Pretreatment of seawater for transuranic analysis

Pretreatment of seawater and other water samples for transuranic analysis

- Sample size of 100-200 liter seawater

1. Add conc. HNO3 to the sample until pH about 2, (approx 50 ml HNO3/30 l water)

2. Transfer the water sample into a precipitation vessel and add 300 mg Fe-carrier, Pu-242 tracer (if Pu is to be determined) and Am-243 tracer (if Am is to be determined). Mix overnight.

3. Precipitate by adding 32 % NaOH at pH 8 - 9 . Mix for 2 hours. Let the precipitate settle down overnight.

4. Transfer the precipitate to several buckets through the tap in the bottom of the precipitation vessel. Let the precipitation settle down in the buckets and transferthe solution away using suction. Combine the precipitates from each bucket into a 5 l beaker. Rinse the buckes with a mixture of water + few drops NH3 (pH 8-9). Leth the precipitate settle down (figure 1) and remove extra solution with suction. Dissolve the precipitate with min. amount of conc. HCl (Figure 2) and dilute into 2-3 l with distilled water

Figure1. Settled precipitate (with Pu) from seawater sample

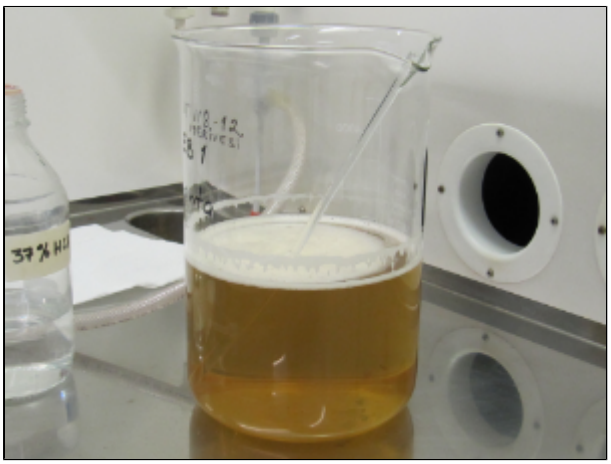
Figure 2.Precipitate has been dissolved with HCl.

5. Heat the solution and precipitate Fe(OH)3 by adding carb.free NH3 at 8 – 9. Stir for 1 h using magnetic stirrer (Figure 3).

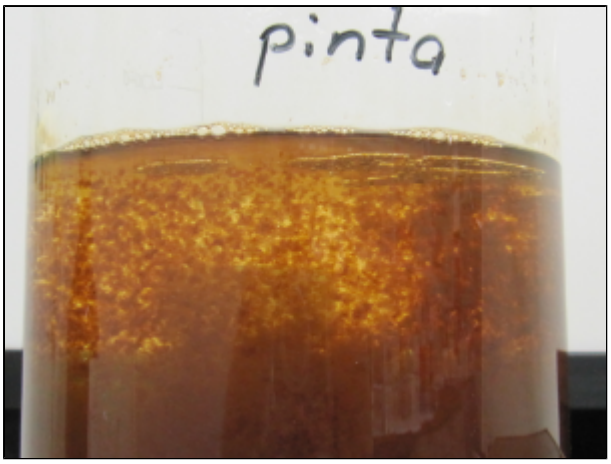

Figure 3. Fe(OH)3 precipitate

7. Let the sample cool and precipitate settle down for at least 3 h. Solution is removed with suction.

8. Transfer the precipitate using basic water (dist water+ NH3, pH 8-9) into 250 ml centrifuge bottles and centrifuge. Discard the solution. Wash the precipitate with bascic water (dist water+ NH3, pH 8-9) and centrifuge. Discard the solution. Dry the precipitate in centrifuge bottles at 105 oC in a heat cabinet for at least 4 h.

9 Dissolve the precipitate with 100 ml of 8 M HNO3 while heating in a 400 ml beaker. Add 3 ml of H2O2 and heat under watch glass at 90 oC for 1 h *(If needed carry out HF digestion)*. Filter the sample through glass fiber filter using Bühner funnel. Residual is discarded.

10. Heat the solution and add 1 ml H2O2 and continue heating the beaker covered with watch glass for 1 h. Add approx. 2 g (1 teaspoon) NaNO2 into the solution. Let cool for 1 h. Continue Pu analysis by conducting anion exchange as described for vegetation samples.