BEYOND MEASURE

SCION INSTRUMENTS

A Complete Solution for the Analysis of Volatile Organic Compounds (VOCs) in Water

Application Note AN0015

INTRODUCTION

Demand for lower detection limits of volatile organic compounds in drinking water requires the use of a mixed mode GC-MS system. The ability for simultaneous full scan and selection ion monitoring (SIM) is vital for low-level quantitation. The United State Environmental Protection Agency (US EPA) 524.3 method details the analytical method for the measurement of purgeable organic compounds in water by capillary gas chromatography with mass spectrometry.

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.

EXPERIMENTAL

The SCION 456-GC coupled with the SCION SQ mass spectrometer and Tekmar Atomx XYZ Purge and Trap sample concentrator was used to achieve a highly automated and robust solution for VOC analysis.

Calibration standards were prepared at 0.1, 0.5, 1,2,5,10,20 and 40ppb with the method preservative. Standards at lower concentrations were required for SIM, typically in the range of 5-100ppt. A 5mL sample size was used for the analysis as determined by US EPA 524.3.

The purge and trap and GC parameters are listed in Table 1 and 2. The purge and trap conditions for drinking water come factory installed on the Atomx.

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

Variable	Value	Variable	Value
Valve Oven/ Transfer Line Temp	150°C	Sample Preheat Time/ Temp	1 min/40°C
Transfer Line Temp	150°C	Purge Time/Flow	11 min, 40mL/min
Sample Mount Temp	60°C	Desorb Preheat Temp	245°C
Pre-purge Flow	40mL/min	Desorb Time/Temp	1 min, 250°C
Condenser Purge Temp	20°C	Desorb Flow	100mL/min
Bake Time/Temp	7 min, 260°C	Condenser Bake Temp	200°C
Bake Flow	300mL/min		

Table 2. Analytical conditions of the SCION GC and MS

Variable	Value
Injector	S/SL, 1:100
Column	SCION 624-MS 20m x 0.25mm x 1.0µm
Oven	35°C (2min), 10°C/min to 170°C, 50°C/min to 240°C
Scan Range	35-300m/z
Emission	15-20µA
Manifold Temp	60°C

RESULTS

The SCION SQ was tuned to meet the requirements for spectral resolution for Bromofluorobenzene (BFB), using target ion ratio tuning (built directly into the software). All acceptance criteria was exceeded so therefore passed.

Using CBS, a mixed-mode method was created by importing all target compounds from a library containing all of the associated SIM ions. Compound graphs can be created, based upon the retention time and retention time window. CBS optimised the placement of SIM ions throughout the run for maximum sensitivity and optimal dwell times.

The calibration standards were injected and calibration curves were generated. Calibration correlation coefficient for Method 524.3 analytes were 0.9995 respectively. t-butyl alcohol (TBA) is usually a poor compound to analyse by purge and trap. However, Figure 1 shows the calibration curve for TBA and it's excellent response using the SCION analyser whereas Table 3 shows calibration statistics for the analytes in the EPA method. Precision and accuracy were determined after the completion of the validated calibration.

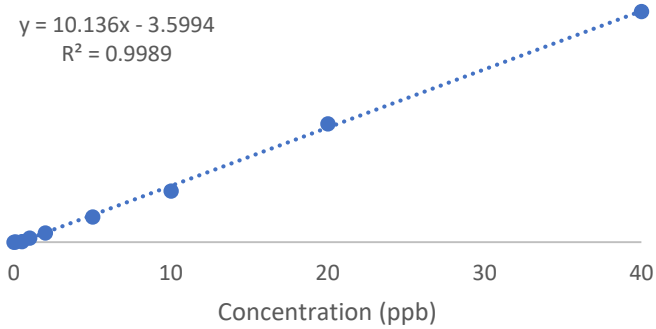

Fig 1. TBA calibration curve 0.1-40ppb

Demonstration of capability includes the analysis of VOCs in water with a minimum of seven replicates. Table 3 also provides a summary of the precision and accuracy data as well as the method detection limit (MDL), for ten replicates. All compounds analysed easily met the requirements, with an average recovery of 99.27% and %RSD of 4.49. Method 524.3 states that the recovery must be within 70-150%; with all compounds passing this criteria.

Table 3. Calibration statistics of selected compounds (n=10)

Compound	R^2	%RSD	MDL (ppb)	Recovery (%)
4-chlorotoluene	1	6.77	0.037	93
1,3,5-trimethylbenzene	0.9999	6.52	0.034	93
tert-butylbenzene	0.9997	7.01	0.043	100
1,2,4-trimethylbenzene	0.9998	7.44	0.028	89
sec-butylbenzene	0.9998	8.68	0.067	95
1,3-dichlorobenzene	1	6.48	0.044	93
p-isopropyltoluene	0.9999	8.32	0.066	91
1,4-dichlorobenzene	0.9999	6.52	0.022	91
1,2-dichlorobenzene	1	6.96	0.042	95
n-butylbenzene	0.9998	7.77	0.056	94
DBCP	0.9982	4.57	0.088	97
1,2,4-trichlorobenzene	0.9998	5.82	0.058	99
Hexachlorobutadiene	0.9998	8.85	0.078	99
Napthalene	0.9996	5.25	0.035	86
1,2,3-trichlorobenzene	1	6.97	0.031	93
Dichlorodifluoromethane	0.9964	8.78	0.026	89
Chloromethane	0.9990	5.62	0.044	84
Vinyl Chloride	0.9985	3.70	0.037	94
Bromomethane	0.9972	2.93	0.051	90
Chloroethane	0.9982	5.38	0.056	85
Trichlorofluoromethane	0.9981	5.40	0.064	92
1,1-dichloroethane	0.9998	3.15	0.028	94
Methylene Chloride	0.9995	3..14	0.024	128
cis-1,2-dichloroethane	0.9999	2.13	0.037	117
trans-1,2-dichloroethane	0.9996	1.16	0.062	113
bromochloromethane	0.9998	2.50	0.027	117
chloroform	0.9999	2.72	0.040	103
1,1,1-trichloroethane	0.9996	2.78	0.056	101
carbon tetrachloride	0.9994	6.21	0.050	97
1,1-dichloropropane	0.9992	4.20	0.049	103
benzene	0.9999	2.45	0.036	106
trichloroethane	0.9998	3.16	0.039	106
1,2-dichloropropane	0.9998	3.13	0.054	119
1,2-dibromomethane	0.9999	1.96	0.034	111
bromodichloromethane	0.9998	2.62	0.045	105
trans-1,3-dichloropropane	0.9995	1.76	0.022	109
toluene	0.9997	3.72	0.036	98
cis-1,3-dichloropropane	0.9997	1.94	0.024	117
1,1,2-dichloroethane	0.9999	1.56	0.059	96
tetrachloroethane	0.9997	5.72	0.067	98
1,3-dichloropropane	0.9999	2.13	0.034	97
Dibromochloromethane	0.9988	2.32	0.022	96
EDB	0.9999	1.73	0.032	91
chlorobenzene	0.9999	2.87	0.029	99
1,1,1,2-tetrachloroethane	0.9997	3.25	0.036	100
ethylenebenzene	0.9999	3.57	0.041	98
m,p-xylene	0.9998	4.22	0.048	98
o-xylene	0.9999	3.50	0.030	101
Styrene	0.9998	2.44	0.036	95
bromoform	0.9999	1.49	0.027	111
isopropylbenzene	0.9998	5.13	0.052	100
bromobenzene	0.9999	5.79	0.029	100
1,1,2,2-tetrachloroethane	0.9995	4.55	0.038	96
1,2,3-trichloroproane	0.9998	3.33	0.032	108
n-propylbenzene	0.9998	5.99	0.036	96
2-chlorotoluene	1	7.39	0.035	94
t-butyl alcohol	0.9986	5.15	0.038	135

Table 4 shows the data obtained for 1,2-dibromoethane when the individual calibration points were calculated against the calibration curve.

Table 4. Recovery of 1,2-dibromoethane

Target Conc. (ppb)	Result	% Recovery
0.5	0.56	111
1	1.03	103
2	1.85	92
5	4.71	94
10	9.67	97
20	20.26	101
40	40.42	101

CONCLUSION

The Tekmar Atomx XYZ purge and trap sample concentrator coupled with the SCION 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, with all requirements of EPA Method 524.3 being exceeded.

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