Gas Chromatography and Sulfur Chemiluminescence Detection for Low-Sulfur Diesel

Technical Overview

Introduction

Environmental regulations are driving down sulfur levels in fuels. These regulations place many challenges on the refining industry, not the least of which is low sulfur measurement. This technical overview briefly describes the ability of the Agilent 355 Sulfur Chemiluminescence Detector (SCD) to help refiners meet some of the sulfur measurement challenges for low-sulfur diesel fuels.

Sulfur compounds occur naturally in petroleum and feedstocks for fuel production. Their presence creates processing challenges and impacts product quality. Many sulfur compounds are toxic, reactive, and corrosive to processing equipment and damage catalysts used in hydrocarbon processing. In terms of the product quality of fuels, sulfur compounds impart undesirable odors and contribute to gum formation in gasoline and diesel fuel, and turbine deposits in jet fuel, especially in military aircraft [1, 2].

From a pollution standpoint, all fuel sulfur contributes to acid rain. It is also known that sulfur compounds poison catalytic converters used in modern automobiles, contributing to other emission problems. In industrialized nations, strict environmental regulations directed toward sulfur have been critical to improving air quality. The U.S. EPA and other environmental regulatory bodies, particularly those in Europe, Japan, and other industrialized nations, have developed regulations aimed at further reducing air pollution from mobile sources. Sulfur plays a key role in this. The allowable levels of sulfur in liquid fuels, such as gasoline and diesel fuels, are being driven to lower levels.

Numerous instrumental methods are used to measure sulfur in gasoline and diesel fuels. [2] It is beyond the scope of this overview to cover these techniques. Nevertheless, it is safe to say that the sensitivity required for the fuels of tomorrow can only be achieved with a limited number of technologies, and only gas chromatography with sulfur-selective detection can provide the means to measure both trace total sulfur and sulfur speciation.

Total sulfur measurement alone provides very little information about the best manner to process a feed or even to determine whether a process is functioning as designed. For example, in a process that converts mercaptans to disulfides, total sulfur measurements would show no difference between the feed and the product, while a sulfur distribution measurement via gas chromatography and sulfur-selective detection tells if the intended chemical conversion is occurring. Simulated distillation methodologies can also tell how best to process a feed. Gas chromatographic techniques provide the means for determining individual sulfur species, types, and distribution, in addition to total sulfur.

This technical overview briefly illustrates the capability of gas chromatography with sulfur chemiluminescence detection using the Agilent 355 SCD for the analysis of sulfur in a low-sulfur diesel fuel. Figure 1 shows the response obtained from a diesel sample that contained approximately 6 mg/kg total sulfur, as determined by calibration with an NIST standard and independently by another total sulfur instrument. A brief description of the experimental details is given in the figure caption.

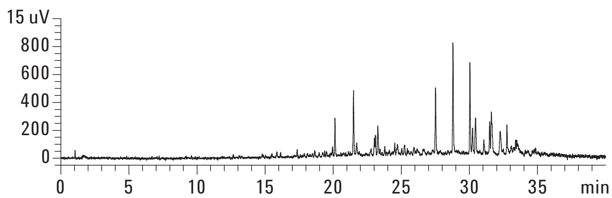

Figure 1. Low-sulfur diesel analysis by gas chromatography with the Agilent 355 SCD.

Experimental conditions:	
Injection:	1 mL 1:8 split, 350 °C
Column:	30 m, 0.32 mm id, 0.25 mm HP-5
Oven temperature:	100 °C for 3 min to 250 °C at 4 °C/min.

References

1 K. H. Altgalt and M. M. Boduszynski, "Composition and Analysis of Heavy Petroleum Fractions," 1994, Marcel, Dekker, N.Y., Chapter 1.

2 R. A. Kishore Nadkarni, American Laboratory, 2000, 32, 16.

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