# Metrohm Ion Chromatograph Quick Start Guide



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## Introduction

## Preparing Reagents, Standards and Samples

### Reagents

**Dosino Reservoir**:

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

- Metrohm “A Supp 5 Eluent Snips”
  - 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.
- Add three more 2 L flasks filled with DI water.

## Standards

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 80) | standard 7 DF(67) | Standard 8 (DF1000) (Pipette from DF100) | Standard 9 (DF2000) (Pipette From DF100) | Standard 10 (Pipette From DF100) | Standard 11 (Pipette From DF100) |
|---|---|---|---|---|---|---|---|---|---|---|
| **IAPSO (µL)** | 100 | 66.7 | 50 | 28.6 | 125 | 150 | 1000 | 500 | 100 | 50 |
| **DI water (µL)** | 9900 | 9933.3 | 9950 | 9971.4 | 9875 | 9850 | 9000 | 9500 | 9900 | 9950 |

- Make sure that the vials are vortexed before analysis.

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

## Samples

All samples are diluted 1:100 with DI water.

- Make sure that the vials are vortexed before analysis

## Check Standards

- Prepare 10 check standards of DF100 by making 1 mL of IAPSO up to 100 mL with DI water in a 100 mL volumetric flask.
- Shake the flask, then decant the solution into 10 separate IC vials.

# Instrument Operation

## Startup

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).

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

5. To start the instrument, select **Equilibration** tab > **Start HW** button.

   To shut down the instrument, select the **Stop HW** button.

6. 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.

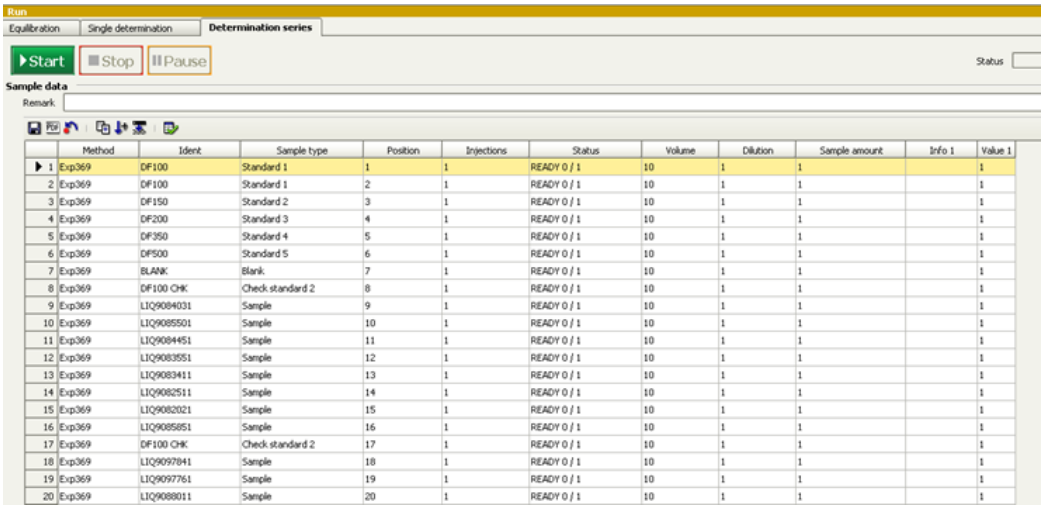
## Sequences

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.
    - Then the samples.
    - 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*

From Figure 1:

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

**Value** : 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 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.

## LIMS

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.

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**.

Copy these csv files to the MUT uploader directory (**C:\DATA\in**).

Run MUT to upload the results to the LIMS.

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.

## 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 |

## 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 o n 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.

# Credits

This document originated from Word document IC_QSG_369_draft.docx (see Archived Versions below for a pdf copy) that was written by L. Brandt (2013-21-08) and edited by E. Moortgat (2017-10). Credits for subsequent changes to this document are given in the page history.

# Archived Versions

IC_QSG_369_draft.pdf