Analysis of the aroma components of spearmint essential oil by GC-TOFMS and AI structure analysis

Product used: Mass Spectrometer (MS)

Introduction

Spearmint is a plant in the genus Mentha of the Family Lamiaceae. It has long been known as one of the herbs with a sweet and refreshing aroma. Fresh leaves are often used in cooking, herbal teas, and desserts, and it is also widely used as a flavor for toothpaste and chewing gum. The aroma of spearmint has aromatherapy effect, making it a recommended aroma when you want to energize and refresh a tired body and mind, or when you want to clear your head.

In this MSTips, we introduce the results of analysis of the aroma components of spearmint essential oil by gas chromatography-time-of-flight mass spectrometry (GC-TOFMS). Since essential oils are natural materials extracted from flowers, leaves, and seeds of plants, they are expected to contain substances not registered in the NIST database. Therefore, we used JMS-T2000GC and msFineAnalysis AI, which are capable of AI structural analysis, for the analysis.

Experiment

Commercially available spearmint essential oil was used, diluted by 1/2 with a hexane solution. JMS-T2000 GC AccuTOF™ GC-Alpha (JEOL) was used for GC-MS measurements (Figure 1). The GC-TOFMS measurements were performed using both EI and field ionization (FI) modes with the EI/FI/FD combination ion source. The qualitative data processing was performed with msFineAnalysis AI (JEOL). Measurement conditions are shown in Table 1.

Figure 1 JMS-T2000GC AccuTOF™ GC-Alpha

Table 1 Measurement conditions

GC conditions	
Gas Chromatograph	8890 GC (Agilent Technologies)
Column	HP-1MS (Agilent Technologies) 30m x 0.32mm, 0.25µm
Inlet	250 °C, Split 50:1
Oven temperature	40°C(1min) - 15°C/min - 250°C(1min)
Carrier flow	He, 1.5mL/min

MS conditions	
Spectrometer	JMS-T2000GC (JEOL Ltd.)
Ion source	EI/FI combination
Ionization	EI(70eV, 250 °C), FI
Mass range	*m/z* 33-800
Analysis software	msFineAnalysis AI

Results

Figure 2 shows the TIC chromatogram of spearmint essential oil. Thirty-seven components were detected as having a height threshold of 0.015% or higher. Carvone, which has a sweet and refreshing scent, was detected as the main component, followed by Limonene, which has a sweet-sour and refreshing scent.

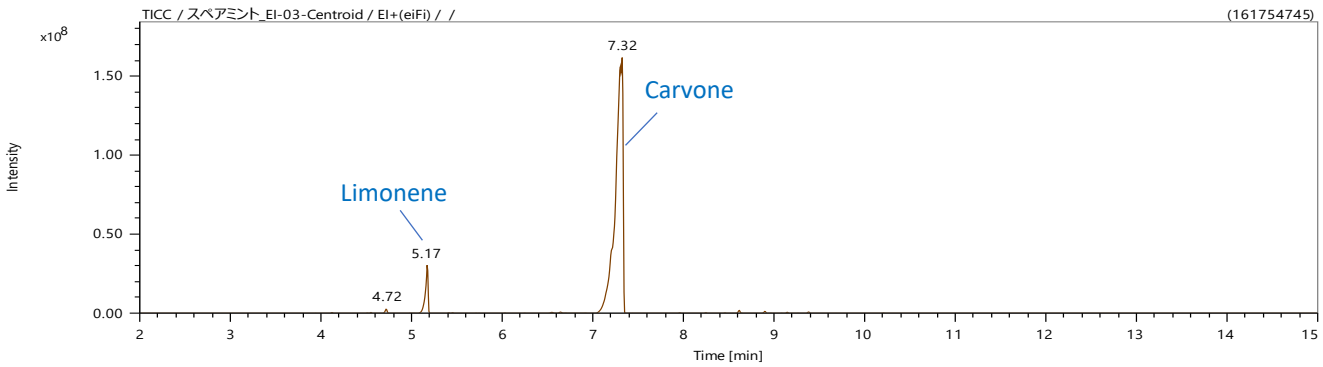

Figure 2 TIC chromatograms of spearmint essential oil

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Table 2 shows the results of the integrated analysis of the 37 aroma compounds detected in spearmint essential oil. Six of the 37 identified components were not registered in the NIST database, it was able to obtain the compound name and structural formula by AI structural analysis. Figure 3 shows an enlarged TICC around limonene, the second most strongly identified compound. The deconvolution peak detection identified and detected even the low-intensity component as a co-eluting component. The co-eluting component ID010 was also an unknown substance not registered in the NIST library database, but a candidate structural formula could be calculated by the AI structural analysis function as shown below.

Table 2 List of integrated analysis results of spearmint essential oil components

General					Total Result								
ID	RT [min]	RI [iu]	Height [%]	IM m/z	Compound Name	Lib.	Similarity / AI Score	Formula	DBE	Calculated m/z	Mass Error [mDa]	Isotope Matching	EI Fragment Coverage
001	3.65	887	0.02	128.11939	Furan, 2,5-diethyltetrahydro-	mainlib	823	C8 H16 O	1.0	128.11957	-0.18	0.92	100
002	3.94	914	0.03	136.12440	5,5-Dimethyl-1-vinylbicyclo[2.1.1]hexane	mainlib	865	C10 H16	3.0	136.12465	-0.25	0.93	100
003	4.12	931	0.26	136.12439	α-Pinene	mainlib	928	C10 H16	3.0	136.12465	-0.27	0.98	100
004	4.51	967	0.03	136.12426	1-methyl-5-(2-methylpropyl)cyclopenta-1,3-diene	AI	833	C10 H16	3.0	136.12465	-0.39	0.99	75
005	4.55	970	0.25	136.12445	β-Pinene	mainlib	920	C10 H16	3.0	136.12465	-0.20	0.98	100
006	4.72	986	1.63	136.12465	β-Myrcene	mainlib	806	C10 H16	3.0	136.12465	0.00	0.72	88
007	4.83	996	0.02	136.12422	Bicyclo[3.1.0]hexane, 4-methylene-1-(1-methylethyl)-	mainlib	731	C10 H16	3.0	136.12465	-0.44	0.98	100
008	4.99	1011	0.08	136.12399	(+)-4-Carene	mainlib	909	C10 H16	3.0	136.12465	-0.66	0.97	100
009	5.03	1015	0.03	134.10854	Benzene, 1-methyl-3-(1-methylethyl)-	mainlib	874	C10 H14	4.0	134.10900	-0.46	0.99	100
010	5.13	1024	0.36	154.13503	3-ethyl-4-methylhept-3-en-2-one	AI	830	C10 H18 O	2.0	154.13522	-0.18	0.90	90
011	5.17	1028	18.99	136.12463	Limonene	mainlib	931	C10 H16	3.0	136.12465	-0.02	0.36	100
012	5.30	1040	0.02	136.12411	β-Ocimene	mainlib	835	C10 H16	3.0	136.12465	-0.55	0.96	100
013	5.41	1051	0.17	136.12447	γ-Terpinene	mainlib	880	C10 H16	3.0	136.12465	-0.18	0.94	100
014	5.45	1055	0.17	154.13487	7-Oxabicyclo[2.2.1]heptane, 1-methyl-4-(1-methylethyl)-	mainlib	772	C10 H18 O	2.0	154.13522	-0.35	0.98	100
015	5.71	1080	0.05	136.12413	Cyclohexene, 3-methyl-6-(1-methylethylidene)-	mainlib	856	C10 H16	3.0	136.12465	-0.52	0.99	100
016	5.74	1083	0.03	168.11387	(2R)-6-oxo-2-prop-2-enylheptanal	AI	792	C10 H16 O2	3.0	168.11448	-0.61	0.75	93
017	6.03	1111	0.11	172.14573	3-Octanol, acetate	mainlib	915	C10 H20 O2	1.0	172.14578	-0.05	0.96	100
018	6.24	1132	0.08	154.13508	Cyclohexanone, 5-methyl-2-(1-methylethyl)-, (2R-cis)-	mainlib	840	C10 H18 O	2.0	154.13522	-0.13	0.93	100
019	6.34	1142	0.02	154.13484	Cyclohexanone, 5-methyl-2-(1-methylethyl)-, trans-	mainlib	748	C10 H18 O	2.0	154.13522	-0.38	0.99	100
020	6.54	1162	0.32	154.13495	3-Cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)-, (R)-	mainlib	851	C10 H18 O	2.0	154.13522	-0.27	0.81	100
021	6.64	1172	0.43	152.11957	(+)-Dihydrocarvone	mainlib	886	C10 H16 O	3.0	152.11957	0.00	0.71	100
022	6.92	1200	0.05	154.13478	Cyclohexanol, 2-methyl-5-(1-methylethenyl)-, [1S-(1α,2β,5α)]-	mainlib	754	C10 H18 O	2.0	154.13522	-0.44	0.96	100
023	7.30	1241	100.00	152.11158	(-)-Carvone	mainlib	906	C10 H14 O	4.0	150.10392	-1.00	0.67	90
024	7.44	1256	0.03	166.09856	Carvone oxide	mainlib	782	C10 H14 O2	4.0	166.09883	-0.27	0.55	100
025	7.74	1288	0.02	194.16600	2-tert-butyl-3,4-dimethyl-5-propan-2-ylfuran	AI	776	C13 H22 O	3.0	194.16652	-0.51	0.72	100
026	7.96	1312	0.19	196.14593	Dihydro carveol acetate	mainlib	896	C12 H20 O2	3.0	196.14578	0.15	0.88	100
027	8.24	1344	0.22	194.12967	2-Cyclohexen-1-ol, 2-methyl-5-(1-methylethenyl)-, acetate, cis-	mainlib	904	C12 H18 O2	4.0	194.13013	-0.46	0.92	100
028	8.46	1368	0.14	164.11951	2-Cyclopenten-1-one, 3-methyl-2-(2-pentenyl)-, (Z)-	mainlib	880	C11 H16 O	4.0	164.11957	-0.06	0.76	100
029	8.62	1386	1.13	204.18671	(-)-β-Bourbonene	mainlib	889	C15 H24	4.0	204.18725	-0.54	0.90	100
030	8.90	1419	0.74	204.18654	Caryophyllene	mainlib	919	C15 H24	4.0	204.18725	-0.71	0.91	95
031	8.96	1427	0.04	204.18648	4-heptan-4-yl-1,2-dimethylbenzene	AI	827	C15 H24	4.0	204.18725	-0.77	0.81	100
032	9.08	1441	0.03	204.18640	(4S,7R)-2,7-dimethyl-4-propan-2-yl-1a,3a,4,5,6,7-hexahydro-1H-cyclopropa[i]indene	AI	857	C15 H24	4.0	204.18725	-0.85	0.93	100
033	9.14	1449	0.38	204.18671	cis-β-Farnesene	mainlib	937	C15 H24	4.0	204.18725	-0.54	0.90	100
034	9.26	1463	0.04	204.18656	Germacrene D	mainlib	711	C15 H24	4.0	204.18725	-0.69	0.97	100
035	9.38	1477	0.50	204.18649	Germacrene D	mainlib	887	C15 H24	4.0	204.18725	-0.76	0.85	100
036	9.68	1514	0.03	204.18637	Naphthalene, 1,2,3,5,6,8a-hexahydro-4,7-dimethyl-1-(1-methylethyl)-, (1S-cis)-	mainlib	823	C15 H24	4.0	204.18725	-0.89	0.93	100
037	10.21	1581	0.10	222.19707	1H-Cycloprop[e]azulen-4-ol, decahydro-1,1,4,7-tetramethyl-, [1aR-(1aα,4β,4aβ,7α,7aβ,7bα)]-	mainlib	828	C15 H26 O	3.0	222.19782	-0.75	0.99	100

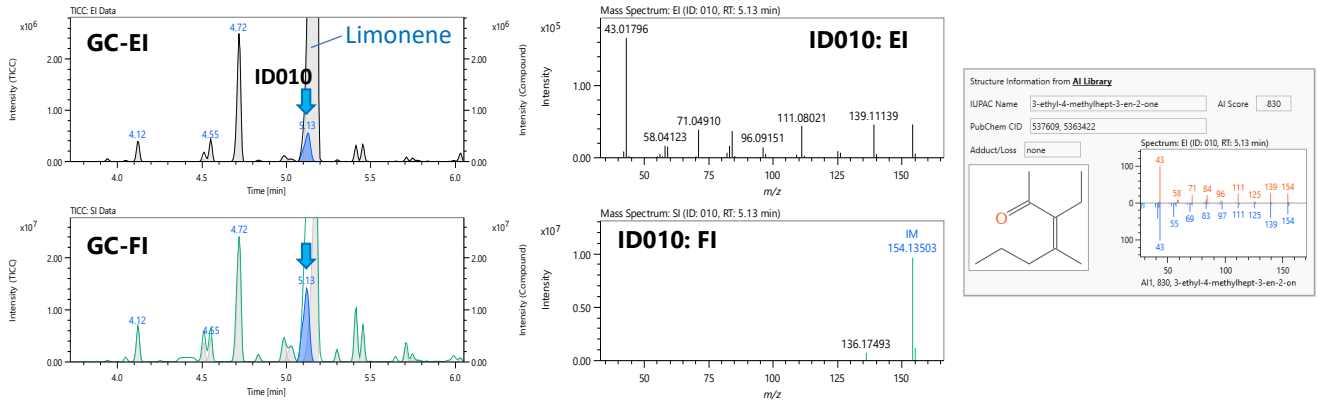

Figure 3 TIC chromatograms (left), mass spectrum (center), AI structural analysis results summary (right) of spearmint essential oil components

Conclusion

GC-TOFMS using JMS-T20000GC and msFineAnalysis AI were used to analyze aroma components in spearmint essential oil. Deconvolution peak detection by msFineAnalysis AI enabled the detection of co-eluted components without missing them. Although some components were not registered in NIST database, it was able to obtain the compound name and structural formula by AI structural analysis. So, it was confirmed that this device and software are effective for analyzing aroma components in naturally derived samples.

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