF.)
26. Place the filter directly on a suitable mounting disc. Secure with a mounting ring.
27. Place the mounted sample under a heat lamp (sample to lamp distance should be about 10 cm) for 10 min prior to  $\alpha$ -spectrometry measurement.

## PRECIPITATION OF U

1. The U bearing solution must be in HCl solution.
2. Transfer 1-2 mL of the U bearing solution (1N HCl) to a 10-mL plastic culture tube. Wash the original sample vessel twice with 1-mL portions of 1N HCl. Transfer the washings to the culture tube. Mix by gently shaking the tube.
3. Add 100  $\mu\text{L}$  of the 0.5 mg mL<sup>-1</sup> Nd carrier with an Eppendorf pipette. Gently shake the tube to mix the solution.
4. Add four drops of 20% Ti trichloride to the tube and gently shake the tube. A strong permanent violet color should appear. If the color fails to appear, add a few more drops of Ti trichloride.
5. Continue the precipitation from Step 4, Precipitation of Th, Pu, Am, and Cm.

## REFERENCES

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