

USP Total Organic Carbon System Suitability Test for the Aurora 1030W TOC Analyzer

Application Note 22310807

Keywords

USP Method 643
 Suitability Standard
 Sucrose
p-benzoquinone

Introduction

As a result of the development of U.S. Pharmacopoeia (USP) Method <643>, which replaces the oxidizable substances test, there is a need to analyze purified water using a total organic carbon (TOC) analyzer for calibration and verification using two standards. The two standards are referred to as a *Standard Solution* and a *System Suitability Standard*. Current USP guidelines require that the unit compare a 500-ppb C Sucrose Standard Solution with a 500-ppb C (carbon) *1,4*-benzoquinone (*p*-benzoquinone) System Suitability Standard. Reagent water (RW) is also analyzed as a standard.

By USP guidelines, the range of acceptable recovery of the System Suitability Standard is 85–115%. Failure to achieve this percentage of recovery requires repeating the calibration or adjusting the method to oxidize the *p*-benzoquinone more effectively. USP guidelines also require the preparation of stock solutions at 10 ppm for both standards and that 500-ppb C standards are made from these stocks. This experimental method discusses the preparation and analysis of these standards.

An OI Analytical Aurora 1030W TOC Analyzer (Figure 1) was used to perform the experiment. The Aurora 1030W enables analysis of SST by NPOC only, TICNPOC, and TOC by subtraction modes.



Figure 1. Aurora 1030W TOC Analyzer

For this experiment, NPOC internal was chosen since it uses two steps to calibrate for total organic carbon (TOC).

1. A set amount of sample and phosphoric acid is added to the digestion vessel, the temperature is set, and the sample undergoes sparging by a carrier gas to remove all total inorganic carbon (TIC), which is swept to the outside.
2. A preset amount of sodium persulfate is added to the reaction vessel, the temperature is raised for the reaction to occur, and the carrier gas is started after the react time. This sparge allows the carbon dioxide to be swept to the NDIR (nondispersive infrared detector) where it is converted to a signal.

The resulting mass of carbon in the carbon dioxide is proportional to the mass of carbon present as TOC in the sample and reported at the proper concentration.

Experiment

The system suitability test (SST) evaluates the TOC analyzer's capability to oxidize one easy component and one more difficult component. The test analyzes sucrose and *p*-benzoquinone, the latter considered more difficult. A feature of the Aurora 1030W allows the operator to create a method with the necessary calibration data and easily adjust the operating parameters to optimize the efficiency of the reaction process. This feature also allows for setting up a sequence with an SST method built into the system to test both compounds. See Figure 2.

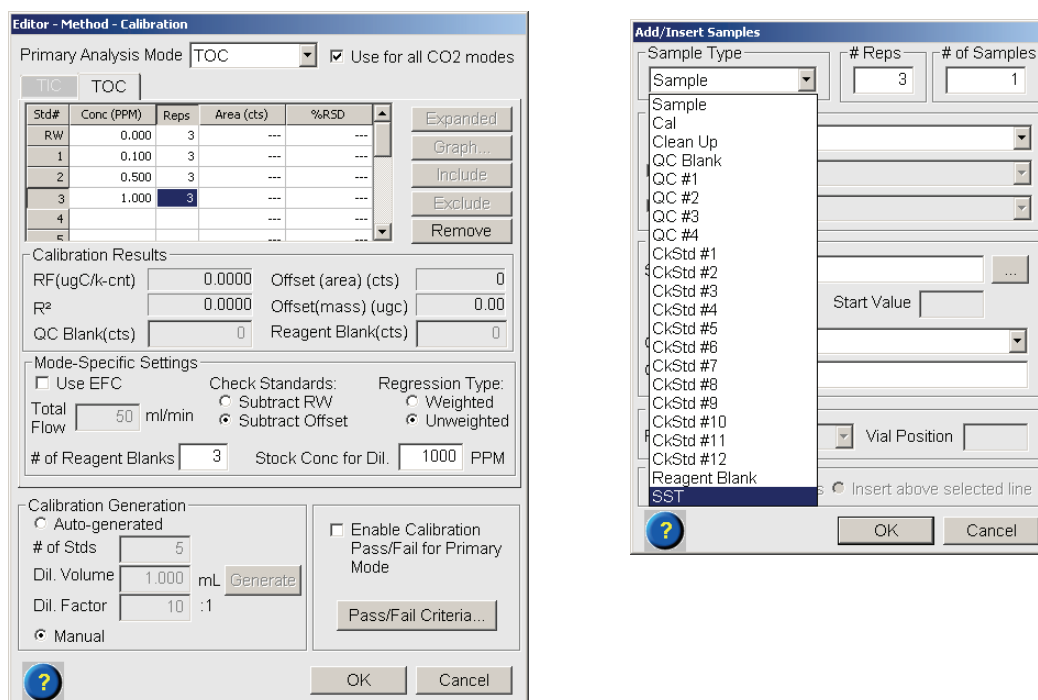


Figure 2. Editor method calibration screen (left) and editor sequence SST test screen (right)

As a result, calibration and analysis is easily setup without creating multiple sequences to test the unit's capability,

minimizing system operator effort (Figure 3).

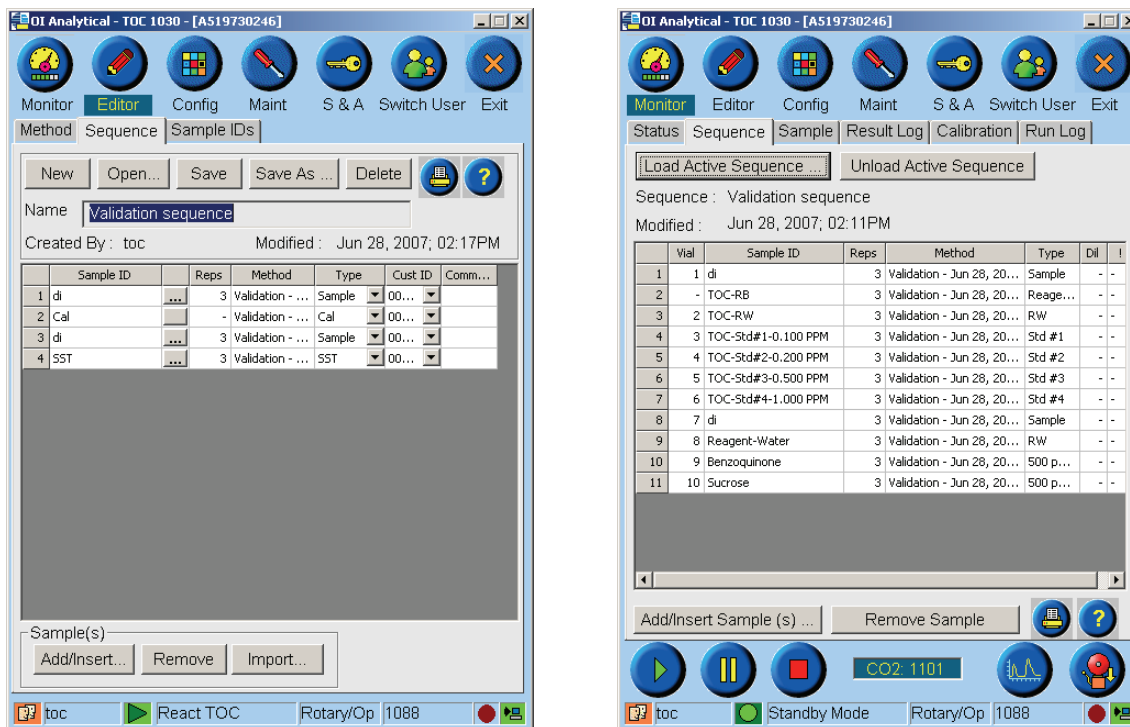


Figure 3. Editor Sequence screen (left) and SST active sequence (right)

Response efficiency is measured and recorded. Again, USP requires the efficiency be 85–115 percent.

Calibration

The TOC analyzer was calibrated using a 4-point calibration curve according to the method in Figure 4. The standards were RW, 100 ppb C KHP, 500 ppb C KHP, and 1 ppm C KHP. Running to 1 ppm is necessary to bracket the 500 ppb C standard for analysis. The results are provided in Figure 5.

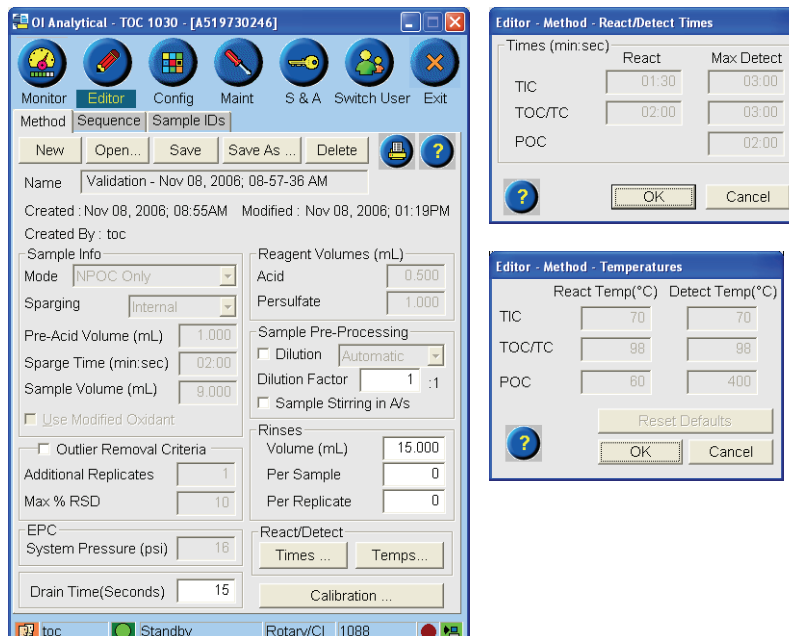


Figure 4. Method Parameters

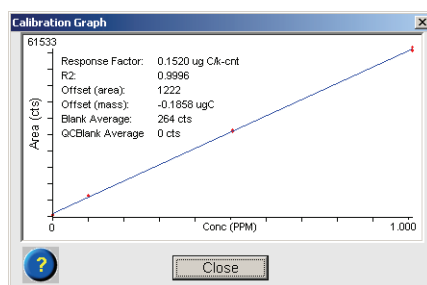


Figure 5. Calibration Results

The response factor for the Aurora 1030W was 0.1520 $\mu\text{g C/k-cnt}$, and the coefficient of correlation was 0.9996. Once within acceptable limits, the system is ready for sample analysis.

After completion of the calibration sequence, the curve must be validated using the check standard principle as described previously. The same three standards were run using the check standard defaults found in the Sample Type of the Editor -> Sequence tab.

It is important to note the RW or offset is used to subtract the background of the instrument from the check standard automatically. By selecting “subtract RW”, the value of the water will be taken into account. By selecting “subtract offset”, the linear regression as it passes through the y-axis is taken into account. For the examples in this experiment, “subtract offset” was used, which is the default configuration on the Aurora 1030. USP 643 allows a maximum concentration of 0.25 ppm C in RW. Results are displayed in Table 1.

Table 1. Check Standards results

Chkstd	ppm	%RSD	ppm Error	Recovery
0.1	0.095	3.07	0.0029	95.00%
0.5	0.498	0.65	0.0032	99.60%
1	0.994	0.59	0.0059	99.40%

Standard Solution

Prepare a 10-ppm C sucrose stock solution by weighing 24 mg of sucrose (dried at 105 °C for 2 hours); weighing paper is suggested because the dried sucrose is less likely to adhere to weighing paper than to weigh boats. The 42.1% carbon sucrose is added to a 1-L volumetric flask containing 500–750 mL of low-TOC water. Swirl the mixture until the sucrose is completely dissolved. When no particles remain visible, fill the flask to the 1-L mark with low-TOC water.

To prepare the 500-ppb C standard, remove 50 mL from the 10-ppm C stock solution with a volumetric pipette and place in a 1-L volumetric flask. Add low TOC water to the 500–750 mL level and swirl to mix. Complete preparation of the standard by adding low-TOC water to the 1-L mark on the flask and mixing again.

Prepare a 1-ppm C standard by transferring 100 mL from the 500-ppb C standard to a 1-L volumetric flask and fill with low-TOC water to the 1-L mark on the flask.

System Solubility Standard

Prepare the 10-ppm C stock solution of *p*-benzoquinone (66.7%) carbon by weighing 15 mg of undried *p*-benzoquinone on a weigh boat and rinse with low-TOC water into a 1-L volumetric flask. Add low-TOC water up to the 500–750 mL level and allow the *p*-benzoquinone to completely dissolve. When no more particles are visible, add low TOC water to fill the flask to the 1-L mark.

To prepare the 500-ppb C standard, remove 50 mL from the 10-ppm C stock solution with a volumetric pipette and place in a 1-L volumetric flask. Add low TOC water to the 500–750 mL level and swirl to mix. Complete preparation of the standard by adding low TOC water to the 1-L mark on the flask and mix again.

Similar to a check standard as described above, the 1030W automatically calculates the SST by analyzing the RW, *p*-benzoquinone, and sucrose as samples and then subtracts the RW from each standard to calculate the percent response efficiency using Equation 1:

$$E(\text{PercentResponseEfficiency}) = \frac{(R_{ss}(\text{p-benzoquinone}) - RW)}{(R_{s}(\text{sucrose}) - RW)} \times 100 \quad (1)$$

Results

Once the standards are made, the Aurora 1030W runs the System Suitability Test. Data is reported in Table 3.

Table 2. Check Standards results

Reagent Water	0.018 ppm
Benzoquinone	0.538 ppm
Sucrose	0.531 ppm
% Error	101.36%

As shown in Table 3, the system properly oxidized the sample within the percent error as prescribed in USP 643.

To demonstrate the stability of the instrument, the SST was setup for seven consecutive days from the same stock standard, including the data from the previous table. In addition vials of ultra pure water (UPW) were made from the same water source to show stability of the instrument at low levels over the course of time. All vials (both water and system suitability standards) not used within the first day were stored at 4 °C until later use. Each day a sequence was run of an initial DI water vial for cleanup, the SST standards, and a series of ten UPW vials picked at random. The vials were brought to room temperature before use and then sampled in triplicate per vial. The results are shown in the Table 3.

Table 3. SST Days 1-7

	Nov. 9	Nov. 10	Nov. 11	Nov. 12	Nov. 13	Nov. 14	Nov. 15
DI Water	0.014	0.086	0.695	0.046	0.039	0.049	0.07
Reagent Water	0.018	0.059	0.018	0.019	0.022	0.029	0.047
Benzoquinone	0.538	0.531	0.552	0.557	0.543	0.552	0.549
Sucrose	0.531	0.551	0.568	0.545	0.541	0.536	0.559
%	101.36%	95.94%	97.09%	102.28%	100.39%	103.16%	98.05%
UPW #1							
UPW #1	0.021	0.019	0.042	0.023	0.056	0.037	0.020
UPW #2	0.010	0.014	0.021	0.066	0.018	0.033	0.035
UPW #3	0.013	0.020	0.025	0.027	0.053	0.026	0.034
UPW #4	0.028	0.020	0.037	0.033	0.031	0.015	0.021
UPW #5	0.015	0.011	0.018	0.051	0.116	0.017	0.018
UPW #6	0.007	0.112	0.039	0.026	0.032	0.034	0.021
UPW #7	0.009	0.020	0.067	0.030	0.063	0.042	0.024
UPW #8	0.017	0.015	0.023	0.033	0.044	0.019	0.033
UPW #9	0.023	0.028	0.019	0.020	0.045	0.025	0.033
UPW #10	0.013	0.034	0.008	0.031	0.081	0.031	0.032

As shown in Table 3, all SST runs for the 7 days fell within the passing criteria of 85-115% set by USP <643>, with the greatest divergence at 4% of the true 100%. These data demonstrate that the instrument exhibits solid stability during the 7-day time frame.

Because the water was obtained from a faucet and capped by hand as opposed to retrieval by on-line sampling, a potential for carbon contamination exists. Resulting vial-to-vial variations are therefore inherent to the sampling process. However, it can be concluded that low background carbon levels were consistently obtained. To further demonstrate that consistent low background carbon levels were obtained, the error from replicate to replicate is shown in Table 4 after breaking the RSDs into an actual amount of ppm error. Due to the volume of data, only results from 3 days are provided.

Table 4. Percent RSD to ppm Error Data

	November 9		November 10		November 11	
	RSD	ppm Error	RSD	ppm Error	RSD	ppm Error
UPW #1	11.12	0.0023	8.53	0.0016	1.15	0.0005
UPW #2	7.6	0.0008	19.93	0.0028	7.9	0.0017
UPW #3	17.35	0.0023	3.26	0.0007	13.28	0.0033
UPW #4	4.47	0.0013	6.78	0.0014	1.29	0.0005
UPW #5	17.15	0.0026	18.9	0.0021	5.67	0.0010
UPW #6	18.75	0.0013	11.35	0.0127	7.6	0.0030
UPW #7	24.29	0.0022	6.19	0.0012	3.52	0.0024
UPW #8	11.95	0.0020	10.32	0.0015	12.27	0.0028
UPW #9	7.09	0.0016	6.44	0.0018	2.03	0.0004
UPW #10	2.78	0.0004	3.51	0.0012	3.55	0.0003

One additional benefit of the Aurora 1030W when determining the System Suitability is that the Aurora will run the calculation for you when utilizing the SST sample type in the sequence. As a result, instant visual clues indicate whether the instrument passes. An example is shown in Figure 6.



Figure 6. Result Log

Summary

When the standard and stock solutions are made correctly, the results produced by the Aurora 1030W TOC Analyzer comply with the USP regulations. Shelf life of the System Suitability Standards must be considered when setting up a standard operating protocol for the laboratory because the carbon content of *p*-benzoquinone can decrease over time since *p*-benzoquinone sublimates.



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