Uranium

Environmental Measurements Laboratory
U.S. Department of Energy

U-01-RC

ENRICHED URANIUM IN URINE

(see Volume II)

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U-02-RC

ISOTOPIC URANIUM IN BIOLOGICAL AND ENVIRONMENTAL MATERIALS

Contact Person(s) : Isabel M. Fisenne

APPLICATION

This procedure has been used to analyze soft tissue, vegetation, water, and air filter samples (Hindman, 1983; Sill and Williams, 1981; Welford et al., 1960).

Uranium from acid leached, dry-ashed and wet-ashed materials is equilibrated with ^{232}U tracer, and is isolated by anion exchange chromatography. The separated U isotopes are microprecipitated for α spectrometry.

SPECIAL APPARATUS

1. Ion exchange columns (see Specification 7.5).

2. Polyethylene dispensing bottles (see Specification 7.11).

3. Special apparatus for the microprecipitation of U are listed under the generic procedure, G-03.

* *Environmental Protection Agency - Guidelines Establishing Test Procedures for the Analysis of Pollutants, Under the Clean Water Act; National Primary Water Regulations and National Secondary Drinking Water Regulations; Methods Update, tentatively slated for approval, 66FR3466-3497, January 16, 2001.*

SPECIAL REAGENTS

1. Uranium-232 tracer solution - about 0.3 Bq g^{-1} of solution in a dispensing bottle.

2. Bio Rad AG 1-X4 (100-200 mesh), anion exchange resin (see Specification 7.4).

SAMPLE PREPARATION

A. Vegetation and soft tissue.

1. Dry ash the sample according to the method described in Procedure Sr-02-RC (see **Note 1**).

2. Weigh out 10 g of ash and transfer to a 400-mL beaker.

3. Add a weighed amount of ^{232}U tracer solution (~ 0.03 Bq) from the dispensing bottle (see **Note 2**).

4. Add 200 mL of HNO_3 to the beaker and evaporate slowly to dryness.

5. Add 25 mL of HNO_3 to the beaker. Repeat the acid addition and evaporation until a white residue is obtained. (**Note:** If silicious material is present, transfer the sample to a 100 mL platinum dish or a 100 mL Teflon beaker with HNO_3. Add 10 mL of HF to the vessel and evaporate to dryness. Repeat additions of 25 mL HNO_3 - 10 mL HF as necessary to volatilize the silica. Remove the HF by adding three successive 10-mL volumes of HNO_3 to the vessel and evaporate to dryness.)

6. Add 25 mL of HCl and evaporate to dryness. Repeat the acid addition and evaporation twice more.

7. Heat to dissolve the residue in 50-100 mL of 7N HCl.

8. Continue with **Determination**.

B. Water.

1. Evaporate the H_2O sample to a small volume.

2. Add a weighed amount of ^{232}U tracer solution (~ 0.017 Bq) from a dispensing bottle and evaporate slowly to dryness (see **Note 2**).

3. Add 50 mL of HNO_3 and evaporate to dryness. Add 25 mL of HNO_3 and evaporate twice more.

4. Dissolve the residue in 25 mL of HCl and evaporate to dryness. Repeat the HCl addition and evaporation.

5. Heat to dissolve the residue in ≤ 50 mL of 7N HCl.

6. Continue with **Determination**.

C. Air filters.

Cellulose filters:

1. Add a weighed amount of ^{232}U tracer solution (~ 0.017 Bq) from a dispensing bottle to the filter in a platinum dish and dry ash in an electric muffle at 550°C (see **Note 2**).

2. Dissolve the residue in HNO_3 and transfer to a 250-mL beaker.

3. Add 25 mL of HNO_3 and evaporate to dryness. Repeat the acid addition and evaporation twice more.

4. Dissolve the residue in 25 mL of HCl and evaporate to dryness. Repeat the HCl addition and evaporation twice more.

5. Heat and dissolve the residue in ≤ 50 mL of 7N HCl.

6. Continue with **Determination**.

Glass fiber filters:

1. Place the filter and a magnetic stirring bar in a 400-mL Teflon beaker. Add a weighed amount of ^{232}U tracer solution (~ 0.033 Bq) from a dispensing bottle.

2. Add 100 mL of HNO_3, mechancially stir while heating for 1 h. Reduce the solution volume to ~ 25 mL. Remove the stirring bar and rinse with H_2O.

3. Add 10 mL of HF and evaporate to dryness.

4. Repeat the 25 mL HNO_3 - 10 mL HF additions and evaporations as necessary to volatilize the silica.

5. Add 25 mL of HNO_3 to the beaker and evaporate to dryness. Repeat twice more.

6. Heat and dissolve the residue in 25 mL of HCl and evaporate to dryness. Repeat the HCl addition and evaporation twice more.

7. Dissolve the residue in ≤50 mL of 7N HCl.

8. Continue with **Determination**.

DETERMINATION

1. Pass the 7N HCl sample solution obtained during sample preparation through a prepared anion exchange column (see **Note 3**). Discard the column effluent.

2. Wash the column with 400 mL of 7N HCl. Discard the washings.

3. Elute the uranium with 200 mL of 1N HCl, collecting the eluate in a 250-mL beaker. Discard the resin.

4. Evaporate the eluate to near dryness.

5. Destroy any residual organic material with dropwise additions of HNO_3.

6. Evaporate the solution to dryness. Dissolve the residue in a few drops of HCl.

7. Convert the solution to the chloride with three 5-mL additions of HCl.

8. Add 1-2 mL of 1N HCl, prepared with filtered water (see Procedure G-03, Microprecipitation Source Preparation for Alpha Spectrometry). Cool to room temperature.

9. Continue the analysis under Procedure G-03, Microprecipitation Source Preparation for Alpha Spectrometry.

Notes:

1. Freeze-dried or wet tissue may be wet ashed directly in HNO_3. Proceed with Step 3 of **Vegetation and Soft Tissue**.

2. It is necessary to analyze reagent blanks with each batch of samples to correct the U results.

3. 20 mL of Bio-Rad AG1-X4, prepared according to Specification 7.4 are conditioned with 500 mL of 7N HCl.

LOWER LIMIT OF DETECTION (LLD)

Uranium Isotopes

Counter Efficiency	(%)	40
Counter Background	(cps)	3.33×10^{-6} for ^{238}U
		3.33×10^{-6} for ^{234}U
Yield	(%)	85
Blank	(cps)	3.33×10^{-6} for ^{238}U
		3.33×10^{-5} for ^{234}U
LLD (400 min)	(mBq)	0.23 for ^{238}U
		0.53 for ^{234}U
LLD (1000 min)	(mBq)	0.21 for ^{238}U
		0.48 for ^{234}U
LLD (5000 min)	(mBq)	0.065 for ^{238}U
		0.15 for ^{234}U

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"Neodymium Fluoride Mounting for Alpha Spectrometric Determination of Uranium, Plutonium and Americium"
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"Urinary Uranium Levels in Non-Exposed Individuals"
Am. Ind. Hyg. Asso. J., 21 (1960)

U-03-RC

ISOTOPIC URANIUM IN BONE ASH

Contact Person(s) : Isabel M. Fisenne

APPLICATION

This procedure has been used to analyze 50 g human bone ash samples (Fisenne et al., 1980; Hindman, 1983; Sill and Williams, 1981).

Bone ash is dissolved in acid, and the U is equilibrated with ^{232}U tracer and isolated by solvent extraction. The purified U isotopes are microprecipitated for α spectrometry.

SPECIAL APPARATUS

1. Mechanical shaker.

2. Polyethylene dispensing bottle - see Specification 7.10.

3. Special apparatus for the microprecipitation of U are listed under the generic procedure, G-03.

SPECIAL REAGENTS

1. Uranium-232 tracer solution - about 0.1 Bq g^{-1} of solution in a dispensing bottle.

2. Alamine-336, tertiary tricaprylyl amine (Henkel Corporation, 2430 N. Huachuca Dr., Tucson, AZ 85745-1273) 3:7 in toluene. Wash twice with an equal volume of 1:1 HCl. Prepare 100 mL of acid-washed 3:7 Alamine-336 for each sample.

3. Standardized sodium hydroxide 0.1N - dissolve 4 g of NaOH in H_2O and dilute to 1 L. Standardize the solution against potassium acid phthalate.

4. Phenolphthalein indicator - dissolve 0.5 g of reagent in 100 mL of 95% ethanol.

SAMPLE PREPARATION

1. Weigh 50 g of ground, dry-ashed bone and transfer to a 400-mL beaker.

2. Add a weighed amount of ^{232}U tracer (~ 0.01 Bq per sample) from the dispensing bottle. (**Note:** It is necessary to analyze reagent blanks with each batch of samples to correct the U results.)

3. Add 100 mL of HCl and heat gently on a hot plate for 10 min with occasional stirring.

4. Add 70 mL of H_2O and stir to obtain a clear solution. If insoluble material is present, filter the sample through a glass fiber filter. Wash the filter with 1:1 HCl and discard the residue.

5. Cool the solution. Transfer a 100-µL aliquot of the sample into a 150-mL beaker containing 25 mL of H_2O. Add two to three drops of 0.5% phenolphthalein indicator. Stir and titrate the solution with 0.1N NaOH to the pink endpoint. Calculate the normality of the sample solution.

6. If the normality is >5.8N in HCl, proceed directly to the extraction. If the normality is <5.8N, transfer the sample to a 250-mL graduated cylinder and record the volume. Return the sample to the beaker and add an appropriate volume of HCl to the cylinder to increase the sample acid concentration to 6N. Transfer the acid to the sample beaker and proceed with the extraction.

DETERMINATION

1. Transfer 50 mL of acid-washed Alamine-336 into each of two 500-mL separatory funnels.

2. Transfer the sample to the first separatory funnel. Wash the beaker with 1:1 HCl and add the washings to the funnel.

3. Shake the separatory funnel for 5 min. Allow the phases to separate and draw off the aqueous (lower) phase into the second separatory funnel. Retain the organic phase in the first funnel.

4. Shake the second separatory funnel for 5 min. Allow the phases to separate, draw off, and then discard the aqueous (lower) phase.

5. Combine the two organic phases in one of the separatory funnels.

6. Wash the organic phase four times for 5 min with equal volumes of 1:1 HCl. Discard the washings.

7. Strip the U from the organic phase by shaking twice for 5 min with 100-mL portions of 0.1N HCl. Combine the strip solutions in a 400-mL beaker.

8 Evaporate the solution to near dryness.

9. Destroy any residual organic material with dropwise additions of HNO_3.

10. Evaporate the solution to dryness. Dissolve the residue in a few drops of HCl.

11. Convert the solution to the chloride with three 5-mL additions of HCl.

12. Add 1-2 mL of 1N HCl, prepared with filtered water (see Procedure G-03, Microprecipitation Source Preparation for Alpha Spectrometry). Cool to room temperature.

13. Continue the analysis under Procedure G-03, Microprecipitation Source Preparation for Alpha Spectrometry.

LOWER LIMIT OF DETECTION (LLD)

Counter Efficiency	(%)	40
Counter Background	(cps)	3.33×10^{-6} for ^{238}U 6.67×10^{-6} for ^{234}U
Yield	(%)	90
Blank	(cps)	3.33×10^{-6} for ^{238}U 3.30×10^{-5} for ^{234}U
LLD (400 min)	(mBq)	0.2 for ^{238}U 0.5 for ^{234}U
LLD (1000 min)	(mBq)	0.1 for ^{238}U 0.3 for ^{234}U
LLD (5000 min)	(mBq)	0.05 for ^{238}U 0.1 for ^{234}U

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Fisenne, I. M., P. M. Perry, and G. A. Welford
"Determination of Uranium Isotopes in Human Bone Ash"
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