Xi'an Jiaotong-Liverpool University
西交利物浦大学

General Operating Procedure for:

ICS-1100 for inorganic anions

Department of Health and Environmental Sciences

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Contents

1. Purpose

The purpose of this GOP is to regulate the operation, usage and maintenance of ICS-1100

2. Scope

This GOP applies to all personnel using and managing ICS-1100.

3. Procedure

3.1 Switching on the machine

3.1.1 Check the eluent. It should be replaced if the color change or some sediment appeared. The fresh prepared one can't be mixed with the old eluent.

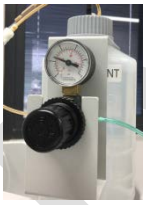
Currently the eluent we used (for column AS22) is 4.5mmol/L Na2CO3 and 0.8mmol/L NaHCO3. (0.9540g Na2CO3 and 0.1344g NaHCO3 into 2L ultrapure water. Use Sigma's chemicals.)

3.1.2 The room temperature should be 20-25ºC and the humidity should be less than 80%.

3.1.3 Open the valve for N2 gas cylinder, contact Yili or Xiao if it is less than 3MPa.

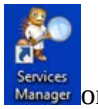
3.1.4 Open the valve at the pressure reducer of the gas supply and make sure the preliminary pressure is less than 5 bar. (2 bar is recommended.)

3.1.5 Check the pressure of the eluent, it should be less than 10Psi. Normally 4psi. If not, contact Xiao or Yili.

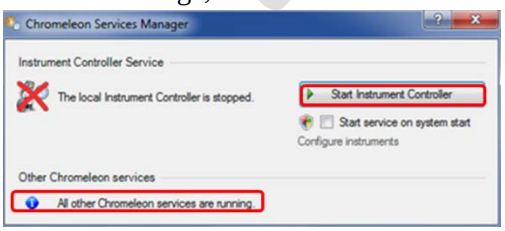


3.1.6 Tap the eluent bottle to remove any bubble on the bottle wall.

3.1.7 Switch on the analyzer, sampler and the computer.

3.1.8 Click on the services manager 

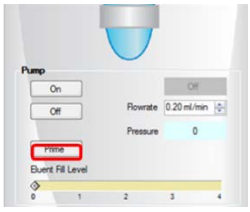
on the desktop, wait till it shows "all other Chromeleon services are running", then click "start instrument controller".



3.1.9 Click on “Chromeleon 7” 

to start the software.

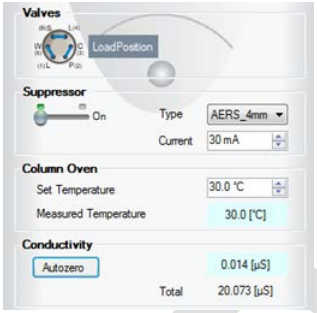
3.1.10 Open the waste valve, then click “prime” on the software. Click “ok” for the reminder of opening the waste valve. The pressure should be “0” during the prime. At least 5 minutes is needed for new eluent.



3.1.11 Click “off” under the pump and close the waste valve.

3.1.12 Click “on” under the pump and increase the flowrate gradually. The working flowrate should be 1.0ml/min. It is set only after you have press “enter” on the keyboard.

3.1.13 Click the scrollbar under “Suppressor” to open the suppressor. Adjust the current to 30 mA.

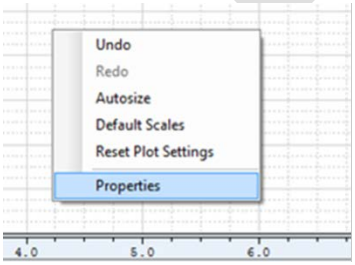


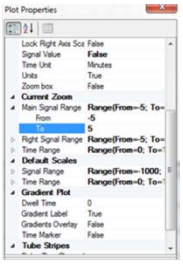
3.1.14 Record the time and conductivity in the logbook. (It should be about 20µs for the eluent we currently use. If the stable value is larger, your eluent may have been contaminated.)

3.1.1-3.1.14 is also the SOP for weekly maintenance. It should be down each week to protect the suppressor and column.

3.1.15 Click “monitor baseline” to collect the baseline.

3.1.16 Right click the mouse in the plot zone. And choose “properties” in the option window. You can change the signal range in the “plot properties” window.



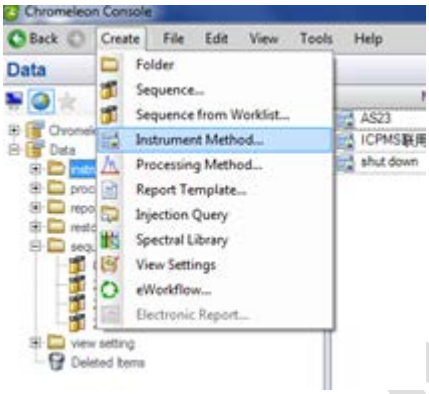


3.1.17 Click “stop” to stop monitoring the baseline when it is stable.

3.2 Create new method and sequence.

3.2.1 Click data.

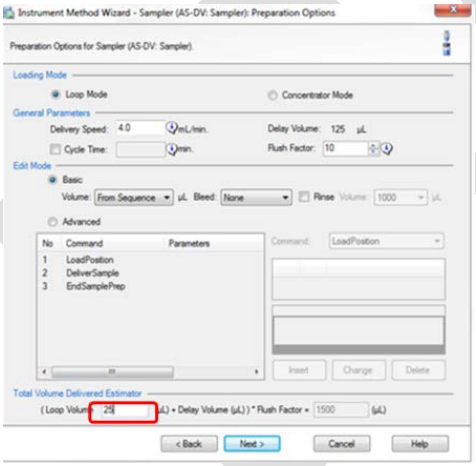
3.2.2 Create instrument method.



3.2.3 Run time, 30 min recommended.

3.2.4 Pressure limit: 200-3000.

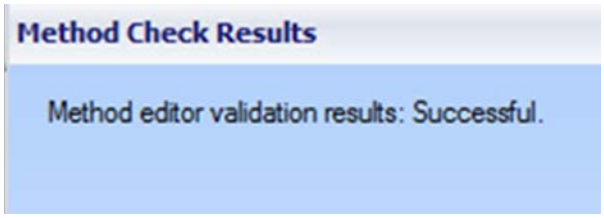
3.2.5 Loading mode: loop mode. Loop volume is 25µL currently.



3.2.6 Channels: ECD_1, cell temperature: 35ºC and column temperature: 30 ºC.

3.2.7 Suppressor type: ASRS_4mm. Set the eluent concentration: carbonate 4.5mM and bicarbonate 0.8mM.Click “next”. Click “finish” in next window.

3.2.8 Click “check method”, it will show the check results.

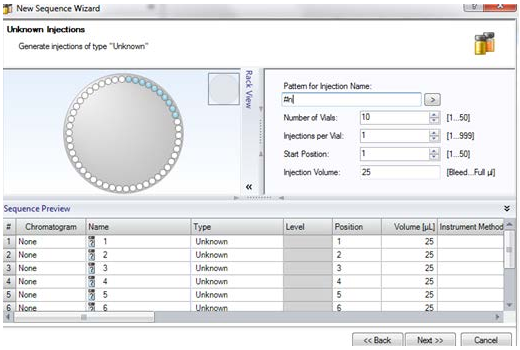


3.2.9 Save and name the instrument method.

3.2.10 For processing method and report, we can use the existed one.

3.2.11 Click “create” and choose “sequence” in the pull-down menu.

3.2.12 Set injection name, number of vials and so on, as the picture show. Then click next.

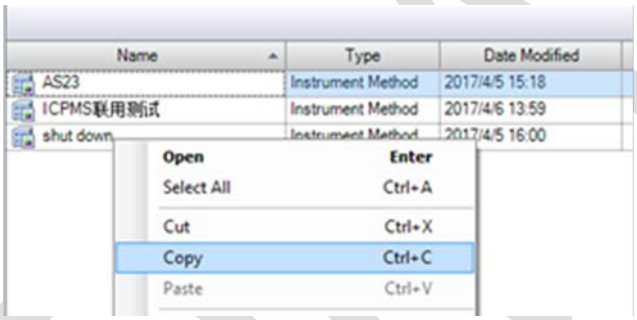


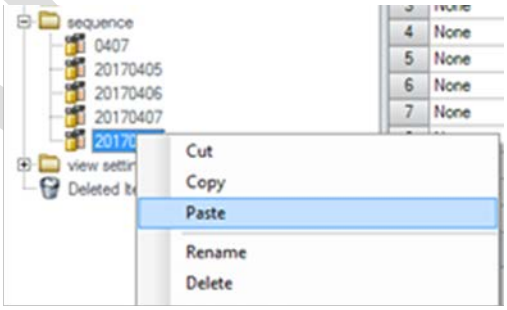
3.2.13 Choose instrument method, processing method and report template. Following subsequent guidance.

3.2.14 Set “type” for samples. For calibration standard, you should also choose level.

3.2.15 Name and save sequence.

3.2.16 Auto shut down: click instrument method, right click on “shut down” and choose copy. Right click on the sequence and choose “paste”. Then you can add a new injection and choose “shut down” as the instrument method.

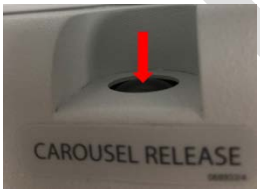




3.3 Loading sample and start sequence

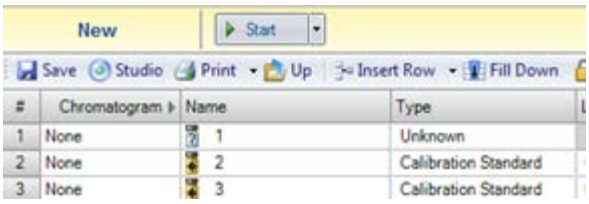
3.3.1 Put samples into the sampling bottle to full scale line and make sure there is no bubble at the bottom. Put the cap to certain depth; shake to move the redundant solution.

3.3.2 Open the cap of auto sampler and press the carousel release button, then the carousel light is off and you can rotate the sample plate and load your samples.



3.3.3 Click the carousel release button again to lock the sample plate.

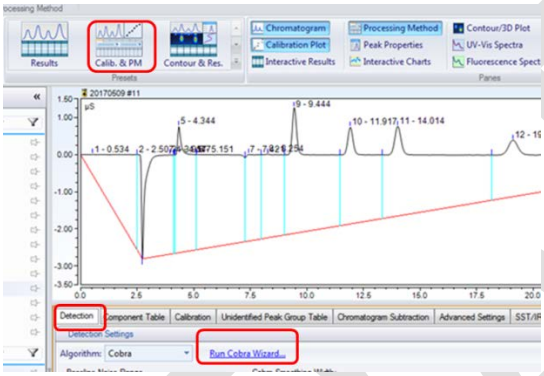
3.3.4 Click “start” to start sequence.



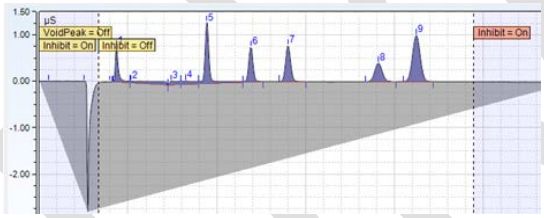
3.4 Date reprocess

3.4.1 Click “data” and double click the first standard to open this injection in the chromatograph studio.

3.4.2 Click “calib.& PM”, “detection”, then click “run cobra wizard”.



3.4.3 Click in the chromatogram and drag the mouse to pick the integration area. Then click “next”.



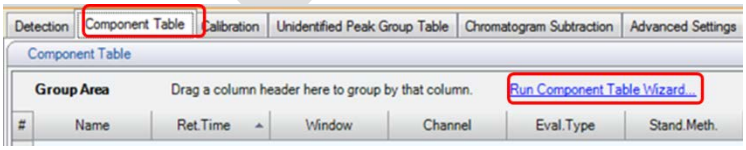
3.4.4 Click “next”.

3.4.5 Set “smoothing witch” and click “next”.

3.4.6 Set “minimum area” and click “next”.

3.4.7 Click “finish”.

3.4.8 Click “component table” and then click “run component table wizard”.



3.4.9 “Next”, “next”, “finish”.

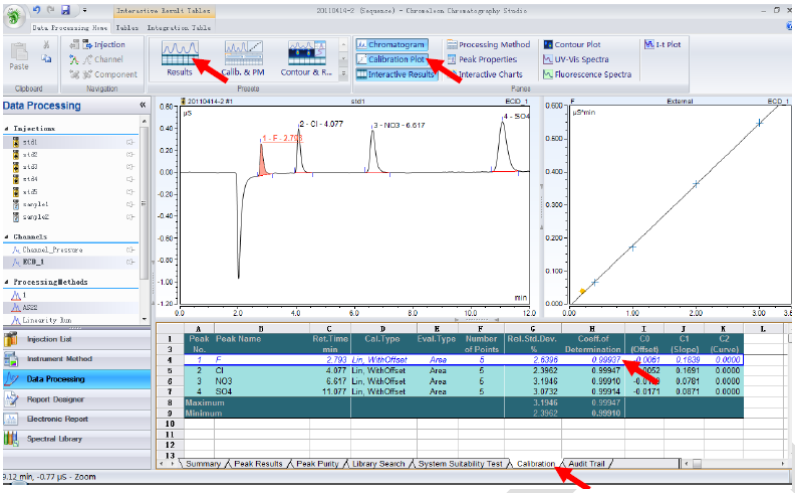
3.4.10 Set “name” and concentration for standards.

3.4.11 Double click “window” to set retention time.

3.4.12 Double click “Cal. Type” and choose “ignore origin (with offset).

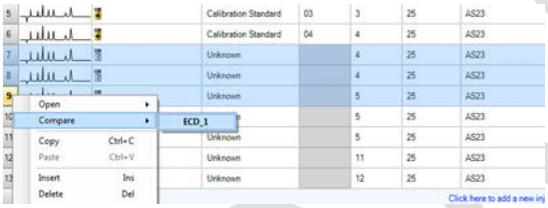
3.4.13 Save.

3.4.14 Click “results” and “calibration plot” to see the calibration curve. Click “calibration” to see R^2 (Coeff. Of determination), C0 (offset) and C1 (slop).

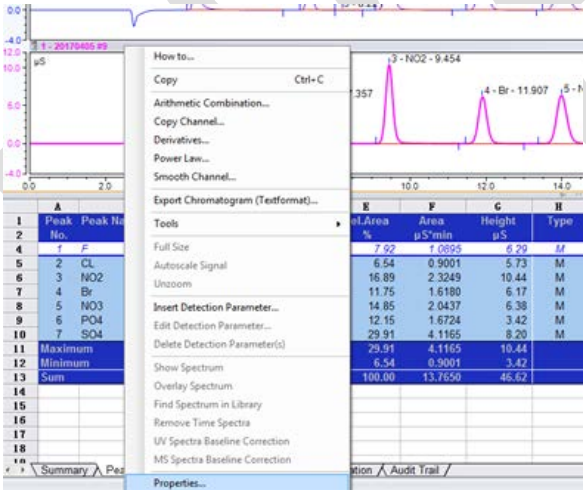


3.4.15 Click “summary” or “peak results” to see the amount.

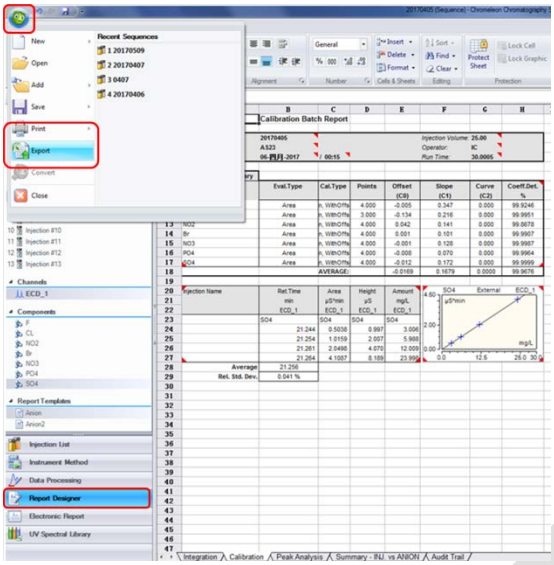
3.4.16 For parallel samples, select them, right click and choose “compare”.



3.4.17 Right click and choose “properties”, click on “comparison” then you can choose to compare them in different arrangement or offset.

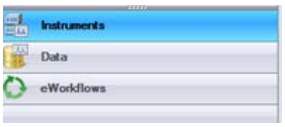


3.4.18 Click “report designer”, then click  , chose export or print to export your data.



3.5 Shut down

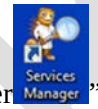
3.5.1 Click instruments.



3.5.2 Turn off suppressor.

3.5.3 Turn off pump.

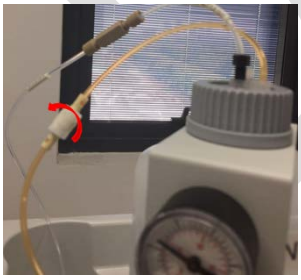
3.5.4 Close the software.

3.5.5 Click “services manager 

” and click “stop instrument controller”.

3.5.6 Close the analyzer, auto sampler, gas and computer.

3.5.7 Release the residual gas and then close it again.



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