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Application Note SI-02393

EPA Method 524.2: Determination of Volatile Organic Compounds in Drinking Water by Capillary Column GC/MS Using the 210-MS Ion Trap and V:Results™ GC/MS software

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Introduction

EPA Method 524.2, "Measurement of Purgeable Organic Compounds in Water by Capillary Column Gas Chromatography/Mass Spectrometry", is a general-purpose method for the identification and simultaneous measurement of purgeable volatile organic compounds in surface water, groundwater, and drinking water. This note provides assistance for chemists to rapidly set up the 210-MS Ion Trap GC/MS system for the analysis of 60 standard purgeable volatile organic compounds in water by EPA Method 524.2.

Initial Calibration

EPA Method 524.2 does not specify the calibration levels or ranges to be used. Both linear (5 levels minimum) and quadratic fitting (6 levels minimum) are allowed with a minimum correlation coefficient of 0.99. Calibration levels of 0.2, 0.5, 1, 2, 5, 10, 20, 50, and 100 ppb were used in this analysis. There are no minimum response factors for select compounds in the method. The %RSD of the initial calibration for each analyte should be less than 20% if a linear regression or relative response factors are used to calculate results.

Purge-and-Trap and GC Conditions

Column: FactorFour™ VF-624ms 20 m × 0.15 mm × 0.84 μm (Part No. CP9100)
GC Conditions: 35 °C for 2 min, to 200 °C at 10 °C/min, hold 0 min, to 240 °C at 50 °C/min, hold 0 min
Purge Volume: 20 mL at 40 mL/min
Split Ratio: 1:200

MS Conditions

Target TIC: 12000 counts
Scans Averaged: 2 μs
Max Ion Time: 25000 μs
Emission Current: 15-20 μA
Manifold Temp: 60 °C
Transfer line Temp: 250 °C
Ion Trap Temp: 160 °C



Figure 1. Varian Archon™ Purge and Trap AutoSampler with Tekmar Stratum concentrator (top) and 210-MS Ion Trap GC/MS (bottom).

Instrumentation

- Varian 210-MS Ion Trap Mass Spectrometer with 431-GC Gas Chromatograph
- Varian 1177 Split/Splitless Injector with 4 mm open liner
- Varian Archon™ Purge and Trap AutoSampler
- Tekmar Stratum concentrator with #9 StraTrap
- V:Results GC/MS software

Results and Discussion

Figure 2 shows volatile gases at 0.2 ppb, extracted ion chromatograms (EIC). Excellent peak shape and sensitivity are easily obtained with the system.

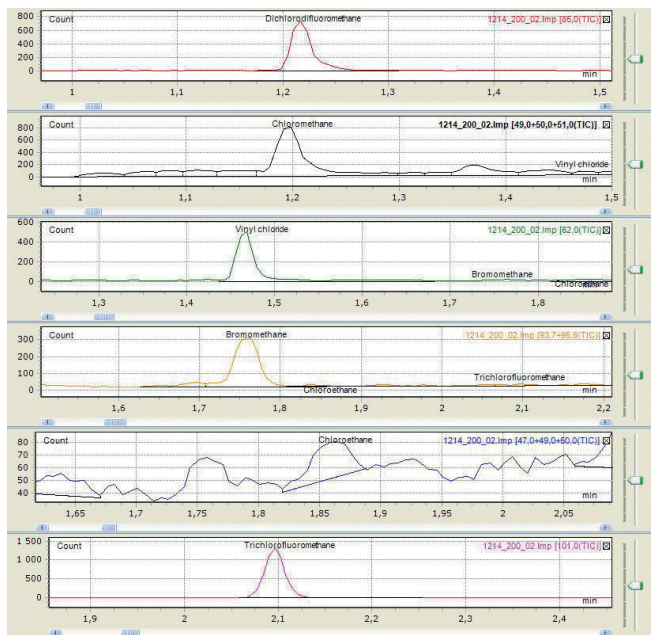


Figure 2. Extracted ions for the six volatile gases in US EPA 524.2. Concentration is 0.2 ppb with 20 mL purge volume and 1:200 split.

Table 1. Calibration data for 59 volatile compounds from 0.2 ppb to 100 ppb, 20 mL purge volume.

Compound Name	Correlation Coefficient (r^2)	Average RF	%RSD
Dichlorodifluoromethane	0.9954	0.7324	13.54
Chloromethane*	0.9918	0.0969	13.86
Vinyl chloride	0.9958	0.5687	11.15
Bromomethane	0.9932	0.6668	6.96
Chloroethane	0.9981	0.0981	16.91
Trichlorofluoromethane	0.9992	1.5098	9.43
1,1-Dichloroethene	0.9959	0.6471	7.74
Methylene chloride	0.9991	0.3716	14.69
cis-1,2-dichloroethene	0.9984	0.6032	3.89
1,1-Dichloroethane	0.9979	0.5087	26.25
2,2-Dichloropropane	0.9987	0.3555	11.91
trans-1,2-dichloroethene	0.9976	1.1094	5.00
Bromochloromethane	0.9980	0.3991	5.71
Chloroform	0.9954	0.6914	8.69
1,1,1-Trichloroethane	0.9975	0.9644	3.87
Carbon Tetrachloride	0.9990	0.4738	5.86
1,1-Dichloropropene	0.9959	0.3034	5.92
Benzene	0.9958	0.8214	5.15
1,2-Dichloroethane	0.9974	0.5662	8.38
Trichloroethene	0.9965	0.3059	5.61
1,2-Dichloropropane	0.9966	0.2868	7.24
Dibromomethane	0.9961	0.3400	9.24
Bromodichloromethane	0.9983	0.5066	7.06
trans-1,3-dichloropropene	0.9961	0.3639	6.03

Compound Name	Correlation Coefficient (r^2)	Average RF	%RSD
Toluene	0.9999	1.9304	6.10
cis-1,3-dichloropropene	0.9958	0.3040	4.28
1,1,2-trichloroethane	0.9999	0.2509	9.20
Tetrachloroethane	0.9998	0.2924	9.16
1,3-dichloropropane	0.9995	0.2319	4.58
Dibromochloromethane	0.9990	0.3412	5.10
1,2-Dibromoethane	0.9997	0.2726	3.75
Chlorobenzene	0.9999	1.0771	4.20
1,1,1,2-Tetrachloroethane	0.9984	0.4292	7.86
Ethylbenzene	0.9987	2.1615	4.65
m,p-Xylene	0.9990	3.8453	4.90
o-Xylene	0.9993	1.9260	3.54
Styrene	0.9997	1.0937	4.70
Bromoform	0.9998	0.1752	6.25
Isopropylbenzene	0.9994	1.6598	3.76
Bromobenzene	0.9993	0.5062	5.22
1,1,2,2-Tetrachloroethane	0.9998	0.2743	10.78
1,2,3-Trichloropropane	0.9984	0.1834	6.72
n-Propylbenzene	0.9989	2.3621	6.51
2-Chlorotoluene	0.9991	0.4980	5.09
4-Chlorotoluene	0.9894	0.4574	9.47
1,3,5-Trimethylbenzene	0.9995	1.9294	3.29
tert-Butylbenzene	0.9988	1.5751	6.39
1,2,4-Trimethylbenzene	0.9978	1.9094	4.13
sec-Butylbenzene	0.9987	2.0434	5.85
1,3-Dichlorobenzene	0.9993	1.7268	4.56
p-Isopropyltoluene	0.9998	3.5297	4.83
1,4-Dichlorobenzene	0.9994	1.7076	6.49
1,2-Dichlorobenzene	0.9991	1.5205	4.71
n-Butylbenzene	0.9999	3.8631	6.77
1,2-Dibromo-3-chloropropane	0.9998	0.4284	22.39
1,2,4-Trichlorobenzene	0.9996	1.0888	4.32
Hexachlorobutadiene	0.9991	0.8379	8.20
Naphthalene	0.9991	1.5656	7.01
1,2,3-Trichlorobenzene	0.9996	0.9087	4.62
Average	0.9981	0.9864	7.45

* Chloromethane was measured from 2 to 100 ppb.

All compounds showed excellent calibration coefficients and relative standard deviations at a concentration range from 0.2 to 100 ppb (Table 1). The average r^2 and %RSD of all compounds were 0.998 and 7.45%, respectively.

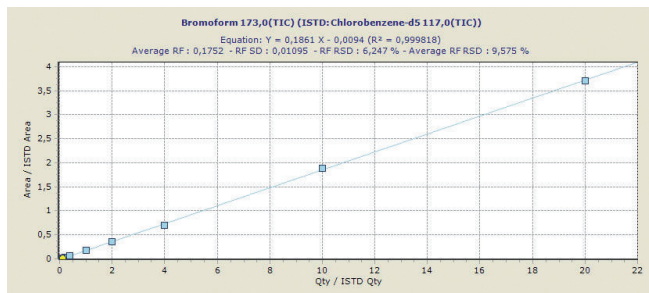


Figure 3. Calibration curve for bromoform, typically a very reactive purge-and-trap compound, from 0.2 to 100 ppb on the 210-MS.

Conclusion

The Varian 210-MS system with the Varian Archon™ AutoSampler and Tekmar Stratum purge-and-trap concentrator showed excellent linearity and %RSD over the calibration range. The entire GC/MS system is proven to meet and exceed the performance required by US EPA method 524.2 and validated to meet all of the QC criteria outlined in the method.

These data represent typical results.

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