Calibrating Gasoline Range Organics (GROs) using an automated dilution scheme with the Atomx Concentrator/Multi-matrix Autosampler

Application Note

Abstract

In developing the **Atomx Automated Sample Prep System,** Teledyne Tekmar integrated both a P&T concentrator and a multi-matrix autosampler into a single platform system that can be used in the analysis of soil, water, and soils requiring methanol extraction. This "all-in-one" setup has allowed for greatly increased throughput and efficiency through the built in features it provides.

In this study the sample syringe dilution feature was used to generate a calibration curve from a single stock solution for a subset of EPA method 8260b compounds[1]. This study not only validates the capability to perform accurate and precise dilutions but also removes the risk of calibration errors that can occur from manually preparing multiple standards. Lastly, it clearly demonstrates that a considerable amount of labor time can be saved when using this feature.

Experimental-Instrument Conditions

GC Parameters		MSD Parameters		Atomx Water Parameters	
GC:	Agilent 7890A	MSD:	5975C inert XL	Desorbs Temp	250°C
Column	Restek RTX-624 20m x 0.18mmID x 1µm	Source:	230°C	Dry Purge Flow	100mL/min
Oven Program:	35°C for 4 min; 16°C/ min to 85°C for 0 min; 30°C /min to 210°C for 3 min, 14.29 min runtime	Quad:	150°C	Dry Purge Temp	20°C
Inlet:	220°C	Solvent Delay:	0.5 min	Desorbs Time	2.00 min
Column Flow	1.2 mL/min	Scan Range:	m/z 35-300	Sample Volume	5.0mL
Gas:	Helium	Scans:	5.19 scans/sec	Purge Time	11.00 min
Split:	80:1	Threshold:	400	Purge Flow	40mL/min
Pressure:	25.8 psi	MS Transfer Line Temp:	230°C	Purge Temp	20°C
				Condensate Purge Temp	20°C
				Desorbs Temp	250°C
				Bake Time	2.00 min
				Bake Flow	200mL/min
				Trap Bake Temp	280°C
				Condensate Bake Temp	200°C

Table 1: GC, MSD, and Atomx Parameters

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Calibration and Minimum Detection Limits

A 100ppb working calibration standard containing the target and surrogate compounds (PVOC/GRO Mix) was prepared in 500mL of de-ionized water. The standard was then transferred to 6 separate VOA vials for analysis. The calibration range was 2-100ppb and was accomplished using the following dilution volumes:

1:1= 5mL, 1:2= 2.5mL, 1:5= 1mL, 1:10= 500µL, 1:25= 200µL, and 1:50= 100µL.

All dilutions were automatically brought to a 5mL final volume with deioninzed water using the instrument supplied water reservoir. Additionally the 1:100 dilution was used to generate the MDLs at 1ppb for this study. A 125ppm Internal Standard (IS) was prepared in methanol and transferred to the standard vessel on the Atomx. Using the internal standard addition feature 1µL volumes were automatically injected to the calibration standards providing a constant 25ppb final concentration.

Compound	MDL	Calibration %RSD
MTBE	0.73	4.76
Dibromofluoromethane (surr)	0.72	4.47
Benzene	0.61	4.59
Toluene-d8 (Surr)	0.66	8.02
Toluene	0.60	5.4
Ethylbenzene	0.55	7.58
M&P Xylene	1.11	7.44
Ortho Xylene	0.58	6.46
BFB (Surr)	0.52	11.69
1,3,5-Trimethylbenzene	0.57	10.08
1,2,4-Trimethlybenzene	0.53	8.22
Naphthalene	0.53	6.99

Table 2: MDL and Calibration %RSD for Target and Surrogate Compounds

Agilent Chemstation software was used to process the calibration data. The relative response factors (RRF) of all the analytes were evaluated for %RSD. The calibration met EPA method 8260 performance criteria. The calibration results and a typical chromatogram are listed in Figure 1.

Insert Figure 1 Here

Figure 1: Static Experimental Results and TIC of 50ppb GRO calibration standard.

Conclusions

Teledyne Tekmar's Atomx Automated Sample Prep System continues to improve efficiency and throughput. By automating the calibration process using the sample dilution feature in conjunction with the automated internal standard addition feature, the calibration curve met the performance criteria of EPA method 8260b[1]. Using the Atomx, only one stock standard solution needs to be made, rather than an entire series of calibrations. Switching to an automated calibration allows for the same performance, but with the benefit of less prep time. This automated calibration, as well as the other enhancement tools, aid the user in both productivity and cost savings.

References

1. USEPA Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Revision 2, December 1996.

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