

Thermal Sampling of Lubricants

Engine lubricants are prepared using a base oil (either natural or synthetic) to 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 classes 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.

Pyrolysis is a thermal technique that uses a controlled temperature to produce bond cleavage. The sample may be thermally treated at either a high or low temperature with the products analysed by GC or GC/MS. Here, the technique is used to identify additives in selected lubricant samples.

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.

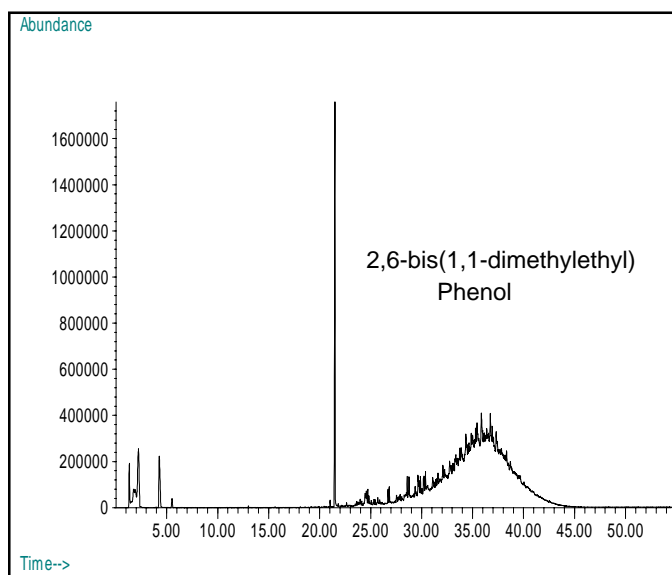


Figure 1. Antioxidant and Detergent

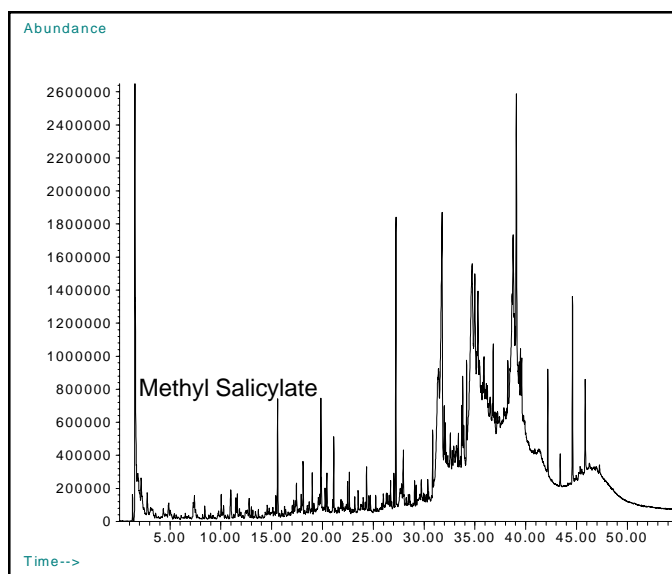


Figure 2. Detergent Ester

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: 15sec
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, 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.



CDS Analytical, Inc. has been a leader in the design and manufacture of laboratory instruments for sample preparation and analysis since 1969. We are dedicated to providing the best possible instruments for both research and routine analysis. Well known in the field of pyrolysis, CDS manufactures the Pyroprobe® 1000, 2000 and 2500 autosampler for the introduction and analysis of solid materials by GC, MS and FT-IR. CDS offers a complete line of dynamic headspace instruments for the analysis of volatile organic compounds in environmental, pharmaceutical and food applications, including the model 6500 16 position autosampler for complex, multicomponent materials investigation. Our customers, their requirements and applications are important to us. To help meet your needs, we offer a wide range of analytical information and the services of our applications laboratory. If you would like additional information, please contact us at the address below, call us at 1 800 541 6593, or log onto www.cdsanalytical.com.