Agilent Model 355 Sulfur Chemiluminescence Detector (SCD): Sulfur Compounds in Peppermint Oil

Technical Overview

Introduction

The reliable measurement of sulfur compounds at low levels in flavorings is extremely important. Volatile sulfur compounds are critical for the flavor and aroma of foods; however, they are more often associated with off-flavors. In addition to sensory properties, sulfur compounds are studied for other functional properties, including use as antioxidants, antimicrobial agents, and anticarcinogens. [1]

Volatile sulfur compounds are major contributors to the flavor and odor of many foods. They often are not present in an intact cell or plant, but may be formed through enzymatic processes when plants are cut or chewed, releasing flavor precursors and enzymes from the rupturing cells. Food preparation is another source of volatile sulfur compounds, where thermal breakdown of these enzymatically produced flavorants can form hydrolysis products and other recombinants. These products are necessary for the sensory quality of flavors and fragrances at low levels, while higher levels of the same compounds result in off-flavors.

Essential oils isolated from plants are used for their odoriferous qualities as well as for flavoring enhancement. Sulfur compounds characteristically exhibit very low flavor thresholds such that even trace amounts of these compounds affect the flavor profile of foods. [2–4] The selective and sensitive detection of sulfur in food products is therefore important to verify food quality and product stability and to maintain consistent sensory properties of food and fragrance products.

Gas chromatography coupled with the Agilent Sulfur Chemiluminescence Detector (SCD) provides an efficient means to isolate and analyze volatile sulfur compounds. The Model 355 produces a linear and equimolar response to sulfur compounds without hydrocarbon quenching or interferences. The patented mechanism of operation produces one response factor for sulfur, as opposed to other detectors where individual response factors for each analyte of interest must be determined. This allows for speciated quantitative analysis of sulfur, as well as total sulfur determinations from a single standard.

The following chromatogram illustrates the ability of the SCD to speciate and quantitate sulfur compounds at levels less than 1 ppm in peppermint oil without preconcentration. Chromatographic conditions were as follows: Model 355 SCD operated according to standard conditions; 2-µL neat liquid sample split 20:1 onto a 60-m, 0.53-mm id, 5.0-µm HP-1 column. The gas chromatograph was an Agilent Technology Model 5890 Series II equipped with electronic pressure programming. The initial temperature of 30 °C was held for two minutes, followed by a ramp at 10°/minute to the final temperature of 275 °C.

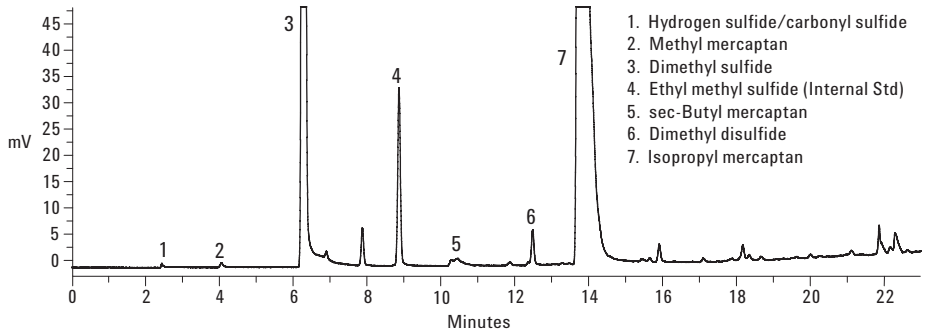

Figure 1. Sulfur chromatogram of natural oil of peppermint.

Table 1. Sulfur Concentrations of Identified Peaks

Compound	Concentration (ppb as S)
Hydrogen sulfide/carbonyl sulfide	110
Methyl mercaptan	200
Dimethyl sulfide	167,000
Ethyl methyl sulfide (Internal Standard)	5,000
sec-Butyl mercaptan	80
Dimethyl disulfide	1,080
Isopropyl mercaptan	84,100
Total	259,000

The Agilent 355 SCD is the most sensitive and selective sulfur detector commercially available with detection limits of less than 500 nanograms of sulfur per second. The equimolar response of the SCD provides for rapid quantitative analysis of samples using either an external standard, or an internal standard as illustrated above. In this case, ethyl methyl sulfide, which was not present in the sample, was chosen as an internal standard and spiked into the analyte at 5 ppm. Retention time comparison to the spiked peak allowed for peak identification and confirmation. Direct referencing of peak area to the spiked standard provided the concentration of each component, and the total area of all of the peaks corresponded to the total sulfur content.

References

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