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Application News

No. G340A

Gas Chromatograph Nexis SCD-2030

Examination of Analysis of the Total Sulfur Content Using Nexis™ SCD-2030

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User Benefits

- Nexis SCD-2030 can easily quantify the total sulfur content in a sample without column separation.
- It can quantify the total sulfur content in a sample using a calibration curve created from a sulfur compound which concentration is already known.
- It can be applied to monitoring the level of sulfur compounds in a sample.

■ Introduction

The Sulfur Chemiluminescence Detector (SCD) is a selective and highly sensitive GC detector for sulfur compounds, and has the following characteristics:

- No peaks will be detected in principle when samples with no sulfur compound content are introduced into the SCD
- The SCD responds linearly in proportion to the number of sulfur atoms (S atoms) introduced into the detector. Thus, it displays the same sensitivity (i.e. equimolar sensitivity) regardless of compound species provided the same number of S atoms (i.e. S mol number) is introduced.

These characteristics suggest that the total sulfur content can be quantified more easily by omitting column separation step when a sample is introduced into the SCD.

This article examined the feasibility of the quantification of the total sulfur content in a sample using the SCD by comparing the results of the analysis with and without column separation.

■ Sample Preparation, Analytical Methods and Conditions

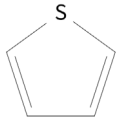
To examine the analysis of the total sulfur content, the standards for the calibration curve were prepared by diluting thiophene with hexane. Trial samples postulated as having an unknown sulfur content were prepared by mixing three types of sulfur compounds, S-methyl thioacetate, diisopropyl sulfide, and dimethyl trisulfide, and then diluting with hexane. Their specific concentrations are shown in Figs. 1 and 2. These samples were analyzed using the detector with column separation, and subsequently they were analyzed without column separation by changing the column to a deactivated tubing. The analytical conditions are shown in Tables 1 and 2. The analytical methods are as follows:

[Quantification by analysis with column separation (analytical conditions in Table 1)]

(1) The calibration curve is created by analyzing the standards.
(2) Trial samples postulated as having an unknown sulfur content are analyzed.
(3) Each of the sulfur compounds in the "unknown" samples is quantified using the calibration curve created in (1).
(4) The total sulfur content is calculated by summing the values of all the sulfur compounds quantified in (3).

[Quantification by analysis without column separation (analysis conditions in Table 2)]

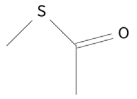
(1) The calibration curve is created by analyzing the standards.
(2) Trial samples postulated as having an unknown sulfur content are analyzed.
(3) The "unknown" samples analyzed in (2) are quantified using the calibration curve in (1). The quantified values are the total sulfur content in those samples.



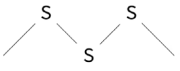
Thiophene
No. of S atoms: 1

Standards for the Calibration Curve	The Sulfur Content* (M)
1	1.19×10^{-3}
2	1.19×10^{-4}
3	1.19×10^{-5}

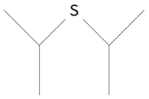
Fig. 1 The Compound Structures of the Standards for the Calibration Curve and their Sulfur Content after Preparation (* Molar Concentration of S)



S-methyl thioacetate
No. of S atoms: 1



dimethyl trisulfide
No. of S atoms: 3



diisopropyl sulfide
No. of S atoms: 1

Samples for Examination	Total Sulfur Content* (M)
A	4.33×10^{-4}
B	4.33×10^{-5}

Fig. 2 Sulfur Compound Structures and Total Sulfur Content in the Unknown Samples for Examination (*Total Molar Concentrations of S)

Table 1 Conditions for Analysis with Column Separation

Model	:	Nexis GC-2030 (SPL) / SCD-2030
Injection Volume	:	0.5 µL
Injection Temp	:	220 °C
Injection Mode	:	Split (Split Ratio 1:30)
Carrier Gas	:	He
Carrier Gas Control	:	Linear velocity (30.0 cm/s)
Column	:	SH-1 (30 m × 0.25 mm I.D., 0.25 µm) *1
Column Temp.	:	50 °C (3.5 min) - 30 °C/min – 200 °C
Interface Temp.	:	200 °C
Electric Furnace Temp.	:	850 °C
Detector Gas	:	H_2 100.0 mL/min N_2 10.0 mL/min O_2 12.0 mL/min O_3 25.0 mL/min

*1 P/N: 221-75719-30

Table 2 Conditions for Analysis without Column Separation

Model	:	Nexis GC-2030 (SPL) / SCD-2030
Injection Volume	:	0.5 µL
Injection Temp	:	220 °C
Injection Mode	:	Split (Split Ratio 1:50)
Carrier Gas	:	He
Carrier Gas Control	:	Pressure (10 kPa)
Column	:	Deactivated fused silica tubing (15 m × 0.2 mm I.D.)
Column Temp.	:	200 °C (Held for 10 min *1)
Interface Temp.	:	200 °C
Electric Furnace Temp.	:	850 °C
Detector Gas	:	H_2 100.0 mL/min N_2 10.0 mL/min O_2 12.0 mL/min O_3 25.0 mL/min

*1 An analysis interval of about 10 minutes is required for stable analysis.

■ Chromatograms of the Standards and the Calibration Curve

Chromatograms of the standards and the calibration curve created from the analysis with separation are shown in Fig. 3, and those from the analysis without separation are shown in Fig. 4. The calibration curve is created with the sulfur content in thiophene as the abscissa and the area as the ordinate.

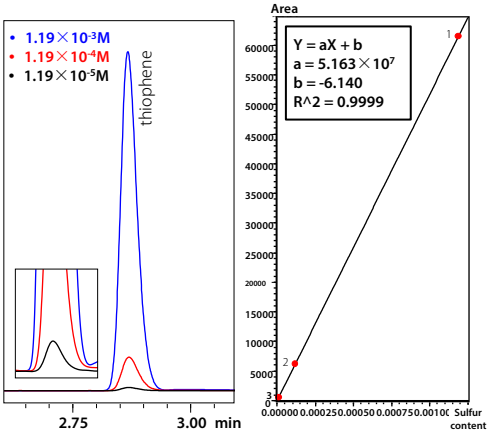


Fig. 3 Chromatograms and the Calibration Curve from the Analysis with Separation

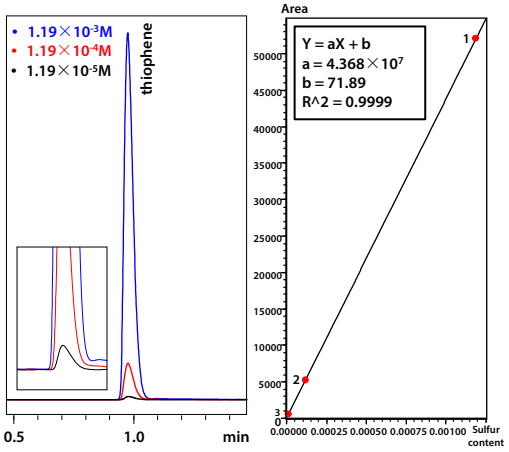


Fig. 4 Chromatograms and the Calibration Curve from the Analysis without Separation

■ Chromatogram of Unknown Samples and Results of Quantification

Chromatogram of "unknown" sample B (total sulfur content: 4.33 x 10^{-5} M) with separation is shown in Fig. 5, and that without separation is shown in Fig. 6.

The total sulfur content in the "unknown" samples analyzed with separation was obtained by summing the sulfur content of each of the sulfur compounds in the samples using the calibration curve created with thiophene. The total sulfur content in the "unknown" samples analyzed without separation was quantified using the calibration curve created with thiophene. Each of the sulfur compounds in the "unknown" samples cannot be quantified individually because the analysis was performed without separation.

Results of quantification of the total sulfur content are shown in Table 3. As shown in the Table.3, the values of the total sulfur content obtained from the analysis with and without separation were almost equal. The quantified values of the total content were also equal to their theoretical values.

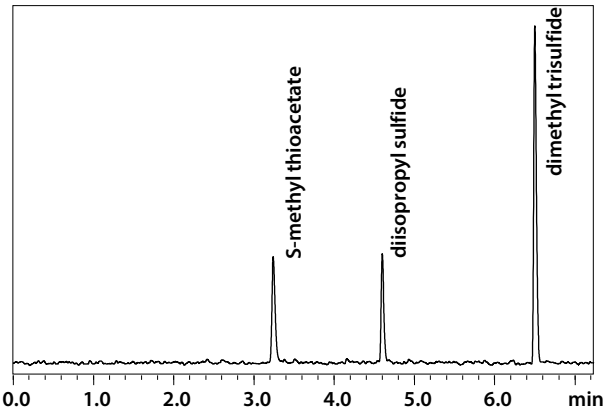


Fig. 5 Chromatogram of the Unknown Sample B Obtained from the Analysis with Separation

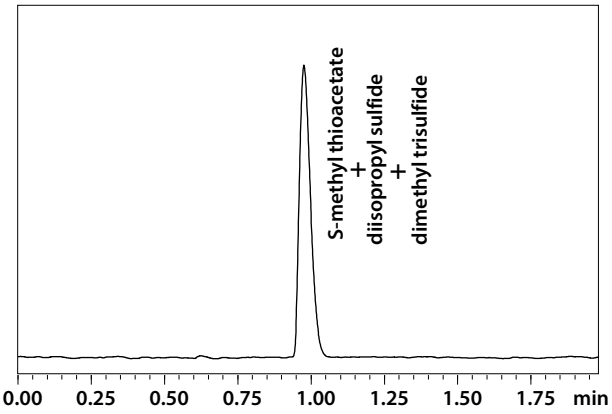


Fig. 6 Chromatogram of the Unknown Sample B Obtained from the Analysis without Separation

Table 3 Results of Quantification of the Total Sulfur Content (M)

	Unknown Sample A for Examination			Unknown Sample B for Examination		
Compound	Theoretical Sulfur Content	Quantified Content with Separation	Quantified Content without Separation	Theoretical Sulfur Content	Quantified Content with Separation	Quantified Content without Separation
S-methyl thioacetate	1.11×10^{-4}	1.01×10^{-4}		1.11×10^{-5}	9.31×10^{-6}	
diisopropyl sulfide	8.46×10^{-5}	9.26×10^{-5}		8.46×10^{-6}	8.34×10^{-6}	
dimethyl trisulfide	2.38×10^{-4}	2.67×10^{-4}		2.38×10^{-5}	2.44×10^{-5}	
Total Sulfur Content	4.33×10^{-4}	4.61×10^{-4}	4.74×10^{-4}	4.33×10^{-5}	4.21×10^{-5}	4.23×10^{-5}

■ Conclusion

Analysis of the total sulfur content in samples was examined by taking advantage of the characteristics of the SCD. The total sulfur content was easily quantified without separation using deactivated tubing, which provided one peak as the total sulfur level. Further, the total content quantified by analysis without separation was found to be equal to that quantified by analysis with separation, a typical method of SCD. Using the characteristics of Nexis SCD-2030, comparison and quantification of the total sulfur content in samples can be easily performed through the analysis under appropriate conditions. This detector can be used for simple analysis of the total sulfur content in samples including petroleum or chemical products, resins, and foods.

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