

Concentrating a Suite of Semi-Volatile Compounds from EPA Method 625 Using the New Biotage TurboVap® II

Introduction

There are a wide range of volatile and semi-volatile contaminants finding their way into both terrestrial bodies and water sources worldwide. In the United States (US), the contaminants are analyzed according to stipulated US-EPA methods. In the European Union (EU), a large number of these same compounds are tested according to the European Water Framework Directive. Though these analytes are approached differently from a regulatory perspective, it is clear that background monitoring occurs on a global basis. Initial extraction of these analytes depends on the matrix being analyzed and is often a multifaceted process, but ultimately analysts are presented with some form of extraction/organic solvent they must concentrate to achieve instrumental limits of quantification. Presented within this technical note are the results of such an evaporative process using the new Biotage TurboVap® II.

The new TurboVap® II is built on the solid foundations of reliability and performance that made it the market leader for solvent evaporation. The modern design incorporates many new customer driven features for easier use and expanded functionality. TurboVap II still utilizes the highly efficient patented gas vortex shearing technology, which is synonymous with the TurboVap brand.

This modern design features many new enhancements, including a well-lit glass tank for much greater visibility of the samples, improved sensors with automatic end-point detection, user replaceable nozzles, easy access drain port, and a color, menu driven touchscreen for simple operation and monitoring. The system can be vented from the bench or placed in the fume hood. Because the footprint is noticeably smaller on this modern unit, less space is required.



Experimental

A TurboVap® II evaporation system (P/N 415001) was operated with a TurboVap II Rack with End-Point Sensors 6 Positions, 200 mL Tubes (P/N 415100) using 1 mL end-point evaporation tubes (P/N C128506).

Dichloromethane (DCM), a mixed analyte standard P/N 506559 and p-Terphenyl were all purchased from Sigma-Aldrich.

180 mL of DCM was added into six 200 mL, 1 mL end-point evaporation tubes and spiked with 20 µg of each analyte to provide a concentration of ~111 ng/mL.

The system was initially operated using the parameters documented in table 1.

Table 1.

Parameter	Setting
Inlet pressure	6 bar
Operating flow rate	2.8 L/min
Water bath temperature	40 °C

Evaporation was automatically halted once the end-point sensors detected the solvent had reached the cut off volume (~ 0.7 mL). p-Terphenyl was added at a concentration of 20 µg/mL to each of the six tubes to act as an external standard and enable response factor (Response Factor) generation. Using a pipette, the concentrated extract was transferred to a screw capped autosampler vial and the inner walls of the evaporator tube rinsed with DCM ensuring a final volume of 1 mL.

The experimental procedure was repeated a second time but in place of a constant 2.8 L/min gas flow, a ramped gradient flow was applied as detailed in table 2.

Table 2.

Parameter	Setting
Inlet pressure	6 bar
Initial flow rate	2.8 L/min hold for 4 minutes
Final flow rate	7.0 L/min over 30 minutes
Water bath temperature	40 °C

Analysis

Gas chromatography-mass spectrometry (GC-MS) was used for the quantitative determination of each analyte following evaporation. GC separation was carried out on an Agilent 7890A equipped with QuickSwap. Separation of target analytes was achieved using an Agilent J&W DB-5ms, 30 m x 0.25 mm ID x 0.25 µm column. Oven parameters were as follows; initial temperature of 40 °C with a 2 minute isocratic hold. Ramp conditions were 20 °C/min to 290 °C and a hold for 1.5 minutes, followed by a second ramp 100 °C/min to 325 °C held for 4.6 minutes providing a total run time of 20.95 minutes. Post run the column was back flushed for 2.4 minutes (approximately 3 void volumes).

Injection volume was set at 2 µL, whilst using helium at a flow rate of 1 mL/min (constant flow) as the carrier gas. The inlet was set to operate in splitless mode and inlet temperature was maintained at 300 °C with a purge flow of 50 mL/min at 0.8 minutes.

The GC system was coupled directly to an Agilent 5975C mass spectrometer, with the transfer line temperature set at 300 °C. The MS was operated in electron impact ionization (EI), acquiring data in full scan mode between 40 and 285 m/z. Source and quadrupole temperature were maintained at 230 and 150 °C respectively, whilst a 4 minute solvent delay was maintained.

Table 3. Results of the evaporation and instrumental analysis.

Analyte	% Recovery Fixed Flow	% RSD Fixed Flow	% Recovery Ramped Flow	% RSD Ramped Flow
<i>n</i> -Nitrosodimethylamine	90.8	5.0	89.8	2.2
Phenol	83.3	4.5	86.5	2.7
Bis(2-Chloroethyl) ether	80.2	5.6	82.7	2.3
2-Chlorophenol	80.8	4.7	83.1	2.1
1,3-Dichlorobenzene	78.6	5.2	79.5	1.9
1,4-Dichlorobenzene	78.6	5.3	80.3	1.6
1,2-Dichlorobenzene	79.8	5.2	81.6	1.5
Bis(2-Chloroisopropyl) ether	81.9	4.7	85.4	1.5
<i>n</i> -Nitrosodi- <i>n</i> -propylamine	99.2	2.9	101.2	2.1
Hexachloroethane	80.0	3.8	80.5	2.6
Nitrobenzene	85.3	4.7	88.9	2.7
Isophorone	88.2	4.9	95.1	2.2
2-Nitrophenol	84.3	3.3	86.9	2.0
2,4-Dimethylphenol	85.1	5.0	88.6	4.2
Bis(2-Chlorethoxy) methane	84.5	4.9	90.0	2.3
2,4-Dichlorophenol	83.7	4.2	89.8	3.7
1,2,4-Trichlorobenzene	80.0	5.1	84.0	2.7
Naphthalene	81.2	4.9	85.7	2.6
Hexachloro-1,3-butadiene	80.8	6.4	84.6	3.3
4-Chloro-3-methylphenol	89.1	3.7	97.7	3.3
Hexachlorocyclopentadiene	90.9	8.6	88.9	3.2
2,4,6-Trichlorophenol	89.5	3.6	99.0	3.2
2-Chloronaphthalene	84.4	4.6	92.8	2.5
Dimethyl phthalate	90.4	4.6	101.2	2.7
Acenaphthalene	85.9	4.2	95.1	2.5
2,6-Dinitrotoluene	91.0	4.0	101.1	3.3
Acenaphthene	86.9	3.8	95.2	3.1
2,4-Dinitrotoluene	96.9	2.1	102.7	3.4
Diethyl phthalate	93.7	4.8	103.2	3.0
2-Methyl-4,6-dinitrophenol	88.8	4.1	98.6	2.9
Fluorene	92.0	2.5	99.2	2.9
Azobenzene	89.7	4.7	99.3	3.1
4-Bromodiphenyl ether	89.5	4.7	100.7	2.8
Hexachlorobenzene	92.7	4.3	100.6	3.1
Phenanthrene	94.0	4.1	101.0	2.9
Anthracene	94.7	3.1	101.4	3.3
Carbazole	97.1	3.1	108.1	3.5
Dibutyl phthalate	101.2	4.9	103.7	2.5
Fluoranthene	100.4	3.4	103.0	3.0
Pyrene	99.4	3.0	101.4	3.5
Benzyl butyl phthalate	112.4	0.8	104.2	2.3
Benzanthracene	94.0	3.8	103.2	4.5
Bis(2-Ethylhexyl) phthalate	109.7	3.8	106.9	3.3
Chrysene	94.9	3.1	103.3	4.2
Di- <i>n</i> -octyl phthalate	111.0	4.2	110.6	3.6
Benzo[b]fluoranthene	96.0	6.8	105.5	4.3
Benzo[k]fluoranthene	104.6	5.8	104.0	5.3
Benzo[a]pyrene	94.6	6.0	105.4	4.8
Dibenz[a,h]anthracene	98.5	8.6	109.6	6.9
Benzo[g,h,i]perylene	98.2	8.3	110.0	5.7

Results

Table 3 shows the results of the evaporation and instrumental analysis. A total of n=6 replicates for each of the two evaporation methods were analyzed and the averages are presented. Recovery and RSD values were calculated by comparing the peak area response of p-Terphenyl in a standard and the processed samples and generating a RF. This RF was then used to volumetrically normalize the results for the processed samples when compared to the standard.

Conclusion

The new TurboVap® II evaporation system provides excellent recoveries and RSDs for a wide range of semi-volatile compounds. With two options available for tube sizes 50 or 200 mL and either 0.5 or 1.0 mL end-point the system can be used with solvent extracts derived from a wide range of extraction methodologies, including Solid Phase Extraction, Supported Liquid Extraction, Liquid-Liquid Extraction, Continuous Liquid-Liquid Extraction, TLCP Extracts, Pressurized Fluid Extraction, and Ultrasonic Extraction.

Ordering Information

Part Number	Description	Quantity
415001	TurboVap II (Includes 415222 Manifold, but no Rack)	1
415222	TurboVap II Manifold (6 Nozzles)	1
415100	TurboVap II Rack with End-Point Sensors (6 Positions, 200 mL Tubes)	1
415535	TurboVap II Rack with End-Point Sensors (6 Positions, 50 mL Tubes)	1
415494	TurboVap II Multi Rack without End-Point Sensors (6 Positions, 50/200 mL Tubes)	1
C128506	Evaporation Tube TurboVap II, 200 mL, 1 mL EndPoint	12
C128507	Evaporation Tube TurboVap II, 200 mL, 0.5 mL EndPoint	12
C128508	Evaporation Tube TurboVap II, 50 mL, 0.5 mL EndPoint	12
C128511	Evaporation Tube TurboVap II, 50 mL, 1 mL EndPoint	12
C128512	Evaporation Tube TurboVap II, 50 mL, Centrifuge	12

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