

# Application News

## No. M289

### Gas Chromatography

## New Approach to Food Smell Analysis Using Combination of GCMS™ and GC-SCD (2)

Gas chromatography mass spectrometry (GCMS), which has outstanding qualitative analysis capabilities, is used in smell analyses of food products, but because sulfur compounds contribute to the smell of food, even when present in trace amounts, detection by GCMS is difficult in some cases due to insufficient sensitivity and separation of sulfur compounds from other components. In this experiment, we studied a new approach to analysis of food smell by using a combination of GCMS, which is particularly effective for comprehensive qualitative analysis of odor components, and a GC device equipped with a sulfur chemiluminescence detector (GC-SCD), which enables selective and highly sensitive detection of only sulfur compounds.

As the study method, using kimchi (Korean pickles), which has a particular strong smell among food products as the sample, we conducted an analysis of the odor components remaining in a container, in which kimchi had been stored, after washing the container with water.

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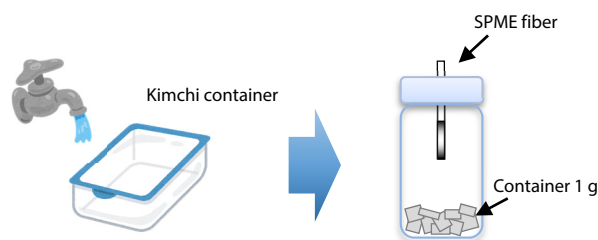


Appearance of GCMS-QP™2020 NX + Nexis™ SCD-2030 + AOC-6000

### Sample and Analysis Method

Using commercially-available kimchi as the sample material, a sample was prepared by the following procedure.

- (1) Wash a container, in which kimchi had been stored, with water and then cut the container into small pieces.
- (2) Measure 1 g of the pieces prepared in (1), introduce into a 20 mL screw vial bottle, and immediately seal with the screw cap.
- (3) Heat the sample in (2) at 80 °C for 30 min, then concentrate the headspace part to solid-phase microextraction fiber (SPME fiber; DVB/CAR/PDMS, SUPELCO).



Wash kimchi container with water

SPME extraction

### Analysis Conditions

Table 1 shows the composition of the GC instrument and the analysis conditions used in this experiment. Table 2 shows the composition of the GCMS instrument and the analysis conditions.

**Table 1 Composition of GC Instrument and Analysis Conditions**

Model	: Nexis GC-2030/SCD-2030
Sample Desorb Time	: 2 min (vaporizing chamber temperature: 250 °C)
Injection	: SPL
Injection Temp.	: 250 °C
Injection Mode	: Split
Split Ratio	: 1:5
Carrier Gas	: He
Carrier Gas Control	: Constant pressure (44.5 kPa)
Column	: InertCap 5MS/Sil (30 m×0.32 mm I.D., 0.50 μm)
Column Temp.	: 50 °C (5 min) – 10 °C/min – 250 °C (10 min)
Detector	: Sulfur chemiluminescence detector (SCD)
Interface Temp.	: 200 °C
Electric Furnace Temp.	: 850 °C
Detector Gas	: H <sub>2</sub> 80.0 mL/min N <sub>2</sub> 40.0 mL/min O <sub>2</sub> 10.0 mL/min O <sub>3</sub> 25.0 mL/min

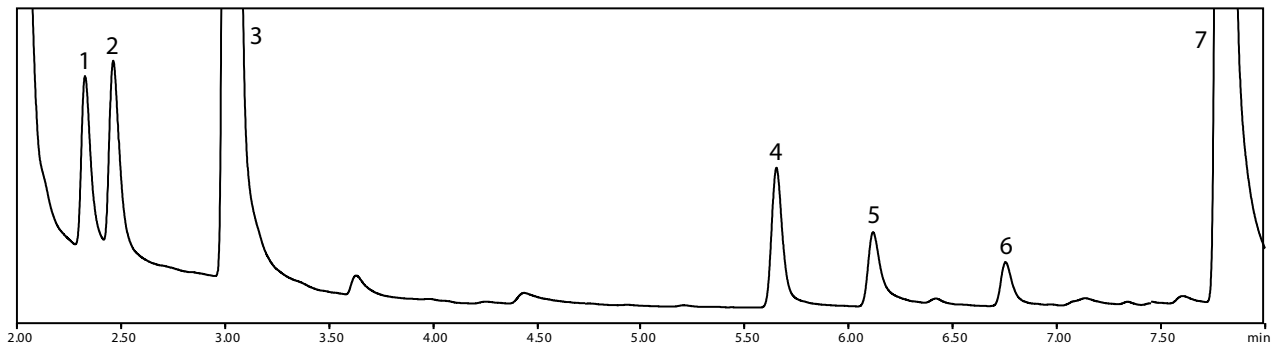
**Table 2 Composition of GCMS Instrument and Analysis Conditions**

Model	: AOC-6000 / GCMS-QP2020 NX
<u>AOC-600</u>	
Incubation Temperature	: 80 °C
Sample Extract Time	: 30 min
Sample Desorb Time	: 2 min
<u>GC</u>	
Injection	: SPL
Injection Temperature	: 250 °C
Injection Mode	: Split
Split Ratio	: 1:5
Carrier Gas	: He
Carrier Gas Control	: Constant pressure (44.5 kPa)
Column	: InertCap 5MS/Sil (30 m×0.32 mm I.D., 0.50 μm)
Column Temp.	: 50 °C (5 min) – 10 °C/min – 250 °C (10 min)
<u>MS</u>	
Ion Source Temp.	: 200 °C
Interface Temp.	: 250 °C
Ionization Mode	: EI
Measurement Mode	: Scan
Event Time	: 0.3 s

Analysis Results

Fig. 1 to Fig. 3 show the GC-SCD chromatograms and GCMS total ion chromatograms (TIC) in the respective retention time ranges. Table 3 shows the compounds that could be identified among the detected sulfur compounds. A large number of sulfur compounds could be identified easily by using a combination of GC-SCD and GCMS.

SCD



GCMS

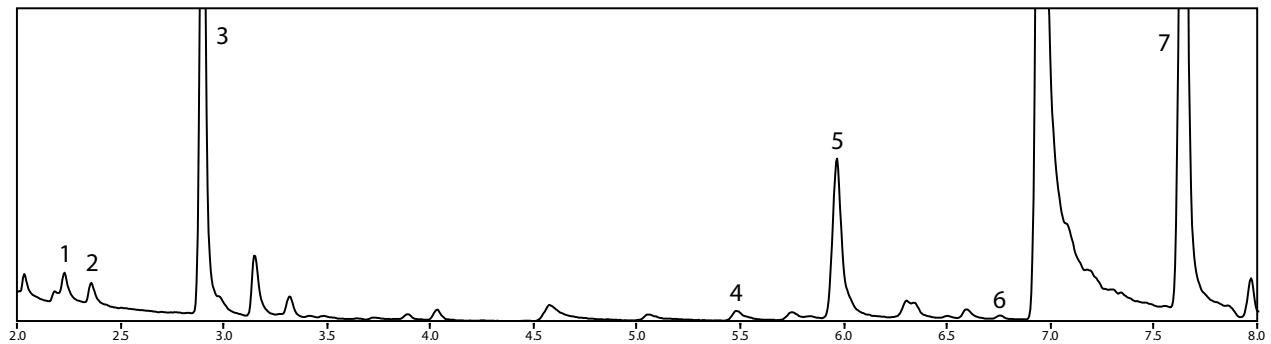
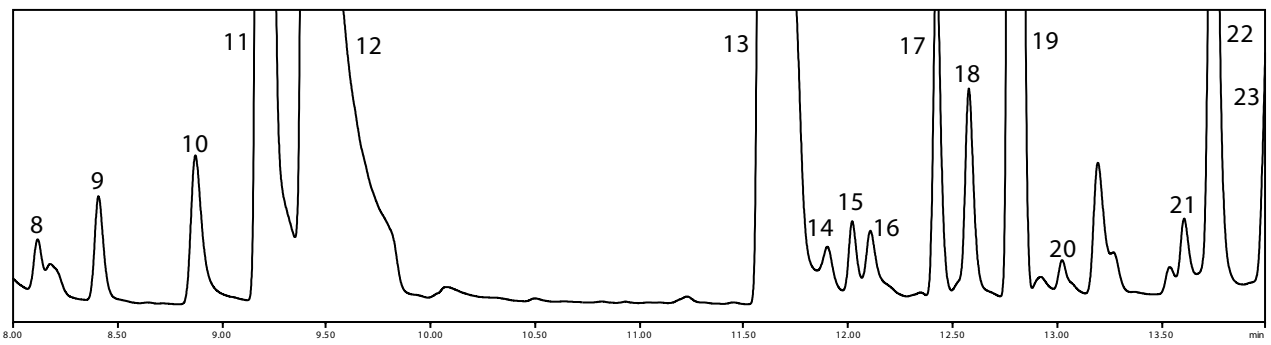


Fig. 1 SCD and GCMS Chromatograms (2 to 8 min)

SCD



GCMS

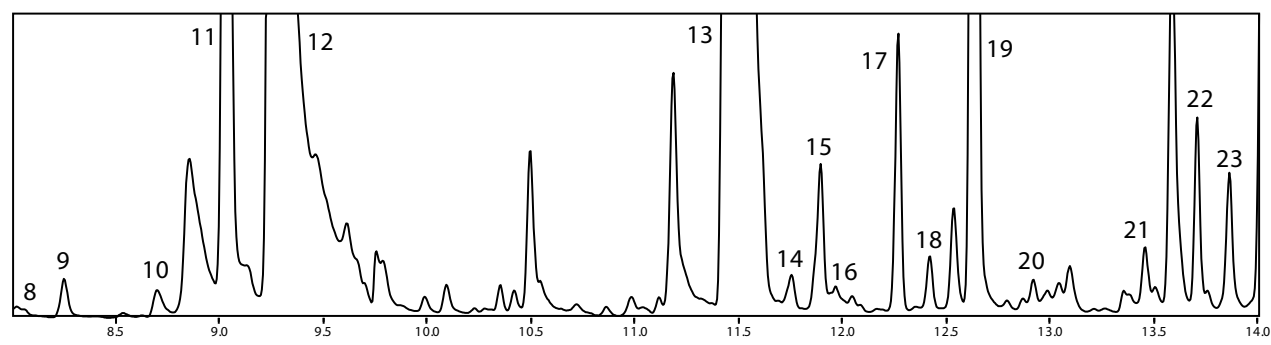
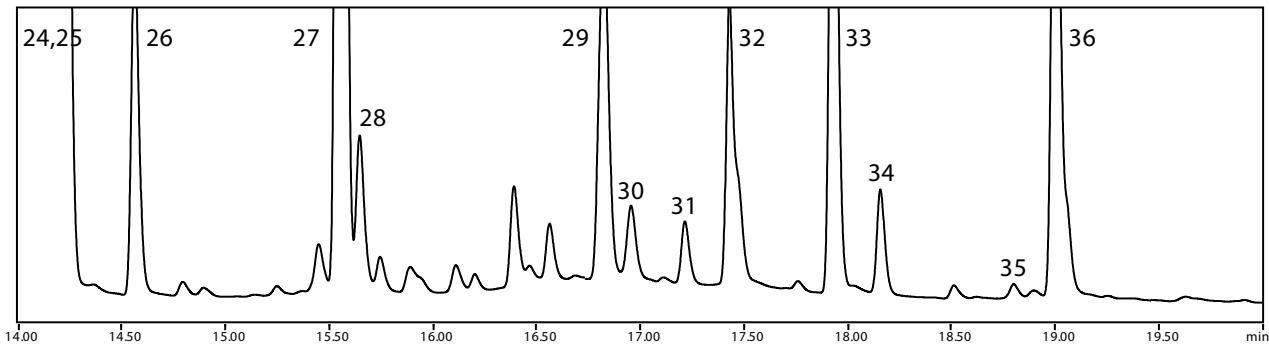


Fig. 2 SCD and GCMS Chromatograms (8 to 14 min)

GC-SCD



GCMS

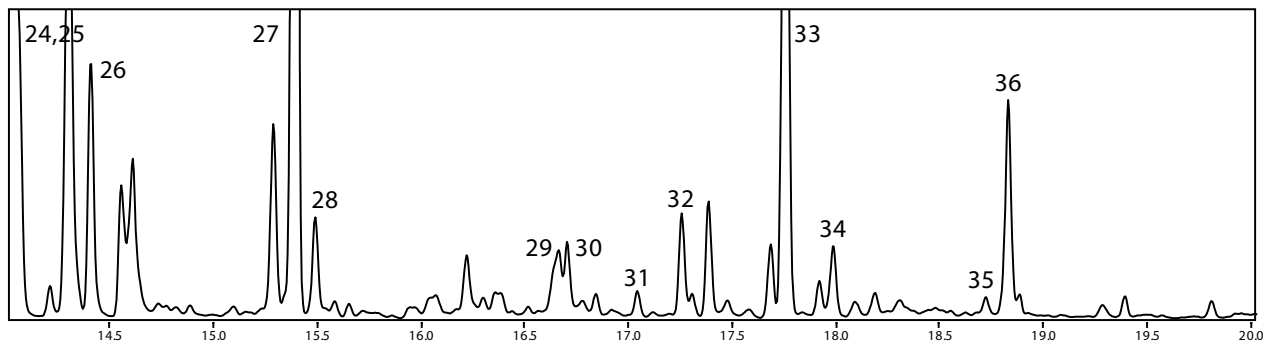


Fig. 3 SCD and GCMS Chromatograms (14 to 20 min)

Table 3 Sulfur Compounds Identified in Experiment

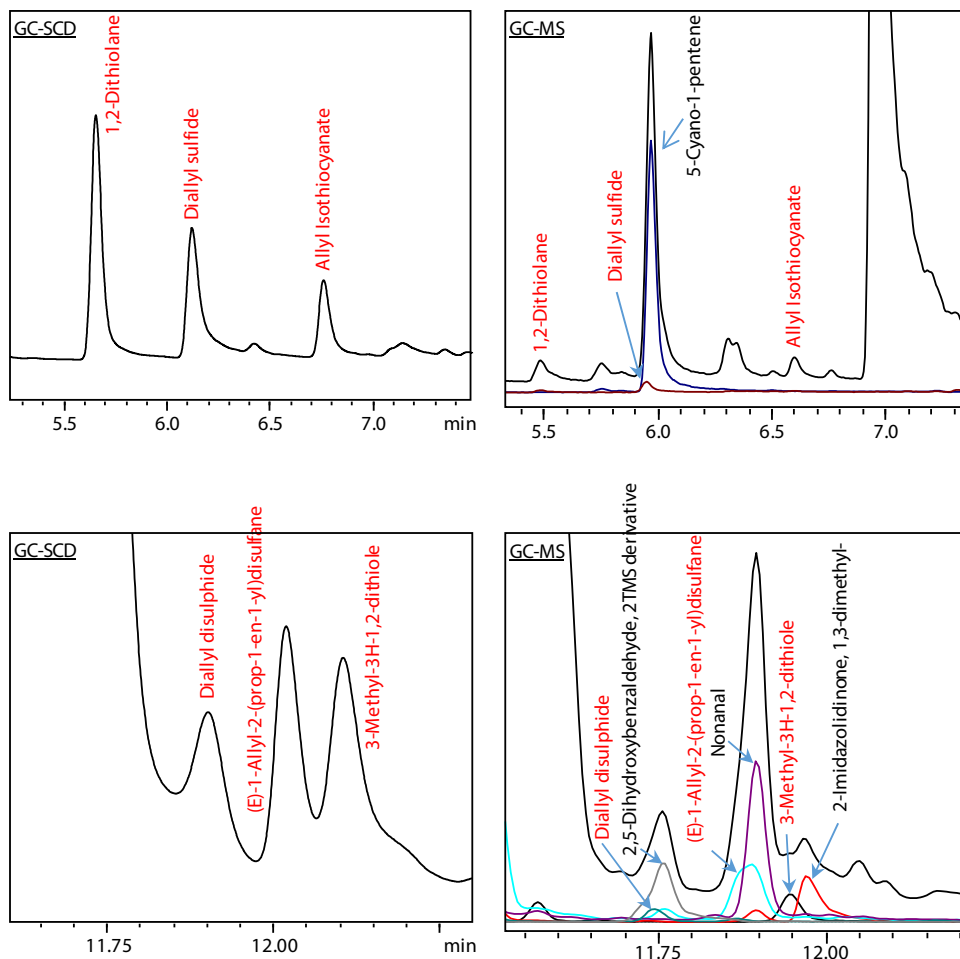
ID	Compound Name	ID	Compound Name
1	Allyl Methyl Sulfide	19	Trisulfide, methyl 2-propenyl
2	S-methyltioacetate	20	Trisulfide, methyl propyl
3	Dimethyl disulfide	21	Isothiocyanic acid, hexamethylene ester
4	1,2-Dithiolane	22	3-Vinyl-1,2-dithiacyclohex-4-ene
5	Diallyl sulfide	23	cis-Raphasatin
6	Allyl Isothiocyanate	24	2-Vinyl-4H-1,3-dithiine
7	Allyl Methyl Disulfide	25	Tetrasulfide, dimethyl
8	Butane, 2-isothiocyanato-	26	1-Cyano-4,5-epithiopentane
9	(E)-1-Methyl-2-(prop-1-en-1-yl)disulfane	27	Trisulfide, di-2-propenyl
10	3H-1,2-Dithiole	28	6-(Methylthio)hexanenitrile
11	Dimethyl trisulfide	29	5-Thiazoleethanol, 4-methyl-
12	4-Isothiocyanato-1-butene	30	Benzene, (isothiocyanatomethyl)-
13	Cyclopentyl isothiocyanate	31	trans-Raphasatin
14	Diallyl disulfide	32	Erucin
15	(E)-1-Allyl-2-(prop-1-en-1-yl)disulfane	33	Benzene, (2-isothiocyanoethyl)-
16	3-Methyl-3H-1,2-dithiole	34	Cyclopentanol, 2-mercapto-
17	Cyano-3,4-epithiobutane	35	Disulfide, methyl 1-propeny
18	Disulfide, methyl (methylthio)methyl	36	Berteroin

In the data analysis, from the GC-SCD chromatograms, it was possible to identify four species of sulfur compounds that had been overlooked by only GCMS analysis due to overlapping with the peaks of other compounds in the TIC acquired by GCMS. Table 4 shows those sulfur compounds, and Fig 4 shows the GC-SCD chromatograms and GCMS TICs.

Use of a combination of GC-SCD and GCMS enabled easy identification of sulfur compounds, and made it possible to identify sulfur compounds that had been overlooked by GCMS analysis alone.

**Table 4 Sulfur Compounds Overlapped with Other Components in GCMS Analysis**

Main peak	Sulfur compound overlapping with main peak
5-Cyano-1-pentene	Diallyl sulfide
2,5-Dihydroxybenzaldehyde, 2TMS derivative	Diallyl disulfide
Nonanal	(E)-1-Allyl-2-(prop-1-en-1-yl)disulfane
2-Imidazolidinone, 1,3-dimethyl-	3-Methyl-3H-1,2-dithiole



**Fig. 4 GC-SCD Chromatograms and GCMS TICs (Red : Sulfur Compounds)**

## Conclusion

In an analysis of the remaining smell in a kimchi container using GCMS and GC-SCD, it was possible to identify 36 species of sulfur compounds. Use of a combination of GCMS and GC-SCD enabled easy identification of sulfur compounds, and it was also possible to identify sulfur compounds which had been overlooked due to overlapping with the peaks of other components in the analysis using only GCMS.

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Gas Chromatograph



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