



Solid-Phase Extraction of Pesticides in Water using Graphitized Carbon Black (GCB)

UCT Part Numbers:

EUCARB1M6 (1000 mg GCB (non-porous, 120/400 mesh), 6 mL)

AD0000AS (cartridge adaptor)

RFV0075P (reservoirs, 75 mL)

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Graphitized carbon black (GCB) is a reverse phase and anion exchange sorbent. GCB retains non-polar compounds, such as organochlorine pesticides, and some very polar compounds, such as surfactants, which are difficult to retain by other reverse phase sorbents. This simple SPE method uses UCT's proprietary, treated GCB for the determination of pesticides in water providing excellent recovery.

Procedure

1. Cartridge Preparation

- a) Transfer 100 mL of aqueous sample to a glass container
- b) Adjust pH to less than 2 using 6N HCl
- c) Spike as necessary
- d) Connect **RFV0075P** reservoirs to the top of the **EUCARB1M6** cartridges using **AD0000AS** adaptor
- e) Wash cartridges with 10 mL dichloromethane (DCM)
- f) Draw full vacuum to remove all DCM
- g) Add 10 mL methanol and draw down to top of frit
- h) Add 10 mL reagent water and draw down to top of frit
- i) Do not let cartridges go dry after step g)

2. Extraction

- a) Add samples to the reservoirs adjusting vacuum to give a drop-wise flow, about 10 mL/min
- b) Rinse sample containers using 10 mL reagent water and add rinsate to cartridges
- c) Dry cartridges using full vacuum for 10 min

3. Elution

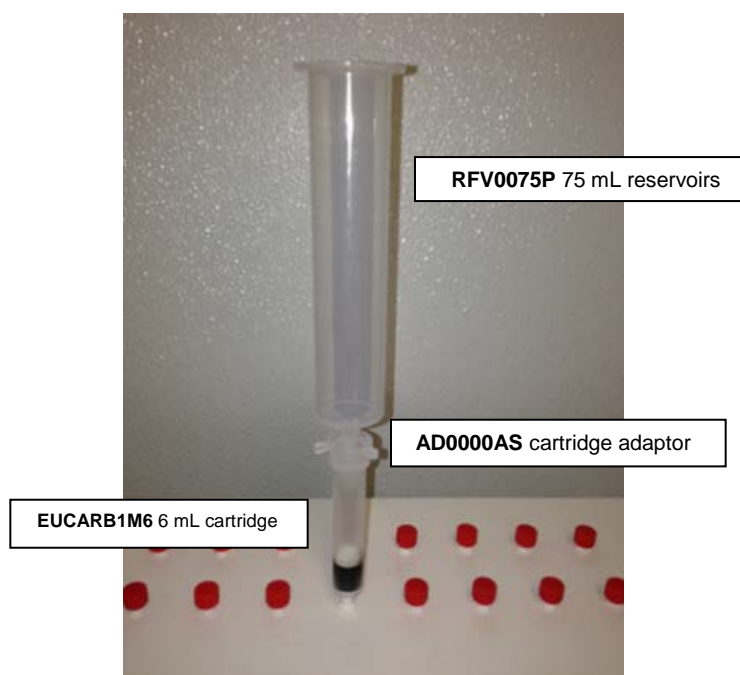
- a) Insert test tubes in the manifold then elute cartridges using 5 mL ethyl acetate dropwise followed by 5 mL of DCM dropwise
- b) Dry extracts by passing through anhydrous Na₂SO₄
- c) Rinse test tubes with DCM and add to Na₂SO₄
- d) Concentrate extracts to 1 mL using a gentle stream of N₂ at 35 °C
- e) Add IS prior to GC/MS analysis

4. Analysis

Parameters

GCMS: Agilent 6890N GC coupled with 5975C MSD
MSD Injector: 1 µL splitless injection at 250 °C
Injection Vol: 1 µL
Liner: 4 mm splitless gooseneck liner with deactivated glass wool (UCT: GCLGN4MM)
Column: Restek Rxi [®] -5sil MS 30m x 0.25mm x 0.25µm
Guard Column: 10 m
Column Flow Rate: 1.2 mL/min
Carrier Gas: He
Full Scan: 45-500 amu
Temperature Program: Initial T 55 °C hold for 1 min; ramp at 10 °C/min to 200 °C; ramp at 7 °C/min to 300 °C; hold for 0.21 min.

Detail of Reservoir, Adaptor, and Cartridge Setup



Accuracy and Precision Data

Compound	Intra-day (n=4)		Inter-day (n=17)	
	Rec%	RSD	Rec%	RSD
alpha Lindane	93	2.1	89	9.3
beta Lindane	96	1.9	91	8.8
gamma Lindane	93	1.7	92	8.3
delta Lindane	95	3.3	89	11.7
Heptachlor	97	3.2	91	11.1
Aldrin	95	1.5	84	12.9
Heptachlor epoxide	102	2.4	97	12.0
trans-Chlordane	93	3.8	90	8.8
Endosulfan I	94	5.0	91	8.4
cis-Chlordane	96	3.3	91	9.7
p,p'-DDE	91	3.5	89	8.8
Dieldrin	98	1.9	93	9.4
Endrin	100	2.1	95	11.8
Endosulfan II	105	1.4	97	10.3
p,p'-DDD	98	2.2	92	9.8
Endrin aldehyde	95	5.4	92	9.3
Endosulfan sulfate	102	3.8	97	10.2
p,p'-DDT	99	3.0	94	9.6

Endrin ketone	106	2.1	99	10.9
Methoxychlor	105	2.7	99	10.5
Dichlofluanid	107	2.8	98	10.8
Dicofol	95	0.7	86	11.6
Tolyfluanide	106	3.1	98	11.6
Captan	119	4.2	105	13.4
Folpet	107	3.9	95	10.0
Overall average	99	2.8	93	10.4

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