

Analysis of brominated flame retardants in polypropylene products by FD method using JMS-T2000GC AccuTOF™ GC-Alpha

Related products: Mass spectrometer (MS)

Introduction

JMS-T2000GC AccuTOF™ GC-Alpha provides advanced analysis results with high throughput by high-resolution TOFMS, multi-ionization mode and automatic analysis software msFineAnalysis. Pyrolysis-GC-MS method is widely used in material analysis using GC-MS, in MSTips No. 330, the result of difference analysis between the non-defective product and the defective product in the PP / PE copolymer product was shown. On the other hand, in the analysis of additives in materials, there are high boiling point components that are difficult to pyrolyze and cannot pass through the column, and components that are difficult to estimate the previous structure after pyrolysis. The FD method is effective for these components. This report shows the result of analysis for two types of polypropylene (PP) products. The difference between sample was not found by the pyrolysis method, but a brominated flame retardants was found as a difference by the FD method.

Overview of FD method

Figure 1 shows a schematic diagram of the FD method. In the FD method, the sample is diluted with a solvent and then dropped onto an emitter probe to be introduced into an ion source. At this time, the sample does not need to be completely dissolved in the solvent. In this case, it is possible to analyze only the additives in the solvent. Since it is a direct sample injection method without using a column, high boiling point (high mass) components can be measured. Since it is soft ionization, molecular ions can be detected. By using the EI/FI/FD combination ion source, it is possible to continuously measure the pyrolysis method and the FD method without releasing the vacuum and exchanging the ion source.

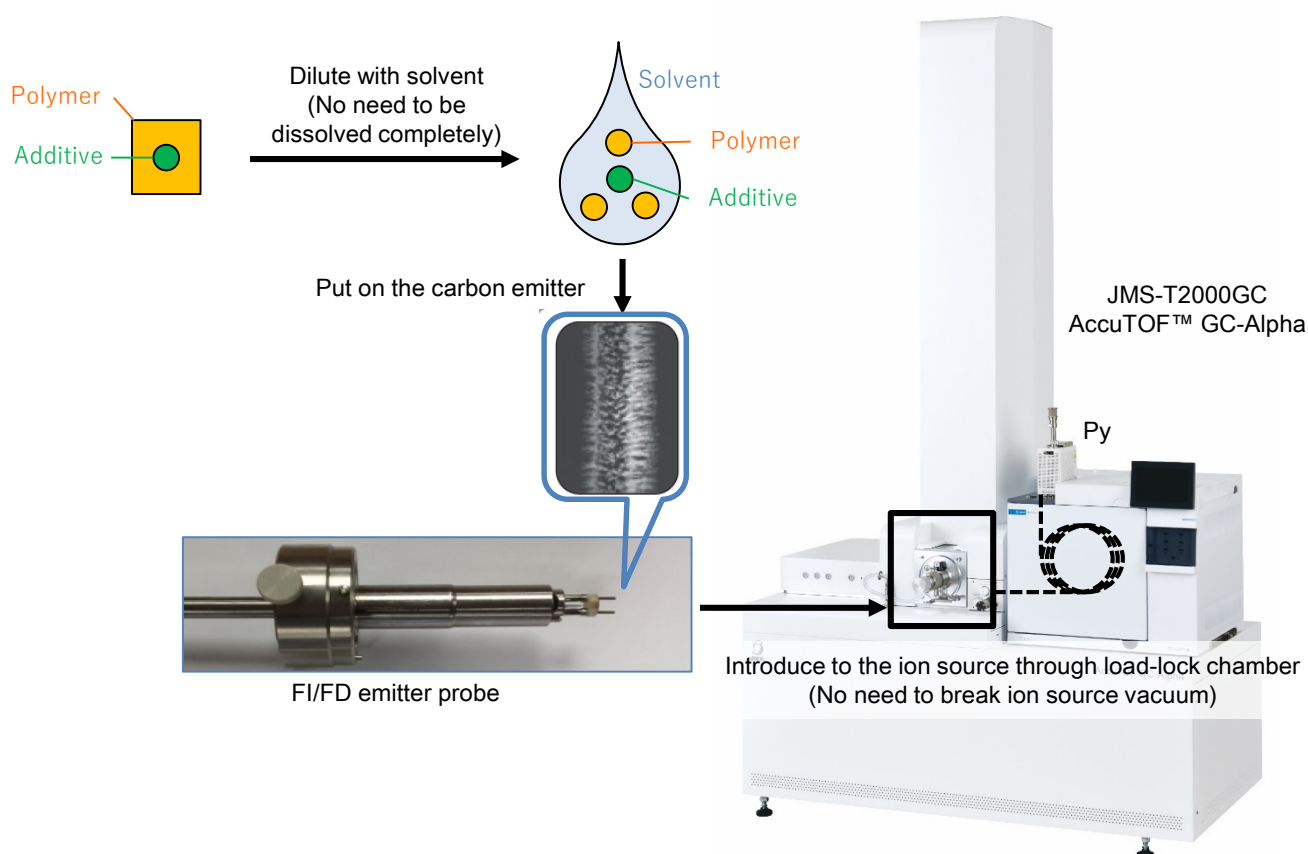


Figure 1. Schematic diagram of the FD method

Experiment

Table 1 shows the details of the measurement conditions in this experiment. Two types of commercially available PP products (flame-retardant and non-flame-retardant) were used as samples. The flame retardant was not detected as a different component between samples in the pyrolysis method. The measurement by the FD method is as short as 1 minute per sample. A high voltage of -10 kV is applied to the emitter, which extracts electrons from the sample molecule and soft-ionizes them. After the start of measurement, the emitter current is gradually increased to assist ionization, and finally the residue is burned out at a high temperature. Therefore, the conditioning operation is not required after the measurement, and the next sample can be measured immediately.

Table 1. Measurement conditions

Sample	2 types polypropylene products (Flame-retardant, non-flame-retardant)
Preprocessing	Dilute 1mg sample with acetone after freezing and grinding
MS	JMS-T2000GC AccuTOF™ GC-Alpha
Ion source	EI/FI/FD combination ion source
Ionization	FD method, Cathode voltage -10kV, Emitter current 0→51.2mA/min→40mA,
Mass range	m/z 50~1,600

Results

Figure 2 shows the mass spectra of the FD measurement results. A clear difference peak was detected at m/z 943.4797 from the flame-retardant PP. It was estimated to be a brominated flame retardant by isotope pattern analysis and composition estimation. Furthermore, analysis was performed using the analysis software Mass Mountaineer (RBC Software). This software automatically performs peak assignment and composition estimation from the mass spectrum. The screening analysis of various components is possible using the composition formula list. In this results, some antioxidants were hit from the composition formula list of additives.

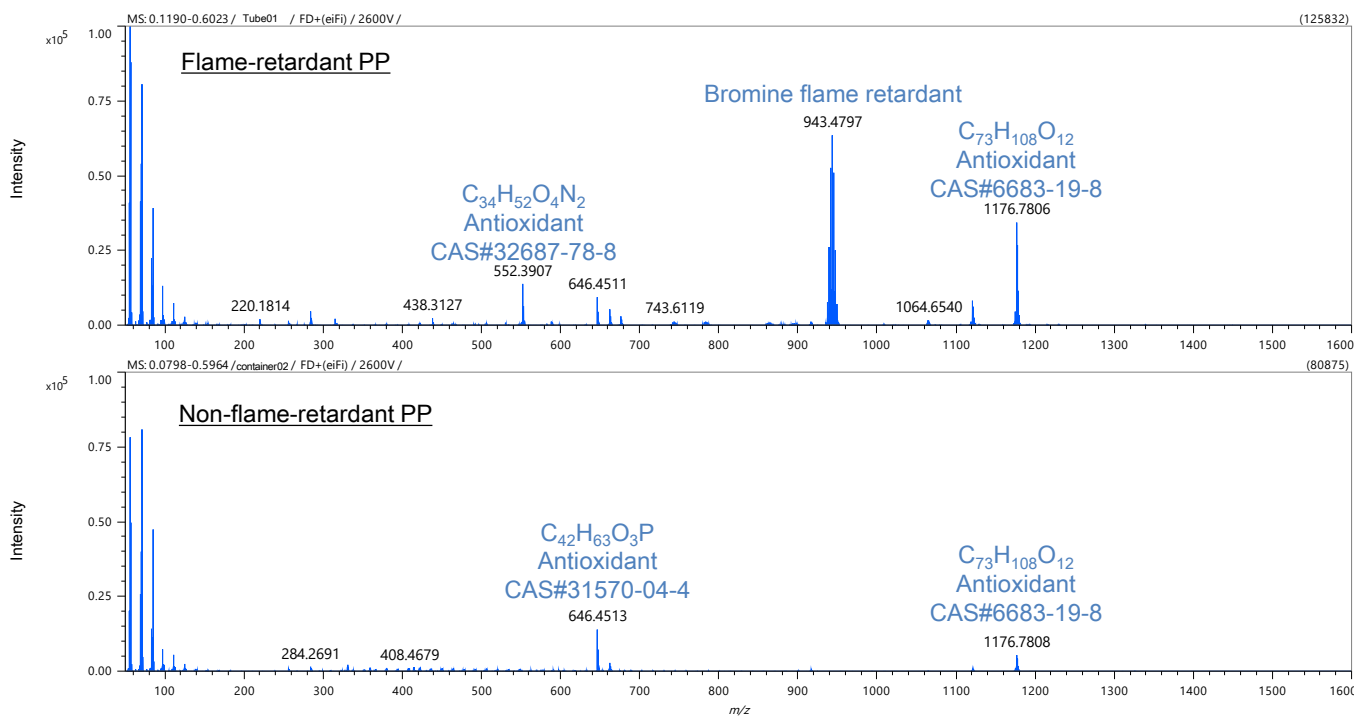


Figure 2. Mass spectra by FD method

Conclusion

FD method of JMS-T2000GC AccuTOF™ GC-Alpha can detect additives that may be missed by the pyrolysis method. Since both method have high affinity, they are expected to be a new analysis flow in material analysis.

Copyright © 2022 JEOL Ltd.

Certain products in this brochure are controlled under the "Foreign Exchange and Foreign Trade Law" of Japan in compliance with international security export control. JEOL Ltd. must provide the Japanese Government with "End-user's Statement of Assurance" and "End-use Certificate" in order to obtain the export license needed for export from Japan. If the product to be exported is in this category, the end user will be asked to fill in these certificate forms.



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

• AUSTRALIA & NEW ZEALAND • BELGIUM • BRAZIL • CANADA • CHINA • EGYPT • FRANCE • GERMANY • GREAT BRITAIN & IRELAND • ITALY • KOREA • MALAYSIA • MEXICO • RUSSIA • SCANDINAVIA
• SINGAPORE • TAIWAN • THE NETHERLANDS • USA

Local office

