

**\*EPA APPROVED\***

*Ra-04-RC*

**RADIUM-226 IN TAP WATER, URINE, AND FECES**

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**APPLICATION**

The procedure presented has been applied to tap water, ashed urine, and feces samples. Only  $^{226}\text{Ra}$  yields  $^{222}\text{Rn}$  progeny that has suitable characteristics for detection by an emanation technique; therefore, the procedure is specific.

After sample preparation, radium is isolated from most other elements by coprecipitation with barium sulfate. Further purification is obtained by the removal of silica with HF and reprecipitation of the sulfate. The sulfate precipitate is dissolved in alkaline EDTA to prepare the emanating solution. The chemical yield of barium is determined with the  $\gamma$ -emitting tracer  $^{133}\text{Ba}$ .

**SPECIAL APPARATUS**

Radon bubblers - see Specification 7.7.

**SPECIAL REAGENT**

1. Barium carrier solution - 20 mg mL<sup>-1</sup> - 30.4 g BaCl<sub>2</sub> L<sup>-1</sup> in 1:99 HCl.
2. Ammonium sulfate solution - 100 g (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> L<sup>-1</sup> in water.

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*\* 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.*

3. Aerosol OT solution - 0.1%.
4. EDTA solution - 300 g tetrasodium salt of EDTA L<sup>-1</sup> in water.
5. EDTA wash solution - 1:9 dilution of EDTA solution.
6. Ammonium acetate solution - 15 g L<sup>-1</sup> in water.
7. Acetic acid solution - 20 mL glacial acetic L<sup>-1</sup> in water.
8. Triethanolamine - 1:1 in water.

## SAMPLE PREPARATION

### A. Tap water.

1. Transfer two 0.5-L of tap water to a 3 L beaker.
2. Add 25 mL of HCl and 1 mL of barium carrier solution. Add a weighed aliquot (about 0.1 g) of the <sup>133</sup>Ba tracer solution.
3. Evaporate and add an additional two 0.5-L aliquots of tap water until a 10-L collection has been obtained. Evaporate gently to about 100 mL.
4. Transfer to a 400-mL beaker with water, policing the sides of the 3-L beaker thoroughly. Evaporate gently to about 100-mL.
5. Adjust the pH to 4 with 1:1 NH<sub>4</sub>OH and proceed with **Determination**.

### B. Urine.

1. Weigh an aliquot of <sup>133</sup>Ba tracer solution (about 0.1 g) into a 2-L beaker containing a small amount of water and 1 mL of barium carrier solution.
2. Add 1500 mL of urine, then 100 mL of HNO<sub>3</sub> and evaporate to about 1-L.

3. Slowly add 100-mL of  $\text{HNO}_3$ .
4. Repeat the evaporation and addition of acid until a total of 500-mL of  $\text{HNO}_3$  has been added.
5. Evaporate to about 20 mL. Transfer to a 400-mL beaker with water.
6. Adjust the pH to 4 with 1:1  $\text{NH}_4\text{OH}$  and proceed with Determination.

### C. Feces ash.

1. Weigh 1 g of fecal ash into a 150-mL beaker. Add a weighed aliquot (about 0.1 g) of  $^{133}\text{Ba}$  tracer solution and 1 mL of barium carrier solution.
2. Cover the ash with a small quantity of water and slowly add 10 mL of  $\text{HNO}_3$ . Evaporate to a small volume on a medium hot plate.
3. Add about 25 mL of water. Add  $\text{NH}_4\text{OH}$  until a permanent hydroxide flock forms (3-5 mL).
4. Dissolve the flock in a few drops of  $\text{HNO}_3$ .
5. Add 2 mL of  $\text{NH}_4\text{Ac}$  solution and 1 mL of acetic acid solution. Dilute to about 100-mL and proceed with **Determination**.

### DETERMINATION

1. Add 1 mL of  $(\text{NH}_4)_2\text{SO}_4$  solution and allow the sample to digest for 1 h at room temperature.
2. Filter by gravity on a 9 cm Whatman No. 42 filter paper. Wash the paper thoroughly with water. Discard the filtrate and washings.
3. Transfer the paper to a platinum dish. Dry the paper and then ash at  $500^\circ\text{C}$  for about 1 h.

4. Add 1 mL of  $\text{H}_2\text{SO}_4$  and 2 mL of HF. Evaporate to  $\text{SO}_3$  fumes.
5. Cool and transfer to a 90 mL centrifuge tube with water.
6. Police the dish and add the washings to the centrifuge tube.
7. Stir and let stand for 0.5 h.
8. Centrifuge at 2000 rpm for 1 h. Add one drop of 0.1% Aerosol OT. Decant carefully and discard the supernate.
9. Heat a solution of EDTA ( $300 \text{ g L}^{-1}$  EDTA) in an  $85^\circ\text{C}$  water bath.
10. Break up the  $\text{BaSO}_4$  precipitate with a stirring rod.
11. Add 1 mL of 1:1 triethanolamine and 5 mL of the hot EDTA solution, and stir. Wash down the sides of the tube with water.
12. Digest in the steam bath for 15 min, stirring occasionally.
13. Transfer the solution to a 30-mL polyethylene bottle.
14. Dilute the sample to the same liquid level as a known aliquot (about 0.1 g) of  $^{133}\text{Ba}$  solution diluted to 25 mL in a 30 mL polyethylene bottle.
15. Gamma count samples and standard on a flat crystal to determine the chemical yield of barium.
16. Transfer the sample solution to a radon bubbler.
17. De-emanate radon by bubbling with forming gas for about 10 min at  $100 \text{ mL}^{-1} \text{ min}$  as described in Procedure Ra-03-RC. Record the time as the starting time for the radon build-up period. Continue the analysis by the emanation technique.

LOWER LIMIT OF DETECTION (LLD)\*

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		A	B	C
Counter Efficiency	(%)	57.5	57.5	57.5
Counter Background	(cps)	0.0028	0.0028	0.0028
Yield	(%)	90	80	85
Blank	(cps)	0.0012	0.0020	0.0012
LLD (400 min)	(mBq)	0.33	0.45	0.39
LLD (1000 min)	(mBq)	0.17	0.29	0.15

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\*Reagent blanks must be analyzed with each set of samples.

Pulse ionization chamber:

A = Tap water

B = Urine

C = Feces