SHIMADZU

Application News

Liquid Chromatography Mass Spectrometry No.C71

Analysis of Polymer Additives Using LCMS-2020

Polymer materials such as plastic and rubber have become indispensable in our daily lives. Additives such as antioxidants and ultraviolet absorbers are added in minute quantities to polymers to affect specific properties. Conducting qualitative and/or quantitative analysis of the additives in these polymers makes it possible to obtain information pertaining to mixing technology and new additives.
Here we present an example of qualitative analysis of several types of polymer additives included in commercial food containers and packaging materials using the LCMS-2020.

■ In-Source CID Analysis of Polymer Additives using the LCMS-2020

Typically, structural analysis is conducted using an MS/MS instrument, but due to the high voltage that can be applied to the lens system of the single quadrupole LCMS-2020, this instrument can be used not only to obtain molecular weight information, but molecular structure information as well. Fig. 1 shows the chromatograms of 14 polymer additive substances, and Fig. 2 and Fig. 3 show the in-source CID mass spectra for Irganox 245 and Irganox 1010, respectively. The molecular structures were obtained using the deprotonated molecule observable using the ESI negative mode, and -100 to -150 V applied to DL and Q-array DC.

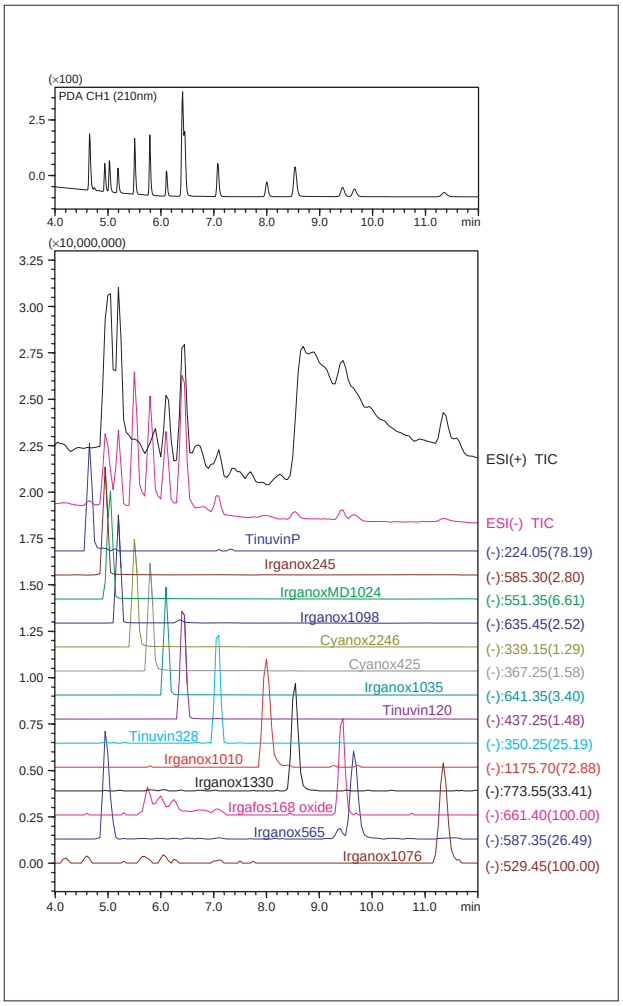

Fig. 1 Chromatograms of 14 Polymer Additives

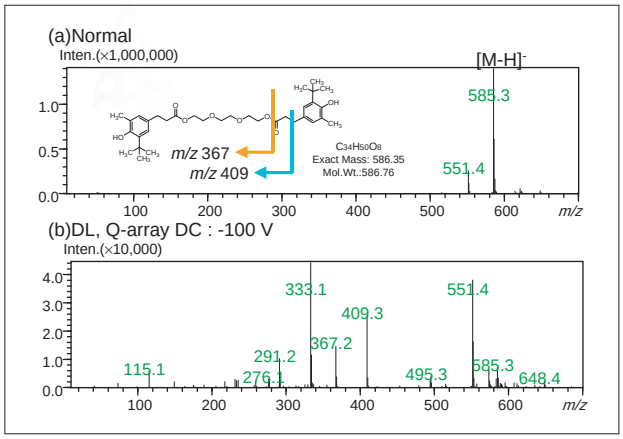

Fig. 2 Mass Spectra of Irganox 245

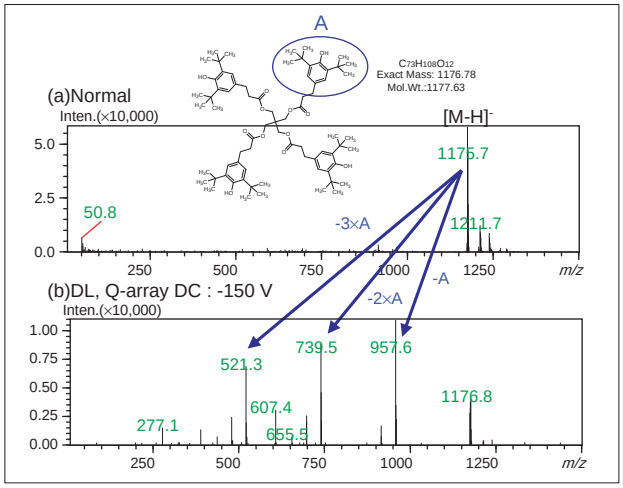

Fig. 3 Mass Spectra of Irganox 1010

■ Analysis of Unknown Polymer Additives Using LCMS-2020

After cutting a plastic food container into very fine pieces, 1 mL of THF/MeOH was added to 0.1 g of the container material and placed in an ultrasonic bath for 30 minutes. The extract obtained was used as the sample. Fig. 4 shows the chromatograms obtained from measurement of the extract, and peaks determined to be ① Cyanox 425, ② Irganox 1010, and ③ Irgafos 168 oxide based on their retention times and *m/z* values were detected. Peak ② at retention time 8 minutes shows the same CID mass spectrum as that of Fig. 3, and was therefore identified as Irganox 1010.

Fig. 5 shows the CID mass spectra of Irgafos 168 oxide and Fig. 6 shows the CID mass spectra of peak ③ at retention time 9.4 minutes. The deprotonated molecular ion $[M-H]^-$ at *m/z* 661could not be observed in Fig. 6 (C) due to contaminant interference. However, peak ③ was ultimately able to be identified as Irgafos 168 oxide because it has the same CID mass spectrum as the standard, demonstrating the usefulness of CID analysis.

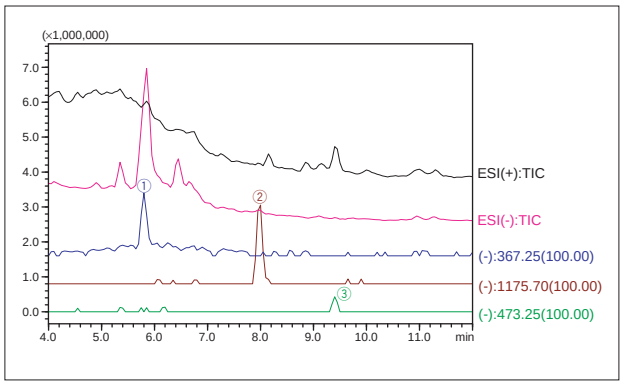

Fig. 4 Mass Chromatograms of THF/Methanol Extract of Plastic Food Container

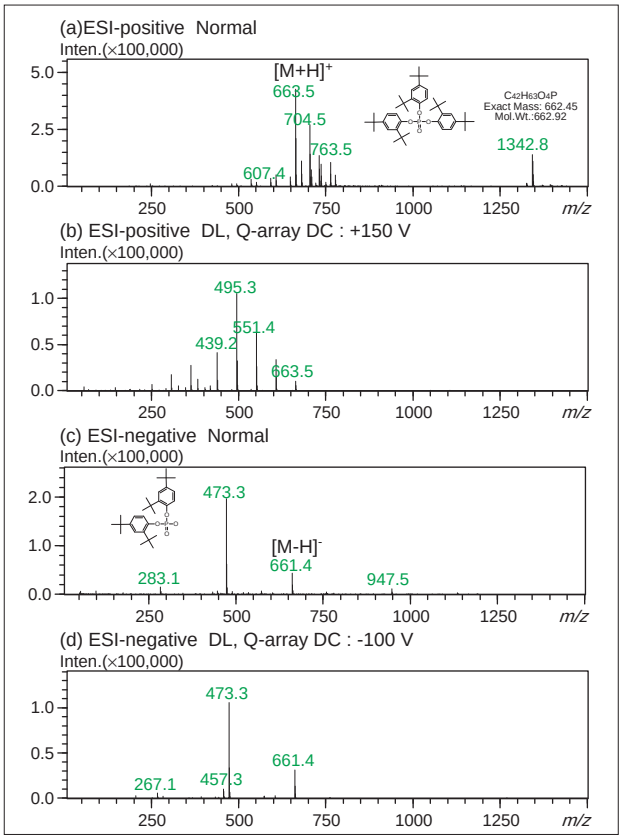

Fig. 5 Mass Spectra of Irgafos 168 Oxide

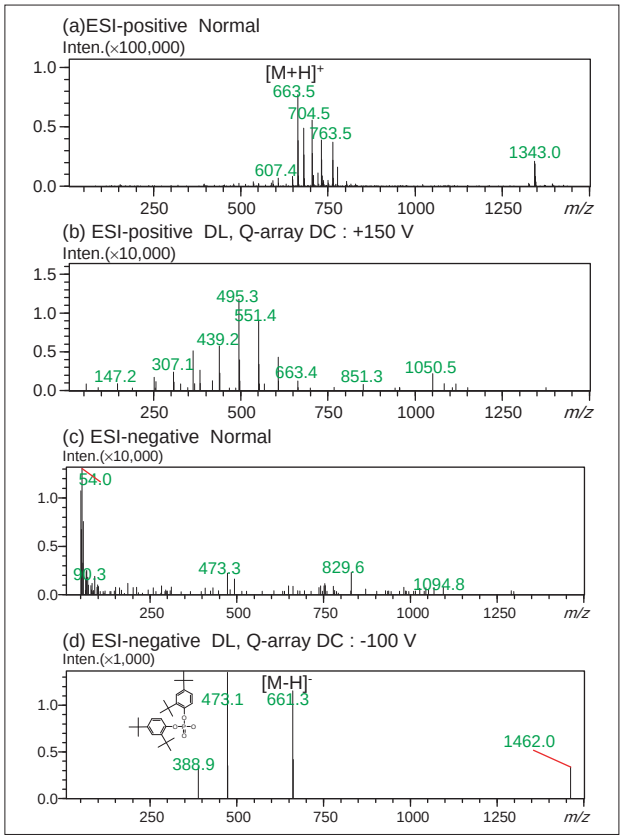

Fig. 6 Mass Spectra of Peak ③

Table 1 Analytical Conditions

Column	: Shim-pack XR-ODS (75 mmL. × 2.0 mmI.D., 2.2 µm)	Probe Voltage	: +4.5 kV (ESI-Positive mode), -3.5 kV (ESI-Negative mode)
Mobile Phase A	: 5 mmol/L ammonium acetate-water	Nebulizing Gas Flow	: 1.5 L/min
Mobile Phase B	: acetonitrile	Drying Gas Flow	: 10 L/min
Gradient Program	: 25 %B (0 min) - 100 %B (5-15 min) - 25 %B (15.01 - 20 min)	DL Temperature	: 250 ˚C
Flow Rate	: 0.5 mL/min	Block Heater Temperature	: 450 ˚C
Injection Volume	: 2 µL		
Column Temperature	: 40 ˚C		

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Printed in Japan 3295-06007-10A-IK