Application Note: 10175

GC/MS Analysis of Volatile Organic Compounds in Drinking Water: Productivity Solution for US EPA Method 524.2

Jessie Butler, Eric Phillips, Thermo Fisher Scientific, Austin, TX, USA

Key Words

- DSQ II GC/MS
- EnviroLab Forms 2.0 Software
- Drinking Water
- EPA Method 524.2
- Volatiles

Overview

The GC/MS analysis of volatile organics in drinking water according to United States Environmental Protection Agency (US EPA) Method 524.2 offers a number of analytical challenges, ranging from water handling to very low target compound concentrations. While a 25 mL purge volume has historically been used for purge and trap concentration prior to GC/MS analysis, the best purging efficiency for polar compounds, such as ketones, can be achieved with a 5 mL sample volume. This, in turn, requires a highly sensitive mass spectrometer in order to continue to achieve the required method detection limits. The Thermo Scientific DSQ™ II GC/MS system provides excellent sensitivity and allows for the use of a 5 mL purge. To take full advantage of this sensitivity and allow environmental laboratories to realize the improved performance and productivity provided by the DSQ II system, a comprehensive turn-key package was created for Method 524. This 524 Productivity Solution integrates the hardware, software, analytical column, standards, consumables and methods to provide a complete solution for the method. Using the 524 Productivity Solution, laboratories can readily meet the method requirements and easily incorporate the DSQ II system into their existing workflows.

Results

The analysis of 84 volatile organics was performed in 11 minutes, as shown in the total ion chromatogram of the mid-level 20 µg/L standard (Figure 1). Over a concentration range of 0.4 to 40 µg/L, the compounds analyzed had an average % RSD of 6. The average method detection limit (MDL) was 0.095 µg/L, using a 5 mL sample volume. A daily calibration check standard was run at 4 µg/L. The average % difference was 2.0%. Table 1 highlights the calibration, daily calibration check, and detection limit study results for selected compounds.

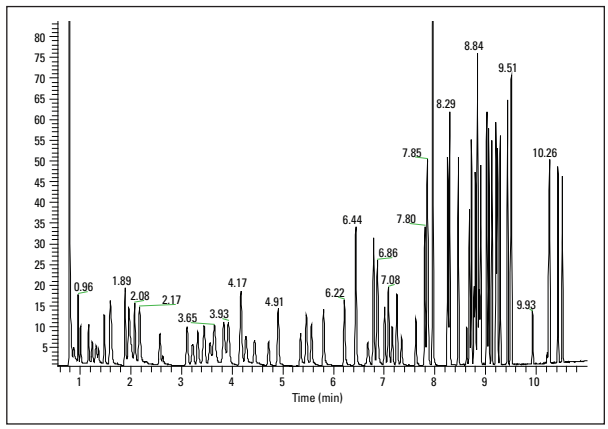

Figure 1: TIC of mid-level standard (20 µg/L)

Methods

In full-scan mode, the DSQ II system has excellent sensitivity, which allows a split injection of 80 to 1 to be made on a conventional split/splitless injector using the FOCUS GC. The high split ratio minimizes adverse effects of co-eluting water inherent in this method of sample transfer. The column flow was set to a constant pressure of 25 psi. A 20 m x 0.18 mm TRACE™ TR-524 column with a film thickness of 1.0 µm was used for the analytical column. The DSQ II mass spectrometer was tuned to meet the criteria for the tuning compound 4-bromofluorobenzene (BFB) using Target Tuning software.

Data was acquired using Xcalibur™ instrument control software and then processed automatically using EnviroLab Forms 2.0. The Method 524 Productivity Solution includes complete methods, and EnviroLab Forms 2.0 reports can be saved in *.pdf, *.doc, *.xls, or *.rtf format. Reports can also be easily uploaded to a laboratory information management system (LIMS) using XML format.

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Conclusion

The Method 524 Productivity Solution provides fast chromatography, with a short run time of 15 minutes, including a 5 minute bake out of the column after the last target compound elutes at about 10 minutes. This allows a high-throughput lab to easily process 36 samples or more per 12 hour shift. The DSQ II system successfully met the QC criteria for EPA Method 524.2 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 eMethods and Standard Operating Procedure, 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. Finally, the Productivity Solution for EPA Method 524 transforms the DSQ II system into a dedicated analyzer for volatiles. This allows laboratories to achieve three times the sample throughput of standard methodologies, greatly improving productivity and streamlining instrument integration.

For more detailed information on methods and results, please visit www. thermo.com and request Technical Note TN10174.

Reference

1. *Method 524.2 Measurement of Purgeable Organic Compounds by Capillary Column Gas Chromatography/Mass Spectrometry,* Rev 4.1, National Exposure Research Laboratory Office of Research and Development U. S. Environmental Protection Agency Cincinnati, Ohio 45268.

Compound	MDL (µg/L)	Curve (0.4-40 µg/L) (%RSD)	Ave. RF	Daily Check (4 ppb) Daily RF	%Diff.
dichlorodifluoromethane	0.137	6	0.137	0.149	9.2
chloromethane	0.057	4	0.462	0.448	-3.2
vinylchloride	0.036	6	0.242	0.240	-0.9
bromomethane	0.093	2	0.178	0.127	2.3
chloroethane	0.082	3	0.124	0.127	2.3
carbon disulfide	0.077	2	0.568	0.566	-0.4
trans-1,2-dichloroethylene	0.089	5	0.209	0.215	3.0
cis-1,2-dichloroethylene	0.110	5	0.233	0.229	-1.8
bromochloromethane	0.087	8	0.097	0.100	3.9
chloroform	0.109	4	0.397	0.419	5.4
benzene	0.045	3	0.756	0.735	-2.8
ethyl methacrylate	0.067	6	0.281	0.286	1.8
dibromochloromethane	0.094	8	0.168	0.171	1.8
2-hexanone	0.128	4	0.359	0.351	-2.1
bromoform	0.110	7	0.197	0.184	-6.7
4-chlorotoluene	0.030	7	0.825	0.841	2.0
tert-butylbenzene	0.061	7	0.675	0.737	9.2
p-isopropyltoluene	0.052	4	0.593	0.605	2.1
n-butylbenzene	0.050	5	0.992	1.028	3.6
nitrobenzene	0.376	8	0.040	0.041	3.0
hexachlorobutadiene	0.131	8	0.154	0.158	2.7
naphthalene	0.051	7	1.013	1.020	0.7
1,2,3-trichlorobenzene	0.073	7	0.463	0.474	2.4

Table 1: Method detection limit, calibration curve and daily calibration check results for selected compounds, demonstrating excellent precision and sensitivity

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