



Using the CDS 8500 Automated Purge and Trap System's Internal Standard EPC Delivery Mechanism for Calibration Curve Preparation

Application Note

Environmental

Abstract

The analytical performance of the CDS 8500 Series Purge and Trap is demonstrated for the electronic pressure control, used for internal standard delivery, to prepare calibration curves.

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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 incorporates a robust electronic pressure control to deliver internal standard, surrogates, and/or matrix spikes through four separate channels simultaneously. 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.

Calibration standards were prepared from a 2000 µg/mL BTEX standard purchased from Restek. From those standards, 5 and 40 µg/mL standards were prepared and loaded into separate internal standard channels. Each standard was added to a deionized water sample in 1, 2, 5, 10, 20, and 25 µL increments. The final result is an eleven point calibration curve between 5 and 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 5 and 200 µg/mL. Table 3 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 utilizes and electronic pressure control to reproducibly add internal standard, surrogates, and matrix spikes to water and soil samples. The flexibility of the fourth channel, along with the different volumes of standard that can be added by the EPC, makes the 8500 internal standard module a tool that can be used to prepare calibration curves for standards.

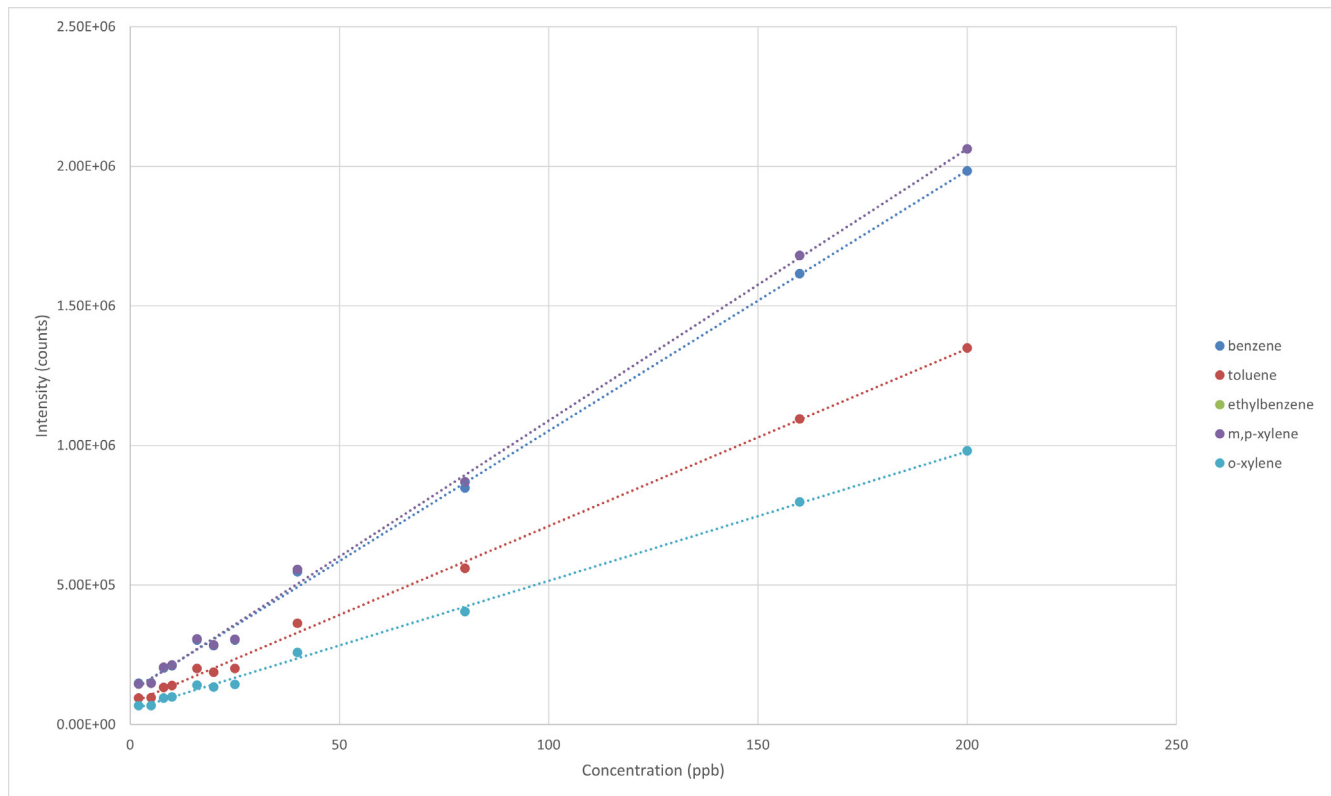


Figure 1. Calibration curves for BTEX target components.

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

	benzene		toluene		ethylbenzene		m,p-xylene		o-xylene	
Concentration (ppb)	Accuracy (%)	RSD (%)	Accuracy (%)	RSD (%)	Accuracy (%)	RSD (%)	Accuracy (%)	RSD (%)	Accuracy (%)	RSD (%)
5	69.5	3.7	72.9	4.5	70.9	5.5	71.8	5.4	73.8	5.6
8	112.8	11.1	115.1	10.6	115.5	10.9	118.3	10.6	120.2	8.0
10	99.0	3.0	102.9	2.3	103.4	2.2	102.2	2.7	104.0	2.8
16	124.0	8.9	125.0	9.8	124.5	10.4	123.8	9.8	122.7	9.0
20	88.3	3.4	89.5	4.0	88.5	3.7	88.4	4.5	89.5	3.9
25	79.6	0.9	80.1	1.8	79.2	1.7	79.2	1.7	80.0	1.6
40	103.2	14.4	101.8	15.2	101.8	15.5	101.4	15.4	99.6	16.2
80	104.9	11.4	102.2	11.1	103.2	11.9	103.7	11.1	101.7	11.1
160	100.2	12.1	100.4	12.1	100.7	11.5	100.4	11.7	100.5	11.5
200	99.9	1.6	100.2	1.8	99.9	2.1	100.0	2.1	100.2	2.7