Laboratory Total Sulfur Analysis by Gas Chromatography and Agilent Model 355 SCD

Technical Overview

Introduction

Gas chromatography with sulfur chemiluminescence detection (SCD) provides a relatively quick means to identify and quantify sulfur compounds that are present in petroleum and petrochemical feeds and products, such as natural gas, ethylene, propylene, liquefied petroleum gases, solvents, gasoline, and diesel fuels. In many cases, it is sufficient to simply measure the total sulfur content of a sample without the need for compound speciation. If that is the case, one can replace the GC column with an appropriate transfer line to connect the injector with the detector. This technical overview briefly covers the rapid determination of total sulfur using a GC as a sample introduction device and the SCD to quantitate the sulfur content of a sample.

Experimental conditions are as follows: Agilent Model 355 SCD operated according to standard conditions with FID adapter; 3-m high-purity stainless-steel transfer line from split/splitless injector to FID; oven, injector, and detector temperatures 200 °C; 1-L injector split 1:10; and 10 psi injector head pressure.

Figure 1 illustrates the measurement of 1 ppm of sulfur (thiophene) by weight in high-purity benzene. Note that the analysis is very rapid, requiring less than 15 seconds for the actual measurement. Analysis times will be longer than this of course because of sample handling, system calibration, and other requirements.

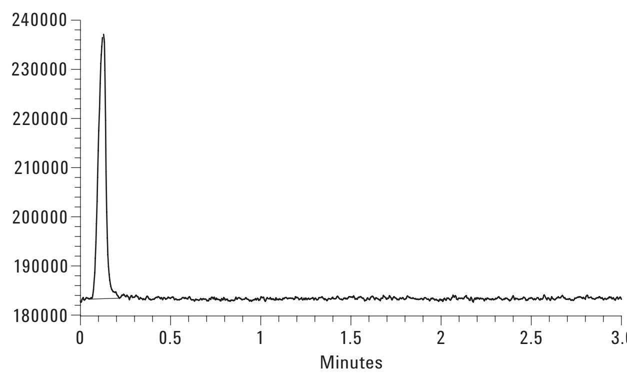

Figure 1. 1 ppm sulfur as thiophene.

Excellent sensitivity, selectivity, precision, and linearity are observed using this technique. For example, the detection limit of total sulfur in benzene is less than 100 ppb sulfur by weight.

The technique demonstrates very good precision at the 1 ppm wt level, around 2% relative standard deviation (RSD). For instance at 1 ppm wt thiophene (as sulfur) in benzene, the mean area was 157,600 area counts with a standard deviation of 3,080 (n = 5), yielding an RSD of 1.95%.

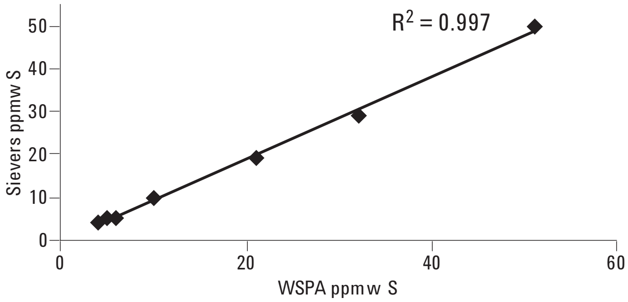

Figure 2. Relationship between the Agilent Model 355 SCD and WSPA results for total sulfur in gasoline.

Both linearity and accuracy are reflected in the results from comparing sulfur analyses obtained by this technique to the results determined by Western States Petroleum Association (WSPA) for seven gasoline samples. This is shown in Figure 2.

Relationship Between Sulfur Results by Laboratory

Chemiluminescent TSA and WSPA results calibration in this analytical technique is achieved through the use of appropriately generated standards. One should match the sample and standard matrices as closely as possible. For example, use a benzene standard matrix when measuring sulfur in a benzene sample. Since all sulfur compounds produce the same response, use of a single sulfur compound standard is sufficient for calibration. Please note that nonvolatile sulfur compounds will not be detected since they will remain in the injection port. Analyzer linearity is greater than three orders of magnitude, so generation of multiple calibration points may not be necessary. In summary, this technique is simple, accurate, and precise.

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Printed in the USA
June 26, 2007
5989-6792EN