Differential analysis of coffee aroma compounds using HS GC-QMS and msFineAnalysis iQ

Product used : Mass spectrometer (MS)

Overview

The msFineAnalysis iQ is an automated qualitative analysis software for gas chromatograph quadrupole mass spectrometer JMS-Q1500GC/JMS-Q1600GC that performs integrated analysis combining library database (DB) search results using EI (Electron Ionization) mass spectra and molecular weight information using soft ionization methods. In addition, the software provides automatic peak detection by deconvolution, differential analysis of two samples, and qualitative analysis by retention index (RI). Using this software, a more accurate qualitative analysis can be achieved in a shorter time.
Coffee aroma contains a very large number of components. Therefore, a large number of peaks are observed in TIC chromatogram, and it takes a long time to identify the components of each peak and to analyze differences between samples. In this report, we present the results of differential and integrated analyses performed on coffee aroma components using msFineAnalysis iQ.

Methods

A headspace sampler MS-62071STRAP and a GC-QMS JMS-Q1600GC UltraQuad™ SQ-Zeta were used for the measurements. Data were acquired using the EI method and the low ionization energy EI method as a soft ionization (SI) method for the components collected using the trap mode. Samples were 2 mL of coffee extracted from commercially available instant drip-type packets (A: fresh coffee immediately after opening, B: oxidized coffee for 5 days after opening) and measured by the EI (n=5) and SI (n=1) methods for components extracted into the headspace by heating. Comparison of two samples (differential analysis between A and B) was attempted using the measurement data obtained under the measurement conditions shown in Table 1. Please refer to MSTips No. 348 for the function of differential analysis.

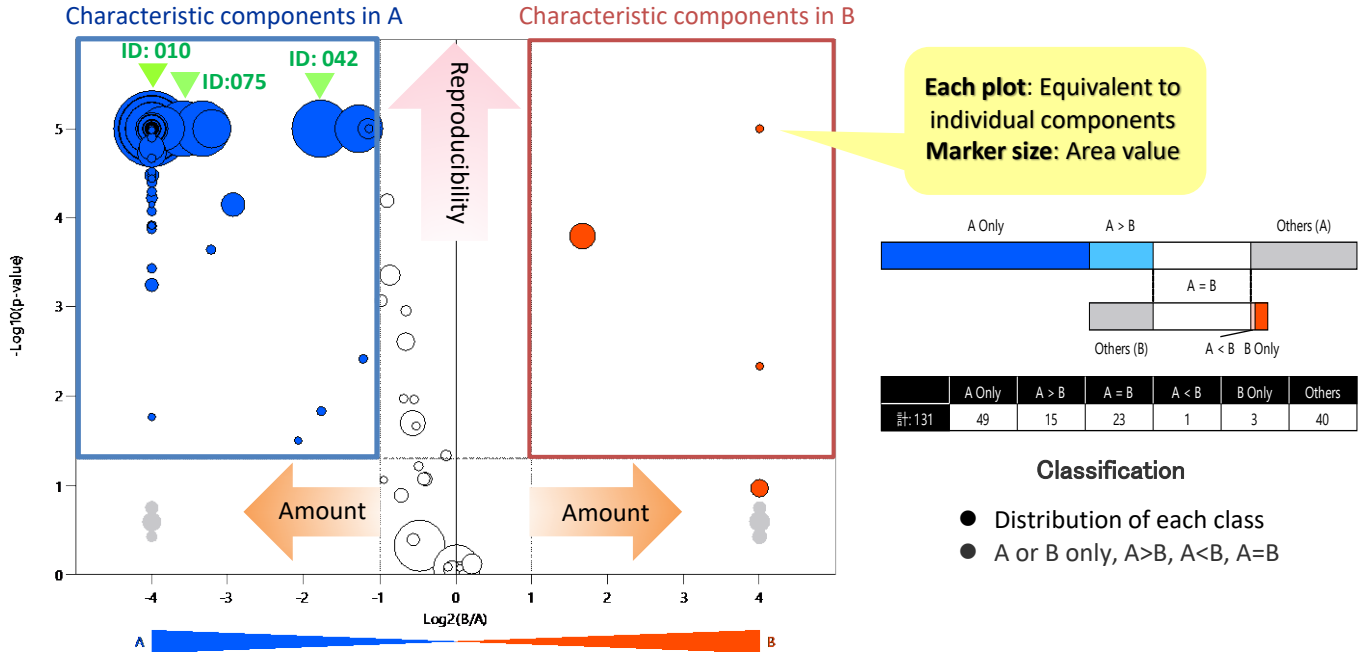
JMS-Q1600GC *UltraQuad™ SQ-Zeta* w/ MS-62071STRAP

Table 1 Measurement Condition

GC		HS		MS	
Column	InertCap WAX (GL Sciences Inc.) 60 m × 0.32 mm id, 0.5 µm film thickness	Sample temp.	60 °C	Interface temp.	250°C
Oven temp.	40°C(3min)→10°C/min→250°C(10min)	Heating time	15 min	Ion source temp.	250°C
Carrier gas	1.5 mL/min (Constant Flow)	Sampling mode	Trap	Acquisition mode	Scan (*m/z* 29-400)
Injection temp.	250°C	Number of sampling	3	Ionization	EI(70eV, 50µA) SI(15eV, 30µA)
Injection mode	Split 30:1	Trap tube	AQUATRAP1 (GL Sciences Inc.)		

Results

Figure 1 shows the results of the differential analysis (volcano plots). The difference in the number of components and the detected amount (peak area value) between Sample A and Sample B was visualized and easily confirmed. The characteristic components of Sample A were 64 peaks (A Only 49 peaks, A>B 15 peaks), the common components of Sample A and Sample B were 23 peaks (A=B), and the characteristic components of Sample B were 4 peaks.
Next, the results of the integrated analysis of the characteristic components and the mass spectra (ID: 010, 042, 075) are shown as an example.



	A Only	A > B	A = B	A < B	B Only	Others
計:131	49	15	23	1	3	40

Figure 1 Volcano plot of variance component analysis result between fresh coffee (A) and oxidized coffee (B)

Table 2 shows the names of compounds with a similarity of 900 or more that were estimated as characteristic aroma components of Sample A by the integrated analysis. Estimated compounds were aldehydes, furans, esters, ketones, pyrroles, and pyridines. As an example, Figure 2 shows the mass spectra of "Furan,2-methyl-" (ID: 010), "Pyridine" (ID: 042), and "2-Furanmethanol, acetate" (ID:075) acquired by the EI and SI (low ionization energy EI) methods. Integrated qualitative analysis was able to complement the search results not only by the molecular ions in the SI method, but also by ΔRI (tolerance = |50|).

Table 2 Integrated qualitative analysis result of characteristic aroma components in fresh coffee (A)

General			Variance Component Analysis Result			Total Result								Spectrum Info	
ID	RT [min]	Height [%]	Class	Count (A)	Count (B)	Library Name	CAS#	Lib.	Similarity	ΔRI [iu]	Formula	MW	Molecular Weight Check	IM m/z	IM Ionization
003	4.79	69.32	A > B	5	5	Acetaldehyde	75-07-0	replib	903	22	C2 H4 O	44	✔	44	SI
004	5.36	17.15	A Only	5	0	Methyl formate	107-31-3	replib	934	7	C2 H4 O2	60	✔	60	SI
006	5.80	22.86	A Only	5	0	Furan	110-00-9	replib	937	18	C4 H4 O	68	✔	68	SI
008	6.05	100.00	A > B	5	5	Acetone	67-64-1	replib	907	14	C3 H6 O	58	✔	58	SI
009	6.22	51.36	A > B	5	5	Acetic acid, methyl ester	79-20-9	mainlib	946	17	C3 H6 O2	74	✔	74	SI
010	6.90	97.47	A Only	5	0	Furan, 2-methyl-	534-22-5	replib	926	19	C5 H6 O	82	✔	82	SI
012	7.14	7.30	A Only	5	0	Ethyl Acetate	141-78-6	replib	930	16	C4 H8 O2	88	✔	88	SI
013	7.26	8.23	A > B	5	5	Methyl Alcohol	67-56-1	mainlib	921	8	C H4 O	32	✔	32	SI
015	7.40	44.90	A > B	5	5	2-Butanone	78-93-3	replib	911	11	C4 H8 O	72	✔	72	SI
022	8.36	7.94	A Only	5	0	Furan, 2,5-dimethyl-	625-86-5	replib	934	32	C6 H8 O	96	✔	96	SI
023	8.79	29.40	A > B	5	5	2,3-Butanedione	431-03-8	mainlib	927	16	C4 H6 O2	86	✔	86	SI
027	9.75	2.75	A Only	5	0	Thiophene	110-02-1	replib	910	22	C4 H4 S	84	✔	84	SI
029	10.06	2.17	A Only	5	0	Toluene	108-88-3	replib	904	22	C7 H8	92	✔	92	SI
034	10.70	7.08	A Only	5	0	Disulfide, dimethyl	624-92-0	mainlib	918	22	C2 H6 S2	94	✔	94	SI
040	11.87	20.14	A Only	5	0	1H-Pyrrole, 1-methyl-	96-54-8	mainlib	909	21	C5 H7 N	81	✔	81	SI
042	12.63	75.12	A > B	5	5	Pyridine	110-86-1	replib	956	24	C5 H5 N	79	✔	79	SI
070	16.95	20.65	A Only	5	0	Furfural	98-01-1	replib	928	41	C5 H4 O2	96	✔	96	SI
075	17.74	82.31	A > B	5	5	2-Furanmethanol, acetate	623-17-6	mainlib	953	29	C7 H8 O3	140	✔	140	SI

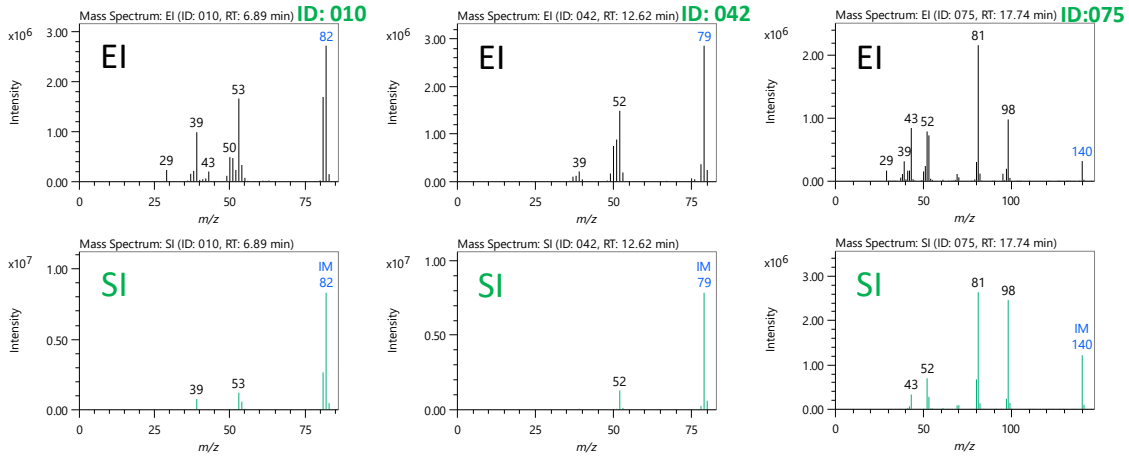


Figure 2 An example of the mass spectra detected from fresh coffee (A)

Table 3 shows the names of all compounds estimated as characteristic aroma components of Sample B by the integrated qualitative analysis. The components detected in Sample A decreased and disappeared, while ethanol was detected as a characteristic component of Sample B.

Table 3 Integrated qualitative analysis result of characteristic aroma components in oxidized coffee (B)

General			Variance Component Analysis Result			Total Result								Spectrum Info	
ID	RT [min]	Height [%]	Class	Count (A)	Count (B)	Library Name	CAS#	Lib.	Similarity	ΔRI [iu]	Formula	MW	Molecular Weight Check	IM m/z	IM Ionization
020	7.90	14.16	A < B	5	5	Ethanol	64-17-5	replib	888	14	C2 H6 O	46	✔	46	SI
025	9.39	1.48	B Only	0	5	Acetonitrile	75-05-8	replib	929	15	C2 H3 N	41	✔	41	SI
088	19.99	1.27	B Only	0	5	2-Furanmethanol, 5-methyl-	3857-25-8	mainlib	687	27	C6 H8 O2	112	✔	112	SI
090	21.62	7.43	B Only	0	5	Hexanoic acid	142-62-1	replib	880	42	C6 H12 O2	116	✔	116	EI

Table 4 shows the names of compounds with a similarity of 850 or more that were estimated as common components of Sample A and Sample B by the integrated qualitative analysis. Acetic acid, 2-Furanmethanol, and pyrazines were estimated as characteristic compounds.

Table 4 Integrated qualitative analysis result of characteristic aroma components in both coffee of A and B

General			Variance Component Analysis Result			Total Result								Spectrum Info	
ID	RT [min]	Height [%]	Class	Count (A)	Count (B)	Library Name	CAS#	Lib.	Similarity	ΔRI [iu]	Formula	MW	Molecular Weight Check	IM m/z	IM Ionization
047	13.11	3.28	A = B	5	5	Pyrazine	290-37-9	replib	859	29	C4 H4 N2	80	✔	80	SI
052	14.00	15.92	A = B	5	5	Pyrazine, methyl-	109-08-0	replib	947	30	C5 H6 N2	94	✔	94	SI
056	14.62	9.18	A = B	5	5	2-Propanone, 1-hydroxy-	116-09-6	replib	916	36	C3 H6 O2	74	✔	74	SI
057	14.86	3.87	A = B	5	5	Pyrazine, 2,5-dimethyl-	123-32-0	replib	916	34	C6 H8 N2	108	✔	108	SI
058	14.94	4.32	A = B	5	5	Pyrazine, 2,6-dimethyl-	108-50-9	replib	916	32	C6 H8 N2	108	✔	108	SI
059	15.06	4.66	A = B	5	5	Pyrazine, ethyl-	13925-00-3	replib	924	31	C6 H8 N2	108	✔	108	SI
062	15.78	2.83	A = B	5	5	Pyrazine, 2-ethyl-6-methyl-	13925-03-6	replib	918	31	C7 H10 N2	122	✔	122	SI
064	15.89	1.81	A = B	5	5	Pyrazine, 2-ethyl-5-methyl-	13360-64-0	mainlib	896	38	C7 H10 N2	122	✔	122	SI
068	16.71	3.04	A = B	5	5	2-Propanone, 1-(acetyloxy)-	592-20-1	replib	870	10	C5 H8 O3	116	✔	116	SI
069	16.89	49.57	A = B	5	5	Acetic acid	64-19-7	replib	965	47	C2 H4 O2	60	✔	60	SI
072	17.55	7.88	A = B	5	5	Ethanone, 1-(2-furanyl)-	1192-62-7	replib	937	49	C6 H6 O2	110	✔	110	SI
083	19.27	53.60	A = B	5	5	2-Furanmethanol	98-00-0	mainlib	967	23	C5 H6 O2	98	✔	98	SI
084	19.44	10.74	A = B	5	5	Butyrolactone	96-48-0	replib	877	66	C4 H6 O2	86	✔	86	SI

Summary

In this report, we presented the results of integrated qualitative analysis and differential analysis using msFineAnalysis iQ for coffee aroma components acquired by HS GC-QMS. It has been shown that this method can be used to quickly identify multiple components with different contents. Integrated qualitative analysis with msFineAnalysis iQ will improve the accuracy of qualitative analysis, reduce work time and increase work efficiency.

JEOL Ltd.
3-1-2 Musashino Akishima Tokyo 196-8558 Japan Sales Division Tel. +81-3-6262-3560 Fax. +81-3-6262-3577
www.jeol.com ISO 9001 • ISO 14001 Certified
▼ Local office
• AUSTRALIA & NEW ZEALAND • BELGIUM • BRAZIL • CANADA • CHINA • EGYPT • FRANCE • GERMANY • GREAT BRITAIN & IRELAND • ITALY • KOREA • MALAYSIA • MEXICO • RUSSIA • SCANDINAVIA • SINGAPORE • TAIWAN • THE NETHERLANDS • USA