

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

Summary:

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

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

2. Extraction

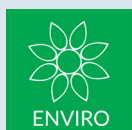
- Add samples to the reservoirs adjusting vacuum to give a dropwise flow, about 10 mL/min.
- Rinse sample containers using 10 mL reagent water and add rinsate to cartridges.
- Dry cartridges using full vacuum for 10 min.

3. Elution

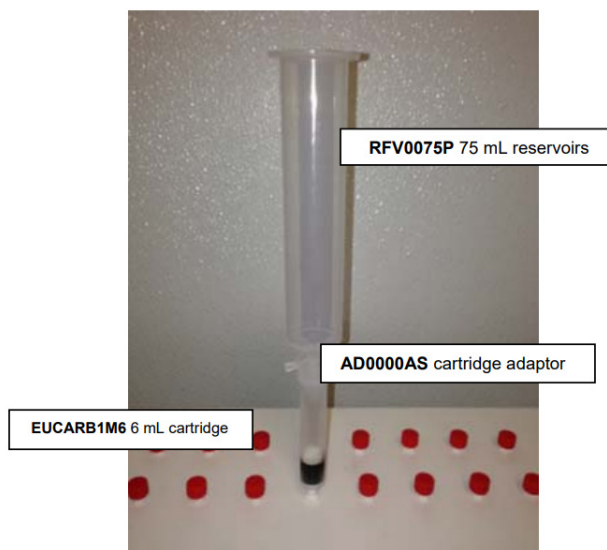
- Insert test tubes in the manifold then elute cartridges using 5 mL ethyl acetate dropwise followed by 5 mL of DCM dropwise.
- Dry extracts by passing through anhydrous Na_2SO_4
- Rinse test tubes with DCM and add to Na_2SO_4
- Concentrate extracts to 1 mL using a gentle stream of N_2 at 35°C.
- Add IS prior to GC/MS analysis.

4. Analysis

Parameters	
GC/MS	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 0.25 μm
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	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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