Agilent Model 355 Sulfur Chemiluminescence Detector (SCD): Sulfur Speciation in Grapefruit Juice

Technical Overview

Introduction

The ability to measure sulfur compounds at trace levels is extremely important in the food, flavor, and beverage industries. Sulfur compounds, especially volatile ones, impact flavor and aroma both positively and negatively, although they are more often associated with off-flavors or odors. Interest concerning the measurement and speciation of sulfur compounds in the citrus industry is growing. To that end, the analysis of sulfur compounds in citrus juices, like grapefruit juice, using the Agilent Sulfur Chemiluminescence Detector (SCD) is described in this technical overview.

Sulfur-containing compounds are major contributors to the flavor and fragrance of many foods and beverages. It has been demonstrated that they may have both positive and negative impacts depending on sulfur compound type and concentration. This is true for citrus juices, especially for grapefruit juice, which contains numerous sulfur compounds. In fact, sulfur compounds, such as the mercaptopinanes, are responsible for the characteristic grapefruit fragrance at low concentration levels. At high levels, these compounds produce an unpleasant sulfurous odor.

The concentration of various sulfur compounds in citrus juices is affected by factors such as fruit variety, ripeness, growing conditions, and handling during processing and storage. Citrus processors can control some but not all of these factors. Juice processors must deal with these variables and still produce citrus products of high quality and consistency.

Research in the field continues to paint an ever more detailed picture that provides a better understanding of relationships between the various processing parameters over which there is control. In the area of sulfur compounds, this task is complicated because concentration levels of sulfur compounds may be important at levels in the parts per billion range, while there is the potential for interference from hundreds of other compounds. A sensitive and selective method of analysis for sulfur compounds in citrus products is needed because of the low levels that affect product quality and the inherent complexity of the sample matrices. The SCD is very useful for this because of its extreme sensitivity and selectivity. It is the most sensitive and selective sulfur detector available.

The following data were kindly provided to Agilent by Professor Russell Rouseff of the University of Florida Citrus Research and Education Center. An approach for the analysis of sulfur compounds in citrus juices is described. Results comparing sulfur compounds in grapefruit juice from different times of the season are shown. This type of information is useful in refining processing strategies.

An internal standard of s-methyl thiobutanoate is added to juice samples prior to extraction with ethyl acetate. The extract is analyzed by gas chromatography using the SCD. The equimolar response of the SCD, high extraction efficiency, and known concentration of the internal standard are used to quantitate the levels of sulfur compounds in the juice samples. External standards of known sulfur compounds are used for identification, sometimes in conjunction with mass spectrometry. Overall precision of the extraction, injection, and reproducibility of the SCD detector is excellent.

Figure 1 illustrates differences in sulfur levels related to grapefruit juices of different maturity; however, all three are from legally mature fruit.

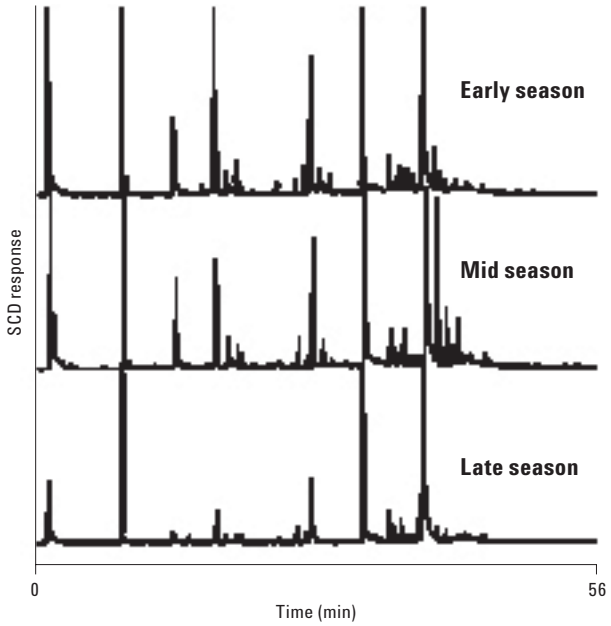

Figure 1. Sulfur chromatograms of legally mature grapefruit juices from different season intervals.

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