



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.

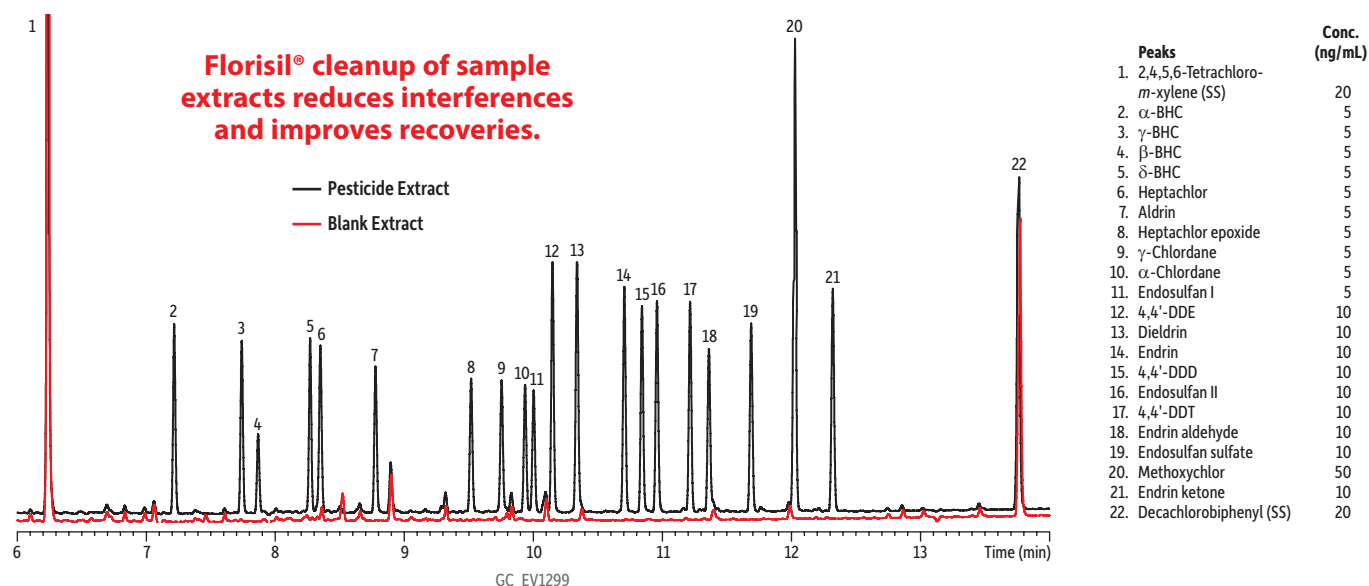
Standard EPA methods for the preparation and analysis of pesticide-containing hazardous wastes require initial liquid/liquid extraction with dichloromethane, gel permeation chromatography (GPC) fractionation of higher molecular weight interferences, and a final cleanup of polar contaminants (like trichlorophenol) 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- <i>m</i> -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

Visit www.restek.com/florisil today!

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



Column: Rtx®-CLPesticides2, 30 m, 0.32 mm ID, 0.25 μ m (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 μ L 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/min 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:** N₂; **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

*PTFE frits

**Glass tubes with PTFE frits

Contact your Restek representative and order yours today!

Visit www.restek.com/Contact-Us to find a distributor or representative.

PATENTS & TRADEMARKS

Restek® patents and trademarks are the property of Restek Corporation. (See www.restek.com/Patents-Trademarks for full list.) Other trademarks appearing in Restek® literature or on its website are the property of their respective owners. The Restek® registered trademarks used here are registered in the United States and may also be registered in other countries.

RESTEK

U.S. • 110 Benner Circle • Bellefonte, PA 16823 • 1-814-353-1300 • 1-800-356-1688 • fax: 1-814-353-1309 • www.restek.com

China • phone: +86-10-5629-6620 • fax: +86-10-5814-3980 • cn.restek.com

France • phone: +33 (0)1 60 78 32 10 • fax: +33 (0)1 60 78 70 90 • www.restek.fr

Germany • phone: +49 (0)6172 2797 0 • fax: +49 (0)6172 2797 77 • www.restekgmbh.de

Italy • phone: +39-02-7610037 • fax: +39-02-70100100 • www.superchrom.it

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

UK • phone: +44 (0)1494 563377 • fax: +44 (0)1494 564990 • www.thamesrestek.co.uk

Lit. Cat.# EVTS1681-UNV

© 2012 Restek Corporation. All rights reserved.

Printed in the U.S.A.

