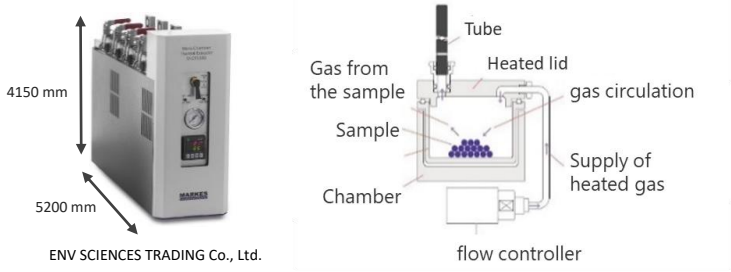
Soy Meat Aroma Components Analysis Using Microchamber and Thermal Desorption GC-MS

Product used：Mass Spectrometer (MS)

1. Introduction

One of the challenges of plant-based meat alternatives is replicating the flavor of animal-based meat, and aroma plays a significant role in that flavor. In particular, the reduction of the beany odor is a challenge in soy-based meat products, and it is important to identify the trace compounds responsible for this odor in the aroma components of the samples. The microchamber/heated extraction device is used to collect volatile organic compounds from samples heated in the chamber by a sample tube containing adsorbent. Samples can be placed directly into the chamber and a wide range of sample volumes can be set. Thermal Desorption (TD) is a pretreatment device that heats the sample tube and introduces the desorbed gas into the GC. The gas desorbed from the sample tube is trapped in a cooled focusing trap and then desorbed by rapid heating of the focusing trap. The two-stage thermal desorption system provides high analytical sensitivity. These methods are used in a wide range of fields such as food, fibers, plastics and wood products because of their ability to collect volatile compounds efficiently (MSTips No.435, 436, 459). In this report, we show the useful analysis results of soy meat aroma compounds using this method.



μ-CTE250

TD100-Xr,
JMS-Q1600GC UltraQuad™ SQ-Zeta

2. Experiment

Figure 1 shows the collection method using the microchamber. A commercial soy meat products (hamburger steak) weighing 10 g was used as the sample. The sample including all ingredients (bun, patty, sauces, pickles) on aluminum foil was placed in the microchamber (μ-CTE250, MARKES International Ltd.). The temperature of the chamber was set at 50 ℃, and Volatile compounds were collected by passing through the sample tube for 15 minutes. The measurement was performed using a GC-MS (JMS-Q1600GC, JEOL Ltd.) attached to a TD pre-treatment device (TD-Xr100, MARKES International Ltd.). The ionization methods used were electron ionization (EI) and photoionization (PI) as soft ionization (SI). The analysis of the obtained data was performed using the software "Integrated Qualitative Analysis[1)] " (hereafter referred to as msFineAnalysis iQ) from JEOL Ltd. The measurement conditions are shown in Table 1.

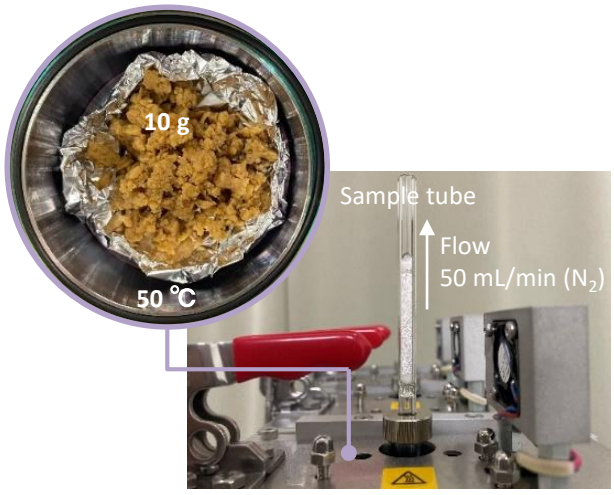


Figure 1 Sampling process

Table 1 Measurement Condition

Micro-Chamber/ Thermal Extractor	μ-CTE250 (Markes International Ltd.)
Sampling temp.	50 ℃, 15 min
Sampling flow	50 mL/min (N_2)
TD	TD100-xr (Markes International Ltd.)
Sample tube type	Tenax TA
Flow path temp	150 ℃
Dry-purge	5 min, 50 mL/min
Tube desorb	250 ℃, 10 min, 50 mL/min
Focusing trap type	U-T11GPC-2S
Trap cooling	0 ℃
Trap-purge	1 min, 50 mL/min
Trap desorb	300 ℃, 3 min
TD split	10:1 (split flow:18 mL/min)
GC	8890 GC (Agilent Technologies)
Inlet temp.	250 ℃
Column	DB-5MS (Agilent Technologies), 30 m x 0.25 mm, 0.25 μm
Oven temp.	40 ℃(1 min)-10 ℃/min-300 ℃(3 min)
Column flow	2.0 mL/min (He)
MS	JMS-Q1600GC (JEOL Ltd.)
Interface temp.	250 ℃
Ion source temp.	250 ℃
Ionization	EI (70 eV, 50 μA), PI
Scan range	*m/z* 19～500

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3. Results

3.1 TICC and Deconvolution peaks

The TICC and deconvolution peaks from the TD-GC-MS measurement are shown in Figure 2. The deconvolution detection function of msFineAnalysis iQ can identify multiple components in a peak that appears to be a single component, as well as small amounts of components that cannot be detected in the TICC. This function detected Top 50 peaks (ID:[001]-[050]) based on relative intensity. These peaks were compounds originating from hamburger steak such as dimethyl disulfide from onion, hexanal from soy, 4-terpineol from nutmeg (basic seasonings of a hamburger steak) and so on. Therefore, it was shown that TD-GC-MS enables the analysis of aroma components in food.

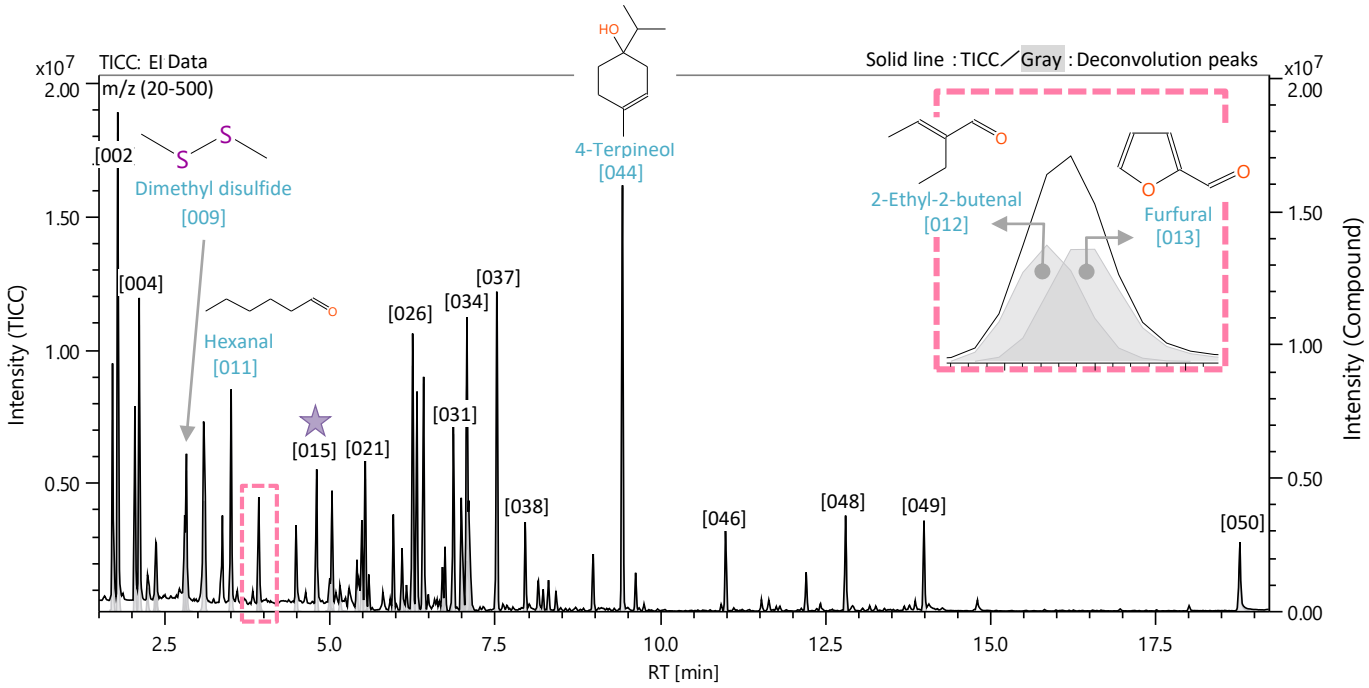

Figure 2 TIC chromatogram and Deconvolution peaks

3.2 Results of msFineAnalysis iQ

Table 2 shows the integrated analysis results of 50 peaks（ID:[001]～[050]）obtained using msFineAnalysis iQ. The msFineAnalysis iQ“ provides high quality results through integrated qualitative analysis using identification functions such as confirmation of molecular ions in EI and SI data, retention index (RI) and isotope ratio matching in addition to database searching of EI data. For example, peak [015] ★ was identified as 2-heptanone based on the integrated analysis. Figure 3 shows the mass spectrum. The mass spectrum similarity（Match factor : Max 999）is 969, indicating a good result. Molecular ion peaks were detected in the mass spectra from both EI and PI data, and it was detected as the base peak in the PI data. Furthermore, the isotopic pattern analysis results shown in Figure 4 indicate that the similarity score between the measured and theoretical values is 0.99, indicating a good result. And the difference between the measured RI value of this peak and RI value in the database（ΔRI<30 is good）is 6 [iu], indicating a good result. As a result, several aroma components suspected to be from hamburger steak were confirmed with high accuracy.

Table 2 msFineAnalysis iQ results (ID:[001]～[015])

The background color of the table ・・・Blue : highly accurate result.

General				Total Result									Spectrum Info
ID	RT [min]	Meas. RI [iu]	Height [%]	Library Name	CAS#	Match Factor	ΔRI [iu]	Formula	MW	Molecular Weight Check	Adduct/ Loss	Isotope Matching	IM Ionization
001	1.70	639	40.01	2-Butanone	78-93-3	923	41	C4 H8 O	72	✔	none	0.79	SI
002	1.78	646	100.00	Ethyl Acetate	141-78-6	922	34	C4 H8 O2	88	✔	none	0.91	SI
003	2.04	669	31.82	Butanal, 3-methyl-	590-86-3	874	17	C5 H10 O	86	✔	none	0.83	SI
004	2.11	674	59.79	Butanal, 2-methyl-	96-17-3	931	12	C5 H10 O	86	✔	none	0.99	SI
005	2.23	685	4.95	1-Penten-3-ol	616-25-1	909	2	C5 H10 O	86	✔	none	0.79	SI
006	2.35	696	9.64	Pentanal	110-62-3	852	4	C5 H10 O	86	✔	-C2H5	0.03	EI
007	2.37	697	7.61	Furan, 2-ethyl-	3208-16-0	893	6	C6 H8 O	96	✔	none	0.99	SI
008	2.80	734	16.56	2-Butenal, 2-methyl-, (E)-	497-03-0	907	8	C5 H8 O	84	✔	none	0.97	SI
009	2.82	737	29.45	Disulfide, dimethyl	624-92-0	972	9	C2 H6 S2	94	✔	none	0.99	SI
010	3.08	759	23.09	1-Pentanol	71-41-0	801	6	C5 H12 O	88	✔	-H2O	0.99	EI
011	3.50	795	44.28	Hexanal	66-25-1	883	6	C6 H12 O	100	✔	-H2O	0.73	EI
012	3.91	824	12.69	2-Ethyl-trans-2-butenal	63883-69-2	946	5	C6 H10 O	98	✔	none	0.98	SI
013	3.92	825	12.24	Furfural	98-01-1	945	8	C5 H4 O2	96	✔	none	0.89	SI
014	4.48	863	16.02	1-Hexanol	111-27-3	924	5	C6 H14 O	102	✔	-H2O	0.82	SI
015	4.79	885	27.68	2-Heptanone	110-43-0	969	6	C7 H14 O	114	✔	none	0.99	SI

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Table 2 msFineAnalysis iQ results (ID:[016]〜[050])

General				Total Result								Spectrum Info
ID	RT [min]	Meas. RI [iu]	Height [%]	Library Name	CAS#	Match Factor	ΔRI [iu]	Formula	MW	Molecular Weight Check	Adduct/ Loss	Isotope Matching	IM Ionization
016	4.99	898	4.09	Heptanal	111-71-7	848	3	C7 H14 O	114	✔	-H2O	N/A	EI
017	5.03	901	22.17	Thiophene, 2,4-dimethyl-	638-00-6	909	26	C6 H8 S	112	✔	none	0.72	SI
018	5.15	908	3.88	Pyrazine, 2,6-dimethyl-	108-50-9	842	8	C6 H8 N2	108	✔	none	0.91	SI
019	5.40	924	9.17	Bicyclo[3.1.0]hex-2-ene, 2-methyl-5-(1-methylethyl)-	2867-05-2	923	5	C10 H16	136	✔	none	0.97	SI
020	5.48	929	13.57	Disulfide, methyl propyl	2179-60-4	791	3	C4 H10 S2	122	✔	none	0.98	SI
021	5.53	932	27.69	α-Pinene	80-56-8	922	5	C10 H16	136	✔	none	0.95	SI
022	5.58	935	5.91	(E)-1-Methyl-2-(prop-1-en-1-yl)disulfane	23838-19-9	876	5	C4 H8 S2	120	✔	none	0.74	SI
023	5.95	959	19.26	Benzaldehyde	100-52-7	925	3	C7 H6 O	106	✔	none	0.98	SI
024	6.09	967	11.99	Dimethyl trisulfide	3658-80-8	938	4	C2 H6 S3	126	✔	none	0.96	SI
025	6.16	971	3.96	Bicyclo[3.1.0]hexane, 4-methylene-1-(1-methylethyl)-	3387-41-5	881	3	C10 H16	136	✔	none	0.98	SI
026	6.25	977	51.32	β-Pinene	127-91-3	912	2	C10 H16	136	✔	none	0.99	SI
027	6.31	981	40.05	5-Hepten-2-one, 6-methyl-	110-93-0	922	5	C8 H14 O	126	✔	none	0.96	SI
028	6.41	987	47.62	Furan, 2-pentyl-	3777-69-3	855	6	C9 H14 O	138	✔	none	0.99	SI
029	6.70	1005	4.83	α-Phellandrene	99-83-2	812	0	C10 H16	136	✔	none	0.90	SI
030	6.74	1007	11.16	β-Ocimene	13877-91-3	923	30	C10 H16	136	✔	none	0.94	SI
031	6.86	1015	37.05	(+)-4-Carene	29050-33-7	889	6	C10 H16	136	✔	none	1.00	SI
032	6.98	1023	21.06	o-Cymene	527-84-4	919	1	C10 H14	134	✔	none	0.93	SI
033	7.01	1025	8.48	1-Hexanol, 2-ethyl-	104-76-7	866	5	C8 H18 O	130	✔	-H2O	0.95	EI
034	7.07	1028	47.92	Limonene	138-86-3	876	2	C10 H16	136	✔	none	0.92	SI
035	7.10	1030	14.31	β-Phellandrene	555-10-2	746	1	C10 H16	136	✔	none	0.93	SI
036	7.12	1032	5.16	Eucalyptol	470-82-6	775	0	C10 H18 O	154	✔	none	0.92	SI
037	7.52	1057	65.61	γ-Terpinene	99-85-4	910	3	C10 H16	136	✔	none	0.99	SI
038	7.95	1084	17.70	Cyclohexene, 3-methyl-6-(1-methylethylidene)-	586-63-0	899	2	C10 H16	136	✔	none	0.98	SI
039	8.15	1097	5.34	Linalool	78-70-6	922	2	C10 H18 O	154	✔	-H2O	0.89	SI
040	8.22	1101	3.93	Nonanal	124-19-6	871	3	C9 H18 O	142	✔	-H2O	N/A	EI
041	8.30	1107	6.20	Disulfide, dipropyl	629-19-6	804	0	C6 H14 S2	150	✔	none	0.99	SI
042	8.42	1114	4.09	(E)-1-(Prop-1-en-1-yl)-2-propyldisulfane	23838-21-3	878	4	C6 H12 S2	148	✔	none	0.95	SI
043	8.98	1152	11.17	Trisulfide, methyl propyl	17619-36-2	937	2	C4 H10 S3	154	✔	none	0.99	SI
044	9.42	1181	88.46	Terpinen-4-ol	562-74-3	936	4	C10 H18 O	154	✔	none	0.99	SI
045	9.62	1194	7.77	L-α-Terpineol	10482-56-1	938	4	C10 H18 O	154	✔	-H2O	0.94	SI
046	10.98	1289	16.51	Safrole	94-59-7	947	2	C10 H10 O2	162	✔	none	0.98	SI
047	12.20	1379	7.97	Copaene	3856-25-5	928	3	C15 H24	204	✔	none	0.89	SI
048	12.80	1424	19.79	Caryophyllene	87-44-5	980	5	C15 H24	204	✔	none	0.87	SI
049	13.99	1519	18.19	1,3-Benzodioxole, 4-methoxy-6-(2-propenyl)-	607-91-0	976	0	C11 H12 O3	192	✔	none	1.00	SI
050	18.77	1953	14.13	n-Hexadecanoic acid	57-10-3	941	15	C16 H32 O2	256	✔	none	0.95	EI

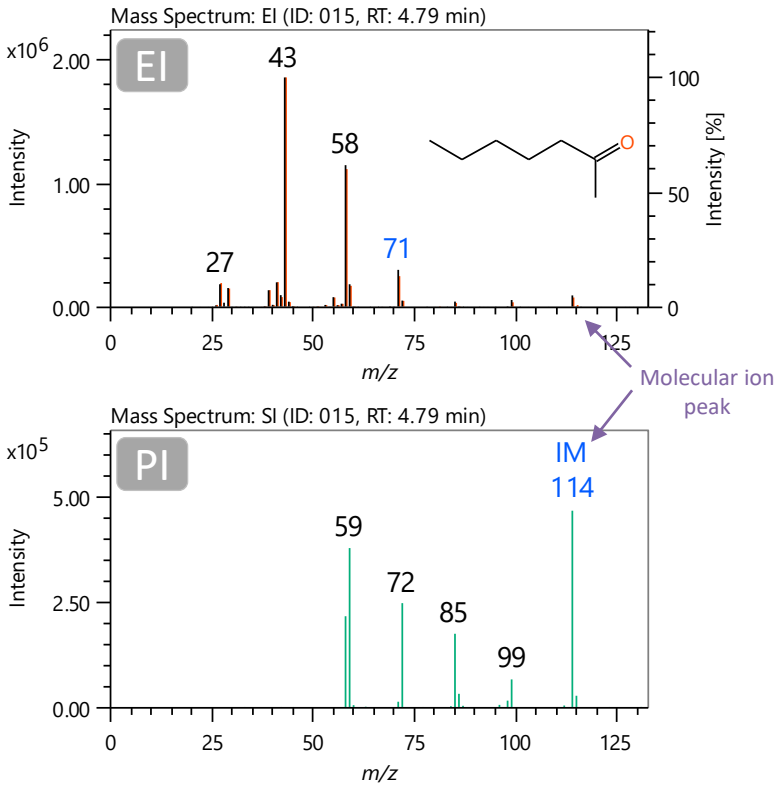
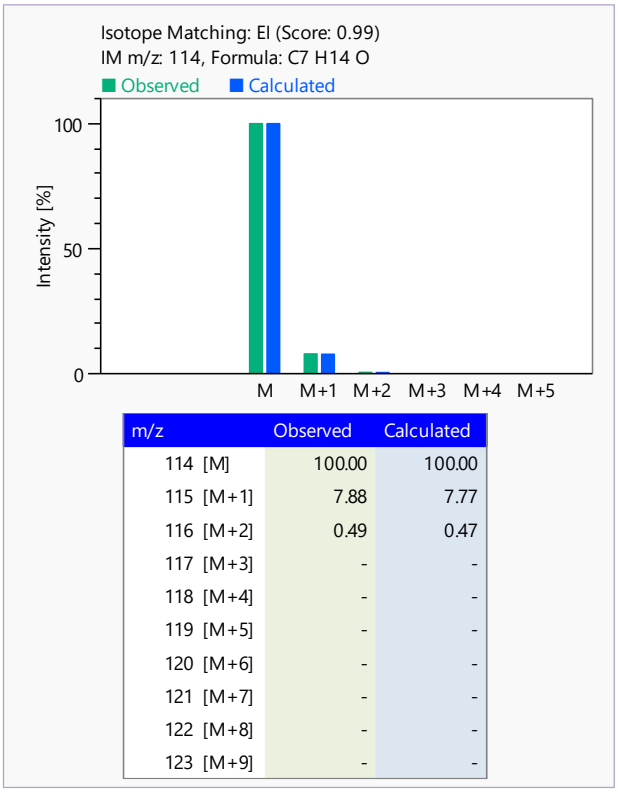


Figure 3 Mass spectra of ID:[015]



m/z	Observed	Calculated
114 [M]	100.00	100.00
115 [M+1]	7.88	7.77
116 [M+2]	0.49	0.47
117 [M+3]	-	-
118 [M+4]	-	-
119 [M+5]	-	-
120 [M+6]	-	-
121 [M+7]	-	-
122 [M+8]	-	-
123 [M+9]	-	-

Figure 4 Isotope pattern matching results for ID:[015] molecular ion

4. Conclusion

We analyzed the aroma compounds in hamburger steak using microchamber and TD-GC-MS. As a result, several aroma compounds were identified from the hamburger steak, demonstrating the usefulness of this method for food aroma analysis.

References 1) M. Ubukata et al, Rapid Commun Mass Spectrum., 34 (2020). DOI: 10.1002/rcm.8820

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