



# THE ANALYSIS OF DRINKING WATER FOR VOLATILE ORGANIC COMPOUNDS BY GC-MS



The United States Environmental Protection Agency Method (US EPA) 524.3 describes the analytical method for 'measurement of purgeable organic compounds in water by capillary column gas chromatography/mass spectrometry'. EPA 524.3 is a method widely used with a purge and trap system to determine over 70 volatile organic compounds (VOCs) in drinking water to ensure water supplies are safe for consumption.

Table 1. Analytical conditions of the instrumentation

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

Demand for lower detection limits of VOCs 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 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.

The SCION 456-GC coupled with the SCION SQ MS and Tekmar Atomx XYZ Purge and Trap sample concentrator was used to

achieve a highly automated and robust solution for the analysis of VOCs in drinking water, to EPA 524.3 specification. Table 1 details the analytical conditions of the Atomx Purge and Trap and SCION GC-MS.

The SCION SQ was tuned to meet the requirements for the spectral resolution of Bromofluorobenzene (BFB) using target ion ratio tuning (built directly into the mass spectrometer software; mass spec work station MSWS). All acceptance criteria were exceeded so passed specifications, requirements of the method. Using CBS, a mixed-mode method was created by importing all target compounds from a library containing all of the associated SIM ions. Table 2 details the tune report for BFB including acceptance criteria and obtained values.

Table 2. BFB acceptance criteria and obtained values

m/z	Acceptance Criteria	Value
50	15-40% of mass 95	23.0
75	30-80% 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

Calibration standards were prepared at 0.1, 0.5, 1, 2, 5, 10, 20 and 40ppb. Standards at lower concentrations were required for the SIM acquisition; typically, in a range of 5-100ppt. A 5mL sample size was used for the analysis as specified in EPA 524.3. The purge and trap conditions for EPA 524.3 come factory installed on the Atomx XYZ system. Figure 1 shows the chromatogram of the 5ppb calibration standard in full scan mode.

Post calibration standard analysis, calibration curves were generated in the MSWS software. The calibration correlation coefficient for EPA 524.3 analytes were 0.9995. t-butyl alcohol (TBA) is usually poor compound to analyse on a purge and trap system. However, Figure 2 shows the calibration curve for TBA with an excellent R2 of 0.999.

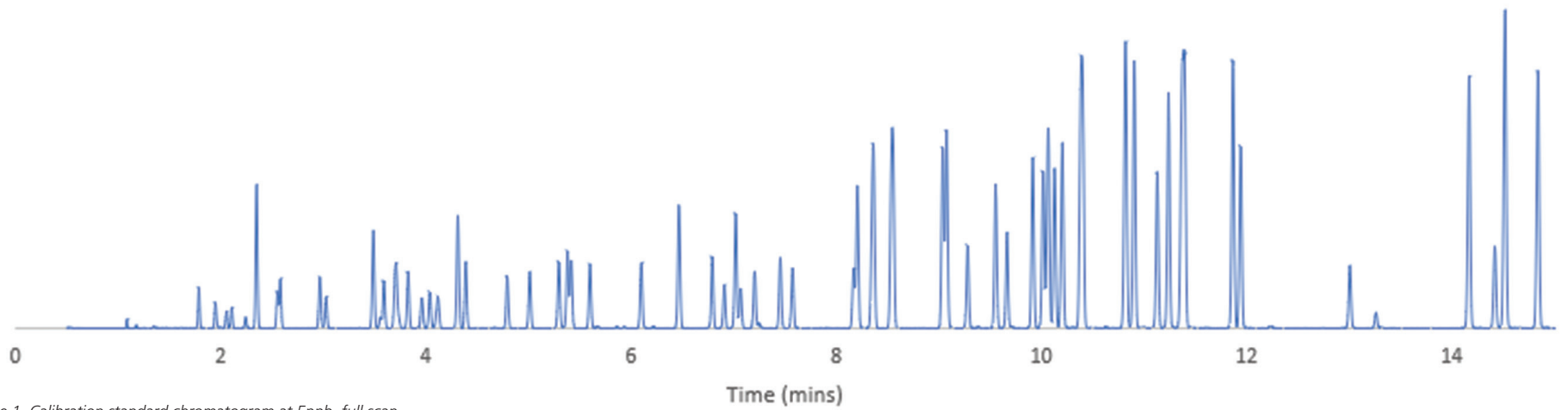


Figure 1. Calibration standard chromatogram at 5ppb, full scan.

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

Compound	R2	%RSD	MDL (ppb)	Recovery (%)
4-chlorotoluene	1	6.7	0.037	93
1,3,5-trimethylbenzene	0.999	6.5	0.034	93
tert-butylbenzene	0.999	7.0	0.043	100
1,2,4-trimethylbenzene	0.999	7.4	0.028	89
sec-butylbenzene	0.999	8.6	0.067	95
1,3-dichlorobenzene	1.000	6.4	0.044	93
p-isopropyltoluene	0.999	8.3	0.066	91
1,4-dichlorobenzene	0.999	6.5	0.022	91
1,2-dichlorobenzene	1.000	6.9	0.042	95
n-butylbenzene	0.999	7.7	0.056	94
DBCP	0.998	4.5	0.088	97
1,2,4-trichlorobenzene	0.999	5.8	0.058	99
Hexachlorobutadiene	0.999	8.8	0.078	99
Napthalene	0.999	5.2	0.035	86
1,2,3-trichlorobenzene	1.000	6.9	0.031	93
Dichlorodifluoromethane	0.996	8.7	0.026	89
Chloromethane	0.999	5.6	0.044	84
Vinyl Chloride	0.999	3.7	0.037	94
Bromomethane	0.997	2.9	0.051	90
Chloroethane	0.998	5.3	0.056	85
Trichlorofluoromethane	0.998	5.4	0.064	92
1,1-dichloroethane	0.999	3.1	0.028	94
Methylene Chloride	0.999	3.1	0.024	128
cis-1,2-dichloroethane	0.999	2.1	0.037	117
trans-1,2-dichloroethane	0.999	1.1	0.062	113
bromochloromethane	0.999	2.5	0.027	117
chloroform	0.999	2.7	0.040	103
1,1,1-trichloroethane	0.999	2.7	0.056	101
carbon tetrachloride	0.999	6.2	0.050	97
1,1-dichloropropane	0.999	4.2	0.049	103
benzene	0.999	2.4	0.036	106
trichloroethane	0.999	3.1	0.039	106
1,2-dichloropropane	0.999	3.1	0.054	119
1,2-dibromomethane	0.999	1.9	0.034	111
bromodichloromethane	0.999	2.6	0.045	105
trans-1,3-dichloropropane	0.999	1.7	0.022	109
toluene	0.999	3.7	0.036	98
cis-1,3-dichloropropane	0.999	1.9	0.024	117
1,1,2-dichloroethane	0.999	1.5	0.059	96
tetrachloroethane	0.999	5.7	0.067	98
1,3-dichloropropane	0.999	2.1	0.034	97
Dibromochloromethane	0.999	2.3	0.022	96
EDB	0.999	1.7	0.032	91
chlorobenzene	0.999	2.8	0.029	99
1,1,1,2-tetrachloroethane	0.999	3.2	0.036	100
ethylenebenzene	0.999	3.5	0.041	98
m,p-xylene	0.999	4.2	0.048	98
o-xylene	0.999	3.5	0.030	101
Styrene	0.999	2.4	0.036	95
bromoform	0.999	1.4	0.027	111
isopropylbenzene	0.999	5.1	0.052	100
bromobenzene	0.999	5.7	0.029	100
1,1,2,2-tetrachloroethane	0.999	4.5	0.038	96
1,2,3-trichloropropane	0.999	3.3	0.032	108
n-propylbenzene	0.999	5.9	0.036	96
2-chlorotoluene	1.000	7.3	0.035	94
t-butyl alcohol	0.999	5.1	0.038	125

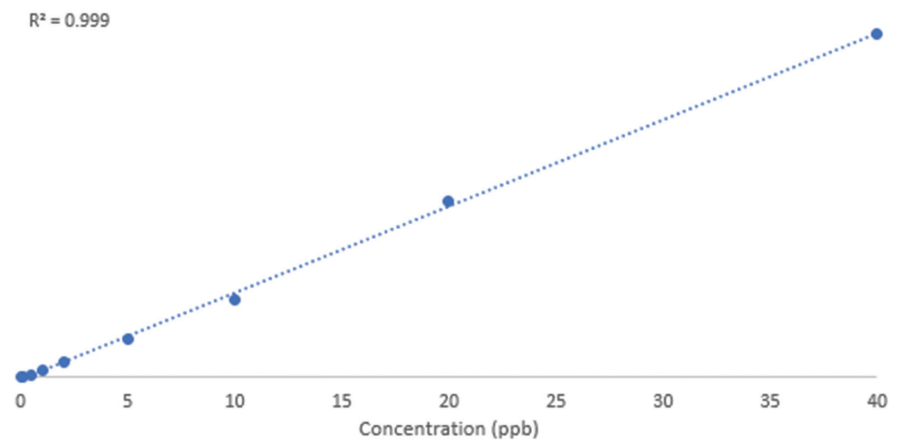


Figure 2. TBA calibration curve 0.1ppb to 40ppb

The robustness of the analytical system was tested through replicate analysis of VOCs in water with a minimum of seven replicates. Linearity, repeatability, MDL and the recovery of each target compound can be found in Table 3. All replicates analysed met the method requirements; RSD % must be below 20% with recovery of analytes being between 70% and 130%. MDL was measured in full scan mode at 0.1ppb.

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 demonstrates the excellent performance of the SCION system, will all requirements of EPA Method 524.3 being exceeded.

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