APPLICATION NOTE

EPA 8260: Analysis of Volatile Organic Compounds by GC-MS

AN034v1; SCION Instruments

Introduction

The United States Environmental Protection Agency (US EPA) was established in 1970 with the aim to protect human health and the environment. Since then environmental contamination has been at the forefront of government policy and regulation through US EPA methods for the analysis of environmental pollutants.

EPA 8260 is the standard method for the analysis of volatile organic compounds (VOCs) in ground water and solid waste by purge and trap (P&T) gas chromatography with mass spectrometry (GC-MS). EPA 8260 is a comprehensive method with more than 100 VOCs in the target compound list. The method is used to identify and quantify VOCs, with a boiling point <200°C, in a variety of solid waste matrices, regardless of water content.

The SCION Single Quad (SQ) mass spectrometer has a unique feature, Compound Based Scanning (CBS), for easy automated setup and optimisation of complex mixed mode methods. CBS makes use of libraries that store all the essential information about a compound such as retention time, time window, qualifier and quantifier ions. Compounds are loaded directly into a method, scan times are optimised with data acquisition and processing tables synchronised. Managing large number of SIM acquisitions is made easy in mixed mode.

This application note describes the analytical operating conditions for analysis of US EPA 8260 including Bromofluorobenzene (BFB) tune parameters and calibration details.

Experimental

The SCION 8500-GC coupled with the SCION 8700 SQ MS and Tekmar Atomx XYZ P&T sample concentrator was used to achieve a highly automated and robust solution for VOC analysis. The system can be visualized in Figure 1.

Calibration curves were generated using five multi level calibration samples ranging from 0.5 to 200 ppb (µg/L), with four internal standards held at a constant concentration. The purge and trap and GC-MS parameters are listed in Tables 1 and 2. The purge and trap conditions for EPA 8260 come factory installed on the Atomx.

Figure 1. SCION Instruments 8500 GC equipped with a 8700 SQ MS.

Table 1. Analytical conditions of the Atom XYZ Purge and Trap.

Variable	Value	Variable	Value
Valve Oven	140˚C	Bake Flow	200 mL/min
Transfer Line Temp	140˚C	Sample Preheat Time/Temp	1min/45˚C
Sample Mount Temp	90˚C	Purge Time/Flow/Desorb Preheat Temp	11 min, 40 ml/min, 245˚C
Pre-purge Flow	40 mL/min	Desorb Time/Temp	2 min, 245˚C
Condenser Purge Temp	20˚C	Desorb Flow	100 mL/min
Bake time/Temp	2 min, 280˚C	Condenser Bake Temp	200˚C

Table 2. Analytical conditions of the SCION GC-MS.

Variable	Value
Injector	S/SL, 1:100, 180˚C
Carrier	1mL/min
Column	SCION-624MS 30mm x 0.25mm x 1.4µm
Oven	40˚C (2 mins), 10˚C/min to 170˚C (1min), 50˚C/min to 240˚C (2mins)
Scan range	50-300 m/z
Manifold temp	60˚C

Results

EPA Method 8260 specifies that the MS must be tuned via analysis of a BFB standard. The SCION SQ was tuned to meet these requirements for spectral resolution of BFB using target ion ratio tuning; built directly into the software. All acceptance criteria was met, thus passing specification as detailed in Table 3.

Table 2. BFB acceptance criteria and obtained values.

m/z	Acceptance criteria	Value
50	15-40% of mass 95	23.0
75	30-60% of mass 95	52.4
95	Base Peak	100
96	5-9% of mass 95	6.1
173	<2% of mass 174	0.5
174	>50% of mass 95	70.0
175	5-9% of mass 174	6.1
176	>95% but <101% of mass 174	97.3
177	5-9% of mass 176	6.1

Figure 2 details the chromatogram of the 120ppb calibration standard whilst Table 4 details peak identification of the calibration standard along with retention times and linearity values.

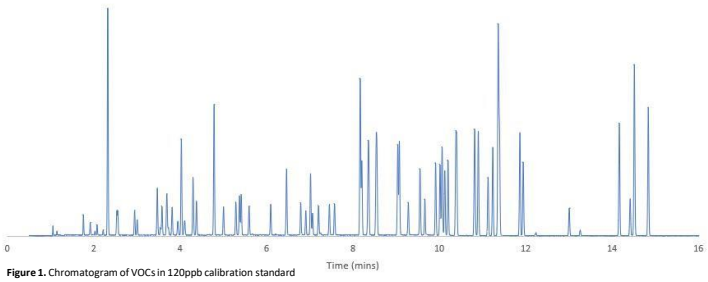

Figure 2. Chromatogram of VOCs in 120 ppb calibration standard.

Table 4. Peak identification, retention time and linearity of target compounds.

Compound	RT	R^2
1,1-Dichloroethene	1.949	0.9995
Iodo-methane	2.057	0.9997
Carbon Disulphide	2.109	0.9999
Allyl Chloride	2.110	0.9991
Methylene Chloride	2.351	0.9999
2-propenenitrile	2.555	0.9996
1,2-dichloroethylene	2.582	0.9998
1,1-dichloroethane	2.968	0.9999
2-chloro-1,3-butadiene	3.028	0.9998
t-1,2-dichloroethane	3.489	0.9995
Propanenitrile	3.559	0.9982
Bromochloro-methane	3.698	0.9995
Chloroform	3.818	0.9999
1,1,1-trichloroethane	3.962	0.9995
Carbon Tetrachloride	4.108	0.9991
1,1-dichloropropene	4.122	0.9995
Benzene	4.313	1
1,2-dichloroethane	4.390	0.9992
Trichoroethylene	5.014	1
1,2-dichloropropane	5.298	0.9999
Dibromomethane	5.382	0.9997
Methyl methacrylate	5.418	0.9998
Bromodichloromethane	5.601	0.9997
Cis-1,3-dichloropropene	6.101	0.9999
Toluene	6.468	1
Methacrylic acid, ethyl ester	6.910	0.9999
1,1,2-trichloroethane	7.022	0.9998
Tetrachloroethylene	7.068	0.9999
Tetrachloroethene	7.068	1
1,3-dichloropropane	7.206	0.9999
Dibromochloromethane	7.456	0.9998
1,2-dibromomethane	7.576	1
Chlorobenzene	8.207	0.9999
1,1,1,2-tetrachloroethane	8.351	0.9998
Ethylbenzene	8.548	0.9999
m,p-xylene	8.548	0.9998

Compound	RT	R^2
o-xylene	9.039	0.9999
Styrene	9.076	0.9999
Bromoform	9.282	0.9996
Isopropylbenzene	9.554	0.9996
Bromobenzene	9.917	0.9999
1,1,2,2-tetrachloroethane	10.018	0.9998
1,2,3-trichloropropane	10.065	0.9997
1,4-dichloro-2-butene	10.065	0.9998
N-propylbenzene	10.129	0.9999
2-chlorotoluene	10.129	0.9998
4-chorotoluene	10.383	0.9998
1,3,5-trimethylbenzene	10.403	0.9996
tert-butylbenzene	10.818	0.9995
Pentachloro-ethane	10.830	0.9999
1,2,4-trimethylbenzene	10.906	0.9999
Sec-butylbenzene	11.130	0.9999
4-isopropyltoluene	11.375	0.9996
1,2-dichlorobenzene	11.396	0.9998
1,3-dichlorobenzene	11.396	0.9998
1,4-dichlorobenzene	11.868	0.9998
N-butylbenzene	11.943	0.9999
1,2-dibromo-3-chloropropane	13.007	0.9999
Nitrobenzene	13.262	0.9998
1,2,4-trichlorobenzene	14.169	0.9999
Hexachlorobutadiene	14.420	0.9998
Naphthalene	14.518	0.9995
1,2,3-trichlorobenzene	14.841	0.9998

Conclusions

The Tekmar Atomx XYZ purge and trap sample concentrator coupled with a SCION 8500 GC and SCION 8700 SQ Mass Spectrometer is a total solution for EPA VOC methods. The method is easily setup for both full scan and SIM methods using the unique Mass Spec Work Station software. Excellent repeatability, recovery and linearity of the VOCs demonstrate the excellent performance of the SCION system, under EPA 8260 specifications.

Order Information

Ordering Information for the 8300 GC	
Part	**Part Number**
SCION 8500 GC + SCION 8700 SQ PREMIUM EI ONLY (120V)	SCIONSQ85PRE531
SCION 8500 GC + SCION 8700 SQ PREMIUM EI ONLY (230V)	SCIONSQ85PRE532
SCION 8500 GC + SCION 8700 SQ PREMIUM EI+CI (120V)	SCIONSQ85PRC531
SCION 8500 GC + SCION 8700 SQ PREMIUM EI+CI (230V)	SCIONSQ85PRC531

Suggested Consumables	
Part	**Part number**
15% Graphite/85% Vespel Ferrule 1/16" with 0.4 mm hole pk/10	41312148
BTO Septa 9 mm, pk/50	CR298713
SCION-624MS 30mm x 0.25mm x 1.4µm	SC32591

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