Standard Operating Procedure IC Metrohm

With this device the analysis of cations and anions can be done (separately).

Calibration range	Maximum acceptable concentration range for each ion
Cation: 0.05 - 2 mg/L	Cation: < 50 mg/L
Anion: 0.02 - 5 mg/L	Anion: <10 mg/L

- **Eluent solution** (a separate pdf file for the preparation)

Anion eluent (2 L): 7.5 mM Na_2CO_3 and 0.75 mM NaOH and regenerant: 250 mM H_2SO_4

Cation eluent (2 L): 1.7 mM HNO_3 and 1.7 mM PDCA

Regenerant solution (only applicable for anion analyses): 0.2 M oxalic acid ($C_2H_2O_4$ x 2 H_2O), 500 mM H_2SO_4 and 10% aceton

In order to have proper results renew the eluents after two weeks, the regenerant solution can be used for several months.

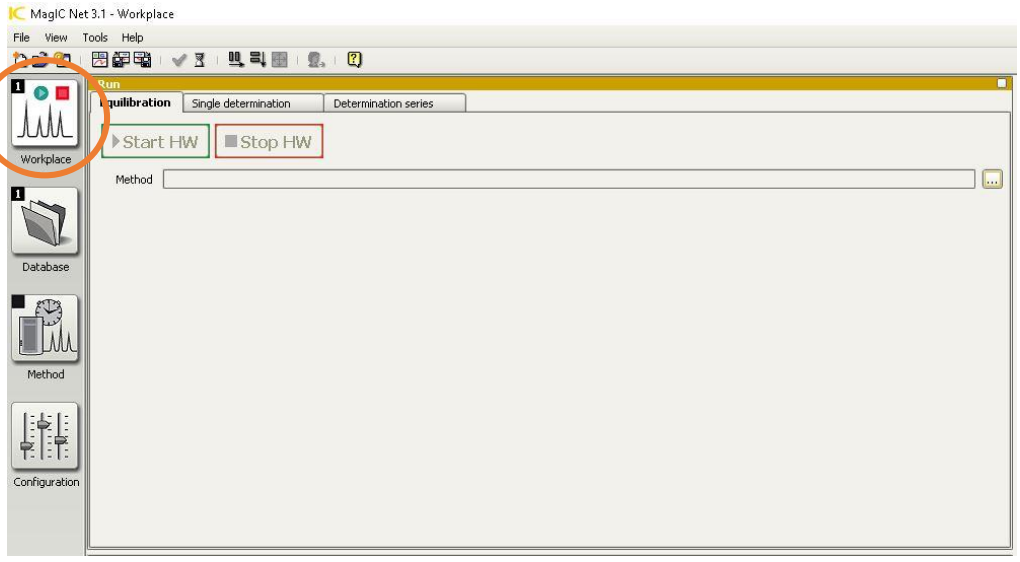
Always use the glassware specially assigned for the different chemicals and cover them with paraffin paper to prevent contamination!

READ the pdf file in order to follow the proper eluent preparation procedure

Approximately, the flow rate of the eluent is 1 mL/min. A single sample analysis takes 35 min. With these values, the acquired eluent volume can be calculated. Take into account that the eluent level should always be higher than the tube tip in the eluent bottle, to avoid air suction. When extra eluent solution is added to the eluent bottle during refill, make sure that the tubing is always submerged in the liquid when samples are running in order to avoid suction of air.

- **Equilibration**

Before analysing any samples, the IC needs an equilibration in order to set all parameters to the desired values (oven temperature, eluent flow rate, pressure). Go to 'Work place - Equilibration' and add the appropriate method in the PaInT folder, according to column and the type of analysis. Afterwards, Start HW (Hardware).



	Anion	Cation
Pressure	12 MPa	6 MPa
Temperature	45 °C	35 °C
Flow rate	0.8 mL/min	0.9 mL/min

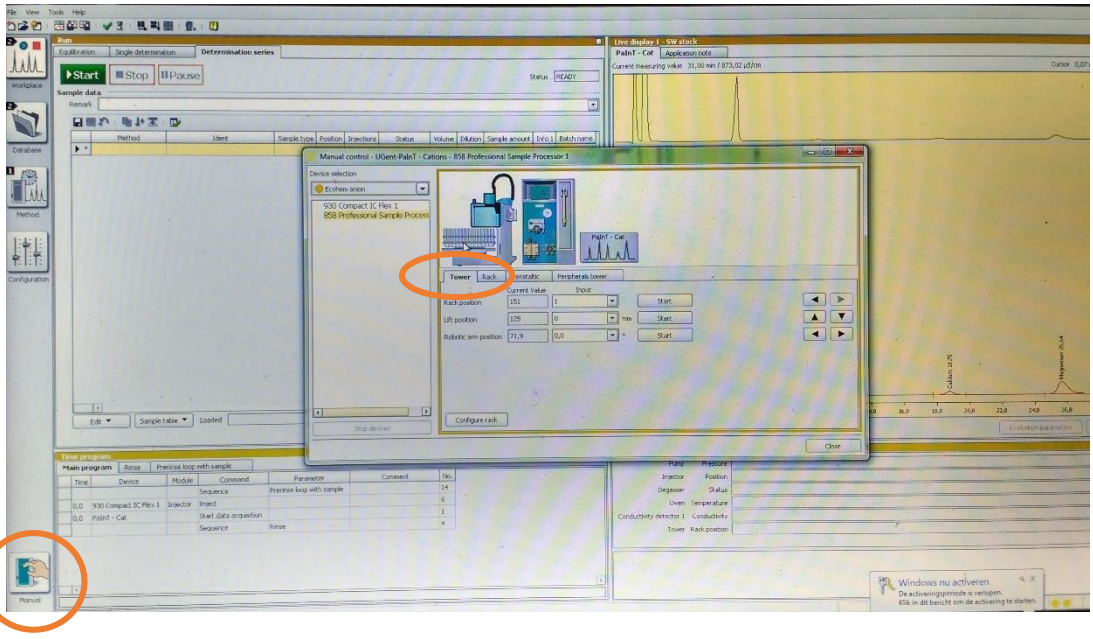
These values should be met after 45 min with a stable background noise level around 880 µS/cm or 1.8 µS/cm, for cation and anion analysis respectively. If not, please contact one of the responsibles for more information (your first contact is your tutor).

- **<u>Preparation of samples</u>**

The required sample amount is 7 ml for each analysis, so for a cation and anion analysis 14 ml is required. If a dilution is required, always use **fresh Milli Q** from the 6th floor. In addition to the samples and the eluent solution, make sure the three bottles on the sample rack are completely filled. One with 10% (v/v) methanol in Milli Q (position 149) and the other two with Milli Q. Put them always back at the same positions!

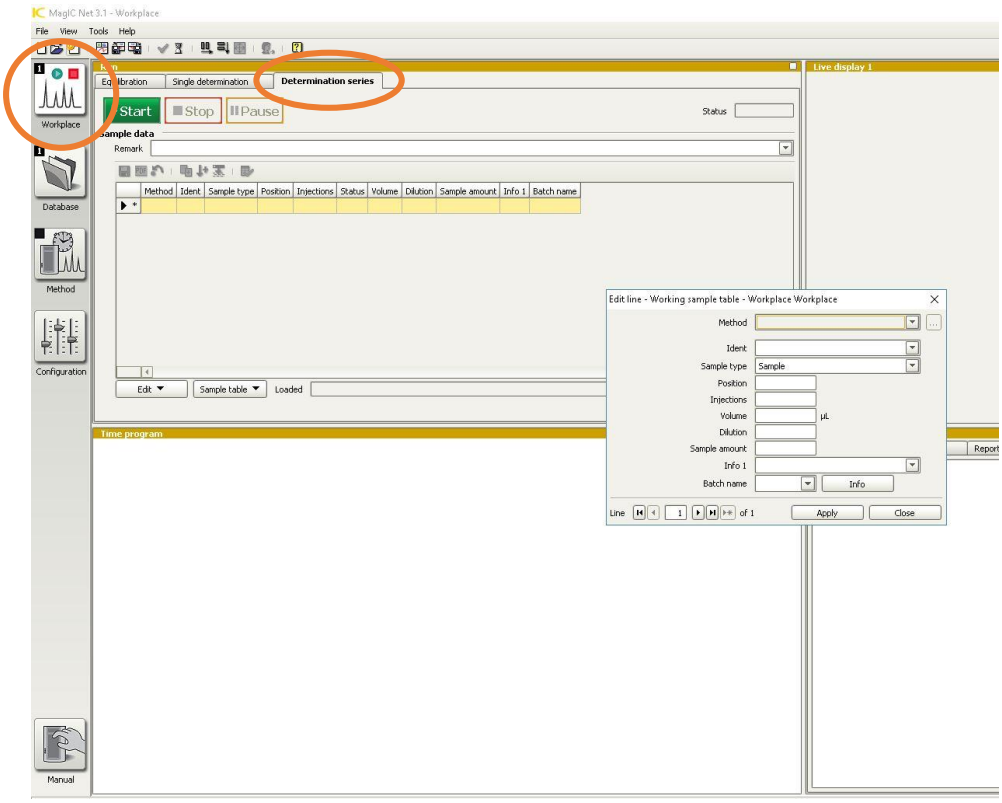
In order to get access to the bottles, turn the rack of the IC. Do this by a 'Manual Operation', click on the picture of the sample rack and insert the value 25 in the position command. After filling the bottles, no other proceedings are required. The rack will automatically take a sample when it is desired.

The IC is used for clean samples only, highly contaminated samples need to be diluted. When non-dissolved solids are visible in a sample, prefilter it before analysis. **Fe^{3+} is dangerous for the suppressor**, so don't analyse samples with a high metal content on the IC.



- **<u>Addition of samples</u>**

Add samples to the table list in 'Work place – Determination series'. If an old sample table is shown, go to 'Sample table' and choose new. Double click on the blank row to edit the table. The required parameters are Method, Ident, Position, Dilution and Sample amount. Method is the type of analysis that will be performed according to the used column, always pay attention on the appropriate department name. Ident is a tag of your choice, Position relates to the sample position in the sample rack, Dilution and Sample amount should get the value 1. If preferred, choose a batch name to group your samples, then press 'Enter' in order to go to the next sample.



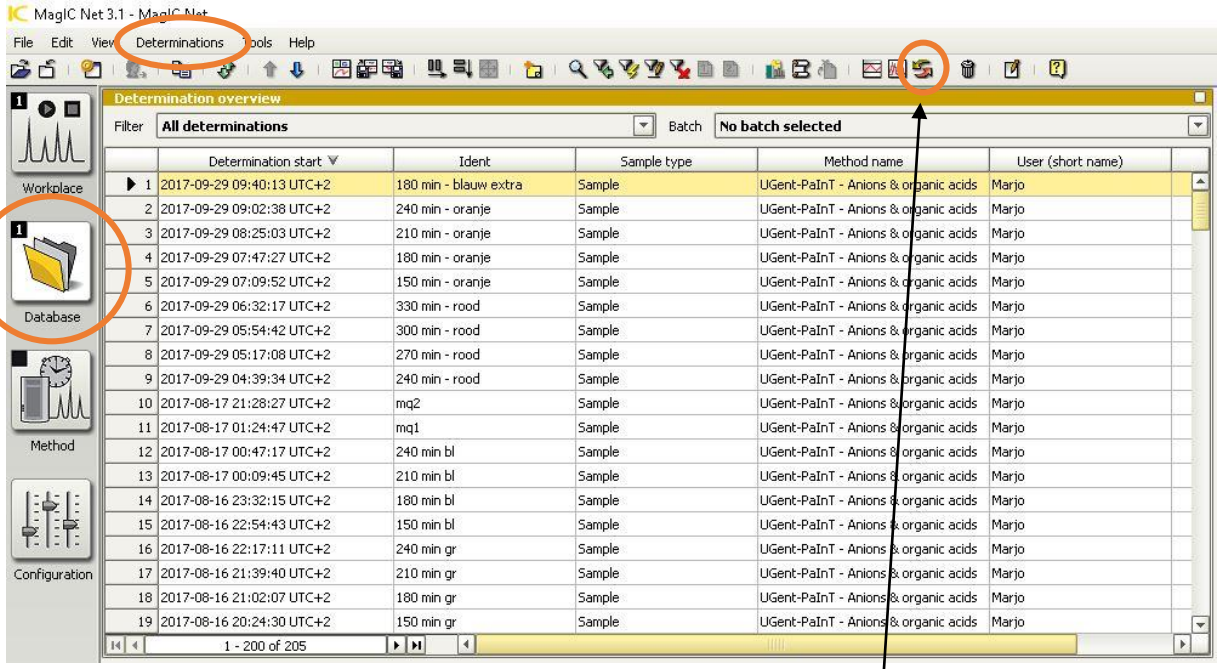
It is obliged to **run a test** on your table with 'Sample table - Run test…'. Make sure that there is a check mark at the box 'Stop hardware when the sample table is finished'! To start the analysis, press 'Start'.

Samples are analysed according to their sequence in the table not their position.

Put at least one blank sample (Milli Q sample) in front of your samples and put blank samples before and after a standard sample or a complete calibration series. Make your own standards or check if there are still available. In addition, put at least one standard in front of your samples to check the current calibration curve. If your standard is in line with the calibration curve, a new calibration is unnecessary. If not, run all standards after your samples in order to achieve a new calibration curve and reprocess everything afterwards on your **own PC**.

- **<u>Export/Import of data and reprocessing</u>**

First, check in which format you want to export the data. For the results, export as an excel file, but if reprocessing is required, export as an Idet file. Make sure the **Metrohm USB stick** is inserted in the PC. In 'Database', select the data you want to export. Then go to 'Determinations' and select 'Export…'. Choose the appropriate export template and click 'OK'.



Always reprocess on your own PC, so the original data are always available on the Metrohm PC!
Reprocessing is done at your own PC after installation of the MagicNet software (available on the USB stick). First, import the data in 'Database - Determinations' and select 'Import…'. Select the data which you want to reprocess and click on the reprocess button as shown above. In the new tab, you can assign a new retention time to a certain detected component peak (update retention time). A difference in retention time, compared to the calibration, can be due to concentration and matrix of the sample. For cations you only need to do this once, for the anions you need to do this twice, for the high and low calibration curve respectively. Under the known compounds you can also add other components of interest and their respective retention time. When this is done for one sample, click 'Update'. Next, you can reprocess all the other data by 'Reprocessing - from selected determination'. At last, you can save the reprocessed data by clicking on 'OK'.

For help with the reprocessing, ask one of the responsibles.

- **<u>Switch a column</u>**

A method is available to guide you through a switch. Go to 'Work place - determination series' and add a sample. This is not a real sample but this will start your guide for a column switch. As method choose the desired column switch method (from cation to anion or the opposite). Fill in the parameters, as explained for a normal sample. Press 'Start' to start the analysis and the switch guide will pop up. Follow the instructions in the exact order! If you never did a switch before, ask one of the responsibles to guide you through your first switch. Some of the handlings cannot be explained on paper…

Whenever you removed a column, close both sides of the column with end caps, in order to prevent the column to dry out during storage, and put it in the appropriate box. When opening and closing the column oven pay attention on the tubings, in order to prevent pinched tubes. With every switch you have a certain period of time you need to flush the IC with Milli Q, so never put cation eluent directly on an anion column or the opposite. Switching an anion column with another anion column which uses the same eluent can be done whitout the switch method.

Whenever you switch the eluent solution, **never touch the tubes** inside the bottles! In addition, always mention which kind of column is in use with the list on top of the IC.