Application Note: 10173

# GC/MS Analysis of Semi-volatile Organic Compounds in Drinking Water: Productivity Solution for US EPA Method 525.2

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**Keywords:**

- DSQ II GC/MS
- Drinking Water
- EnviroLab Forms 2.0 Software
- Semi-Volatile Analysis
- Splitless Injection Method
- US EPA Method 525.2

## Overview

The analysis of drinking water for semi-volatile organics including thermally labile pesticides of varying polarity is quite challenging. Using a surged splitless injection with the DSQ™ II GC/MS enables laboratories to easily achieve the method detection limits required by United States Environmental Protection Agency (USEPA) Method 525.2. By combining the DSQ II GC/MS with all of the methodologies, consumables, operating procedures, and layered application software, the 525.2 Productivity Solution streamlines method development and validation.

## Results

A mixture of 115 compounds, including polynuclear aromatics, phthalates, Aroclors, and pesticides was analyzed using the DSQ II GC/MS. Over a concentration range of 0.1 to 10 ng/µL, the calibration curve had an average RSD of 9%. The average method detection limit (MDL) at a 99% confidence level was 0.04 µg/L. Seven replicate injections were made at 5 ng/µL to demonstrate the accuracy of quantitation at a mid-level concentration, which resulted in an average RSD of 1.9% for the compounds, easily meeting the percent recovery of 70 to 130%. US EPA Method 525.2 identifies problem compounds that tend to show higher MDLs, lower recoveries, and potential for non-detection. Table 1 gives an overview of the published MDLs in the EPA Method, the Instrument Detection Limits (IDLs) achieved on the DSQ II, and the calibration curve fit for each problem compound as called out in the method. The EPA Method 525 Productivity Solution produces Gaussian peak shapes and good results for these problematic compounds.

| Problem Compounds Listed in Method* with Stated Low Recoveries | | MDL Listed in Method (µg/L) | DSQ II Calibration Results | | DSQ II IDLs |
|---|---|---|---|---|---|
| | | | % RSD | $R^2$ | (µg/L) |
| merphos | breakdown to DEF | Not Detectable (ND) | 24 | 0.9967 | 0.1320 |
| fenamiphos | poor GC peak | 0.95 | 24 | 0.9978 | 0.0320 |
| fenarimol | poor GC peak | 1.2 | 14 | 0.9993 | 0.0280 |
| fluridone | poor GC peak | 0.55 | 32 | 0.9989 | 0.0490 |
| hexazinone | poor GC peak | 0.11 | 13 | 0.9991 | 0.0420 |
| norflurazon | poor GC peak | 0.13 | 19 | 0.9994 | 0.0710 |
| stirofos | poor GC peak | 0.13 | 18 | 0.9997 | 0.0140 |
| tebuthiuron | poor GC peak | 2.8 | 16 | 0.9993 | 0.0300 |
| tridemefon | poor GC peak | 0.33 | 11 | 0.9998 | 0.0420 |
| tricyclazole | poor GC peak | 2.6 | 26 | 0.9959 | 0.0610 |
| anthracene | photodegradation | 0.068 | 3 | 0.9995 | 0.0140 |
| benzo(a)anthracene | photodegradation | 0.2 | 3 | 0.9993 | 0.0270 |
| dimethyl phthalate | in lab at ~ 2 µg/L | 0.058 | 3 | 0.9999 | 0.0270 |
| atraton | ND at pH of 2 | 0.16 | 10 | 0.9998 | 0.0240 |
| prometon | ND at pH of 2 | 0.38 | 10 | 0.9999 | 0.0540 |
| carboxin | ND in water | 1.4 | 16 | 0.9995 | 0.0270 |
| disulfoton | ND in water | 0.62 | 8 | 0.9999 | 0.0270 |
| metribuzin | < 50% | 0.16 | 8 | 0.9999 | 0.0200 |
| cyanazine | ND at pH of 2 | 0.17 | 10 | 0.9999 | 0.0580 |
| pentachlorophenol | loss of sensitivity < 5 ng | ND | 14 | 0.9948 | 0.4900 |

Table 1: EPA Method Problem Compounds, published MDLS, and DSQ II Instrument Detection Limits (IDLs)

**Characteristics as described in the method in Section 13.2 Problem Compounds*[1]

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## Methods

In full-scan mode, the DSQ II offers excellent sensitivity, which allows a conventional splitless injection to be used instead of more difficult techniques, such as cold on-column or temperature programmed injections. A 1 µL injection was made in the splitless mode with a surge pressure of 37 psi and a 1 mL/min column flow. A proprietary phase, 30 m x 0.25 mm i.d. x 0.25 µm film thickness TRACE™ TR-525 column was used (Thermo Scientific PN 26RX142P). The column and inlet were tested for activity by injecting the performance mix, a solution of DFTPP, endrin, and 4,4'-DDT at 5 µg/mL in iso-octane (Thermo Scientific PN 60181-311). The mass spectrometer was tuned automatically using Autotune Tuning software to meet DFTPP tuning criteria. The TIC for the mid-level standard is shown in Figure 1.

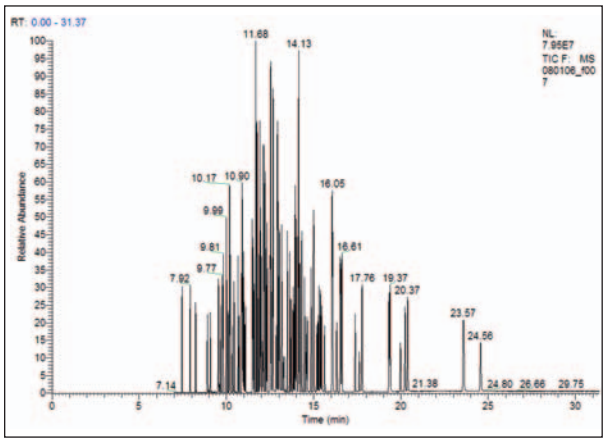


Figure 1: TIC of mid-level standard (5 ng/µL)

Data was acquired using Xcalibur™ instrument control software and then processed automatically using the EnviroLab™ Forms 2.0 software. Complete methods are available for EPA Methods and reports can be saved in *.pdf, *.doc, *.xls, or *.rtf format. Reports can also be uploaded to a laboratory information management system (LIMS) using *.XML format files.

## Conclusion

This method provides fast chromatography while maintaining optimal separations of the chromatographic peaks. The DSQ II successfully met the QC criteria for EPA Method 525.2 in a splitless mode using the TRACE GC Ultra™. Combined with a fast scanning rate, this Productivity Solution features excellent separation and sensitivity, enabling the generation of MDLs required in the EPA method. The Productivity Solution (Figure 2) contains all of the consumables needed to perform the instrument validation, including standard solutions and vials. The electronic methods, Standard Operating Procedure, and How To Manual with Quick Start Guide can be automatically downloaded to the local computer by installing the Interactive Reference CD (PN 120296-CD). A Validation Data CD (PN 120296-VCD) may be used as a reference of typical data for the method.


Figure 2: EPA Method 525.2 Semi-volatiles in Water Productivity Solution

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. Now, adding a DSQ II system to an existing laboratory workflow is easier than ever.

For detailed information on the instrument and processing parameters, as well as calculated values for all compounds analyzed, please visit our website at *www.thermo.com/gc* and request TN10172.

## References

1. *Method 525.2 Determination of Organic Compounds in Drinking Water by Liquid-Solid Extraction and Capillary Column Gas Chromatography/ Mass Spectrometry*, Rev 2.0, National Exposure Research Laboratory Office of Research and Development U. S. Environmental Protection Agency Cincinnati, Ohio 45268

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