

Application of Thermal Extraction-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 additives in a food packaging material by the thermal extraction-GC/MS method, assuming food contamination. A Shimadzu OPTIC-4 multimode inlet for GC/MS was utilized in the analysis by thermal extraction-GC/MS, and the Shimadzu Polymer Additives Library, a GC/MS mass spectra library, was used in the qualitative analysis of the additives.

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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). In calculations of the retention indices of the detected peaks, we used the retention time of n-alkane (a hydrocarbon species) obtained in the analysis of polyethylene (PE) by the pyrolysis-GC/MS method (For the analysis conditions, refer to Application News No. M291.).

Table 1 shows the instrument system and analysis conditions.

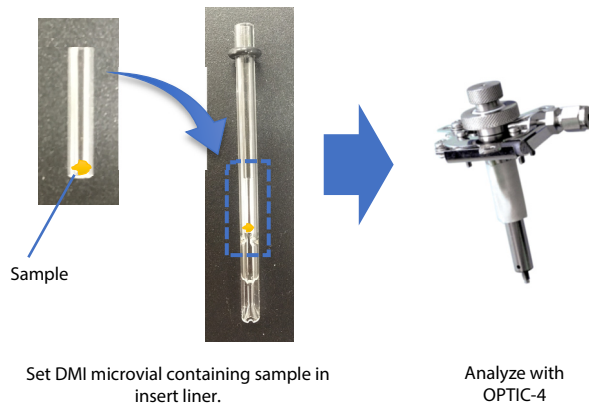


Fig. 2 Sampling Procedure for Thermal Extraction Analysis Using OPTIC-4

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) → 340 °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		

Results

Fig. 3 shows the total ion chromatogram (TIC) obtained by thermal extraction-GC/MS. A library search of the obtained peaks was carried out using the Polymer Additives Library, which contains the mass spectra of a wide range of additives used in polymer materials and the decomposition products of the additives. Because information on retention indices is registered for all compounds, compounds can be filtered by using the retention index, enabling compound identification with a high degree of accuracy. Information on the

classification of additives is also registered, allowing the user to identify the additives associated with compounds found in the library, even without a detailed knowledge of additives.

Fig. 4 shows the peaks for produced compounds hit in the search with the Polymer Additives Library. Multiple antioxidants and their degradation products (fragments) were identified. In this process, highly accurate qualitative analysis of additives was possible by filtering using the retention index.

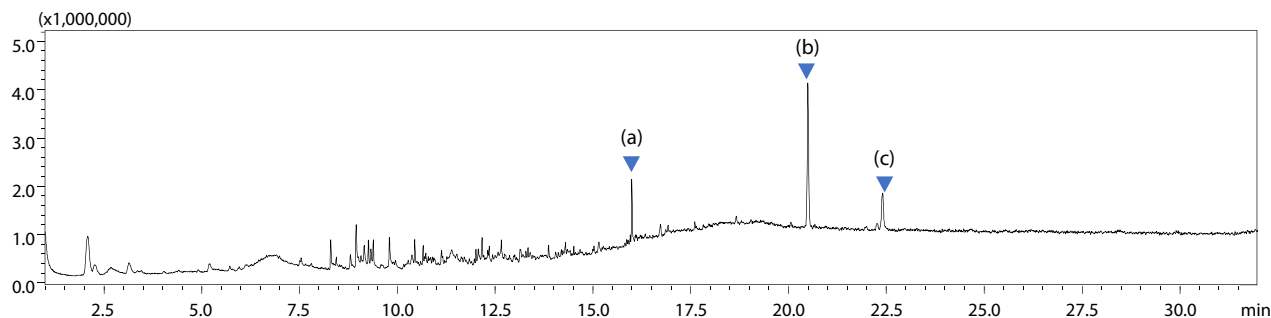
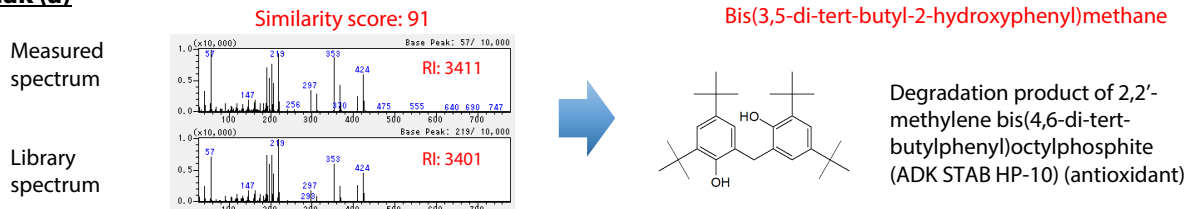
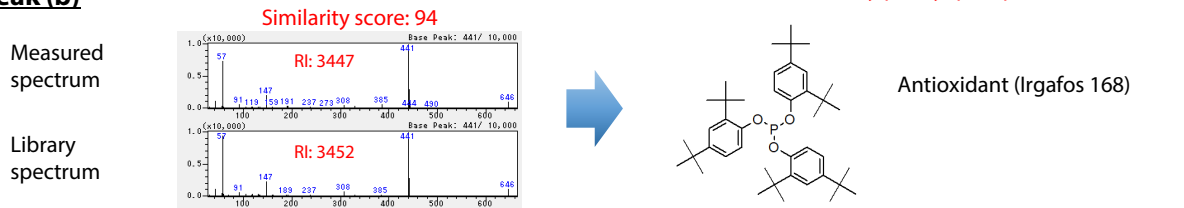


Fig. 3 TIC Obtained by Thermal Extraction-GC/MS Method

Peak (a)



Peak (b)



Peak (c)

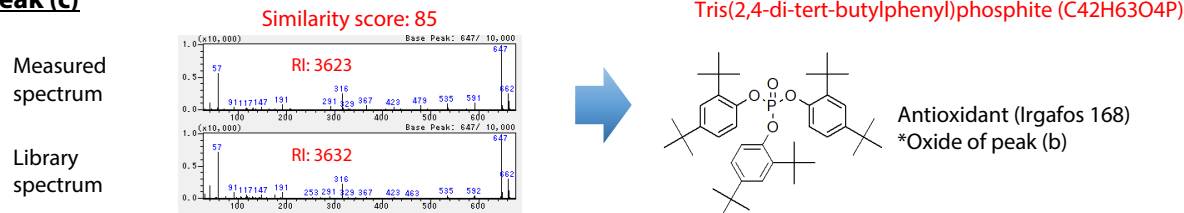


Fig. 4 Results of Qualitative Analysis of Peaks (a), (b), and (c) by Polymer Additives Library

Conclusion

An analysis of additives contained in a food product contaminant was conducted using the thermal-extraction GC/MS method with an OPTIC-4 multimode inlet and the Polymer Additive Library. As a result, the additives in a resin

could be identified with good accuracy. By using the analysis technique described here, it is considered possible to identify the source of contaminants and take appropriate measures.

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