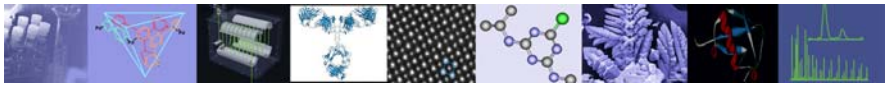

AccuTOF-GCv Series

Analysis of Additives in Plastic by Thermal Desorption (TD)

Accurate Mass Measurement and Isotope Pattern Matching

Introduction

Among the various methods used for characterizing plastics, pyrolysis (Py) GC/MS and thermal desorption (TD) GC/MS are widely used for both qualitative and quantitative analyses. These are simple techniques that provide detailed information about the samples.
In this application note, we report the analysis of additives in plastic by using a thermal desorption system and a JEOL JMS-T100GCV "AccuTOF GCv" GC-TOFMS. Identification of the analytes was accomplished by library search and accurate mass measurement. Additionally, isotope cluster pattern matching was performed using the "Mass Spec Tools™" software to help identify an unknown compound that was present in the sample.

Methods

Sample: Plastic (0.4 mg)

Thermal desorption
Instrument: PY-2020iD (Frontier Laboratories Ltd., Fukushima, Japan)
Temperature: 150 °C → 10 °C/min → 350 °C (20 minutes total)

GC
Instrument: 6890N (Agilent)
Column: DB1-HT, 8 m x 0.25 mm x 0.1 µm

MS
Instrument: JMS-T100GCV (JEOL)
Ionization mode:
EI(+): Electron energy: 70 eV
Ionization current: 300 µA
Acquired mass range: *m/z* 35 – 1,400
Spectral recording interval: 0.4 sec

Software: Mass Spec Tools™ (ChemSW, Inc., Fairfield, CA, U.S.A.)

Results and Discussion

The TIC chromatogram is shown in Fig. 1. Several of the peaks were identified by the NIST mass spectral library search as squalene, decabromodiphenyl ethane (a brominated flame retardant), and Irganox® 1330 (an antioxidant). While this NIST search worked well for identifying several compounds in the TICC, there was an "unknown" component just prior to the Irganox® 1330 peak that could not be identified by the library search (see Fig. 1 inset). The mass spectra of Irganox® 1330 and the unknown component are shown in Fig. 2. Although the *m/z* values for the major peaks in the spectra are different, the fragmentations observed among these peaks were very comparable, suggesting that they have similar structures. The mass spectrum of this unknown component was then further analyzed using the "Mass Spec Tools Elcomp™" software to determine the possible elemental compositions for the observed peaks.

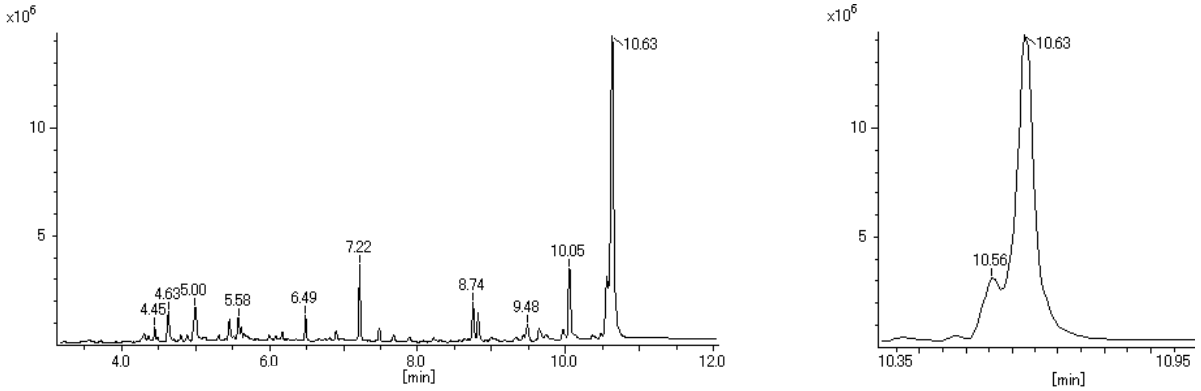

Fig. 1 TIC chromatogram

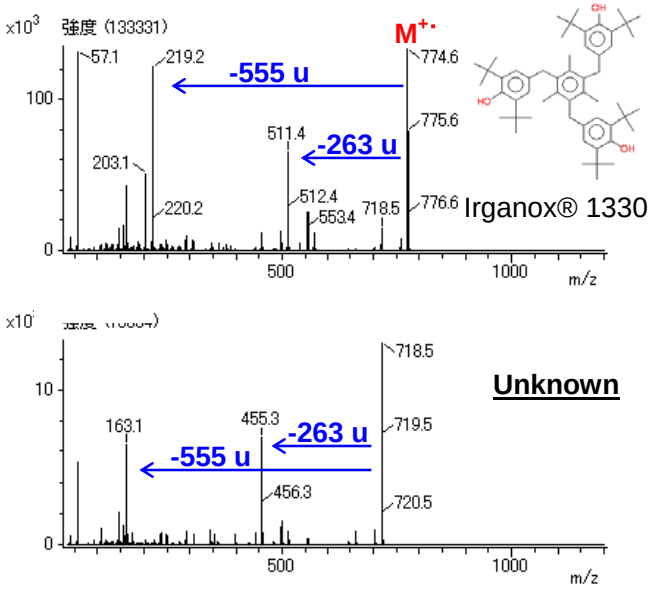

Fig. 2 Mass spectra of Irganox® 1330 (top) and unknown component (bottom)

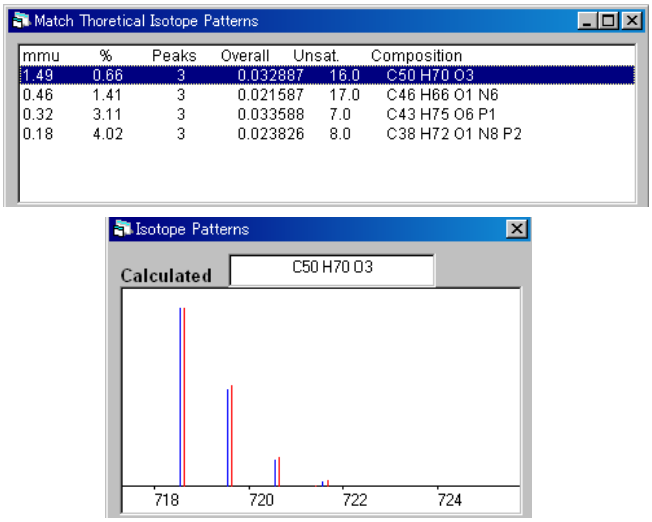

Fig. 3 Analysis results table (top) and isotope patterns (bottom)

The elemental composition of the unknown compound was calculated based on the observed exact mass by using a combination of C, H, O, N, and P, even though the spectral similarities suggested that it likely consisted of only C, H, and O. There were 21 candidates calculated that were within the error range of ± 5 mmu. By using isotope pattern matching, this total was narrowed down to 4 possibilities, as shown in the top window of Fig. 3. Among them, $C_{50}H_{70}O_3$ had the smallest matching error (the smaller the "%" in Fig. 3, the better the match) and indeed showed the best match between the measured and calculated isotope patterns, as shown in the bottom window of Fig. 3. The structure of the unknown was identified as an analog of Irganox® 1330 in which one of the t-butyl groups has been substituted with a hydrogen, which is also consistent with the fragmentation information observed for both compounds in Fig. 2.

Conclusions

The JEOL "AccuTOF GCv" GC-TOFMS used in conjunction with thermal desorption is a powerful tool for identifying compounds found within plastics. Additionally, AccuTOF GCv analyses result in library searchable fragmentation data, accurate mass measurements, and isotopic pattern data that are useful for identifying compounds present in these types of samples.