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Application Note

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Analysis of Thiophene in Benzene Using ASTM Method D7011 and a Pulsed Flame Photometric Detector (PFPD)

PETROCHEMICAL SERIES

Introduction

High purity benzene is key in numerous chemical manufacturing processes. Even very small amounts of thiophene in benzene can poison catalysts making the determination of trace level amounts essential to the petroleum industry. ASTM Method D7011covers the determination of thiophene in refined benzene using gas chromatography and sulfur selective detection.

This application note presents instrument configuration and operating parameters for the detection and quantitation of trace levels of thiophene in benzene using ASTM Method D7011 and PFPD.

Figure 1. OI Analytical 5383 Pulsed Flame Photometric Detector

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Instrumentation

Instrumentation for this study included an OI Analytical Model 5383 PFPD (Figure 1) mounted on an Agilent 7890A GC system with split/splitless injection port.

Experimental

Instrument operating conditions are shown in Table 1. The PFPD was tuned for optimum sulfur response. It was configured for simultaneous sulfur and hydrocarbon detection with sulfur run in the linearized mode, ie., with the square root on.

The instrument was calibrated by injecting 1µl thiophene in benzene standards at 0.02, 0.2, 0.5, 1, and 2 ppm. A quality assurance check standard and benzene blank were also analyzed. All injections were made in duplicate.

Table 1. Instrument Configuration & Operating Conditions

Inlet	220 °C Split mode Split ratio 5:1 (manual injections) Sulfinert coated
GC Column	Agilent J&W DB - WAX 30-m x 0.25-mm ID x 0.5-µm film Helium carrier gas, 1.2 mL / min
Oven Program (Agilent 7890A)	50 °C for 1 minute 10 °C / minute to 100 °C 30 °C / minute to 200 °C Hold for 1 minute Total run time 10.33 minutes
Sulfur Detection	Pulsed Flame Photometric Detector (PFPD) 2-mm combustor, BG-12 filter, R1924 PMT Detector base temperature 250 °C H_2/air ratio tuned for optimum sulfur emission 6-24 milliseconds sulfur gate (linear mode; square root on) 1-2 milliseconds hydrocarbon gate

Results and Discussion

Calibration

A five-point calibration was analyzed and the Agilent GC ChemStation OpenLab data system was used to generate a calibration curve using average response. Linearity was established with a correlation coefficient of 0.9995 and residual standard deviation of 14.35. See Figure 2.

Figure 2. Calibration 0.02 - 2.0 ppm

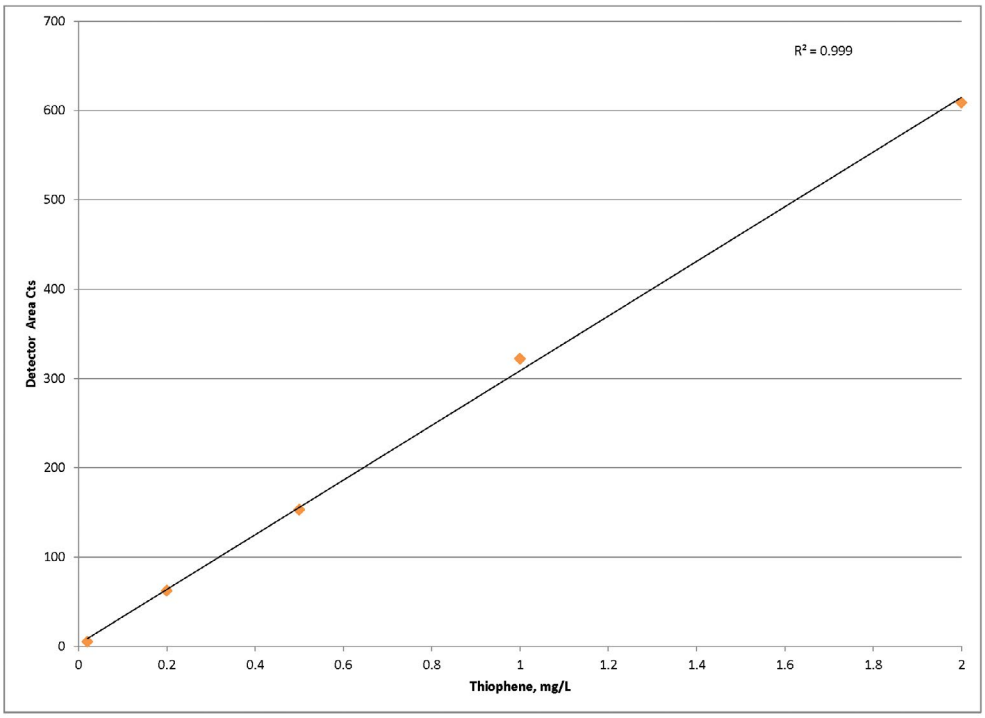


Figure 3. 0.02 ppm Standard

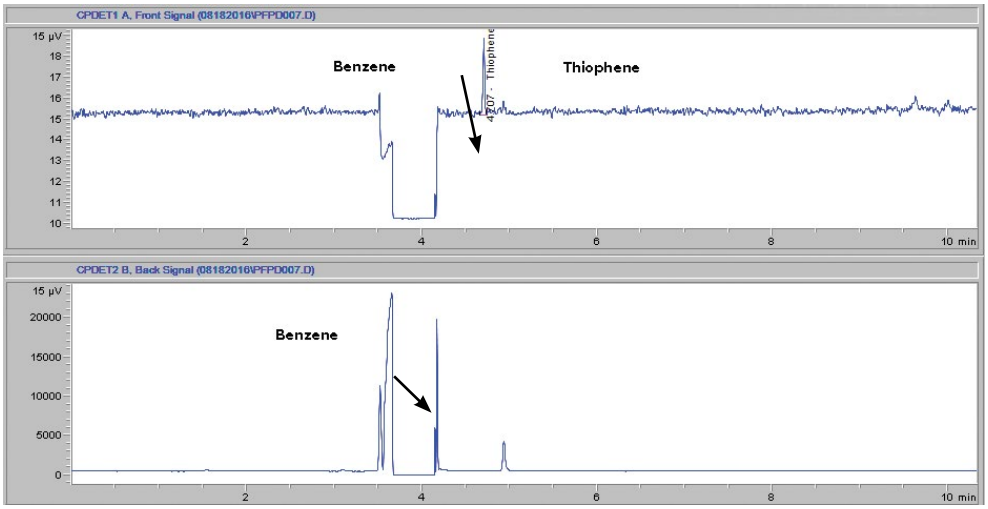
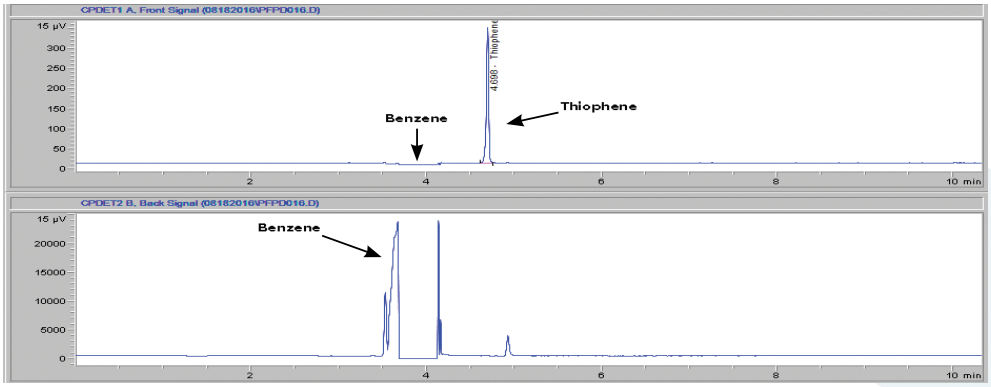


Figure 4. 2.0 ppm Standard



Method Detection Limit Study

Seven injections of 0.01 ppm thiophene in benzene were made. The calculated MDL is 4.4 ppb.

System Stability

A repeatability study was performed over a 10 day period with 80 replicates (manual injections) of 0.2 ppm. A fairly low RSD of 9.01% exemplifies the stability of the PFPD and GC system.

Conclusion

The OI Analytical 5383 PFPD coupled with the Agilent 7890A is ideally suited for the analysis of thiophene in benzene using ASTM D7011. All method requirements and QC criteria were easily met using a fast and reliable method.

Reference

ASTM D7011, 2015, "Standard Test Method for Determination of Trace Thiophene in Refined Benzene by Gas Chromatography and Sulfur Selective Detection," ASTM International West Conshohocken, PA.

Acknowledgement

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