Procedure for Filter-Fe

Instructions for analysing corrosion products on a membrane filter as given by Maja Skou Jensen in the 2018/2019 round robin.

The instructions assume that 1 L of sample has been filtered on a 0.45 µm membrane filter shortly after sampling. The filters may be dried in a place protected from dust or analysed directly.

It is advisable to run a blind test on the membrane filters to ensure that they do not contain iron.

For ICP:

1. *Transfer the filter to a small beaker, diameter a little larger than the filter. Add 25 mL of 2 % HCl (or HNO3).*
2. *Cover the beaker with e.g. a watch glass and heat it in water bath at 90-95 ®C for ½ h, let it cool down.*
3. *Transfer the solution to a 50 mL volumetric flask, then flush filter and beaker with 2 x 10 mL UPW and add this to the flask, Fill to the mark with UPW to make up to 50 mL.*
4. *Run the analysis at the ICP, using calibration standards in 1 % HCl as reference, e.g. 10, 20, 50, 100, and 200 µg/kg.*
5. *Convert the result to concentration in the sample by division by 20.*

And for the FerroZine method:

1. *Transfer the filter to a bottle, add 50 mL of ultrapure water (UPW) and standard amount of chemicals for digestion (125 µL of thioglycolic acid for the Ferrozine method). Cap the bottle and heat to 90 °C for ½ hour. Let cool to <30 °C.*
   Remark: For filters with high amount of solids, it may be necessary to double or quadruple the amount of thioglycolic acid to dissolve all particles.
2. *Add remaining chemicals for the spectrophotometric method (e.g. Ferrozine reagent/buffer) and allow appropriate time for color development.*
3. *Run the analysis normally from here.*
4. *Divide the result by 20 to get a figure comparable to the grab sample results.*