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**TENTATIVE METHOD FOR THE DETERMINATION OF URANIUM
ISOTOPES IN SOIL AND AIR SAMPLES**

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PREFACE

The analytical procedure described in this document is a tentative method for the determination of uranium isotopes in soil and air samples. It is being collaboratively tested according to an interagency agreement between the U.S. Environmental Protection Agency (USEPA) and the U.S. Department of Energy (USDOE). Data from the collaborative test will be examined and information on the precision and accuracy of the method will be obtained. A final report describing the results of the collaborative study will be prepared.

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1. SCOPE AND APPLICATION

1.1 General Considerations

This procedure applies to the determination of uranium isotopes in soil or air filter samples. It has been applied to soils from various parts of the U.S. and to both fiber glass and polystyrene air filters. Analysis of soils for uranium at levels greater than 0.01 dpm/g can be performed using this method for 10 gram samples. Although 10 g samples are recommended, it can be applied to samples of up to 50g of soil.

The method described in this procedure can be carried out by an experienced technician under the supervision of a chemist who fully understands the concepts involved in the analysis. It involves relatively simple operations, but it should be utilized only after satisfactory results are obtained by the analyst when replicate standard samples are analyzed.

1.2 Minimum Detectable Activity

The minimum detectable concentration of a given uranium isotope is dependent upon the size of the sample analyzed and the minimum detectable activity (MDA) of the isotope. In a given laboratory situation this will also depend upon the observed blank values (cf. Section 3). The MDA is defined as that amount of activity which in the same counting time, gives a count which is different from the background count by three times the standard deviation of the background count.

The MDA can be calculated from some typical parameters that might be expected from the method described in this procedure. Suppose a sample is counted for 1000 minutes using a silicon surface barrier detector with a 25% counting efficiency, the background in the energy region of interest is 5 counts in 1000 minutes, and the chemical recovery of the uranium tracer is 80%. From the definition of the MDA,

the sample count would have to be seven (ca. $3 \times \sqrt{5}$) in 1000 minutes, and to achieve this count under the stated conditions would require 0.034 d/m of activity. Thus, a typical MDA for a given uranium isotope by this procedure would be 0.03 d/m.

1.3 Sensitivity

Using the minimum detectable activity computed in the last section and considering a 10 gram soil sample, the lowest concentration of uranium-238, as mg U-238/g of soil, that could be detected by this method would be:

$$0.034 \text{ d/m} / (0.739 \text{ d/m/mg} \times 10\text{g}) = 0.0046 \text{ mg/g.}$$

The lowest concentration of U-238 detectable per air filter is the same as the MDA, i.e. 0.046 mg/filter.

The sensitivity of this isotopic method can be compared (Bishop, 1977) to methods which determine total uranium concentrations fluorometrically. Although fluorometric methods are comparable in sensitivity to counting methods for U-238 (natural uranium), fluorometric methods could not detect other uranium isotopes which are readily detectable by counting methods. This is because the isotopic method can be used to detect much smaller masses of these other uranium isotopes which have high specific activities. The long lived isotopes of uranium along with some of their properties of interest to this procedure are given in Table 1.

PROPERTIES OF URANIUM ISOTOPES
OF INTEREST IN ENVIRONMENTAL SAMPLES

Isotope	Half-Life (Years)	Specific Activity (dis/min/ g)	Alpha Energies in MeV (Abundance)
U-232	72	4.75×10^7	5.32 (68%), 5.27 (32%)
U-233	1.62×10^5	21,000	4.82 (83%), 4.78 (15%)
U-234	2.47×10^5	13,730	4.77 (72%), 4.72 (28%)
U-235	7.1×10^8	4.76	4.58 (8%), 4.40 (57%) 4.37 (18%)
U-236	2.39×10^7	141	4.49 (76%), 4.44 (24%)
U-238	4.51×10^9	0.739	4.20 (75%), 4.15 (25%)

1.4 Precision and Accuracy

The precision of the method has not yet been evaluated, but based on experience with a similar procedure for uranium in water, the precision is expected to approach that of counting statistics errors. The accuracy is expected to be within the limits propagated from counting statistics and the uncertainty in the specific activity of the tracer used.

2. SUMMARY

The present procedure involves a nitric acid-hydrofluoric acid dissolution, coprecipitation of uranium with iron hydroxide, an ether extraction if the sample contains iron as a major constituent, separation of uranium by anion exchange, and electrodeposition of the uranium followed by alpha pulse height analysis.

After adding uranium-232 tracer, organic matter is removed from the sample by heating. Subsequently, the sample is decomposed by a nitric acid-hydrofluoric acid digestion, and the uranium is coprecipitated by making this solution basic with ammonium hydroxide (carbonate free). The hydroxide precipitate is dissolved in 8 M hydrochloric acid, which is extracted with isopropyl ether to remove the bulk of the iron present. For samples with relatively low iron concentrations, such as air filters containing little particulate matter, this extraction can be omitted.

The 8 M hydrochloric acid solution is passed through an anion exchange resin column. Uranium, polonium and bismuth will be adsorbed on the resin while thorium and radium will pass through the column. Plutonium and any unextracted iron are also retained by the resin, but are eluted with 6 M HCl containing hydrogen iodide. The iodide ion reduces plutonium (IV) to plutonium (III) and reduces the iron (III) to iron (II), and neither of these ions are retained by the ion exchange resin in 6 M HCl. The uranium is eluted from the column with 1.0 M HCl, while any zinc adsorbed on the column will remain. Electrodeposition of the uranium onto a stainless steel slide is performed, and the slide is counted by alpha pulse height analysis using a silicon surface barrier detector.

When uranium-232 is used as a tracer, the other isotopes of uranium listed in Table 1 can be detected in the alpha spectrum of an unknown sample. From the alpha energies given in Table 1, it can be seen that the alpha energy of uranium-232 is at least 0.50 MeV higher than the energy of any other uranium isotope. Thus, there should be little interference from tailing of the uranium-232 into the lower energy alpha peaks. If a sample contained both uranium-233 and uranium-234, it would be very difficult to resolve these two peaks since their principal alpha energies differ by only 0.05 MeV. The other uranium peaks can be resolved if the resolution is good.

3. INTERFERENCES

- 3.1 The only possible alpha activity that may come through the chemistry of the procedure is protactinium-231 (Edwards, 1968). Protactinium-231 has the following alpha energies in MeV, the abundance being given in parantheses: 5.06 (10%), 5.02 (23%), 5.01 (24%), 4.95 (22%) and 4.73 (11%). Thus, from Table 1, it can be seen that this protactinium-231 could possibly interfere with the determination of uranium-233 or uranium-234.
- 3.2 In determining very low levels of uranium isotopes in environmental samples, detector backgrounds and laboratory blanks must be accurately determined. Blank determinations must be made in order to ascertain that the contamination from reagents, glassware and other laboratory apparatus is negligible compared to the sample that is being analyzed. A blank determination should be made in exactly the same way a sample determination is made. For accurate analyses blank determinations should be consistent, and should be an order of magnitude lower than activities obtained when analyzing samples.

4. APPARATUS

4.1 Instrumentation

- 4.1.1 Alpha Pulse Height Analysis System - A system consisting of a silicon surface barrier detector capable of giving a resolution of 50 keV or better with samples electrodeposited on flat mirror finished

stainless steel slides is used. The resolution here is defined as the width of the alpha peak in keV, when the counts on either side of the peak are equal to one-half of the counts at the maximum of the peak. The counting efficiency of the system should be greater than 15% and the background in the energy region of each peak should be less than 10 counts in 1000 minutes.

- 4.1.2 Electrodeposition Apparatus - A direct current power supply, 0-12 volts and 0-2 amps, is required for the electrodeposition described in this procedure. A disposable electrodeposition cell is also recommended. An apparatus similar to that shown in Figure 1 has been used in the present procedure. In the present procedure, the cell itself is surrounded by water, but the water is not circulated. The electrodeposition can be carried out without the water cooling. The cathode is a stainless steel slide with a polished mirror finish. The diameter of the slide is 1.91 cm (3/4 in.) and the thickness is ca. 0.05 cm (0.02 in.). The exposed cathode area during electrodeposition is 2 cm².

The anode is a 1 mm diameter platinum wire with an 8 mm diameter loop at the end above the cathode.

4.2 Laboratory Equipment

- 4.2.1 Balance - top loading, capacity 1200 g, precision ± 0.1 g.
- 4.2.2 Balance - analytical, capacity 160 g, precision ± 0.1 mg
- 4.2.3 Hot plate - magnetic stirrer and stirrer bar.
- 4.2.4 Centrifuge - capable of handling 100 ml or larger centrifuge bottles.

Figure 1. Water Cooled Electrodeposition Apparatus

4.3 Labware

- 4.3.1 Graduated cylinders - 25 ml to 100 ml.
- 4.3.2 Beakers - glass, 100 ml to 250 ml.
- 4.3.3 Beakers - teflon, 250 ml with teflon covers.
- 4.3.4 pH paper - pH range 2 to 10.
- 4.3.5 Automatic pipettes - with disposable tips, volumes between 100 λ and 1000 λ .
- 4.3.6 Centrifuge bottles - 250 ml.
- 4.3.7 Ion exchange columns - approximately 1.3 cm ID, 15 cm long with 100 ml reservoir.
- 4.3.8 Pipettes - glass, Class A.
- 4.3.9 Disposable pipettes - 2 ml glass eye-dropper type, with rubber bulb.
- 4.3.10 Dropping bottles.
- 4.3.11 Watch glasses.
- 4.3.12 Polyethylene washing bottles.
- 4.3.13 Glass stirring rods
- 4.3.14 Safety glasses or goggles.
- 4.3.15 Rubber gloves.
- 4.3.16 Crucible tongs - platinum tipped.
- 4.3.17 Beaker tongs.
- 4.3.18 Spatulas.
- 4.3.19 Heat lamp-mounted on ring stand for drying slides.

5. STANDARDS, ACID, REAGENTS

5.1 Standards

5.1.1 Standardized uranium-232 solution.

5.2 Acids

Reagent grade, meeting American Chemical Society (ACS) specifications; diluted solutions prepared with distilled deionized water.

5.2.1 Nitric acid - concentrated (16 M).

5.2.2 Hydrochloric acid - concentrated (12 M, 8 M, 6 M, 1.0 M, 0.5 M).

5.2.3 Sulfuric acid - concentrated (18 M), 1.8 M.

5.2.4 Hydrofluoric acid - concentrated (48% solution).

5.2.5 Hydroiodic acid - concentrated (48% solution).

5.3 Reagents

Reagent grade, meeting ACS specifications; solutions prepared with distilled deionized water.

5.3.1 Ferric chloride - in 0.5 M HCl containing 10 mg of iron per ml of solution.

5.3.2 Ammonium hydroxide - concentrated (15 M), 1.5 M, 0.15 M.

5.3.3 Anion exchange resin - Bio Rad AG1-X4 (100-200 mesh) chloride form. (Available from Bio Rad. Laboratories, 3rd and Griffin Avenues, Richmond, California, 94804). A column is prepared by slurring this resin with 8 M HCl and pouring it onto a column of inside diameter approximately

1.3 cm. The height of the column of resin should be about 10 cm.

- 5.3.4 Sodium hydrogen sulfate solution ca. 5% in 1 M H_2SO_4 ; dissolve 10 g of the $\text{NaHSO}_4 \cdot \text{H}_2\text{O}$ in 100 ml of water and then carefully add 100 ml of 2 M H_2SO_4 .
- 5.3.5 Preadjusted electrolyte - 1 M ammonium sulfate adjusted to pH 3.5 with 15 M NH_4OH and 18 M H_2SO_4 .
- 5.3.6 Thymol blue indicator, sodium salt (available from Fisher Scientific Company) - 0.04% solution.
- 5.3.7 Ethyl alcohol - made slightly basic with a few drops of 15 M NH_4OH per 100 ml of alcohol.

6. CALIBRATION AND STANDARDIZATION

6.1 Standardization of the Uranium-232 Tracer Solution

If a standard uranium-232 solution is not available, a freshly purified sample of uranium-232 could be standardized by mixing a known amount of another standard solution such as uranium-236, with a known amount of the solution to be standardized, mixing thoroughly and electroplating the mixture. From an alpha pulse height analysis of the mixture the specific activity of the uranium-232 solution could be determined. Alternately, a freshly purified solution of uranium-232 could be standardized by 2π counting. Weighed aliquots of the solution free of hydrochloric acid could be evaporated on platinum or stainless steel slides and counted with a 2π proportional counter. The efficiency of the 2π proportional counter can be accurately determined with a National Bureau of Standards alpha-particle standard. When using these standards, corrections for resolving time and backscattering must be made if necessary.

6.2 Determination of Alpha Spectrometer Efficiency

Determination of the alpha spectrometer counting efficiency is not necessary to get an accurate concentration of the uranium isotopes in the sample being analyzed, since the counting efficiency of the electroplated sample is the same for the tracer uranium isotope and for the unknown uranium isotope. This efficiency cancels out when the concentration of the unknown uranium isotope is calculated (cf. Section 8.1).

A determination of the alpha spectrometer counting efficiency is required to calculate the uranium recovery of a particular analysis. To determine this efficiency requires that one count an alpha particle source of known alpha particle emission rate under the same conditions that the samples are counted. The alpha particle counting efficiency is then calculated as illustrated in Section 8.2.

7. STEP BY STEP PROCEDURE FOR ANALYSIS

7.1 Acid Dissolution

- 7.1.1 Weigh a representative 10.0 ± 0.1 grams of 100 mesh soil sample or the glass fiber filter(s) and transfer to a casserole. If a Microsorban (polystyrene) filter(s) was used, place the filter(s) into a 150-ml or 250-ml Pyrex beaker and cover with a watch glass.
- 7.1.2 Add an appropriate quantity of uranium-232 tracer. If the activity is expected to be less than 1 dpm/g, or is unknown, add 10 dpm of tracer. For higher levels add as much uranium-232 tracer as the estimated activity of uranium in the sample. If a Microsorban filter(s) was used, add the uranium-232 tracer after Step 7.1.3.
- 7.1.3 Heat the casserole containing the soil sample or glass fiber filter(s) at 600°C for four hours in a muffle furnace; remove, and cool. If a Microsorban filter(s) was used, place the Pyrex beaker in a muffle furnace at 200°C for 1 hour. Increase the temperature at 1 hr intervals to 300°C , 400°C and 550°C . Muffle for 2 or 3 hrs at 550°C or until only a brown podery ash remains. Remove and cool.
- 7.1.4 Transfer the sample to a 250-ml Teflon beaker, rinsing the casserole with 10 ml portions of 16 M HNO_3 to a final volume of 60 ml of nitric acid. For Microsorban filter(s) rinse the beaker with 5 ml portions of 16 M HNO_3 to a final volume of 30 ml of nitric acid.

- 7.1.5 Carefully add 30 ml of 48% hydrofluoric acid (20 ml for Microsorban filters), cover with a teflon watchglass and heat on a hotplate with frequent stirring for about 1 hr. Remove from the hotplate and cool. (CAUTION: HF is very hazardous. Wear rubber gloves, safety glasses or goggles, and a laboratory coat. Avoid breathing HF fumes. Clean up all spills and wash thoroughly after using HF).
- 7.1.6 Carefully add 30 ml (20 ml for Microsorban filters) each of 16 M HNO_3 and HF (48%) and digest with some stirring for an additional hour.
- 7.1.7 Remove from the hotplate and cool to room temperature. Slowly add 20 ml of 12 M HCl and heat on a hotplate with some stirring until the solution has evaporated to a liquid volume of approximately 10 ml.
- 7.1.8 Add 50 ml of distilled water and digest on a hot plate with stirring for 10 minutes to dissolve the soluble salts.
- 7.1.9 Cool and transfer the total sample into a 250-ml centrifuge bottle with a minimum of distilled water from a wash bottle. If any insoluble residue is present, the sample should be centrifuged and the supernate should be transferred into another centrifuge bottle. This residue should be washed with 10 ml of 1.0 M HCl which is added to the supernate. Discard the insoluble residue. Proceed to section 7.2, Coprecipitation.

7.2 Coprecipitation

- 7.2.1 Add 20 mg of iron as FeCl_3 in 0.5 M HCl to the centrifuge bottle and stir. (This step is usually not necessary for soil samples containing iron).
- 7.2.2 Add 15 M NH_4OH (CO_3^{2-} free) while stirring to precipitate the iron. Continue adding 15 M NH_4OH to raise the pH to 9-10 as determined by pH paper,⁴ then add 5 ml in excess.

- 7.2.3 Centrifuge for approximately 5 minutes, decant and discard the supernate.
- 7.2.4 Dissolve the precipitate with a minimum of 12 M HCl and add 8 M HCl to a volume of about 50 ml. Transfer the solution to a 250-ml separatory funnel using two 5-ml rinses of 8 M HCl and proceed to Section 7.3, Ether Extraction.

7.3 Ether Extraction

- 7.3.1 Add 60 ml of isopropyl ether to the separatory funnel and shake the solution for two minutes. Allow the phases to separate and then transfer the aqueous phase (lower phase) to a second separatory funnel. Add 5-ml of 12 M HCl to the aqueous phase.
- 7.3.2 Repeat this ether extraction two more times. The bulk of the iron is removed as evidenced by the appearance of a yellow color in the organic phase. (If the sample has a very high concentration of iron, additional extractions may be necessary.)
- 7.3.3 Transfer the aqueous phase to a 150-ml beaker, boil for 15 min, and proceed with Section 7.4, Anion Exchange Separation.

7.4 Anion Exchange Separation

- 7.4.1 Condition the anion exchange resin column (prepared as described in Section 5.3) by rinsing the column with 4 column volumes of 8 M HCl.
- 7.4.2 Transfer the sample from Step 7.3.3 to the conditioned anion exchange resin.
- 7.4.3 After the sample has passed through the column, elute any unextracted iron (and plutonium if present) with 6 column volumes of 6 M HCl containing 1 ml of concentrated HI per 50 ml of 6 M HCl (freshly prepared).

- 7.4.4 Rinse the column with an additional two column volumes of 6 M HCl.
- 7.4.5 Elute the uranium with six column volumes of 1.0 M HCl.
- 7.4.6 Evaporate the sample to about 20 ml and add 5 ml of 16 M HNO₃.
- 7.4.7 Evaporate the sample to near dryness.

7.5 Electrodeposition

- 7.5.1 Add 2 ml of a 5% solution of NaHSO₄·H₂O in 1 M H₂SO₄ to the sample.
- 7.5.2 Add 5 ml of 16 M HNO₃, mix well and evaporate to dryness, but do not bake.
- 7.5.3 Dissolve the sample in 5 ml of the preadjusted electrolyte (cf. Section 5.3), warming to hasten the dissolution.
- 7.5.4 Transfer the solution to the electrodeposition cell using an additional 5-10 ml of the electrolyte in small increments to rinse the sample container.
- 7.5.5 Add three or four drops of thymol blue indicator solution. If the color is not salmon pink, add 1.8 M H₂SO₄ (or 15 M NH₄OH) until this color is obtained.
- 7.5.6 Place the platinum anode into the solution so that it is about 1 cm above the stainless steel slide which serves as the cathode.
- 7.5.7 Connect the electrodes to the source of current, turn the power on, and adjust the power supply to give a current of 1.2 amps. (Constant current power supplies will require no further adjustments during the electrodeposition.)
- 7.5.8 Continue the electrodeposition for a total of 1 hr.

- 7.5.9 When the electrodeposition is to be terminated, add 1 ml of 15 M NH_4OH and continue the electrodeposition for 1 min.
- 7.5.10 Remove the anode from the cell and then turn off the power.
- 7.5.11 Discard the solution in the cell and rinse the cell 2 or 3 times with 0.15 M NH_4OH .
- 7.5.12 Disassemble the cell and wash the slide with ethyl alcohol that has been made basic with NH_4OH .
- 7.5.13 Touch the edge of the slide to a tissue to absorb the alcohol from the slide.
- 7.5.14 Dry the slide, place it in a box, and label for counting. (The sample should be counted within a week, since uranium-232 daughters grow into the sample and possibly interfere with the determination of certain other uranium activities.)

7.6 Alpha Pulse Height Analysis

- 7.6.1 Count the samples for at least 1000 minutes or longer if the detector efficiency is less than 15%, if the tracer recovery is low, or if the unknown uranium activity is low.
- 7.6.2 Check the alpha pulse height analysis spectrum for peaks at the uranium-233, uranium-234, uranium-235, uranium-236 and/or uranium-238 alpha energies (see Table 1) and determine the total counts in each peak. Where two isotopes are close in energy, complete resolution may not be possible. (This is the case with uranium-236 and uranium-235, for example.)
- 7.6.3 Make the necessary background corrections. (The background should be determined by a 4000 minute or longer count.)

- 7.6.4 Make a blank correction for each peak, if necessary.

8. CALCULATION OF RESULTS

8.1 Calculations of Uranium Concentrations

By integrating the appropriate energy peak along with the uranium-232 tracer peak, the concentrations of the corresponding uranium isotope can be determined (cf. Table 1). This concentration is calculated as follows:

$$X_i = \frac{C_i \times A_t}{C_t \times W_s} \quad (8.1)$$

where X_i = the concentration of the unknown uranium isotope in d/m/g of soil or d/m/ filter.

C_i = the net sample counts in the energy region corresponding to the uranium isotope being measured.

A_t = the activity of the uranium-232 tracer added to the sample in d/m.

C_t = the net sample counts in the uranium-232 tracer energy region of the alpha spectrum.

W_s = the weight in grams of the soil sample taken for analysis; W_s is omitted from this formula ($W_s=1$) when the sample is an air filter.

8.2 Calculation of Alpha Spectrometer Efficiency

The absolute counting efficiency of the alpha spectrometer, ϵ , must be determined in order to calculate the uranium recovery of the analytical procedure.

To determine this efficiency requires a standard source of a known alpha particle emission rate:

$$\epsilon = R_s / R_\alpha \quad (8.2)$$

where R_s = the net counting rate of the standard source in the energy region of the alpha emitter of interest in counts per minute.

R_α = the absolute alpha particle emission rate of the alpha emitter of interest in alpha disintegrations per minute.

8.3 Calculation of Uranium Recovery of the Chemical Analysis

The uranium recovery efficiency E (%) expressed in percent is given by:

$$E(\%) = \frac{C_t \times 100\%}{t \times A_t \times \epsilon} \quad (8.3)$$

where t =the counting time in minutes and the other terms are as defined in Sections 8.1 and 8.2.

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