

Solid Phase Extraction of PCBs from Transformer Oil and Waste Oil, and Analysis by Capillary GC

Solid phase extraction is the preferred method for cleanup of polychlorinated biphenyls. The procedure allows you to quantify these regulated compounds in transformer oil and waste oil at concentrations ranging from 5 to 500ppm.

Key Words:

- polychlorinated biphenyls • transformer oil • waste oil

The adverse effects of polychlorinated biphenyls (PCBs) on wild-life have alerted the public to a potential danger and made PCBs regulated substances (1). Because PCBs have been used widely in dielectric, hydraulic, and heat transfer systems, oils used in these systems often must be monitored.

Our solid phase extraction (SPE) cleanup procedure will enable you to quantify PCBs in transformer oil and waste oil at concentrations of 5 to 500ppm. You can process single samples in less than five minutes on a Visi™-1 Single SPE Tube Processor or, alternatively, up to 12 samples simultaneously in a Visiprep™ SPE Vacuum Manifold. PCB recovery rates approach 100% and recovery on different SPE tube lots is highly consistent.

This SPE procedure is a model of simplicity. For extraction of transformer oil, add a 500µL aliquot of isooctane to a Supelclean™ LC-Florisil® solid phase extraction tube, then quickly weigh 0.2g of the transformer oil sample onto the frit. (For accuracy, set the tube on the analytical balance.) Flush PCBs in the oil through the tube with five 2mL aliquots of isooctane, and collect the eluant in a 10mL volumetric flask. Prior to the GC analysis, dilute the sample to the mark and mix thoroughly.

The SPE procedure can also be modified to allow the extraction of PCBs from waste oil samples. The procedure is identical to the procedure used for transformer oil except an unconditioned tube containing 1 gram of silica is attached to the outlet of the Florisil tube using an adapter. A maximum of 0.1 gram of waste oil can be extracted before breakthrough of matrix impurities is observed.

To test our SPE procedure we extracted, in triplicate, transformer oil standards containing 5ppm, 50ppm or 500ppm each of Aroclor® 1260 PCB mixture, 50ppm of Aroclor 1254 PCB mixture, or 50ppm of Aroclor 1242 PCB mixture. We analyzed the extracts on a 30m x 0.32mm ID SPB™-5 capillary column (Figure A). Average absolute recoveries were consistently in the vicinity of 100% (Table 1).

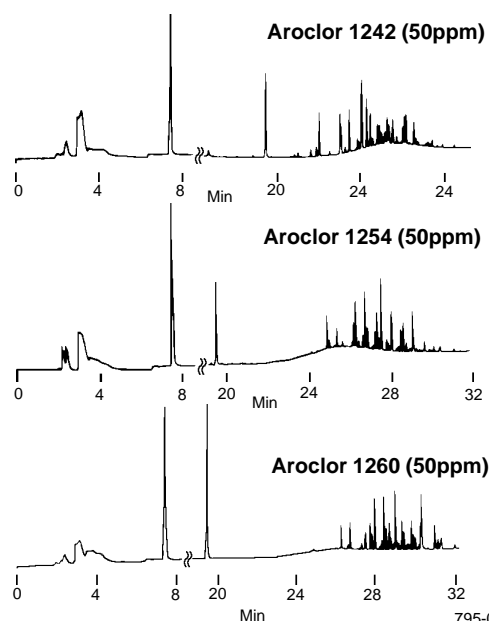
Lot-to-lot reproducibility of extractions on Supelclean LC-Florisil tubes was determined by extracting, from three lots of packing, an oil sample containing 50ppm of Aroclor 1260 PCB mixture. Average absolute recoveries for the three lots were almost identical, and again were approximately 100% (Table 2).

To confirm the usefulness of this SPE procedure for real-life analyses, we obtained two samples of used transformer oil from a power company. PCB content of these oils was determined by extracting the samples, analyzing the extracts, and comparing the peak areas to those for 5ppm, 50ppm and 500ppm standards. We determined that these samples contained 9.8ppm and 57.6ppm of Aroclor 1260 PCBs mixture (Figure B).

For comparison, we also extracted an oil sample having 58ppm Aroclor 1260 according to the currently used sulfuric acid extraction. We diluted 0.2g of oil to 100mL with hexane, then transferred 10ml of the diluted oil to a 40mL screw-cap vial (Teflon® -lined cap) containing 5mL of concentrated sulfuric acid. After shaking the vial for one minute, we allowed the layers to settle, then transferred the upper (organic) layer to a second vial. We analyzed this extract by GC under the conditions previously described.

Figure A. PCBs in Transformer Oil Extract

Extraction Tube: **Supelclean LC-Florisil, 6mL, 1.0g packing**
Cat. No.: **57057**
Column: **SPB-5, 30m x 0.32mm ID, 0.25µm film**
Cat. No.: **24048**
Col. Temp.: **40°C (4 min) to 300°C at 10°C/min, hold 5 min**
Carrier: **nitrogen, 36.3cm/sec**
Det.: **ECD (16 x 10⁻¹¹ AFS)**
Inj.: **1µL of extract or standard, splitless injection (30 sec delay, 50:1 split ratio)**



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Figure C shows that PCBs recovery was significantly better from our SPE method than from the acid extraction. Other significant advantages of the SPE method include: use of concentrated sulfuric acid is avoided, less glassware is used, and solvent costs and disposal problems are significantly reduced (11 mL organic

Table 1. Consistently High Recovery of PCBs from Transformer Oil by Solid Phase Extraction

PCB Mixture	Percent Recovery			
	Trial 1	Trial 2	Trial 3	Mean ± S.D.
Aroclor 1260 (5ppm)	99	118	88	102 ± 15
Aroclor 1260 (50ppm)	109	105	97	104 ± 6
Aroclor 1260 (500ppm)	97	98	97	97 ± 1
Aroclor 1242 (50ppm)	95	94	96	95 ± 1
Aroclor 1254 (50ppm)	104	86	100	97 ± 4
Aroclor 1260 (50ppm)	109	105	97	104 ± 6

Table 2. Consistent Lot-to-Lot Performance of Supelclean LC-Florisil SPE Tubes

Packing Lot. No.	Percent Recovery of 50ppm Aroclor 1260 PCBs			
	Trial 1	Trial 2	Trial 3	Mean ± S.D.
1	109	105	97	104 ± 6
2	99	107	106	104 ± 4
3	100	109	104	104 ± 4

solvent vs. 100mL organic solvent plus 5mL concentrated acid). Furthermore, the SPE method is faster than the acid extraction method (5 minutes vs. 10 minutes), and you can process up to 12 samples simultaneously on the SPE vacuum manifold.

Because no internal standards are used in this procedure, volume must be consistent among all samples and standards. Although most oil components are retained on the packing bed by this procedure, small amounts of material pass through the bed with the PCBs. These materials are eluted from the SPB-5 column well away from the PCBs portion of the chromatogram, however, and thus are not an important consideration (Figure B).

Supelco offers an extensive line of PCBs standards. See our current catalog.

Reference

1. Public Law 94-469. U.S. Toxic Substances Control Act, October 11, 1976.

Trademarks

Aroclor — Monsanto Co.
 Florisil — U.S. Silica Co.
 Hall — Tracor Instruments, Austin, Inc.
 Teflon — E.I. du Pont de Nemours & Co., Inc.
 SPB, Supelclean, Sylon, Visiprep, Visi-1 — Sigma-Aldrich Co.

- US Pat. Nos. D289,861; 4,810,471; other patents pending.
- ■ US Pat. No. 4,810,471; other patents pending.

Fused silica columns manufactured under HP US Pat. No. 4,293,415.

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Figure B. PCBs from Used Transformer Oil 58ppm Aroclor 1260 in Transformer Oil

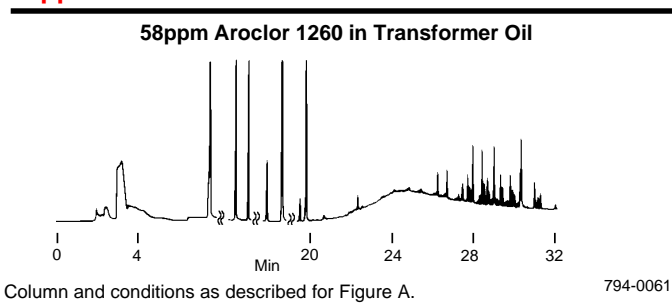
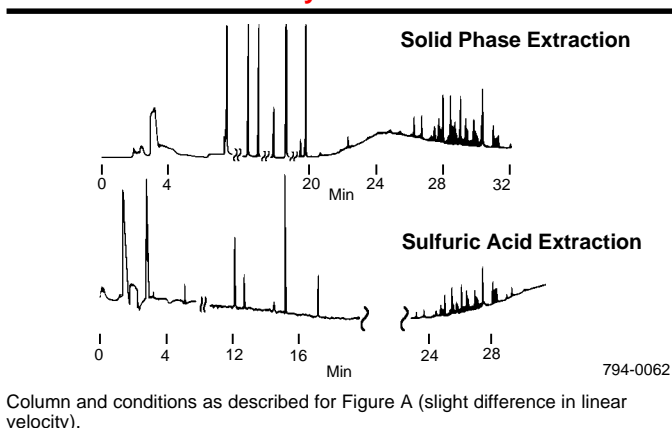


Figure C. Solid Phase Extraction Offers More Efficient PCBs Recovery



Ordering Information:

Description	Cat. No.
Supelclean LC-Florisil SPE Tubes, pk. of 30	57057
Supelclean LC-Si SPE Tubes, pk. of 30	57051
Tube Adapters, pk. of 12	57020-U
Visiprep SPE Vacuum Manifold [■]	57030-U
Visi-1 Single SPE Tube Processor ^{■ ■}	57080-U
SPB-5 Capillary Column 30m x 0.32mm ID, 0.25µm film	24048

Aroclors in PCB-Free Oil (Quality Control Check Samples)

Prepared w/w in 5mL of Diala AX transformer oil, according to US EPA specifications for "quality control check samples." Use these standards to quantify PCBs in transformer fluids and waste oils. Each solution is evaluated by GC, using an EC or Hall[®] detector.

Description	Cat. No.	
	50mg/kg oil Cat. No.	500mg/kg oil Cat. No.
Aroclor 1242	48732	48731
1254	48734	48733
1260	48736	48735

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