Excellence in Science

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

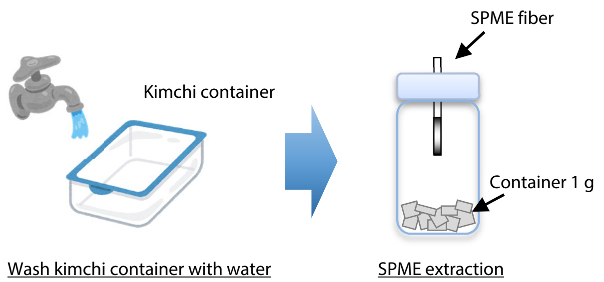
Sample and Analysis Method

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).



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_2 80.0 mL/min N_2 40.0 mL/min O_2 10.0 mL/min O_3 25.0 mL/min

Table 2 Composition of GCMS Instrument and Analysis Conditions

Model	: AOC-6000 / GCMS-QP2020 NX
AOC-600	
Incubation Temperature	: 80 °C
Sample Extract Time	: 30 min
Sample Desorb Time	: 2 min
GC	
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)
MS	
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.

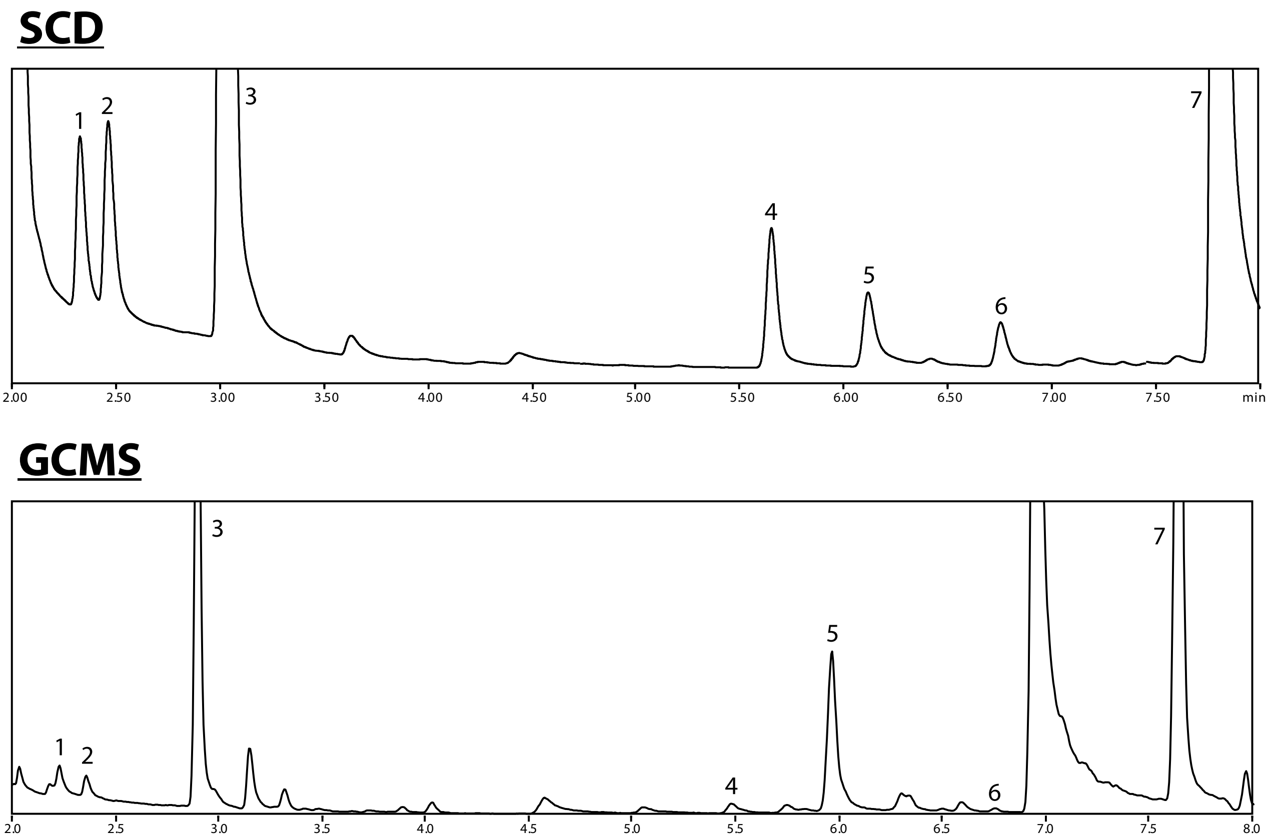


Fig. 1 SCD and GCMS Chromatograms (2 to 8 min)

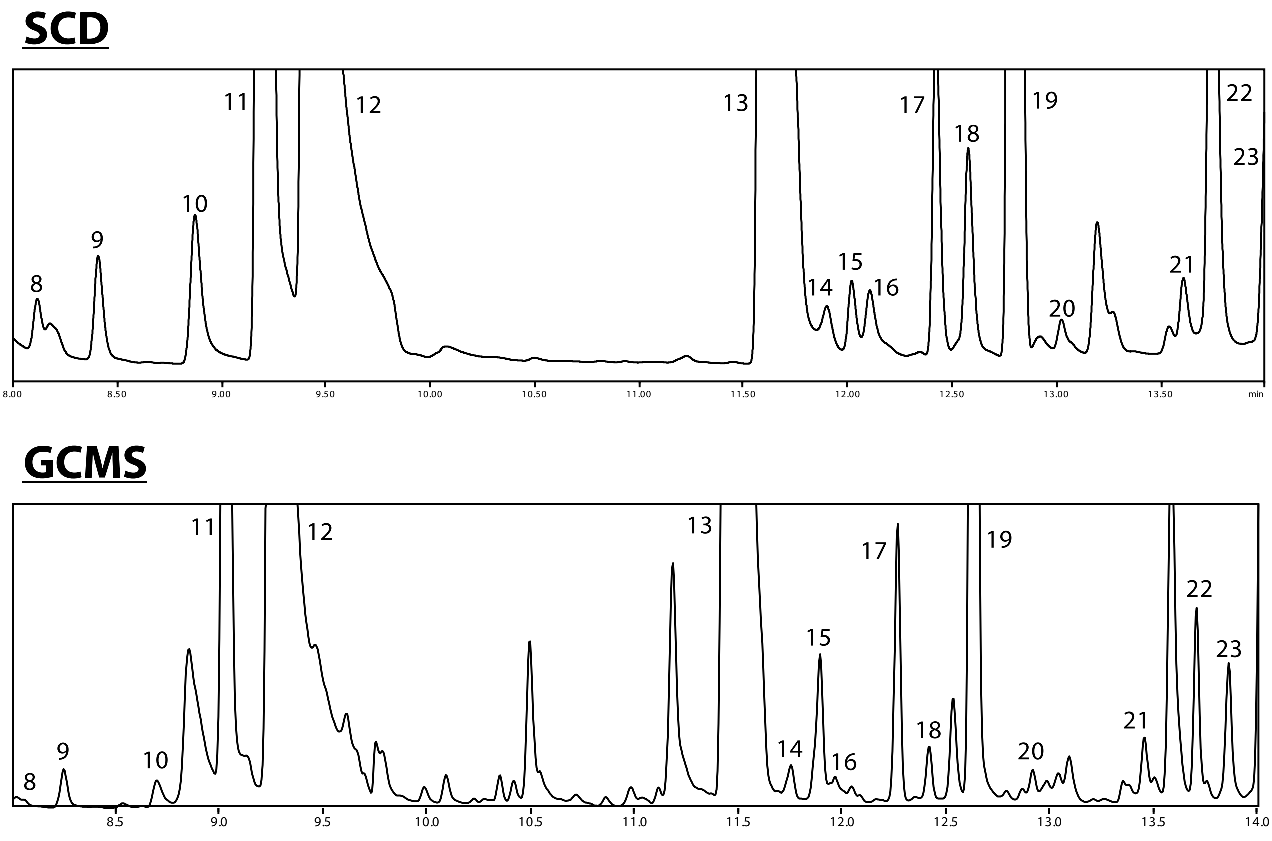
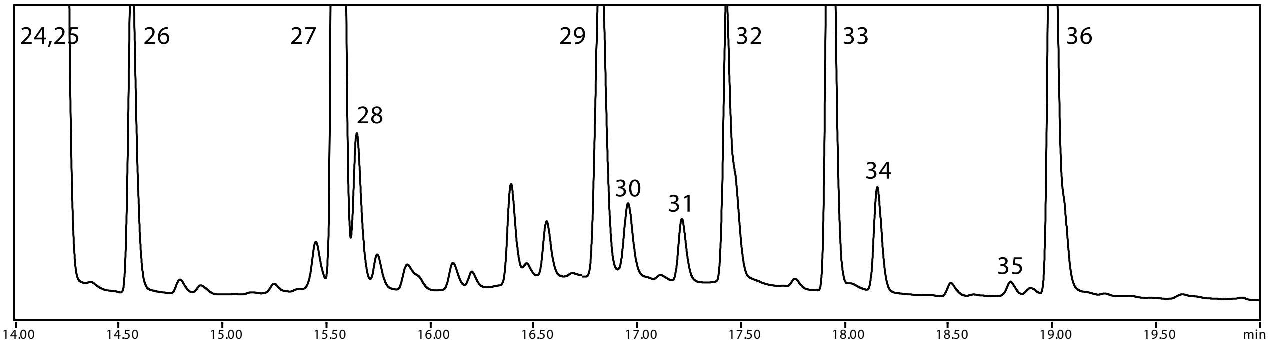


Fig. 2 SCD and GCMS Chromatograms (8 to 14 min)

GC-SCD



GCMS

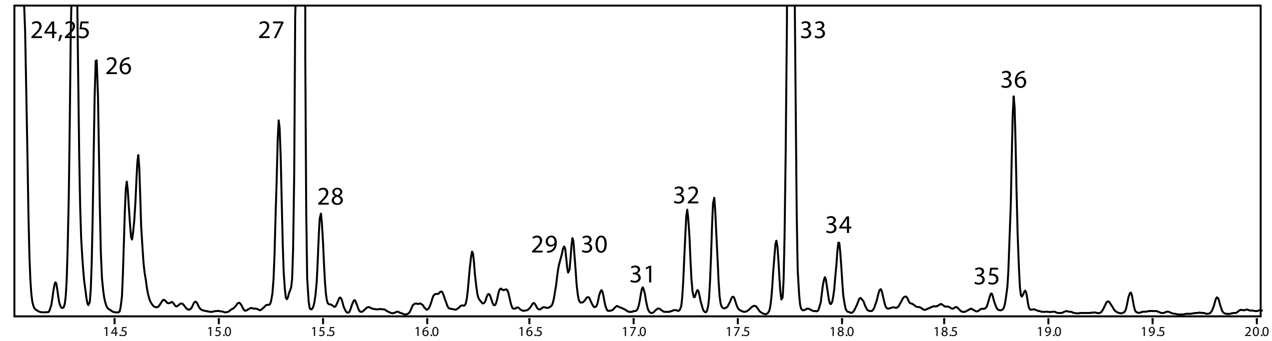


Fig. 3 SCD and GCMS Chromatograms (14 to 20 min)

Table 3 Sulfur Compounds Identified in Experiment

ID	Compound Name
1	Allyl Methyl Sulfide
2	S-methyltioacetate
3	Dimethyl disulfide
4	1,2-Dithiolane
5	Diallyl sulfide
6	Allyl Isothiocyanate
7	Allyl Methyl Disulfide
8	Butane, 2-isothiocyanato-
9	(E)-1-Methyl-2-(prop-1-en-1-yl)disulfane
10	3H-1,2-Dithiole
11	Dimethyl trisulfide
12	4-Isothiocyanato-1-butene
13	Cyclopentyl isothiocyanate
14	Diallyl disulfide
15	(E)-1-Allyl-2-(prop-1-en-1-yl)disulfane
16	3-Methyl-3H-1,2-dithiole
17	Cyano-3,4-epithiobutane
18	Disulfide, methyl (methylthio)methyl

ID	Compound Name
19	Trisulfide, methyl 2-propenyl
20	Trisulfide, methyl propyl
21	Isothiocyanic acid, hexamethylene ester
22	3-Vinyl-1,2-dithiacyclohex-4-ene
23	cis-Raphasatin
24	2-Vinyl-4H-1,3-dithiine
25	Tetrasulfide, dimethyl
26	1-Cyano-4,5-epithiopentane
27	Trisulfide, di-2-propenyl
28	6-(Methylthio)hexanenitrile
29	5-Thiazoleethanol, 4-methyl-
30	Benzene, (isothiocyanatomethyl)-
31	trans-Raphasatin
32	Erucin
33	Benzene, (2-isothiocyanatoethyl)-
34	Cyclopentanol, 2-mercapto-
35	Disulfide, methyl 1-propeny
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

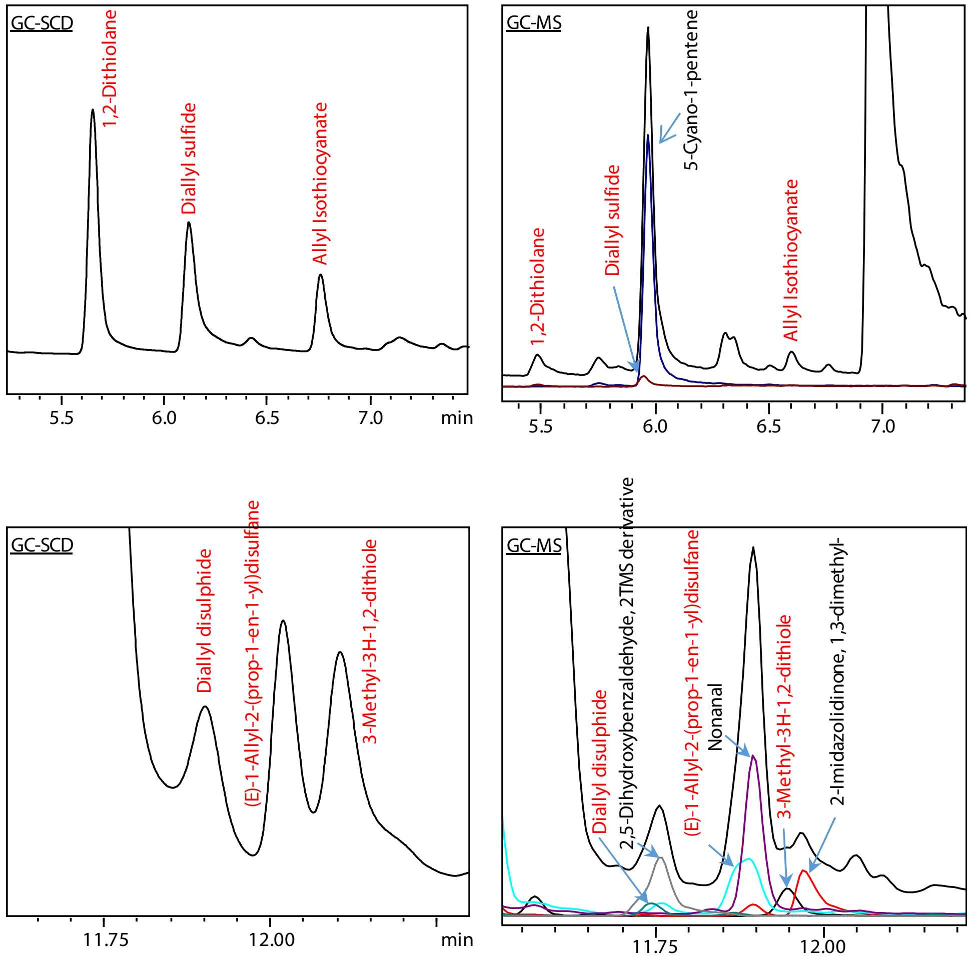


Fig. 4 GC-SCD Chromatograms and GCMS TICs (Red : Sulfur Compounds)

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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