vial selection guide

1. introduction

SUEZ, through its Sievers product line of TOC analyzers and consumables, recognizes the importance of having traceable, certified low TOC vials. In addition to conventional Sievers certified TOC vials, two specialty vial types were developed to meet customer needs: Sievers Pre-Acidified TOC Vials and Sievers Dual Use Conductivity & TOC (DUCT) Vials. These specialty vials may improve performance such as TOC % recovery on specific product families. The following Vial Selection Guide is written to help customers test all three types of vials and identify the optimal vial choice for specific applications. After completing the test plan and data worksheet below, the optimal vial choice can be selected based TOC recovery and linearity.

2. experimental test plan

2.1 Materials

- Sievers M9 or 900 TOC Analyzer[1]
- Sievers Autosampler[2] with DataPro/DataPro2 Software
- One 1L volumetric flask
- Five 500 mL volumetric flasks
- 20 Certified TOC Vials
- 18 Certified DUCT Vials
- 20 Certified Pre-Acidified TOC Vials
- Deionized water

2.2 Procedure

1. Create a 1000 ppm stock solution of the compound to be tested. Do so by dissolving 1000 mg (1g) of the compound in 1L of deionized water (<20 ppb TOC) in a 1L volumetric flask. If the compound is not soluble at 1000 ppm, a lower concentration stock solution can be made. The volumes listed in Step 2 of this procedure will need to be changed to reflect the different stock solution concentration.

2. Using the 1000 ppm stock, create solutions of 10 ppm, 5 ppm, 3 ppm, 1 ppm, and 0.5 ppm in volumetric flasks. The following table describes the volume of stock solution to add to 500 mL volumetric flasks to make the desired concentrations. Fill each flask to 500 mL total volume with <20 ppb TOC deionized water. (Please note that at least 360 mL of solution of each concentration will be needed for the vial selection testing protocol.)

Volume of Stock Solution	Compound Concentration (in 500 ml)
0.25 mL	**0.5 ppm**
0.5 mL	**1 ppm**
1.5 mL	**3 ppm**
2.5 mL	**5 ppm**
5.0 mL	**10 ppm**

3. Select one type of vial (Certified, Pre-Acidified, or DUCT). Of the 18 total vials of this type, 15 will be filled in Step 4 below, and three will be filled in Step 5.

4. Fill three vials with each solution created in Step 2, for a total of 15 vials. For example, fill three vials with the 0.5 ppm solution, three vials with the 1 ppm solution, etc. until there are three vials of each concentration. Be sure to label all vials to track concentrations.

a. When filling the Pre-Acidified Vials, be sure volumes are equal to ensure consistent pH across vials. For example, fill all Pre-Acidified Vials to the top of the label. After filling and capping each Pre-Acidified Vial, gently invert the vial several times to thoroughly mix the acid into the sample.

5. Three vials should remain. These three vials will be blanks or negative controls and should be filled with deionized water.

6. Repeat Steps 3-5 for the other two types of vials. 54 vials should be filled and labelled.

7. Fill two additional Certified TOC Vials with deionized water and label as Rinse Vials. Also, fill two additional Pre-Acidified Vials with deionized water and label as Acidified Rinse Vials.

8. Place the vials in the Autosampler[2] in the following order:

a. **Rack 1** (closest to the needle of the Autosampler)

Certified Blank*	Certified Blank	Certified Blank	Certified 0.5 ppm	Certified 0.5 ppm	Certified 0.5 ppm	Certified 1 ppm
Certified 1 ppm	Certified 1 ppm	Certified 3 ppm	Certified 3 ppm	Certified 3 ppm	Certified 5 ppm	Certified 5 ppm
Certified 5 ppm	Certified 10 ppm	Certified 10 ppm	Certified 10 ppm	Rinse Vial	Acidified Rinse Vial	Acidified Rinse Vial

* Certified Blank refers to the negative control vials in Certified TOC vials prepared in Step 5.

b. **Rack 2**

Acidified Blank**	Acidified Blank	Acidified Blank	Acidified 0.5 ppm	Acidified 0.5 ppm	Acidified 0.5 ppm	Acidified 1 ppm
Acidified 1 ppm	Acidified 1 ppm	Acidified 3 ppm	Acidified 3 ppm	Acidified 3 ppm	Acidified 5 ppm	Acidified 5 ppm
Acidified 5 ppm	Acidified 10 ppm	Acidified 10 ppm	Acidified 10 ppm	Rinse Vial	DUCT*** Blank	DUCT Blank

**Acidified blank refers to the negative control vials in Pre-Acidified Vials prepared in Step 5.
***DUCT blank means the negative control vials in DUCT Vials prepared in Step 5.

c. **Rack 3** (furthest from the needle)

DUCT Blank***	DUCT 0.5 ppm	DUCT 0.5 ppm	DUCT 0.5 ppm	DUCT 1 ppm	DUCT 1 ppm	DUCT 1 ppm
DUCT 3 ppm	DUCT 3 ppm	DUCT 3 ppm	DUCT 5 ppm	DUCT 5 ppm	DUCT 5 ppm	DUCT 10 ppm
DUCT 10 ppm	DUCT 10 ppm	***EMPTY***	***EMPTY***	***EMPTY***	***EMPTY***	***EMPTY***

***DUCT blank refers to the negative control vials in DUCT Vials prepared in Step 5.

9. Once all vials are placed in the Autosampler[2], set up a protocol in DataPro or DataPro2. Use the settings[1] outlined in the table below to set up the method for each vial.

Compound Concentration (ppm)	Acid Setting (µl/min)	Oxidizer Setting (µl/min)	Repetitions	Rejects
blank	1.0	0.0	5	1
0.5	1.0	0.0	5	1
1	Auto	Auto	5	1
3	Auto	Auto	5	1
5	Auto	Auto	5	1
10	Auto	Auto	5	1

10. Perform calculations outlined in Appendix A, and record TOC data in Appendix B. For all calculated statistics, the average of all non-rejected repetitions for the three sample vials should be used. Do not use the average of the averages that the instrument displays for each vial. Statistically correct data for averages and standard deviations will only be achieved by taking the grand average of all non-rejected repetitions for the three sample vials at each concentration.

[1] It is recommended that the M9 or 900 TOC Analyzer be multi-point calibrated. If single point calibrated, do so at or above 10 ppm. The Autoreagent (AR) feature must be verified for the M9, and both calibrated and verified for the 900.

[2] If a Sievers Autosampler is not available, run the vials in the order stated in Step 8 with the grab mode settings outlined in Step 9.

appendix a: calculations

Percent Recovery

1. Calculate the carbon content of the compound.

$$\% \text{ Carbon}=\frac{\#\text{of Carbon molecules x MW of Carbon}}{\text{Total MW of Compound}} \text{ x } 100$$

MW= Molecular Weight (g/mole)

   a. Example: Sucrose ($C_{11}H_{22}O_{11}$)

$$\% \text{ Carbon}=\frac{(11 \text{ x } 12.01)}{(11 \text{ x } 12.01)+(22 \text{ x } 1.01)+(11 \text{ x } 16.00)} \text{ x } 100 = 40.0 \%$$

2. Calculate the theoretical TOC concentration for each solution (0.5 ppm, 1 ppm, 10 ppm, etc.).

$$\text{Theoretical TOC Concentration=Solution TOC concentration x } \left(\frac{\% \text{ Carbon}}{100}\right)=$$

   a. Example: 10 ppm sucrose

$$\text{Theoretical TOC concentration=10 ppm x } \left(\frac{40.0\%}{100}\right)=4 \text{ ppm TOC}$$

3. Calculate the percent recovery for each TOC concentration and each vial type.

$$\% \text{ Recovery=}\left(\frac{\text{Measured TOC-Blank TOC}}{\text{Theoretical TOC}}\right)*100\%$$

Measured TOC = TOC average from the triplicate vials at each concentration
Blank TOC = TOC average of the negative controls for each vial type

Linearity

1. Establish linearity using the measured and theoretical TOC concentrations. Create a graph with expected TOC concentrations on the x-axis and measured TOC concentrations on the y-axis for each vial type. Perform a least-squares regression on the data and calculate the slope, intercept, and R^2. If using Microsoft Excel, use the following steps:

   a. Right click on any data point in the graph and select "Add Trendline".
   b. In the pop-up menu select: "linear" and check the boxes near the bottom for "display equation on chart" and "display R-squared value on chart".
   c. Click on the close button.
   d. A line should now be on the graph with an equation for the least-squares regression line through the data and R^2 value for the correlation coefficient of the data.

2. Compare the R^2 value for each set of vials. An R^2 close to one indicates a high degree of linearity among the data set.

3. Using the % Recovery data and the Linearity data, choose the optimal vial type for this compound. The best vial choice will be the one with the highest degree of linearity and highest % recovery for the range of TOC concentrations tested.

appendix b: data worksheet

Calculated By: ______________________________ Date: ______________

Verified By: ______________________________ Date: ______________

Compound: ______________________________ % Carbon: ____________

Sievers Certified TOC Vial Percent Recovery:

Concentration (ppm)	Expected TOC (ppm)	Measured TOC (ppm)	Standard Dev (ppb)	% RSD	% Recovery
Blank					
0.5					
1					
3					
5					
10					

Linearity of Certified TOC Vial, R^2 = __________

Sievers Pre-Acidified TOC Vial Percent Recovery:

Concentration (ppm)	Expected TOC (ppm)	Measured TOC (ppm)	Standard Dev (ppb)	% RSD	% Recovery
Blank					
0.5					
1					
3					
5					
10					

Linearity of Certified TOC Vial, R^2 = __________

Sievers DUCT Vial Percent Recovery:

Concentration (ppm)	Expected TOC (ppm)	Measured TOC (ppm)	Standard Dev (ppb)	% RSD	% Recovery
Blank					
0.5					
1					
3					
5					
10					

Linearity of Certified TOC Vial, R^2 = __________

Attach and label graph for each of the above to this worksheet.

Optimal Vial Choice = ________________