Eichrom Industries provides total solutions for radiochemical separations challenges. Eichrom not only manufactures state of the art extraction chromatography and ion-exchange products, but also provides R&D and consulting services to enhance the value of these products. ORTEC has organized a number of the procedures developed by Eichrom (and its collaborating partners) for convenient access to our Customers, Sales Team, and Technical Support Specialists. For questions about radiochemical separations and alpha-spectrometry please contact:

Michael Schultz, Application Specialist, ORTEC 801 S. Illinois Avenue, Oak Ridge, TN, 37830-0895, Phone: (865) 481-2446, Fax: (865) 483-0396, Email: michael.schultz@ortec-online.com

Lead-210 in Water

1. Scope

1.1 This procedure describes a method for the separation and measurement of lead-210 in water.

2. Summary of Method

2.1 Pb-210 is concentrated from a 500 mL sample by iron hydroxide scavenge or cation exchange column. Lead is separated from the other elements on an Eichrom Pb resin column. The purified lead fraction is collected and, after allowing for Bi-210 ingrowth, is counted by gas flow proportional counting.

3. Significance of Use

3.1 This method is a rapid, reliable method for measurement of radioactive lead-210 in water samples that is more efficient and cost-effective than traditional procedures.

4. Interference

4.1 Any beta emitter can cause an interference with the measurement of the Bi-210 daughter of Pb-210. This method effectively eliminates these possible interferences.

5. Apparatus

5.1 *Aluminum foil*
5.2 *Beta detector*- Gas-flow proportional counter
5.3 *Centrifuge*
5.4 *Centrifuge tubes (50 mL, plastic)*
5.5 *Column reservoirs, 25 mL available from Eichrom*
5.6 *Fume hood*
5.7 *Filter*- 0.45 micron
5.8 *Filter apparatus*- Gelman apparatus: 0.1 micron, 25 mm filters with polycarbonate base and metal screen, polysulfide funnel and 100 mL polypropylene flask
5.9 *Glass beakers*
5.10 *Glass stir rods*
5.11 *Hot plate*
5.12 *Ion exchange columns* - 1 to 1.5 cm diameter, 10 ml resin volume
5.13 *Stainless steel planchets*- 50.8 mm diameter, 6.4 mm deep flat bottom, cupped planchet.

6. Reagents

6.1 Unless otherwise indicated, all references to water should be understood to mean deionized distilled water.
6.2 *Ammonium hydroxide*, concentrated.
6.3 *Cation exchange resin* - C8-B500-M-H, hydrogen form, 100 to 200 mesh. Available from Eichrom.
6.4 *Ethanol, USP, 100%*
6.5 *Hydrochloric Acid (0.5M)* - Add 42 mL concentrated HCl (sp gr 1.19) to 800 mL water and dilute to 1 liter with water.
6.6 *Iron carrier* (*20 mg/mL*) - Dissolve 9.6 grams of ferric chloride ($FeCl_3 \cdot 6H_2O$) in 70 mL 0.5M HCl and dilute to 100 mL with 0.5M HCl.
6.6 *Lead carrier (10 mg/mL)* - Dissolve 1.6 grams of $Pb(NO_3)_2$ in water and dilute to 100 mL with water
6.7 *Nitric Acid (15.7M)* - concentrated HNO_3.

6.8 *Nitric acid solution (8M)* - Add 510 mL of concentrated nitric acid (sp gr 1.42) to 400 mL of water and dilute to 1 liter with water.
6.8 *Nitric Acid (1M)* - Add 63.5 mL of concentrated HNO_3 to 800 mL water and dilute to 1 liter with water.
6.9 *Nitric Acid (0.1M)* - Add 6.4 mL of concentrated HNO_3 to 800 mL water and dilute to 1 liter with water.
6.10 *Pb Resin prepacked 2mL column* - 100-150 micron particle size.
6.11 *Sulfuric Acid (18.0M)* - concentrated H_2SO_4

7. Procedure

7.1 *Water Sample Preparation:*

7.1.1 If required, filter the sample through a 0.45 micron filter.

7.1.2 If samples larger than 1 L are analyzed, evaporate the sample to approximately 1 L.

7.1.3 Aliquot 500 to 1000 mL of the sample (or enough to meet required detection limit) into an appropriate size beaker.

7.1.4 Acidify the sample to pH 2, with concentrated HNO3 (sp gr 1.42). (0.6 mL per 1000 mL).

7.1.5 Add 1 ml of stable lead carrier and appropriate amount of spike.

7.1.6 *Iron hydroxide precipitation option:*

7.1.6.1 Add 1 mL of iron carrier to sample from step 7.1.5. Cover beaker with a watch glass and heat at near boiling for an hour.

7.1.6.2 Remove watch glass. Add 12 mL concentrated ammonium hydroxide to precipitate iron hydroxide. Stir the solution with a glass rod if necessary. Allow the beakers to heat for another 30 minutes.

7.1.6.3 Let the precipitate settle for at least 2 hours. (preferably overnight) Decant the supernatant. Transfer the precipitate into a 50 mL plastic centrifuge tube.

7.1.6.4 Centrifuge and discard the supernatant.

7.1.6.5 Wash the precipitate with 10 mL water. Centrifuge and discard the supernatant.

7.1.6.6 Repeat step 7.1.6.5 twice.

7.1.6.7 Add 10 mL 1M HNO_3 to dissolve the precipitate. Go to section 7.2

7.1.7 *Cation Exchange option*

7.1.7.1 Prepare a cation exchange column containing 10 ml (10 g) of C8-B500-M-H, 100-200 mesh for a sample size of 500 mL. A larger resin column may be required for a larger sample size. (20 g resin column for 1.0 L sample)

7.1.7.2 Place columns on rack with large volume reservoirs (250 ml to 500 ml).

7.1.7.3 Ensure that a suitable container is below each column.

7.1.7.4 Add 20 ml of 0.1 M HNO3 to each column to condition columns.

7.1.7.5 Load each sample from step 7.1.5 onto the appropriate column and allow to drain.

7.1.7.6 Add 25 ml of 0.1 M HNO3 to each column to rinse.

7.1.7.7 Discard the feed and rinse the solution collected.

7.1.7.8 Ensure that a labeled 150 ml beaker is below each column.

7.1.7.9 Add 50 ml of 8 M HNO3 to each column to elute lead.

7.1.7.10 Place each beaker on a hot plate in a fume hood and evaporate to dryness.

7.1.7.11 Redissolve the residue in 10 mL of 1M HNO_3. Go to section 7.2.

7.2 *Pb Separation using Pb resin*

7.2.1 Condition an Eichrom Pb Resin column with 10 mL 1M HNO_3.

7.2.2 Load the dissolved precipitate from step 7.1.6.7 or 7.1.7.11 onto the column. Discard eluent.

7.2.3 Add 10 mL 1M HNO_3 to the column. Record the time and date of this addition. This will be used to calculate the ingrowth of bismuth-210. Discard the eluent.

Note: This will remove bismuth and iron if present.

7.2.4 Add 10 mL 0.1M HNO_3 to the column and discard the eluent.

7.2.5 Place a clean and labeled centrifuge tube under the column. Add 20 mL of water to the column to elute lead.

7.2.6 Very carefully add 4 mL of concentrated sulfuric acid to each Pb eluent and allow the solution to cool for 10-15 minutes

7.2.7 Cap the tubes and mix the solution well. A white precipitate is formed.

7.3 *Sample preparation for counting*

7.3.1 *Planchet option*

7.3.1.1 Centrifuge the solution from step 7.2.7 and discard the supernatant.

7.3.1.2 Add 10 mL of water to the precipitate and mix well. Centrifuge the solution and discard the supernatant.

7.3.1.3 Repeat step 7.3.1.2 one more time

7.3.1.4 Add 5 mL of DI water to the precipitate and mix well to slurry (a plastic disposable pipet could be used).

7.3.1.5 For each sample analyzed, clean a 2 inch diameter counting planchet by moistening a paper towel with ethanol, wiping the dish and letting it dry.

7.3.1.6 Weigh the counting planchet on an analytical balance and record the weight to 0.0001 gram.

7.3.1.7 Place each planchet under a heat lamp in a hood or on a hot plate with low heat.

7.3.1.8 Transfer the slurry in step 7.3.1.4 to the planchet in 2-3 mL aliquots and evaporate to near dryness between additions.

Note: If the samples evaporate completely between additions, allow the planchets to cool slightly before adding more sample. This will minimize splattering and losses of the sample.

7.3.1.9 Rinse the tube containing the slurry with 2-3 mL of water and transfer to the planchet.

7.3.1.10 After all the solution has evaporated to dryness, cool each planchet.

7.3.1.11 Reweigh each planchet, and record the weight to 0.0001 gram.

7.3.1.12 Cover planchet with aluminum foil and wait at least three days for bismuth-210 ingrowth.

7.3.1.13 Count on a gas flow proportional counter.

7.3.2 *Filter option*

7.3.2.1 Set up a 0.1 micron 25 mm filter, glassy side down on a Gelman filter apparatus with stainless steel screen, 50 mL polysulfide funnel and 100 mL polypropylene Erlenmeyer flask.

7.3.2.2 Add 3-5 mL of 80% ethanol to each filter, applying vacuum and ensuring there are no leaks along the sides. Add 2-3 mL of water to each filter.

7.3.2.3 Filter the sample from step 7.2.7 and rinse 50 mL centrifuge tube with 5 mL water, transferring this rinse to the filter apparatus.

7.3.2.4 Wash each filter with 3-5 mL of ethanol.

7.3.2.5 Remove filters, place in plastic Petri dishes, and dry under heating (IR) lamps for a few minutes.

7.3.2.6 Mount filters in the center of the planchets, using double-sided tape or glue stick.

7.3.2.7 Cover planchet with aluminum foil and wait for atleast 3 days for Bi-210 ingrowth.

7.3.2.8 Count on a gas-flow proportional counter.

8. Calculations

Gravimetric: Pb carrier

$$\text{Y (carrier yield)} = \frac{Rw - Tw - Bw}{Cw}$$

where:

R_w = residue + planchet or filter, mg
T_w = tare weight of planchet or filter, mg
B_w = blank weight, mg
C_w = $PbSO_4$ added, mg

Calculate Pb-210 activity based on Bi-210 ingrowth

$$^{210}Pb\ (\text{pCi / L}) = \frac{S - B}{2.22 \times E \times Y \times (1 - e^{-\lambda(t_0 - t_1)}) \times V}$$

where:

S = sample counts per minute
B = background counts per minute
E = efficiency of counter
λ = 0.138 day^{-1} (decay constant for ^{210}Bi)
t_0 = time of Bi-210 separation, recorded in step 7.2.3
t_1 = time of midpoint of sample count
V = volume of sample in liters
Y = Pb carrier yield