



Using the CDS 8500 Automated Purge and Trap System's Automated Sample Dilution Tool to Prepare Calibration Curves

Application Note

Environmental

Abstract

The analytical performance of the CDS 8500 Series Purge and Trap is demonstrated for the sample syringe to prepare calibration curves by performing automated sample dilution.

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Introduction

CDS Analytical's 8500 Series Purge and Trap System is a new, fully automated Purge and Trap concentrator for the trace measurement of purgeable volatile organic compounds (VOCs) in water. The 8500 utilizes a 25 mL sample syringe to deliver water samples to the concentrator. An added feature of the sample syringe is that it can perform automated dilutions of samples up to a ratio of 1:100. This application note demonstrates how having four internal standard channels provides additional flexibility and requisite reproducibility to prepare calibration curves.

Experimental Setup

The 8500 Series Purge and Trap System was used to process the water samples. The Purge and Trap method parameters are shown in Table 1, which are standard for the analysis of VOCs defined in the EPA Method 8260D.



Valve Oven Temperature	130°C
Transfer Line Temperature	130°C
Standby Flow	10 mL/min
Trap Ready Temperature	35°C
Wet Trap Ready Temperature	45°C
Spurge Vessel Heater	On
Purge Time	11 min
Purge Flow	40 mL/min
Purge Temperature	40°C
Dry Purge Time	2 min
Dry Purge Flow	200 mL/min
Dry Purge Temperature	35°C
Foam Sensor	On
Desorb Parameters:	
Water Rinse Volume	5 mL
Number of Water Rinses	3
Overflow Sensor	On
Desorb Preheat Temperature	245°C
Desorb Time	4 min
Desorb Drain Flow	250 mL/min
Desorb Temperature	250°C
Bake Parameters:	
Bake Time	4 min
Bake and Vessel Flow	200 mL/min
Trap Bake Temperature	260°C
Wet Trap Bake Temperature	260°C

Table 1. Purge and Trap Method Parameters.

A Shimadzu single quad GCMS-QP 2010 was used. GC/MS conditions are listed in Table 2. Carrier gas was supplied to the 8500 Series Purge and Trap and a heated transfer line from the 8500 Series Purge and Trap concentrator was plumbed into the carrier supply line of the split/spitless inlet.

Analytical Column	RTX-VMS (30 m x 25 mm x 140 µm)
Injector Temperature	240°C
Carrier Gas	He at 1.00 mL/min
Split Ratio	40:1
Oven Program	35°C Hold 4 min 90°C at 5°C/min 100°C at 12°C/min 220°C at 30°C/min Hold 2.67min
Mass Spectrometer:	
Interface Temperature	220°C
Ion Source Temperature	200°C
Scan Mode and Range	Full scan 35-260m/z
Scan Time	0.3 min
Scan Speed	833

Table 2. GCMS Conditions.

From a 2000 µg/mL BTEX standard, purchased from Restek, an aqueous solution of 200 ng/mL BTEX was prepared and directly added to the purge and vials. The 25 mL sample syringe was used to dilute samples at ratios of 1:100, 1:50, 1:25, 1:10, 1:5, 1:2, and 1:1. The resulting concentration range was 2 to 200 ng/mL. The internal standard was a 4 component 8260 internal standard mix, and the 8260 surrogates was a 3 component mix. Both were diluted to a concentration of 25 µg/mL. 5 µL of this internal standard was added to each sample.

Results and Discussion

Figure 1 depicts the calibration curves from the BTEX standard between 2 and 200 µg/mL. Table 2 summarizes the calibration accuracy and reproducibility. The calibration curve meets the requirements set by EPA methods such as 524 and 8260.

Conclusion

The 8500 Series Purge and Trap System a 25 mL sample syringe for transferring water samples from the autosampler to the purge and trap concentrator. An added feature of the sample syringe is that it supports automated sample dilution. This application note demonstrates that dilution can be quantitatively performed to prepare calibration curves.

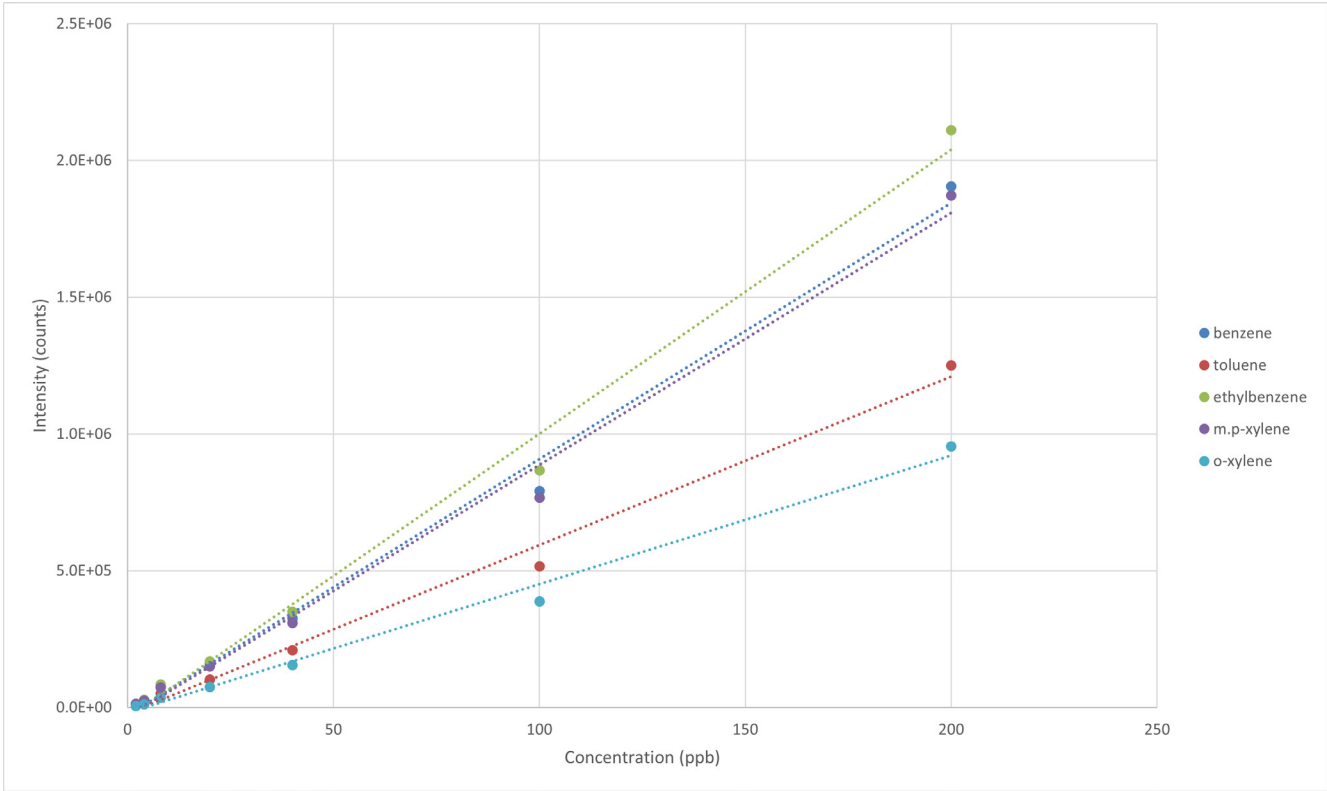


Figure 1. TIC of 8260D volatile organic standard mix at 50 µg/L.

Table 3. Accuracy and RSD for the BTEX components at each calibration level (n=3).

	benzene		toluene		ethylbenzene		m-xylene		o,p-xylene	
Concentration (ppb)	Accuracy (%)	RSD (%)	Accuracy (%)	RSD (%)	Accuracy (%)	RSD (%)	Accuracy (%)	RSD (%)	Accuracy (%)	RSD (%)
2	105.2	6.9	108.1	1.0	113.2	5.9	113.8	5.6	115.3	5.4
4	91.8	5.7	92.4	6.4	92.4	5.5	91.9	4.7	92.6	5.6
8	111.6	0.7	110.6	0.3	107.9	0.2	108.0	0.4	106.9	1.3
20	93.6	6.1	92.3	6.2	89.8	6.9	90.5	6.6	89.6	6.8
40	92.9	3.2	91.9	3.1	91.0	3.0	90.6	2.8	90.5	3.5
100	89.2	2.8	89.0	2.9	89.0	3.3	88.8	2.8	88.5	3.4
200	106.8	1.5	107.2	1.8	107.7	1.7	107.8	1.4	108.1	1.2