Application Note SI-02334

EPA Method 8260B: Volatile Organic Compounds by GC/MS Using the Varian 210-MS Ion Trap Mass Spectrometer and V:Results™ GC/MS Software

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Introduction

EPA Method 8260B *Volatile Organic Compounds by Gas Chromatography Mass Spectrometry (GC/MS)* is used to determine volatile organic compounds in a variety of solid-waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air-sampling-trapping media, ground and surface water, aqueous sludges, caustic liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments. This note assists in the rapid setup of the 210-MS GC/MS system for the analysis of standard volatiles by EPA Method 8260B.

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

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
- Teledyne Tekmar Stratum concentrator with #9 Trap
- V:Results GC/MS software

Initial Calibration

EPA Method 8260B 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 0.5, 1, 2, 5, 10, 20, 50 and 100 ppb were used in this analysis. The %RSD for each analyte should be less than 15%, and the %RSD of the calibration check compounds (CCCs) must be below 30%.

Conditions

Purge-and-Trap and GC Conditions

Column:	FactorFour™ VF-624ms, 20 m x 0.15 mm x 0.84 µm (Part No. CP9100)
Program:	35 °C for 2 min, to 200 °C at 10 °C/min, to 240 °C at 50 °C/min, hold 0 min
Purge Vol:	5 mL at 40 mL/min
Split Ratio:	1:100

MS Conditions

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

Results and Discussion

Figure 1 shows volatile gases at 0.5 ppb, extracted ion chromatograms (EIC) using the V:Results™ GC/MS software. Excellent peak shape and sensitivity are easily obtained with the system.

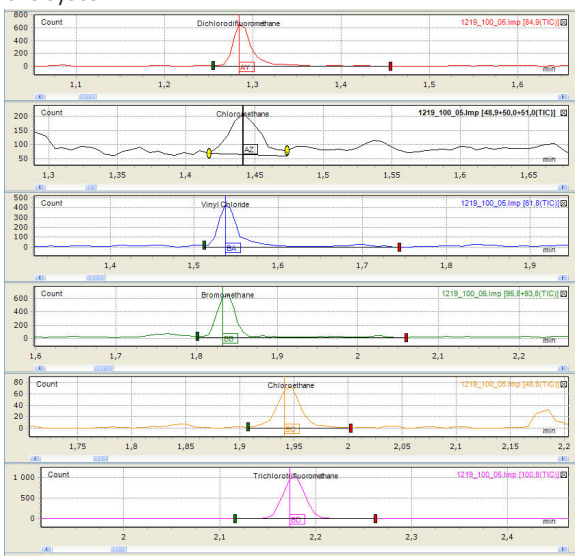

Figure 2. Extracted ions for the six volatile gases in US EPA 8260B using the V:Results™ GC/MS software. Concentration was 0.5 ppb with 5 mL purge volume and 1:100 split.

Table 1. Calibration data for 60 volatile organic chemicals.

Compound Name	"Correlation Coefficient (r^2)"	Average RF	%RSD	CCC
Dichlorodifluoromethane	0.9712	0.4451	22.2	
Chloromethane	0.9975	0.1126	9.7	PASS
Vinyl chloride	0.9937	0.4752	14.5	
Bromomethane	0.9967	0.4850	26.2	
Chloroethane	0.9991	0.0723	8.1	
Trichlorofluoromethane	0.9908	1.2628	16.4	
1,1-Dichloroethene	0.9968	0.5673	7.9	PASS
Methylene chloride	0.9969	0.3458	16.1	
cis-1,2-dichloroethene	0.9985	0.5548	5.6	
1,1-Dichloroethane	0.9997	0.6662	7.6	PASS
2,2-Dichloropropane	0.9962	0.3336	11.3	
trans-1,2-dichloroethene	0.9985	1.1310	3.4	
Bromochloromethane	0.9991	0.4912	7.2	
Chloroform	0.9982	0.7033	3.1	
1,1,1-Trichloroethane	0.9981	0.9254	4.1	
Carbon Tetrachloride	0.9979	0.4441	8.0	
1,1-Dichloropropene	0.9994	0.2919	7.6	
Benzene	0.9999	0.8558	4.7	
1,2-Dichloroethane	0.9998	0.7632	4.1	
Trichloroethene	0.9971	0.2904	5.5	
1,2-Dichloropropane	0.9997	0.3029	3.2	
Dibromomethane	0.9993	0.5030	4.6	
Bromodichloromethane	1.0000	0.5901	4.9	
trans-1,3-dichloropropene	0.9992	0.4388	3.9	
Toluene	0.9995	1.9545	3.6	
cis-1,3-dichloropropene	0.9989	0.4302	5.8	
1,1,2-trichloroethane	0.9993	0.3639	4.6	
Tetrachloroethene	0.9936	0.2658	9.8	
1,3-dichloropropane	0.9989	0.3377	3.6	
Dibromochloromethane	0.9983	0.4832	6.1	
1,2-Dibromoethane	0.9972	0.4525	7.2	
Chlorobenzene	0.9995	1.0519	3.3	PASS
1,1,1,2-Tetrachloroethane	0.9979	0.4459	4.7	
Ethylbenzene	0.9979	2.1928	4.1	
m,p-Xylene	0.9980	3.9660	4.4	
o-Xylene	0.9993	2.0490	3.5	
Styrene	0.9995	1.1206	5.2	
Bromoform	0.9984	0.3021	8.5	PASS
Isopropylbenzene	0.9970	1.6806	5.1	
SS-4-Bromofluorobenzene	Not computed	0.4761	5.0	
Bromobenzene	0.9996	0.5420	3.4	
1,1,2,2-Tetrachloroethane	0.9974	0.5474	4.7	PASS
1,2,3-Trichloropropane	0.9962	0.3835	9.5	
n-Propylbenzene	0.9968	2.4631	6.2	
2-Chlorotoluene	0.9992	0.4988	5.3	
4-Chlorotoluene	0.9946	0.4897	6.7	
1,3,5-Trimethylbenzene	0.9974	2.1027	5.2	
tert-Butylbenzene	0.9955	1.6213	6.0	
1,2,4-Trimethylbenzene	0.9973	2.1188	5.6	
sec-Butylbenzene	0.9948	2.1303	6.6	
1,3-Dichlorobenzene	0.9967	1.5603	4.5	
p-Isopropyltoluene	0.9977	3.0818	5.2	
1,4-Dichlorobenzene	0.9946	1.6067	5.0	
1,2-Dichlorobenzene	0.9980	1.5397	4.2	
n-Butylbenzene	0.9974	3.5415	7.7	
1,2-Dibromo-3-chloropropane	0.9977	0.8055	9.0	
1,2,4-Trichlorobenzene	0.9993	1.2560	2.8	
Hexachlorobutadiene	0.9881	0.6973	13.5	
Naphthalene	0.9996	2.9235	8.0	
1,2,3-Trichlorobenzene	0.9997	1.2521	2.8	
Mean	**0.9972**		**6.9**	**6.9**

All compounds showed excellent correlation coefficients and relative standard deviation at a concentration range from 0.5 to 100 ppb (Table 1). The average r^2 and %RSD of all compounds were 0.997 and 6.9%, respectively.

The calibration check compounds (CCCs) have average %RSD of 6.9%, easily passing method criteria of 30%. The average %RSD of all compounds was 6.9%, significantly better than the method criteria of 15%.

Using the V:Results™ EPA Tune criteria testing capabilities, compound SS-4-bromofluorobenzene was used to validate the tuning of the instrument according to EPA 8260B section 7.3.

The system performance check compounds (SPCCs) were evaluated based on average RRF. Table 2 shows RRFs over the concentration range of 0.5 to 100 ppb.

Table 1. Calibration data for 60 volatile organic chemicals.

Compound Name	"8260 B Criteria"	"Average RF"
Chloromethane	0.100	0.113
1,1-Dichloroethene	0.100	0.567
1,1-Dichloroethane	0.100	0.666
Chlorobenzene	0.300	1.052
Bromoform	0.100	0.302
1,1,2,2-Tetrachloroethane	0.300	0.547

Conclusion

The Varian 210-MS system with the Varian Archon™ AutoSampler, Tekmar Stratum purge-and-trap concentrator and V:Results GC/MS software showed excellent linearity and %RSD over the calibration range. The entire GC/MS system is proven to meet or exceed the performance required by US EPA method 8260B and is validated to meet all of the QC criteria outlined in the method.

These data represent typical results.
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