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Application Note SI-02361

EPA 8270D: Determination of Semi-Volatile Organic Compounds in Waste Matrices by GC/MS Using the Varian 210-MS Ion Trap and V:Results™ GC/MS Software

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Introduction

EPA Method 8270D Semi-Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) is a commonly used method for semi-volatile compound analysis in a variety of waste matrices, including solid waste, air, water, and soil. The method lists over 200 compounds that can be analyzed, but only a subset of the listed target compounds are monitored in a given measurement. The method also refers to a variety of sample preparation and clean-up methods. This note describes a method to rapidly set up the 210-MS GC/MS system for the analysis of 64 semi-volatile compounds using EPA 8270D.



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

Materials and Methods

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)
- Varian 8400 AutoSampler
- V:Results GC/MS software

Initial Calibration

EPA 8270D does not specify the calibration levels or ranges to be used. Both linear (five levels minimum) and quadratic fitting (six levels minimum) are allowed with a minimum correlation coefficient of 0.99. Calibration solutions of 1, 2, 5, 10, 20, 50, 80, 120, and 160 ppm were used in this analysis. The average %RSD for all of the analytes should be less than 15%, and the %RSD of the calibration check compounds must be below 30%.

GC Conditions

Column: FactorFour™ VF-5ms, 30 m x 0.25 mm,

(Part no: CP8809)

Program: 40 °C hold for 2 min, to 220 °C at

10 °C/min, hold 0 min, to 270 °C, hold for 1 min, to 320 °C at 15 °C/min, hold for

3.87 min

Pressure Pulse: 40 psi for 0.4 min

Split Ratio: 20:1

MS Conditions

Target TIC: 6000 counts Scans Averaged: 3 μ scans Max Ion Time: 25000 μ s Emission Current: 15 μ A Manifold Temp: 60 °C Transfer Line Temp: 280 °C Ion Trap Temp: 210 °C

Results and discussion

A typical total ion chromatogram is shown in Figure 2.

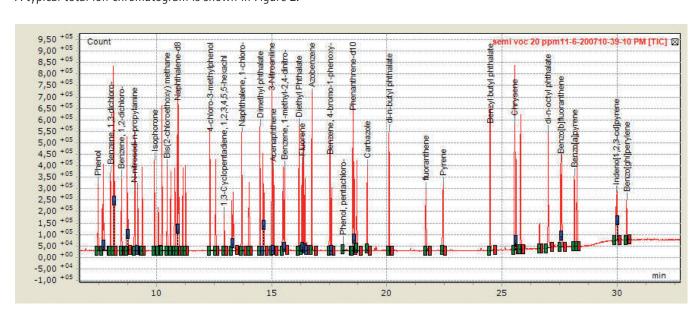


Figure 2. Total ion chromatogram of a 20 ppm calibration standard of 64 semi-volatile compounds for EPA 8270D on a Varian 210-MS. Internal standards are at 40 ppm.

All compounds showed good correlation coefficients and relative standard deviation at a concentration range from 1 to 160 ppm (Table 1). The average r^2 and %RSD of all compounds were 0.997 and 10.94%, respectively.

The calibration check compounds had average %RSD of 14.4%, easily passing the method criteria of 30%. The average %RSD of all compounds was 10.9%, again significantly better than the method criteria of 15%.

The system performance check compounds were evaluated based on average RF. Table 2 shows RFs over the concentration range of 1 to 160 ppm.

Table 1. System performance check compounds' comparison of average RF.

Compound	870D minimum criteria	Average RF	
N-nitroso di-n-propylamine	0.050	0.651	
Hexachlorocyclopentadiene	0.050	0.848	
Phenol, 2,4-dinitro-	0.050	0.062	
4-Nitrophenol	0.050	1.013	

Conclusion

The Varian 210-MS Ion Trap Mass Spectrometer 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 8270D. The V:Results GC/MS software QC features permitted automatic validation of the method, meeting all the outlined QC criteria.

Table 2. Calibration data of semi-volatile compounds.

Compound	Correlation coefficient (r²)	Average RF	%RSD	Calibration check compound
1,4-Dichlorobenzene-d4				
Naphthalene-d8	1			
Acenaphthene-d10]			
Phenanthrene-d10	1	Internal sta	ndards	
Chrysene-d12	1			
Perylene-d12	[
N-nitroso dimethylamine	0.990	0.374	14.68	
Phenol	0.999	1.743	6.55	PASS
Bis(2-chloroethyl) ether	0.999	1.035	8.63	
Phenol, 2-chloro-	0.999	1.781	3.80	
Benzene, 1,3-dichloro-	0.997	0.720	7.90	
Benzene, 1,4-dichloro-	0.998	0.728	8.42	
Benzene, 1,2-dichloro-	0.995	0.704	7.90	PASS
o-Cresol	0.999	1.418	3.29	
Bis(2-chloroisopropyl) ether	0.997	1.036	5.74	
N-nitroso di-n-propylamine	0.999	0.651	4.54	
p-Cresol	0.999	1.973	5.65	
Ethane, hexachloro-	0.996	0.331	9.37	
Benzene, nitro-	0.996	1.253	10.57	
Isophorone	0.998	2.124	7.02	
Phenol, 2-nitro-	0.999	0.270	12.91	PASS
Phenol, 2,4-dimethyl-	0.997	1.564	7.20	
Bis(2-chloroethoxy) methane	0.998	1.266	5.03	
Phenol, 2,4-dichloro-	0.997	0.407	10.01	PASS
Benzene, 1,2,4-trichloro-	0.995	0.406	5.97	
Naphthalene	0.999	3.010	5.32	
p-Chloroaniline	0.997	1.207	14.65	
Hexachlorocyclopentadiene	0.999	0.848	9.40	
4-Chloro-3-methylphenol	0.999	3.178	4.87	PASS
Naphthalene, 1-methyl-	0.999	0.810	6.77	
1,3-Cyclopentadiene, 1,2,3,4,5,5-hexachloro	0.992	0.456	9.62	
Phenol, 2,4,6-trichloro-	0.994	0.541	14.19	PASS
Phenol, 2,4,5-trichloro-	0.998	0.691	14.11	
Naphthalene, 1-chloro-	0.998	1.897	7.33	
2-Nitroaniline	0.996	0.969	7.47	
Dimethyl phthalate	0.998	3.594	4.78	
2,6-Dinitro-toluene	1.000	0.736	9.58	
Acenaphthylene	1.000	1.921	6.37	
Acenaphthene	0.999	1.401	6.76	PASS
Phenol, 2,4-dinitro-	1.000	0.062	56.19	
4-Nitrophenol	1.000	1.013	13.84	
Dibenzofuran	0.999	2.564	5.80	
Benzene, 1-methyl-2,4-dinitro-	0.998	1.055	15.79	
Diethyl Phthalate	0.999	3.418	25.12	PASS
Fluorene	1.000	1.553	4.82	

Compound	Correlation coefficient (r²)	Average RF	%RSD	Calibration check compound
4-Chlorophenyl phenyl ether	0.997	1.802	6.71	
4-Nitroaniline	0.995	1.993	6.76	
3-Nitroaniline	0.999	2.168	26.67	PASS
Phenol, 2-methyl-4,6-dinitro-	0.999	0.228	48.07	
Azobenzene	0.997	3.580	5.39	
Benzene, 4-bromo-1-phenoxy-	0.996	0.711	5.55	
Hexachlorobenzene	0.994	0.216	12.03	
Phenol, pentachloro-	0.999	0.330	45.18	PASS
Phenanthrene	1.000	1.221	4.01	
Anthracene	0.999	1.201	4.46	
Carbazole	0.999	1.205	7.16	
Di-n-butyl phthalate	0.995	2.974	9.66	
Fluoranthene	0.999	1.286	4.94	PASS
Pyrene	1.000	1.217	3.11	
Benzyl butyl phthalate	0.999	1.334	8.35	
Chrysene	0.997	1.316	7.11	
Benz[a]anthracene	0.998	1.134	6.31	
Bis(2-ethylhexyl) phthalate	0.993	2.025	8.88	PASS
Di-n-octyl phthalate	0.999	3.396	5.99	
Benzo[b]fluoranthene	0.994	1.451	13.16	
Benzo[k]fluoranthene	0.997	1.426	12.72	PASS
Benzo[a]pyrene	0.997	1.402	14.35	
Indeno[1,2,3-cd]pyrene	0.997	0.766	15.03	
Dibenz[a,h]anthracene	0.995	0.718	17.31	
Benzo[ghi]perylene	0.995	0.604	13.46	
Average	0.997		10.94	

These data represent typical results.

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