Validation of EPA Method 8260C on a Thermo Scientific TRACE 1300 Series GC with New Helium Saver Inlet

Application Note

Abstract

As helium continues to become more expensive, many labs are searching for more cost effective carrier gas options. This study demonstrates the effectiveness of the Atomx automated sample prep system's nitrogen purge feature in conjunction with Thermo Scientific's TRACE 1300 series Helium Saver inlet at producing quality data while also reducing helium consumption. Calibration and method detection limit (MDL) samples were prepared, analyzed and quantitated according to US EPA Method 8260C.

Introduction

As helium becomes more difficult and expensive to procure, there has been a push to find alternative carrier gas options for gas chromatography/mass spectrometry (GC/MS) applications. Chief among these is hydrogen, which can be cheaply and easily generated in the lab. However, hydrogen can have several disadvantages as a carrier gas for GC/MS, including both its highly reactive and flammable natures[1].

Thermo Scientific has developed the Helium Saver inlet as an alternative to using a different carrier gas. This GC inlet greatly reduces the amount of helium used in GC analysis. To further reduce helium consumption, this study utilized the nitrogen purge option of the Atomx. Thanks to the Atomx mass flow controller, accurate purge volumes can be achieved with nitrogen as well as helium.

The purpose of this application note is to evaluate the effectiveness of the Atomx nitrogen purge option in conjunction with Thermo Scientific's Helium Saver inlet for the analysis of EPA method 8260C[2].

Experimental Conditions

Instrument Conditions

The Atomx automated sample prep system equipped with a K trap, and a Thermo Scientific TRACE 1310 GC with Helium Saver inlet, coupled to an ISQ mass spectrometer detector (MSD) were used in this study. The conditions used in this study are reported in Tables I-III.

Table I GC Parameters	
GC:	TRACE 1310
Column	20 m x 0.18 mm x 1.00 µm TG-VMS (Part # 26080-4950)
Oven Program:	35°C, for 3 min, 14°C/min to 100°C, 25°C/min to 210°C, hold for 2 min, run time 14 min
Inlet:	Thermo Scientific Helium Saver (HeS-S/SL)
Column Flow:	1.50 mL/min
Gas:	Helium
Split Flow:	12.0 mL/min,
Purge Flow:	5 ml/min
Inlet Temp:	200°C

Table II MS Parameters	
MSD:	ISQ
Source Temp:	325°C
MS Transfer Line Temp:	230°C
Scan Range:	35-260 amu
Scan Time:	0.2 sec
Detector Gain:	$1e^{+5}$
Solvent Delay:	1.75 min

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Table III Atomx Purge and Trap Parameters			
Trap:	K Trap (Part# 14-5864-403)	Dry Purge Time:	2.00 min
Sample Volume:	5.0 mL	Dry Purge Flow:	100 mL/min
Sparge Vessel Temp:	40°C	Desorb Time:	0.50 min
Purge Gas:	Nitrogen	Desorb Temp:	250°C
Purge Time:	11.00 min	Transfer Line Temp:	140°C
Purge Flow:	40 ml/min		

Calibration

A nine point calibration curve was prepared in deionized water, ranging from 0.5-200 ppb. A mixed internal standard and surrogate standard solution was prepared in methanol and injected to a concentration of 25 ppb via the Atomx standard addition system. The preparation of standards was done according to the procedure detailed in EPA Method 8260C[2]. The TIC chromatogram of the 2 ppb calibration standard is presented in Figure 1.

Thermo Scientific's TraceFinder EFS software was used for target compound calibration. All calibrations are average response factor (Avg RF), unless otherwise noted. Those that did not meet the minimum requirements for Avg RF utilized a 1/x weighted quadratic calibration. The relative standard deviation (%RSD) and average response factor (Avg RF) is reported for each target compound, internal standard and surrogate standard in Table IV.

Method Detection Limits

Method detection limits (MDL) were determined for all 91 target compounds. Seven replicates spiked to a concentration of 0.5 ppb were used to determine the MDL of each target compound. One exception was the handling of the para and meta isomers of xylene, which were combined, and therefore analyzed at a concentration of 1 ppb. The MDL's, as well as their %RSD's are presented in Table IV.

Table IV Experimental Results

Compound	Calibration		MDL		Compound	Calibration		MDL	
	%RSD	Avg RF	(ppb)	%RSD		%RSD	Avg RF	(ppb)	%RSD
Dichlorodifluoromethane	11.80	0.21	0.15	9.70	2-Chloroethylvinyl Ether	13.91	0.21	0.13	11.04
Chloromethane	5.20	0.45	0.16	10.52	cis-1,3-Dichloropropene	6.73	0.59	0.05	3.52
Vinyl Chloride	7.30	0.50	0.08	5.40	Toluene	4.43	1.33	0.04	3.00
Bromomethane	15.70	0.35	0.19	11.20	Toluene-d8 (Surr)	0.94	1.31		
Chloroethane	14.27	0.25	0.58	20.18	2-Nitropropane	13.94	0.20	0.19	16.64
Trichlorofluoromethane	12.27	0.53	0.17	11.86	4-Methyl-2-pentanone (MIBK)	9.09	0.30	0.09	6.83
Ethyl Ether	11.84	0.44	0.14	10.66	Tetrachloroethylene	7.48	0.53	0.10	6.15
1,1-Dichloroethene	10.32	0.55	0.12	8.23	trans 1,3-Dichloropropene	7.36	0.56	0.10	7.68

Table IV Experimental Results

Compound	Calibration		MDL		Compound	Calibration		MDL	
	%RSD	Avg RF	(ppb)	%RSD		%RSD	Avg RF	(ppb)	%RSD
Carbon Disulfide	11.44	1.37	0.13	7.58	1,1,2-Trichloroethane	5.85	0.28	0.10	7.62
1,1,2-Trichlorotrifluoroethane	11.62	0.30	0.12	8.43	Ethylmethacrylate	7.87	0.63	0.07	4.96
Iodomethane	*0.99		0.18	5.47	Dibromochloromethane	10.02	0.32	0.06	4.62
Allyl Chloride	6.87	0.76	0.16	12.55	1,3-Dichloropropane	5.44	0.62	0.07	5.63
Methylene Chloride	4.91	0.63	0.09	6.95	1,2-Dibromoethane (EDB)	5.50	0.36	0.04	2.90
Methyl Acetate	9.29	0.92	0.11	7.54	Butyl Acetate	8.26	0.91	0.06	4.56
trans-1,2-Dichloroethene	8.33	0.64	0.06	4.27	2-Hexanone	4.20	0.67	0.04	2.94
Acetone	13.63	0.36	0.22	15.08	Ethylbenzene	4.99	1.42	0.07	4.26
Methyl-tert-butyl-Ether (MTBE)	7.49	1.82	0.05	3.23	Chlorobenzene	5.09	0.92	0.07	5.06
tert-Butyl Alcohol	9.37	1.09	0.11	8.49	Chlorobenzene-d5 (IS)	3.09			
Acetonitrile	8.79	0.19	0.28	10.66	1,1,1,2-Tetrachloroethane	9.76	0.81	0.06	4.48
Chloroprene	10.77	0.70	0.04	2.90	m,p-Xylene	5.83	1.18	&0.14	4.79
1,1-Dichloroethane	9.96	0.92	0.06	4.77	o-Xylene	4.99	1.24	0.06	4.14
Acrylonitrile	9.48	0.42	0.12	9.68	Styrene	5.79	1.09	0.07	5.14
Ethyl Acetate	14.24	0.04	0.15	12.80	Bromoform	12.88	0.26	0.10	7.58
Ethyl-tert-Butyl-Ether (ETBE)	8.67	1.69	0.06	4.53	Isopropylbenzene	6.72	1.39	0.07	4.75
Isobutanol	6.28	0.74	0.04	5.42	Amyl acetate	10.54	0.81	0.06	4.68
Isopropyl acetate	6.30	0.73	0.03	5.27	4-Bromofluorobenzene (Surr)	2.31	0.53		
cis-1,2-Dichloroethene	7.33	0.69	0.07	5.32	n Propylbenzene	7.33	1.60	0.10	6.75
2,2-Dichloropropane	6.50	0.69	0.11	9.28	Bromobenzene	6.52	0.86	0.07	4.88
Bromochloromethane	10.29	0.48	0.08	5.75	1,1,2,2-Tetracholorethane	6.11	0.53	0.05	4.82
Chloroform	6.38	0.82	0.05	3.63	2-Chlorotoluene	6.47	1.19	0.09	6.31
Carbon Tetrachloride	11.44	0.46	0.12	9.66	1,2,3-Trichloropropane	6.31	0.52	0.09	6.39
Tetrahydrofuran	*0.99		0.07	3.12	trans-1,4-Dichloro-2-Butene	9.25	0.93	0.05	3.95
Vinyl Acetate	9.76	1.23	0.08	6.09	4-Chlorotoluene	3.26	3.41	0.26	18.60
1,1,1-Trichloroethane	9.83	0.62	0.09	6.90	tert-Butylbenzene	5.58	3.41	0.10	7.32
Dibromofluoromethane (Surr)	1.82	0.50			Pentachloroethane	*0.98		0.18	-7.93
2-Butanone (MEK)	7.38	0.43	0.07	5.05	1,2,4-Trimethylbenzene	5.67	3.91	0.07	4.36
1,1-Dichloropropene	9.20	0.65	0.14	9.60	sec-Butylbenzene	7.68	4.54	0.08	5.22
Benzene	5.14	1.36	0.07	5.08	p-Isopropyltoluene	7.40	3.84	0.10	6.48
Propionitrile	13.15	0.20	0.20	16.09	1,3-Dichlorobenzene	2.60	2.26	0.11	6.93
Pentafluorobenzene (IS)	2.86				1,4-Dichlorobenzene	2.59	2.28	0.06	3.68
tert-Amyl Methyl Ether (TAME)	4.91	1.84	0.14	9.20	1,4-Dichlorobenzene-d4 (IS)	4.48			
1,2-Dichloroethane	8.44	0.41	0.03	2.36	n-Butylbenzene	9.74	3.30	0.10	5.90
Propyl acetate	8.95	0.94	0.04	3.29	Nitrobenzene	*0.98		0.82	-6.92

Table IV Experimental Results									
Compound	**Calibration**		**MDL**		**Compound**	**Calibration**		**MDL**	
	%RSD	**Avg RF**	**(ppb)**	**%RSD**		**%RSD**	**Avg RF**	**(ppb)**	**%RSD**
Trichloroethene	6.98	0.33	0.09	5.79	1,2-Dibromo-3-chloropropane (DBCP)	6.17	0.70	0.22	13.73
1,4-Difluorobenzene (IS)	3.35				1,2-Dichlorobenzene	3.89	2.21	0.08	5.16
Dibromomethane	6.85	0.19	0.07	5.90	Hexachlorobutadiene	8.87	0.24	0.10	6.87
1,2-Dichloropropane	5.46	0.35	0.12	9.22	1,2,4-Trichlorobenzene	7.35	1.46	0.16	9.31
Brodichloromethane	8.24	0.37	0.08	6.47	Naphthalene	9.38	5.33	0.10	7.23
Methyl Methacrylate	11.00	0.74	0.06	4.44	1,2,3 Trichlorobenzene	6.92	1.45	0.08	4.78

*Denotes r^2 value of weighted quadratic calibration

[&]Denotes MDL calculated at 1ppb.

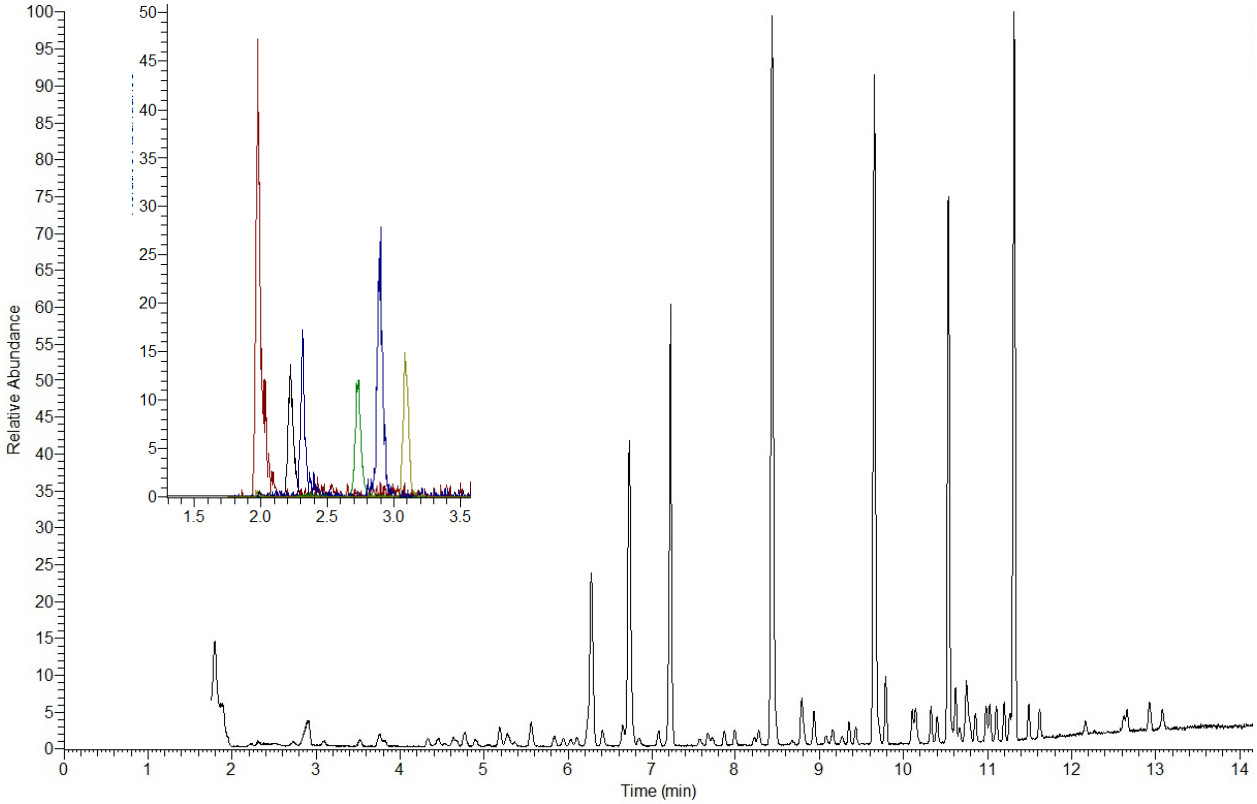

Figure 1 TIC Chromatogram for 2 ppb standard. Inset depicts the quantitation ions for the first six gases.

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Conclusion

Calibrations meeting the criteria were achieved in all but two compounds tested, representing a failure rate of 0.02%, well below the limit of 10% for method validation[2]. Excellent sensitivity was obtained for the target analytes listed.

The Atomx nitrogen purge gas feature, when combined with Thermo Scientific's Helium Saver inlet provides a substantial decrease in helium use while providing a platform that continues to meet the needs of analysts utilizing EPA Method 8260C.

References

1. Heseltine, James. January 2010. Hydrogen as a Carrier Gas for GC and GC–MS. LCGC North America. Vol 28, Issue 1.
2. US EPA Method 8260C Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) Revision 3, August 2006.

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