

Use Thermal Desorption to Monitor C5-C30 Organic Compounds in Polymers

Using a system composed of a Carbotrap 370 thermal desorption tube, a Supelco Thermal Desorption Unit, and an SPB-5 capillary column, analysts can rapidly and reliably monitor C5 to C30 organic compounds offgassed from many polymer materials without cryogenic focusing. Carbotrap 370 tubes efficiently trap – then desorb – C5-C30 polymer fractions. Using this system, typical polymer samples were analyzed over a wide range of controlled temperatures. (ChromFax: 100866)

Key Words:

- adsorbent tubes • Carbotrap tubes • graphitized
- carbon black • organic compounds • polymers
- offgassing, polymer • thermal desorption

To maintain product quality and to evaluate product content for potentially toxic components, polymer manufacturers routinely need information about the composition of their products. By using a system composed of a Carbotrap™ 370 thermal desorption tube, a Supelco™ Thermal Desorption Unit, and a 30m x 0.32mm ID (1.0µm film) SPB™-5 capillary column, analysts can quickly and reliably obtain profiles (fingerprints) of C5-C30 components offgassed from polymer products at specific temperatures.

Monitoring this wide range of compounds requires an adsorbent tube that will efficiently trap – then desorb – all of the offgassed materials. Carbotrap 370 tubes contain equal bed lengths of

Carbopack™ F, Carbopack C, and Carbopack B graphitized carbon black adsorbents (surface areas 5, 10, and 100m²/gram respectively) in an 11.5cm x 2mm ID glass tube. These adsorbent tubes will efficiently trap C5-C30 polymer fractions. As thermally desorbed material from the polymer passes through the tube, larger molecules are adsorbed by the adsorbents with the smaller surface areas (Carbopack F and Carbopack C). Smaller molecules not trapped by these first two beds are adsorbed by the third bed – the Carbopack B adsorbent.

Several features of the Supelco Thermal Desorption Unit are also critical to the success of the procedure. The ballistic heating capability of the unit is essential. Temperature in the unit can be rapidly and accurately raised. For example:

30°C → 100 ± 1°C in 12 ± 2 seconds

30°C → 200 ± 1°C in 16 ± 2 seconds

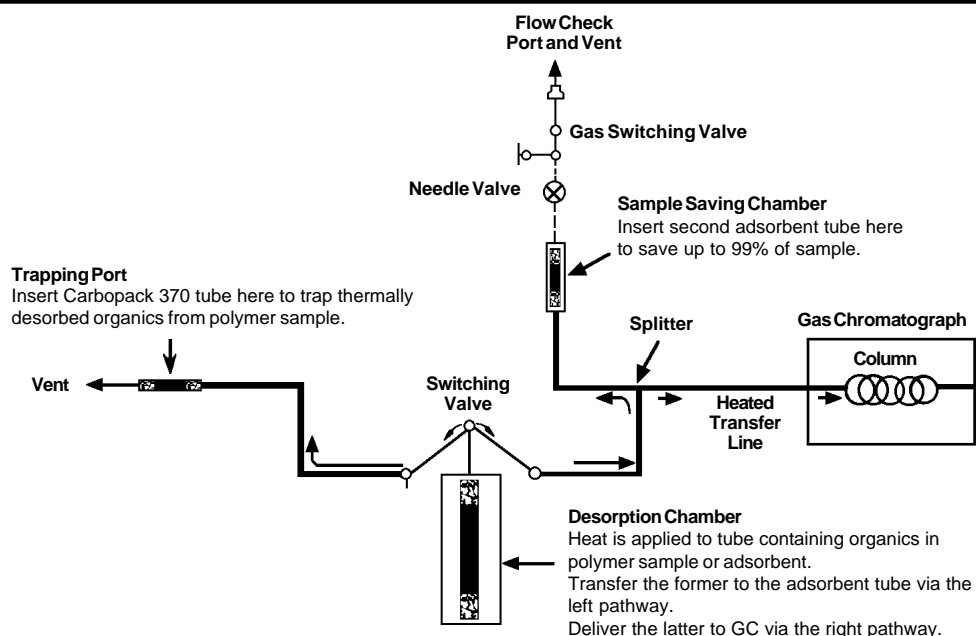
30°C → 300 ± 1°C in 20 ± 2 seconds

30°C → 400 ± 4°C in 26 ± 2 seconds

Because the temperature at which a polymer is characterized can be precisely duplicated from one sample to the next, reproducibility is ensured.

To obtain a fingerprint of a polymer sample, insert a Carbotrap 370 tube into the secondary trapping chamber of the thermal desorption unit (Figure A), and place a known amount of the polymer in an empty 4mm ID glass tube. Insert the latter tube into the sample desorption chamber, set the switching valve to divert the sample to the Carbotrap 370 tube, and quickly raise the temperature to the

Figure A. Thermal Desorption Unit Flow Pathways



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set level. Offgassed components are carried from the desorption chamber to the Carbotrap 370 tube through the *left* flow pathway in Figure A. Because of the nature of the adsorbents in the Carbotrap 370 tube, offgassed components are trapped at room temperature (there is no need for cryotrapping). Once trapped, they do not migrate from the adsorbents.

When offgassed compounds are trapped, remove the 4mm ID tube from the sample desorption chamber and insert the Carbotrap 370 tube (now containing the trapped compounds) into the desorption chamber. Activate the switching valve and the compounds will be thermally desorbed to the capillary GC column via the *right* flow pathway in Figure A.

Because the sample has been focused onto a narrow (2mm ID) adsorbent tube, and the desorption temperature is rapidly attained and accurately controlled, the sample is well focused at the column inlet – again without need for cryofocusing. This ensures sharp chromatographic peaks and, therefore, maximum resolution. Because alternative thermal desorption units provide slower temperature rises and require larger gas flow volumes, samples must be cryofocused at the GC column inlet.

C5-C30 fraction profiles, for typical polymer samples analyzed using this system, are shown in Figures B-E. For comparison, consistent analytical conditions were used to obtain these chromatograms (Table 1). All of these parameters are easily set on the Supelco Thermal Desorption Unit. Just dial the desired value.

Figure B shows a series of fingerprints for a sample of polyethylene beads. These fingerprints were made as the volatile offgas components were desorbed to the Carbotrap 370 tube at successively higher sample chamber temperatures. The effect of temperature on offgas composition is apparent.

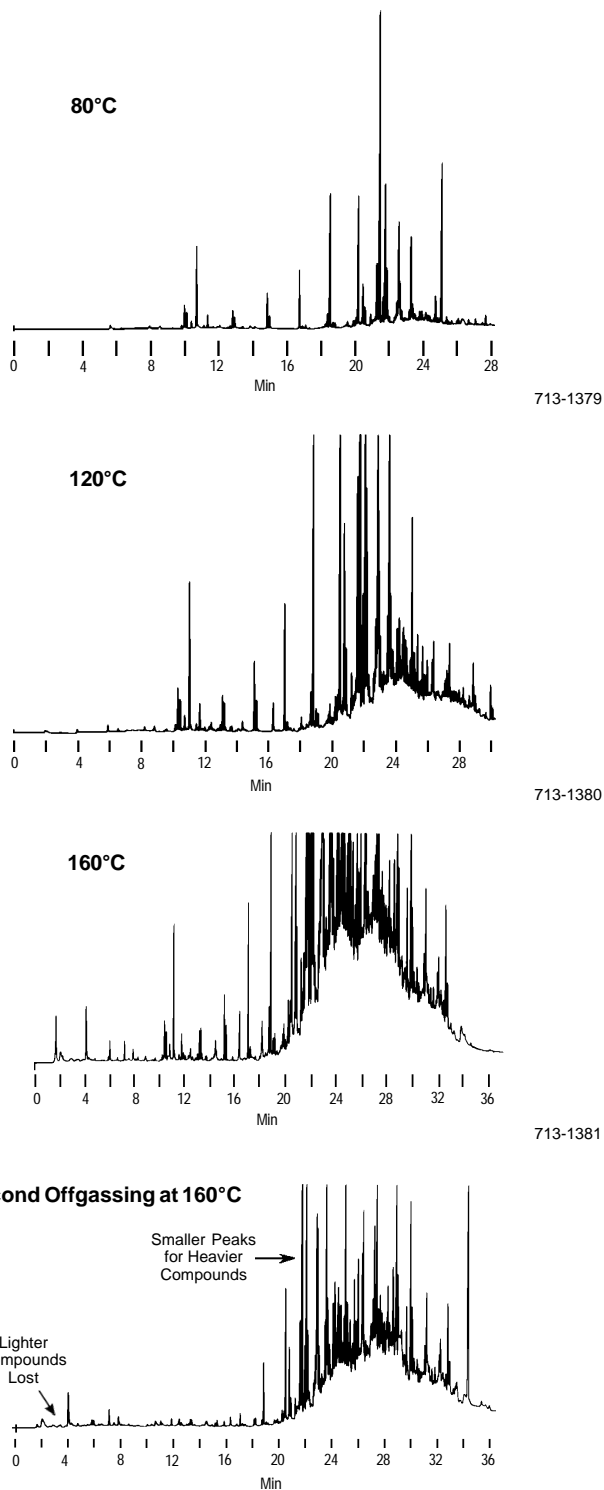
To obtain the fourth chromatogram in Figure B, the sample heated to 160°C was *reheated* to the same temperature. Lighter compounds were almost completely offgassed in the first analysis. Peaks for the heavier compounds are noticeably smaller. This shows that, by adjusting sample chamber temperature, ranges of sample components can be completely offgassed from a polymer sample. Because temperatures and other conditions can be consistently reproduced, it is valid to compare fingerprints obtained under the same conditions.

Table 1. Analytical Conditions

Samples	
polyethylene beads, styrene butadiene, polysulfone, granular polypropylene homopolymer	
Thermal Desorption	
Sampling Tube:	Carbotrap 370 (conditioned at 330°C for 20 min before use)
Cat. No.:	2-0373
Sample Purge:	8mL/min, helium, 15 min (polymer sample to Carbotrap 370 tube)
Sample Desorption:	helium, 6mL/min (5 min), 330°C (sample split 1:1 in thermal desorption unit)
Valve Compartment Temp.:	235°C
Transfer Line Temp. (desorption unit to GC column):	230°C
Chromatography	
Column:	SPB-5, 30m x 0.32mm ID, 1.0µm phase film
Oven:	40°C (2 min), then to 280°C at 8°C/min (0-8 min)
Det.:	FID

Using a system composed of a Carbotrap 370 thermal desorption tube, a Supelco Thermal Desorption Unit, and an SPB-5 capillary column, you can rapidly and reliably monitor C5 to C30 organic compounds offgassed from many polymer materials – over a wide range of controlled temperatures. Because a wide range of analytical conditions can be established, then reproduced consistently, this system will prove invaluable in evaluating and ensuring consistent product quality.

Figure B. Effect of Sample Chamber Temperature on Offgas from a Sample of Polyethylene Beads



Conditions: see Table 1.

Figure C. Styrene Butadiene (100°C)

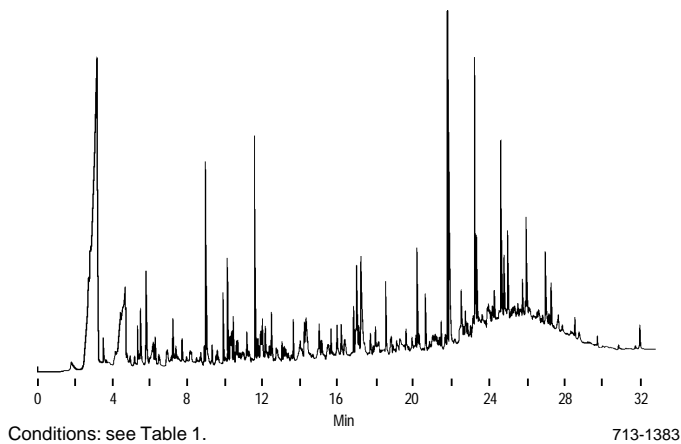


Figure D. Polysulfone (180°C)

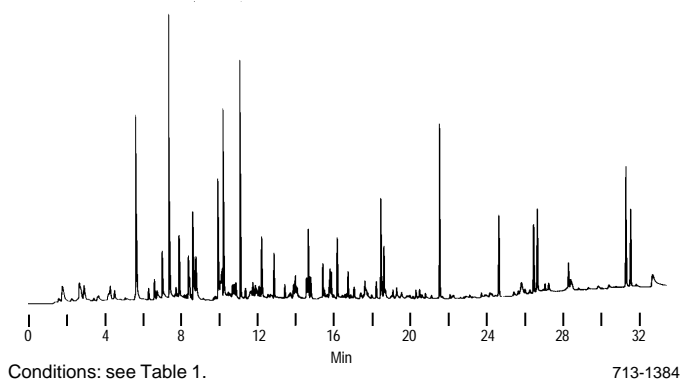
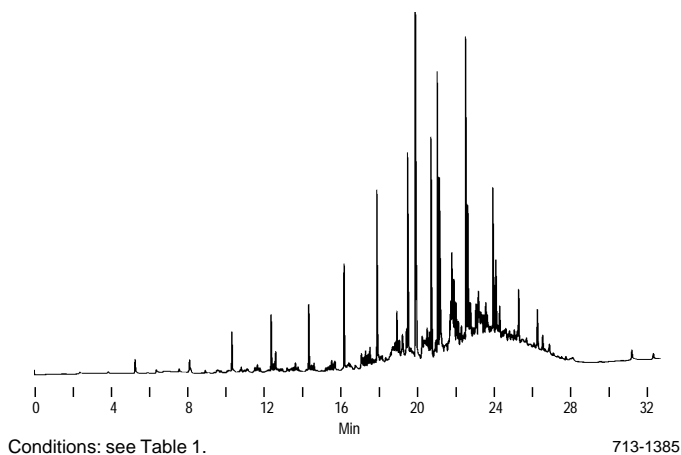


Figure E. Granular Polypropylene Homopolymer (80°C)



Thermal Desorption Saves Time and Solvent

Thermal Desorption Unit

Use the Supelco Thermal Desorption Unit 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.

Carbotrap Adsorbents

Carbotrap graphitized carbon blacks are ideal adsorbents for trapping a wide range of organic compounds. For example, you can efficiently adsorb and thermally release all hydrocarbons listed in US EPA Methods TO-1, TO-2, and TO-3 – individually or in mixtures – on a Carbotrap 300 thermal desorption tube.

Beds of Carbotrap C, Carbotrap, and Carbosieve™ S-III* respectively trap the heaviest compounds, the C5-C8 compounds, and the lightest compounds. The alternative to using a Carbotrap 300 tube for these three methods is to use *two* less versatile adsorbent traps – *plus* a cold trap.

Other reusable Carbotrap thermal desorption tubes are described in our general catalog.

*German Pat. No. 1935500. Patent holder – Badische Anilin- & Soda-Fabrik Aktiengesellschaft.

Ordering Information:

Supelco Thermal Desorption Unit*

110 VAC	22819
220 VAC	22829

Carbotrap 370 Thermal Desorption Tube 20373

Empty Thermal Desorption Tube

11.5cm x 4mm ID	20235-U
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SPB-5 Fused Silica Capillary Column

30m x 0.32mm ID, 1.0µm film	24049
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Desorption Tube Conditioner** (110 VAC)

For single tube	22831
For 1 to 6 tubes	22833

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

*Manufactured by Dynatherm, Inc. Marketed exclusively by Supelco, Inc.

**The Desorption Tube Conditioner enables you to prepare up to six tubes simultaneously for this or other thermal desorption analysis.

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

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