EPA 525.2: Trace Level Determination of Semi-Volatile Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column GC/MS Using the 240-MS Ion Trap and V:Results™ GC/MS software

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Introduction

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 compound analysis in drinking water. This note provides a basic overview of 25 regulated compounds analyzed on the 240-MS GC/MS system for the analysis of water samples by EPA Method 525.2. The 240-MS provides the ultimate in sensitivity, easily exceeding the required detection limits cited in the method. The system can also be configured in both the internal or external ionization mode for added flexibility.

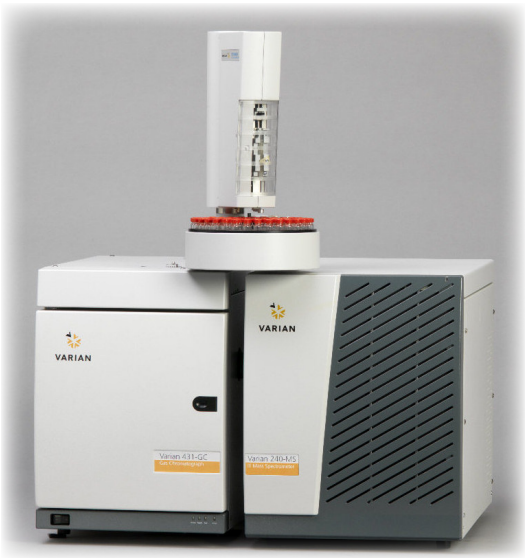

Figure 1. Varian 240-MS Ion Trap Mass Spectrometer with 431-GC.

Instrumentation

- Varian 240-MS Ion Trap Mass Spectrometer
- Varian 431-GC Gas Chromatograph
- Varian 1177 Split/Splitless Injector with Siltek frit insert (Part No. RT210462145)
- 8400 AutoSampler
- V:Results GC/MS software

GC Conditions

Column: FactorFour™ VF-5ms 30 m × 0.25 mm x 0.25 µm (Part No. CP8944)
Program: 45 °C for 1.5 min, to 240 °C for 2 min at 20 °C/min, to 300 °C for 4 min at 40 °C/min

MS Conditions

MS Configuration:	Internal ionization
Target TIC:	6000 µs
Scan Range:	45-450 m/z
Max Ion Time:	25000 µs
Emission Current:	20 µA
Manifold Temp:	50 °C
Transfer line Temp:	280 °C
Ion Trap Temp:	220 °C

Results and Discussion

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

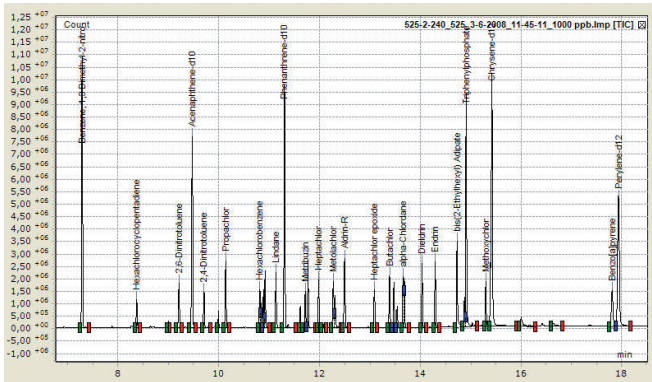

Figure 2. TIC at 1 ppm of regulated 525.2 mixture, 240-MS internal.

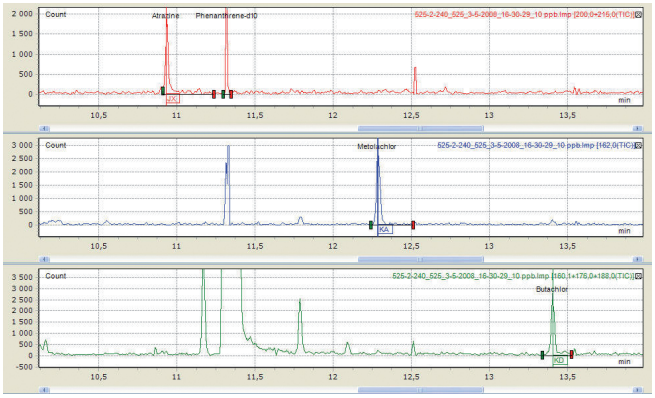

Figure 3. EIC of select compounds at 0.01 ppm.

Initial Calibration

Calibration solutions of 0.01, 0.02, 0.05, 0.1, 0.2, 0.5, 1, 2, 5 and 10 ppm were prepared for all the analytes. The %RSD for each analyte should be less than 30% in order to use the mean response factors or linear regression fitting for calculating results. Quadratic calculations may not be used in EPA 525.2.

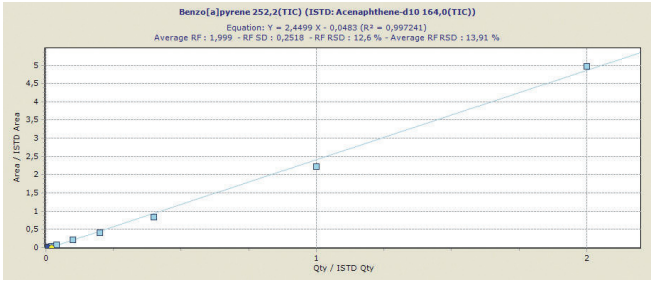

Figure 4. Calibration of benzo(a)pyrene from 0.01 to 10 ppm.

All compounds meet the method QC criteria. They showed excellent calibration coefficient and relative standard deviation at concentrations ranging from 0.01 to 10 ppm (Table 1), even for late eluting compounds such as benzo[a] pyrene (Figure 4). The average r^2 and %RSD of all 25 regulated compounds are 0.9975 and 14.58%, respectively.

Table 1. Calibration data of 25 regulated compounds.

Compound Name	Correlation Coefficient (r^2)	Average RF	%RSD
Hexachlorocyclopentadiene	0.9988	0.6588	11.06
2,6-Dinitrotoluene	0.999	0.7087	12.97
2,4-Dinitrotoluene	0.9997	0.6845	16.34
Propachlor	0.9953	2.1286	12.65
Hexachlorobenzene	1.0000	0.5952	6.71
Simazine	0.9925	0.3924	11.08
Atrazine	0.9957	0.6690	16.17
Lindane	0.9999	1.7157	10.46
Metribuzin	0.9979	0.5000	13.6
Alachlor	0.9996	0.8753	9.53
Heptachlor	0.9985	0.7339	8.98
Metolachlor	0.9995	1.2759	8.63
Cyanazine	0.9958	0.5948	13.48
Aldrin-R	0.9941	0.9424	14.94
Heptachlor epoxide	0.9980	0.3972	8.72
Butachlor	0.9990	1.1007	11.55
gamma-Chlordane	0.9982	0.5831	14.45
alpha-Chlordane	0.9997	0.8379	9.02
trans Nonachlor	0.9997	0.2656	9.15
Dieldrin	0.9995	1.3265	8.92
Endrin	0.9996	0.1338	46.59
bis(2-Ethylhexyl) adipate	0.9963	3.2848	17.38
Methoxychlor	0.9924	1.3232	29.65
bis(2-Ethylhexyl) phthalate	0.9912	1.7660	29.84
Benzo[a]pyrene	0.9972	1.9991	12.6
Average	0.9975	1.0197	14.58

Method detection limits (MDLs) of these 25 compounds were calculated based on the standard deviation of seven replicates at 0.01 ppm multiplied by Student's t at 99% confidence level (Table 2).

Table 2. MDL replicates at 0.01 ppm.

Compound Name	Average Amount (ppm)	%RSD	MDL (ppm)
Hexachlorocyclopentadiene	0.0100	7.52	0.002372
2,6-Dinitrotoluene	0.0100	14.48	0.004568
2,4-Dinitrotoluene	0.0088	14.78	0.004087
Propachlor	0.0145	3.94	0.00179
Hexachlorobenzene	0.0126	21.21	0.008421
Simazine	0.0170	7.48	0.003996
Atrazine	0.0166	6.47	0.003372
Lindane	0.0133	6.07	0.002537
Metribuzin	0.0123	12.54	0.004859
Alachlor	0.0087	5.19	0.001418
Heptachlor	0.0115	13.49	0.004862
Metolachlor	0.0100	8.03	0.002522
Cyanazine	0.0100	26.48	0.008337
Aldrin-R	0.0098	11.45	0.003514
Heptachlor epoxide	0.0087	10.85	0.002957
Butachlor	0.0110	15.95	0.005505
gamma-Chlordane	0.0076	8.74	0.002097
alpha-Chlordane	0.0082	6.76	0.001739
trans Nonachlor	0.0091	13.47	0.003864
Dieldrin	0.0113	16.10	0.005729
Endrin	0.0108	28.40	0.009107
bis(2-Ethylhexyl) adipate	0.0116	9.07	0.003296
Methoxychlor	0.0048	14.22	0.002135
bis(2-Ethylhexyl) phthalate*	0.0267	9.30	0.007796
Benzo[a]pyrene	0.0098	11.26	0.003484
Average	0.0100	12.13	0.0042

*bis(2-Ethylhexyl) phthalate is at 0.05 ppm

Conclusion

The Varian 240-MS showed excellent sensitivity and linearity for all 25 regulated analytes. The 240-MS has the best full scan sensitivity of all GC/MS products on the market. V:Results GC/MS software allowed for automatic calculation of the MDLs. The average MDL of all 25 analytes is 3.5 ppb, significantly lower than that required in the EPA method.

These data represent typical results.
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