

# CDSolutions

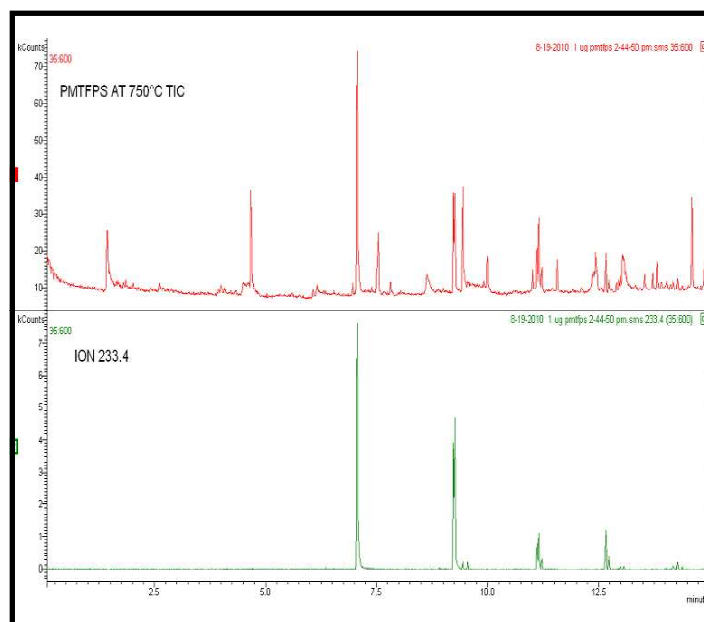
## APPLICATIONS INFORMATION USING ADVANCED SAMPLE HANDLING TECHNOLOGY

### Determination of Polymethyltrifluoropropyl Siloxane in Crude Oil

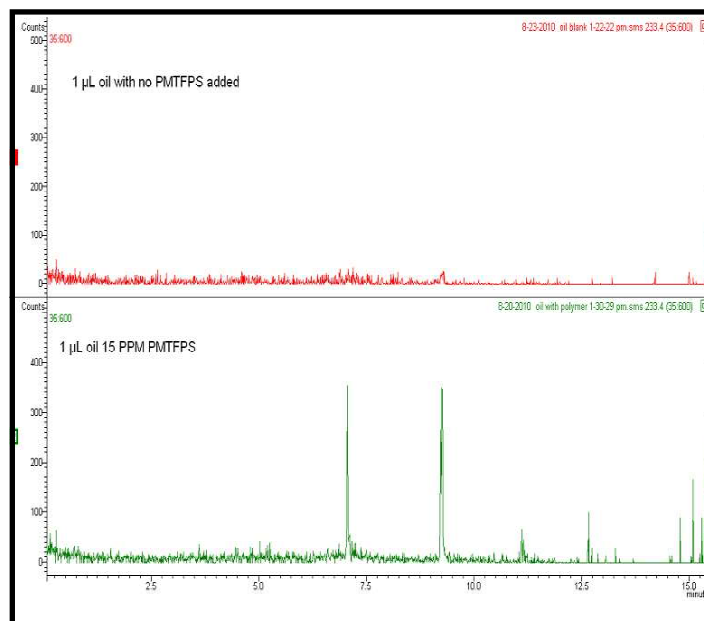
Poly[methyl(trifluoropropyl)siloxane], (PMTFPS) is, among other things, an antifoaming agent added to crude oils, generally in the low PPM levels. Since it is a polymer, it cannot be determined by GC directly, but may be pyrolyzed to produce fragments that are compatible with GC. When the polymer itself 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  $\mu\text{L}$  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 PMTFPS 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.



**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 PMTFPS (top) and for a sample with 15 PPM (bottom).

## Experimental Conditions

The samples were pyrolyzed in a quartz tube inserted into the coil filament rod of a Pyroprobe 5200, which was interfaced to a GC/MS.

### Pyrolysis

Interface:

Initial	300°C	5 minutes
Final	325°C	3 minutes

Pyrolysis:

750°C 15.00 seconds

Iso zones:

Transfer Line:	325°C
Valve oven:	325°C

### GC/MS

Injection Port: 325°C

Carrier: Helium, 1.2 mlmin

Split: 50:1

Oven: 40°C for 2 min  
10°C/min to 320°C

Mass range: 35 to 600 amu

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

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

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® 5000, 5150, 5200 and 5250 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 8400 four-position autosampler. CDS also manufactures the Dynatherm line of thermal desorption instruments including the 9000 series for air monitoring and the 9300 TDA. 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](http://www.cdsanalytical.com).