

Pb-01-RC

**LEAD-210 IN BONE, FOOD, URINE,
FECES, BLOOD, AIR, AND WATER**

Contact Person(s) : Isabel M. Fisenne

APPLICATION

This procedure is applicable to samples of bone, food, urine, feces, blood, air, and water and is based on the solvent extraction of a lead bromide complex into Aliquat-336 (Petrow and Cover, 1965; Morse and Welford, 1971).

Lead-210 is isolated from most interferences. Its progeny ^{210}Bi is separated from ^{210}Pb , and the β activity is measured radiometrically after ingrowth.

SPECIAL APPARATUS

1. Atomic absorption (AA) spectrometer.
2. Aluminum foil - 7.2 mg cm^{-2} .
3. Rings and discs - see Specification 7.2.
4. Mylar film - see Specification 7.3.
5. Teflon filter holder.
6. Combination magnetic stirrer and hot plate.
7. Plastic scintillation phosphors - see Specification 7.9.

SPECIAL REAGENTS

1. Aliquat-336, methyltricapryl-ammonium chloride (Henkel Corporation, 2430 N. Huachuca Dr., Tucson, AZ 85745-1273), 3:7 in toluene and washed twice with an equal volume of 1.5M hydrobromic acid.
2. Hydrobromic acid 48%.
3. Hydrobromic acid 3.0M - 340 mL of 48% HBr L⁻¹ of water.
4. Hydrobromic acid 1.5M - 170 mL of 48% HBr L⁻¹ of water.
5. Hydrobromic acid 0.1M - 10 mL of 48% HBr L⁻¹ of water.
6. Toluene.
7. Standard Pb solution - 1000 µg mL⁻¹.
8. Lead carrier: 20 mg Pb mL⁻¹ - 32 g Pb(NO₃)₂ L⁻¹ in 1:19 HNO₃.
9. Bismuth carrier: 10 mg Bi mL⁻¹ - 23.2 g Bi(NO₃)₃·5 H₂O L⁻¹ in 1:19 HNO₃.

SAMPLE PREPARATION

A. Water.

1. To daily collections of 20 L of tap water add 100 mL of HNO₃ and evaporate to about 100 mL (see **Note 1**).
2. Add 100 mL of HNO₃ and transfer to a 400-mL beaker. Complete the destruction of organic material and evaporate to near dryness.
3. Add 50 mL of 10% HNO₃ to the beaker and warm to affect dissolution of any residue.

4. Cool the solution to room temperature and transfer the sample solution to a 100-mL volumetric flask. Bring the solution to volume with 10% HNO₃.
5. Pipette 1 mL of sample solution into a 10-mL volumetric flask. Bring the solution to volume with 10% HNO₃. Reserve this solution to determine the stable Pb content of the sample by AA spectrometry (see **Note 2**).
6. Return the sample solution in the 100-mL volumetric flask (Step 4) to the 400-mL beaker. Add 1 mL of Pb carrier and evaporate the solution to dryness.
7. Add 100 mL of 3M HBr to the sample beaker and warm the solution. Cool the solution to room temperature and proceed with the **Determination**.

B. Urine, blood, feces and air filters.

1. Measure 2-L of urine and transfer to a 3-L beaker. Place a measured volume of blood or 24 h fecal sample in a 1-L beaker. Place the air filter in a 600-mL beaker (see **Note 1**).
2. Destroy most of the organic material by carefully heating with HNO₃. Hydrogen peroxide can be used to complete the oxidation of organic material (see **Note 3**).
3. Add 50-mL of 10% HNO₃ to the beaker and warm to affect dissolution of any residue.
4. Cool the solution to room temperature and transfer the sample solution to a 100-mL volumetric flask. Bring the solution to volume with 10% HNO₃.
5. Pipette 1 mL of sample solution into a 10-mL volumetric flask. Bring the solution to volume with 10% HNO₃. Reserve this solution to determine the stable Pb content of the sample by AA spectrometry (see **Note 2**).
6. Transfer the sample solution in the 100-mL volumetric flask (Step 4) to the 400-mL beaker. Add 1 mL of Pb carrier and evaporate the solution to dryness.
7. Add 100 mL of 3M HBr to the sample beaker and warm the solution. Cool the solution to room temperature and proceed with the **Determination**.

C. Bone (see Note 4)

1. Weigh 20 g of bone ash and transfer to a 400-mL beaker.
2. Dissolve the ash in about 80 mL of 3M HBr and warm to complete the dissolution.
3. Cool the solution to room temperature and transfer the sample solution to a 100-mL volumetric flask. Bring the solution to volume with 3M HBr.
4. Pipette 1 mL of sample solution into a 10-mL volumetric flask. Bring the solution to volume with 10% HNO₃. Reserve this solution to determine the stable Pb content of the sample by AA spectrometry (see Note 2).
5. Transfer the sample solution in the 100-mL volumetric flask (Step 3) to the 400-mL beaker and add 1 mL of Pb carrier.
6. Proceed with the **Determination**.

D. Food.

1. Depending upon food type, freeze drying should be used to remove excess water prior to wet ashing the sample (see Note 1).
2. Destroy most of the organic material by carefully heating with HNO₃. Hydrogen peroxide can be used to complete the oxidation of organic material (see Note 3).
3. Add 50 mL of 10% HNO₃ to the beaker and warm to affect dissolution of any residue.
4. Cool the solution to room temperature and transfer the sample solution to a 100-mL volumetric flask. Bring the solution to volume with 10% HNO₃.
5. Pipette 1 mL of sample solution into a 10-mL volumetric flask. Bring the solution to volume with 10% HNO₃. Reserve this solution to determine the stable Pb content of the sample by AA spectrometry (see Note 2).

6. Transfer the sample solution in the 100-mL volumetric flask (Step 3) to the 400-mL beaker. Add 1 mL of Pb carrier and evaporate the solution to dryness.
7. Add 100-mL of 3M HBr to the same beaker and warm the solution. Cool the solution to room temperature and proceed with the **Determination**.

Notes:

1. It is necessary to analyze reagent blanks with each batch of samples to correct the ^{210}Pb results.
2. The stable Pb content of some samples may be high enough to contribute a significant fraction to the total stable Pb measured by AA. This would result in an inflated estimate of the Pb carrier yield.
3. Hydrogen peroxide contains measurable and variable amounts of stable Pb and should be used sparingly.
4. It has been shown at this Laboratory that no ^{210}Pb loss occurs from bone dry ashed below 700°C (Fisenne, 1994). The absence of ^{210}Pb loss was determined for three bone types — ribs, vertebrae, and femur. It is the practice at EML to dry ash bones for ^{210}Pb analyses at 550°C .

DETERMINATION

1. Transfer the 3M HBr solution to a 250-mL separatory funnel containing 75 mL of Aliquat-336.
2. Shake for 30 sec. Let the phases separate and discard the aqueous (lower) phase.
3. Wash the organic phase three times with 50-mL portions of 0.1M HBr and discard all washes (lower phases).
4. Wash the organic phase twice with an equal volume of water. Transfer the washed organic phase to a suitable disposal container.

5. Combine the strip solutions in a 400-mL beaker and add 100 mL of HNO_3 .
6. Wait for any reaction to subside and heat gently until the organic residue is destroyed. Evaporate the solution to ~ 10 mL.

A. First milking.

1. Transfer the sample to a 40-mL centrifuge tube with water. Add 1 mL of Bi carrier.
2. Adjust the pH of the sample to 8 with NH_4OH .
3. Stir the sample and heat in a hot water bath.
4. Cool and centrifuge the tube for 10 min. Decant and discard the supernate.
5. Dissolve the precipitate with five drops of HCl.
6. Add 40 mL of water and heat with constant stirring.
7. Cool and centrifuge for 10 min. Decant and reserve the supernate in a 250-mL beaker.
8. Repeat Steps 5-7 twice more, combining the supernates. Discard the precipitate. Record the time and date for ingrowth of ^{210}Bi .
9. Add 1 mL of Bi carrier and 3-5 mL of HCl to the combined supernates. Reduce the volume to <100 mL.
10. Cool, transfer to a 100-mL volumetric flask and bring to volume with 0.5N HCl.
11. Pipette 1 mL of sample into a 10-mL volumetric flask. Bring to volume with 0.5N HCl.
12. Measure the quantity of Pb in both the sample and the separated Pb fraction in the 10-mL volumetric flasks on an AA spectrometer at 283 μm . (The calibration curve should have a working range of 0-50 $\mu\text{g mL}^{-1}$.)
13. Subtract the Pb content of the dissolved sample and the reagent blank from the total Pb content determined in Step 12 to obtain the Pb carrier yield.

14. Allow 2-3 weeks for ingrowth of ^{210}Bi into the main portion of the sample (Step 10).

B. Second milking.

1. Transfer the solution from the 100-mL volumetric flask to a 250-mL beaker and evaporate to about 15 mL.
2. Transfer the sample to a 40-mL centrifuge tube and adjust the pH to 8 with NH_4OH . Centrifuge the tube for 10 min. Decant and discard the supernate.
3. Dissolve the precipitate with five drops of HCl and bring volume of sample to 30 mL with H_2O . (Record the time and date for decay of ^{210}Bi .)
4. Heat with constant stirring in a hot water bath. Cool and centrifuge the tube for 10 min. Reserve the supernate for additional ^{210}Pb analysis in a 150-mL beaker.
5. Dissolve the precipitate with five drops of HCl and dilute to 30 mL with water.
6. Heat in a hot water bath with constant stirring. Cool and centrifuge the tube for 10 min. Combine the supernate with that from Step 4.
7. Dissolve the precipitate with five drops of HCl. Stir and dilute to 30 mL with water.
8. Heat the tube in a hot water bath with constant stirring. Cool, filter with suction on a preweighed 2.4 cm Whatman No. 42 filter paper using a Teflon filter holder.
9. Wash the tube and the precipitate with water and alcohol. Dry the paper and precipitate for 30 min at 110°C in a drying oven.
10. Cool and reweigh the filter to determine weight of BiOCl precipitate.
11. Mount the filtered sample on a nylon ring and disc, covering the sample with aluminum foil (7.2 mg cm^{-2}), a plastic scintillation phosphor and Mylar film.
12. Measure the ^{210}Bi on a low-level β -scintillation counter. (Record the time and date for decay of ^{210}Bi .)

- Standardize the counter with a known amount of ^{210}Pb from which ^{210}Bi has been separated and prepared in the same way as the sample.

DATA PROCESSING AND ANALYSES

The ^{210}Pb activity of the sample is calculated using the following formula:

$$\text{Bq of } ^{210}\text{Pb} = \frac{R_s Y_1 Y_2 E}{GD}$$

where

R_s = net counting rate of the sample,

Y_1 = yield factor for Pb carrier,

Y_2 = yield factor for Bi carrier,

E = counter efficiency factor,

G = growth factor (growth of ^{210}Bi from first milking to final milking), and

D = decay factor (decay of ^{210}Bi from final milking to time of counting).

LOWER LIMIT OF DETECTION (LLD)

		A	B
Counter efficiency	(%)	35	-
Counter background	(cps)	0.005	-
Yield	(%)	80	80
LLD (400 min)	(Bq)	0.01	0.007
LLD (1000 min)	(Bq)	0.005	0.005

A = ^{210}Pb separation, ^{210}Bi ingrowth, ^{210}Bi separation.

B = ^{210}Bi separation only.

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