

Thermal Sampling of Lubricants

Engine lubricants are prepared using a base oil (either natural or synthetic) with which a wide variety of additives can be mixed. Additive selectivity is dependent upon the specific properties that are desired from the lubricant. For example, the neutralization of combustion acid residues from fuels employs additives that are naturally alkaline, such as salicylates. Compound such as certain phenols, aromatic amines, and thiophosphates are excellent antiwear additives. Deposits and varnishes that accumulate on engine parts are removed by the detergents in the oil formulation. These detergents include calcium or magnesium salts of alkylphenates or alkylsalicylates.

Thermal sampling techniques permit the introduction of materials like lubricating oils without the use of solvents. The sample may be thermally treated at either a high or low temperature with the products analyzed by GC or GC/MS. At lower temperatures, intact molecules are volatilized, while at higher temperatures pyrolysis takes place - a thermal technique that uses a controlled temperature to produce bond cleavage. Here, thermal sampling is used to identify additives in selected lubricants.

The chromatogram in Figure 1 shows a detergent motor oil containing a hindered phenol. This additive acts as an antioxidant and alkaline detergent on engine parts.

Figure 2 is a chromatogram of a completely synthetic machine cutting oil. The methyl salicylate is added as a detergent.

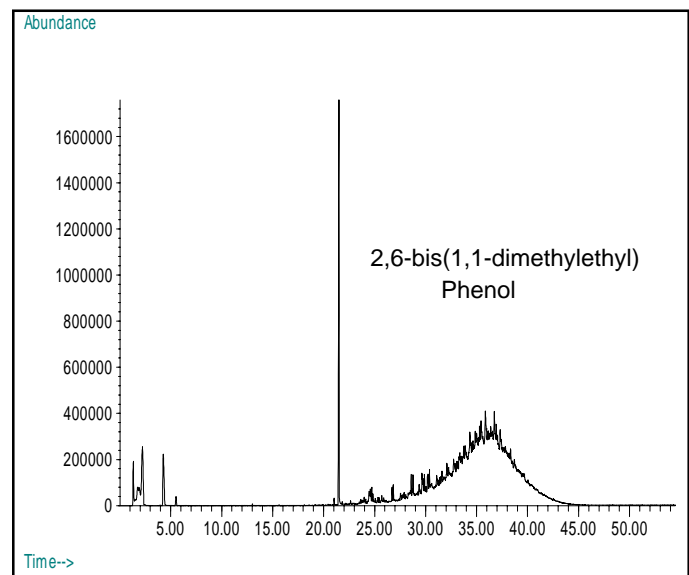


Figure 1. Antioxidant and Detergent

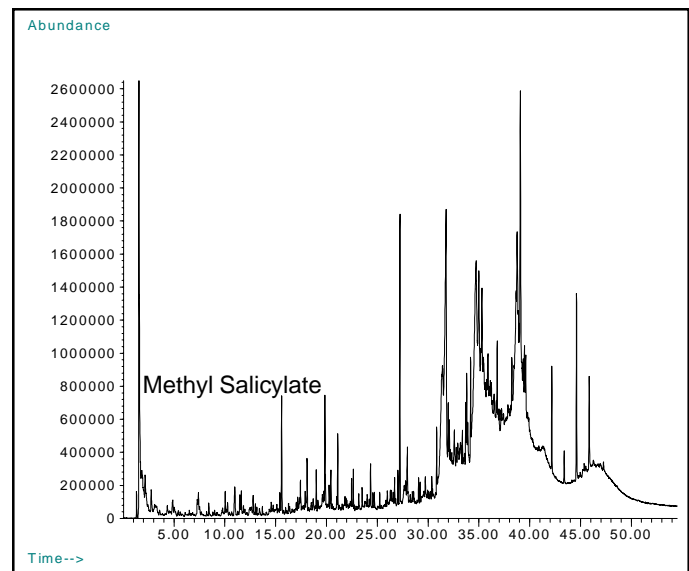


Figure 2. Detergent Ester

Equipment

All samples were pyrolysed using the CDS 2500 Pyrolysis Autosampler interfaced To a Hewlett-Packard 6890 Gas chromatograph with a Hewlett-Packard 5972A mass selective detector.

CDS 2500 Autosampler Conditions

Interface Temperature: 300°C
Pyrolysis Temperature: 600°C
Time: 15 sec
Clean Temperature: 1000°C for 10 sec
Sample Amount:: 25-50 µg

Gas Chromatograph Conditions:

Carrier: Helium
Column: HP-5M
30 m x 0.25 mm
Split: 50:1
Initial Temperature: 40°C for 2 min
Ramp: 6°C/min
Final Temperature: 295°C for 10 min

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

T. Wampler, Analytical Pyrolysis: An Overview, in *Applied Pyrolysis Handbook*, T. P. Wampler (Editor), Marcel Dekker, New York, 1995

A. G. Borrego et al., Chromatographic Study of Spanish Shale Oils, *JAAP*, 58-59, 285 (2001).

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.



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