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APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Antioxidant Detection in Petroleum Wax

Wax is the name given to a broad range of either natural or synthetic products, used in a myriad of applications.They include use as a fuel, lubrication, mold release, coating and lamination in flexible packaging, and moisture proofing in fiber and chipboard. Waxes can further be categorized as natural, synthetic, mineral hydrocarbon or petroleum waxes. Although there are four main types of petroleum waxes, this application note deals with paraffin, a deoiled slack wax (a wax obtained from dewaxing the base distillate of a lube oil stream).

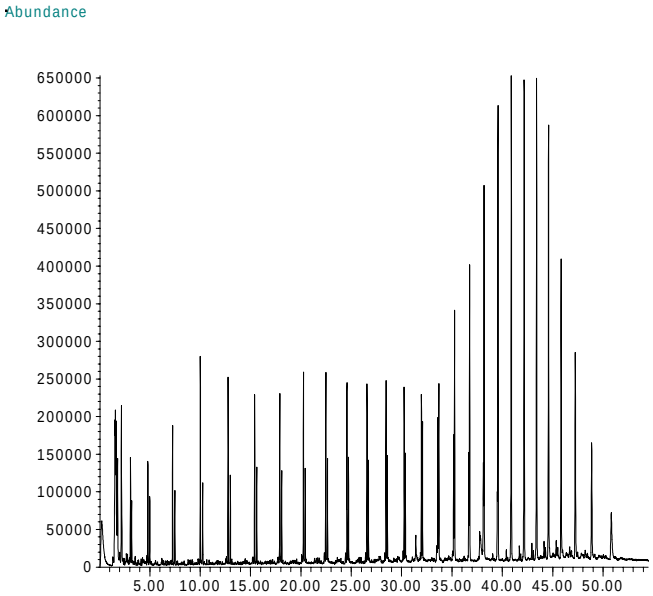

Figure 1. Petroleum Wax Chromatogram (TIC)

The sample material is a food grade paraffin that contains butylated hydroxytoluene (BHT). The BHT functions as an antioxidant and is used to retard oxidative degradation of fats and oils. This particular paraffin is used in coating fiberboard boxes containing foods such as grains and dried cereals. The levels of BHT in the paraffin are in the 10 ppm range.

Conventional analysis employs the use of solvents, but the BHT analysis shown here uses the thermal treatment of pyrolysis to volatilize the analytes and introduce them to the GC column without solvents. The advantage of this technique is an increase in sensitivity in addition to simplified sample preparation.

Figure 1 is a chromatogram of the paraffin containing 10 ppm of BHT. This was run using the mass spectrometer in the scan mode, and the presence of BHT was not discerned in the total ion chromatogram. Figure 2 shows the same paraffin run in the single ion mode (SIM) for the BHT

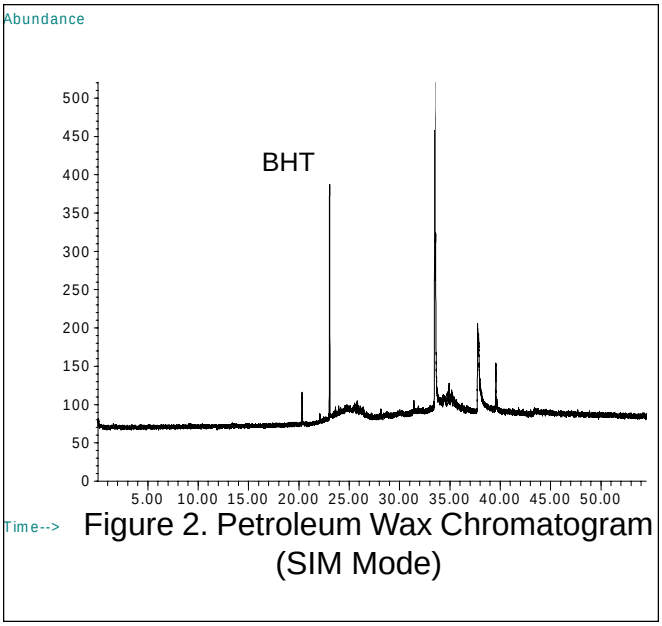

Figure 2. Petroleum Wax Chromatogram (SIM Mode)

molecular ion 220, showing that the compound is easily detected using SIM mode mass spectrometry.

Pyrolysis-GC/MS is clearly a powerful analytical system using minimal preparation time and the absence of solvent work up.

Equipment

The sample was analyzed using a CDS Analytical Pyroprobe 2500 Autosampler, interfaced to an Agilent Instruments gas chromatograph/mass spectrometer.

Pyrolysis

Pyrolyzer:	CDS 2500 Autosampler
Interface Temperature:	300°C
Temperature:	750°C
Time:	15 sec
Sample Probe:	Coil, Quartz Tube
Sample Amount:	~75mcg

Chromatography

Gas Chromatograph:	HP6890
Column:	HP5M, 30 m x.250 µm x .25 µm
Initial Temperature:	40°C for 2 minutes
Ramp:	6°C/minute
Final Temperature:	295°C for 10 minutes
Detector:	HP5972A MSD
Carrier	He, Split 25:1

FOR MORE INFORMATION CONCERNING THIS APPLICATION, WE RECOMMEND THE FOLLOWING READING:

W. J. Irwin, Analytical Pyrolysis: A Comprehensive Guide, Marcel Dekker, Publisher, 1981.

T. P. Wampler, *Introduction to Pyrolysis-Capillary Gas Chromatography,* Journal of Chromatography A, 842 (1999) 207.

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

CDS Analytical, Inc.

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CDS Analytical, Inc. 465 Limestone Road, P.O. Box 277, Oxford, PA USA 19363-0277 PH: 610 932 3636 FX: 610 932 4158