

SPE of Chlorinated Pesticides in Water

Application Brief

Condition: Add 5 mL acetone to the cartridge. Apply vacuum and discard the eluant. Repeat with 5 mL methanol then 5 mL water. Do not allow the sorbent to go dry at any point during this step.

Load: Attach a sample reservoir to the top of the cartridge. Add 0.2 mL methanol to 20 mL of the water sample.¹ Mix, then add to the cartridge. Apply the vacuum and discard the eluant. The flow rate should be no greater than 10 mL/min.²

Rinse: Add 3 mL water to the cartridge. Apply vacuum and discard the eluant. Leave the vacuum on for 30 seconds after all of the water has passed through the cartridge. Centrifuge the cartridge at 1000–1500 rpm for 5 minutes.³

Elution: Place a collection tube beneath the cartridge. Add 3 mL of acetone to the cartridge. Apply vacuum and collect the eluant. Concentrate to dryness under a stream of dry nitrogen⁴ (transfer to a small vial may be necessary). Dissolve the residue in 200 μ L acetone. Inject 2 μ L into the GC.

¹ Volume up to 100 mL may be used. Adjust the amount of added methanol so that the final concentration is 1%.

² Using slower flow rates will result in slightly better recovery values.

³ Centrifuging removes additional water which aids in sample concentration.

⁴ The use of heat to aid in sample concentration may result in reduced recovery values.

Equipment

- AccuBOND[®] ODS (C18) 6 mL/500 mg cartridge (P/N 188-1356)
- 25 mL sample reservoir (P/N 700-4007)
- coupling fitting (P/N 5185-5794)
- vacuum manifold (P/N 5185-5754, 10-port) (P/N 5185-5765, 20-port)

Reagents

- water (HPLC grade)
- methanol (pesticide grade)
- acetone (pesticide grade)



Compound	k	\bar{x}	Std. Dev.
Trifluralin	5.52	90	12
Chloroneb	5.63	90	6
Propachlor	6.65	87	7
Hexachlorobenzene (HCB)	7.10	69	13
α -BHC	7.43	73	3
γ -BHC (lindane)	8.16	49	18
β -BHC	8.26	83	4
Heptachlor	8.75	62	7
Chlorothalonil	8.76	86	2
δ -BHC	8.93	97	4
Alachlor	9.03	83	5
Aldrin	9.38	67	5
DCPA	9.80	66	3
Heptachlor epoxide	10.86	72	7
o,p'-DDE	11.49	66	11
Endosulfan 1	11.55	81	9
p,p'-DDE	12.15	88	5
Dieldrin	12.52	86	11
o,p'-DDD	13.12	68	9
Chlorobenzilate	13.35	86	8
Endrin	13.85	97	12
p,p'-DDD	14.17	80	8
o,p'-DDT	14.25	64	14
Endosulfan II	14.48	91	7
p,p'-DDT	15.27	79	6
Endrin aldehyde	15.64	85	3
Endosulfan sulfate	16.07	90	7
Methoxychlor	18.50	92	11

Water spiked at 50 ppb

$n = 4$

k = partition ratio (a measure of retention)

\bar{x} = % recovery

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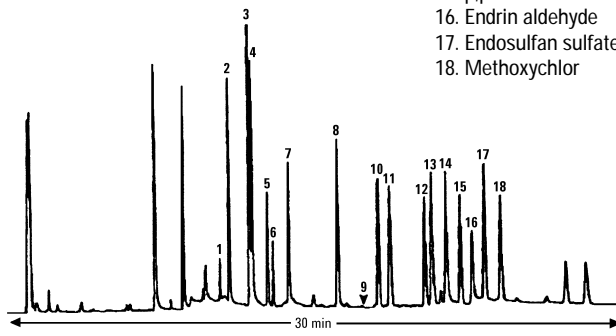
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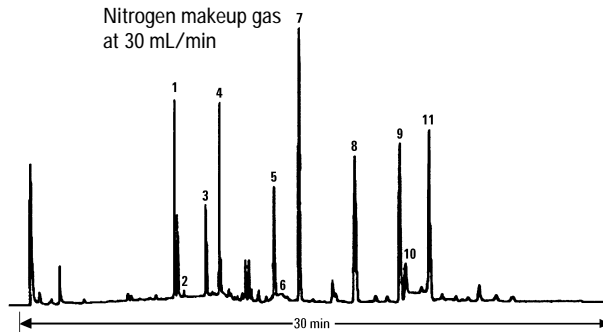
Pond Water Extract Containing Chlorinated Pesticides

Column:	DB-608 30 m x 0.53 mm I.D.	1. Hexachlorobenzene
P/N:	125-1730	2. α -BHC
Film Thickness:	0.83 μ m	3. γ -BHC
Carrier:	Helium at 40 cm/sec (measured at 140°C)	4. β -BHC
Oven:	140°C for 2 min	5. Heptachlor
	140-240°C at 10°/min	6. δ -BHC
	240°C for 5 min	7. Aldrin
	240-265°C at 5°/min	8. Heptachlor epoxide
	265°C for 5 min	9. Endosulfan 1
Injector:	Megabore® direct, 250°C	10. p,p'-DDE
Detector:	ECD, 325°C	11. Dieldrin
	Nitrogen makeup gas at 30 mL/min	12. Endrin
		13. p,p'-DDD
		14. Endosulfan II
		15. p,p'-DDT
		16. Endrin aldehyde
		17. Endosulfan sulfate
		18. Methoxychlor



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