Direct lead determination in aqueous samples – Version 1.0 – 14/09/15 - TKI

Summary

1 Scope

This procedure describes a method for the direct separation and measurement of Pb-210 in aqueous samples (drinking water, mineral water and ground water samples with limited mineral content).

2 Summary of Method

Pb-210 is concentrated and separated using TK101 Resin prior to gas proportional counting or liquid scintillation counting. TK101 resin is used to concentrate lead from 1 – 5 L aqueous samples and to subsequently separate it from matrix elements and other beta emitters. Stable lead is used to monitor method yields and correct results to improve precision and accuracy.

3 Significance of Use

This method is a rapid and reliable method for measurement of lead in aqueous samples.

4 Interferences

4.1 The presence of elemental strontium in the sample may bias the gravimetric yield determination. If it is suspected that natural strontium is present in the sample, its concentration should be determined by a suitable means and the yield calculation appropriately modified.

4.2 Lead must be separated from interfering isotopes of other elements to enable measurement by beta counting.

4.3 A rinse with 2M HCl is used to effectively remove barium-140, strontium/yttrium-90 and potassium-40 isotopes as well as other matrix interference's.

5 Apparatus

5.1 Analytical balance- 0.0001 g sensitivity

5.2 Vacuum box

5.3 Cartridge reservoirs - 20 mL or similar

5.4 Beta detector -gas proportional counter, liquid scintillation or Cerenkov counter

5.5 Counting dishes - 50.8 mm diameter, 6.4 mm deep flat bottom, cupped planchet. – GPC option only

5.6 Liquid scintillation vials

5.7 Fume hood

5.8 Filter- 0.45 micron

5.9 Fume hood

5.10 Hotplate

5.11 Plastic Petri dishes, 5-1/2 x 1 cm

6 Reagents

6.1 Unless otherwise indicated, all references to water should be understood to mean deionized distilled water.

6.2 Acetone

6.3 Nitric acid (15.7 M)- concentrated HNO_3

6.4 Ethyl alcohol -USP, 100%

6.5 Liquid scintillation cocktail (LSC counting only)

6.6 Nitric acid (15.7M) - concentrated nitric acid

6.7 Hydrochloric acid (12M) - concentrated hydrochloric acid

6.8 Hydrochloric acid (2M) -Add 167 mL of concentrated HCl to 800 mL of water and dilute to 1 liter with water.

6.9 Hydrochloric acid (6M) -Add 500 mL of concentrated HCl to 800 mL of water and dilute to 1 liter with water.

6.10 Nitric acid solution (8M) - Add 510 mL of concentrated nitric acid to 400 mL of water and dilute to 1 liter with water.

6.11 Nitric acid solution (0.01M) - Add 0.64 mL of concentrated nitric acid (sp gr 1.42) to 950 mL of water and dilute to 1 liter with water.

6.12 Nitric acid solution (0.05M) - Add 3.2 mL of concentrated nitric acid (sp gr 1.42) to 900 mL of water and dilute to 1 liter with water.

6.13 Sulfuric acid (18M) – concentrated sulfuric acid

6.14 TK101 Resin -prepacked 2mL column (0.7 grams resin), or smaller particle size (50-100 µm) in appropriate size column. Pre-packed cartridges may also be used. Refer to VBS01, Setup and Operation Instructions for Eichrom's Vacuum Box System (VBS)

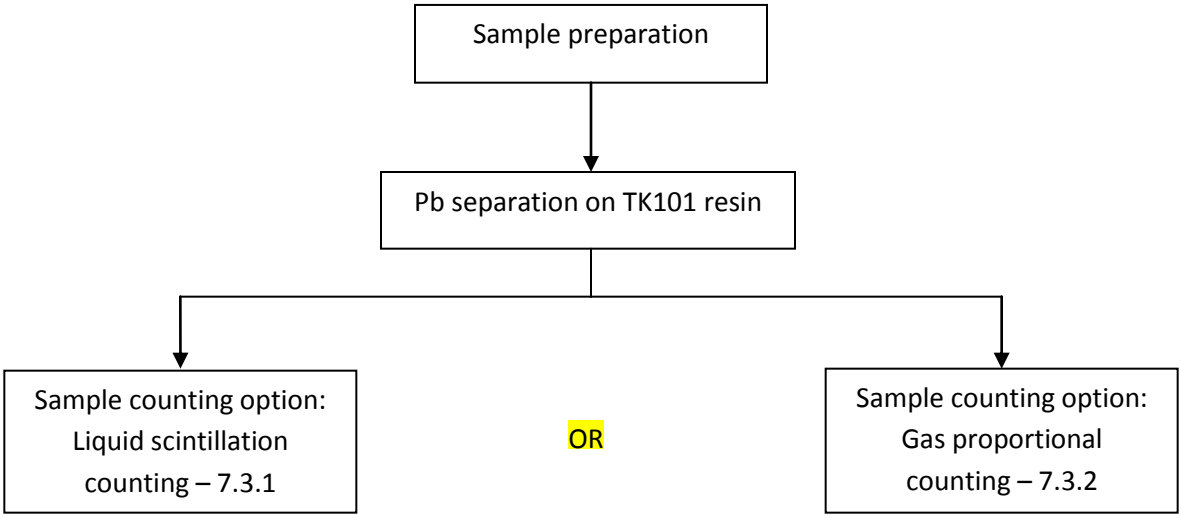
6.15 Pb-210 standard

6.16 Lead (Pb) carrier (10 mg/mL), gravimetric - Dissolve 1.6 grams of $Pb(NO_3)_2$ in water and dilute to 100mL with water

Note: In case of spectrometric yield determination concentration of the lead carrier solution can be adjusted to lower quantities of Pb.

7 Procedure

7.1 Synopsis



7.2 Lead Separation using TK101 Resin:

7.2.1 If samples larger than 5L are analyzed, evaporate the sample to approximately 5L.

Note: Smaller volumes may be analyzed

7.2.2 Measure the sample volume using a standard graduated cylinder (or equivalent) and transfer volume to an appropriate size plastic bottle or volumetric flask.

7.2.3 If necessary filter the sample

7.2.4 Acidify the sample to pH 2 using concentrated nitric acid.

Note: Alternatively the sample can be loaded without acidification (pH ≤ 8)

7.2.5 Add 1 ml of 10 mg/ml lead carrier (for gravimetric yield determination option, for spectrometric yield determination lower amounts may be added) into each sample aliquot.

7.2.6 Place columns or cartridges on vacuum box. Attach suitable reservoirs.

7.2.7 Ensure that a suitable container is below each column/cartridge.

7.2.8 Add 5 ml of 0.01 M HNO_3 to each column/cartridge to condition columns.

7.2.9 Adjust flow rate to 10 - 20 mL/min.

7.2.10 Load each sample onto the appropriate column and allow to drain.

7.2.11 Add 15 ml of 8 M HNO_3 to each column to rinse.

7.2.12 Add 20 ml of 2 M HCl to each column to rinse.

7.2.13 Discard the feed and rinse solutions collected.

7.2.14 Place fresh LSC vial below each column/cartridge.

7.2.15 Add 20 ml of 6 M HCl to each column to elute lead.

7.2.16 Cap and shake Pb eluate.

7.3 Sample preparation for counting

7.3.1 Liquid scintillation counting option:

7.3.1.1 Case of yield determination via spectrometry (ICP-MS, AAS,…):

7.3.1.1.1 Homogenize eluate and withdraw appropriate sample aliquot for yield determination by spectrometry.

7.3.1.1.2 Transfer eluate into beaker

7.3.1.1.3 Evaporate eluate to dryness

7.3.1.1.4 Add 5 mL of conc. nitric acid and evaporate to dryness

7.3.1.1.5 Repeat until white residue is obtained

7.3.1.1.6 Redissolve residue in 10 mL 0.01 HNO_3

7.3.1.1.7 Add 10 mL LSC cocktail, cap and shake

7.3.1.2 Case of yield determination via gravimetry (LSC option):

7.3.1.2.1 For each sample analyzed, clean a counting dish by moistening a paper towel with ethanol, wiping the dish and letting it dry.

7.3.1.2.2 Weigh the counting dish(s) on an analytical balance and record the weight.

7.3.1.2.3 Place each counting dish on a hot plate under a heat lamp in a hood.

7.3.1.2.4 Evaporate the eluate from 7.2.164 onto each dish in successive 3 mL volumes.

7.3.1.2.5 Allow each 3 mL volume to evaporate to near dryness between additions.

7.3.1.2.6 Rinse the vial containing the column strip solution with 2 mL of 0.05M HNO_3 and transfer to the counting dish.

7.3.1.2.7 After all the solution has evaporated to dryness, cool each dish.

7.3.1.2.8 Reweigh each counting dish, and record the weight.

7.3.1.2.9 Redissolve residue in 4 mL 0.05M HNO_3, transfer solution into LSC vial

7.3.1.2.10 Rinse dish two times with 3 mL 0.05M HNO_3, transfer solution into LSC vial

7.3.1.2.11 Add 10 mL LSC cocktail, cap and shake

7.3.2 Gas Proportional Counting Option:

Note: Gas proportional counting provides lower detection limits than liquid scintillation counting or Cerenkov counting.

7.3.2.1 For each sample analyzed, clean a counting dish by moistening a paper towel with ethanol, wiping the dish and letting it dry.

7.3.2.2 Weigh the counting dish(s) on an analytical balance and record the weight.

7.3.2.3 Place each counting dish on a hot plate under a heat lamp in a hood.

7.3.2.4 Evaporate the eluate from 7.2.164 onto each dish in successive 3 mL volumes.

7.3.2.5 Allow each 3 mL volume to evaporate to near dryness between additions.

7.3.2.6 Rinse the vial containing the column strip solution with 2 mL of 0.05M HNO_3 and transfer to the counting dish.

7.3.2.7 After all the solution has evaporated to dryness, cool each dish.

7.3.2.8 Reweigh each counting dish, and record the weight.

7.3.2.9 Cover planchet with aluminum foil

7.3.2.10 Store samples for at least 5 days prior to measurement

7.3.2.11 Count samples sufficient time to achieve the desired counting statistics and minimum detectable concentration.

Note: Alternatively the Pb can be precipitated as $PbSO_4$ and filtered using a weighed filter. The filter is then dried and weighed, the weight is recorded.

8 Preparation of Pure Pb-210 and Pure Bi-210 for Counter Calibration Sources:

8.1 Add an appropriate volume of calibrated Pb-210 standard solution (in equilibrium with Bi-210) to a small beaker, add 1 mL of Pb carrier and evaporate the solution to dryness.

8.2 Redissolve the residue in 5 mL of 8M HNO_3

8.3 Place a beaker below each column.

8.4 Pipette 5 mL of 8M HNO_3 into each TK101 resin column to condition resin and allow to drain.

8.5 Ensure that a clean beaker or vial is below the column (Bi-210 fraction).

8.6 Transfer the redissolved residue into the appropriate TK101 Resin column by pouring or by using a plastic transfer pipette and allow to drain.

8.7 Add 5 mL of 8M HNO_3 to rinse to the beaker and transfer each solution into the TK101 Resin column and allow to drain.

8.8 Repeat step 8.7.

8.9 Add 5 mL of 8M HNO_3 to the TK101 Resin column and allow to drain.

8.10 Replace container.

8.11 Ensure that a clean beaker or vial is below the column (Pb-210 fraction).

8.12 Elute Pb with 20 mL 6M HCl

8.13 Prepare the standard solutions as appropriate for use as a calibration standard (evaporation on planchet, etc.).

9 REFERENCES

(1) Banavali, A.D., et al., "Strontium-89/90 Analysis by Eichrom Column Chemistry and Cerenkov Counting," 38th Annual Conference on Bioassay, Analytical and Environmental Radiochemistry. Santa Fe, NM, November, 1992.

(2) Nelson, D.M, "Purification of Strontium in Water before Strontium-89/Strontium-90 Measurement," DOE Methods Compendium, RP500.

(3) Jake Surman, Jackie Pates, Hao Zhang and Steffen Happel: "Development of a new resin for the rapid determination of strontium-90 in environmental waters", oral presentation at the INTERNATIONAL SYMPOSIUM ON ENVIRONMENTAL RADIOACTIVITY, PLYMOUTH (UK), 4-5 September, 2012

(4) J.J.Surman, J.M.Pates, H.Zhang, S.Happel: "Development and characterization of a new Sr selective resin for the rapid determination of 90Sr in environmental water samples", Talanta, 129 (2014) 623–628