Ra-03-RC

RADIUM-226 IN SOIL, VEGETATION ASH, AND ION EXCHANGE RESIN

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APPLICATION

This procedure is applicable to 5 g samples of soil and 10 g of vegetation ash or to ion exchange resin from sampling columns.

Soil, vegetation ash or ion exchange resin are prepared for ^{222}Rn emanation measurement. The sample is pretreated with HNO_3-HF, fused with KF and transposed to pyrosulfate (Sill, 1961). The cake is dissolved in dilute HCl. $RaBaSO_4$ is precipitated, filtered, and dissolved in alkaline EDTA. The chemical yield is determined with the γ-emitting tracer ^{133}Ba.

SPECIAL APPARATUS

1. Radon bubblers - see Specification 7.7.

2. 100 mL platinum dishes or 250 mL platinum crucibles.

3. Millipore filter setup - 47 mm diameter.

4. Millipore filters - 47 mm diameter, 0.45 μm pore size.

SPECIAL REAGENTS

1. Barium-133 tracer solution - about 50 cps per 0.1-g aliquot, prepared in 1:99 HCl.

2. Barium carrier solution (20 mg mL^{-1}) - 30.4 g $BaCl_2$ L^{-1} in 1:99 HCl.

3. EDTA solution - 300 g tetrasodium salt of EDTA L^{-1} of water.

4. Triethanolamine - 1:1 in water.

SAMPLE PREPARATION

A. Soil and vegetation.

1. Weigh 5 g of soil or 10 g of vegetation ash into a 100-mL platinum dish. Add a weighed aliquot (about 0.1 g) of ^{133}Ba tracer solution and 1 mL of Ba carrier solution.

2. Slowly add 10 mL of HNO_3 and 10 mL of HF to the sample and evaporate on a hot plate to near dryness.

3. Continue the analysis as described below.

B. Ion exchange resin.

1. Transfer the resin and paper pulp from the collection column to a 250-mL platinum crucible. Dry under a heat lamp and ash at 500ºC in a muffle furnace for 48 h.

2. To the cooled crucible, add a weighed aliquot (about 0.1 g) of ^{133}Ba tracer solution and 1 mL of barium carrier solution.

3. Continue the analysis as described below.

DETERMINATION

1. Weigh out 15 g of KF and add to the sample. Press the KF into the sample with a plastic spatula.

   Caution - wear rubber gloves and safety glasses during Steps 2-5.

2. Fuse the sample over an air fed Meker burner, gradually increasing the temperature until a clear melt is obtained. Cool the melt.

3. Using a burette, slowly add 17.5 mL of H_2SO_4 to the melt. Heat the dish on a hot plate until a clear melt accompanied by dense fumes is obtained. Cool the melt.

4. Weigh out 10 g of Na_2SO_4, add to the dish and fuse over the Meker burner until a clear melt accompanied by dense fumes is obtained. Cool the melt.

5. Transfer the cake to a 600 mL beaker containing 350 mL of hot water and 25 mL of HCl. Stir the solution to dissolve the cake. Cool for 1 h.

6. Filter the precipitate onto a 47 mm diameter, 0.45 μm pore size Millipore filter into a 1 L sidearm flask, police the beaker, and wash with water. Add the washing to the funnel. Discard the filtrate.

7. Using a strong stream of water from a wash bottle, transfer the precipitate from the filter into a 150 mL beaker. Discard the filter.

8. Add 5 mL of EDTA solution and 1 mL of 1:1 triethanolamine to the beaker. Heat on a warm hot plate for about 15 min, stirring occasionally. Reduce the sample volume to ~15 mL.

9. Gravity filter the warm solution through a Whatman No. 41 filter paper into a 30-mL polyethylene bottle.

10. Wash the beaker dish and filter with hot water. Discard the filter.

11. Dilute the sample to the same liquid level as a known aliquot (about 0.1 g) of the ^{133}Ba tracer solution and dilute to 25 mL in a 30 mL polyethylene bottle.

12. Gamma count the samples and standard to determine the chemical yield of barium.

13. Transfer the sample to a ^{222}Rn bubbler with water.

14. De-emanate ^{222}Rn by bubbling with forming gas for about 10 min at 100 mL min^{-1} as described in ^{226}Radium - Emanation Procedure (see Ra-03-RC). Record the time as the starting time for ^{222}Rn buildup. Continue the analysis by the emanation technique.

LOWER LIMIT OF DETECTION (LLD)*

Counter Efficiency	(%)	57.5
Counter Background	(cps)	0.0028
Yield	(%)	90
Blank	(cps)	0.0020
LLD (400 min)	(mBq)	3.3
LLD (1000 min)	(mBq)	1.7

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

REFERENCE

Sill, C. W.
"Decomposition of Refractory Silicates in Ultramicro Analysis"
Anal. Chem., 33, 1684 (1961)