

Environmental Applications note

cat.# 59557

SPE Extraction for EPA Method 525.1

EPA Method 525.1 Overview

EPA Method 525.1 is used for the determination of organic compounds in drinking water by liquid-solid extraction and capillary column gas chromatograph/mass spectrometry. It is applicable to a wide range of organic compounds that are efficiently partitioned from the water sample onto a C18 organic phase chemically bonded to a solid silica matrix in a cartridge or disk.

SIMDisk™-GF extraction disks can be used in EPA Method 525.1

This procedure was followed to demonstrate method equivalency:

1 Sample Pretreatment:
Allow 1 liter of deionized water to equilibrate to room temperature in a narrow-mouth amber glass bottle. Adjust sample pH to less than 2 with 6M hydrochloric acid, being careful not to over acidify the sample. Addition of too much HCl can cause the formation of acidic extracts.

2 Apparatus Assembly:
Assemble the 47mm apparatus. Place the SIMDisk™-GF disk in the Diskcover™-47 filter support, wrinkled side up.

3 Disk Precleaning:
Add 5ml of methylene chloride to the top surface of the disk and immediately draw through under vacuum at 15 in. Hg (50 kPa). Continue to draw vacuum at 15 in. Hg (50 kPa) for 5 minutes to remove all solvent.

4 Disk Conditioning:
Add 5ml methanol to the top surface of the disk and immediately apply low vacuum (1-2 in. Hg, 3-7 kPa). Draw through until the top surface of the methanol is just above the disk. Do not allow any air to pass through the disk or reach the top surface of the disk. Immediately add 5ml of deionized water to the disk and draw through at

low vacuum until the water almost reaches the top surface of the disk. It is preferable to leave extra liquid above the disk rather than allow any air to contact the surface of the disk.

5 Sample Addition:
Add the sample onto the disk, directly to the film of water left from the conditioning step. Adjust the vacuum to 10 in. Hg (35 kPa) for a flow rate of approximately 100ml per minute until the entire sample has been passed through the disk.

6 Disk Drying:
After the sample has been processed, draw air through the disk under vacuum at approximately 15 in. Hg (50 kPa) for approximately 5 minutes.

7 Analyte Elution:
Release system vacuum. Insert the sample collection rack and collection vessels. Reassemble the apparatus. Add 5ml methylene chloride directly to the sample bottle and gently swirl to rinse all inner surfaces of the bottle. Allow the sample bottle to stand for 1 to 2 minutes and transfer the methylene chloride to the disk using a glass pipet. Rinse the reservoir sides in the process. Draw the solvent through the disk at 5 in. Hg (17 kPa). Repeat the bottle rinse

and disk elution twice with fresh aliquots of methylene chloride, combining all eluates in the collection tube.

8 Final Analysis:
Remove water from sample eluate by passing it through approximately 3 grams of anhydrous sodium sulfate. Concentrate to 1ml and analyze 1µl by GC/MS.

SIMDisk™-GF:
cat.# 24004, 20-pack

*EPA methods are available from the National Technical Information Service:
703-487-4650*

Accuracy/Precision data from four determinations of Method 525.1 analytes at 2µg/L with Liquid-Solid SIMDisk™-GF 47mm extraction disk and the Finnigan MAT ITS40 Ion Trap MS

compound	target conc. (µg/L)	mean (µg/L)	std. dev. (µg/L)	% RSD	accuracy (% of target)	% REC. in method
acenaphthalene-d10	5	-----	-----	-----	-----	-----
henanthrene-d10	5	-----	-----	-----	-----	-----
chrysene-d12	5	-----	-----	-----	-----	-----
hexachlorocyclopentadiene	2	1.6	0.30	2.1	80	55
dimethylphthalate	2	1.8	0.17	9.4	90	95
acenaphthylene	2	2.0	0.60	3.1	100	95
2-chlorobiphenyl	2	2.0	0.50	2.4	100	95
diethylphthalate	2	2.1	0.70	3.3	105	100
fluorene	2	2.1	0.60	3.1	105	110
2,3-dichlorobiphenyl	2	2.0	0.70	3.2	100	115
hexachlorobenzene	2	2.0	0.60	2.8	100	85
simazine	2	1.9	0.19	10.2	95	105
atrazine	2	2.1	0.16	7.5	105	110
pentachlorophenol	8	9.7	0.79	8.2	121	97
gamma-BHC	2	2.1	0.40	2.2	105	105
phenanthrene	2	2.2	0.40	1.9	110	120
anthracene	2	2.0	0.90	4.6	100	85
2,4,5-trichlorobiphenyl	2	1.9	0.40	1.9	95	85
alachlor	2	2.1	0.40	1.7	105	-----
heptachlor	2	1.9	0.40	2.2	95	110
di-n-butylphthalate	2	2.5	0.24	9.5	125	110
2,2',4,4'-tetrachlorobiphenyl	2	1.9	0.20	1.2	95	75
aldrin	2	1.6	0.20	12.7	80	80
heptachlor epoxide (isomer B)	2	2.1	0.50	2.5	105	115
2,2',3',4,6-pentachlorobiphenyl	2	1.9	0.50	2.4	95	95
gamma-chlordane	2	1.9	0.80	4.1	95	110
pyrene	2	2.0	0.40	2.0	100	95
alpha-chlordane	2	1.9	0.50	2.8	95	100
trans-nonachlor	2	1.9	0.70	3.7	95	135
2,2',4,4',5,6'-hexachlorobiphenyl	2	1.7	0.14	8.0	85	80
endrin	2	2.2	0.50	2.2	110	90
butylbenzylphthalate	2	2.2	0.12	5.4	110	100
bis(2-ethylhexyl)adipate	2	1.8	0.21	11.9	90	80
2,2',3,3',4,4',6'-heptachlorobiphenyl	2	1.8	0.40	1.9	90	70
methoxychlor	2	2.1	0.50	2.5	105	90
2,2',3,3',4,5',6,6'-octachlorobiphenyl	2	1.7	0.20	1.2	85	90
benzo(a)anthracene	2	1.9	0.20	0.9	95	90
chrysene	2	1.9	0.20	0.9	95	110
bis(2-ethylhexyl)phthalate	2	2.2	0.40	2.0	110	95
benzo(b)fluoranthene	2	2.0	0.90	4.2	100	-----
benzo(k)fluoranthene	2	2.0	0.80	4.2	100	105
benzo(a)pyrene	2	2.0	0.10	5.1	100	40
perylene-d12	5	4.7	0.34	7.3	94	100
indeno(1,2,3-cd)pyrene	2	1.9	0.22	11.6	95	20
dibenzo(a,h)anthracene	2	1.8	0.20	11.3	90	15
benzo(g,h,i)perylene	2	1.8	0.18	9.9	90	35

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