

Strontium analysis of plant samples

Analysis of strontium from vegetation samples

Determination of strontium is based on radiochemical separation and liquid scintillation or proportional counting. Detailed description is presented below.

Dissolving the sample

1. Ash is used in the analysis as follows:

- a) Kelp (*Fucus vesiculosus*) and bear moss (*Polytrichum*) 5 g
- b) Others 10 g

Additional ashing is done at 600 C (1- 2 h) if needed, if sample contains too much carbon (black colour).



Figure 1. Additional ashing of soil sample in the oven

2. Two alternative methods can be used for to convert the ashed samples into liquid samples: microwave digestion or melting with sodium carbonate.

- Mars5-burn:
Choose "sample specific burn" in the "MARS-cookbook". Transfer the liquid to a beaker (litre volume) or to a Teflon covered beaker in case of HF. Dissolve the residue to dry with care. Centrifuge if needed, and wash the pre-precipitate with HCl in ratio 1:4. If the sample is rich in silicate, follow the instructions given in point 4.
- Melting:
Place/put anhydrous sodium carbonate (Na_2CO_3) on the bottom of a platinum bowl and on top of it, put about 90 mg strontium chloride ($\text{SrCl}_2 \cdot 6 \text{H}_2\text{O}$), and 1:4 (weight ratio) ash mixed with sodium carbonate. Add another thin layer of sodium carbonate. Melt the mixture in muffle furnace at 900C 30 minutes (Remember fiberglass gloves and calf-length tongs!)

3. Stir and cool the melt by dipping the bowl bottom into ice-water. Place the cooled bowl (outer side rinsed with distilled water) in a 600 ml beaker. Add distilled water about 200 ml. Extract the sample with a heating magnetic stirrer until the pre-precipitate unsticks the bowl. Centrifuge the sample and discard the liquid.

4. Dissolve the carbonate precipitate into 200 ml HCl in ratio 1:4 and transfer the solution to a beaker (volume of 1 litre).

- If the sample is rich in silicate, dissolve it gently into 37 % HCl, until the carbonates are dissolved. Mix and evaporate the sample with care to dry in a beaker (volume of 600 ml) on a hot plate. Add a further 37% HCl and evaporate to dry. Add about 50 ml of 1:4 HCl. Heat the sample in water bath, centrifuge and transfer it to a beaker (volume 1 litre). Wash the residual precipitate two times with 50 ml of 1:4 HCl, centrifuge and add it to the previous solution.

Oxalate precipitation

5. Add 25 ml of 25% ammonium acetate ($\text{CH}_3\text{COONH}_4$) and 30 g of oxalic acid ($\text{C}_2\text{H}_2\text{O}_4$) to the solution. Heat the solution on a hot plate and adjust the pH to 5-6 with aid of C. 25% of ammonia (NH_3) (checked with pH paper). Dilute the solution to 900 ml, boil it and left the solution to stand for at least 4 hours in order to descent the precipitate.

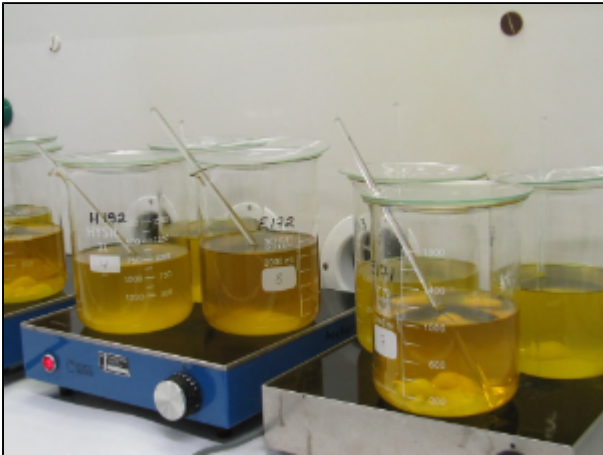


Figure 2. Oxalate precipitation for soil sample

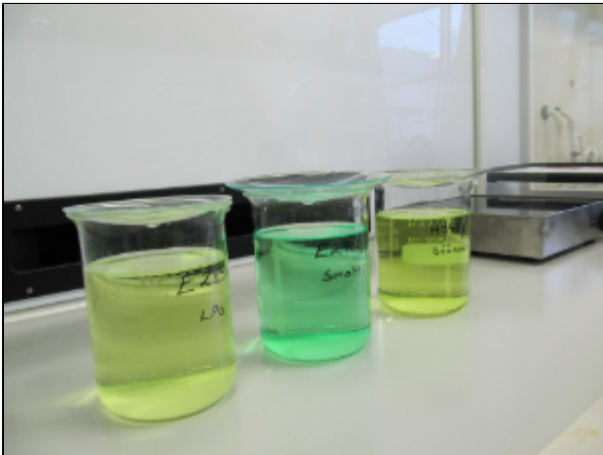


Figure 3. Oxalate precipitation has settled (deposition sample)

6. The liquid is then sucked into drains and the sediment is transferred to a 250 ml centrifuge tube with water. Centrifuge and suck the solution into the drain. Wash the precipitate once with cold water, and centrifuge it.

7. Transfer the oxalate precipitation to a quartz beaker with a small amount of water, dry it with an infrared lamp and ash the oxalates to carbonates in a muffle furnace at 600 - 700 ° C for about ½ hour.



Figure 4. Ashed oxalate precipitation in crucibles

Iron precipitation and chromate precipitation

8. **If the sample does not contain Pb-210**, dissolve the carbonate sediment in 20 ml of 8 M HNO₃ and follow the instructions given in section 12.

9. **If the sample contains Pb-210**, proceed as follows: add 60 ml of distilled water and 65% HNO₃ to the cooled silica crucible, until no dissolution of carbonates can be detected. Boil for at least 10 min covered with a watch glass.

10. Heat the the solution well. Precipitate iron with carbonate free ammonia (pH 8-9), heat the solution a few minutes and filter while hot (41 Wh). Wash the precipitate with hot with ammonia water. Add 10 mg of lead carrier and a few drops of methyl red indicator, adjust pH by the addition of 6 M HNO₃ until the solution turns red and then add 6 M NH₄OH until the solution turns back to yellow. Add 1ml 30% acetic acid and 2 ml of 25% ammonium acetate solution.



Figure 5. colour of the sample turns red

11. Heat the solution well and precipitate lead by adding 1ml 30% Na chromate. Heat, cool and centrifuge the solution. Filter through the Watman paper (nro 40). Solution is made basic with ammonia (lemon yellow color change). Precipitate Sr with ammonium carbamate (3.5 to 4 g), heat, cool and centrifuge. Dissolve the precipitate in 20 ml of 8 M HNO₃.

Chromate filtrate and lead chromate precipitate is treated as hazardous waste/ toxic waste

Extraction chromatography with Sr resin

12. Prepare a Sr-Spec column

Add glass wool on the bottom of the column. Add dist. water to the column (half full). Weigh 3 g of Sr resin (100-150 μ) and mix it with small amount of distilled water. Transfer the resin into the column. Add glass wool on the top of the resin bed. Condition the column with 30 ml 3 M HNO₃. (Resin can be used 10 times but it must be reconditioned after each use by washing it with 30 ml of distilled water followed by 30 ml 3 M HNO₃. If needed to remove Pb and daughters, resin is washed with 30 ml 8 M HCl followed by 30 ml of water and then 30 ml of 3 M HNO₃)

13. Transfer the sample to the column and allow it to drip freely into a clean beaker. Rinse a quartz beaker or centrifuge tube with 10 ml of 8 M HNO₃ and transfer the rinsing solutions to the column.

14. Wash the column twice with 20 ml of 8 M HNO₃ followed by 20 ml of 3 M HNO₃. **Record the exact time when washing was completed (yttrium ingrowth starts)**

15. Elute Sr from the resin with 70 ml 0.05 M HNO₃ and collect the eluate into a 250 ml centrifuge tube.

16. Add 25 % NH₃ to make the solutions basic, pH 8-9 and add 3.5 - 4 g of solid ammonium carbamate (NH₄CO₂NH₂) to the solution. Heat in water bath until precipitate settles in the bottom of the tube. Cool in ice water bath.



Figure 6. Preconditioned Sr-resin columns ready to be used

Measurement of Sr-90

Samples can be counted by liquid scintillation counter (Quantulus or Guardia) or by low background proportional counter (Berthol or Risö). For Sr-90 both counters can be used but if Sr-89 is to be determined proportional counter should be used preferably.

Measurement of strontium with liquid scintillation counter

1 Filter the sample solution from step 16 (above) using **tornisuodatin** on preweighed Whatman 42 filter paper (diameter 3 cm) that has been dried at 105 C for 30 minutes and weighed. Wash the precipitate with 3 x 5 ml distilled water and then with 3 x 5 ml technical alcohol. Dry the sample at 105 C for 30 min and weigh the precipitate.

2. Transfer the precipitate into a preweighed 20 ml scintillation bottle and dissolve it with 1.7 ml of 1M HCl. Shake well.

3. Weigh the scintillation bottle. To determine the yield with AAS take an aliquot of 0,15 ml with pipette and transfer it into a preweighed 50 ml measuringbottle and weigh. Add 1 ml of 1:1 HCl and fill the measuring bottle with distilled water. Weigh the scintillation bottle and determine the weigh of the final solution.

4. Add 6 ml Ultima Gold uLLT scintillation cocktail into the LS bottle containing the sample and shake well. Prepare background sample by pipetting 1,5 ml of 1M HCl and 6 ml Ultima Gold uLLT into a LS bottle. Shake well.

5. Wait 18 days until Sr and Y are in equilibrium. Store samples in dark and cool place. Check that phases have not been separated in LS bottle before counting the samples.

6. Before placing the LS bottles into LS counter (Quantulus) wash the closed LS bottles in ultrasound washing system first 3 minutes in distilled water and then 3 minutes in alcohol.

7. Start measurement next day after samples have been placed in Quantulus (constant temperature achieved).