

Sample preparations for ICP-OES

June 2020

1. Overview

The samples analyzed must all be filtered (minimum 0.45 μ m) and acidified. The recommended volume is 4mL per sample, in principle 3.5mL is sufficient.

NB:

- Peek nebulizer: 10% TDS maximum; 2% recommended.
- Glass nebulizer: 1% TDS maximum; 0.2% recommended.

Samples should normally be prepared in 2% nitric acid as STD. If the matrix is too different, plan to make customized STDs in the same matrix.

NB: HF is strictly forbidden on ICP-OES.

2. Soils and sediments sample preparation by aquaregia extraction

In principle on a sample dried for one week in a 40°C oven sieved at 2mm and crushed.

- Weigh 1.0g (+-0.1g) of dried sample into a 100 ml Erlenmeyer flask
- Add 2-3 ml of water milli-Q → to obtain a paste.
- Add 10 ml of AquaRegia solution (in a ratio of 3:1 HCL supra pure and HNO₃ supra pure: 7.5ml and 2.5ml respectively)
- Close the Erlenmeyer flask with a stopper and leave the solution to stand overnight at room temperature.
- Install refrigerants, circulate water at a low flow rate and reflux the solution for 2 hours.
- Rinse the refrigerant with 30ml HNO₃ at 2M; collect the rinse solution
- Filter the solution through a 45 μ m cellulose filter directly into a 50 or 100mL volumetric flask
- Rinse Erlenmeyer flask and filter with HNO₃ at 2M
- Fill the volumetric flask with MiliQ water.
- Analyze the solution with ICP-OES.

3. As : Sample preparation hydride: speciation mode

Standards and samples for speciation must be prepared on the day of analysis.

All samples and STDs should be diluted 50:50 with 0.4M citrate buffer at pH=5.0 (prepared with citric acid at pH=5.0 with NaOH). 8mL of each is required for analysis.

NB: plan sufficient volumes for the check and put some regularly.

Preparation of NaBH₄:

Weigh 0.5g of NaOH in a PP vial, add 200mL of MiliQ water and dissolve them. Add 5g of NaBH₄ and then add MiliQ water to a final volume of 1000mL.

The NaBH₄ can be used a maximum of 1 week after its preparation.

4. As : Sample preparation hydride: low concentration analysis mode

Samples for total arsenic analysis can be prepared in advance and kept for a few weeks.

In order to be sure to analyze all the arsenic a pre-reduction must be done on all samples.

NB: as long as the samples have been prereduced they must be analyzed as soon as possible.

Preparation of NaBH₄ :

Weigh 0.5g of NaOH in a PP vial add 200mL of MiliQ water and dissolve them. Add 5g of NaBH₄ and then add MiliQ water to a final volume of 1000mL.

The NaBH₄ solution can be used a maximum of 1 week after preparation.

Preparation of the 5% KI / ascorbic acid solution

Weigh 5g KI and 5g ascorbic acid in a polypropylene bottle and dissolve them with 100mL MiliQ water

Pre-reduction of samples:

Prepare 15mL falcon tubes: about 20 at a time

Add 4mL of sample, 0.4mL of ascorbic acid KI solution and 1.6mL of concentrated HCl. Allow the mixture to react for 20min then make up to 10mL by adding 4mL of purified water.