Application Note: 10209

Analysis of Wastewater for Volatile Organics (EPA Method 8260C) by GC/MS

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Key Words

- DSQ II Single Quadrupole GC/MS
- EnviroLab Forms
- EPA Method 8260C
- Purge and Trap
- Volatile Organics

Overview

United States Environmental Protection Agency (US EPA) Method 8260 is an analytical method for the measurement of volatile organic pollutants in a wide range of matrices from ground water to aqueous sludge, oily waste, and sediment.[1] Method 8260C lists 100 organics with diverse physical properties, from a gas at room temperature, like vinyl chloride, to an aromatic like naphthalene that elutes from the analytical column at 230 °C. There are also very polar compounds, like tert-butanol, and very reactive components such as 2-chloroethyl vinyl ether. In addition, the linear calibration range covers 1 µg/L to 200 µg/L, with detection limits cited in the method at 5 µg/L for ground water, 5 µg/kg for soil, and 0.5 mg/kg for waste oils.[1] These factors present a significant challenge for any instrument and analyst in the environmental laboratory.

The Thermo Scientific 8260 Productivity Solution offers a comprehensive start-up package for the installation of the Thermo Scientific DSQ™ II single quadrupole mass spectrometer, Thermo Scientific FOCUS™ GC, and a purge and trap system in your laboratory as a dedicated analyzer for EPA Method 8260C. For this Productivity Solution, the DSQ II MS and FOCUS GC (Figure 1), along with an OI Analytical Eclipse 4660 Purge and Trap and an OI Analytical 4551 Autosampler, were put through rigorous QC tests to measure the precision, linearity, accuracy, and sensitivity of the entire instrument system for EPA Method 8260C. This application note highlights the performance of the Productivity Solution approach. The results show compliance to all QC tests with a run time of 11 minutes, with an average method detection limit of 0.35 µg/L.

This comprehensive approach to EPA Method 8260C ensures your laboratory's success in the measurement of precision, linearity, accuracy, and sensitivity for the analysis of wastes and wastewater according to the guidelines specified in EPA Method 8260C.

Figure 1: Thermo Scientific DSQ II GC/MS with FOCUS GC

Methods

All standards were prepared in organic-free water as specified in the 8260 Productivity Solution Standard Operation Procedure, *Determining Volatile Organics in Wastewater Using the DSQ II*.[2] Approximately 100 target compounds were analyzed to determine their linear range, method detection limit (MDL), and accuracy and precision of analysis in the method validation (MVD) study. A split injection was made using a purge and trap adapter interface to integrate the concentrator to the GC for sample introduction. The mass spectrometer was operated in EI Full Scan mode. The chromatography was optimized to provide a short run time, with all compounds eluting in less than 11 minutes. The mass spectrometer was tuned using Target Tune to meet the mass resolution criteria for 4-bromofluorobenzene (BFB) as specified in the EPA method.

Results

The 8260 Productivity Solution for the DSQ II GC/MS system was evaluated against the QC criteria for EPA Method 8260C. The results showed compliance to all QC tests set in the method. The linear range was determined to be 1 to 200 µg/L with an average percent relative standard deviation (%RSD) of 6.9. The average MDL was 0.35 µg/L. A total ion chromatogram for a 20 µg/L mid-level standard acquired during the method validation study is shown in Figure 2. Uniformity of spectral identification for detection was ensured by applying tuning compound ion ratio criteria for BFB. Table 1 lists method validation, method detection and calibration results for selected compounds from the 8260C target list. This table includes system performance check compounds, and demonstrates the accuracy, precision, and sensitivity of the method when using the DSQ II system.

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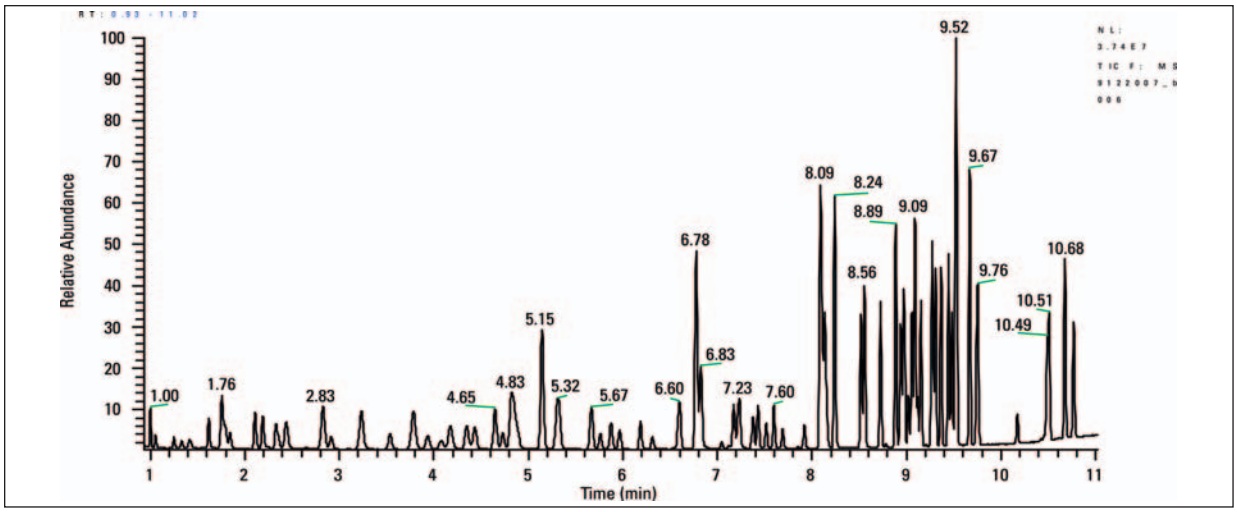

Figure 2: Total ion chromatogram of 20 µg/L method validation standard

	Linearity	Method Validation (MVD)		Method Detection Limit (MDL)
Component	**%RSD**	**% D**	**%RSD**	**(µg/L)**
dichlorodifluoromethane	8	-16.2	6	0.22
chloromethane	5	-3.8	5	0.21
vinyl chloride	12	-10.6	6	0.38
bromomethane	6	-5.0	5	0.38
chloroethane	10	-1.5	5	0.47
trichlorofluoromethane	11	-10.6	6	0.29
carbon disulfide	1	-10.8	6	0.22
1,1,2-trichloro-1,2,2-trifluoroethane	2	-13.9	6	0.37
methyl tert-butyl ether (MTBE)	5	-2.0	2	0.20
tert-butanol	16	29.4	7	1.07
diisopropyl ether (DIPE)	5	-2.7	3	0.49
allyl alcohol	7	-1.4	3	0.28
ethyl tert butyl ether (ETBE)	7	-2.3	3	0.24
benzene	5	-3.3	4	0.21
tert-amyl methyl ether (TAME)	7	1.3	3	0.19
dibromomethane	2	5.5	2	0.14
methyl methacrylate	8	-1.7	2	0.15
2-chloroethyl vinyl ether	6	-2.3	1	0.18
4-methyl-2-pentanone (MIBK)	11	5.7	5	0.70
ethyl methacrylate	10	-1.0	2	0.17
chlorobenzene	3	-0.3	3	0.17
o-xylene	6	0.5	4	0.20
bromoform	7	6.7	2	0.19
bromobenzene	8	3.8	4	0.18
trans-1,4-dichloro-2-butene	4	-1.3	3	0.29
1,3-dichlorobenzene	6	2.1	3	0.14
benzyl chloride	9	5.5	5	0.33
1,2-dibromo-3-chloropropane	9	1.7	4	0.30
naphthalene	9	12.3	3	0.35
1,2,3-trichlorobenzene	9	10.1	4	0.37

Table 1: Method linearity, validation, and detection limit study results for selected 8260C compounds

Conclusions

The 8260 Productivity Solution provides fast chromatography, with the last target compound eluting at less than 11 minutes. The DSQ II system successfully met the QC criteria for EPA Method 8260C in a split mode of injection using the FOCUS GC. Combined with a fast scanning rate, this method features excellent separation and sensitivity and generates MDLs required by the EPA method. The methods and standard operating procedure (SOP), along with a How To Manual with Quick Start Guide, can be automatically downloaded to the local PC by installing the Interactive Reference CD. A Validation Data CD may be used as a reference of typical data for the method.

EnviroLab™ Forms 2.0 software matches the workflow in environmental laboratories around the world, is simple to use, and allows novice users to be instantly productive. The DSQ II offers excellent sensitivity and reliably meets the method QC criteria. The 8260 Productivity Solution transforms the DSQ II system into a dedicated analyzer for volatiles. This allows laboratories to improve productivity and streamline new instrument integration.

For more detailed information on methods and results, please visit www. thermo.com and request Technical Note TN10210.

References

1. *US EPA Method 8260C Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)*, Rev 3, 2006
2. 8260 Productivity Solution Standard Operation Procedure, *Determining Volatile Organics in Wastewater Using the DSQ II*. Thermo Fisher Scientific Part #120294-0001

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