# GC Analysis of Sulfur Components in Propylene using a Pulsed Flame Photometric Detector

Application Note

Hydrocarbon Processing


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## Abstract

The Agilent J&W Select Low Sulfur column measures trace levels of target components in C3 hydrocarbon streams without any matrix interference.


## Introduction

Hydrogen sulfide ($H_2S$), carbonyl sulfide (COS) and methyl mercaptan ($CH_3SH$) are common components in light hydrocarbon streams. They have corrosive and toxic properties, causing damage to pipes and equipment. The emission of undesired odors caused by volatile sulfur compounds in intermediates and final products have serious economic and environmental impact. In addition, the presence of sulfur can affect the performance of industrial processes, causing chemical reactions, loss of catalyst activity (catalyst poisoning), and ultimately lower yield.

These sulfur components must be quantified at low ppb levels. They can be measured with sulfur specific detection devices like the Pulsed Flame Photometric Detector (PFPD) but large sample volumes are needed to reach the desired low parts per billion (ppb) detection limits. This creates matrix overload and quenching effects (decreased signal/sensitivity due to background interferences) on most sulfur specific detectors, limiting the detector's sensitivity and linearity and raising quantification limits. The capillary PLOT column, Agilent J&W Select Low Sulfur column, with a novel stationary phase was developed for the analysis of sulfur species such as $H_2S$, COS and $CH_3SH$ in light hydrocarbon C3 matrices, with high loadability properties and unique selectivity giving baseline resolution for sulfur components and matrix components.

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## Experimental

| | |
|---|---|
| Technique: | GC-PFPD |
| Column: | Agilent J&W Select Low Sulfur, 60 m × 0.32 mm (p/n CP8575) |
| Oven: | 65 °C isotherm |
| Carrier gas: | Helium, constant flow, 2.0 mL/min |
| Injector: | 200 °C, split 1:20 |
| Detector: | PFPD, 200 °C |
| Sample: | Propylene matrix containing ~500 ppb $H_2S$, COS, and $CH_3SH$ |
| Injection volume: | 1 mL |
| Injection: | Gas sampling valve |

## Results and Discussion

The stationary phase shows good selectivity between $H_2S$, COS and low mercaptans in various C3 hydrocarbon matrices. Therefore, co-elution of the sulfur components and the matrix, which causes "quenching", is avoided.

The system was equipped with a gas sampling valve. The gas sampling valve event table is shown in Table 1. The detector settings are shown in Table 2.

*Table 1. Gas Sampling Valve Event Table*

| Time (min) | Gas sampling valve |
|---|---|
| Initial | Fill |
| 0.01 | Inject |
| 1.00 | Fill |

*Table 2. Detector PFPD Settings*

| Combustion gases | |
|---|---|
| Air (1) | 17 mL/min |
| $H_2$ | 13 mL/min |
| Air (2) | 10 mL/min |
| Trigger level | 250 mV |
| Tube voltage | 550 V |
| Sampling delay | 6 ms |
| Sampling width | 20 ms |

Figure 1 shows the chromatogram of sulfur compounds $H_2S$, COS, and $CH_3SH$ in a propylene matrix. Methyl mercaptan shows peak broadening from column overloading by the large amount of propylene. The propylene matrix elutes between COS and methyl mercaptan.

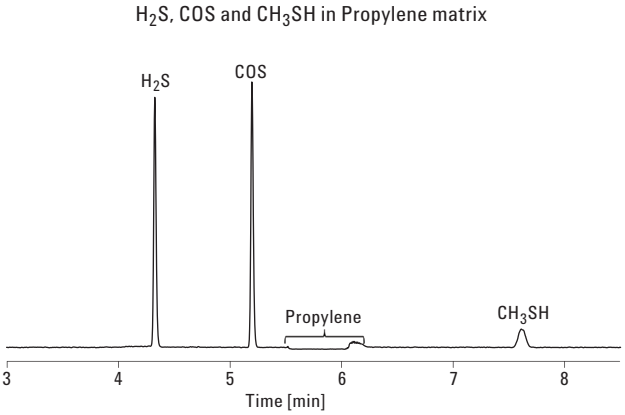


*Figure 1. Chromatogram of sulfur compounds $H_2S$, COS and $CH_3SH$ in a propylene matrix, using the Agilent J&W Select Low Sulfur with GC-PFPD.*

## Conclusion

The Agilent J&W Select Low Sulfur used in a GC with a sulfur specific detector, such as a PFPD, can detect $H_2S$, COS and $CH_3SH$ at trace levels in a propylene matrix as a result of excellent separation of the sulfur compounds and the matrix. Separating the matrix from the sulfur components eliminates the "quenching" effects caused by the matrix. This provides a better response for the sulfur compounds. The column provides a good response for reactive sulfur compounds, such as $H_2S$, which makes detections of 20 ppb possible.

Although this is a PLOT column, no spikes will be observed because this column does not shed particles. It can therefore be used safely in combination with valves.

## References

1. W. Wardencki (1998) Review "Problems with the determination of environmental sulphur compounds by gas chromatography." J. Chromatog. A. 793: 1-19.
2. Roger L. Firor and Bruce D. Quimby, "Comparison of Sulfur Selective Detectors for Low-Level Analysis in Gaseous Streams," Agilent Technologies publication 5988-2426EN.

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