

Desorption of PCBs from Soil Using the HTD

The presence of pollutants such as polychlorinated biphenyls (PCBs) in soil is a serious environmental problem. Frequently it is desirable to obtain a rapid measurement of the level of contaminants present in order to discern the sample size required for more elaborate testing. For these screening tests, a rapid and reliable method of removing and analyzing the volatiles and semivolatiles present in soil is required. The use of thermal desorption techniques, in which the sample material is desorbed directly to a gas chromatograph, simplifies the testing procedure by eliminating both solvent extraction and sample sparging.

To desorb the volatiles and semivolatiles from soil quantifiably, the thermal desorption system used must rapidly heat the entire soil sample in a controlled manner, so that no portion overheats, which could result in breakdown products, and no cold spots remain, which would make the analysis unreliable. The **CDS Analytical High Temperature Desorber (HTD)** is controlled by the CDS Analytical Pyroprobe temperature controller using the same resistive heating system as the Pyroprobe. Sample sizes up to 200 mg can be accurately heated to any desired temperature up to 1400 C. Using the Pyroprobe 2000 control system, the temperature can be increased in multiple steps.

Samples for the HTD are placed into a quartz tube 25 mm in length and 6 mm inside diameter. This tube is then inserted into the large platinum heating coil, which is controlled by the Pyroprobe. The sample coil is

Figure 1

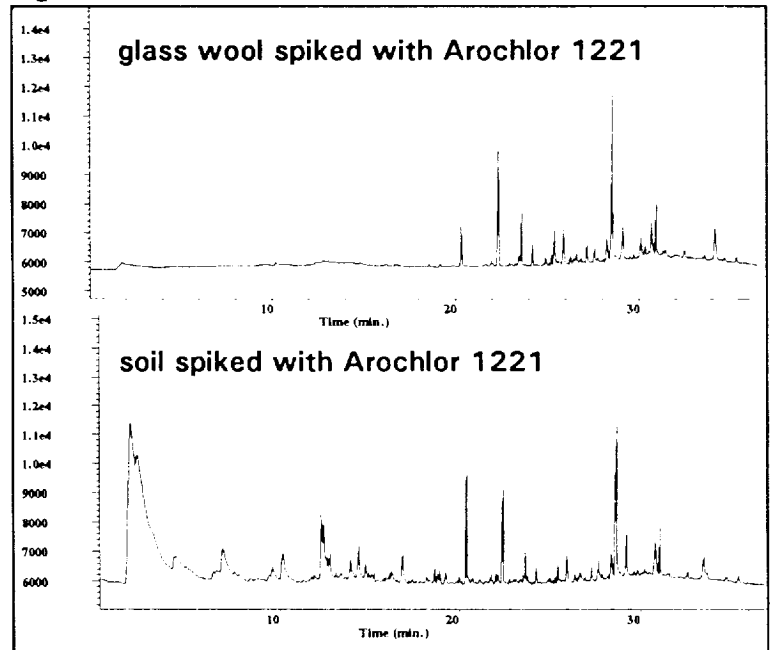
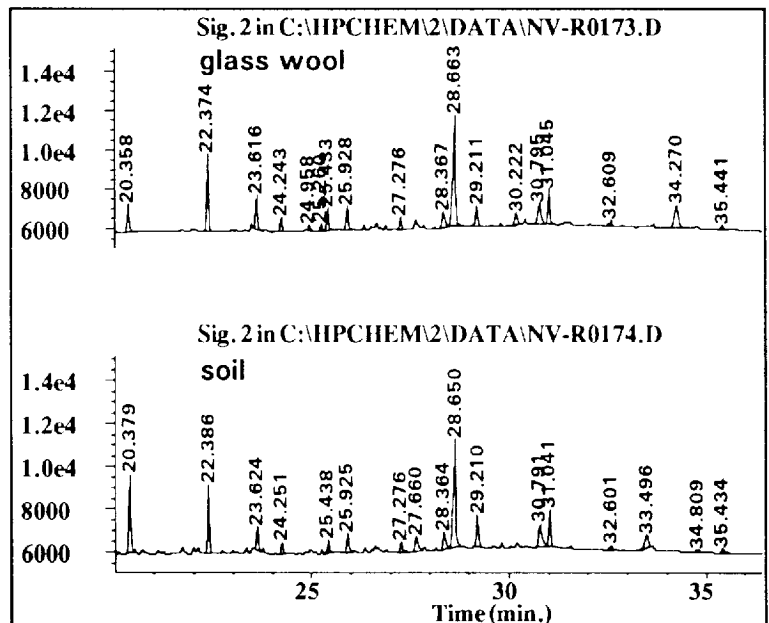


Figure 2



placed into the GC carrier gas stream in a heated interface, then rapidly heated to desorb the volatile and semivolatile contaminants. Starting the analysis with a temperature burst to 1000 C for a few seconds facilitates heating the interior of the soil sample.

In these experiments, a solution of Arochlor 1221 in methanol was placed onto soil samples and allowed to sit overnight to evaporate the solvent. The samples were desorbed in the HTD using the conditions given below.

Figure 1 compares the GC/FID chromatogram of the desorption of Arochlor 1221 from an inert substrate (glass wool) with the same sample from soil. The glass wool or 30 mg soil was spiked with Arochlor at 27 ppm. The other substances present in the chromatogram of the sample desorbed from soil are organic materials found in the soil.

Because of the levels of other contaminants present in soil samples, the peak assignments can be questionable. Figure 2 compares the section of the two chromatograms found in Figure 1 that encompasses the retention times of Arochlor 1221. The perfect match of times for most of the peaks indicates that this section of the chromatogram from soil is indeed caused by Arochlor. A comparison of the area counts of these peaks shows that 98% of the Arochlor was desorbed from the soil under these conditions.

Experimental Conditions

Desorption

Instrument: CDS Analytical HTD

Program:

	Interface	Probe
Start	150 C	0
Step 1	300 C at 60 C/min hold 10 min	1000 C at 20 C/mSec hold 3 sec
Step 2		450 C at 20 C/sec hold 3 min

Gas Chromatography

Instrument: HP 5890 GC
Column: 30 m x 0.53 mm SE-54
Carrier: Helium at 10 ml/min
Program: 50 C, 4 min; 10 C/min
to 280 C.
Detector: FID

Analyte Recovery from Soil: 98%

Additional literature on this and related topics may be obtained by contacting your local CDS Analytical representative, or directly from CDS at the address below.

ABOUT CDS

CDS Analytical, Inc. is a leader in the design and manufacture of laboratory instruments for sample preparation and analysis. With 25 years experience in the field, CDS is dedicated to providing the best possible instruments for both research and routine analysis. Well known in the field of analytical pyrolysis, CDS manufactures the Pyroprobe 1000 and 2000 for the introduction and analysis of solid materials by GC, MS and FT-IR. CDS offers a complete line of purge and trap instruments for the analysis of volatile organic compounds in the environmental, food and pharmaceutical areas, as well as custom systems for complex, multicomponent materials investigation. Our customers, their requirements and applications are important to us. To help meet their needs, we offer a wide range of analytical information and the services of our applications laboratory. If you would like additional information, please contact us at the address below, or call us at 1 800 541 6593.