CDS solutions

APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

Introduction of Liquid Samples using a Pyroprobe

Once a Pyroprobe has been connected to the injection port of the GC, it may still be desirable to inject liquid samples, for example to provide standards, without disconnecting the Pyroprobe from the GC. Injecting the solution into a quartz tube filled with quartz wool works for semivolatile analytes, but, as shown in Figure 1, even a sample like crude oil can lose a considerable portion of the more volatile compounds this way. Even if the quartz tube is only exposed to air for a minute before being placed into the Pyroprobe, the early eluters begin to evaporate. If the tube is allowed to sit for 5 minutes, most of the compounds below decane are lost, as shown in Figure 2.

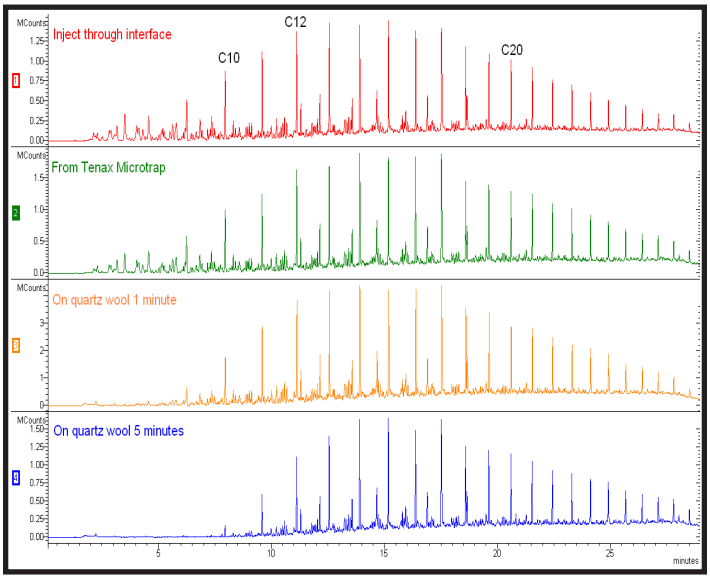

Figure 1. Crude oil transferred from a Pyroprobe to the GC.

A simple solution is to remove the probe rod and ferrule from the thumb wheel and replace them with a 9.5 mm septum. By heating the interface to injection port temperature, a syringe injection may be made into the interface zone, with the sample transferred directly to the GC or to the trap of a 5200.

Another solution is to make a microtrap from a quartz tube by filling it with a sorbent like Tenax (see Figure 3 on the back). The liquid sample may be injected into the sorbent inside the tube, and then desorbed using the Pyroprobe coil to heat the tube.

Figures 1 and 2 show the chromatograms of a crude oil injected through a septum on the interface, and desorbed from a Tenax microtrap. The chromatograms are essentially identical, and each retains the volatiles lost when the oil was just placed into a quartz tube for analysis.

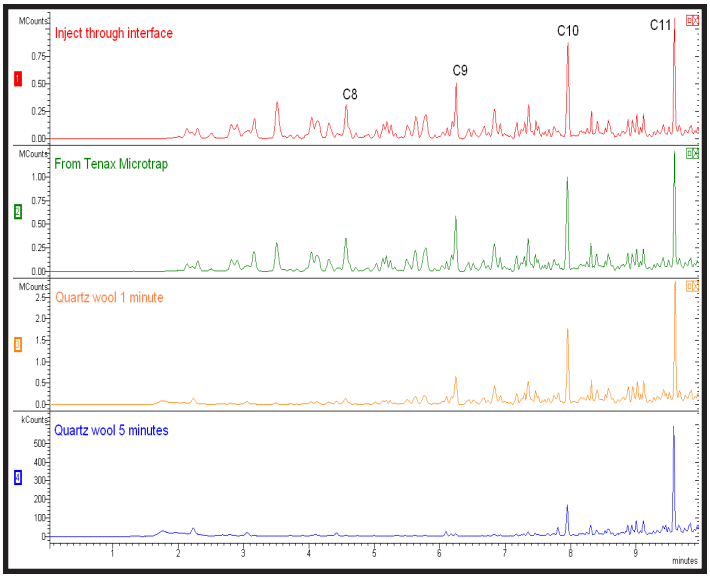

Figure 2. Crude oil chromatograms expanded to show effect on early eluters.

Equipment

Pyroprobe 5200

Interface:	325°C for 4 minutes
Filament:	350°C for 30 seconds
Valve Oven:	325°C
Transfer line:	325°C
Trap desorption:	325°C for 4 minutes
Dry (recondition):	350°C for 60 seconds

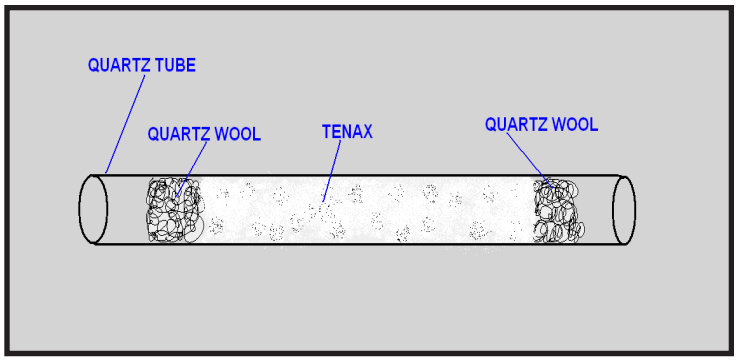

Figure 3. Quartz tube microtrap made by filling the center half of a standard Pyroprobe quartz tube with a sorbent, held in place with plugs of quartz wool. The Tenax can be conditioned before use by heating to 350°C in the coil of the Pyroprobe for several minutes.

GC/MS

Column:	30 m x 0.25 mm 5% phenyl methyl silicone
Carrier:	Helium
Split:	50:1
Oven program:	40°C for 2 minutes 10°C/min to 300 °C

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