

Introduction

Method 525.2 describes the procedure to determine low ppb levels of semi-volatile organic material in drinking water using solid phase extraction (SPE) or liquid–solid extraction (LSE) techniques. The City of Fort Worth, Water Department implemented an automated SPE process for the analysis of semi-volatiles by EPA Method 525.2 using the Atlantic C18 solid phase extraction disk. Ethyl acetate, methanol and water were used to condition the Atlantic C18 disk prior to the extraction step. The extraction solvents used were a 1:1 mixture of methylene chloride and ethyle acetate. Extracts were then analyzed by GC/MS using a splitless injection technique.

Automated sample handling equipment manufactured by Horizon Technology, Inc. was used in this method, including the SPE-DEX[®] 4790 Automated Extraction System, the Envision[™] Platform Controller, and the DryVap[™] Automated Drying and Concentration System. These units are designed to streamline the sample handling required for analyzing environmental samples.

The SPE-DEX 4790 provides automated extraction of liquid samples by solid phase extraction methods. It can handle samples that range from 20ml to 4L. The Envision Platform provides a user-friendly, web-based controller capable of interacting with up to eight extractors via a PC. The DryVap Concentrator System provides automatic sample drying with a patented membrane technology and automatically concentrates each dried extract by applying heat, vacuum, and sparge flow for up to six samples at once.

Instrumentation

- Horizon Technology SPE-DEX 4790 Automated Extractor System
- Horizon Technology Envision Platform (hardware/software system to control extractors)
- Horizon Technology DryVap Concentrator System with DryDisk[™] Separation Membranes
- Atlantic C18 Disks (47mm)
- Agilent 6890 GC
- Agilent 5973 inert MSD
- Agilent 7683B Autosampler
- Column A: Agilent HP-5ms, 30m x 0.250mm x 0.25um
- Liner: 4mm single taper, deactivated, splitless
- Merlin MicroSeal[®] High Pressure Septum



Method Summary

- 1) A 1L aliquot of sample is used.
- 2) Adjust samples pH to <2.
- 3) Spike surrogate and internal standard compounds into samples.
- 4) Spike analyte standards into samples.
- 5) Start extraction method, collect extract (approximately 30ml).
- 6) Add extract to the Dry Disk holder and start concentration process on the DryVap system.
- 7) Concentrate the extract to less than 1.0ml and quantitatively bring the extract volume to 1.0ml (DryVap concentration vessels are graduated to 0.5ml and 1.0ml).
- 8) Transfer a portion of the extract to a GC vial with insert.
- 9) Analyze by GC/MS.

Table 1 on the next page shows the parameters that were programmed into the Envision Platform for the extraction process. All of the steps in the extraction process are automated. The DryVap Concentrator settings are shown in Table 2, also on the next page.

Table 1: Extractor Program for SPE-DEX Automated 525.2 Method.

STEP	SOLVENT	SOAK TIME	DRY TIME
Prewet #1	Ethyl Acetate	1:30 min	1:30 min
Prewet #2	Methanol	1:30 min	0 min
Prewet #3	UP Water	1:30 min	0 min
Sample Process			
Air Dry			8:00 min
Rinse Step #1	Ethyl Acetate	1:30 min	1:00 min
Rinse Step #2	Methylene Chloride	1:30 min	1:00 min
Rinse Step #3	MeCl ₂ :EtAc (1:1)	1:30 min	2:00 min
Rinse Step #4	MeCl ₂ :EtAc (1:1)	1:30 min	2:00 min

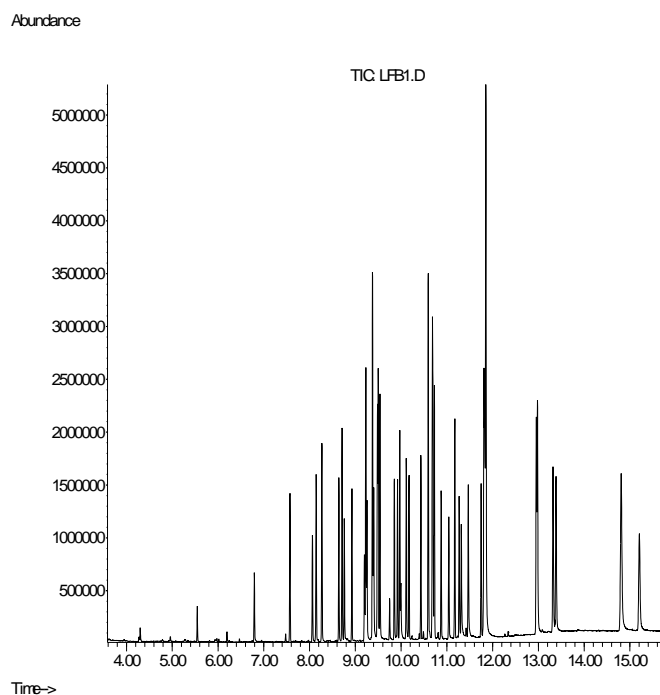
Table 2: Parameters used for the DryVap Concentrator System.

PARAMETER	SETTING
Dry Volume	20
Heat Power	5
Auto Rinse Mode	OFF
Heat Timer	OFF

Results

Ultra-Pure (UP) water was spiked with 525.2 standard at a theoretical concentration of 5.0ppb shown in figure 1. The results for the Atlantic C18 disks are listed in Table 3 on the following page. Forty two compounds were included in the target compound list. The table shows the compound names, amount recovered, and percent recovery.

The Atlantic disk had a recovery range between 61%-122%. Twenty seven compounds fell within 100-122% recovery range and twelve compounds fell within 90%-100% recovery range. One compound fell within each range: 80-90%, 70-80% and 60%-70%. Atlantic Disk 2 had a recovery range between 58%-119%. Twenty eight compounds fell within 100%-119%. Nine compounds fell within 90%-100%. Three compounds fell between the 80-90%. One compound fell within each range 70-80% and 50-60%. †Note the excellent recovery for Prometon with an acidic extraction (pH<2). This analyte usually requires a separate extraction under neutral pH conditions.

Figure 1. EPA Method 525.2 Semi-Volatile Analytes

Conclusions

This data demonstrates that the equipment used in this study is capable of fully automating EPA method 525.2 that results in data that is both accurate and precise. The SPE-DEX 4790 Automated Extractor System with the Envision Platform and DryVap Concentrator System reduces analyst labor, solvent usage, turnaround time, and improves accuracy and precision.

Acknowledgements

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Table 3. Concentration and % Recovery of EPA 525.2 Analytes

Analyte	Recovery (ppb)	% Recovery	Recovery (ppb)	% Recovery
	Atlantic Disk 1		Atlantic Disk 2	
Hexachlorocyclopentadiene	4.92	98	5.27	105
Dimethylphthalate	4.70	94	4.56	91
Acenaphthylene	5.07	101	4.87	97
Diethylphthalate	6.11	122	5.86	117
Fluorene	5.49	110	5.34	107
Propachlor	5.73	115	5.53	111
Trifluralin	6.07	121	5.89	118
Hexachlorobenzene	5.35	107	5.12	102
Atrazine	5.37	107	4.96	99
Prometon [†]	3.93	79	4.16	83
Simazine	4.00	80	3.96	79
gamma-BHC	5.34	107	5.25	105
Phenanthrene	4.82	96	4.86	97
Anthracene	4.74	95	4.75	95
Pentachlorophenol	19.74	99	20.03	100
Metribuzin	3.06	61	2.92	58
Alachlor	5.35	107	5.34	107
Heptachlor	5.67	113	5.52	110
Di-n-butylphthalate	5.36	107	5.27	105
Bromacil	4.57	91	4.46	89
Metalachlor	5.43	109	5.40	108
Aldrin	4.58	92	4.24	85
Heptachlor Epoxide	5.40	108	5.50	110
gamma-Chlordane	5.36	107	5.16	103
Butachlor	5.55	111	5.55	111
alpha-Chlordane	5.37	107	5.22	104
trans-Nonachlor	5.34	107	5.14	103
Pyrene	5.45	109	5.29	106
Dieldrin	5.74	115	5.65	113
Endrin	5.50	110	5.16	103
cis-Nonachlor	4.80	96	4.86	97
Butylbenzylphthalate	5.51	110	5.59	112
Di-(2-ethylhexyl) Adipate	5.75	115	5.95	119
Methoxychlor	5.67	113	5.80	116
Benzo (a) Anthracene	5.03	101	5.02	100
Chrysene	5.19	104	5.11	102
Benzo (b) Fluoranthene	5.24	105	5.59	112
Benzo (k) Fluoranthene	5.08	102	4.83	97
Benzo (a) Pyrene	4.75	95	4.81	96
Indeno (1,2,3-cd) Pyrene	4.75	95	4.92	98
Dibenz (a,h) Anthracene	4.87	97	5.09	102
Benzo (g,h,i) Perylene	4.79	96	5.01	100