Petrochemical Applications

Rt®-XLSulfur Packed GC Column for Analysis of Low-Level Sulfur Compounds in C1-C6 Hydrocarbon Streams

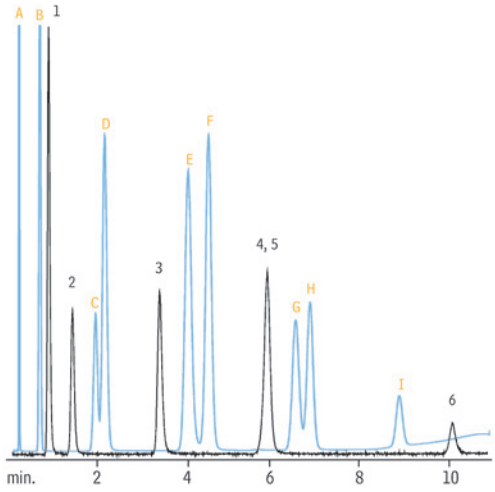
The analysis of sulfur compounds in C1-C6 hydrocarbon streams by gas chromatography (GC) is an important application in the petrochemical field. The presence of sulfur compounds in petroleum products can affect the longevity and performance of catalysts used in hydrocarbon processing. As requirements for sulfur detection become more stringent, the importance of good chromatographic separation of the hydrocarbons from the sulfur compounds and the inertness of the analytical column increases. Detectors used for sulfur determination generally are specific (e.g., sulfur chemiluminescence detection, FPD, PFPD) and help eliminate positive response from chromatographic interferences. Unfortunately, when high levels of hydrocarbons elute through the detector simultaneously with sulfur compounds, the signal for sulfur is quenched and area counts are nonlinear. For a successful analysis, the analytical column must resolve the hydrocarbons from the sulfur compounds listed in Figure 1.

Hydrocarbons are non-reactive but sulfur compounds, especially hydrogen sulfide and methyl mercaptan, are easily adsorbed by undeactivated surfaces. Therefore, there are two areas of concern with micropacked or packed column sulfur analysis: one is the inertness and selectivity of the solid support, and the other is the inertness of the tubing walls. Packed and micropacked columns typically use metal tubing for ruggedness but the surface is very adsorptive for sulfur compounds. PTFE tubing is also an option, but it has a limited temperature range, is permeable, and will expand and contract during temperature changes. These characteristics will negatively affect column efficiency and stability.

Restek designed the Rt®-XLSulfur column to accomplish the challenging separation of hydrocarbons from sulfur compounds. All parts of the column have been optimized for inertness. The packing material is extensively deactivated for the analysis of low ppbv levels of hydrogen sulfide and methyl mercaptan, and then is prepared to achieve the proper selectivity and required resolution (Figure 1). Analysis of 50 ppbv sulfur compounds using a 1 mL gas loop and a sulfur chemiluminescence detector (SCD) shows excellent response (Figure 2).

The interior tubing walls of the Rt®-XLSulfur column are treated with a Sulfinert® coating, a passivation technique designed to deactivate metal surfaces. This coating is found to be very inert for all sulfur compounds, including hydrogen sulfide and methyl mercaptan. Another issue that is routinely overlooked with packed columns is that their end plugs are known to adsorb sulfur compounds. For that reason, Restek also treated the end plugs in the Rt®-XLSulfur column with Sulfinert® passivation. The extra care taken with the surfaces in this column result in a more accurate analysis of trace sulfur compounds in hydrocarbon processes.

Figure 1 The Rt®-XLSulfur micropacked column separates hydrocarbons from sulfur compounds.



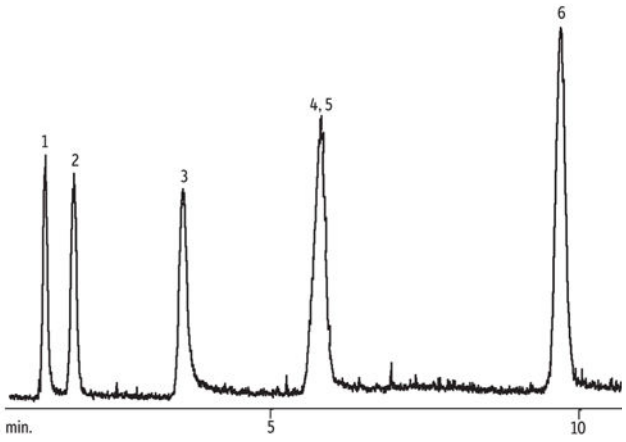
GC_PC00436

Sulfurs
1. Hydrogen sulfide
2. Carbonyl sulfide
3. Methyl mercaptan
4. Ethyl mercaptan
5. Dimethyl sulfide
6. Dimethyl disulfide

Hydrocarbons
A. Methane
B. Ethane
C. Propylene
D. Propane
E. Isobutane
F. Butane
G. Isopentane
H. Pentane
I. Hexane

Column	Rt®-XLSulfur, 1m, 0.95mm OD, 0.75 mm ID (cat.# 19806)
Sample	
Conc.:	50 ppb each
Injection	packed not on-column
Oven	
Oven Temp:	60 °C to 230 °C at 15 °C/min.
Carrier Gas	He, constant flow
Flow Rate:	9 mL/min.
Detector	SCD/FID
Acknowledgement	Sulfur standards courtesy of DCG Partnership 1 Ltd., Pearland, TX.

Figure 2 The Rt®-XLSulfur column is sensitive enough for 50 ppbv sulfur compounds.



GC_PC1221

Peaks
1. Hydrogen sulfide
2. Carbonyl sulfide
3. Methyl mercaptan
4. Ethyl mercaptan
5. Dimethyl sulfide
6. Dimethyl disulfide

Column	Rt®-XLSulfur, 1 m, 0.75 mm ID (cat.# 19806)
Sample	
Conc.:	1 mL of 50 ppbv each sulfur compound
Injection	sample valve
Oven	
Oven Temp:	60 °C to 230 °C at 15 °C/min.
Carrier Gas	He, constant flow
Flow Rate:	9 mL/min.
Detector	SCD

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