Integrated qualitative analysis of fatty acid methyl esters(FAMEs) by msFineAnalysis iQ

Product used : Mass spectrometer (MS)

Overview

Electron Ionization (EI) used in gas chromatograph quadrupole mass spectrometer is one of a hard ionization method which tends to generate fragment ions. Compounds can be identified by comparing the mass spectra acquired by EI with libraries. On the other hands, soft ionization such as photoionization(PI) is used to find the molecular ion. Comparing the molecular weights of the compounds identified in the library with the molecular ions enables highly accurate qualitative analysis. Therefore we have developed an integrated qualitative analysis method called msFineAnalysis iQ. Fatty acid methyl esters（FAMEs）are important compounds for measuring the amount of lipids in food, and are used as biodiesel fuels due to their low environmental impact. Numerous unsaturated compounds with a double bond in the alkyl chain are contained in FAMEs. As the number of the double bonds increases, it tends to become more difficult to detect their molecular ions by EI. Therefore FAMEs standard sample was measured by EI and PI to confirm the detection of molecular ions. We report the results of the integrated qualitative analysis combining library database search and molecular ion confirmation when performing msFineAnalysis iQ.

Results

Restek's 37 fatty acid methyl ester mixture standard reagent (2-6 wt/wt%, P/N: 35077) was used as the measurement sample. The measurement conditions are shown in Table 1. Fig.1 shows TICC of GC/EI and GC/PI. All of 37 components could be separated and detected using medium polarity column DB-23.

In PI mass spectra, each molecular ion of FAMEs with 3 or less double bonds in the alkyl group was observed remarkably, while the relative intensity decreased as the double bonds increased. When the relative intensity of molecular ions is small, it becomes difficult to distinguish between ions and noise. But even in such cases, it was possible to search for molecular ions based on the library search results in integrated qualitative analysis.

As examples, Fig. 2 shows the mass spectra and the structural formulas of six components with 20 carbon atoms excluding ester bonds and 0 to 5 double bonds.

Table 1 Measurement condition

[GC condition]	
GC system:	8890 (Agilent Technologies)
Column:	DB-23 (Agilent Technologies), 30m x 0.25mm, 0.15mm
Oven temp.:	50℃(1min)→25℃/min→175℃→4℃/min→230℃
Inj. mode:	Split mode (100:1)
Inj. volume:	GC/EI: 0.5mL, GC/PI: 1.0mL
[QMS condition]	
MS system:	JMS-Q1600GC (JEOL Ltd.)
Ion source:	EI/PI combination ion source
Ionization:	EI+, 70eV, 50μA
	PI+, 10.8eV (115-400nm, D2 lamp)
Mass range:	*m/z* 29-400 (SCAN mode)

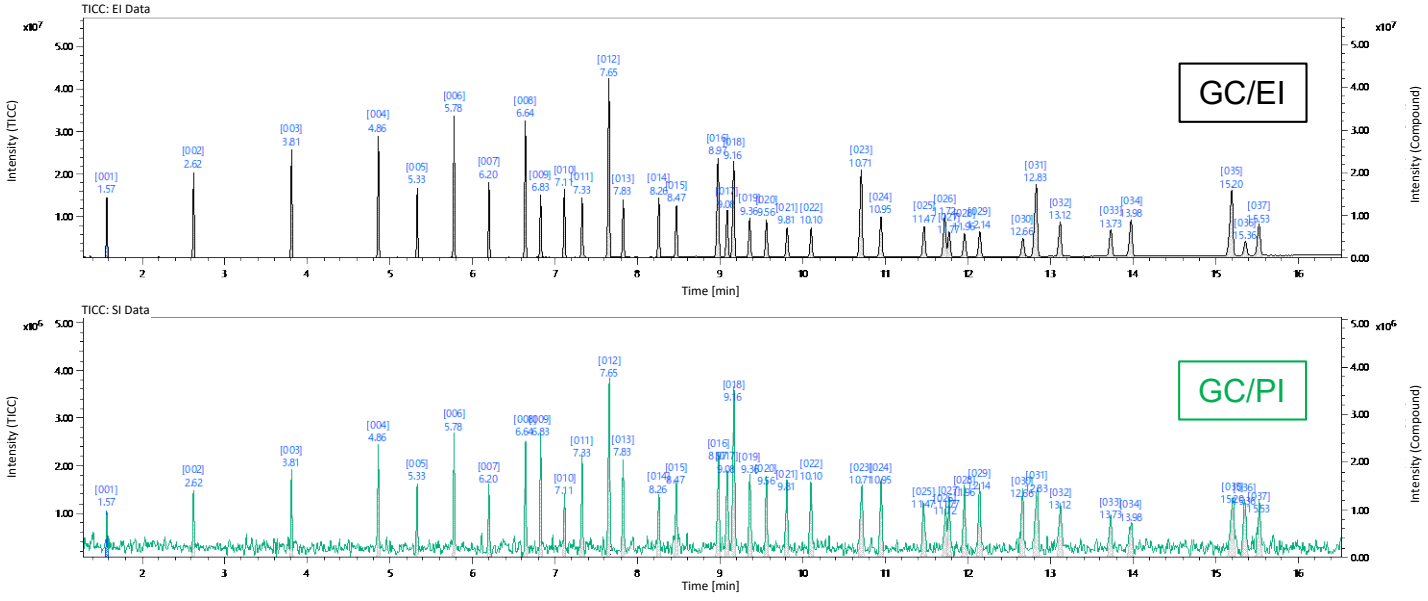
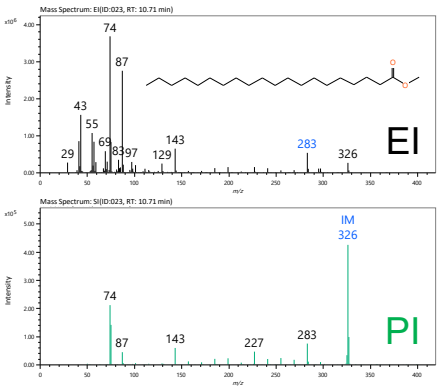


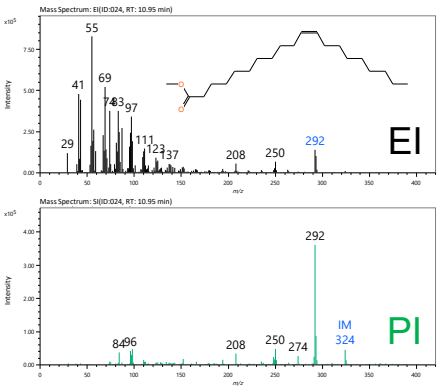
Fig.1 GC/EI and GC/PI total ion current chromatograms for the FAME 37 mix sample.

Table 2 Integrated qualitative analysis result using the msFineAnalysis iQ

General						Total Result									Spectrum Info			
ID	RT [min]	Width [s]	Area	Height	Link	Library Name	CAS#	Similarity	Reverse Similarity	Formula	DBE	MW	Molecular	Adduct/Loss	IM m/z	IM Ionization	EI Base Peak	SI Base Peak
001	1.57	2.44	13927657	14154520	✔	Butanoic acid, methyl e	623-42-7	924	924	C5 H10 O2	1.0	102	✔	none	102	SI	43	74
002	2.62	2.44	19403662	19911061	✔	Hexanoic acid, methyl e	106-70-7	964	967	C7 H14 O2	1.0	130	✔	none	130	SI	74	74
003	3.81	2.44	24035381	25151344	✔	Octanoic acid, methyl e	111-11-5	936	936	C9 H18 O2	1.0	158	✔	none	158	SI	74	74
004	4.86	2.44	27846900	28296508	✔	Decanoic acid, methyl e	110-42-9	957	960	C11 H22 O2	1.0	186	✔	none	186	SI	74	74
005	5.33	2.44	14996779	16268702	✔	Undecanoic acid, meth	1731-86-8	955	957	C12 H24 O2	1.0	200	✔	none	200	SI	74	74
006	5.78	2.74	31752507	33059575	✔	Dodecanoic acid, meth	111-82-0	937	937	C13 H26 O2	1.0	214	✔	none	214	SI	74	74
007	6.20	2.44	16630505	17571475	✔	Tridecanoic acid, meth	1731-88-0	963	963	C14 H28 O2	1.0	228	✔	none	228	SI	74	74
008	6.64	2.75	34468629	31990427	✔	Methyl tetradecanoate	124-10-7	956	958	C15 H30 O2	1.0	242	✔	none	242	SI	74	74
009	6.83	2.74	15391076	14511274	✔	Methyl myristoleate	56219-06-8	961	961	C15 H28 O2	2.0	240	✔	none	240	SI	55	208
010	7.11	2.74	17688344	15848200	✔	Pentadecanoic acid, me	7132-64-1	952	952	C16 H32 O2	1.0	256	✔	none	256	SI	74	74
011	7.33	3.96	16283048	13977019	✔	Methyl (Z)-10-pentade	-	925	961	C16 H30 O2	2.0	254	✔	none	254	SI	55	222
012	7.65	3.35	55196271	41646818	✔	Hexadecanoic acid, me	112-39-0	949	949	C17 H34 O2	1.0	270	✔	none	270	SI	74	270
013	7.83	3.35	16555571	13484391	✔	9-Hexadecenoic acid, m	1120-25-8	956	956	C17 H32 O2	2.0	268	✔	none	268	SI	55	236
014	8.26	3.66	18695837	13826058	✔	Heptadecanoic acid, me	1731-92-6	924	924	C18 H36 O2	1.0	284	✔	none	284	SI	74	284
015	8.47	3.66	16788522	11953172	✔	cis-10-Heptadecenoic a	-	959	959	C18 H34 O2	2.0	282	✔	none	282	SI	55	250
016	8.97	4.27	38337178	23096407	✔	Methyl stearate	112-61-8	952	952	C19 H38 O2	1.0	298	✔	none	298	SI	74	298
017	9.09	3.66	16988977	10789984	✔	9-Octadecenoic acid, m	1937-62-8	925	925	C19 H36 O2	2.0	296	✔	none	296	SI	55	264
018	9.16	3.66	33909149	22037270	✔	9-Octadecenoic acid (Z)	112-62-9	951	951	C19 H36 O2	2.0	296	✔	none	296	SI	55	264
019	9.36	4.27	14285941	9060563	✔	9,12-Octadecadienoic a	2566-97-4	895	895	C19 H34 O2	3.0	294	✔	none	294	SI	67	294
020	9.56	4.27	13994383	8567383	✔	9,12-Octadecadienoic a	112-63-0	950	950	C19 H34 O2	3.0	294	✔	none	294	SI	67	262
021	9.81	5.49	11815613	6870547	✔	Methyl γ-linolenate	16326-32-2	961	961	C19 H32 O2	4.0	292	✔	none	292	SI	79	150
022	10.10	4.57	11505776	6795477	✔	9,12,15-Octadecatrieno	301-00-8	958	958	C19 H32 O2	4.0	292	✔	none	292	SI	79	108
023	10.71	4.88	39492253	20336258	✔	Eicosanoic acid, methyl	1120-28-1	892	892	C21 H42 O2	1.0	326	✔	none	326	SI	74	326
024	10.95	4.88	17401011	9362419	✔	cis-Methyl 11-eicoseno	2390-09-2	964	964	C21 H40 O2	2.0	324	✔	none	324	SI	55	292
025	11.47	5.49	14088137	7170334	✔	11,14-Eicosadienoic aci	2463-02-7	911	942	C21 H38 O2	3.0	322	✔	none	322	SI	67	290
026	11.72	3.66	14908261	8272499	✔	Heneicosanoic acid, me	6064-90-0	915	917	C22 H44 O2	1.0	340	✔	none	340	SI	74	340
027	11.77	2.74	6422169	4109245	✔	8,11,14-Eicosatrienoic a	21061-10-9	899	903	C21 H36 O2	4.0	320	✔	none	320	SI	67	150
028	11.96	5.49	10960531	5371285	✔	5,8,11,14-Eicosatetraen	2566-89-4	970	970	C21 H34 O2	5.0	318	-	-	247	SI	79	150
029	12.14	5.49	11896854	5756162	✔	11,14,17-Eicosatrienoic	55682-88-7	883	911	C21 H36 O2	4.0	320	✔	none	320	SI	79	108
030	12.66	6.10	9382224	4368089	✔	5,8,11,14,17-Eicosapent	2734-47-6	971	971	C21 H32 O2	6.0	316	-	-	215	SI	79	108
031	12.83	6.10	40124001	16683017	✔	Docosanoic acid, methy	929-77-1	946	946	C23 H46 O2	1.0	354	✔	none	354	SI	74	354
032	13.12	5.79	17406003	7976546	✔	13-Docosenoic acid, me	1120-34-9	892	892	C23 H44 O2	2.0	352	✔	none	352	SI	55	320
033	13.73	5.79	14106349	6224634	✔	cis-13,16-Docasadienoi	-	956	956	C23 H42 O2	3.0	350	✔	none	350	SI	67	350
034	13.98	5.79	19984303	8485938	✔	Tricosanoic acid, methy	2433-97-8	947	947	C24 H48 O2	1.0	368	✔	none	368	SI	74	368
035	15.20	6.71	40099524	15274311	✔	Tetracosanoic acid, met	2442-49-1	907	907	C25 H50 O2	1.0	382	✔	none	382	SI	74	382
036	15.36	6.71	8396494	3309815	✔	4,7,10,13,16,19-Docosal	2566-90-7	975	975	C23 H34 O2	7.0	342	-	-	166	SI	79	108
037	15.53	5.79	17925096	7293540	✔	15-Tetracosenoic acid,	56554-33-7	891	896	C25 H48 O2	2.0	380	✔	none	380	SI	55	348



ID: 23, Eicosanoic acid, methyl ester (C20:0)



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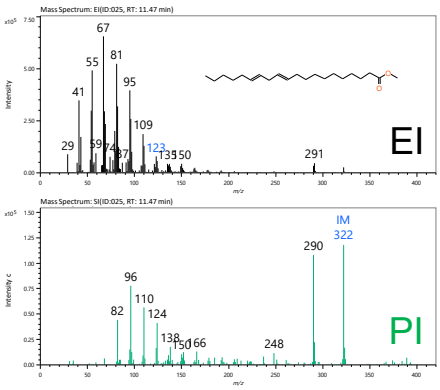

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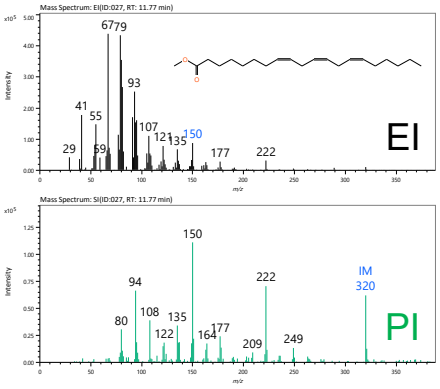

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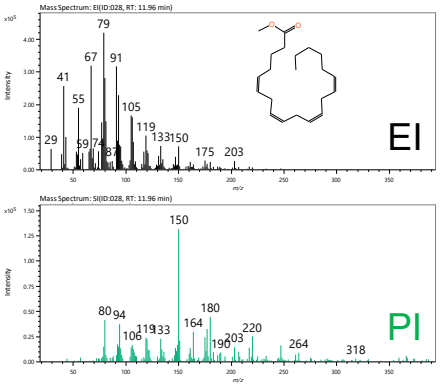

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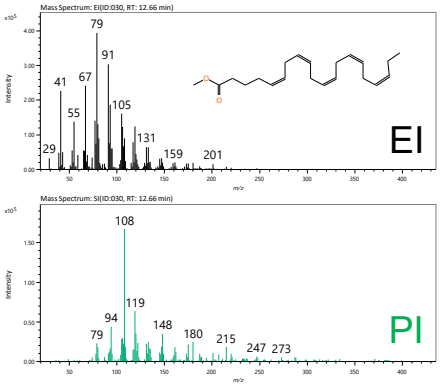

ID: 30, 5,8,11,14,17-Eicosapentaenoic acid, methyl ester, (all-Z)- (C20:5n3)

Fig.2 EI and PI mass spectra for C20 FAMEs.

Table 2 shows the integrated qualitative analysis result using msFineAnalysis iQ. We were able to improve the quality of the analysis results by the integrated qualitative analysis that combined the results of the library database search with the results of molecular ion confirmation. Both the soft ionization such as PI and the integrated qualitative analysis using msFineAnalysis iQ were shown to be effective in the qualitative analysis of compounds such as FAME whose molecular ions are difficult to obtain by EI.

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