General information and sample preparation for ICP-OES analysis

*Version 1.2, Sebastiaan Derese (+32 473 47 13 06)*

# Responsibles for the analysis

The responsible for the analysis is Sebastiaan (+32 473 47 13 06 or extension 6048). For any questions regarding the ICP-OES analysis, or to transfer your samples for analysis, please contact them.

# General introduction

ICP-OES is a reliable, accurate and easy-to-use technique to determine the concentration of (primarily) **cations** in environmental samples. ICP-OES consists of two parts, namely the ICP (Inductively Coupled Plasma) and OES (Optical Emission Spectrum) parts. This means that your samples are nebulized and brought into a hot Argon plasma (which is being held stable and initiated by an electric induction coil), by which the ions in the solution are being excited (valence electron is brought into a higher energy scale). After the excitation, the element emits light of a certain wavelength, when the electrons that have been excited to a higher energy level drop down to their ‘base energy level’. This emission is detected by a CCD (charge-coupled device), which allows for simultaneous multi-wavelength detection.

Table 1: Quantification range of the ICP-OES methods

|  |  |
| --- | --- |
| Element | Quantification range (ppm, mg/L) |
| Na | 0.01 – 50 |
| K | 0.01 - 50 |
| Ca | 0.01 – 50 |
| Mg | 0.01 – 50 |
| Ag | 0.01 – 50 |
| Al | 0.01 - 50 |
| B | 0.01 – 50 |
| Ba | 0.01 – 50 |
| Bi | 0.01 – 50 |
| Cd | 0.01 - 50 |
| Co | 0.01 – 50 |
| Cr | 0.01 – 50 |
| Cu | 0.01 – 50 |
| Fe | 0.01 - 50 |
| Ga | 0.01 – 50 |
| In | 0.01 – 50 |
| K | 0.01 – 50 |
| Li | 0.01 - 50 |
| Mn | 0.01 – 50 |
| Ni | 0.01 – 50 |
| Pb | 0.01 – 50 |
| Sr | 0.01 - 50 |
| Tl | 0.01 – 50 |
| Zn | 0.01 – 50 |

As can be seen, the quantification range is very similar and rather wide for a variety of elements. This, together with the use of the CCD and the absence of interferences allows to prepare multi-standards (standards with a known concentration of multiple elements), but also to measure several elements directly from one sample.

# Costs of the analysis

A new system for the costs of the analysis has been defined at 60 euro per hour of use of the ICP-OES. As one sample takes about 3 minutes (to be exact: 172 seconds) from start to finish, you can analyze some 20 samples per hour. However, as per 20 samples usually a calibration of 7 samples is necessary, you can **calculate that the cost of one sample is 3.87 euro.**

It is clear that a great number of samples also entails high costs. Therefore, please **plan** your sampling in advance and reduce the amount of samples as much as possible. Additional measurements can be performed later, should the need arise.

# Time costs and analysis interval

Outside of cost limitations, the analysis itself also consumes a lot of time. A good measurement (three measurements, of which the average is withheld) takes 3 minutes. Please, let us know **well in advance** how many samples and how many elements need to be measured. Preferably, do not provide us with more than 120 samples for one analysis day on a regular basis, as this means more than 8 hours of analysis time.

# Calibration standards

**IMPORTANT REMARK:** Please check beforehand whether there are still calibration standards (lab 3, calibration cabinet – the first cabinet as you enter lab 3)!

For the preparation of your calibration standards, please always use the certified reference solutions (please ask the responsibles where you can find them).

The calibration standards are prepared as follows (you need about 20 mL of calibration standard for 2 methods):

1. Take a 100 mL volumetric flask. Add some **fresh** Milli-Q (a few mL suffice). **If you have a specific matrix containing e.g. a large amount of organics or HCl, use this as the dilution matrix instead of Milli-Q!** Make sure the concentration of the matrix in the standards is similar to what you expect in your samples.
2. Add 2% HNO3 to your samples (this corresponds to 3 mL 65% HNO3-solution for a 100 mL flask).
3. Pipette (**with analytical pipettes!**) certified reference solution (CRS). These CRS can be found in the ‘acid and base’ cupboard in Lab 2. Please note this counts for 100 mL flasks. Also, **prepare a blank** (which does however have the same matrix and does contain HNO3).

Table 3: Pipetting volumes for the calibration standards

|  |  |  |  |
| --- | --- | --- | --- |
| Element/Standard | Volume to pipette (standard, µL) | Volume to add (mQ water, mL) | Remarks |
| 50 | 5000 | 92 |  |
| 20 | 2000 | 95 |  |
| 5 | 500 | 96.5 |  |
| 1 | 100 | 96.9 |  |
| 0.2 | 20 | 96.98 |  |
| 0.05 | 1000 | 96 | *Use 5 ppm standard!* |
| 0.01 | 200 | 96.8 | *Use 5 ppm standard!* |
| Blank | 0 | 100 |  |

1. Fill up to the mark with either Milli-Q or your matrix. Cap with Parafilm.
2. Homogenize by turning the bottles up and down a few times.
3. Store in the standards cupboard in Lab 3.

# Sample preparation

**Important remark:** all samples are nebulized before being carried to the ICP. Please note that the nebulizer is in fact a Venturi, and can thus clog easily. Therefore, always filter your samples until they are **perfectly clear**. You can use 0.45 µm syringe-filters for this.

The sample preparation is very similar to the calibration standard preparation. The minimum amount of sample required per method is 8 mL, but please prepare a bit more in case of problems.

1. Take a PS test tube (analys box ‘new ICP-OES tubes’). There is a box filled with these test tubes in the chemicals room.
2. Add 2% HNO3 to your samples (this corresponds to 300 µL 65% HNO3-solution for a 10 mL sample, which is the preferred size of the sample after dilution).
3. Pipette (**with analytical pipettes**) your sample until the end concentration is within the quantification range. Preferably, use the **same dilution factor** for all your samples, so that you can quantify the matrix effect in your standards. You have to have 5 mL samples, but we prefer to use 10 mL samples.
4. Homogenize.

# Sample transfer

When you have finished all of your samples, please conserve them in the dark. They do not have to be cooled or frozen, but preferably store them in the fridge in lab 2. Make sure your samples are labeled **only** with a number (1, 2, 3,…) **or** that you provide us with a sample list (including dilution rate). Of course, ensure that on the global sample rack, your name and the date is labeled correctly. **Please, do not individually wrap your samples with parafilm. Instead, use a sheet of parafilm to prevent dust or water to enter the samples.**

Send an e-mail to either Sebastiaan or Marjolein, with attached an Excel file with the sample names and their dilution, and a picture of where we can find your samples. They will let you know when the next analysis is planned and when you can expect your results. After the analysis, we will send you this Excel file with the concentrations. Standard deviations can be transferred as well.

# After analysis

After you have measured your samples, discard the contents of the test tubes in the ACID liquid waste.

The test tubes have to be rinsed twice with demi water, and are then transferred to the acid bath for 24 h of soaking.

After 24h, remove your tubes, rinse them twice with mQ water, and store them bottoms up in a tube rack until dry. When dry, place them back in the ICP-OES tube box.