

Helium conservation in volatile organic compound analysis using U.S. EPA Method 8260C

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Goal

To demonstrate the effectiveness of the Thermo Scientific™ Helium Saver Module coupled with the nitrogen purge feature of the Teledyne Tekmar™ Atomx™ automated VOC sample prep system for producing quality data while reducing helium consumption.

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. Hydrogen, which can be cheaply and easily generated in the lab, is chief among these alternative carrier gases. However, as a carrier gas for GC-MS, hydrogen has several disadvantages, including its highly reactive and flammable nature.

Thermo Fisher Scientific developed the Helium Saver Module to provide 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 used the nitrogen purge option of the Teledyne Tekmar Atomx automated VOC sample prep system. Thanks to the Atomx system mass flow controller, accurate purge volumes can be achieved with nitrogen as well as helium.

Here, we evaluate the effectiveness of a system that combines the Atomx nitrogen purge option with the Helium Saver Module for VOC analysis using U.S. EPA method 8260C. EPA method 8260C is one of the most widespread methods for the analysis of VOCs. One of the techniques recommended by the method for the sampling and introduction of the analytes is purge and trap. Purge and trap coupled with GC-MS enables the greatest potential selectivity and sensitivity for the analysis.

Experimental

Recommended instrument conditions

TRACE 1310 GC system

Injection volume:	1 µL
Liner:	Splitless w/glass wool (P/N 453A1925)
Carrier gas:	Helium
Column type:	Thermo Scientific™ TraceGOLD™ TG-VMS 20 m, 0.18 mm, 1 µm (P/N 260804950)
Column oven:	Ready delay 1 min, initial temperature 35 °C, hold 3 min, ramp 14 °C/min to 100 °C, ramp 25 °C/min to 210 °C, hold 2 min
Helium Saver injector:	200 °C; split with a split flow of 12 mL/min, helium delay 0.1 min
Column flow:	Constant flow at 1.5 mL/min

ISQ LT Mass spectrometer

Ion source type:	Thermo Scientific™ ExtractaBrite™
Ionization mode:	El, 70 eV
Source temperature:	325 °C
Transfer line temperature:	230 °C

FS Conditions

Acquisition start	Mass range	Dwell time
1.75	35–260	0.2

Atomx purge and trap parameters

Trap	K Trap	Dry purge time	2.00 min
Sample volume	5.0 mL	Dry purge flow	100 mL/min
Spurge vessel	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		

Methods

This analysis used the Thermo Scientific™ ISQ™ LT Single Quadrupole GC-MS system* connected to the Thermo Scientific™ TRACE™ 1310 gas chromatograph. The system was equipped with a Thermo Scientific™ Instant Connect Helium Saver Module with a splitless liner (P/N 453A1925). The column used was a TraceGOLD TG-VMS 20 m, 0.18 mm, 1 µm column (P/N 26080-4950).

The analysis was run in full-scan mode. Samples were acquired and quantified using the Thermo Scientific™ TraceFinder™ Environmental and Food Safety (EFS) software. The Atomx automated sample prep system was equipped with a K trap.

Results and discussion

A nine-point calibration curve was prepared in deionized water, ranging from 0.5 to 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 U.S. EPA Method 8260C. The TIC chromatogram of the 2 ppb calibration standard is presented in Figure 1.

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 Avg RF is reported for each target compound, internal standard, and surrogate standard in Table 1.

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 MDLs, as well as their %RSDs are presented in Table 1.

*Equivalent or better performance with the Thermo Scientific™ ISQ™ 7000 single quadrupole GC-MS system

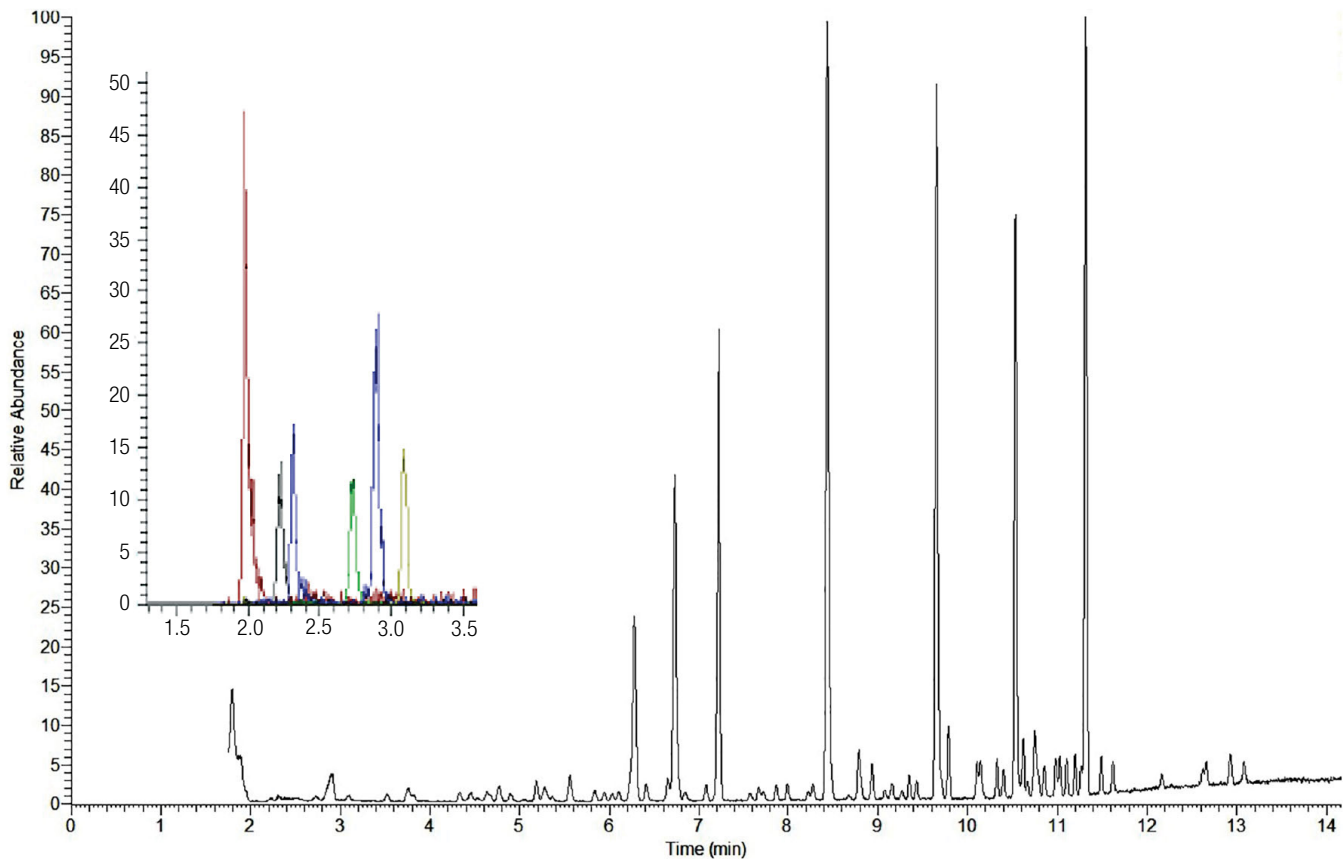


Figure 1. Chromatogram with VOC standard at 2 ppb in full scan mode. The inset depicts the extracted ions for the first six gases.

Table 1. Calibration and MDL results.

Compound	Calibration		MDL	
	%RSD	Avg RF	(ppb)	%RSD
Carbon Disulfide	11.44	1.37	0.13	7.58
1,1,2-Trichlorotrifluoroethane	11.62	0.3	0.12	8.43
Iodomethane	*0.99		0.18	5.47
Allyl Chloride	6.87	0.76	0.16	12.55
Methylene Chloride	4.91	0.63	0.09	6.95
Methyl Acetate	9.29	0.92	0.11	7.54
trans-1,2-Dichloroethene	8.33	0.64	0.06	4.27
Acetone	13.63	0.36	0.22	15.08
Methyl-tert-butyl-Ether (MTBE)	7.49	1.82	0.05	3.23
tert-Butyl Alcohol	9.37	1.09	0.11	8.49
Acetonitrile	8.79	0.19	0.28	10.66
Chloroprene	10.77	0.7	0.04	2.9
1,1-Dichloroethane	9.96	0.92	0.06	4.77
Acrylonitrile	9.48	0.42	0.12	9.68
Ethyl Acetate	14.24	0.04	0.15	12.8
Ethyl-tert-Butyl-Ether (ETBE)	8.67	1.69	0.06	4.53

Table 1. Calibration and MDL results (continued).

Compound	Calibration		MDL	
	%RSD	Avg RF	(ppb)	%RSD
Isobutanol	6.28	0.74	0.04	5.42
Isopropyl acetate	6.3	0.73	0.03	5.27
cis-1,2-Dichloroethene	7.33	0.69	0.07	5.32
2,2-Dichloropropane	6.5	0.69	0.11	9.28
Bromochloromethane	10.29	0.48	0.08	5.75
Chloroform	6.38	0.82	0.05	3.63
Carbon Tetrachloride	11.44	0.46	0.12	9.66
Tetrahydrofuran	*0.99		0.07	3.12
Vinyl Acetate	9.76	1.23	0.08	6.09
1,1,1-Trichloroethane	9.83	0.62	0.09	6.9
Dibromofluoromethane (Surr)	1.82	0.5		
2-Butanone (MEK)	7.38	0.43	0.07	5.05
1,1-Dichloropropene	9.2	0.65	0.14	9.6
Benzene	5.14	1.36	0.07	5.08
Propionitrile	13.15	0.2	0.2	16.09
1,1,2-Trichloroethane	5.85	0.28	0.1	7.62
Ethylmethacrylate	7.87	0.63	0.07	4.96
Dibromochloromethane	10.02	0.32	0.06	4.62
1,3-Dichloropropane	5.44	0.62	0.07	5.63
1,2-Dibromoethane (EDB)	5.5	0.36	0.04	2.9
Butyl Acetate	8.26	0.91	0.06	4.56
2-Hexanone	4.2	0.67	0.04	2.94
Ethylbenzene	4.99	1.42	0.07	4.26
Chlorobenzene	5.09	0.92	0.07	5.06
Chlorobenzene-d5 (IS)	3.09			
1,1,1,2-Tetrachloroethane	9.76	0.81	0.06	4.48
m,p-Xylene	5.83	1.18	**0.14	4.79
o-Xylene	4.99	1.24	0.06	4.14
Styrene	5.79	1.09	0.07	5.14
Bromoform	12.88	0.26	0.1	7.58
Isopropylbenzene	6.72	1.39	0.07	4.75
Amyl acetate	10.54	0.81	0.06	4.68
4-Bromofluorobenzene (Surr)	2.31	0.53		
n-Propylbenzene	7.33	1.6	0.1	6.75
Bromobenzene	6.52	0.86	0.07	4.88
1,1,2,2-Tetrachloroethane	6.11	0.53	0.05	4.82
2-Chlorotoluene	6.47	1.19	0.09	6.31
1,2,3-Trichloropropane	6.31	0.52	0.09	6.39
Pentafluorobenzene (IS)	2.86			
tert-Amyl Methyl Ether (TAME)	4.91	1.84	0.14	9.2
1,2-Dichloroethane	8.44	0.41	0.03	2.36
Propyl acetate	8.95	0.94	0.04	3.29
Trichloroethene	6.98	0.33	0.09	5.79

Table 1. Calibration and MDL results (continued).

Compound	Calibration		MDL	
	%RSD	Avg RF	(ppb)	%RSD
1,4-Difluorobenzene (IS)	3.35			
Dibromomethane	6.85	0.19	0.07	5.9
1,2-Dichloropropane	5.46	0.35	0.12	9.22
Bromodichloromethane	8.24	0.37	0.08	6.47
Methyl Methacrylate	11	0.74	0.06	4.44
1,4-Dichlorobenzene	2.59	2.28	0.06	3.68
1,4-Dichlorobenzene-d4 (IS)	4.48			
n-Butylbenzene	9.74	3.3	0.1	5.9
Nitrobenzene	*0.98		0.82	-6.92
1,2-Dibromo-3-chloropropane (DBCP)	6.17	0.7	0.22	13.73
1,2-Dichlorobenzene	3.89	2.21	0.08	5.16
Hexachlorobutadiene	8.87	0.24	0.1	6.87
1,2,4-Trichlorobenzene	7.35	1.46	0.16	9.31
Naphthalene	9.38	5.33	0.1	7.23
1,2,3 Trichlorobenzene	6.92	1.45	0.08	4.78

*Denotes r^2 value of weighted quadratic calibration; **denotes MDL calculated at 1 ppb.

Conclusions

Calibrations meeting the criteria of U.S. EPA Method 8260C were achieved in all but two compounds tested, representing a failure rate of 0.02%, well below the limit of 10% for method validation. Excellent sensitivity was obtained for the target analytes listed.

The system including a Helium Saver Module combined with the Atomx system nitrogen purge gas inlet provides a substantial decrease in helium use while providing a platform that continues to meet the needs of analysts utilizing U.S. EPA Method 8260C.

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