Uranium from water sample

Analysis of uranium from water sample

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

Amount of water

Amount of the sample taken in uranium analysis is estimated from gamma spectrometric measurement (sediment or soil sample) or from gross alpha measurement (water sample). Normally the amount of uranium in the preparate is 50 µg. Very high amount of uranium may thicken the preparate and then it is reasonable to decrease amount of the sample taken in analysis.Tracer is added in the sample by tested pipet or weighing. When water amount taken in analysis is less than 100 ml then concentrated water (0.05M HCl) is added so that total amount of water is 100 ml.

Equilibrium of water sample

The amount of water taken in analysis is weighted and amount is written down in following chart. Known amount of tracer (U-232) is added in water sample after it is shaken in glass beaker for one hour (with magnetic stirrer).

Co-precipitation with iron hydroxide

Check that pH value is less than 1. Add stable iron (Table 1) and shake the sample with magnetic stirrer for 10 minutes. In the end, add 1ml 30 % H_2O_2 in order to make sure that oxidation state of iron, Fe(III), is correct.

Table 1. The amount of stable iron added in water sample.

Amount of water (ml)	**The amount of stable iron added in water sample (mg)**
0 – 200	10
200 – 400	15
400 – 700	20
700 –	25

Take magnetic stirrer off and put a clock glass onto the beaker. Heat the sample on the heating plate about 20 minutes. After 20 minutes, there are no visible bobbles seen in the sample. Decrease the heat down.

Add 6N NH_3 solution until the precipitate start to form (Figure 1). Keep glass beaker on the heating plate for few minutes (temperature is low and water is not boiling). In the end, add NH_3 solution so that pH value is approximately 8. Cool the sample. Let the precipitate to settle down. Separate solution from the precipitate (suck the most of the solution by water suction pump). The precipitate and rest of the water are removed to the centrifuge funnel (Figure 2). Centrifuge sample 10 minutes using rotation speed 3000 rpm. Suck the solution and dissolve the $Fe(OH)_3$ precipitate in 15 ml of concentrated HCl acid.

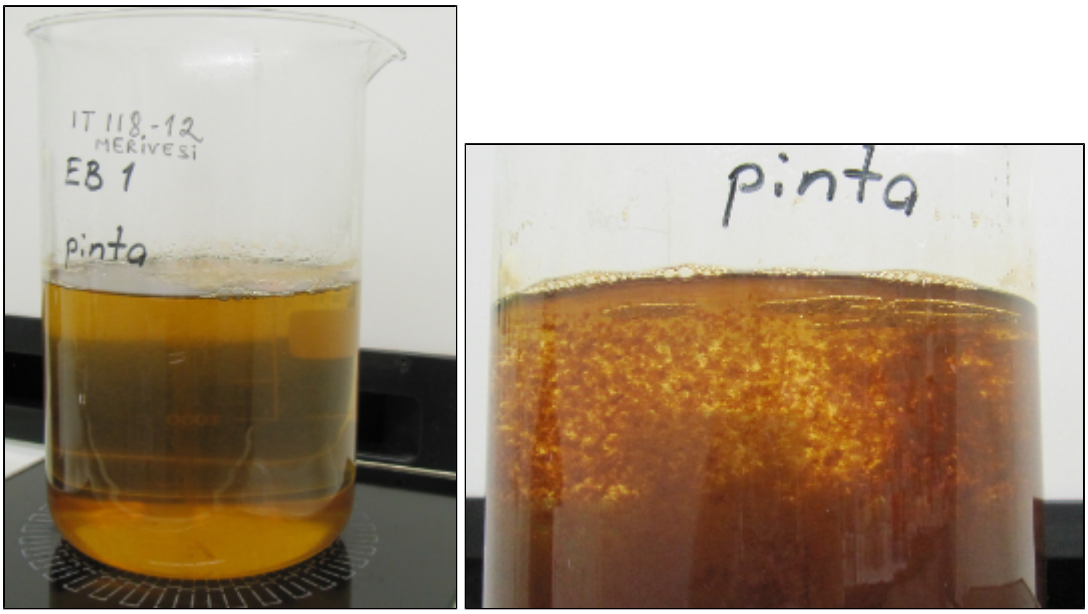


Figure 1. Forming of iron precipitate in water sample

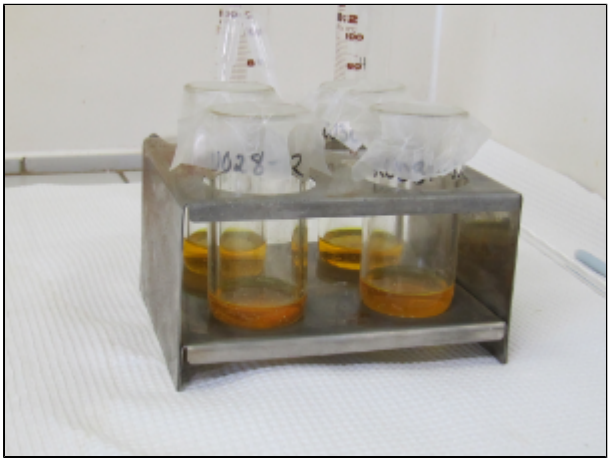


Figure 2. The precipitate in the centrifuge funnel

Separation of uranium isotopes

Before uranium separation, the column need to be conditioned and regenerated (if the resin is used earlier):

Conditioning:

- 30 ml conc. HCl

Regeneration:

- 40 ml 0,1M HCl
- 40 ml 1 % NaHSO3 in 6M HCl
- 2 x 40 ml 6M HCl
- 40 ml ultra pure water (H2O)
- Backwash with syringe and H2O

Put the sample, dissolved in concentrated HCl (Figure 3), into the anion exchange column, Dowex 1x8, 50/100 mesh (Figure 4). Adjust the dropping rate (about 1 drop in 5 seconds). After loading the uranium into the columns, the column is washed with 60 ml of concentrated HCl in order to get rid off isotopes of thorium. Abandon the washing solutions (contains isotopes of thorium and radium). Eluted the uranium from the column with 40 ml of 0.1 M HCl (Figure 6). Evaporate the solution with mild temperature. Be careful that sample is not burned on the bottom.

Figure 3. Dissolved sample in concentrated HCl

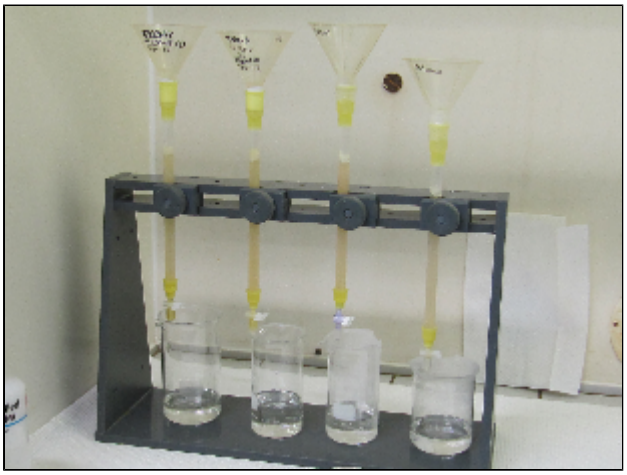


Figure 4. Uranium columns before sample load.

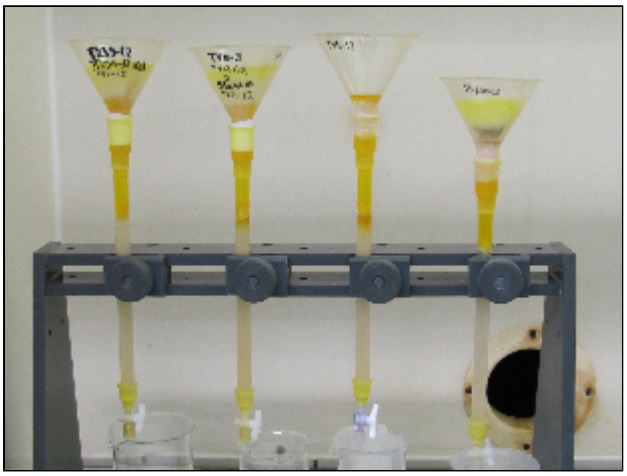


Figure 5. Uranium columns after sample load.

Figure 6. Eluted uranium from the columns

Uranium preparate

Dissolve the residue in 2 - 5 ml 1M HCl acid. If precipitates do not dissolve easy you may use also ultrasonic equipment.

Move solution into plastic test tube, size 50 ml (Figure 7). Rinse the teflon beaker twice with 2 ml of 1M HCl acid

and add them together with the original sample. Add 0,5 ml Ce-carrier and mix the plastic test tube using Vortex mixer.Add 15 % of TiCl3 by drops (about 4 - 5 drops). This will reduce Fe and U. The amount of TiCl3 is enough when colour of the solution change from yellow to light violet (Video 1). Close the TiCl3 bottle (oxygen destroy the solution).

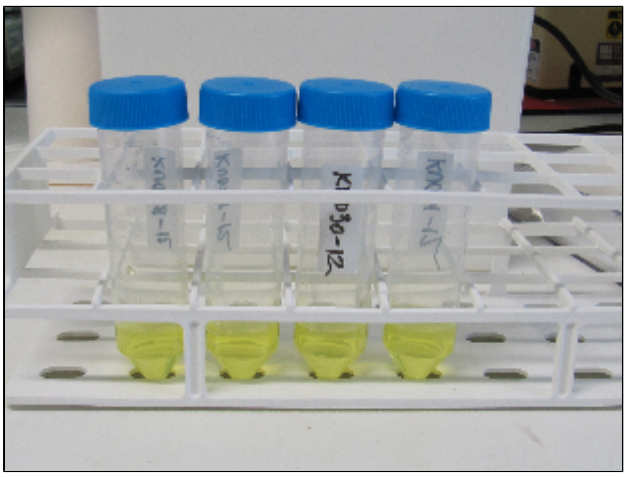


Figure 7. Sample in plastic test tube before reducing uranium

Video 1. Reducing Fe and U using TiCl3

Make the precipitate by adding 1.5 ml of 40% HF acid. Uranium will co-precipitate as fluorine. Mix carefully using Vortex mixer and let precipitate to settle. Put the sample in ice and let it be there at least 45 minutes. Shake the sample 2 - 3 times during that time.

After the filtration system has been prepared put the pump on (video 2). Test the system using 5 ml of ethanol. Open the tap (in order to have pressure in the system). If the system is tight then you may filtrate your sample, which is carefully mixed. After you have filtrate your samples wash the plastic test tube twice with 5 ml of ultra pure water. Filtrate the washing solutions. At the end wash the membrane with 5 ml of ethanol before you take it from the filtration system. Dry the membrane. After membrane is dry glue the membrane onto the steel plane. Write the name of the sample onto the other side of the steel plane.

Video 2. Preparing the uranium preparate

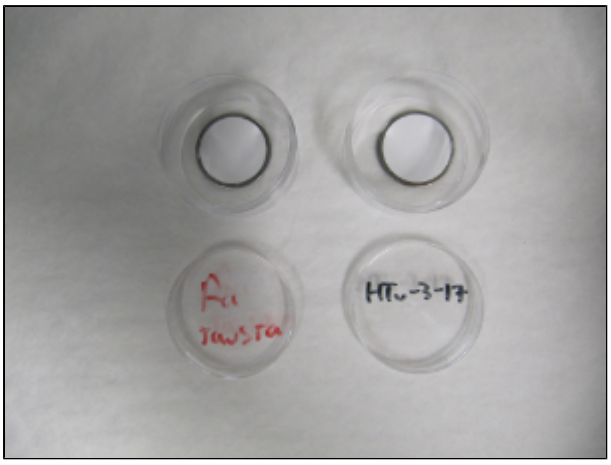


Figure 8. Uranium preparate for alpha measurement.

Measurement of activity concentration of uranium

The radioactivity of uranium isotopes deposited on the membrane is measured by counting the alpha particles from U-232, U-234, U-235 and U-238 in an alpha spectrometer. The purification of uranium by the ion exchange procedure and deposited on the membrane normally gives alpha peaks with a FWHM of about 20 – 30 keV.

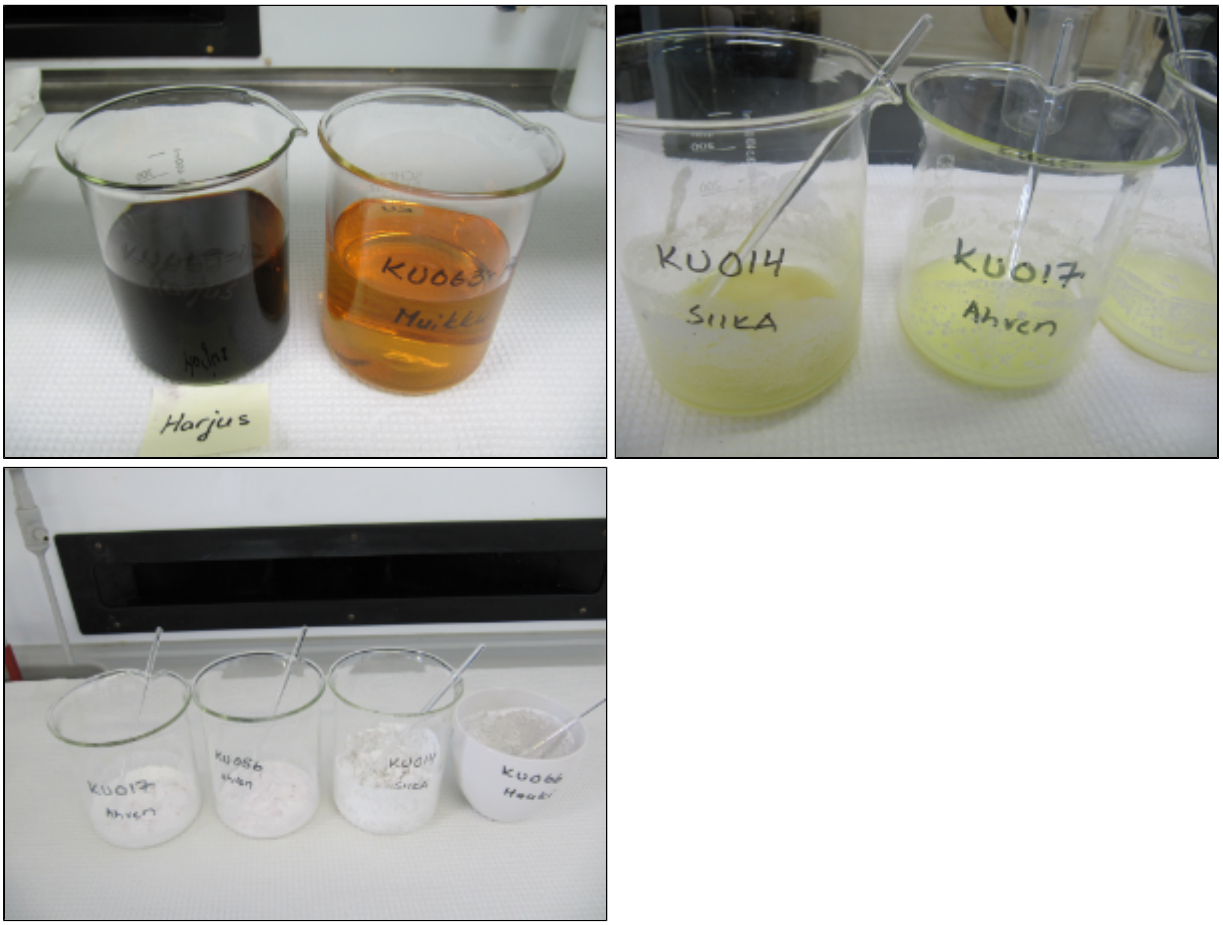
Problems and solutions

Water samples with high organic mater content

Recovery of the uranium decreases if water contains high amounts of organic matter. In the pretreatment water samples need to be oxidized using hydrogen peroxide H2O2 and amount of iron carried need to be increased.

Plants, fish and berries

These matrixes contain a lot of incombustible carbon and organic compounds. Before uranium separation sample is combusted in 450 degree, then sample is digested using micro wave digestion using HNO3 acid and hydrogen peroxide H2O2 (picture 9). Colour of the samples may vary from yellow to dark black. After digestion sample is evaporated into dryness. New combustion in 450 degree is made.



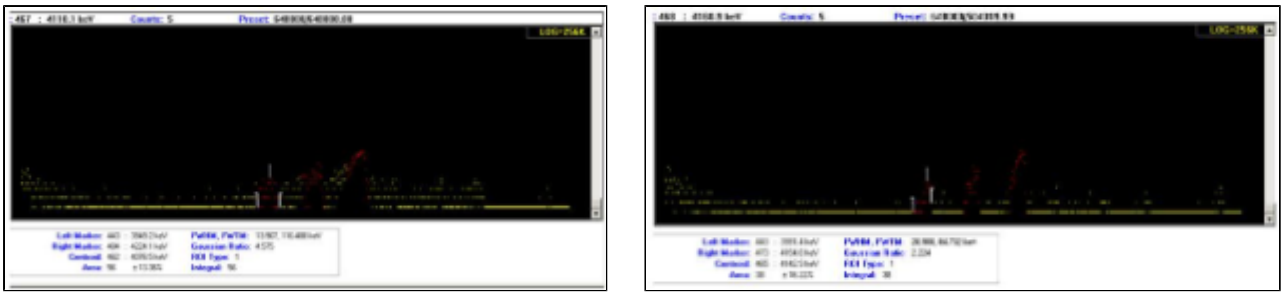
Picture 10. After digestion sample is evaporated into dryness (left), digested samples using micro vawe digestion (in the middle), sample after second combustion (right).

Ion exchange

If sample contains a lot of silicate, the column may block up. Removing the faucet and washing it during ion exchange may help. Also dissolving the sample into 9 - 10 M HCl acids (not in conc. acid) may reduce the problem.

Spectra

Two examples from bad and good uranium spectrums.



Picture 11. Examples from bad (left) and good (right) uranium alpha spectrum.

Waste

In the analysis waste is produced. The acids are discharged into the drain after neutralization. Used ion exchange resin and HF acid are transport to the waste disposal plant.