Determination of Polymethyltrifluoropropyl Siloxane in Crude Oil

Application Note

Energy
Polymers

Author:
T. Wampler

Poly[methyl(trifluoropropyl)siloxane], (PMTFPS) is, among other things, an antifoaming agent added to crude oils, generally in the low PPM levels. Since it is a polymer, it cannot be determined by GC directly, but may be pyrolyzed to produce fragments that are compatible with GC. When the polymer itself is pyrolyzed at 750°C, the pyrogram looks like the top chromatogram in Figure 1. Several of the major peaks in the pyrolysate have mass spectra with a large peak for ion 233. This ion was selected from the TIC, and is shown in the lower chromatogram in Figure 1. To determine the presence of PMTFPS in crude oil, the peaks at about 7 and 9 minutes were selected.

Ion 233 is not a significant ion in the chromatogram of crude oil, especially in the first 10 minutes. To prepare the sample, 1 μl of crude oil was injected into a quartz tube filled with quartz wool. The sample was first heated to 300°C while being purged to vent, to remove many of the early eluting compounds from the oil itself. This was done automatically during the Pyroprobe accessory initial step. After this, the remaining sample was pyrolyzed at 750°C.

Crude oil with no PMTFPS showed no peaks at the retention times of the PMT-FPS products (top pyrogram in Figure 2). When PMTFPS is present at the 15 PPM level, however, the characteristic peaks from the polymer are clearly visible in the pyrogram displaying ion 233.

Pyroprobe Conditions:

Interface Initial:	300°C for 4 minutes
Interface Final:	325°C for 3 minutes
Pyrolysis:	750°C for 15 seconds
Valve oven:	325° C
Transfer line:	325° C

GC Conditions:

GC/MS	
Column:	30m x .25μm 5% phenyl
Carrier:	Helium, 1.2ml/min
Split:	50:1
Injector:	325°C
Oven:	40°C for 2 min
	10°C/min to 320°C
Mass range:	35 to 600 amu

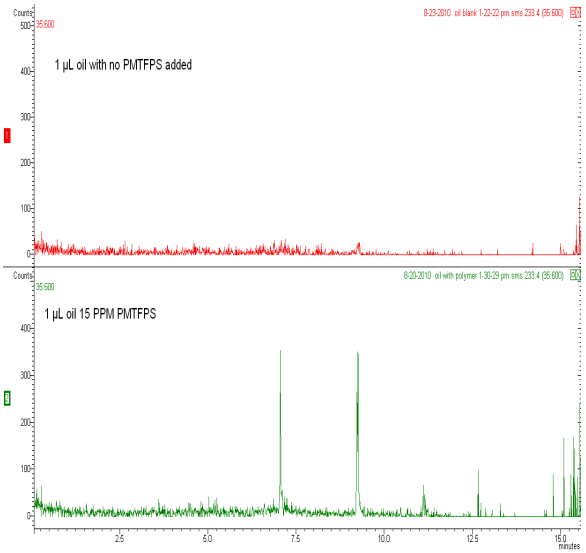

Figure 1. Pyrolysis of PMTFPS at 750°C, TIC (top) and ion 233 (bottom).

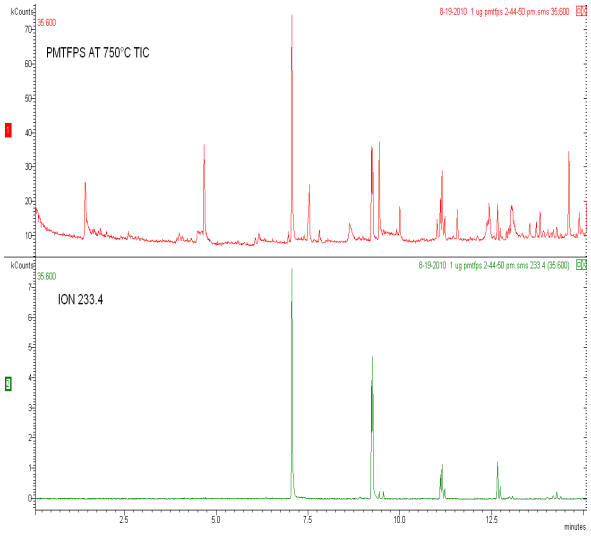

Figure 2. Ion 233 for a sample of crude oil with no PMT-FPS (top) and for a sample with 15 PPM (bottom).

For more information on this and related applications, we recommend the following readings:

K. D. Jansson et al., Determination of polymer additives using analytical pyrolysis, J. Anal. Appl. Pyrolysis 79 (2007) 353-361.

.