Agilent Model 355 Sulfur Chemiluminescence Detector (SCD): Thiophene in Benzene

Technical Overview

Introduction

This overview discusses the analysis of benzene for sulfur contamination. The sensitivity, selectivity, and linear response of the Agilent 355 Sulfur Chemiluminescence Detector (SCD) in the analysis of trace levels of thiophene in benzene illustrates that the Model 355 is well-suited for low-level sulfur analysis.

Benzene is the basic unit of the aromatic class of compounds. The primary sources of benzene are from extraction of hydrocarbon crude distillates, refinery catalytic reforming, carbonization of coal, and the hydrodealkylation of a toluene charge stock. [1] The hydrodealkylation reaction results in the conversion of about 90% of the aromatics in the feed with a selectivity factor of about 95%. Thiophene, which also occurs in the light hydrocarbon fractions distilled from crude stocks and coal tar, is present in levels from 0.4 to 1.4 wt. %. [2] Traditionally, thiophene has been extracted by washing with H_2SO_4 to produce a sweeter product. However, this and other processes of thiophene removal still have difficulty reaching the level of purity required by many chemical markets. Consequently, it is often important to monitor trace levels of thiophene in benzene. The three main applications for benzene are production of ethylbenzene, cumene, and cyclohexane. These three products account for 80% of the benzene consumed as a chemical feedstock. [2]

These species can be classified as intermediates for a wide range of final products, including dyes, resins, solvents, and polymers, including nylon. The purity requirements for synthetic applications continue to become more confining, thereby increasing the need to monitor sulfur removal efficiency and to verify the purity of starting materials.

The data in Figure 1 illustrate the sensitivity of the 355 SCD for trace level analysis of sulfur in a hydrocarbon matrix without interference. Figure 2 displays the linear response of the SCD at trace levels. Correlation coefficients for five orders of magnitude were better than $R^2 = 0.999$. The selectivity of the 355 SCD for sulfur over hydrocarbon is shown in Figures 1 and 3, where sulfur chromatograms show no hydrocarbon interference from the eluting benzene.

The data in Figures 1 and 2 were collected on a Agilent 6890 gas chromatograph with a Agilent 355 SCD directly attached. The chromatograms in Figure 3 were collected simultaneously without column splitting, using the flame ionization detector attachment to the SCD. The chromatographic conditions for the trace analysis are summarized in Table 1.

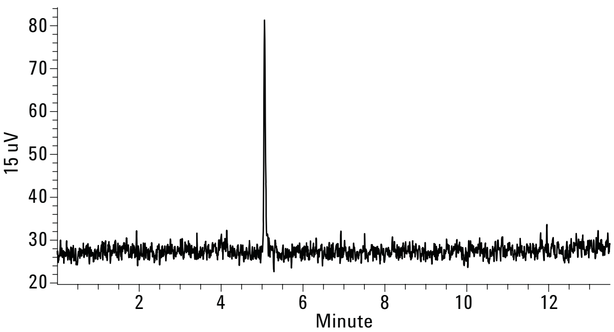

Figure 1. Chromatogram illustrating the analysis of 15 ppb thiophene (as sulfur, split 1:10).

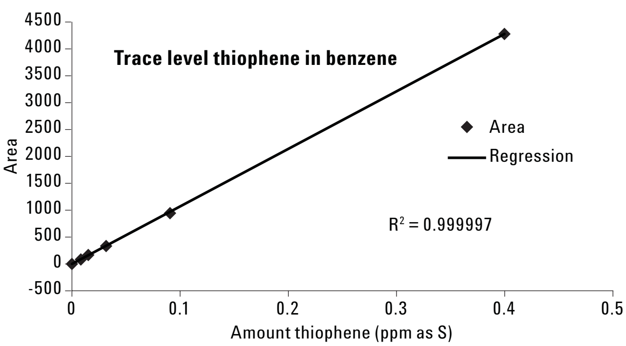

Figure 2. Linearity of trace level analysis of thiophene in benzene.

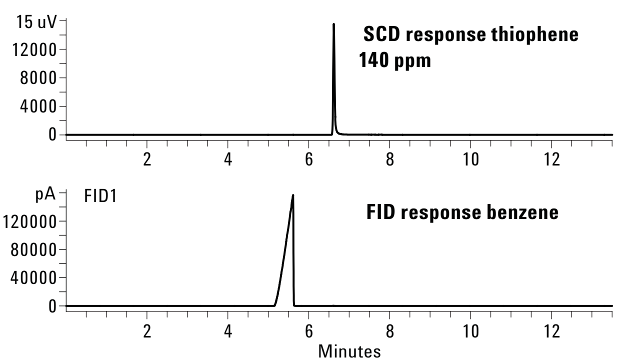

Figure 3. Simultaneous FID-SCD chromatogram on SUPELCOWAX-10.

Table 1. Chromatographic Conditions

Injection temperature	120 °C
Injection volume	1 µL
Initial temperature	30 °C
Initial time	2 min
Rate	10 °C/min
Final temperature	125 °C
Final time	2 min
Split ratio	1:10
Flow mode	Constant flow
Column flow	2 mL/min
Column type	SUPELCOWAX-10
Length	30 m
Internal diameter	0.32 mm
Film thickness	1 µm

References

1. James H. Gary and Glenn E. Handwerk, "Petroleum Refining Technology and Economics," Marcel Dekker Inc., New York, NY, 1984, Chapter 14.

2. Wolfgang Y. Gerhartz, Stephen Yamamoto, et. al. eds., "Ullmann's Encyclopedia of Industrial Chemistry," VCH Publisher, Deerfield Beach, FL, 1985, vol. A3.

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Printed in the USA
June 7, 2007
5989-6786EN

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