

## Florisil® SPE cleanup for organochlorine pesticides and polychlorinated biphenyl compounds (PCBs)

*This procedure is adapted from the US Environmental Protection Agency (EPA) Contract Laboratory Program, Statement of Work for Organic Analyses, Multimedia, Multi-Concentration, OLM04.0. It is not intended to be a replacement or substitute for the official procedure. Please refer directly to the published procedure for additional information.*

### Preparing for Sample Cleanup

**A. Determine the size of Florisil® solid phase extraction (SPE) tube needed for sample cleanup.** If the gas chromatography (GC) autosampler can operate reliably with 1mL of sample extract, then a 500mg tube (cat.# 24031 & 24032) is used and the required final volume is 1mL. If the GC autosampler requires a larger sample volume, prepare 2mL of sample extract using a 1g tube (cat.# 24034). Manual injection requires only 1mL final extract and a 500mg tube. See *Tips for Better Results* below.

**B. Every lot of Florisil® adsorbent must be quality control-tested for activity level before using for sample cleanup.** Add 0.5mL of 2,4,5-trichlorophenol solution (0.1µg/mL in acetone) and 0.5mL of Standard Mixture A (midpoint concentration) to 4mL of hexane. Reduce the final volume to 0.5mL using nitrogen. Place mixture onto the top of a washed Florisil® cartridge, and elute it with 9mL of hexane:acetone (90:10) v/v. Use two additional 1mL hexane rinses to ensure quantitative transfer of standard from the Florisil® tube. Reduce the final volume to 1mL using nitrogen, and analyze the solution by GC/electron capture detector (ECD) using at least one of the GC columns specified for sample analysis. Determine the recovery and percent recovery for each analyte. The check sample must be analyzed on a GC/ECD and meet the initial calibration and calibration verification technical acceptance criteria. The Florisil® lot is acceptable if all pesticides are recovered at 80% to 120%, if the recovery of 2,4,5-trichlorophenol is less than 5%, and if no peaks interfering with the target analytes are detected.

### Tips for Better Results

- 1g tubes will give the most consistent results regardless of final sample volume.
- Flow rate during elution should be either dropwise or gravity feed (no vacuum). This will reduce trichlorophenol breakthrough.

### Procedure for Sample Cleanup Using Florisil® SPE Tubes

1. Attach the vacuum manifold (cat.# 26077 or 26080) to a vacuum pump, install a trap between the manifold and the vacuum source. Adjust the vacuum pressure in the manifold to <20" Hg. Place one Florisil® tube onto the vacuum manifold for each sample extract.
2. Prior to cleanup of samples, the tubes must be washed with hexane:acetone (90:10). This is accomplished by placing the tube on the vacuum manifold, pulling a vacuum, and passing at least 5mL of the hexane:acetone solution through the tube. While the tubes are being washed, adjust the vacuum applied to each tube so that the flow rate through each cartridge is approximately equal. Do not allow the cartridges to dry after they have been washed.
3. After the tubes on the manifold are washed, release the vacuum and place a rack containing labeled 10mL collection vessels inside the manifold.
4. After the collection vessels are in place, restore the vacuum to the manifold. Transfer a volume of extract equal to the required final volume (1 or 2mL)\* from each sample, blank or matrix spike to the top frit of the appropriate Florisil® tube. Use a syringe or volumetric pipette to transfer the extract to the cleanup tube.
5. Elute the pesticides and PCBs in the extract concentrates through the column with 8mL of hexane:acetone (90:10) and collect into the 10mL collection vessels that are held in the vacuum manifold rack.
6. Transfer the elute in each collection vessel to a clean, appropriate vessel for nitrogen blowdown. Perform two additional 1mL hexane rinses to ensure quantitative transfer of the tube elute.
7. Adjust the extract to the final volume (1 or 2mL) by using either nitrogen blowdown or a micro Snyder column. Measure the final volume with a syringe or by transferring the extract to a volumetric flask. The extract is ready for GC/ECD analysis.

\*This volume must equal the final volume after Florisil® cleanup.

# Product Listing

## Rtx<sup>®</sup>-CLPesticides Column

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.25	-60 to 310/330°C	11120	11123
0.32mm	0.50	-60 to 310/330°C	11136	11139
0.53mm	0.50	-60 to 310/330°C	11137	11140

## Rtx<sup>®</sup>-CLPesticides2 Column

ID	df (µm)	temp. limits	15-Meter	30-Meter
0.25mm	0.20	-60 to 310/330°C	11320	11323
0.32mm	0.25	-60 to 310/330°C	11321	11324
0.53mm	0.42	-60 to 310/330°C	11337	11340

## Resprep<sup>™</sup> SPE Cartridges

(All cartridges are polypropylene and have polyethylene frits unless otherwise noted):

### Florisil<sup>®</sup>

3mL	500mg	cat.# 24031	50-pk.
3mL†	500mg	cat.# 24032*	50-pk.
6mL	1000mg	cat.# 24034	30-pk.
6mL†	500mg	cat.# 26086**	30-pk.
6mL†	1000mg	cat.# 26085**	30-pk.

†These cartridges are specified in the CLP Method.

\*Stainless steel frits

\*\*Glass cartridges with Teflon<sup>®</sup> frits.

## Resprep<sup>™</sup> 12- & 24-Port Tube Manifold

Complete manifolds include glass basin with built-in vacuum regulator, polypropylene top plate with 12 or 24 individual control valves, 12- or 24-position collection rack, and 12 or 24 Teflon<sup>®</sup> sample guides.

**Complete Resprep<sup>™</sup>-12 Port Manifold:** cat.# 26077

**Complete Resprep<sup>™</sup>-24 Port Manifold:** cat.# 26080

## Florisil<sup>®</sup> Cartridge Check Mix

1000µg/mL in acetone, 1mL/ampul

	Each	5-pk.	10-pk.
	32017	32017-510	
w/data pack	32017-500	32017-520	32117

**Restek Trademarks:** Diskcover, Resprep, Rtx, and the Restek logo.

**Other Trademarks:** Florisil (U.S. Silica Co.), Teflon (E.I. du Pont de Nemours & Company, Inc.).

For permission to reproduce any portion of this application note, please contact Restek's publications/graphics department by phone (ext. 2128) or FAX.



© Copyright 2000, Restek Corporation

**USA:** 110 Benner Circle, Bellefonte, PA 16823 • phone: (800) 356-1688 • fax: (814) 353-1309  
**Germany:** Schaberweg 23, 61348 Bad Homburg • phone: (49) 06172 2797 0 • fax: (49) 06172 2797 77  
**France:** 1, rue Montespan, 91024 Evry Cedex • phone: (33) 01 60 78 32 10 • fax: (33) 01 60 78 70 90  
**Thames Restek UK Ltd.:** Fairacres Industrial Centre, Dedworth Road, Windsor, Berkshire, SL4 4LE  
 phone: (44) 01753 624111 • fax: (44) 01753 624666  
**Thames Restek Scotland Ltd.:** Dept. 10, 44-46 Morningside Road, Edinburgh, EH10 4BF  
 phone: 0870 241 1247 • fax: 0870 241 0781  
**Restek Ireland:** 8 Baronscourt Lane, Carryduff, Belfast, BT8 8RR • phone/fax: (44)2 890 814 576