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CDS Solutions

APPLICATIONS INFORMATION USING ADVANCED GC SAMPLE HANDLING TECHNOLOGY

Dynamic Headspace and Pyrolysis in Ink Analysis

Printing ink is a complex formulation of pigment, petroleum resins, waxes and oils. Once produced, it may be difficult to analyze since it is a combination of solid and volatile or semivolatile materials. Pyrolysis GC may be useful in the analysis of the nonvolatile constituents, but the presence of volatile materials may interfere. In cases such as this, a two-step process may simplify the situation.

Figure 1 shows a pyrogram of a finished ink product with no pretreatment. The presence of a large, unresolved mass of peaks from 20 to 26 minutes makes interpretation difficult. To simplify the chromatogram, the sample was first heated to 250°C for 5 minutes in the Pyroprobe interface. This process removed the volatile hydrocarbon oil which made up a large part of the ink, leaving behind the solid resins and waxes. The volatilized hydrocarbon oil was then chromatographed, producing Figure 2.

The remaining solids were then pyrolyzed at 700°C for ten seconds, and the resulting pyrolysate chromatographed separately, as seen in Figure 3. Now there is nearly baseline resolution of the major peaks, and specific peaks may be identified and associated with individual solid constituents of the ink formulation. Many of the peaks eluting between 12 and 19 minutes, for

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Figure 1

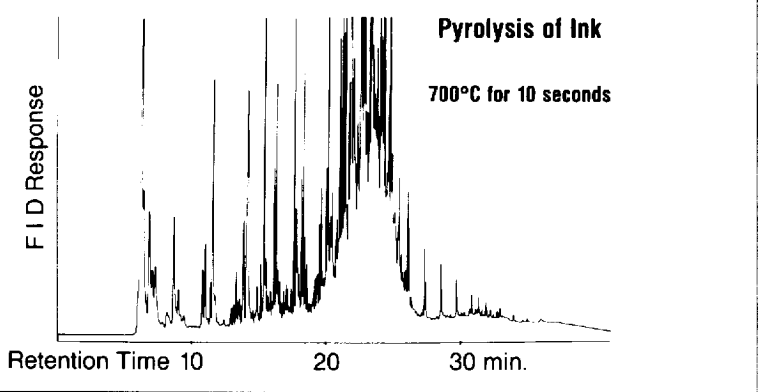


Figure 2

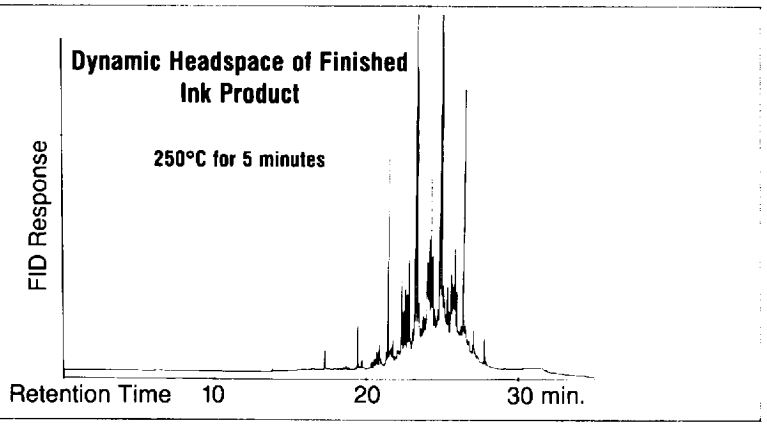
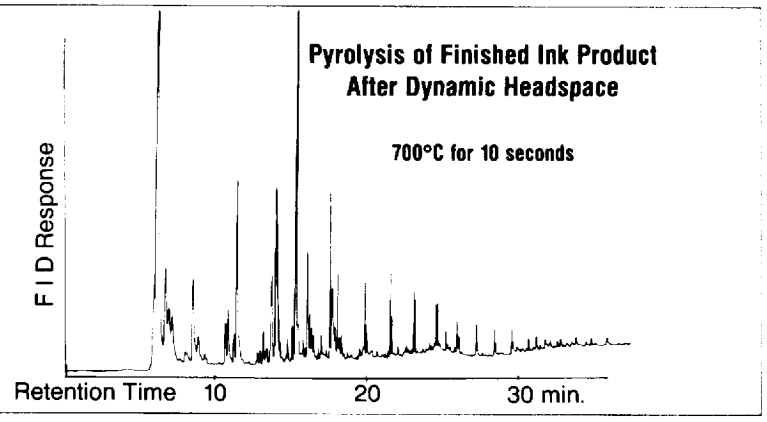


Figure 3



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example, resulted from pyrolysis of the hydrocarbon resins, while the peaks eluting from 20 to 25 minutes are normal alkanes and alkenes produced from the wax.

EQUIPMENT

PYROLYSIS
CDS Pyroprobe 120 with Platinum ribbon probe
Headspace: 250°C for 5 minutes
Pyrolysis: 700°C for 10 seconds

CHROMATOGRAPHY
Column: 50m x 0.25mm SE-54 fused silica capillary
Carrier: Helium, split 70:1
Program: 50°C for 2 min, then 10°/min to 290°
Detector: FID

For more information on this and related applications, we recommend the following readings:

Wampler, T., and Levy, E., "Pyrolysis GC in the Analysis of Ink and Paper," *LC GC*, 4, 11, (1986) 1112-1116

Levy, E., and Wampler, T., "Applications of Pyrolysis Gas Chromnatography/Mass Spectrometry to Toner Material from Photocopiers," *J. Forensic Sci.*, 31, 1, (1986) 258-271.

Wampler, T., and Levy, E., "Analytical Pyrolysis in the Forensic Science Laboratory," *Crime Lab Digest*, 12, 2, (1985) 25-28.

Additional literature may be obtained from CDS by calling 1-800-541-6593 or in Pennsylvania 215-932-3636.

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