

## Analyses of Drilling Tool-related Plastics and Mud-related Polymers Using Pyrolysis Gas Chromatography/Mass Spectrometry

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### Abstract

The type of polymers added to the drilling mud and the plastics used in drilling equipment are critical in a high temperature-high pressure (HTHP) drilling environment.

Pyrolysis gas chromatography/mass spectrometry (pyro-GC/MS) is a powerful instrument for analyzing polymers and plastics. It helps the field engineers identify unknown masses found on the shaker. It also helps lab engineers verify different plastic parts and identify unknown plastic fragments and unknown contaminants.

Several examples are provided to demonstrate pyro-GC/MS capabilities that might be overlooked by field or lab engineers: 1) it identified lumpy unknown fragments collected from the shaker as badly deformed Teflon pieces; 2) it identified that two submitted Viton parts have similar key chemical composition; 3) it identified the black contaminant on a metal part as silicon grease; 4) it identified the jelly-like mass sample from the shaker as an undispersed polymer from a specific mud additive; and 5) it identified an O-ring as high density polyethylene (HDPE) and a plastic fragment as HDPE with silica beads.

The analysis normally takes 20 minutes per sample and the sample preparation is straightforward. Scanning electron microscope (SEM) with energy dispersive spectrometry (SEM/EDS)<sup>1</sup> and thermogravimetric analysis (TGA) are used as supportive instruments with pyro-GC/MS analyses at times.

### Introduction

In a routine pyro-GC/MS analysis, a small amount of the sample is thermally broken down into chemical fragments prior to GC/MS analysis. The results are referred back to identify the original compound(s) with or without lab standards. Five analyses covering different applications are described in this study.

#### Lumpy masses found on a shaker

The lumpy masses (Figure 1) submitted by a field crew were ultrasonic-cleaned and rinsed with isopropanol to remove mud-related material. A small piece of the highly-deformed sample was introduced to the pyro-GC/MS for direct analysis. The results show that the thermal-breakdown chemicals are mainly fluorine-related. SEM/EDS results also show that the major elements in the cleaned sample are fluorine and carbon

with small amounts of oxygen and silicon. A reasonable interpretation is that the lumpy mass found on the shaker is likely a Teflon-based plastic. Total time spent on sample preparations and analysis was less than four hours.

#### Chemical comparison of two compression spacers

An in-house customer submitted two compression spacers to determine if the smaller one was made of similar material to the large one.

The gas chromatograms show that both samples were nearly identical in their chemical composition (Figure 2). The TGA results (Figure 3) show both samples have a very similar thermal degradation pattern, with the smaller spacer containing higher inorganic filler than the larger one.

SEM/EDS analysis results show that the inorganic fillers in both spacers are SiO<sub>2</sub>-based. The large one also contains small amounts of Fe-related material besides SiO<sub>2</sub> filler.

#### Unexpected grease coating on a drilling tool

Trace amounts of the black greasy contaminant were collected from a downhole tool and delivered to the lab on a small cotton swab. The organic material was extracted by methylene chloride and the fluid was left to air dry in the pyro-GC sample container.

Pyro-GC/MS results show that the contaminants are mainly silicon oil/grease (Figure 4) and are not mud or formation-related organics.

#### Undispersed jelly material found on the shaker

In one case the field crew found unexpected gel masses on the shaker. The submitted gel was washed with hexane and dried under the heat lamp. A small amount of the dried sample was analyzed by pyro-GC/MS and was compared with the lab GC polymer library. Figure 5 show that the gel sample is mainly Polymer X (a styrene-acrylate based copolymer) with small amounts of Polymer Y.

#### Identifying a plastic O-ring and a plastic fragment

An internal client submitted an O-ring and a plastic fragment to see if they were made of the same type of material. Pyro-GC/MS analysis results show that both are made of high-density polyethylene HDPE (Figure 6). SEM/EDS results show that the plastic fragments also contain

silica beads in the plastic matrix. It is clear that these samples were not totally identical in composition.

### **Conclusions**

Unexpected material surfacing on the shaker may indicate mechanical or mud-related problems. Drilling engineers might want to identify the unexpected contaminants and plastic material in question. Pyro-GC/MS is a powerful instrument for analyzing polymers and plastics. It helps both the lab and the field engineers solve seemingly complicated problems in a timely manner.

### **Acknowledgments**

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### **References**

1. Yang, S. V.: "Formation Damage Caused by Drilling Mud: A Comprehensive Study Using SEM/EDS, GC/MS and Related Instruments." AADE-10-DF-HO-17, AADE National Technical Conference, Houston, April 6-7, 2010.

## Figures

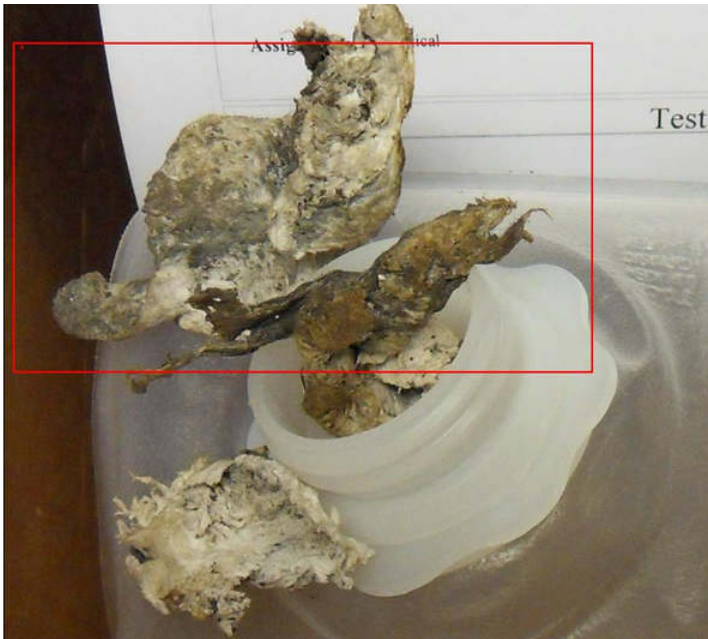


Figure 1. Lumpy material collected from the shaker.

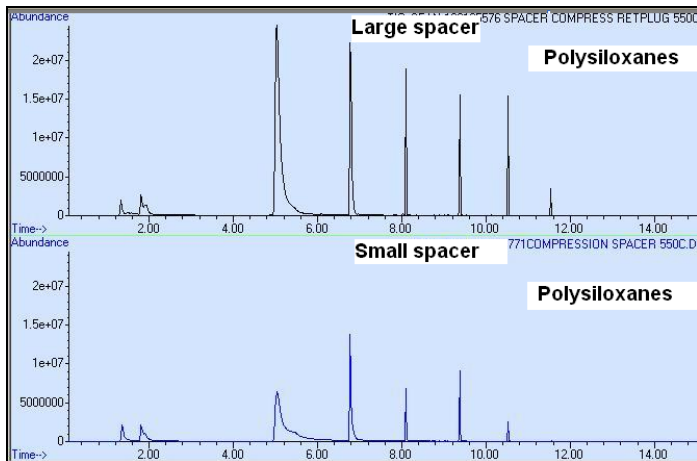


Figure 2. Both spacers are made of polysiloxane based material.

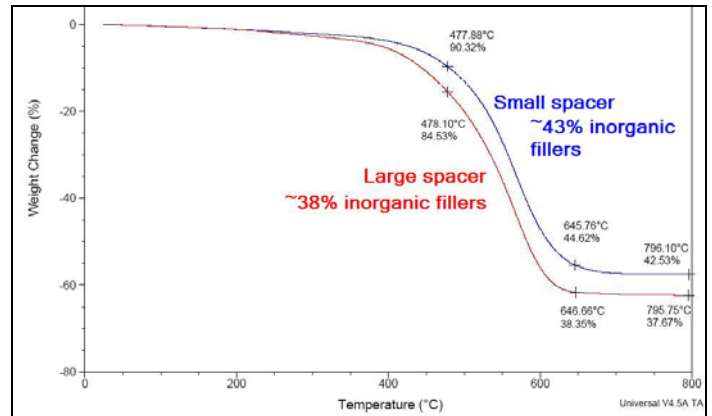


Figure 3. TGA analysis results show that both spacers have very similar thermal degradation paths. Their inorganic fillers are slightly different in quantity.

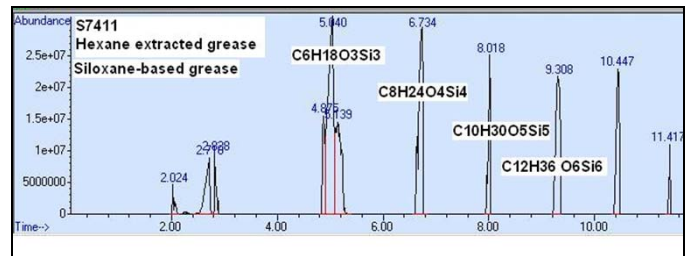


Figure 4. The black contaminant is siloxane based grease.

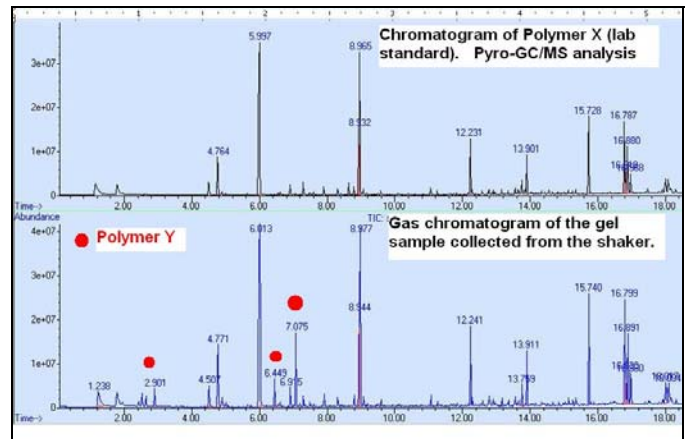
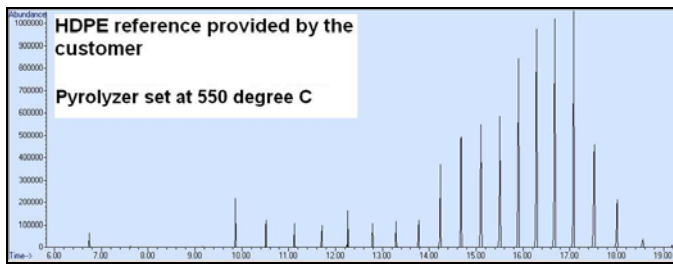


Figure 5. The undispersed gel masses collected from the shaker are positively identified by pyro-GC/MS as Polymer X with small amounts of polymer Y.



**Figure 6. The gas chromatogram of the HDPE standard provided by the customer. The pyrolyzer was set at 550°C prior to GC/MS analysis.**