Agilent 355 Sulfur Chemiluminescence Detector (355 SCD): Sulfur Compounds in Ethylene and Propylene

Technical Overview

Introduction

This technical overview briefly describes the analysis of ethylene and propylene gases for trace amounts of hydrogen sulfide and carbonyl sulfide as well as other volatile sulfur compounds using gas chromatography and sulfur selective detection. The method provides for the determination of individual volatile sulfur-containing compounds, as well as the determination of total sulfur content in chemical feedstocks.

The measurement of trace amounts of volatile sulfur compounds in ethylene and proplyene is important because of the contaminant nature of these compounds in hydrocarbon feedstocks. Accurate gas chromatographic determinations of trace volatile sulfur compounds involve unique analytical difficulties due to the chemical nature of these compounds. Volatile sulfur compounds are particularly reactive and adsorptive in nature, making trace level analysis reliant on exceptionally good chromatographic technique, using inert sample handling systems and valving, and selective detection that is minimally affected by matrix interference. Because of their respective boiling point ranges, the measurement of hydrogen sulfide in ethylene and carbonyl sulfide in propylene is generally of great concern.

This analysis is especially difficult using detectors such as the flame photometric detectors, where coelution of the analyte and solvent contribute to hydrocarbon quenching and interference, which may result in erroneous results. The following chromatograms illustrate the ability of the SCD to selectively detect trace levels of volatile sulfur compounds in hydrocarbon gas samples without suffering from any quenching or interference from the hydrocarbon matrix.

The analyses presented here were performed on an Agilent 5890 Series II gas chromatograph equipped with a split/splitless injector. The Agilent Model 355 Sulfur Chemiluminescence Detector (SCD) was directly connected to an Astec Gaspro capillary column and operated according to standard conditions.

Figure 1 illustrates the power of the 355 SCD for the analysis of COS in propylene. A 1-mL propylene sample containing 60 ppb wt sulfur as COS was introduced to the GC with no pretreatment. The injector was operated with a split ratio of 1:6, and the linear velocity was approximately 38 cm sec^{-1}. Temperature programming for this analysis was as follows: 50 °C for 1 min to 100 °C at 10 °C/min. This chromatogram verifies the selectivity of the Agilent 355 SCD for sulfur over carbon, with no hydrocarbon response or anomalies visible in the baseline. Also evident is the sensitivity of the SCD to sulfur species, making it ideal for trace analysis.

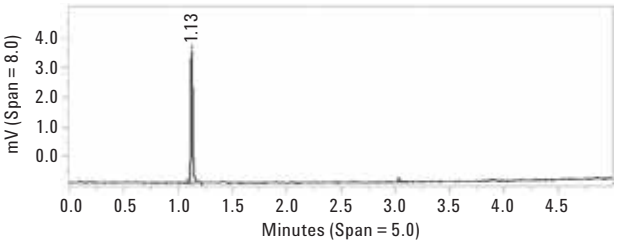

Figure 1. 60 ppb carbonyl sulfide in propylene.

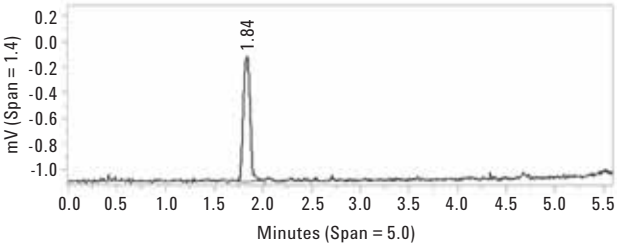

Figure 2. 100 ppb hydrogen sulfide in ethylene.

Figure 2 illustrates the analysis of 100 ppb hydrogen sulfide in ethylene using the GasPro column and SCD. As above, a 1-mL sample (gas) volume was introduced with a split 1:6. The oven temperature program started at 40 °C for 1 min and raised at 10 °C/min to the final temperature of 100 °C. In this case the temperature ramp for the analysis was deliberately set so that there would be a simultaneous elution of the ethylene and the hydrogen sulfide. As expected, this resulted in the exhibited band broadening; however, there was no evident quenching of the sulfur response and no hydrocarbon response or baseline anomalies. Although the SCD works well under these conditions, it is always recommended to separate the analyte from the matrix if at all possible to reduce solvent effects.

Recent developments in chromatographic column technology allow the ambient separation of hydrogen sulfide and carbonyl sulfide in hydrocarbon matrices. For ambient separation of H_2S and COS as well as light mercaptans and sulfides, capillary columns such as the Chrompack CP-SilicaPLOT (30 m 0.32 mm id) or the Astec Gaspro (15 m 0.32 mm id) are ideal. The retention characteristics of these columns are unique and seem to be best suited for light applications.

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Printed in the USA
June 14, 2007
5989-6791EN