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Application News

No. G305B

Gas Chromatography

Performance Assessment of Nexis™ SCD-2030 Using Sulfur Compounds Recommended by ASTM D5623

ASTM D5623 provides guidelines for the analysis of sulfur-containing compounds in light petroleum liquids by gas chromatography with a sulfur chemiluminescence detector (SCD). ASTM D5623 applies to petroleum products with a final boiling point of less than 230 °C at standard atmospheric pressure, such as gasoline.

The SCD provides high selectivity and linearity for sulfur-containing compounds and minimizes the quenching effects of hydrocarbons during sample analysis of complex hydrocarbon matrices.

This document introduces the results of using Nexis SCD-2030 to analyze sulfur compounds standards recommended by ASTM D5623.

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Fig. 1 Nexis™ SCD-2030

Analytical Conditions

Table 1 shows instrument configuration details and analytical conditions used for all evaluations.

In order to prevent adsorption of sulfur compounds at low concentrations, a sample vaporization injector SPL-2030 (P/N: S221-77100-61/-64) was subjected to a deactivation treatment and installed on the system.

LabSolutions GC was used for instrument control and data collection.

Table 1 Instrument Configuration and Analytical Conditions

Main Unit	: Nexis GC-2030 /AOC-20i plus
Column	: SH-I-1MS (30 m × 0.32 mm I.D. df= 4 µm) *1
Detector	: SCD-2030
Injection Volume	: 1 µL
Injection Mode	: Split
Split Ratio	: 1:9
Injection Unit Temp	: 275 °C
Carrier Gas	: He
Carrier Gas Control	: Constant Column Flow Mode (2.8 mL/min)
Column Temp.	: 40 °C (3 min) - 10 °C/min – 250 °C (16 min)
Interface Temp.	: 200 °C
Furnace Temp.	: 850 °C
Detector Gas	: H_2 100 mL/min, N_2 10 mL/min, O_2 12 mL/min, O_3 25 mL/min

*1 P/N: 227-36011-01

Analysis of Sulfur Calibration Standard

Nineteen discrete sulfur compounds recommended by ASTM D5623 were separated into two groups for optimal chromatogram separation. Ten out of the nineteen compounds were diluted in n-hexane to yield standard 1 (STD1) and the remaining nine sulfur compounds were diluted in toluene to yield standard 2 (STD2). Diphenyl sulfide was added to each STD at a concentration of 1 ppm(w/w) as an internal standard. Chromatograms of the STD1 and STD2 are shown in Fig. 2.

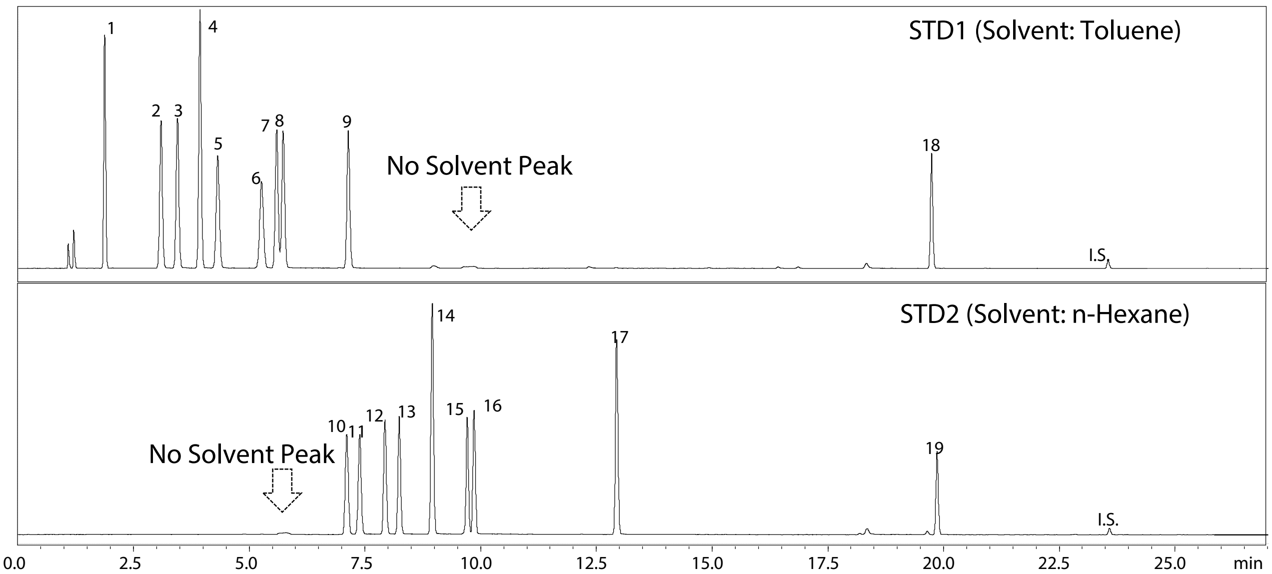

Fig. 2 Chromatograms of 19 Sulfur Compounds (10 ppm)

Repeatability and Linearity Evaluation

Nineteen discrete sulfur compounds were diluted to the following concentrations 0.1 ppm, 1 ppm, 10 ppm, and 100 ppm (w/w) according to ASTM D5623. Table 2 illustrates repeatability (%RSD, n=5) and coefficient of determination (R^2) for the Nexis SCD-2030 analysis of the sulfur compound standards. The area ratio value of the target compound to the internal standard was used in this evaluation. The results were highly reproducible with excellent linear response for all compounds. Fig. 3 illustrates the calibration curves for 2-propanethiol and methyl disulfide which are representative of the analytes.

Table 2 Repeatability and Linearity of Sulfur Compounds

No.	Analytes	0.1 ppm	1 ppm	10 ppm	100 ppm	STD No	R^2 (0.1-100 ppm)
1	Methyl mercaptan	7.1 %	1.8 %	5.1 %	3.0 %	1	0.9999
2	Ethyl mercaptan	6.5 %	1.5 %	3.7 %	2.3 %	1	0.9999
3	Dimethyl sulfide	6.8 %	1.3 %	3.7 %	2.4 %	1	0.9999
4	Carbon disulfide	7.3 %	1.9 %	4.4 %	2.7 %	1	0.9999
5	2-Propanethiol	6.7 %	1.4 %	2.9 %	2.0 %	1	0.9999
6	2-Methyl-2-propanethiol	9.7 %	2.8 %	2.5 %	1.9 %	1	0.9999
7	1-Propanethiol	6.3 %	1.4 %	2.9 %	1.9 %	1	0.9999
8	Ethyl methyl sulfide	7.7 %	1.8 %	2.8 %	2.2 %	1	0.9999
9	Thiophene	6.7 %	1.7 %	2.8 %	2.0 %	1	0.9999
10	2-Butanethiol	8.5 %	3.4 %	1.7 %	2.4 %	2	0.9999
11	2-Methyl-1-propanethiol	8.5 %	3.1 %	1.7 %	2.3 %	2	0.9999
12	Diethyl sulfide	4.8 %	2.9 %	1.9 %	2.3 %	2	0.9999
13	1-Butanethiol	9.1 %	3.8 %	1.8 %	2.4 %	2	0.9999
14	Methyl disulfide	1.9 %	2.6 %	2.1 %	2.3 %	2	1.0000
15	2-Methylthiophene	9.7 %	3.2 %	1.8 %	2.3 %	2	0.9999
16	3-Methylthiophene	6.0 %	2.8 %	1.5 %	2.4 %	2	0.9999
17	Diethyl disulfide	3.7 %	2.8 %	1.8 %	2.4 %	2	0.9999
18	5-Methylbenzothiophene	9.4 %	1.5 %	1.9 %	1.8 %	1	0.9999
19	3-Methylbenzothiophene	9.6 %	4.1 %	1.7 %	2.5 %	2	0.9999

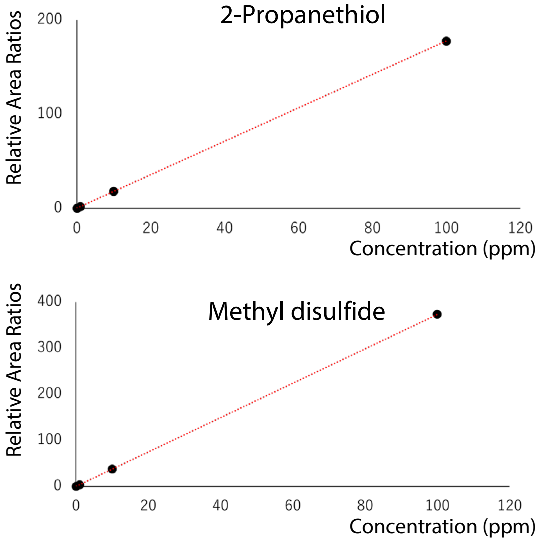

Fig. 3 Calibration Curve for 2-Propanethiol and Methyl Disulfide

Long-Term Stability Evaluation

Long-term stability of a detector is one key measure of performance that directly impacts data reliability. The Nexis SCD-2030 is configured with an industry-first horizontal redox cell, which promotes the complete oxidation-reduction reaction of samples by ensuring sufficient reaction area and reaction time in the cell to achieve stable analysis.

A 10 ppm (w/w sulfur) standard 3 (STD3), containing all nineteen sulfur compounds in Table2, was analyzed for sixteen days continuously. Diphenyl sulfide was added to the sample at a concentration of 1 ppm as an internal standard.

Fig. 4 illustrates the results of relative response factor stability and absolute area stability for the four compounds (1-butanethiol, methyl disulfide, diethyl disulfide, 3-methylbenzothiophene) representing boiling points raining from 98 °C to 273 °C. Each plot represents the daily mean value of the analysis result, and the error bar represents the value of three standard deviations (3σ).

The relative standard deviations of the relative response factors and absolute area for 1-butanethiol, methyl disulfide, diethyl disulfide and 3-methylbenzothiophene are shown in Table 3.

The results of this evaluation showed excellent long-term stability and diurnal variation. The Nexis SCD-2030 not only provides a stable relative response but also a stable absolute response over time.

Table 3 Long-Term Stability for Representative Analytes

Analytes	1-Butanethiol	Methyl disulfide	Diethyl disulfide	3-Methyl-benzothiophene
Response Factor RSD(%)	1.4	1.2	1.9	1.6
Absolute Area RSD(%)	3.5	3.6	3.8	3.4

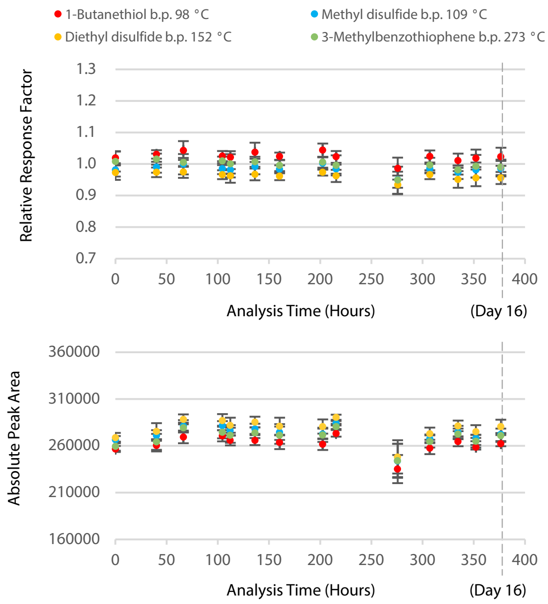

Fig. 4 Relative Response Factors and Absolute Peak Area Over 16 Days

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