APPLICATION BRIEF

Gas Chromatography

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Hydrogenation of Crude Oil at Elevated Pressure

Pyrolysis-GC/MS (Py-GC/MS) is a simple tool for the characterization of various crude oils. The rapid heating of approximately 0.5 - 1 µL of oil volatilizes it directly to the GC, where it is then analyzed like any other complex mixture. For example the bottom chromatogram in Figure 1 shows Py-GC/MS in helium of a crude oil sample.

The addition of a catalytic reactor, a pressure regulator and a trap to the system permits treating the oil in a reactive atmosphere, such as hydrogen, at higher pressures. The sample is volatilized by the Pyroprobe, and the resulting compounds are then carried by the reactant gas through the reactor to the trap, which is positioned after the back-pressure regulator. The hydrogenated products are collected by the trap, which is then thermally desorbed to the gas chromatograph. The middle chromatogram in Figure 1 shows the effect of heating the oil at 600 °C in hydrogen at 100 PSI with the flow moving through a reactor with a platinum catalyst at 500 °C. Under these conditions, compounds with double bonds are hydrogenated, converting olefins to paraffins.

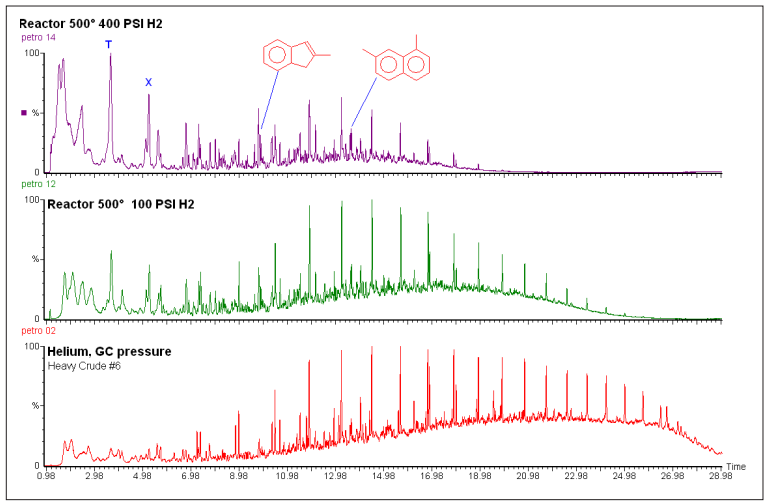

Figure 1. Py-GC of crude oil (bottom), at 100 PSI Hydrogen (center) and at 400 PSI (top)

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This effect is seen more easily in Figure 2. Here a series of olefins is marked with "O"s in the standard Py-GC/MS run on the bottom. Using a hydrogen carrier at 100 PSI, these olefins are completely hydrogenated, so the peaks are absent in the upper chromatogram.

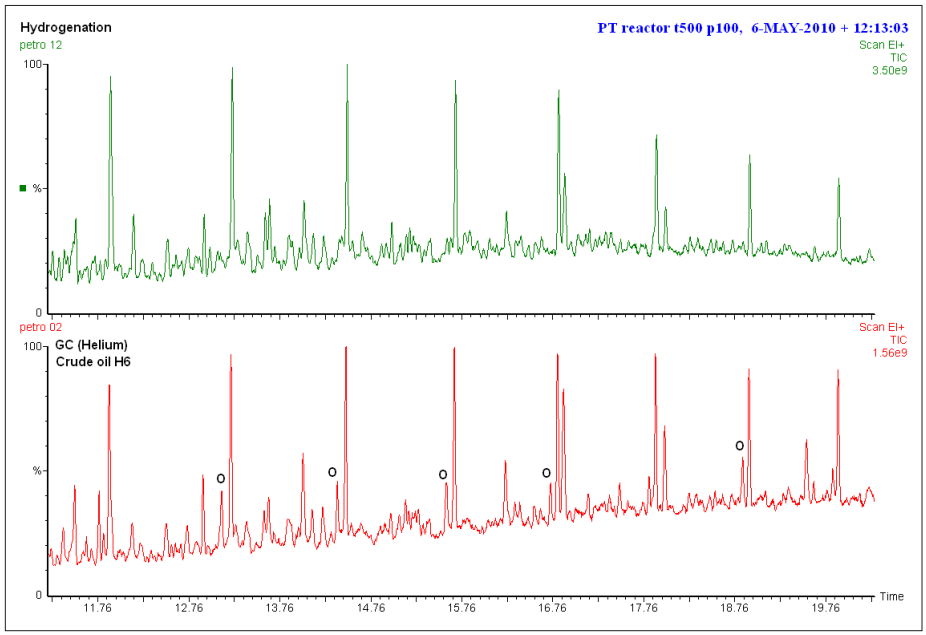

Figure 2. Expanded pyrograms of crude oil in He with olefins marked "O" (bottom), and H_2 (top).

If the pressure is increased further, the effect is to generate aromatics. For the upper chromatogram in Figure 1 the reactor temperature is still 500 °C, but the pressure is now 400 PSI. Benzene, toluene, xylenes and heavier aromatics are generated in large quantities, although some of the aliphatics may still be seen. In addition, the chromatogram is now characterized by an increase in early eluters and a considerable reduction in the less volatile compounds present in the original crude oil.

INSTRUMENT CONDITIONS

PerkinElmer Clarus® GC/MS

Column: 30 m x 0.25 mm 5% phenyl
Carrier: Helium, 50:1 split
Program: 40 °C for 2 minutes,10 °C/min to 300 °C

CDS® Pyroprobe 5200 HP-R

Interface: 325 °C for 4 minutes
Pyrolysis: 600 °C for 15 seconds
Reactor: 500 °C, Platinum
Pressure: 100 PSI, 400 PSI
Trap: 325 °C for 4 minutes

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