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Code: Pu A-2b

H. W. Laboratory Manual
Redox and TBP Methods

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DETERMINATION OF LOW LEVEL PLUTONIUM BY FLUORIDE PRECIPITATION

Safety Precautions

1. Observe the general laboratory Safety Rules.
2. Observe the Assay Methods Safety Rules.

Literature Reference

Andrus, W. S., and Fisher, D. J., Secret Report KAPL-328 (4-11-50)

Principle and Limitations

Low level plutonium is determined radiochemically as the fluoride by precipitation with lanthanum fluoride followed by metathesis with hydroxide and a second precipitation with lanthanum fluoride. The precipitate is then mounted, dried and measured on the specified type A sample tester.

Prior to precipitation the sample is treated with nitric acid in order to oxidize U(IV) to U(VI). Hexone present in the amount of 1% will not interfere.

This method is applicable to sample range of 1 part Pu to 10^9 parts U.

Apparatus

1. Centrifuge, clinical
2. Centrifuge cones, 15 ml.
3. Magnetic stirrer
4. Water bath, 80-90° C.

Chemicals and Solutions

1. Nitric Acid, 1N (Solutions Code: SN-9a)
2. Hydrofluoric acid, 1N (Solutions Code:)
3. Wash solution (Solutions Code: SW-3a)
   1N HNO_3 - 1N HF

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4. Hydroxylamine hydrochloride, 5M (Solutions Code: SH-27c)
5. Potassium hydroxide, 30% (Solutions Code:)
6. Potassium hydroxide, 2% (Solutions Code:)
7. Lanthanum nitrate, 5 mg La^{+3}/ml. (Solutions Code: SL-1c)
8. Collodion solution (Solutions Code: SC-17a)
   0.01 - 0.02 mg solids/drop

Procedure

1. Pipet 5 ml. sample into a 15 ml. centrifuge cone containing 1 ml. of 16N nitric acid.
2. Heat for at least 15 minutes in water bath at 80-90° C. Allow to cool.
3. Add 200 ~~lambdas~~ microliters of lanthanum nitrate and 500 ~~lambdas~~ microliters of 5M hydroxylamine hydrochloride.
4. Dilute to 10 ml. with distilled water and allow to stand for at least 10 minutes after stirring.
5. Add 2.5 ml. of 10N hydrofluoric acid and allow to stand for at least 10 minutes.
6. Centrifuge and ~~slurp~~ remove the supernate into contaminated waste.
7. Wash the precipitate with the wash solution (1N HNO_3 - 1N HF); centrifuge; and ~~slurp~~ remove the supernate into contaminated waste.
8. Repeat step 7 twice.
9. Add 500 ~~lambdas~~ microliters of 30% potassium hydroxide.
10. Heat for at least 20 minutes in water bath at 80-90° C. with occassional stirring.
11. Allow to cool and add 500 ~~lambdas~~ microliters of distilled water.
12. Stir; centrifuge; and ~~slurp~~ remove the supernate into contaminated waste.
13. Wash the precipitate with 2% potassium hydroxide; centrifuge; and ~~slurp~~ remove the supernate into contaminated waste.
14. Repeat Step 13.
15. Add 250 ~~lambdas~~ microliters of 16N nitric acid and heat for at least 5 min.

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Procedure (cont'd)

in water bath at 80-90° C. to dissolve the precipitate.

16. Allow to cool and dilute to about 2 ml. with distilled water.

17. Add 200 microliters of lanthanum nitrate.

18. Add 500 microliters of 10M hydrofluoric acid; stir; centrifuge; and remove the supernatant.

19. Wash the precipitate with the wash solution (1M HNO_3 - 1M HF), centrifuge, and remove the supernate into contaminated waste.

20. Repeat step 19.

21. Add one drop of the wash solution; stir; and transfer onto a clean platinum disc (ringed with zapon).

22. Repeat Step 21 until all the precipitate has been transferred.

23. Complete the drying, flaming, counting and calculations according to the procedure used in the PuA-6 method currently in use for aqueous samples.