
Standard Operating Procedure

Metrohm 930 Ion Chromatorgraph (Anion & Cation Detection)

1. Objective

- a. The following document describes the general procedure to operate the Metrohm 930 Compact IC.

2. Scope

- a. This procedure applies to the setup, analysis, and evaluation of anions and/or cations in aqueous solution using the Metrohm 930 Compact IC.

3. Responsibility

- a. It is the responsibility of the TRACES user to follow the instructions & ensure adherence to this procedure and the TRACES User Protocol
- b. It is the responsibility of the TRACES user to contact the TRACES Staff if:
 - i. Deviation from the procedure is required
 - ii. Changes to the eluent, suppression solution, detector or IC column is necessary

4. Reference

- a. Metrohm 930 Compact IC Flex Manual (8.930.8020EN)
- b. Metrohm MagIC Net 3.2 Online Manual (8.102.8077EN)
- c. Metrohm MagIC Net 3.2 Tutorial (8.102.8001EN)

IC SOP Control Section

5. Instrument*

a. Metrohm 930 Compact IC

- i. Detectors Available
 1. Conductivity (in use for Anion and Cation analysis)
 2. UV-Vis
 3. Amperometric
- ii. Columns
 1. Anion (Metrosep A Supp 5: 4.0 x 150mm)
 2. Cation (Metrosep C 6: 4.0 x 150 mm)

6. Before Getting Started

Contact the TRACES Staff to ensure that the IC has been setup for Automated or Manual analysis. Communicate to the staff your desired eluent composition, volume, AND the IC column for the analysis of choice.

IC Setup Section


1. Initial Setup

- a. Inspect the IC to ensure that the desired eluent composition, volume, and IC Column are installed on the system.

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- i. Volume of eluent required can be estimated from the run time of each method multiplied by the number of samples/blanks/standards multiplied by a factor of X 1.2
- b. Ensure the IC waste bottle (located below the bench top) is $\frac{3}{4}$ empty

2. Instrument & Computer


- a. Turn on the IC power, the LED should flicker on/off
- b. Login onto the computer with the appropriate password and username
 - i. Click MagIC.Net located on the desktop or the taskbar 
 - ii. Keep clear of the autosampler (A/S) and instrument
 - iii. Allow instrument 5 min to communicate and run the diagnostics
 - iv. The A/S should automatically run its diagnostics and setup at Vial#1
 - v. IC power LED should be solid green (Figure 1)

IC Analysis Run Section

1. Select Workplace Method

- a. **Click the Workplace icon**
- b. Select the Method of choice from the dropdown box (Figure 2)
- c. If creating a new Method, please select a previously used Method and save it following the previous naming convention (Lab/Course_Anion/Cation_Auto/Manual)
 - i. Otherwise see the TRACES Staff

2. Start Method/Purge Eluent



- a. Click the 
- b. System will start up slowly
- c. Monitor the Watch Window (Figure 3) and ensure that the flow, oven and pressure are increasing and the conductivity is decreasing (Anion) or increasing (Cation)
- d. Connect the available 10ml syringe to the Purge Valve Hosing (Figure 4)
- e. Click on the Manual Icon on the left (Figure 5)
- f. Select Pump
 - i. Enter flow rate and Click Start
 - ii. Open the Purge Valve $\frac{1}{2}$ way to the left and pull back syringe to about 1-2ml. Allow the syringe to continue filling to about 10ml (Figure 4)
- g. Select Peristaltic (Anion Analysis ONLY!)
 - i. Increase speed from 3 to 6 for the length of time you have the Purge Valve opened
 - ii. Close the Purge Valve to the right till finger tight-pressure should begin to rise
 - iii. Reduce the Peristaltic Pump speed back to 3
- h. Allow the instrument to equilibrate for minimum of 15 -30 min

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3. Load Autosampler

- a. Begin loading the samples starting at the lowest possible A/S
 - i. 15 ml conical tubes are acceptable sample vials (i.e Falcon)
 - ii. CHECK THAT ALL THE 15ml CONICAL TUBES ARE SITTING FIRMLY IN THE LOCATION
 - iii. CHECK THAT THE TOP OF THE CONICAL TUBES DO NOT INTERFERE WITH THE A/S NEEDLE (Figure 6)
 - iv. Use A/S Location 1-83 (when running an Smart Dilution Experiment*)
 - v. Use A/S Location 1-149 (all other runs)
- b. Please recheck the location of each standard and sample BEFORE entering them

4. Setup IC Analysis Run*

- a. Final Checks using the Watch Window
 - i. Check that the oven has reached operational temperature (no longer red)
 - ii. Check that the conductivity readings are stable for several minutes (+/- 5%)
 1. Otherwise see the TRACES Staff
- b. Select Workplace icon and select Determination Series
 - i. Remove all previous samples (if they are already completed)
 - ii. Right Click Mouse (RCM) in an empty field and select Edit Line
 - iii. Select the Method (should be similar to the method you have selected when you started
 - iv. Introduce your sample name/id in the Ident
 - v. Select the type of sample Blank/Sample (Calib/Standards are for advanced users)
 - vi. Ensure the A/S location for each Blank/Sample matches the Pos number
 - vii. Injections=1(minimum 6ml/analysis)
 - viii. Volume=20uL (instrument injection loop)
 - ix. Initial Commands: Initial Rinse (for first blank run of the day); empty for samples
 - x. Instrument Dilution=1 (no instrument dilution)
 - xi. Auto-Vial=84 (all runs other than Smart Dilution Experiment*)
 - xii. Batch Name: naming convention= Course/PI_YYYYMMDD_An/Cat (Figure 7)
 - xiii. Apply
 - xiv. Subsequent samples can be sequentially added (Figure 7); Apply after each addition
- c. Once the entire sample table has been entered, check the Stop Hardware When Sample Table is finished. This will turn the system down once the last analysis has completed (please see Shutdown)
- d. **Select the Sample Table Test. Return message MUST read No errors found.** 
- e. In the Determination Series window select  to run the analysis
- f. Do not interfere with the A/S when it is moving-wait till the sample has injected and the analysis has started

IC Data Evaluation Section

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1. Selecting Files for Reprocessing-The Easy Way

- a. Click the Database icon
- b. Select the appropriate Batch Name to limit your search
- c. Select the file(s) you wish to reprocess
- d. Click on Determinations

- e. Select Reprocess ...

2. Integration*

- a. Settings: leave as default
- b. Peak Detection: Automated
 - i. Minimum Height limit for detection:0.0001uS/cm
 - ii. Minimum Area limit for detection:0.0001(uS/cm) x min
 - iii. Integration Start: Set after the delay time (~2.5 min)
 - iv. Savitzky-Golay Filter available for smoothing
- c. Please see 4. Reprocess Data below for reprocessing procedure

3. Components

- a. Component Table: If necessary, retention time and component name are entered/changed
- b. Peak Detection: Manual
 - i. Right Click Mouse: Add peak
 - ii. Select the area you wish to detect as a peak
 1. Peak in question MUST meet the Minimum limits (2. Integration...)
 2. Rename the peak in the Component Table
- c. Identification: Reference Components: Height, Time, Area, can be selected
- d. Please see 4. Reprocess Data below for reprocessing procedure

4. Reprocess Data*

- a. Update
- b. Processing
- c. OK

IC Data Printing/Exporting Section

1. Print Analysis Report*


- a. Select Print Report
- b. Click on Selected Determinations
- c. Upload D16_results1 as the Report Template
 - i. This is a default report template
- d. Select pdf as the output file
- e. Select the Target Directory and use the Ident for Sample Identification
- f. Ok
- g. Loading a Report Template into the method will automatically print reports after each analysis

2. Exporting Results as CSV*

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- a. Click the Database icon
- b. Select the file(s) to process
- c. Click on Determinations
- d. Export...
- e. Click on selected data records
- f. Export Templates
 - i. CSV: Time vs Conductivity
 1. csv_data_points (csv will be saved to the desktop folder 'CSV_Data_Points')
 - a. Name the file accordingly
 2. OK
 - ii. CSV: Area results for each analyte of interest
 1. Please see TRACES Staff for a method specific template
 2. csv_area_anion_all (csv will be saved to the desktop folder 'CSV_Area_Data')
 3. csv_area_cation_all (csv will be saved to the desktop folder 'CSV_Area_Data')
 - a. Name the file accordingly
 4. OK

IC Shutdown Section

1. **Automated Shutdown**
 - a. Shutdown using the Determination Series please see IC Analysis Run, Section 4 subsection c
2. **Manual Shutdown**
 - a. Click the Workplace icon
 - b. Click the 
3. Remove all samples, standards and other items that are not TRACES property

Figures



Figure 1

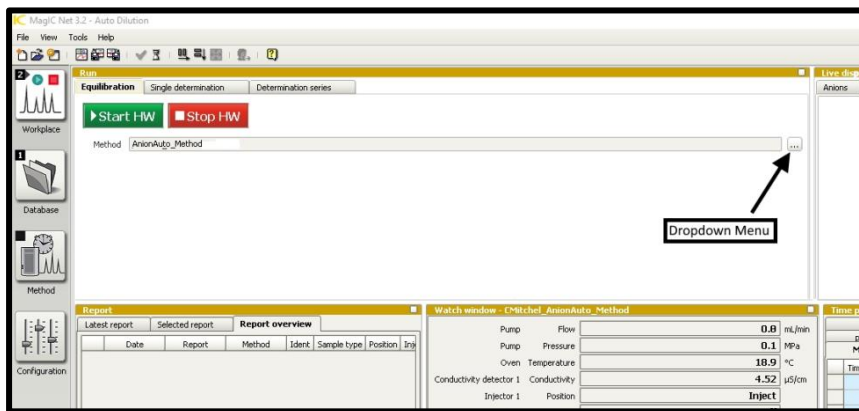


Figure 2

Standard Operating Procedure

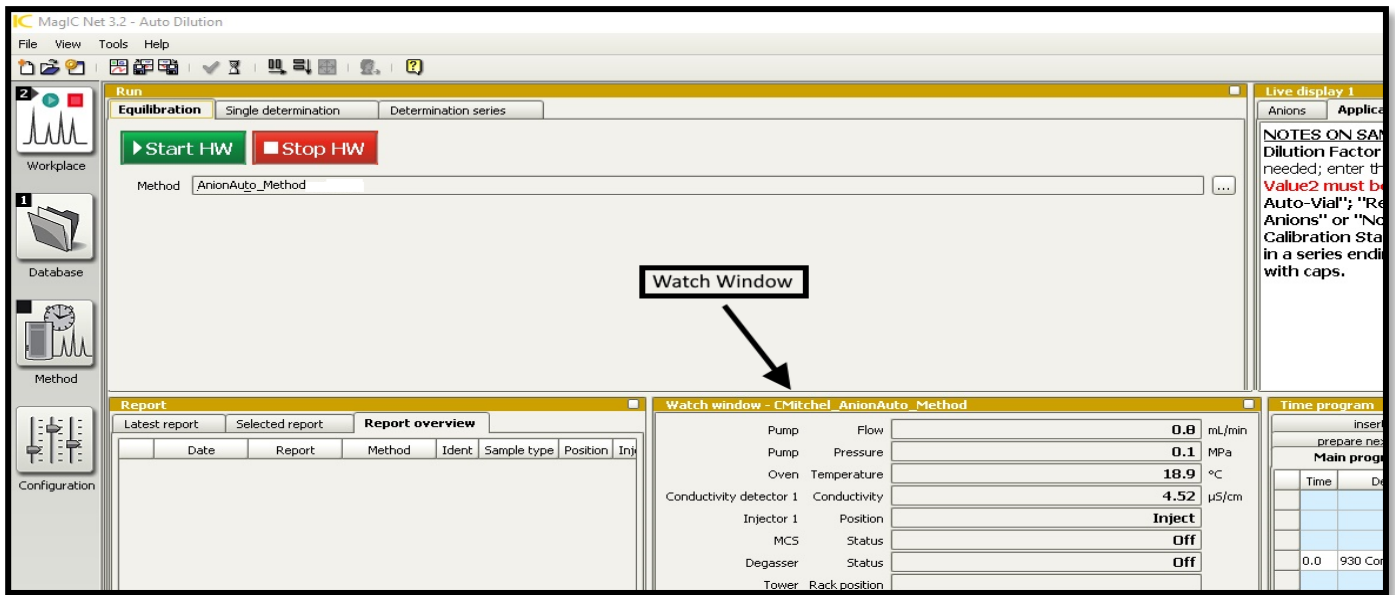


Figure 3



Figure 4

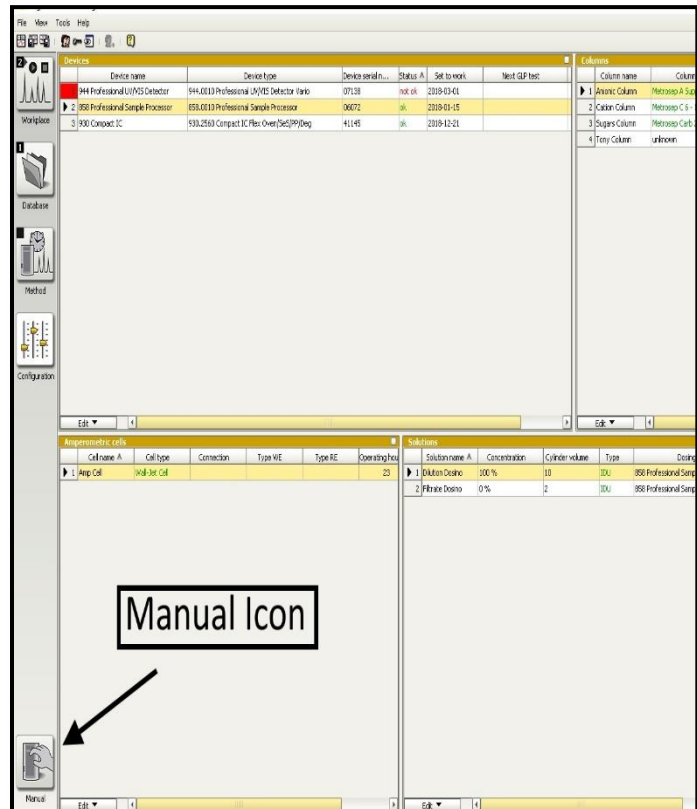


Figure 5

Standard Operating Procedure

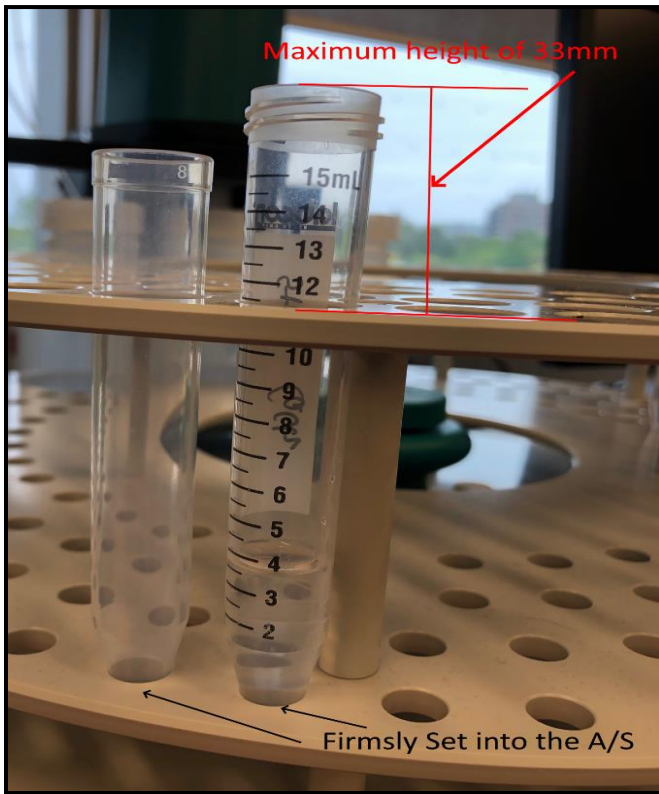


Figure 6

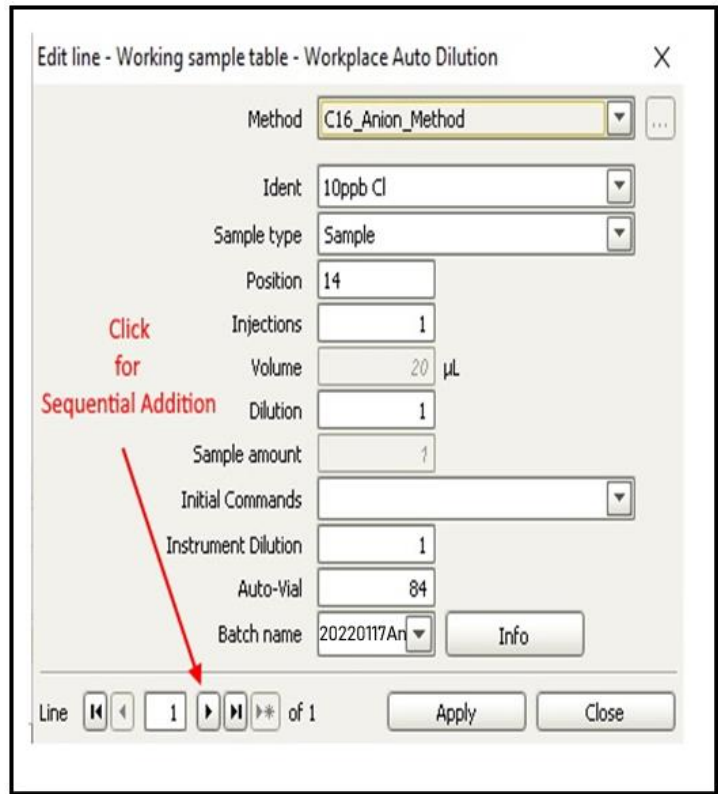


Figure 7

***The TRACES Manager will provide full details during hands-on training.**