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Determination of Lead-210 in Drinking Water

Method 909.0

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Determination of Lead-210 in Drinking Water

Method 909.0

1. Scope and Application

   1.1. Lead-210 is not regulated by the National Interim Primary Drinking Water Regulations (NIPDWR). However, based upon its maximum permissible concentration (MPC) published in NBS Handbook 69, the maximum concentration level (MCL) calculated by applying the formula in the NIPDWR would be 1 pCi/L or less, depending upon the choice of critical organ.

   1.2 The sensitivity of the method as defined in the NIPDWR is approximately 0.7 pCi/L for a one liter sample size using liquid scintillation counting and 0.2 pCi/L using a low background beta counter.

2. Summary of Method

   2.1 Lead carrier is added and concentrated by precipitation as the chromate. It is further purified from its bismuth-210 daughter by selected dissolution of lead sulfide from a 1.5N hydrochloric acid solution. Lead is finally converted to the carbonate and the lead-210 concentration calculated by either counting the lead-210 beta emission by liquid scintillation technique or counting the ingrown bismuth-210 daughter activity by low background end window counting.

3. Sample Handling and Preservation

   3.1 If the sample cannot be analyzed within 24 hours, it is recommended

that the sample be preserved using nitric acid to a concentration of 0.01N (pH 2).

4. Interferences

4.1 Lead-214 will not interfere as the time delay from lead separation and counting (10 half lives) allows for its total decay.

4.2 Lead-212 can interfere with the lead-210 determination and cause a positively biased result. However, a 2 to 3 day storage at the end of Step 8.14 will allow for sufficient decay.

5. Apparatus

5.1 Liquid scintillation counter or low background beta counter

5.2 Millipore 300 mL ground glass filtering assembly

5.3 Membrane filter (PVC), e.g., Gelman 64515

5.4 Centrifuge

5.5 40 mL cone bottom centrifuge tubes

5.6 2.8 cm fiber glass filters

5.7 Convection oven.

6. Reagents

6.1 Acetic acid, glacial

6.2 Ammonium carbonate, 1.5M. Dissolve 144 g ammonium carbonate in 300 mL of water and dilute to 500 mL.

6.3 Ammonium hydroxide, 6M. Transfer 400 mL of concentrated ammonium hydroxide (30%) to 500 mL water and dilute to 1000 mL with water.

6.4 Barium carrier, 5 mg Ba^{++}/mL. Dissolve 4.4713 g of $BaCl_2 \cdot 2H_2O$ in water and dilute to 500 mL.

6.5 Bismuth carrier, 5 mg Bi^{+++}/mL. Dissolve 5.8026 g of $B_i(NO_3)_3 \cdot 5H_2O$ in 1 M HNO_3 and dilute to 500 mL with 1 M HNO_3.

6.6 Hexanoic acid, practical.

6.7 Hydrochloric acid, 12 M.

6 M - Transfer 500 mL of concentrated acid to 400 mL of water and dilute to 1000 mL with water.

1.5 M - Transfer 125 mL of concentrated acid to 700 mL of water and dilute to 1000 mL with water.

6.8 Hydrogen sulfide gas, lecture bottle.

6.9 Lead carrier, 10 mg Pb^{++}/mL. Dissolve 4 grams $Pb(NO_3)_2$ in 250 ml of 0.1 M HNO_3.

6.10 Scintillation solution. Commercially prepared universal liquid scintillation cocktail for aqueous and non-aqueous samples.

6.11 Sodium chromate, 1.5M. Dissolve 175 g of sodium chromate tetrahydrate in 350 mL water and dilute to 500 mL with water.

6.12 Sodium nitrite, 1 M. Dissolve 6.9 g of sodium nitrite in 70 mL water and dilute to 100 mL with water.

6.13 Toluene, reagent grade.

6.14 Water/ethanol wash solution, 1:1. Mix 200 mL of ethanol with 200 mL of water.

7. Calibration and standardization

7.1 Lead carrier solution

7.1.1 Transfer 10 mL of the lead carrier solution to a 150 mL beaker and dilute to 75 mL.

7.1.2 Add 1-2 drops of methyl orange indicator and neutralize by the dropwise addition of 6M NH_4OH.

7.1.3 Reacidify with 2 mL of glacial acetic acid and heat to near boiling.

7.1.4 Slowly bubble H_2S gas into the solution for 3-4 minutes.

7.1.5 Remove H_2S source and heat the solution to just boiling. Cool.

7.1.6 Filter through a tared fritted glass filtering funnel of fine porosity.

7.1.7 Wash several times with 10 mL portion of water.

7.1.8 Dry at 105-110°C. Cool and weigh.

7.2 Counter Efficiency

7.2.1 Transfer 1 mL each of the lead and bismuth carrier to a 40 mL cone bottom centrifuge tube.

7.2.2 Add an aliquot of the lead-210 standard tracer solution approximating 1000 dpm.

7.2.3 Dilute to 20 mL and add 1-2 drops of methyl orange.

7.2.4 Neutralize by the dropwise addition of 6M NH_4OH.

7.2.5 Reacidify with 2 mL of glacial acetic acid.

7.2.6 Heat to near boiling in a hot water bath and slowly bubble H_2S gas into the solution for 2-3 minutes.

7.2.7 Remove H_2S source and continue boiling for 2-3 minutes. Remove from bath and cool.

7.2.8 Centrifuge and discard supernate.

7.2.9 Add 20 mL 1.5M HCl and heat to boiling in a water bath with intermittent stirring, breaking up all large sulfide lumps.

7.2.10 Cool and filter through a 2.8 cm glass fiber filter, saving the filtrate and noting the time of filtration.

7.2.11 Neutralize filtrate by adding 5-6 mL of 6 M NH_4OH using pH paper to verify.

7.2.12 Reacidify by adding 2 mL of glacial acetic acid.

7.2.13 Heat to near boiling in a water bath and slowly bubble H_2S gas into the solution for 2-3 minutes.

7.2.14 Remove H_2S source and continue heating for 2-3 minutes. Cool.

7.2.15 Centrifuge and discard the supernate.

7.2.16 Add 3 mL 6M HCl and heat in a water bath to dissolve the sulfides.

7.2.17 Add 0.5 mL of 1M $NaNO_2$ to oxidize excess sulfide ions. Heat until effervescence ceases and dilute to 20 mL with water.

7.2.18 Filter through a 2.8 cm glass fiber filter, saving the filtrate.

7.2.19 Dropwise add 6M NH_4OH until a pearlescent precipitate persists. Then add 5 mL 1.5M ammonium carbonate solution.

7.2.20 Heat in a hot water bath with stirring until the excess ammonium carbonate begins to decompose (60^oC).

7.2.21 Cool and centrifuge, discarding the supernate.

7.2.22 Add 20 mL 1:1 water/ethanol wash solution breaking up the precipitate with a glass rod.

7.2.23 Filter through a tared 2.8 cm glass fiber filter, washing the tube and precipitate several times with 10 mL volume of the wash solution.

7.2.24 Dry filter at 105-110^oC. Cool and weigh.

7.3 Liquid Scintillation Counting

7.3.1 Place the weighed filter at the bottom of a glass scintillation vial with the precipitate facing upwards.

7.3.2 Add 0.5 mL each of glacial acetic acid and water. Evaporate to dryness in an oven at 120°C.

7.3.3 Cool and add 0.25 mL hexanoic acid wetting the filter completely. Add 3 mL of toluene and swirl occasionally over a period of 30 minutes to solubilize the lead hexanoate.

7.3.4 Add 10 mL of the scintillation solution, mix thoroughly and place in a liquid scintillation counter.

7.3.5 After 30 minutes, determine the beta spectrum of the lead-210 emissions.

7.3.6 Set the beta window to include about 95% of the beta emissions.

7.3.7 Count the standard over a period of two weeks at this window setting, noting the time of each count.

7.4 Low Background Beta Counter

7.4.1 Transfer the filter from step 7.2.24 to a planchet conforming to your standard counting geometry. (It would be desirable to cover the filter to prevent loss of precipitate).

7.4.2 Count the standard over a period of two weeks noting the time of each count.

8. Procedure

8.1 Acidify a 1-liter volume of a tap water sample with 25 mLs of glacial acetic acid.

8.2 Add 10 mgs of lead carrier and 5 mgs of the holdback carriers Bi and Ba. (Five mgs of these additional holdback carriers, Fe, Co, Ni, Ce, Mn, Sr, Zn, and Cu may be added when needed.)

8.3 With constant stirring, add 20 mLs of 0.5M sodium chromate.

8.4 Heat to 70° C on a hot plate with stirring until the precipitate is fully developed.

8.5 Remove from hot plate and cool in a cold water bath.

8.6 Filter with vacuum through a 47 mm 0.45 micron membrane filter.

8.7 Wash precipitate thoroughly with small quantities of distilled water.

8.8 Transfer the filter to a 40 mL cone bottom centrifuge tube and dropwise add 1 mL of conc. HCl contacting the precipitate and heat in a boiling water bath to reduce the chromate and dissolve the precipitate. Dilute to 20 mL with water.

8.9 Remove filter and wash with 10 mL water, adding the wash to the centrifuge tube.

8.10 Add sufficient 6M ammonium hydroxide to neutralize the acid.

8.11 Add 2 mL glacial acetic acid and place centrifuge tube in a boiling water bath for 2-3 minutes.

8.12 Carefully bubble a slight stream of hydrogen sulfide gas into the solution for 2-3 minutes to completely precipitate the lead.

8.13 Remove the hydrogen sulfide source and continue boiling for 5 minutes.

8.14 Remove from the water bath, cool, and centrifuge, discarding the supernate.

8.15 Add 20 mL 1.5N HCl to selectively dissolve PbS, heating in a boiling water bath. (Precipitate is nearly completely solubilized).

8.16 Filter through a 2.8 cm glass fiber filter to remove the Bi_2S_3 precipitate, collecting the filtrate in a clean 40 mL centrifuge tube. (Note time as initial Pb-210 separation.)

8.17 Neutralize by the addition of 5-6 mL 6M NH_4OH. Add 2 mL glacial

acetic acid and reprecipitate the PbS using H_2S gas, heating in a boiling water bath.

8.18 Cool, centrifuge and discard supernate.

8.19 Add 3 mL 6M HCl to dissolve the sulfides and heat in a boiling water bath. Add 0.5 mL 1M sodium nitrite and heat in a hot water bath until effervescence ceases. Remove from water bath and dilute to 20 mL with water.

8.20 Filter through a fiber glass filter to remove any precipitated sulfur or other insolubles into a clean 40 mL cone bottom centrifuge tube. Wash with 10 mL water.

8.21 Add sufficient 6M ammonium hydroxide to neutralize the acid.

8.22 Add 5 mL of 1.5M ammonium carbonate.

8.23 Heat in a boiling water bath for 3 minutes, remove and cool.

8.24 Centrifuge and discard the supernate.

8.25 Wash precipitate with 15 mL of 1:1 water:ethanol solution.

8.26 Filter through a tared 2.8 cm fiber glass filter and rinse with 10 mL 1:1 water/ethanol solution.

8.27 Dry at 105°C, cool and weigh to determine lead carrier recovery. (If liquid scintillation counting is to be used, continue at step 8.28. If Low Background Beta counting is to be used, continue at step 8.33).

8.28 Place filter at the bottom of scintillation vial with the precipitate facing upwards.

8.29 Add 0.5 mL glacial acetic acid and 0.5 mL water and take to dryness in a 120°C oven.

8.30 Cool and add 0.25 mL of hexanoic acid and 3 mL toluene. Mix and let stand for 20 minutes with occasional mixing.

8.31 Add 10 mL of scintillation solution. Mix throughly and place sample into the liquid scintillation counter.

8.32 Using the predetermined window setting for counting only the lead-210 beta emissions, count for sufficient time to meet the method detection limit.

8.33 Place the filter on a planchet conforming to your standard geometry. (It would be desirable to cover the filter to prevent loss of precipitate during storage.)

8.34 Store for about 2 weeks to allow sufficient Bi-210 ingrowth.

8.35 Place in the counter and count for sufficient time to meet the method detection limit and note time of count.

9. Calculation

9.1 Lead standardization

$$\text{Lead, mg/mL} = \frac{\text{mg PbS} \times 0.86599}{10}$$

9.2 Liquid scintillation counter

9.2.1 Bismuth-210 crosstalk (Z)

9.2.1.1 Determine the bismuth ingrowth factors, $(1-e^{-\lambda t})$ where t equals the time difference from time of separation (step 7.2.10) to time of counting for the various count times.

9.2.1.2 Plot the observed count rates as the ordinate against the ingrowth factors.

9.2.1.3 By linear least squares analysis, solve for the intercept, A, and slope, B. (The intercept is the count rate due to the lead-210 emission and the slope is the count rate due to the amount of the

for the various count times where t is the time difference between time of separation and time of count.

9.3.1.2 Plot the observed count rates as the ordinate against the ingrowth factors.

9.3.1.3 By linear least square analysis solve for the intercept A and slope B. (The intercept A represents the count rate due to lead-210 and the slope B represents the count rate of bismuth-210 at equilibrium.)

9.3.1.4 Efficiency determination

Lead-210 efficiency, E_1 = A/dpm recovered

Bismuth-210 efficiency, E_2 = B/dpm recovered

Total efficiency = $E_1 + E_2 (1 - e^{-\lambda t})$

9.3.2 Concentration

$$\text{Lead-210 concentration pCi/L} = \frac{G - B}{V \times (E_1 + E_2(1 - e^{-\lambda t})) \times R \times 2.22}$$

where:

G = gross count rate in lead-210 window

B = background count rate

V = volume of sample, liter

E_1 = Lead-210 efficiency

E_2 = Bismuth-210 efficiency

$(1 - e^{-\lambda t})$ = Bismuth-210 ingrowth factor

R = chemical recovery

2.22 = constant (dpm/pCi)

10. Precision and Accuracy

10.1 Liquid scintillation counting

10.1.1 Accuracy

10.1.1.1 Four samples at lead-210 concentrations ranging from 0 to 41 pCi/L were analyzed. A plot and linear least square solution of pCi/L found versus pCi/L added showed that the intercept was not different from zero and that the slope showed a +1% bias.

10.1.1.2 Seven samples were also analyzed at a single concentration level (7.72 pCi/L). The average of the seven determinations was 7.96 pCi/L. This showed a +3% bias.

10.1.2 Precision

10.1.2.1 Based upon the seven replicate values at 7.72 pCi/L, the relative standard deviation was found to be ± 8%.

10.2 Low background beta counting

10.2.1 Accuracy

10.2.1.1 Eight samples were analyzed at a single concentration level of 7.72 pCi/l. The average concentration found was 7.85 pCi/l. This shows as +2% bias.

10.2. Precision

10.2.2.1 Based upon the eight replicate values at 7.72 pCi/l, the relative standard deviation was calculated to be ± 5%.