Antioxidant Detection in Petroleum Wax

Application Note
General Interest

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Abstract
This application note demonstrates detection of BHT in wax

Wax is the name given to a broad range of either natural or synthetic products, used in a myriadof applications. They include use as a fuel, lubrication, mold release, coating and lamination inflexible 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).

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 (75µg) 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 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.

Experimental Parameters

Pyroprobe
Setpoints:

Pyrolysis:	750°C 15s
Interface:	300°C
Transfer Line:	300°C
Valve Oven:	300°C

GC-MS

Column:	30meter, 5%phenyl, 0.25µm film thickness, 0.25mm ID
Inlet:	300°C
Carrier:	Helium 1.00mL/min, 25:1 split
Initial:	40°C for 2 minutes
Ramp:	6°C/minute
Final:	295°C for 10 minutes

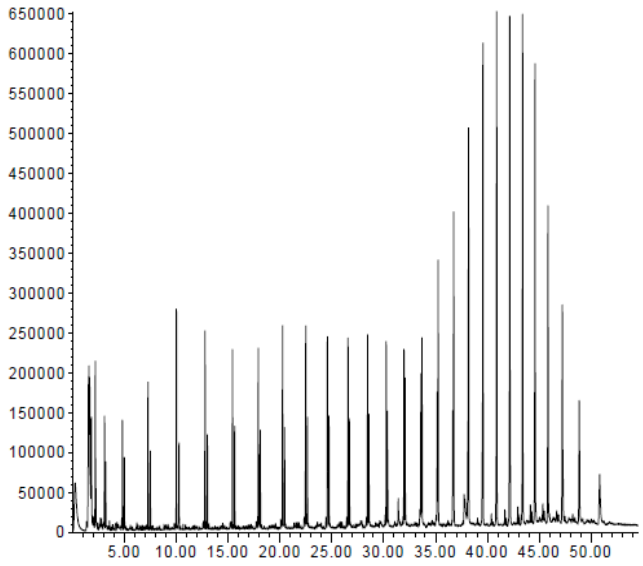

Figure 1: Petroleum Wax Chromatogram Total Ion Chromatogram (TIC).

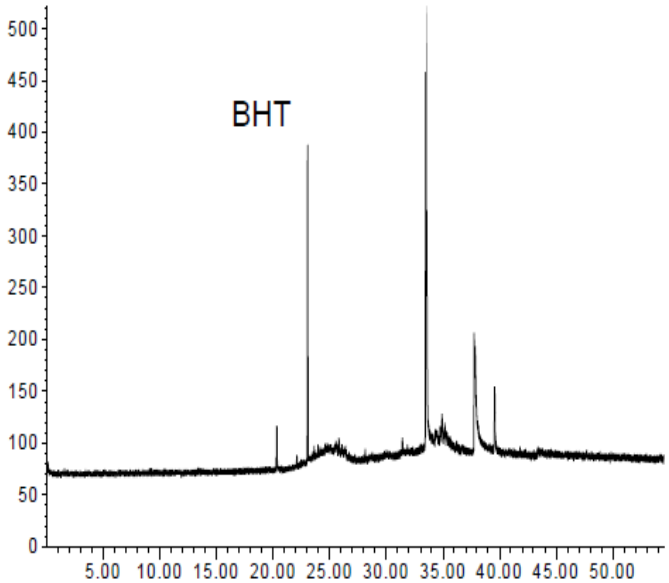

Figure 2: Petroleum Wax Chromatogram (Selected Ion Monitoring (SIM), m/z 220.

Additional 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.