Readme file for iron analysis at NIOZ (2001-09-14)

Principle investigators: P L Croot and P Laan (NIOZ)

Method for the determination of Iron used by NIOZ.

The method used by NIOZ is based on the method developed by Obata et al. 1993 with some slight differences in reagent concentrations and acidifying according to samples by J.T.M de Jong et al. (1998) Analytica Chimica Acta.

The following reagent concentrations were used:
0.3 M HCl by diluting concentrated HCl (Merck supra pure)
0.8 M ammonia by diluting 25% NH4OH (Fluka supra pure)
0.1M H2O2 by diluting 30% H2O2 (Merck supra pure)
TETA, Triethylenetetramine 95% (Organics) and luminol, 3-Aminophthalhydrazide (Fluka) were used as received. Luminol was dissolved with about 40 mg K2CO3 (Merck supra pure).
Luminol 0.3 mM, TETA 0.7 mM.
All reagents were diluted with MQ (Millipore() >18.2(M water.

All filtered and unfiltered samples where collected in 100-ml HDPE bottles which where filled with 0.01 nM acidified MQ. Bottles where rinsed 3 times with sample before filled and accidified to pH (1.8 with 3QD HCl. (we used to acidify our samples with HNO3 but because we used HCl at ANT XVIII/1 we continued using this.)

Concentrations of Fe (III) in the acidified samples were determined with an Fe (III) analyzer (PMT) using chelating resin concentration and chemiluminescence detection (J.T.M de Jong et al. (1998), Analytica Chimica Acta). The determined Fe is total dissolved Fe because the samples where acidified to pH (1.8. The sample is inline buffered with ultra clean NH4Ac buffer to pH 3.5-4.0 for an optimal recovery of the loaded sample onto the 8-hydroquinoline.

The detection limit during the Polarstern cruise was approximately 20-40 pM. More precise information on the analytical aspects can be supplied.

The Fe standard solution we used was a Tritisol 1000 ppm standard solution. (1000+/- 2 mg). The solution was diluted 100x with 0.1M HCl to a 179.1 uM solution (stock 1). This solution was diluted another 100x (0.1 M HCl) to create a 1791 nM Fe (stock 2) solution were from the additions where made.

The system was calibrated by the method of the standard addition. We added the stock2 solution onto a sample. The range of addition was depending on the concentration of the samples. Added amount of stock varied mostly between 0 and 80(L. Of course was a extra spike of standard needed for the higher IN-PATCH stations. The system was calibrated at least once per day but normally every 20-25 samples.

The blank determination is based on two parts, the MQ blank and the reagent blank. Because we are rinsing our system for 1 minute with MQ, to get rid of the salt in the colom, we do a MQ blank by running a complete cycle without loading sample. This will give us a MQ blank value. The reagent blank is determined by analyzing a "known" sample but with a double addition of acid and buffer.

Sensitivity was checked by looking at the counts/Nm Fe during the calibration curve. It seems that the sensitivity was not as constant as we hoped. The sensitivity during the whole cruise was 57945 (18634 (n = 42). The drop in sensitivity can be mainly caused by the luminol that seems to be unstable during the cruise. As soon as there was a new batch made the sensitivity reached a decent number again although the over all trend during the cruise is down.