

Improve Results for GC-ECD Analysis of Chlorinated Pesticides

With Resprep® CarboPrep® SPE Cleanup Cartridges

- Use CarboPrep® SPE cartridge cleanup to remove coextracted sterols, pigments, and nonpolar compounds.
- Improve accuracy for chlorinated pesticide samples by reducing matrix interferences.
- Obtain baseline resolution and excellent recoveries for Method 8081B analytes.



Many chlorinated pesticides have been banned for use because of their short- and long-term toxicity, carcinogenicity, and environmental persistence. An expanded list of these chemicals, some of which are still actively applied in the field, has been included in the updated U.S. Environmental Protection Agency (EPA) Method 8081B. Despite the fact that most of these chlorinated pesticides are now illegal to use, manufacture, and transport in the U.S., organochlorine compounds are still a potential source of pesticide poisoning. Although most of these chlorinated pesticides have limited water solubility and mobility, they do bioaccumulate and persist in the environment. Since there is an ongoing risk for exposure from a number of sources, it is still essential to test soils, wastewater, and sediments for their presence.

One of the primary challenges chemists face with 8081B analysis is chromatographic interference from matrix compounds. This is because the methylene chloride or acetone/methylene chloride solvent used for the extraction of water and soil also removes a wide variety of organic matrix compounds. To reduce interferences in the sample extracts, gel permeation chromatography (GPC) and/or solid phase cleanup is recommended. The GPC cleanup step removes high molecular weight organic compounds, which can create active sites in the injector if they do not volatilize completely. For solid phase cleanup, Florisil® absorbent is commonly used to clean extracts by removing polar contaminants.

Even after GPC and Florisil® cleanup steps, matrix interferences typically still remain. SPE cleanup of chlorinated pesticide samples with Resprep® CarboPrep® SPE cartridges can further improve results by removing sterols, pigments, and nonpolar interferences, without compromising the recovery of pesticides. Extracts will have lower backgrounds, which results in fewer

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chromatographic interferences, extended lifetime for inlet liners and guard columns, and reduced maintenance of sensitive ECD detectors. Results in Table I show that recovery levels are excellent for this chlorinated pesticides list, when following the cleanup procedure described in Figure 1. While many of the components included in EPA Method 8081B are difficult to fully resolve using GC-ECD, target components can be reliably separated using the Rtx®-CLPesticides and Rtx®-CLPesticides2 column pair.

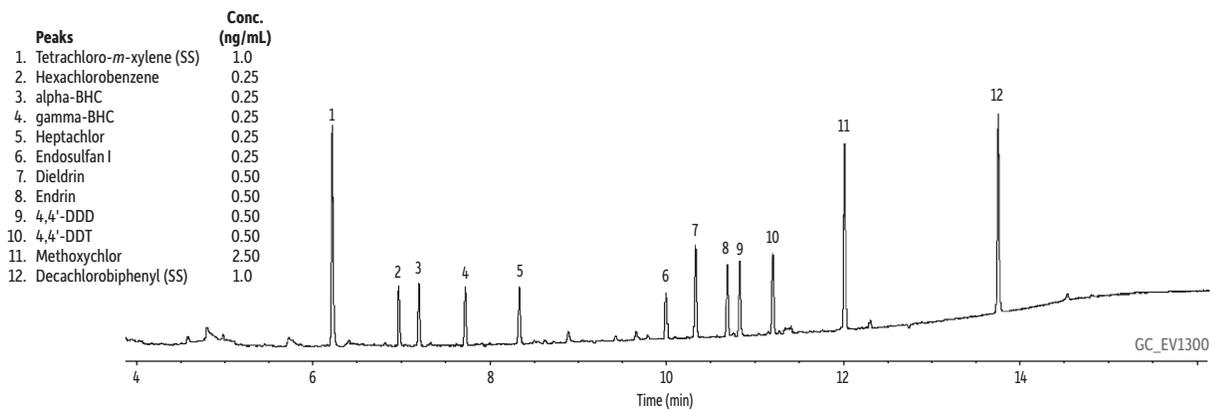
Adding Resprep® CarboPrep® 90 cartridges for SPE cleanup of chlorinated pesticide samples is an effective way to minimize interferences and improve method accuracy. Labs interested in improving results for chlorinated pesticides analysis should consider adding CarboPrep® SPE cleanup to existing procedures and switching to Rtx®-CLPesticides and Rtx®-CLPesticides2 columns.

Table I: Excellent recoveries of chlorinated pesticides are obtained using Resprep® CarboPrep® SPE cartridges.

Compound	%Recovery	%RSD
Tetrachloro- <i>m</i> -xylene	99.3	0.6
Hexachlorobenzene	100.6	1.3
α-BHC	99.6	0.4
γ-BHC	99.3	1.3
Heptachlor	98.9	0.8
Endosulfan I	101.9	4.7
Dieldrin	102.0	1.3
Endrin	103.2	1.8
4,4'-DDD	100.7	1.1
4,4'-DDT	102.8	2.1
Methoxychlor	101.0	4.1
Decachlorobiphenyl	102.4	0.6

See Figure 1 for analyte concentrations, as well as preparation and analysis details (n = 3).

Figure 1: Extract cleanup using Resprep® CarboPrep® SPE cartridges and analysis of chlorinated pesticides on an Rtx®-CLPesticides2 column.



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 Pesticide standard mix A (cat.# 32297)
2,4,5,6-Tetrachloro-*m*-xylene (cat.# 32027)
Decachlorobiphenyl (BZ #209) (cat.# 32029)
Hexachlorobenzene (cat.# 32231)
Hexane

Diluent: Hexane

Injection
Inj. Vol.: 2 μL splitless (hold 0.75 min)
Liner: Sky® 4.0 mm ID single taper inlet liner 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
Carrier Gas: He, constant flow
Flow Rate: 3.5 mL/min

Detector
Detector: μ-ECD @ 330 °C
Make-up Gas Flow Rate: 50 mL/min
Make-up Gas Type: N₂
Data Rate: 50 Hz

Instrument
Instrument: Agilent/HP6890 GC

Notes
Sample: The four standards listed above were diluted in hexane to create a 5-50 ppb sample solution.
Conditioning: Resprep® CarboPrep® SPE cartridges (3 mL, 250 mg, cat.# 26091) were conditioned using 3 mL hexane:dichloromethane (80:20).
Cleanup: 1 mL of the sample solution was applied to the SPE cartridge and eluted using 20 mL of hexane:dichloromethane (80:20).

Product Listings

Resprep® CarboPrep® SPE Cartridges

- Improved recovery of sulfonylurea herbicides, phenols, carbamates, and triazine herbicides, compared to C18 and C8 cartridges.
- Wide range of selectivity for both analytes and their metabolites or degradation products.
- Rapid sampling flow rates; uncompromised recoveries.
- Maximum capacity for contaminant cleanup.
- Controlled manufacturing improves cleanliness, ensures reproducible performance.



Excellent for
Pesticide Residue
Cleanup!

SPE Cartridge	Tube Volume, Bed Weight	qty.	cat.#
CarboPrep 90	3 mL, 250 mg	50-pk.	26091
CarboPrep 90	6 mL, 500 mg	30-pk.	26092

Rtx®-CLPesticides/Rtx®-CLPesticides2

- Application-specific columns for organochlorine pesticides and herbicides.
- Low bleed—ideal for GC-ECD or GC-MS analyses.
- Baseline separations in less than 10 minutes.
- Stable to 340 °C.
- Analyze EPA Method 8081B, 8082A, 8151A, 504.1, 515, 508.1, and 552.2 compounds without time-consuming column change.

Rtx®-CLPesticides Columns (fused silica)

(proprietary Crossbond® phases)

ID	df	temp. limits	15-Meter	20-Meter	30-Meter	60-Meter
0.18 mm	0.18 µm	-60 to 320/340 °C		42102		
0.25 mm	0.25 µm	-60 to 320/340 °C	11120		11123	11126
0.32 mm	0.32 µm	-60 to 320/340 °C			11141	
	0.50 µm	-60 to 320/340 °C	11136		11139	
0.53 mm	0.50 µm	-60 to 300/320 °C	11137		11140	

Rtx®-CLPesticides2 Columns (fused silica)

(proprietary Crossbond® phases)

ID	df	temp. limits	10-Meter	15-Meter	20-Meter	30-Meter	60-Meter
0.18 mm	0.14 µm	-60 to 320/330 °C	42301		42302		
0.25 mm	0.20 µm	-60 to 320/340 °C				11323	11326
0.32 mm	0.25 µm	-60 to 320/340 °C		11321		11324	
	0.50 µm	-60 to 320/340 °C				11325	
0.53 mm	0.42 µm	-60 to 300/320 °C		11337		11340	



Pesticide Standard Mix A (9 components)

α-BHC	5 µg/mL	4,4'-DDT	10	endrin	10
γ-BHC (lindane)	5	dieldrin	10	heptachlor	5
4,4'-DDD	10	endosulfan I	5	methoxychlor	50
In hexane:toluene (90:10), 1 mL/ampul			cat.# 32297 (ea.)		

Decachlorobiphenyl (BZ #209)

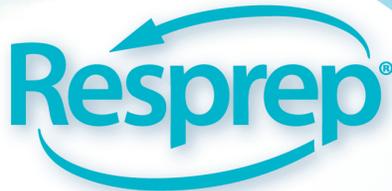
decachlorobiphenyl	
200 µg/mL in acetone, 1 mL/ampul	cat.# 32029 (ea.)
200 µg/mL in acetone, 5 mL/ampul	cat.# 32030 (ea.)
10 µg/mL in isooctane, 1 mL/ampul	cat.# 32289 (ea.)

2,4,5,6-Tetrachloro-*m*-xylene

2,4,5,6-tetrachloro- <i>m</i> -xylene	
200 µg/mL in acetone, 1 mL/ampul	cat.# 32027 (ea.)
200 µg/mL in acetone, 5 mL/ampul	cat.# 32028 (ea.)

Hexachlorobenzene

hexachlorobenzene	
1,000 µg/mL in acetone, 1 mL/ampul	cat.# 32231 (ea.)



Solid Phase Extraction Cartridges from Restek

Proven Quality, Superior Cleanliness, and Method-Specific Performance

Resprep® solid phase extraction (SPE) cartridges are manufactured with specially cleaned sorbents and high-purity materials to minimize background and to help eliminate troublesome interference. In order to ensure reproducibility, every finished product goes through rigorous QC testing, targeted to specific applications whenever possible, and an extensive certificate of analysis details the results.

Available with the following sorbents:

- **Silica:** Multipurpose
- **EPH Silica:** Petroleum
- **Florisil® Adsorbent:** Pesticides
- **CarboPrep® Adsorbent:** Dirty Samples

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