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Application News

No. M291

Gas Chromatography Mass Spectrometry

Application of Pyrolysis-GC/MS to Food Contamination Analysis

In recent years, cases of food contamination by foreign matter have increased, heightening the necessity of contamination analysis by food manufacturers. The Fourier transform infrared spectrometer (FTIR) and energy dispersive X-ray fluorescent spectrometer (EDX) are used in identification of contaminants by instrumental analysis. The pyrolysis-GC/MS method and thermal extraction-GC/MS method are employed in contamination analysis by GC/MS, and enable qualitative analysis of resin materials and additives contained in trace organic contaminants.

This article introduces the results of an analysis of the resins in a food packaging material using the pyrolysis-GC/MS method, assuming food contamination. A Shimadzu OPTIC-4 multimode inlet for GC/MS was used in the analysis by pyrolysis-GC/MS. Because the OPTIC-4 enables high-speed heating (60 °C/s) to a maximum temperature of 600 °C, diverse sample injection modes are available and simple pyrolysis is possible.

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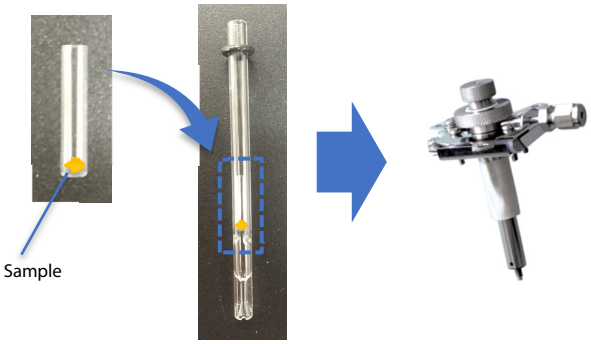
Fig. 1 GCMS-QP™2020 NX + OPTIC-4

Sample and Analysis Conditions

Fig. 1 shows the appearance of the GCMS-QP™2020 NX and OPTIC-4 multimode inlet used in the analysis.

A commercially-available food packaging material was used as the real sample material. The sample material was cut with a cutter knife to obtain a sample weighing approximately 0.2 mg, which was inserted in the DMI microvial of the OPTIC-4 and then set in the DMI insert liner (Fig. 2).

Table 1 shows the instrument system and analysis conditions.



Set DMI microvial containing sample in insert liner.

Analyze with OPTIC-4.

Fig. 2 Sampling Procedure for Pyrolytic Analysis Using OPTIC-4

Table 1 Analysis Conditions

Inlet	:	OPTIC-4
GC/MS	:	GCMS-QP2020 NX
Column	:	UA-5 (MS/HT)-30M-0.25F (L30 m, 0.25 mm I.D., df=0.25 µm) (Frontier Laboratories Ltd.)
OPTIC-4 conditions		
Vent time	:	30 s
Equilibrium time	:	5 s
End time	:	40 min
Injection temperature	:	40 °C (10 s) → (60 °C/s) → 600 °C (3 min) → 320 °C (hold)
Carrier gas	:	Helium
Carrier control mode	:	Flow control
Start column flow	:	1.0 mL/min
End column flow	:	1.0 mL/min
Split flow	:	100 mL/min
Septum purge flow	:	3 mL/min
GC conditions		
Column oven temp	:	40 °C (2 min) → 20 °C/min → 320 °C (16 min)

MS conditions	
Interface temp.	: 280 °C
Ion source temp.	: 230 °C
Ionization method	: EI
Measurement mode	: Scan (*m/z* 29 - 800)
Event time	: 0.3 s

Qualitative Analysis of Resin Material

Fig. 3 shows the obtained pyrogram (total ion chromatogram obtained by pyrolysis-GC/MS). From a reference [1] containing pyrolysis data on resins, this is a distinctive pyrogram of polyethylene (PE), in which hydrocarbon species are arranged at equal intervals. Therefore, it could be inferred that the foreign matter in this experiment contained PE as the base material.

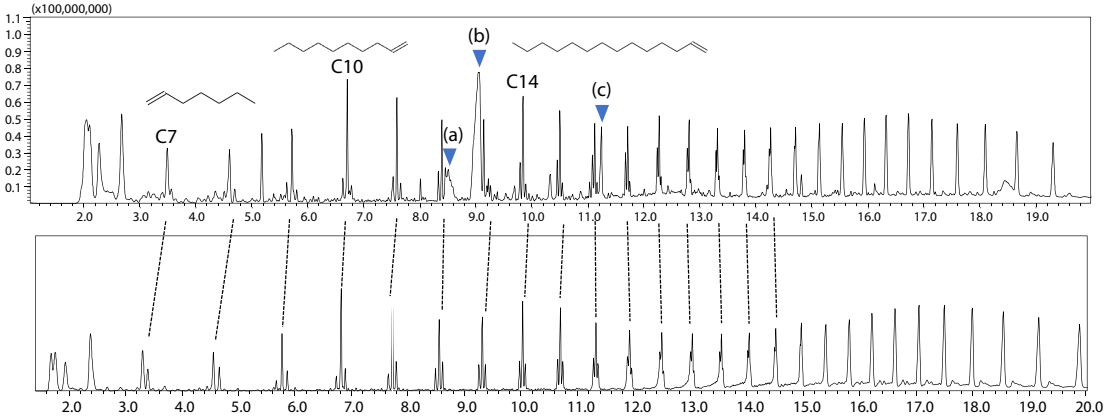


Fig. 3 Top: Pyrogram of Analyzed Foreign Matter, Bottom: Pyrogram of PE

In addition to the peaks seen in the pyrogram of PE, three distinctive peaks ((a) to (c)) were also detected in the pyrogram of the real sample. Compound identification of these peaks was carried out using the NIST Library and the above-mentioned reference [1]. As a result, it was found that (b) is caprolactam, a compound characteristically seen as a pyrolysis product of polyamide (PA), and (a) and (c) were identified respectively as 4-(vinyloxycarbonyl) benzoic acid and benzoic acid, which are compounds characteristically seen as pyrolysis products of polyethylene terephthalate (PET). Based on these results, the foreign matter measured in this experiment was estimated to be a composite resin containing polyamide (PA) and polyethylene terephthalate (PET) in addition to polyethylene (PE).

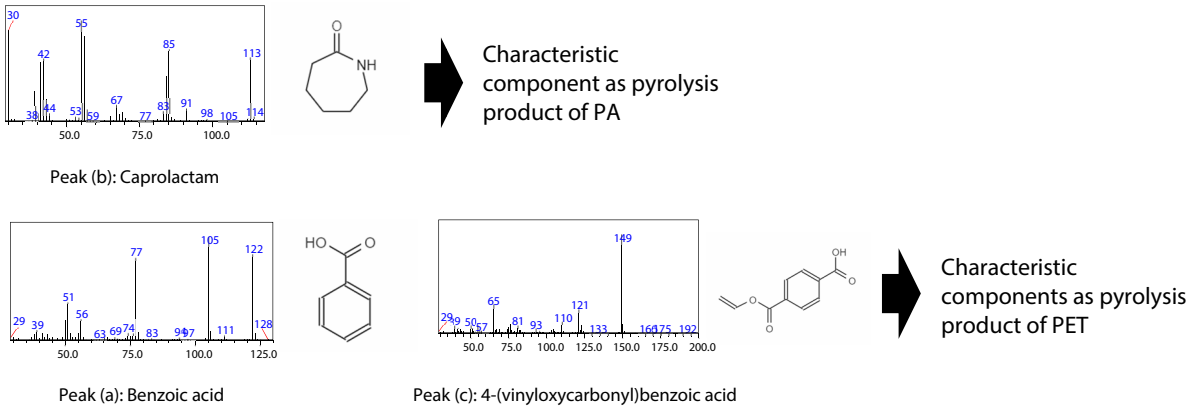


Fig. 4 Mass Spectra of Peaks (a) to (c) and Identified Compounds

Conclusion

As an analysis of contaminants in food products, the resins contained in an assumed foreign matter sample were analyzed by the pyrolysis-GC/MS method in an OPTIC-4 multimode inlet. As a result, qualitative analysis of the composite resin was possible from the pyrogram and pyrolysis products. Thus, this experiment demonstrated the possibility of qualitative analysis of resin materials by using the pyrolysis-GC/MS method, including analysis of trace contaminants and contaminants in multilayer films, which are difficult to analyze by FTIR. By using this analysis technique, it is considered possible to identify the source of contamination and take appropriate countermeasures.

<Reference>
(1) S. Tsuge, H. Ohtani, C. Watanabe: Pyrolysis-GC/MS Data Book of Synthetic Polymers – Pyrograms, Thermograms and MS of Pyrolyzers – , 1st Edition, Elsevier, 420 (2011)

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