Application Note SI-02391

EPA Method 525.2: Determination of Semi-Volatile Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column GC/MS Using the Varian 210-MS Ion Trap and V:Results™ GC/MS software

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Introduction

US EPA Method 525.2, "Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/Mass Spectrometry," is one of the most commonly used methods for semi-volatile organic compound analysis in drinking water. The method lists over 100 compounds, but usually only a subset of the listed target compounds are monitored in any measurement. This note provides a basic overview of 25 key regulated compounds rapidly set up on the 210-MS Ion Trap GC/MS system for the analysis of water samples by EPA Method 525.2.

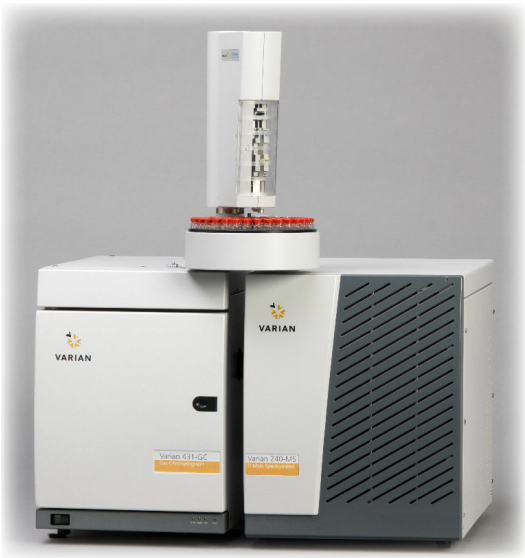

Figure 1. Varian 210-MS Ion Trap Mass Spectrometer with 431-GC.

Instrumentation

- Varian 210-MS Ion Trap Mass Spectrometer with 431-GC Gas Chromatograph
- Varian 1177 Split/Splitless Injector with Siltek frit insert (Part No. RT210462145)
- 8400 AutoSampler
- V:Results GC/MS software

Initial Calibration

Calibration solutions of 0.05, 0.1, 0.2, 0.5, 1, 2, 5, and 10 ppm are used for all the analytes. The %RSD for each analyte should be less than 30% in order to use mean response factors or linear regression fitting for calculating results. Quadratic calculations may not be used in the EPA 525.2 method.

GC Conditions

Column:	FactorFour™ VF-5ms 30 m × 0.25 mm x 0.25 µm (Part No. CP8944)
GC Conditions:	70 °C for 1.5 min, to 200 °C at 10 °C/min, to 270 °C at 5 °C/min, and to 300 °C at 10 °C/min

MS Conditions

Target TIC:	12000 counts
Scan Range:	45-450 *m/z*
Max Ion Time:	25000 µs
Emission Current:	15 µA
Manifold Temp:	80 °C
Transfer line Temp:	280 °C
Ion Trap Temp:	210 °C

Results and Discussion

A typical total ion chromatogram (TIC) and extracted ion chromatogram (EIC) are shown in Figures 2 and 3.

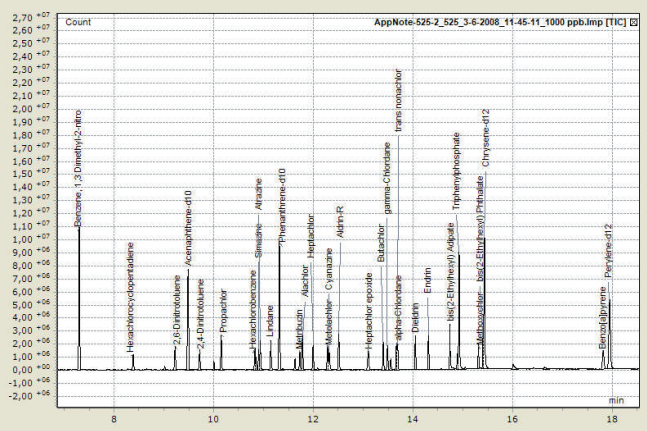

Figure 2. TIC at 1 ppm of regulated 525.2 mixture, 210-MS.

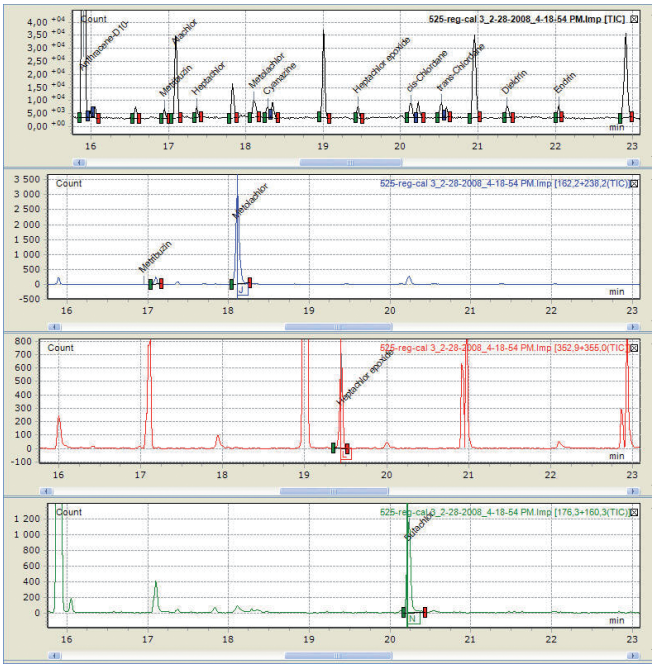

Figure 3. EIC of select compounds at 0.05 ppm.

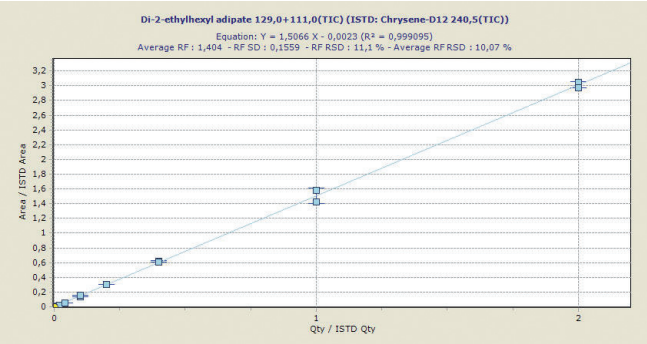

Figure 4. Calibration of Di-2-ethylhexyl adipate from 0.05 to 10 ppm.

All compounds showed excellent calibration coefficients and relative standard deviations at concentrations ranging from 0.05 to 10 ppm (Table 1), even for very challenging compounds, such as Di-2-ethylhexyl adipate (Figure 4). The average r^2 and % RSD of all 25 regulated compounds was 0.9972 and 8.71% respectively.

Table 1. Calibration data of 25 regulated compounds in US EPA Method 525.2.

Compound Name	Correlation Coefficient (r^2)	Average RF	%RSD
Hexachlorocyclopentadiene	0.9996	1.237	3.03
Benzene, 2-methyl-1,3-dinitro-	0.9990	0.775	5.82
Benzene, 1-methyl-2,4-dinitro-	0.9979	1.212	6.90
Benzene, 2,4-dimethyl-1-nitro-	0.9959	1.478	8.80
Benzene, hexachloro-	0.9987	0.367	7.23
Simazine	0.9961	0.635	10.75
Atrazine	0.9869	1.631	17.44
Lindane	0.9996	1.872	5.47
Metribuzin	0.9979	0.722	6.18
Alachlor	0.9978	0.417	14.24
Heptachlor	0.9972	0.356	5.00
Metolachlor	0.9964	2.405	5.80
Cyanazine	0.9992	0.257	9.65
Aldrin	0.9990	0.113	9.23
Heptachlor epoxide	0.9991	0.457	5.47
cis-Chlordane	0.9987	0.381	5.57
Butachlor	0.9964	0.760	10.65
trans Chlordane	0.9993	0.691	5.64
trans Nonachlor	0.9985	0.214	6.99
Dieldrin	0.9978	0.291	11.21
Endrin	0.9975	0.117	6.23
Di-2-ethylhexyl adipate	0.9991	1.404	11.10
Methoxychlor*	0.9871	0.694	23.94
Di-2-ethylhexyl phthalate	0.9963	1.946	8.52
Benzo[a]pyrene	0.9978	0.967	6.83
Average	0.9972	0.856	8.71

* Methoxychlor was measured from 0.05 to 0.5 ppm.

The method detection limits (MDLs) of these 25 compounds were calculated based on the standard deviation of nine replicates at 0.05 ppm multiplied by Student's t at 99% confidence level. The results are listed in Table 2.

Table 2. MDLs based on nine replicates at 0.050 ppm.

Compound Name	Average Amount (ppm)	%RSD	MDL (ppm)
Hexachlorocyclopentadiene	0.0566	3.03	0.0076
Benzene, 2-methyl-1,3-dinitro-	0.0895	5.82	0.0061
Benzene, 1-methyl-2,4-dinitro-	0.1173	6.90	0.0072
Benzene, 2,4-dimethyl-1-nitro-	0.0521	8.80	0.0094
Benzene, hexachloro-	0.0537	7.23	0.0084
Simazine	0.0592	10.75	0.0132
Atrazine	0.0626	17.44	0.0172
Lindane	0.0527	5.47	0.0073
Metribuzin	0.0472	6.18	0.0109
Alachlor	0.0958	14.24	0.0067
Heptachlor	0.1185	5.00	0.0076
Metolachlor	0.0110	5.80	0.0130
Cyanazine	0.0390	9.65	0.0158
Aldrin	0.0392	9.23	0.0087
Heptachlor epoxide	0.0044	5.47	0.0058
cis-Chlordane	0.0324	5.57	0.0053
Butachlor	0.0487	10.65	0.0170
trans Chlordane	0.0377	5.64	0.0087
trans Nonachlor	0.0698	6.99	0.0103
Dieldrin	0.1042	11.21	0.0232
Endrin	0.0688	6.23	0.0053
Di-2-ethylhexyl adipate	0.0202	11.10	0.0158
Methoxychlor	0.0525	23.94	0.0103
Di-2-ethylhexyl phthalate	0.0270	8.52	0.0182
Benzo[a]pyrene	0.0184	6.83	0.0151
Average	0.055	8.71	0.011

Conclusion

The Varian 210-MS Ion Trap GC/MS system showed excellent sensitivity and linearity for the 25 regulated analytes. The entire GC/MS system is proven to produce the required sensitivity and robustness that meets all of the QC criteria outlined in the method.

These data represent typical results.
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