Polonium and lead from water sample

Determination of Po-210 from water sample

Analysis of polonium
Determination of polonium is based on radiochemical separation and alpha spectrometric measurements. Detailed description is presented below.

Amount of water
Amount of the sample taken for polonium analysis is estimated from radon measurement. Normally the amount of water is between 10 - 1000 ml. 1000 Bq/l of radon contains approximate 0.017 Bq of Po-210. Approximaterly 0.33 Bq of polonium is needed for analysis. Po-tracer (Po-209 or Po-208) is added to the sample by tested pipet or by weighing. If tracer is added by weighing then the syringe must be plastic. The amount of tracer (Bq) is calculated according to equation below.

Activity of radon (Bq/l) x 0.01 x amount of water taken in the analysis (l) / 60

When the water amount taken for analysis is less than 90 ml, then concentrated water (0.5M HCl) is added so that total amount of water is 90 ml. If water amount needed in the analysis is higher than 90 ml, original water sample is evaporated in warm water bath (+80 C) so that the final size of the water is around 60 ml. After evaporation, transfer the water sample (60 ml) into Teflon decanter. Rinse the glass con-tainer where the evaporation was done and add rinsing solution into Teflon decanter together with the sample. If needed you may also use ultrasonic cleaning. Add concentrated water (0.5M HCl) so that total size of the sample is 90 ml. Prepare the deposition system. Mixing in Teflon container can be done using magnetic stirrer or nitrogen gas. If nitrogen gas is used as mixing gas then walls of the Teflon container must be rinsed in the middle of the deposition using distilled or RO (reverses osmosis) water.

Adding of HCl

Original size of water taken in analysis (ml)	**Adding of conc. HCl (ml)**
0 – 100	4
100 – 200	3
200 – 400	2
400 – 600	1
800 – 1000	0

Add 0.9 ml conc. hydrazine - monohydrochlorine (reduce Am, Hg ja Pt, which disturbs Po spon-taneous deposition) and 0.18 g ascorbic acid (reduce Fe3+ -> Fe2+). Reagents must be added into Teflon container before silver disk is put in contact with water sample.

- ascorbic acid 0.2 g per 100 ml water sample
- hydrazine - monohydrochlorine 1 ml per 100 ml water sample

Silver disk is put in separate rib if magnetic stirrer is used as deposition container. When mixing is done using nitrogen gas, the piece of plastic is put with the glue on the other side of the silver disk. Otherwise polonium will also deposited both side of the silver disk.

1st Spontaneous deposition onto silver disk

Silver disk are sterling’s disk, which are polished from one side only. Silver content is 99.9%. The diameter if the

disk is 23 mm and thickness 0.3 mm.

- Wash the silver disk. Wipe it first with ethanol, and then keep it in 1M HNO3 for 5 minutes. After this rinse the silver disk with RO water and then with ethanol.
- Heat the water in the pool to + 80 C. Put the Teflon container in the pool. Make sure that the Teflon container is robustly up. Let the polonium spontaneously precipitate onto silver disk for 3 to 4 hours.
- After deposition turn over the remaining solution into 100 glass bottle. Rinse the silver disk with RO (reverse osmosis) water. Put the rinsing solution into the same glass bottle together with original sample. Rinse the silver disk with ethanol (glass A)
- Let the silver disk to dry onto the paper (lintless) few minutes.
- Count the activity of the preparate using AlphaAnalyst spectrometer (Canberra)
- Store the solution from the deposition. Put it into 100 ml glass bottle and store it at least for 6 months.

Figure 1. Spontaneous deposition of Po

2nd spontaneous deposition onto silver disk

- Add Po-208 tracer to the sample (100 ml bottle) that has been stored for 6 months the day before analysis.
- Mix the sample for few hours.
- Transfer the solution into a teflon made precipitation vessel. Rinse ghe glass bottle with 4 ml of conc. HCl and combine the rinse solution into the sample solution. Add ascorbid acid and hydrazine-monohydrochlorine as described above
- Carry out spontaneous deposition on the silver disk (above)

h5 Polonium and lead activities are calculated followingly:

$$A_{Pb} = \frac{e^{\lambda_1 t_{m2}} \times C_{Po}(2.saostus)}{(1 - e^{(-\lambda_1 (t_2 - t_1))})}$$

$$A_{Po} = e^{\lambda_1 t_1}\left[e^{(\lambda_1 t_{m1})} \times C_{Po}(1.saostus) - A_{Pb}(1 - e^{(-\lambda_1 t_1)})\right]$$

CPo(2.saostus) = polonium activity (Bq/l) 2.precipitation
CPo(1.saostus) = polonium activity (Bq/l) 1.precipitation
1 = polonium decay constant (1 = Ln2 / T½ = 0,00501)

t1 = time from sampling to 1st precipitation
t2 = time from sampling to 2nd precipitation
tm1 = time from 1 precipitation to the measurement
tm2 = time from 2. precipitation to the measurement

Critical steps in the analysis

1. Addition of the tracer. The final result is calculated based on tracer yield.
2. Temperature of double-boiler, too low temperature decrease tracer yield.
3. Environmental samples, where is a lot of iron the amount of ascorbic acid (0.2 g) is not enough for reduce iron.
4. Dissolution of environmental sample. If wet combustion is done in the mixture of HNO_3 and HCl, all HNO_3 need to evaporated. Otherwise, it may change density of silver disk. In the worst case the whole sample can be destroyed.