SHIMADZU

LC-MS Application Data Sheet No. 048

Simultaneous Analysis of Irganox Polymer Additive using LC-MS

Irganox, a polymer stabilizer (an additive) with antioxidant and heat stabilization effects, is used for a variety of polymer products. Irganox additives are sometimes used as mixtures to add new properties. A lot of knowledge goes into understanding what types of additives should be used in what ratio.

Irganox has 3,5-di-tert-butyl-4-hydroxyphenyl structures and can be ionized by negative ion electrospray (ESI-negative). In general, to improve the ionization efficiency in the ESI-negative mode, neutral or basic mobile phase conditions are suitable. However, when using a reversed-phase column for separation, Irganox's strong retention to the column causes failure in the elution of target components or serious peak deformation without addition of acid. To solve this problem, in this example, a GPC column was used to prevent component adsorption and ionization was conducted under neutral mobile phase conditions.

Fig. 1 shows the mass chromatograms of a mixture of eleven Irganox components. Because each component clearly provides a deprotonated molecule, they can be easily identified by mass data and retention times. This data can also be used to check for impurities in the additive. Peak (3) is an impurity in the Irganox. The mass spectra of typical Irganox compounds are shown in Fig. 2. The combination of GPC mode separation and MS detection is a suitable method to analyze Irganox, as well as other polymer additives.

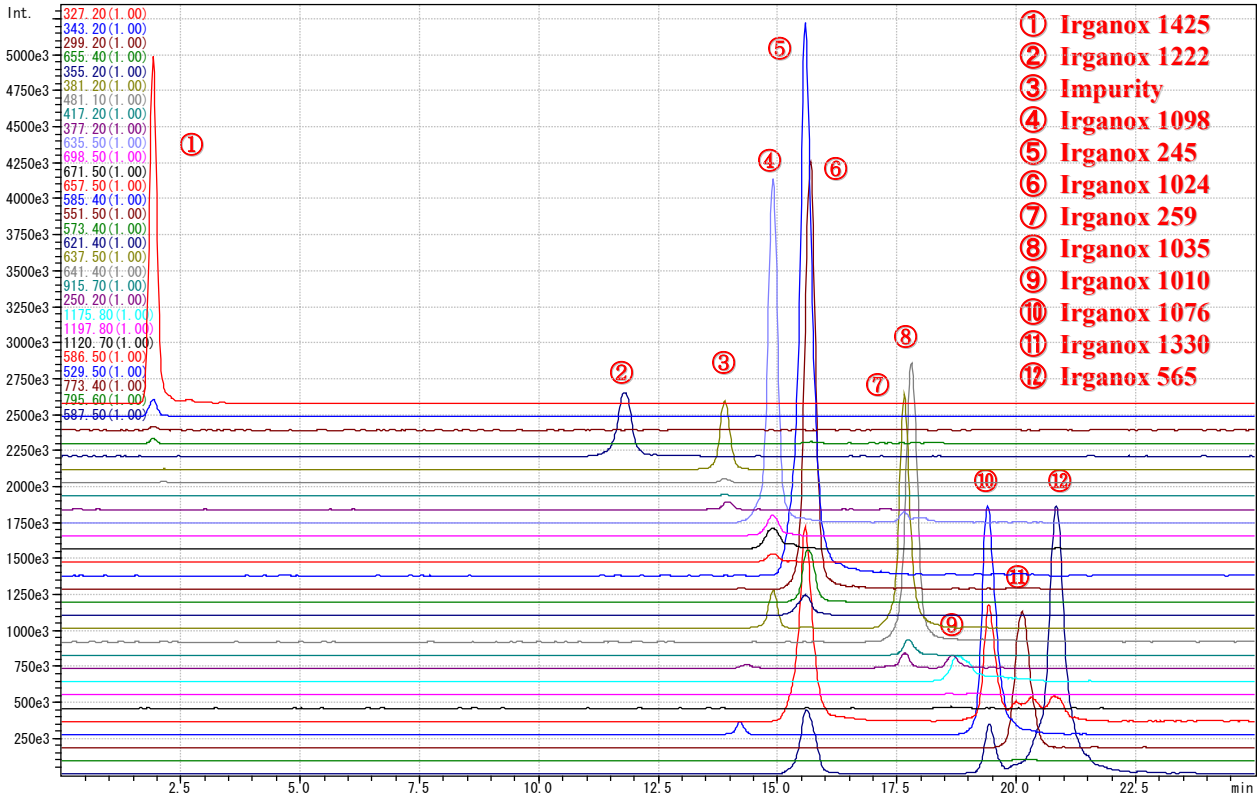

Fig. 1 Mass Chromatograms of Irganox mixture using GPC column

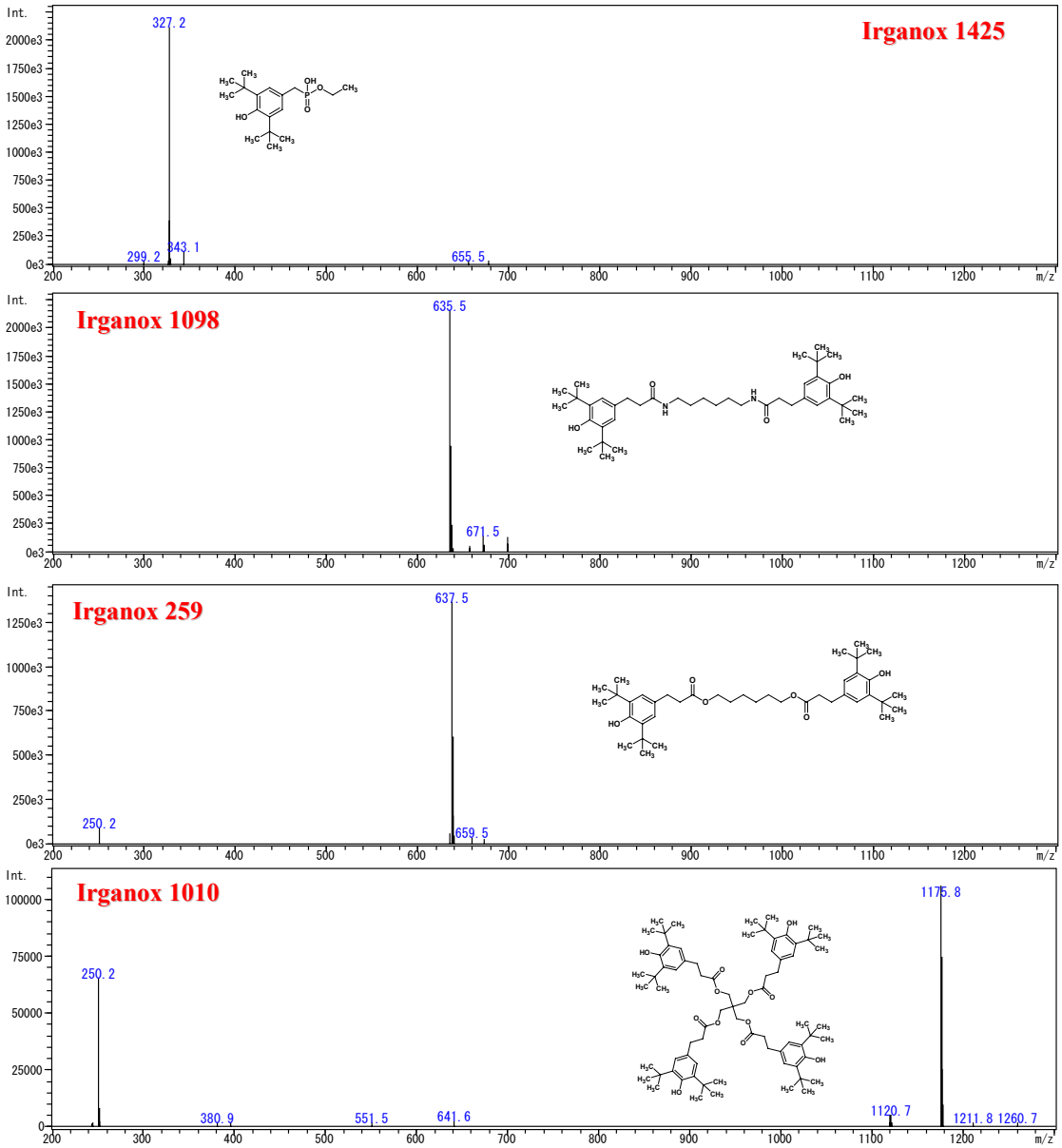

Fig. 2 Mass Spectra of Typical Irganox compounds

Table 1 Analytical conditions for LC-MS

Column	: Shodex GF-310 (4.6 mmI.D. x 50 mmL.)		
Mobile phase A	: water		
Mobile phase B	: acetonitrile		
Time program	: 30%B (0min)→ 90%B (20-25min)		
Flow rate	: 0.25 mL/min		
Injection volume	: 1 μL	**Column temperature**	: 40 ℃
Probe voltage	: -5.0 kV (ESI-Negative mode)		
CDL temperature	: 250 ℃	**Block heater temperature**	: 200 ℃
Nebulizing gas flow	: 1.3 L/min		
Drying gas flow	: 0.1 MPa		
CDL voltage	: Scan-mode		
Q-array DC voltage	: Scan-mode	**Q-array RF voltage**	: Scan-mode
Scan range	: m/z 200-1300 (1.0sec/scan)		

SHIMADZU CORPORATION **International Marketing Division**
3. Kanda-Nishikicho 1-chome, Chiyoda-ku, Tokyo 101-8448, Japan
Phone: 81 (3) 3219-5641 Fax. 81 (3) 3219-5710

Cable Add. SHIMADZU TOKYO