Florisil® SPE Cleanup

for Chlorinated Pesticides Analysis

Many chlorinated pesticides have been banned for use because of their short- and long-term toxicity, carcinogenicity, and environmental persistence. A list of these chemicals, some of which are still actively applied in the field, is included in U.S. EPA Method 8081B. Despite the fact that most of these chlorinated pesticides are now illegal to use, manufacture, and transport in many areas, organochlorines are a common source of pesticide poisoning that results in reportable illness. Although most of these chlorinated pesticides, insecticides, and herbicides have limited water solubility and mobility, they bioaccumulate and persist in the environment. Since there is an ongoing risk of exposure from a number of sources, it is essential to test soils, wastewater, and sediments for their presence.

Standard EPA methods for the preparation and analysis of pesticide-containing hazardous wastes require initial liquid/liquid extraction with dichloromethane, gel permeation chromatography (GPC) fractionation of higher molecular weight interferences, and a final cleanup of polar contaminants (like trichlorophenol) with Florisil® columns or Florisil® solid phase extraction (SPE) tubes before analysis with GC electron capture detection (ECD). Many labs have found that these sample cleanup precautions reduce high background levels that result in difficult quantitation and frequent GC/detector maintenance. With Florisil® cleanup, extracts have lower backgrounds, producing better chromatograms with less interference. In addition, the lifetime of inlet liners and guard columns is extended, and maintenance of sensitive ECD detectors is reduced. Results in Table I show that recovery levels are excellent for chlorinated pesticides, when following the method described in Figure 1.

Table I: Excellent recoveries are obtained for chlorinated pesticides when sample extracts are cleaned with Florisil® SPE tubes. (n=6)

Compound	t_R (min)	% Recovery
Trichlorophenol	4.31	3.7
2,4,5,6-Tetrachloro-*m*-xylene	6.23	85.2
α-BHC	7.21	86.5
γ-BHC	7.74	88.4
β-BHC	7.86	88.9
δ-BHC	8.27	93.1
Heptachlor	8.35	97.0
Aldrin	8.78	88.0
Heptachlor epoxide	9.52	93.4
γ-Chlordane	9.75	94.9
α-Chlordane	9.94	89.5
Endosulfan I	10.00	90.2
4,4'-DDE	10.15	89.4
Dieldrin	10.34	101.2
Endrin	10.71	96.5
4,4'-DDD	10.84	93.7
Endosulfan II	10.96	92.8
4,4'-DDT	11.22	94.5
Endrin aldehyde	11.37	94.1
Endosulfan sulfate	11.69	89.9
Methoxychlor	12.03	108.4
Endrin ketone	12.32	91.5
Decachlorobiphenyl	13.76	108.4

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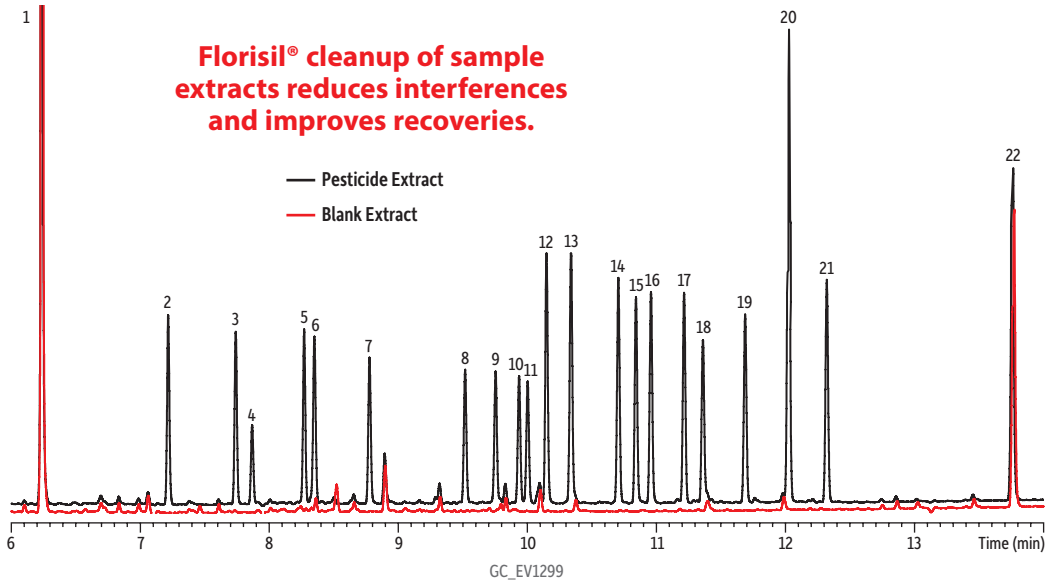
Figure 1: Extracts of a sample and blank demonstrate low background levels and good responses for target pesticides.



	Peaks	Conc. (ng/mL)
1.	2,4,5,6-Tetrachloro-*m*-xylene (SS)	20
2.	α-BHC	5
3.	γ-BHC	5
4.	β-BHC	5
5.	δ-BHC	5
6.	Heptachlor	5
7.	Aldrin	5
8.	Heptachlor epoxide	5
9.	γ-Chlordane	5
10.	α-Chlordane	5
11.	Endosulfan I	5
12.	4,4'-DDE	10
13.	Dieldrin	10
14.	Endrin	10
15.	4,4'-DDD	10
16.	Endosulfan II	10
17.	4,4'-DDT	10
18.	Endrin aldehyde	10
19.	Endosulfan sulfate	10
20.	Methoxychlor	50
21.	Endrin ketone	10
22.	Decachlorobiphenyl (SS)	20

Column: Rtx®-CLPesticides2, 30 m, 0.32 mm ID, 0.25 µm (cat.# 11324) using Rxi® guard column 5 m, 0.32 mm ID (cat.# 10039) with universal "Y" Press-Tight® connector (cat.# 20406-261); **Sample:** 2,4,5,6-Tetrachloro-*m*-xylene (cat.# 32027), Decachlorobiphenyl (BZ #209) (cat.# 32029), Organochlorine pesticide mix AB #2 (cat.# 32292); **Injection:** Inj. Vol.: 2 µL splitless (hold 0.75 min); Liner: Restek Premium 4 mm single taper w/wool (cat.# 23303.5); Inj. Temp.: 250 °C; Purge Flow: 50 mL/min; **Oven:** Oven Temp: 110 °C (hold 0.5 min) to 320 °C at 15 °C/min (hold 5 min); **Carrier Gas:** He, constant flow; Flow Rate: 3.5 mL/min; **Detector:** µ-ECD @ 330 °C; Make-up Gas Flow Rate: 50 mL/min; Make-up Gas Type: N_2; Data Rate: 50 Hz; **Instrument:** Agilent/HP6890 GC; **Notes:** A mixed standard was prepared in 1 mL hexane (see peak list for nominal concentration of each component). For cleanup, a Florisil® tube (cat.# 24034) was first conditioned with 6 mL hexane. The 1 mL standard was then loaded on the tube and eluted with hexane:acetone (90:10), collecting 10 mL of eluent. The eluent was then concentrated down to 1 mL and analyzed.

All cartridges are manufactured using high density polypropylene and have polyethylene frits unless otherwise noted.

Cartridges may be processed by any one or all of these techniques: positive pressure, sidearm flask, centrifuge, or vacuum manifold.

Resprep® SPE Cartridges (Normal Phase)

Hydrophilic (polar) adsorbents used to extract hydrophilic analytes from nonpolar matrices, such as organic solvents (e.g., polar contaminants from sample extracts).

	3 mL/500 mg (50-pk.)	6 mL/500 mg (30-pk.)	6 mL/1000 mg (30-pk.)	15 mL/2 g (15-pk.)
Florisil (EPA SW 846 methods and CLP protocols)	24031 24032*	26086**	24034 26085**	26228

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Japan • phone: +81 (3)6459 0025 • fax: +81 (3)6459 0025 • e-mail: restekjapan@restek.com
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