

Determination of Pb-210 from water sample using Trischem Sr-resin

1. Preparation of the sample

1.1 Weight amount of sample into decanting glass. Depending of activity concentration, amount of water needed in the analysis is between 100 and 1000 ml.

1.2 Add 5 ml Pb-carrier (standard solution 1 mg/ml, pipette using calibrated pipette)

1.3 Add 2 ml Fe-carrier (10 mg/ml). **REMARK! Carrier is made into 1 M HNO₃ solution.**

1.4 Let the water sample stabilize few hours (2 - 3 hours). Water sample can be shaken simultaneously using magnetic stirrer. Because both carriers (Pb and Fe) are in the same chemical form, it is expected that carriers are balanced in the few hours.

1.5 After balancing check the pH of the water. Make sure that it is under 2. Add 1 ml H₂O₂, in order to obtain right oxidation state for Fe(III). Remove oikea hapetustila varmistuu. Remove magnetic rods.

1.6 Heat the sample until it starts to boil and then cook about 20 minutes. During this time all hydrogen peroxide is removed from the sample and color of the sample change into the yellowish. After this add concentrated HN₃ until precipitation begins to form (Fe(OH)₃ precipitation). Then pH of the solution is over 8. Make sure that all iron is precipitated. Turn off the heating plate and let the sample stay about 30 minutes on the plate.

1.7 Let the precipitation to settle down at least two hours. Remove the solution from cooled sample using centrifugation (10 minutes 3000 rpm).

1.8 Dissolve Fe(OH)₃ - precipitate into 10 ml 1M HNO₃. Solution is yellowish. Leave the solution over night, after it become bright.

2. Separation of Pb-210 using Trischem Sr resin

Loading the sample into the column and elution of the sample from the column the recommended speed is 1 ml/min, in the washing 3 ml/min.

2.1 Conditioning the Sr column using 3 ml 15 ml 1M HNO₃.

2.2 Put the sample into the column. Discard the solution.

2.3 Rinse the column using 15 ml 1M HNO₃. In the rinsing Fe and Bi are removed. Discard the solution.

REMARK ! Put down time of the elution. This time is used in ingrowth calculations.

2.4 Rinse the column using 30 ml 0.1M BaCl₂. In the rinsing isotopes of radium (Ra-226 ja Ra-228) are removed. Discard the solution.

2.5 Rinse the column using 15 ml 0.1 M HNO₃. In the rinsing polonium is removed. Discard the solution.

2.6 Put clean decanting glass under the column.

2.7 Eluate the lead using 15 ml 8M HCl.

3. Preparation of Pb sample

3.1 Evaporate the sample into dryness

3.2 Dissolve the precipitate into 1 ml 3M HNO₃ acid and remove sample into weighted liquid scintillation vial (this goes the measurement of Pb-210). Sometimes there are undissolved precipitate in the decanting glass, but it do not contain Pb-210.

3.3 Rinse the evaporation container using two times 0,5 ml 3M HNO₃ and remove solution into liquid scintillation vial.

3.4 Weight the vial and take 0.15 ml aliquote for another weighter liquid scintillation vial (this sample is for AAS determination of stable Pb).

3.5 Weight both liquid scintillation vials.

3.6 Add 20 ml 1M HNO₃ into the AAS vial, weight the bottle and measure content of stable Pb using AAS equipment (atom absorbtion spectrometry).

3.7 Add 20 ml Ultima Gold AB liquid scintillation cocktail into the original liquid scintillation vial (3.2). Store the sample for 18 days (at least 10 days) before measurement using liquid scintillation spectrometry.

Regeneration of Sr-column

- 15 - 20 ml H₂O
- 15 - 20 ml 0,1 M TitriplexIII
- 15 - 20 ml H₂O
- 15 - 20 ml 8M HCl
- 15 - 20 ml H₂O

Conditioning of Sr column

- 15 ml 1M HNO₃

4. Waste

During the analysis following waste is produced.

- Pb-carrier solutions from AAS determination are collected into separate waste containers.
- Used Sr resin are collected into separate waste containers.

5. Solutions needed in the analysis

- Pb carrier (1 mg/ml): standard solution
- Fe carrier (10 mg/ml): 4,8 g FeCl₃ · 6 H₂O into 100 ml 1M HNO₃
- 0,1 M HNO₃ (1000 ml): 7 ml conc. HNO₃ (65%) into one liter
- 1 M HNO₃ (1000 ml): 69 ml conc. HNO₃ (65%) into one liter
- 3 M HNO₃(1000 ml): 208 ml conc. HNO₃ (65%) into one liter
- 0,1 M Titriplex III (500 ml): 18,6 g TitriplexIII into 0,5 liter
- 8 M HCl (1000 ml): 663 ml conc. HCl (37%) into one liter