Analysis of Additives in Commercial Antibacterial Sheets by Combination of Thermal Desorption GC/EI and PI and msFineAnalysis iQ

Product used : Mass Spectrometer (MS)

Introduction

There are many plastic products around us, and they contain various additives according to their functionality. However, food-related plastic products have a direct impact on the human body and the environment, so the use of additives is restricted. Therefore, additive analysis is very important for purposes such as product quality control, cause investigation of molding defects and coloring, and market research in new product development.

A gas chromatograph-quadrupole mass spectrometer (GC-QMS) is widely used as a qualitative/quantitative analysis instrument 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 search 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 results. In this case, it is effective to confirm molecular ions by soft ionization (SI) method including photoionization (PI) method. However, two types of measurement data, the EI method and the SI method, are obtained for one sample, making data analysis more complicated.

We have developed an integrated qualitative analysis software that can quickly and automatically analyze the two data automatically. It is called "msFineAnalysis iQ" .

In this MSTips, thermal desorption GC/MS measurements of a commercial antibacterial lunch box product are performed and the integrated qualitative analysis results are reported using msFineAnalysis iQ.

Experimental

As a sample, a commercial antibacterial sheet for lunch boxes (made of polypropylene) containing the natural antibacterial compound "mustard extract" as a food additive was used. A GC-QMS (JMS-Q1600GC UltraQuad™ SQ-Zeta, manufactured by JEOL Ltd.) was used for the measurement. A pyrolyzer (PY-3030D, manufactured by Frontier Laboratories) was used for sample pretreatment, and the temperature of the heating furnace was raised from 50° C to 360° C at a rate of 20° C per minute. 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

Py	
Sample Volume	EI: 0.27mg, PI: 0.18mg
Furnace Temp.	50℃→20℃/min→360℃
Attachment	micro-JET Cryo Trap MJT-1030E

GC	
Column	ZB-1MS (Phenomenex) 30 m×0.25 mm I.D., df=0.25 μm
Injector Temp.	300℃
Oven Temp.	40℃→ 15℃/min → 340℃ (10min)
Injection Mode	Split 20:1
Carrier Gas	He, 1.0 ml/min (Constant Flow)

MS	
Ionization Temp.	250℃
Interface Temp.	280℃
Ion Source	EI/PI combination ion source
Ionization	EI (70 eV, 50 μA), PI (8~10 eV)
Measurement Mode	Scan (*m/z* 35 - 800)

Results and Discussion

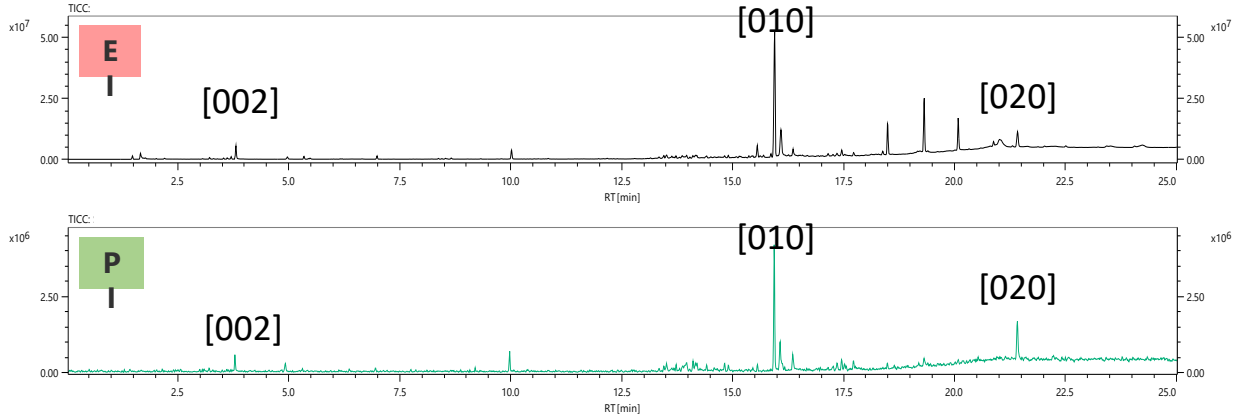

Figure 1 Total ion current chromatograms

Figure 1 shows the results of thermal desorption GC/MS measurement of commercial antibacterial sheets for lunch boxes. Figure 2 shows the mass spectrum of peak [010]. An ion with *m/z* 362, which is presumed to be a molecular ion, was clearly detected by the PI method, although the EI method showed a very small signal. Table 2 shows the integrated analysis result list (top 5 candidates) by msFineAnalysis iQ. From this result, the compound of peak [010] was estimated to be "Octicizer". This compound was presumed to be a plasticizer added to soften the polypropylene resin that is the base material of this sheet. Furthermore, Figure 3 shows the mass spectrum of peak [002]. An ion with *m/z* 99, presumed to be a molecular ion, was detected by both the EI and PI methods. Table 3 shows the integrated analysis result list (top 5 candidates) by msFineAnalysis iQ. "Ally Isothiocyanate", which ranked first in the search results, obtained highly accurate estimation results comprehensively, including not only similarity index but also retention index and isotope matching. This ingredient is a typical "mustard extract". In addition, peak [020] is a compound with a relatively large molecular weight of the molecular ion *m/z* 662, and was presumed to be "Tris(2,4-di-tert-butylphenyl) phosphate". This compound is a typical antioxidant used in food grade resins. In addition, many additive compounds were detected, such as the typical antioxidant "BHT", the ultraviolet absorber "Tricaprylin", and the natural resin rosin "Abietic acid" and its related compounds.

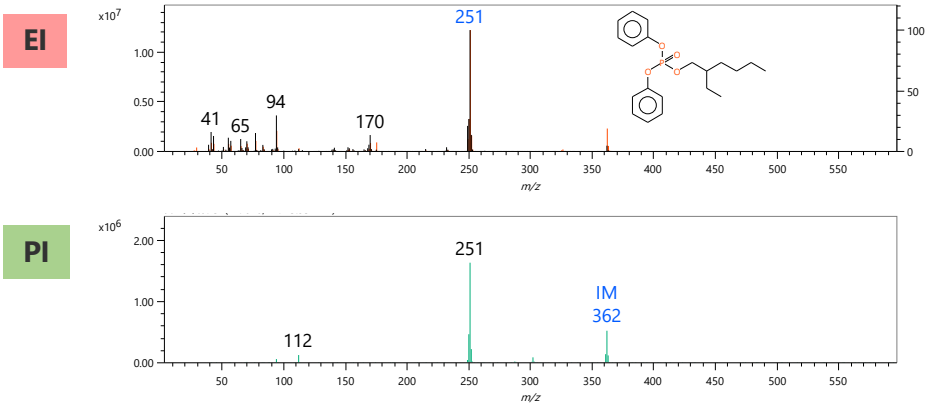

Figure 2 Mass spectra of peak [010]

Table 2 Integrated qualitative analysis result of peak [010]

	Compound	CAS#	Similality	Similality (Reverse)	Lib. RI [iu]	ΔRI [iu]	Formula	EI Base Peak (Lib.)	Mw	Mw confirmation	Isotope Matching
★	Octicizer	1241-94-7	881	881	2410	18	C20 H27 O4 P	251	362	✔	0.98
	Phosphoric acid, octyl diphenyl ester	115-88-8	835	844	N/A	N/A	C20 H27 O4 P	251	362	✔	0.98
	Phosphoric acid, butyl diphenyl ester	2752-95-6	734	751	N/A	N/A	C16 H19 O4 P	94	306	-	-
	N-(6-Bromo-quinolin-8-yl)-2,3,3,3-tetraf	-	584	640	1774-2536	0	C13 H9 Br F4 N2	249	380	✔	0.16
	(+)-1,2-Bis((2S,5S)-2,5-diethylphospholа	136779-28-7	541	547	2504	112	C22 H36 P2	251	362	✔	0.88
	N-(5-Bromo-quinolin-8-yl)-2,3,3,3-tetraf	-	514	561	1774-2536	0	C13 H9 Br F4 N2	249	380	✔	0.16

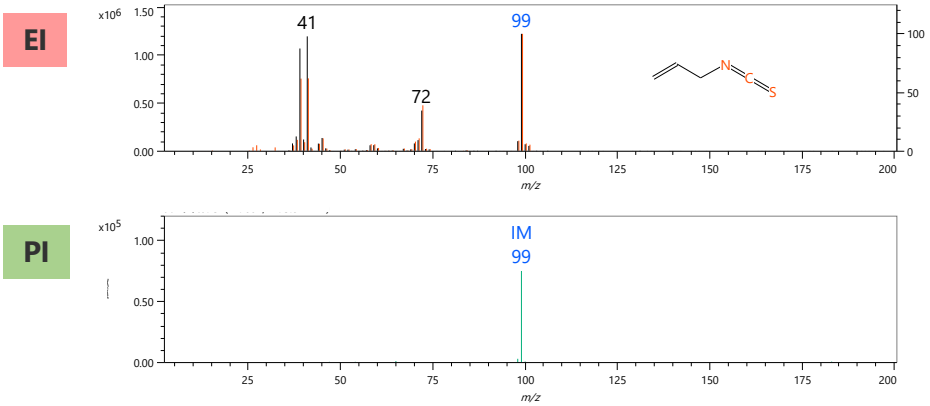

Figure 3 Mass spectra of peak [002]

Table 3 Integrated qualitative analysis result of peak [002]

	Compound	CAS#	Similality	Similality (Reverse)	Lib. RI [iu]	ΔRI [iu]	Formula	EI Base Peak (Lib.)	Mw	Mw confirmation	Addition/ Detachment	Isotope Matching
★	Allyl Isothiocyanate	57-06-7	970	970	885	25	C4 H5 N S	99	99	✔	無し	0.97
	Cyclopropane, isothiocyanato-	56601-42-4	874	874	N/A	N/A	C4 H5 N S	99	99	✔	無し	0.97
	Thiocyanic acid, 2-propenyl ester	764-49-8	755	823	524-1286	0	C4 H5 N S	41	99	✔	無し	0.97
	3-Methyl-5-hydroxy-isoxazole	45469-93-0	666	707	429-1191	0	C4 H5 N O2	41	99	✔	無し	0.74
	4-Methylthiazole	693-95-8	664	666	818	42	C4 H5 N S	99	99	✔	無し	0.97

Conclusion

In this report, an example of integrated analysis by msFineAnalysis iQ was reported for the purpose of qualitative analysis of various additive compounds in food plastic products. 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.

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