# Metrohm Ion Chromatograph (IC): Quick Start Guide

*V1.1; L. Brandt; Approved 8/21/2013 (DJH)*
*362T; Approved July 2016 (DJH)*
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*369 : October 2017 (EM)*

## Reagents

Make sure all reservoirs are filled with the following reagents:

**Dosino Reservoir**:

18 mega-ohm DI water

**Suppressor Rinse Solution**:

0.1% methanol in DI water (1 mL methanol to 1 L DI water)

**Suppressor Regenerant Solution**:

0.05 M sulfuric acid solution (2.7 mL sulfuric acid to 1 L DI water)

**Anions Eluent**:

3.2 mM sodium carbonate/1.0 mM sodium bicarbonate solution (purchase concentrated packs from Metrohm, called "A Supp 5 Eluent Snips")

- snip off tube with scissors and pour contents into a 1 L volumetric flask
- rinse the tube with DI water, adding to the flask
- bring flask to volume

**Cations Eluent**:

1.7 mM nitric acid/1.7 mM PDCA (pyridine-2,6-dicarboxylic acid)

- dissolve 0.248 g PDCA in DI water in a 1 L flask
- add 106 µL concentrated trace metals grade nitric acid
- bring flask to volume with DI water.
- ❖ To make a carboy's worth, in a 2 L volumetric flask, mix 872 µL nitric acid, 2.272 g PDCA with DI water and bring to volume. This may involve using a stir plate with a stir bar in the flask. Pour into the carboy and add three more 2 L flasks filled with DI water.

## Setting up the instrument

1. Load a method (sets instrument's operational parameters)

   Select **METHOD** icon in left panel. Select *File -> Open ->* select the method. You can then save the method with a new name for the current Expedition (*File -> Save As*).

2. Make a new database for the Expedition (saves results in a central Expedition database)

   Select **DATABASE** icon in the left panel. Select *File -> Database Manager*. Select *Edit* pulldown then *New*. Type in a new name.

   Here you can also make a database backup. Select *Database Manager* again and **Backup**, giving a unique Expedition name.

Go back to the **METHOD** panel. In the **Evaluation** window, click on **Results** icon. Select the **Database** tab. Double-click on the *Name Database* field and select your database name for the Expedition.

3. Go back to the **WORKPLACE** icon in the left panel. Select the method that you want the instrument parameters to be set at (**Equilibrium** tab).

   Confirm all reagent reservoirs are filled, peristaltic pump tubes are in good shape and their platens are engaged.

   To start the instrument, select **Equilibration** tab > **green Start HW** button. To shut down the instrument, select the **red Stop HW** button.

   Monitor instrument conditions (**Watch** window) for ~ 60 min before starting a sequence.

   The anions baseline should be ~ 1 µS/cm. Suppressor may cause a small peak every 10 min or so. The anion pump pressure should be around 6.5 – 7.0 MPa.

   The cations baseline should be ~ 870 µS/cm. The cation pump pressure should be around 4 – 4.5 Mpa.

   The column thermostat display appears red until reaching 45°C. Allow the columns to come to temperature before starting sequence.

## Preparing the Calibration Standards and Samples

All samples are diluted 1:100 with DI water before analysis.

Prepare a standard curve, 10 mL per level, in IC vials:

| | **standard 1 (DF 100)** | **standard 2 (DF 150)** | **standard 3 (DF 200)** | **standard 4 (DF 350)** | **standard 6 (DF 67)** | **standard 7 (DF50)** |
|---|---|---|---|---|---|---|
| **IAPSO (µL)** | 100 | 66.7 | 50 | 28.6 | 125 | 150 |
| **DI water (µL)** | 9900 | 9933.3 | 9950 | 9971.4 | 9875 | 9850 |

Make sure that the vials are vortexed before analysis.

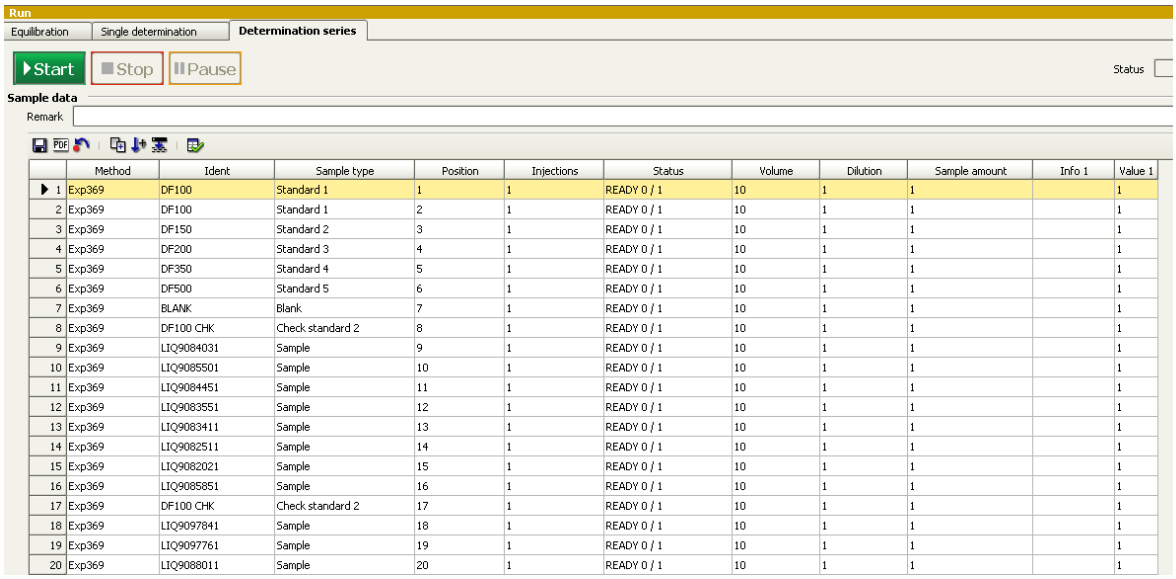
To add/edit standards, select **METHOD** panel and **Standards** in the **Evaluation** window. Here you can also add/edit *check standards* information.

## Setting up a Sequence

1. To set up a sequence, select the **WORKPLACE** panel and **Determination Series** tab.
2. To create a new sample table, use the dropdown menu **Sample Table** -> **New**.

   Go to **Sample Table** drop-down and select **Properties**. In the **Display** tab, make sure that the *Value 1* radio button is checked. Now go to the **Edit** tab and make sure that the *Value 1* radio button is checked.

   Double click the first row to start populating the sample/sequence table. The table should follow a basic guideline as follows (see Figure 1). The *calibration standards* will come first, followed by the *blank* (DI used as a sample), a *check standard* and then the samples with *checks* every ten samples or so.




| | Method | Ident | Sample type | Position | Injections | Status | Volume | Dilution | Sample amount | Info 1 | Value 1 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | Exp369 | DF100 | Standard 1 | 1 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 2 | Exp369 | DF100 | Standard 1 | 2 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 3 | Exp369 | DF150 | Standard 2 | 3 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 4 | Exp369 | DF200 | Standard 3 | 4 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 5 | Exp369 | DF350 | Standard 4 | 5 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 6 | Exp369 | DF500 | Standard 5 | 6 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 7 | Exp369 | BLANK | Blank | 7 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 8 | Exp369 | DF100 CHK | Check standard 2 | 8 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 9 | Exp369 | LIQ9084031 | Sample | 9 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 10 | Exp369 | LIQ9085501 | Sample | 10 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 11 | Exp369 | LIQ9084451 | Sample | 11 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 12 | Exp369 | LIQ9083551 | Sample | 12 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 13 | Exp369 | LIQ9083411 | Sample | 13 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 14 | Exp369 | LIQ9082511 | Sample | 14 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 15 | Exp369 | LIQ9082021 | Sample | 15 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 16 | Exp369 | LIQ9085851 | Sample | 16 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 17 | Exp369 | DF100 CHK | Check standard 2 | 17 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 18 | Exp369 | LIQ9097841 | Sample | 18 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 19 | Exp369 | LIQ9097761 | Sample | 19 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |
| 20 | Exp369 | LIQ9088011 | Sample | 20 | 1 | READY 0 / 1 | 10 | 1 | 1 | | 1 |

Figure 1 : Sample/sequence table example

**Method** : *369 (expedition number)*

**Ident** : Text_ID of the sample. Standards are not yet entered into the LIMS so use something like *DF100* or *DF100 CHECK* (for a check standard).

**Sample Type** : *Sample*, *Standard 1–5*, *Blank* or *Check Standard*

**Position** : Autosampler vial position

**Injections** : 1

**Volume** : 10 µL

**Dilution** : Select "1." Dilution is supposed to be the dilution factor for manual or hand dilutions performed by the analyst prior to the sample being placed in the system; however, the standards are built around 1:100 being baseline, so we don't want the software to calculate dilutions.

**Sample Amount** : Should be "1."

**Info 1** : Here you can enter a comment or *Label ID* for example.

**Value 1** : Dosino Dilution Factor, the dilution factor performed by the automated dilution system. For hand dilution, set this to "1."

3. After the sample/sequence table is complete, go to the **Sample Table** pull-down, select Save As, and give a name.
4. Place the vials in the appropriate chamber according to the sequence table.
5. Click on the **Start** button to start the run. A display of the current aspiration will be shown on the Live Display Window.

## Evaluating the Calibration/Results

Viewing the calibration:

In the ***Database*** panel, select a result of interest, in the **Determination Overview** window. In the **Curves 1** window, are tabs for **Anions** and **Cations**. Select either, and then click on the **Calibration curve** radio button. Select the element to evaluate from the pull-down menu.

Viewing results:

In the same ***Database*** panel, select a result of interest, in the **Determination Overview** window. In the **Curves 1** window, are tabs for **Anions** and **Cations**. Select either, and then click on the **Chromatogram** radio button. You can view the chromatogram, zoom in to check integrations, etc. The results, in a tabular text format, will be in the **Results** window.

## Uploading Data to LIMS

Use MUT to upload results to the LIMS.

1. In the ***Database*** panel, select the result(s) to upload. Either click on a row or hold down the <Ctrl>-key and select multiple results.
2. In the top menu, select **Determinations -> Export**. Select **All Selected Data Records** and export template *Exp368P*. Click **OK**. The Excel csv files will be created in *C:\Metrohm Export*.
3. Copy these csv files to the MUT uploader directory (C:\DATA\in).

   Run MUT.
4. Move the csv files in *C:\Metrohm Export* to an Expedition folder and at the end of the Expedition copy over this folder to the DATA1 volume for backup for the shore db.

## Helpful Hints

Sometimes the autosampler ends at a position where some vials cannot be accessed. To get the autosampler to move, select the ***Manual*** tab on the main panel, then select **858 Professional Sample Processor**. Select the **Tower** tab, change the rack position input, then click **Start**.

The business card for the Service rep is taped to the side of the instrument**.**

The help function in the software is good. Accessed from the top menu.

## Maintenance

For maintenance, there are a couple of great guides, located in the Metrohm_backup*METROHM MANUALS* folder on the desktop.

| | |
|---|---|
| 850_IC.pdf | main instrument guide |
| IC_maintenance_guide.pdf | Outlines maintenance schedules to follow |
| | : changing autosampler ultra-filtration membrane |
| | : changing $H_2O$ and $CO_2$ scrubbers |
| | : replacing peristaltic pump tubing |
| | : changing the guard/inline filters |
| | : pump maintenance |
| Autosampler.pdf | autosampler guide |
| Dosino.pdf | Dosino guide |
| 850_pump_maintenance.pdf | anion/cation pump maintenance |
| Metrohm_parts_guide.pdf | Parts information to send in requisitions |
| Metrohm_ultra_filtration.pdf | More on the autosampler's ultra-filtration |
| MagICNet_tutorial.pdf | Software guide |