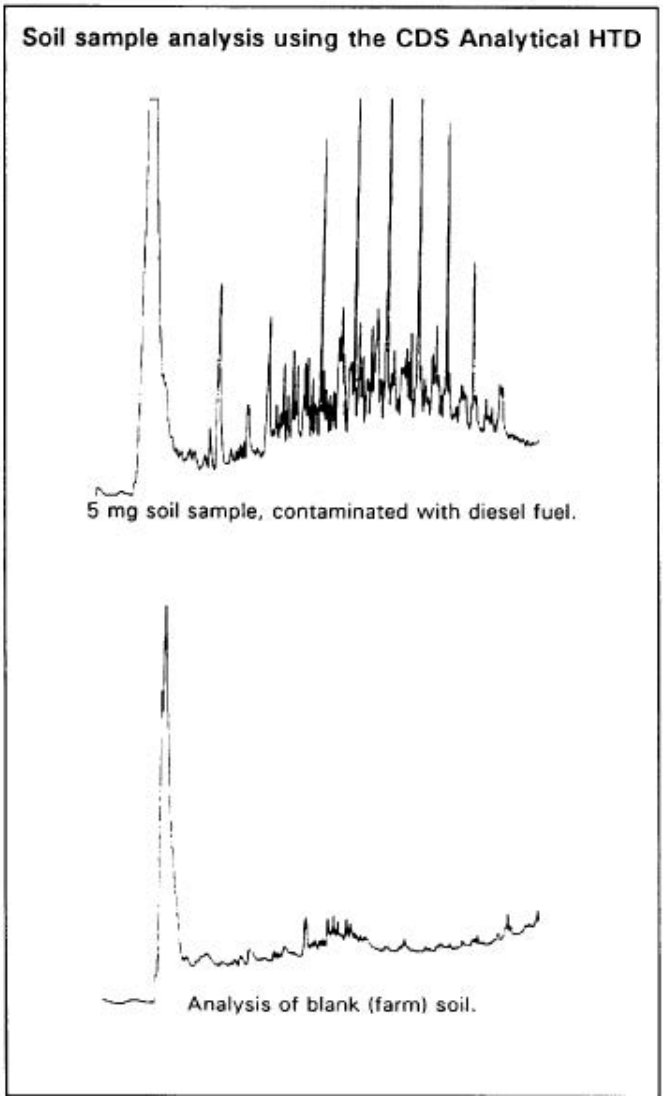
#037

Analysis of Soil Samles using the HTD

The analysis of complex environmental materials such as soils and sludges for semivolatile pollutants may be simplified using rapid thermal desorption techniques, in which the sample material is desorbed directly to the gas chromatograph. This process eliminates solvent extraction by volatilizing the organics of interest rapidly from the sample matrix and transferring them immediately to the injection port of the GC without trapping or dilution.

To achieve such rapid analysis, it is important that the instrument be capable of heating samples which weigh from 5 to 200 mg in a rapid, yet controlled manner. If the samples are heated too slowly, an additional collection or trapping step will be required; if heated too rapidly, or to an excessive temperature, unwanted byproducts may be generated. The CDS High Temperature Desorber (HTD) is controlled by the CDS Analytical Pyroprobe temperature controller for exacting supervision of heating rate and temperature. Using the Pyroprobe 2000, multiple step controls may be programmed for even more versatile temperature management.

Figure 1.



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 a large platinum heating coil, which is controlled by the Pyroprobe. The sample coil is placed into the GC carrier gas stream in a heated interface, then rapidly heated to desorb the volatile and semivolatile organic contaminants.

Figure 1 compares soil contaminated with diesel fuel to a run of blank soil. In each case, a 5 mg sample was placed into the quartz tube and held in place with glass wool. The HTD coil was programmed to heat the sample to 200°C for 50 seconds, with an initial 3 second burst to 1000° to shorten the heating time. The gas chromatograph run was initiated immediately since no collection step is required, so the entire analysis takes only as long as the GC run itself.

Equipment:

DESORPTION

CDS HTD platinum coil, programmed to 1000°C for 3 seconds, then 200° for 50 seconds. Interface temperature: 100°C isothermal.

CHROMATOGRAPHY

30 m x 0.53 mm SE-54 megabore capillary column operated at 7 ml/min. helium flow. Program: Initial temperature 40°C for 2 minutes, then 6°/min. to 225°C. Detection: Flame Ionization Detector.

FOR MORE INFORMATION CONCERNING THIS AND RELATED APPLICATIONS, WE RECOMMEND THE FOLLOWING READING:

Systems approach to automated cryofocusing in purge and trap, headspace and pyrolytic analysis, T.P. Wampler, W. Bowe, J. Higgins, American Laboratory, 17, 8 (1985) 82-87.

A dedicated purge and trap system for environmental analysis, J.W. Washall, T. Wampler, W. Bowe, K. Kristunas, CDS Application paper #155.

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