Directions for running IC samples on the Shimadzu TOC analyzer. (5/18/2015 DER)

Turn on the TOC analyzer by pressing the on switch located in the lower left corner of the panel on the front of the instrument. It will take at least 30 minutes for the instrument to warm up to temperature and stabilize.

Open the "TOC-L sample table editor" by double clicking the shortcut on the desktop. User name and password are both blank.

Open a new sample table by clicking the "New" button near the upper left corner of the window. The system should be "TOC-TN" for internal sparging and "toc-tn2" for external sparging. Press "OK".

Press the "connect" button near the center of the tool bar. A status window should open with a message like" open port 100 %" then "initializing".

Set up calibration curves

In the sample table editor, select the cal curve tab and pres the "New" button.

The System should be "TOC-TN". Leave the user name blank. Add comments as desired and press "next".

Select "normal" and "use dilution from standard solution". Press "Next".

Select IC for the analysis.. The calculation method should be "Linear Regression", the "zero shift" should be off and "Multiple Injections" should be on. Enter a file name; the name should include the full date, a person or project name, the concentration of the top standard, the analysis type. The preferred format for all file names is: "2013 May 30 project analysis" Press "Next".

The standard parameters for DIC are:

Units: mg/l

Injections: 2/3

No. of washes: 2

SD max: 0.1

CV max: 2%

Press "Next"

In the calibration points table, click on "add". The first calibration point is usually 0 ppm so the "Standard Solution Conc." and the "Cal. Point Conc." should both be 0. The "no. of Injections" should be 2/3, SD Max =0.1 and the CV Max=2% for all calibration points. Press "OK" to enter the point into the standard curve.

Press “Add” for more calibration points. Set the value of the “Standard Solution Conc.” to the value of the highest standard and the “Cal. Point Conc.” should be set to the desired value of that point. Press “OK” to enter the point into the standard curve.

Cal curves should have 5 points ranging from 10 % to 100% of the highest standard plus a zero. Curves should be set up starting with the lowest concentration and ending with the highest. Note the Injection volume after entering the highest standard. Normally, 100ul is used for DIC measurements.

Press “Next” after all calibration points have been entered

The “Use Default Setting” box should be checked and the “correlation Coeff. Check” box should not be checked.

Press “Finish” to complete the standard curve.

Create the second calibration curve. The standard practice is to create a calibration curve with a top standard of 10 mg/l as the first calibration curve and a second curve with a top standard of 50 mg/l as the second. If a sample exceeds the 10 mg/l concentration of the first curve, the instrument will automatically jump to the second curve. This minimizes the number of sample needing to be rerun.

After completing the high and low calibration curves, set up a method

Set up a method

Click on the method tab in the lower left corner of the software window, then press “New” button.

The System should be “TOC-TN”. Leave the user name blank. Add comments as desired and press “Next”.

Select IC for the analysis. Enter the default sample name and ID you want (leaving it blank is fine). Manual dilution is 1 and number of determinations is 1. Enter a file name, please include the date and project or person name. The preferred format for all file names is: “ 2013 may 30 project analysis” Press “Next”.

Select the first desired calibration curve which should be your low concentration standard curve as calibration curve 1. Then select the next highest calibration curve as curve 2. Then press “Next”.

The standard parameters for IC are:

Units: mg/l

Injections: 2/3

No. of washes: 2

SD max: 0.1

CV max: 2%

"Multiple Injections" should be selected. Press "Next".

"Use Default Settings" should be selected. Press "Next".

On the "Pharmaceutical Water Testing" page, "None" should be selected.

Press "Finish" to save the method.

Set up a sample table

If there is not a new sample table open already, open a new sample table by clicking the "New" button near the upper left corner of the window. The system should be "TOC-TN" .

Press "OK".

Press "save" to save the sample table. File name should follow the format: "yyyy mon dd project".

To add samples to the table, highlight the line in the table and right click. Choose "insert multiple samples". Use the browse button to find and select the method to use to create the sample parameters, then hit "next". Type in or select the number of samples and the starting vial number. Then hit "finish". A list of the samples and vial numbers will appear along with a diagram of the sampler. Verify that the samples and vial numbers are correct. Make sure the vial type in the lower right corner is "40 ml". Press "OK" to close the diagram window.

To add a standard curve to the table, highlight the line in the table and right click. Select "insert standard curve". Highlight the desire standard curve and press "Open". Check that the vial numbers for the curve are correct. The vial number is in the last column on the right of the sample table. To edit the vial numbers, click the sampler diagram icon in the upper right of the sample table window. A list of the sample/standards and vial number will appear along with a diagram of the sampler. Edit the vial numbers as needed then press "OK".

Repeat the above step for the second(high) standard curve.

Save the sample table when it is complete.

The general setup for a sample run is:

3 samples of DI water at the beginning of the run to prime the instrument.

The low concentration calibration curve.

The high concentration calibration curve.

A DI water and a high and low check standard to verify the calibrations.

10 samples.

A DI water and a check standard.

Repeat the 10 samples followed by a DI water and a check standard to the end of the run. Alternate between the high and low check standards. At the end of the samples, run a DI and both the high and low check standards.

Other notes on sample table setup.

If vial number 0 is enter for a sample or standard, the instrument will sample from the tube on the left side of the instrument next to the lines going to the autosampler. This tube may be placed in a flask or bottle. It is commonly used for the calibration standard as it can be sampled many times.

The auto sampler is random access, meaning it can sample the numbered vials in any order and can sample a vial multiple times. This is useful for check standards and DI waters as the same vial may be sampled up to three times.

A vial filled to the shoulder may be sampled up to 3 times.

Standard Preparation

The excel file: "standard concentration calcultaionsV2" on the desktop of the instrument computer contains a list of commonly used calibration and check standard concentrations as well as a tool for calculating other dilutions.

Typically, two standards are prepared, a calibration standard and a check standard. The calibration standard contains the highest concentration used in the calibration and the instrument is programmed to internally dilute it to create the other points on the calibration curve (see calibration curve section above). The check standard is usually prepared with about half the concentration of the calibration standard and is analyzed periodically though the run to verify instrument performance.

Remove the stock solutions (IC) from the refrigerator and allow them to warm to near room temperature. A warm water bath may be used speed up this step. There are separate stock solutions for the calibration and check standards.

Take the appropriate size volumetric flasks from the acid washed glassware cabinets above the instrument. The flasks are store full of DI water and should be emptied before use.

Select the appropriate size volumetric pipets from the acid washed pipet drawer below the instrument.

Pipet the desired amount of the C stock, into the volumetric flasks for the calibration and check standards. Use different pipets for each component! Add DI water to the volumetric flask to bring the total volume to the line. Cap the flasks, invert and shake gently several times to ensure complete mixing. The used pipets and flasks must be acid washed before the next use.

Return the stock solutions to the fridge.

Starting a run.

Pour the samples and standards into acid washed vials (located in the cupboard under the instrument) and place them into the autosampler according to the sample table.

Check the instrument waste carboy on the floor in front of the instrument. Empty it down the sink if it is more than half full. Be sure to replace both waste tubes after emptying.

Empty, rinse and refill the sampler wash bottle (left side of the sampler) with DI water. Make sure the tube to the sample goes to the bottom of the bottle.

Check the level of the 20% H_3PO_4 bottle between the instrument and the sampler. Add more H_3PO_4 if it is less than half full. Wear safety glasses when handling H_3PO_4 ! Make sure the end of the tube going to the instrument is at the bottom of the bottle.

Empty, rinse and refill the "water for dilution" bottle (between the instrument and the sampler) with DI water. If you are running samples in a matrix other than water, move the tube going into the instrument to a container of sample matrix. Make sure the end of the tube goes to the bottom of the bottle.

Press the "Start" button on the upper tool bar. Select "Shut down Instrument". Press OK.

Cleanup

Users are expected to promptly clean up volumetric pipets, volumetric flasks, sample vials, caps, and any other materials used in running the instrument.

Pipets, flasks and vials should be rinsed 3 times with DI water. Then place them in the 1.5 M HCl bath in room A254. Take care that there are no air bubbles in any of the items. Always wear a lab coat, gloves and safety glasses when working with or around the acid baths! Allow the items to soak in the acid bath overnight.

Remove the items from the acid bath and rinse individually with DI 6 times. Pipets should then be placed in the105 C oven in A258. When they are completely dry, put them back in the drawer under the instrument. Volumetric flasks should be filled with DI and placed in the cabinets above the instrument. Vials should be placed upside down in the plastic racks and air dried before putting them in the cupboard below the instrument.

The caps should be rinsed 6 times with DI water and aired dried before going back into the cupboard under the instrument.

Exporting results

Open the sample table containing the results.

Highlight the rows containing the desired results.

Go into “file”, “ascii export”; there will be 2 choices: “normal” and “detailed”. “Normal” will give one line for each sample with the average result for C and N for that sample. “Detailed” will give one line for each injection. “Normal” is the best one for obtaining the data in a form that can be used for subsequent calculations. “Detailed” is principally used for maintaining a record of the sample run that includes the details like how many injections were performed for each sample/analyte combination, peak areas and standard deviations.