

## Monitor Nonvolatile Organic Compounds in Environmental and Other Samples – With Thermal Stripping and Thermal Desorption

A thermal sparging/thermal desorption system allows rapid and efficient monitoring of polychlorinated biphenyls (PCBs) and other nonvolatile organic molecules in a wide variety of sample matrices. PCB concentrations of less than 1ppb were monitored in samples of river sediment, human blood serum, and a transformer oil/water mixture. Desorption efficiency of the adsorbent tubes, and sparging efficiency of the Dynamic Thermal Stripper were evaluated. Average desorption efficiency was  $103 \pm 7.4\%$ . The average sparging/thermal desorption efficiency was  $100 \pm 6.0\%$ .

### Key Words:

- Carbotrap adsorbent tubes ● Dynamic Thermal Stripper
- Thermal Desorption Unit ● organic pollutants ● PCBs

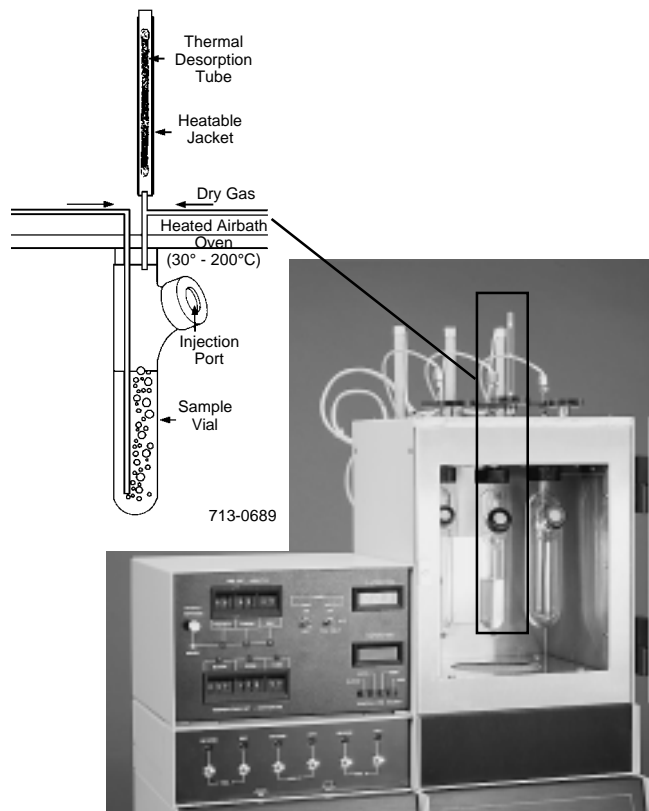
If you monitor large molecular size organic pollutants in foods, environmental samples, biological fluids or tissues, you know that liquid-liquid extractions are tedious, time-consuming, and costly. Recovery can be inefficient, resulting in inaccurate analytical results. Liquid extraction of these pollutants from solid samples (soil, sediment, etc.), blood, or tissue can dilute compounds present in trace quantities to levels that are below detection.

In contrast, a thermal sparging/thermal desorption system composed of a Carbotrap™ 150 adsorbent tube, a Dynamic Thermal Stripper, a Supelco™ Thermal Desorption Unit, and an SPB™-5 capillary gas chromatography column enables you to rapidly and efficiently monitor polychlorinated biphenyls (PCBs) and other nonvolatile (at room temperature) organic molecules in a wide variety of sample matrices.

The Dynamic Thermal Stripper sparges (steam distills) volatile and nonvolatile organic compounds from your sample (mixed with or suspended in water) to the Carbotrap 150 tube. The adsorbent tube is transferred to the Thermal Desorption Unit, which thermally desorbs compounds from the tube and delivers them to the capillary column.

Carbotrap 150 tubes (11.5cm x 6mm OD glass) contain Carbotrap C graphitized carbon black adsorbent (surface area ~ 12m<sup>2</sup>/g) and a small bed of silanized glass beads (to promote a diffuse sample flow through the tube). A Class I, nonspecific adsorbent (1), Carbotrap C adsorbent has no active functional groups to prevent efficient thermal desorption of collected materials. Compared to many other adsorbents, reduced surface-adsorbate interactions enable Carbotrap C adsorbent to selectively trap large size, non-volatile organic molecules, such as PCBs, polynuclear aromatic hydrocarbons, and alkylbenzenes. Potentially interfering smaller molecules migrate through the adsorbent bed. PCBs in sludge samples, for example, are effectively trapped while smaller sulfur compounds are not retained.

Figure A. Dynamic Thermal Stripper Flow Pathways



910-0096

When inserted in the Dynamic Thermal Stripper, a Carbotrap 150 tube traps large organic molecules as they are sparged from the sample (Figure A). The thermal stripper simultaneously accommodates up to three aqueous solutions, suspensions of solid materials, or liquid organic samples, in volumes of 10 to 100mL (larger reservoirs are available for larger sample volumes). Organics in aqueous solutions are recovered through a steam distillation procedure. This technique allows the compounds to be removed from the sample and transported to the adsorbent tube at temperatures much lower than their boiling points (2). Thus, sample breakdown is prevented.

Furthermore, for some samples, steam distillation can provide higher extraction efficiency than alternative liquid-liquid extraction or dynamic headspace techniques. The Dynamic Thermal Stripper features a heated jacket around the adsorbent tube, and plumbing that enables you to add dry gas to the distilled sample.

These features prevent water vapor in the effluent from condensing in the tube (Figure A). This ensures efficient trapping of the organic molecules by the hydrophobic graphitized carbon. Sample-transporting and carrier gas lines are inert (nickel), while oven preheat, purge time, and dry time are individually controlled.

After the appropriate sparging period, the adsorbent tube is transferred to the Thermal Desorption Unit (Figure B). This unit rapidly heats the tube, providing efficient sample desorption and delivery to the GC column. An inert flowpath and a heated transfer line prevent sample loss during the transfer.

**Figure B. Supelco Thermal Desorption Unit Efficiently Transfers Adsorbates from Adsorbent Tube to GC Column**



910-0104

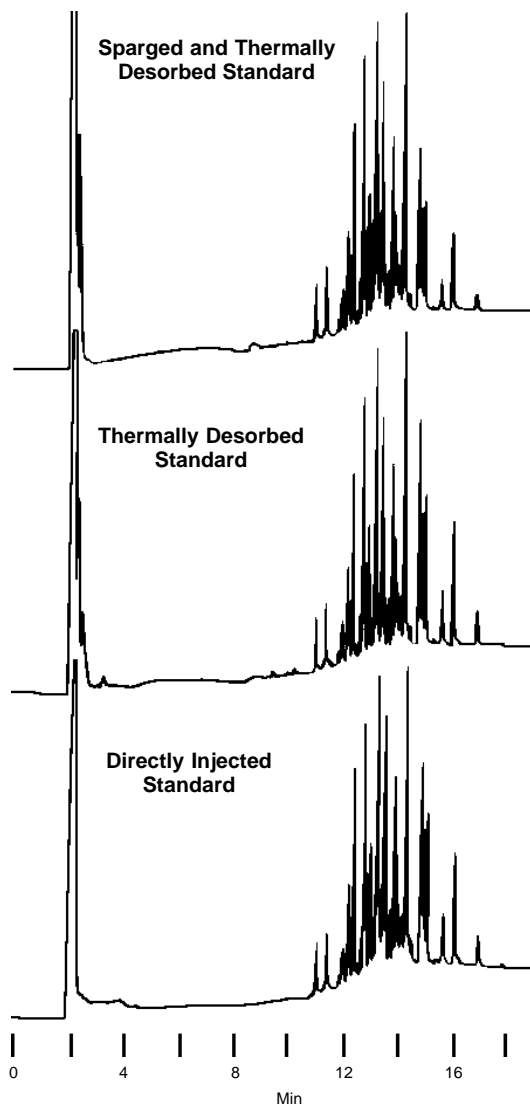
To demonstrate the high efficiency of this system for trapping, desorbing, and analyzing large organic molecules, we simulated analyses for PCBs in a variety of samples (Figures C - E). PCBs in solid samples were simulated by adding Aroclor® 1260 PCB mixture to Hudson River sediment and suspending 30mg of the sediment in 10mL of deionized water. Similarly, PCBs in human blood serum were detected by adding 10µL of PCB-containing serum to 10mL of water. A transformer oil analysis was prepared by mixing 1 mL of PCB-containing transformer oil with 9mL of water. Each 10mL sample contained a total of 6.0ng of Aroclor 1260 PCB mixture – a concentration of less than 1ppb.

For each sample, we inserted a Carbotrap 150 tube in the Dynamic Thermal Stripper and sparged the sample at 110°C for 30 minutes. After transferring the adsorbent tube to the Thermal Desorption Unit, we desorbed the PCBs from the tube to a 0.32mm ID (blood serum) or a 0.75mm ID SPB-5 capillary column.

Figure C shows that chromatograms for sparged and thermally desorbed samples, thermally desorbed standards, and standards injected onto the capillary column are almost identical. We evaluated the desorption efficiency of Carbotrap 150 adsorbent tubes by injecting standards onto a tube and desorbing them onto the column, then comparing peak areas to those for the same standards injected directly onto the column. Average desorption

**Figure C. PCBs Efficiently Recovered from Water**

Sample: 1mL PCB-containing transformer oil/9mL water  
 Desorption Tube: **Carbotrap 150**  
 Cat. No.: **20381**  
 Sample Purge: 110°C (30 min)  
 Thermal Desorption: 350°C (hold 4 min)  
 Adsorb. Tube Heater Temp.: 80°C (40 min)  
 Carrier(desorption tube to column): helium, 100mL/min  
 Column: **SPB-5, 60m x 0.74mm ID, 1.0µm film**  
 Cat. No.: **23721**  
 Oven: 150°C (2 min), then to 290°C at 8°C/min (10 min)  
 Carrier: helium, 10mL/min  
 Det.: ECD



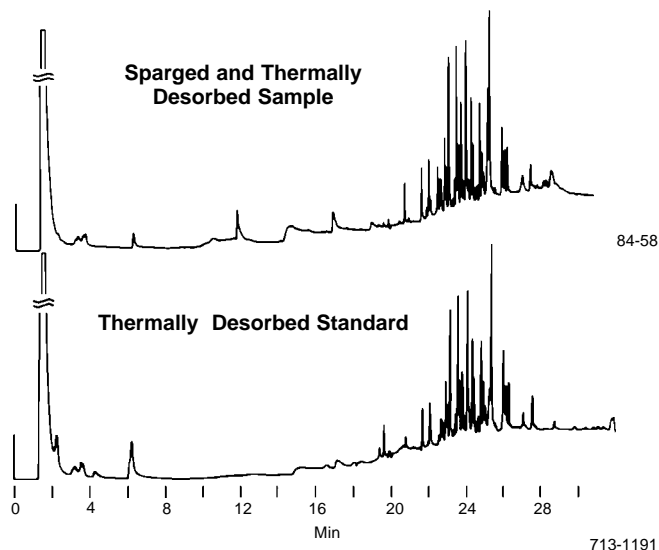
794-0175

efficiency for three trials was  $103 \pm 7.4\%$ . Similarly, to monitor the sparging efficiency of the Dynamic Thermal Stripper for PCBs, we compared peak areas for sparged, trapped, thermally desorbed PCBs with areas for standards injected directly onto the column. The average sparging/thermal desorption efficiency (four trials) was  $100 \pm 6.0\%$ .

Because it is a bonded phase column, the 0.75mm ID SPB-5 capillary column used in most of the PCBs analyses provides several important advantages: inertness, temperature stability, and the high efficiency that is particularly important in PCBs analyses. In

**Figure D PCBs Efficiently Recovered from Blood Serum**

Sample: 6.0ng PCBs/10µL serum in 10mL water  
Desorption Tube: **Carbotrap 150**  
Cat. No.: **20381**  
Sample Purge: 110°C (10 min no sparge gas flow, then 30 min with flow)  
Thermal Desorption: 350°C (hold 4 min)  
Carrier (desorption tube to column): helium, 100mL/min  
Column: **SPB-5, 30m x 0.32mm ID, 1.0µm film**  
Cat. No.: **24049**  
Oven: 40°C (2 min), then to 300°C at 12°C/min (8 min)  
Carrier: helium, 6mL/min (50:50 split)  
Det.: ECD



addition, its sample capacity is almost as large as a packed column's. A 0.75mm ID SPB-5 column can be used in a packed column or a capillary column chromatograph. These columns are compatible with detectors designed for use with packed columns or capillary columns, and are easily interfaced with the thermal desorption unit.

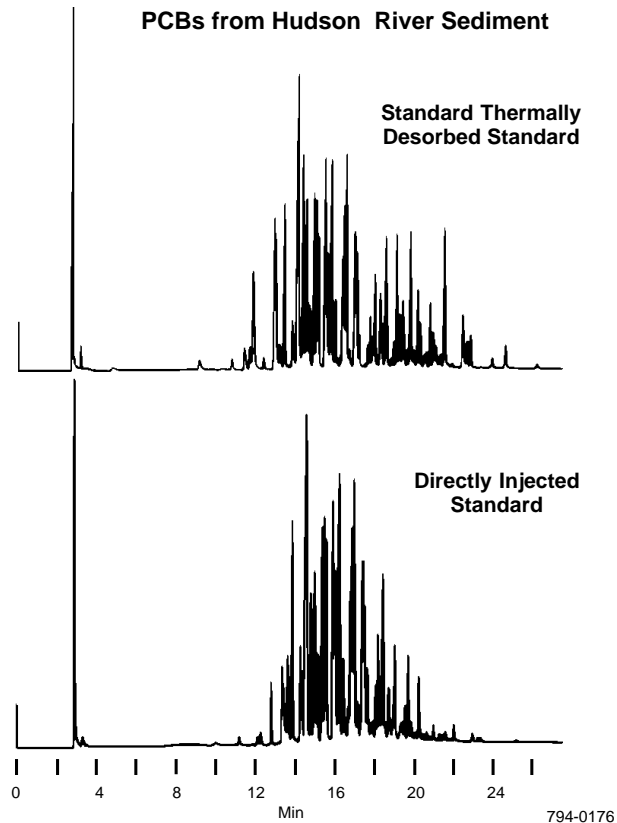
These results show that a system consisting of a Carbotrap 150 adsorbent tube, a Dynamic Thermal Stripper, a Supelco Thermal Desorption Unit, and an SPB-5 capillary column allows you to reliably monitor PCBs—or other large organic molecules—in a wide range of samples.

**References**

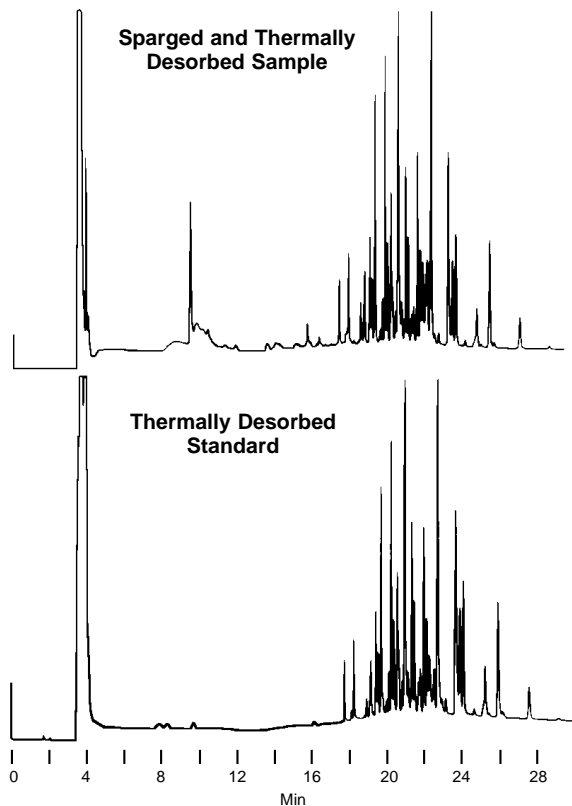
1. Kiselev, A.V. and Y.I. Yashin, Gas Adsorption Chromatography, Plenum Press, New York (1969).
2. Bellar, T.A. and J.J. Lichtenberg, J. Am. Water Works Assoc., 66, 739-744 (1974).

**Figure E. PCBs Efficiently Recovered from Sediment and Transformer Oil, Using Steam Distillation and Thermal Desorption**

Conditions: see Figure C.



**PCBs from Transformer Oil**



## Ordering Information:

### Dynamic Thermal Stripper

Use our dynamic thermal stripper to purge organic compounds from samples and trap them on a thermal desorption tube. Transfer the tube to your Supelco Thermal Desorption Unit for delivery to a GC column.

Purging, trapping, and thermal desorption are highly efficient for PCBs, polynuclear aromatic hydrocarbons, and other water pollutants. You can also use this unit to analyze dry samples (plastics, etc.) by dynamic headspace analysis. The unit will accommodate three samples from 10 to 100mL in size.

110 VAC	22822
220 VAC	22827

### Supelco Thermal Desorption Unit

Use our Supelco Thermal Desorption Unit<sup>†</sup> to deliver airborne organic compounds onto a GC column *without dilution*.

You also save the time and costs involved in solvent desorption. And because a thermal desorption tube can be reused many times, you save on adsorbent tube purchases. The undiluted samples ensure much greater sensitivity.

110 VAC	22819
220 VAC	22829

<sup>†</sup> Manufactured by Dynatherm, Inc.

### Aroclor Solutions Kit

Individual solutions of Aroclor 1016, 1221, 1232, 1242, 1248, 1254, and 1260 in 1mL methanol (200µg/mL). For additional PCB and other pollutant standards, refer to our general catalog.

48825

### Carbotrap 150 Adsorbent Tube

Glass tubes (11.5cm x 6m OD) contain Carbotrap C graphitized carbon black adsorbent.

20381

### SPB-5 Capillary Columns

60m x 0.75mm ID, 1.0µm film	23721
30m x 0.32mm ID, 1.0µm film	24049

Contact our Technical Service Department (phone 800-359-3041 or 814-359-3041, FAX 814-359-5468) for expert answers to your questions.

### Trademarks

Aroclor – Monsanto Corp.  
Carbosieve – Supelco, Inc.  
Carbotrap – Supelco, Inc.  
SPB – Supelco, Inc.  
Supelco – Supelco, Inc.

BULLETIN 861A

For more information, or current prices, contact your nearest Supelco subsidiary listed below. To obtain further contact information, visit our website ([www.sigma-aldrich.com](http://www.sigma-aldrich.com)), see the Supelco catalog, or contact Supelco, Bellefonte, PA 16823-0048 USA.

ARGENTINA · Sigma-Aldrich de Argentina, S.A. · Buenos Aires 1119 AUSTRALIA · Sigma-Aldrich Pty. Ltd. · Castle Hill NSW 2154 AUSTRIA · Sigma-Aldrich Handels GmbH · A-1110 Wien  
BELGIUM · Sigma-Aldrich N.V./S.A. · B-2880 Bornem BRAZIL · Sigma-Aldrich Quimica Brasil Ltda. · 01239-010 São Paulo, SP CANADA · Sigma-Aldrich Canada, Ltd. · 2149 Winston Park Dr., Oakville, ON L6H 6J8  
CZECH REPUBLIC · Sigma-Aldrich s.r.o. · 186 00 Praha 8 DENMARK · Sigma-Aldrich Denmark A/S · DK-2665 Vallensbaek Strand FINLAND · Sigma-Aldrich Finland/YA-Kemia Oy · FIN-00700 Helsinki  
FRANCE · Sigma-Aldrich Chimie · 38297 Saint-Quentin-Fallavier Cedex GERMANY · Sigma-Aldrich Chemie GmbH · D-82041 Deisenhofen GREECE · Sigma-Aldrich (o.m.) Ltd. · Ilioupoli 16346, Athens  
HUNGARY · Sigma-Aldrich Kft. · H-1067 Budapest INDIA · Sigma-Aldrich Co. · Bangalore 560 048 IRELAND · Sigma-Aldrich Ireland Ltd. · Dublin 24 ISRAEL · Sigma Israel Chemicals Ltd. · Rehovot 76100  
ITALY · Sigma-Aldrich s.r.l. · 20151 Milano JAPAN · Sigma-Aldrich Japan K.K. · Chuo-ku, Tokyo 103 KOREA · Sigma-Aldrich Korea · Seoul MALAYSIA · Sigma-Aldrich (M) Sdn. Bhd. · 58200 Kuala Lumpur  
MEXICO · Sigma-Aldrich Química S.A. de C.V. · 50200 Toluca NETHERLANDS · Sigma-Aldrich Chemie BV · 3330 AA Zwijndrecht NORWAY · Sigma-Aldrich Norway · Torshov · N-0401 Oslo  
POLAND · Sigma-Aldrich Sp. z o.o. · 61-663 Poznań PORTUGAL · Sigma-Aldrich Quimica, S.A. · Sintra 2710 RUSSIA · Sigma-Aldrich Russia · Moscow 103062 SINGAPORE · Sigma-Aldrich Pte. Ltd.  
SOUTH AFRICA · Sigma-Aldrich (pty) Ltd. · Jet Park 1459 SPAIN · Sigma-Aldrich Quimica, S.A. · 28100 Alcobendas, Madrid SWEDEN · Sigma-Aldrich Sweden AB · 135 70 Stockholm  
SWITZERLAND · Supelco · CH-9471 Buchs UNITED KINGDOM · Sigma-Aldrich Company Ltd. · Poole, Dorset BH12 4QH  
UNITED STATES · Supelco · Supelco Park · Bellefonte, PA 16823-0048 · Phone 800-247-6628 or 814-359-3441 · Fax 800-447-3044 or 814-359-3044 · email: [supelco@sial.com](mailto:supelco@sial.com)

Supelco is a member of the Sigma-Aldrich family. Supelco products are sold through Sigma-Aldrich, Inc. Sigma-Aldrich warrants that its products conform to the information contained in this and other Sigma-Aldrich publications. Purchaser must determine the suitability of the product for a particular use. Additional terms and conditions may apply. Please see the reverse side of the invoice or packing slip.

H