GC/EI and PI Integrated analysis of water-based inks using msFineAnalysis iQ

Product used : Mass Spectrometer (MS)

Introduction

A gas chromatograph-quadrupole mass spectrometer (GC-QMS) is widely used as a qualitative/quantitative analysis device for volatile compounds, and is very useful as a technique for additive analysis.

Usually, qualitative analysis by GC-QMS is generally performed by library database (DB) search in the measurement data of electron ionization (EI) method. However, when qualitative analysis is performed using only the similarity index with the library spectrum, a plurality of significant candidates may be obtained depending on the compound, or an erroneous candidate may be selected as the identification result. Therefore, it is effective to confirm molecular ions by soft ionization (SI) method including photoionization (PI) method.

In this case, two types of measurement data, the EI method and the SI method, are obtained for a single sample, making data analysis more complicated. Therefore, an integrated qualitative analysis software that can quickly and automatically analyze the two types of data is desired. This is the reason why we have developed msFineAnalysis iQ.

In this MSTips, GC/MS measurements of water-based inks for commercial inkjet printers are performed, and the results of integrated qualitative analysis of the obtained measurement data using msFineAnalysis iQ are reported.

Experimental

A water-based ink (magenta) for inkjet printers was used as the sample. A GC-QMS (JMS-Q1600GC UltraQuad™ SQ-Zeta, manufactured by JEOL Ltd.) was used for the measurement. 1 µL of the undiluted sample was injected into the GC, and the EI method and the PI method were used as ionization methods. Table 1 shows the detailed measurement conditions. The analysis was performed using msFineAnalysis iQ (manufactured by JEOL Ltd.), an integrated qualitative analysis software dedicated to GC-QMS.

JMS-Q1600GC UltraQuad™ SQ-Zeta

Table 1 Measurement condition

GC	
Column	VF-5MS (Agilent Technologies) 30 m×0.25 mm I.D., df=0.25 µm
Injecter Temp.	320℃
Oven Temp.	40℃(1min)→ 10℃/min → 320℃ (3min)
Injection Mode	Split 200:1
Carrier Gas	He, 1.0 ml/min (Constant Flow)

MS	
Ionization Temp.	250℃
Interface Temp.	280℃
Ion Source	EI/PI Conbination Ion Source
Ionization Mode	EI (70 eV, 50 µA), PI (8~10 eV)
mode	Scan (*m/z* 15 - 600)

Results and Discussion

Figure 1 shows the GC/MS measurement results of water-based ink (magenta). The two peaks with early elution times were presumed to be water as solvent and isopropyl alcohol (IPA) as penetrant. Furthermore, a broad peak detected around 9 minutes retention time was presumed to be glycerin as a drying inhibitor. And then, ``Surfynol 104'', a type of surfactant intended for wetting/penetrating, foaming/defoaming functions in water-based ink, was also detected.

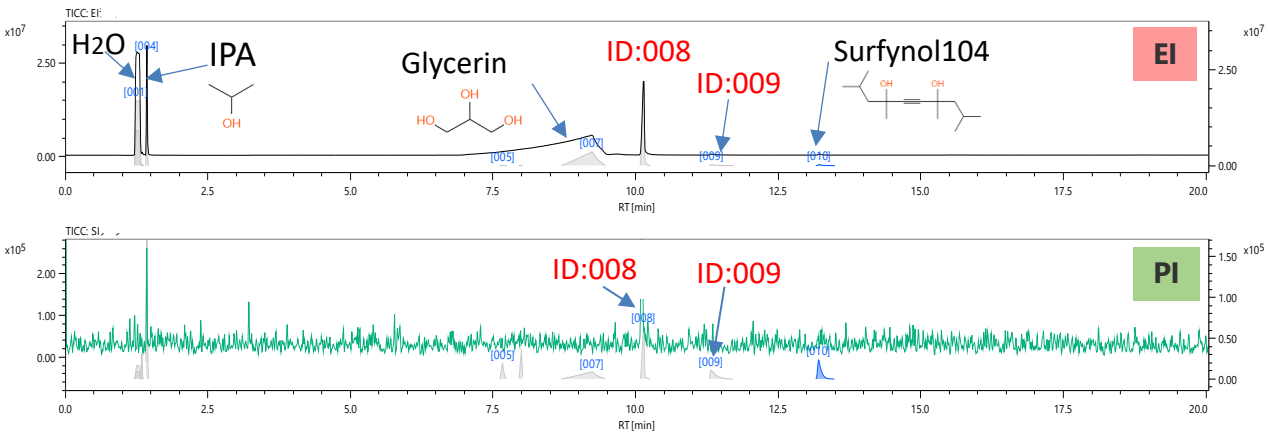


Figure 1 Total ion current chromatograms

Figure 2 shows the mass spectrum of the [ID:008] peak. A correlated mass spectrum was confirmed for both the EI method and the PI method. Table 2 shows the integrated analysis result list (top 5 candidates) by msFineAnalysis iQ. From this result, the compound of peak [ID:008] was estimated to be "Ethanol, 1-(2-butoxyethoxy)-". This compound was presumed to be a kind of solvent contained in water-based ink.

In addition, Figure 3 shows the EI mass spectrum of the peak [ID:009] and the result of isotope matching of the molecular ion (m/z 113) with "Caprolactam", which was the first hit in the library search. Table 3 shows the integrated analysis result list (top 5 candidates) by msFineAnalysis iQ. In the search results, highly accurate estimation results were obtained. This compound t was presumed to be “Caprolactam" which is a condensation monomer compound of water-soluble polyamide resin.

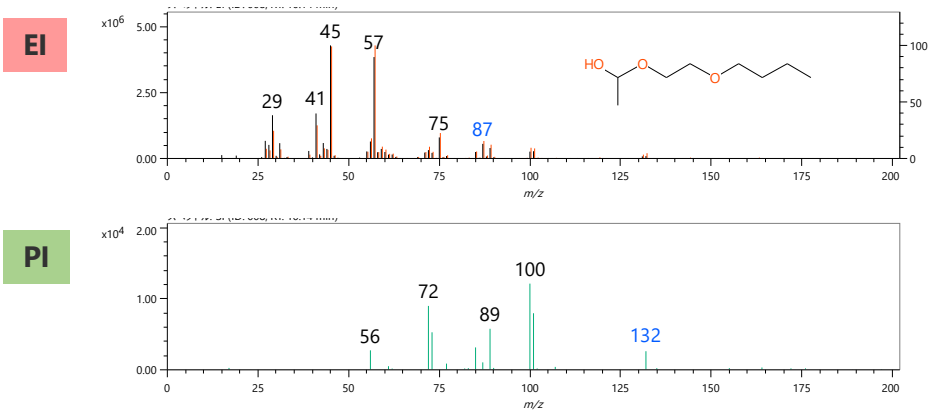
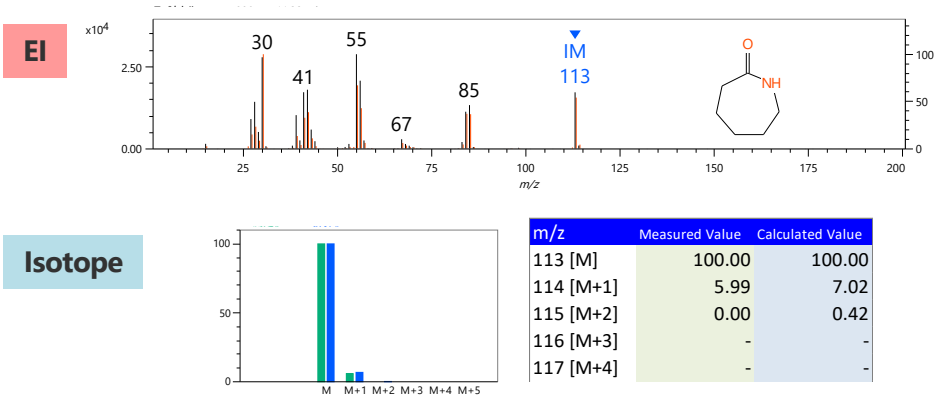


Figure 2 Mass spectra of peak [008]

Table 2 Integrated qualitative analysis result of peak [008]

#	Compound	CAS#	Similarity	Similarity (Reverse)	Lib. RI [iu]	ΔRI [iu]	Formula	EI Base Peak (Lib.)	Mw	Mw confirmation	Isotope Matching
L01	Ethanol, 1-(2-butoxyethoxy)-	54446-78-5	952	953	1187	2	C8 H18 O3	57	162	-	-
★ L02	Ethanol, 2-(2-butoxyethoxy)-	112-34-5	945	952	1192	3	C8 H18 O3	45	162	-	-
L10	Butane, 1-(2-methoxyethoxy)-	13343-98-1	749	771	577-1161	28	C7 H16 O2	57	132	✔	0.98
L22	2-Propanol, 1-(2-methylpropoxy)-	23436-19-3	682	691	510-1272	0	C7 H16 O2	57	132	✔	0.98
L03	2-Propanol, 1-(2-butoxyethoxy)-	124-16-3	830	834	849-1611	0	C9 H20 O3	57	176	-	-



m/z	Measured Value	Calculated Value
113 [M]	100.00	100.00
114 [M+1]	5.99	7.02
115 [M+2]	0.00	0.42
116 [M+3]	-	-
117 [M+4]	-	-

Figure 3 Mass spectra of peak [009]

Table 3 Integrated qualitative analysis result of peak [009]

#	Compound	CAS#	Similarity	Similarity (Reverse)	Lib. RI [iu]	ΔRI [iu]	Formula	EI Base Peak (Lib.)	Mw	Mw confirmation	Isotope Matching
★ L01	Caprolactam	105-60-2	931	931	1253	16	C6 H11 N O	30	113	✔	0.91
L02	Piperidine-2,5-dione	52065-78-8	733	733	673-1435	0	C5 H7 N O2	30	113	✔	0.95
L04	3,4-Dimethyl-isoxazol-5(4H)-one	15731-93-8	723	820	615-1377	0	C5 H7 N O2	55	113	✔	0.95
L05	3,4-Dimethyl-5-hydroxy-isoxazole	29279-99-0	708	808	519-1281	0	C5 H7 N O2	55	113	✔	0.95
L06	2-Oxepanone, 7-methyl-	2549-59-9	698	704	926-1326	0	C7 H12 O2	55	128	✔	0.93

Conclusion

In this report, an example of integrated qualitative analysis by msFineAnalysis iQ was reported for the purpose of compositional analysis of major constituents in water-based ink. msFineAnalysis iQ uses not only library DB search but also multiple identification functions such as retention index and isotope matching, so highly accurate qualitative analysis is possible. This software is expected to improve qualitative accuracy and efficient analysis work in GC-QMS analysis.

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