

Determination of antioxidant Irganox 1098 in ethylene-vinyl alcohol copolymer using Py-GC/MS

[Background] A wide variety of additives are added to polymeric materials to enhance or alter the performance and/or production properties of polymeric materials. Thus, the analysis of additives is of interest and it has been done using thermal desorption (TD)-GC/MS. TD-GC/MS is a simple technique and a sample is heated high enough to quantitatively desorb the additive of interest without thermally decomposing the polymer matrix. However, an antioxidant additive, Irganox 1098, is nonvolatile and TD-GC/MS is not applicable for the analysis of Irganox 1098. In this note, we describe the determination of Irganox 1098 in an ethylene-vinyl alcohol copolymer (EVOH) by pyrolysis (Py)-GC/MS.

[Experimental] A small amount of EVOH spiked with Irganox 1098 (ca. 5000 ppm) was pulverized and determination of the additive was carried out using Py-GC/MS at 600°C. The absolute calibration method was used to generate the calibration curve for the determination. A Multi-Shot Pyrolyzer (EGA/PY-3030D) directly interfaced to the split injector on GC was used to pyrolyze the sample. The pyrolysis products were split, separated on a 15 meter separation column and detected by a quadrupole mass spectrometer.

[Results] The peak for BHT-quinone-methide, one of the thermal decomposition products of Irganox 1098, is observed in the pyrogram of Irganox 1098 (Fig. 1(a)). The mass spectrum contains m/z 161 peak (base peak) and m/z 218 peak (molecular ion). Fig. 1(b) shows the pyrograms of EVOH spiked with Irganox 1098. In the total ion chromatogram (TIC) of EVOH, the peaks for Irganox 1098 are in the midst of the pyrolyzate peaks and not discernible. However, extracted ion chromatograms (EICs) of m/z 161 and m/z 218 show a clear peak at a retention time of 1.8 min. By generating an absolute calibration curve using the area of the m/z 161 peak, the concentration of Irganox 1098 in EVOH was determined to be 5, 217 ppm ($n=2$, RSD=0.9%).

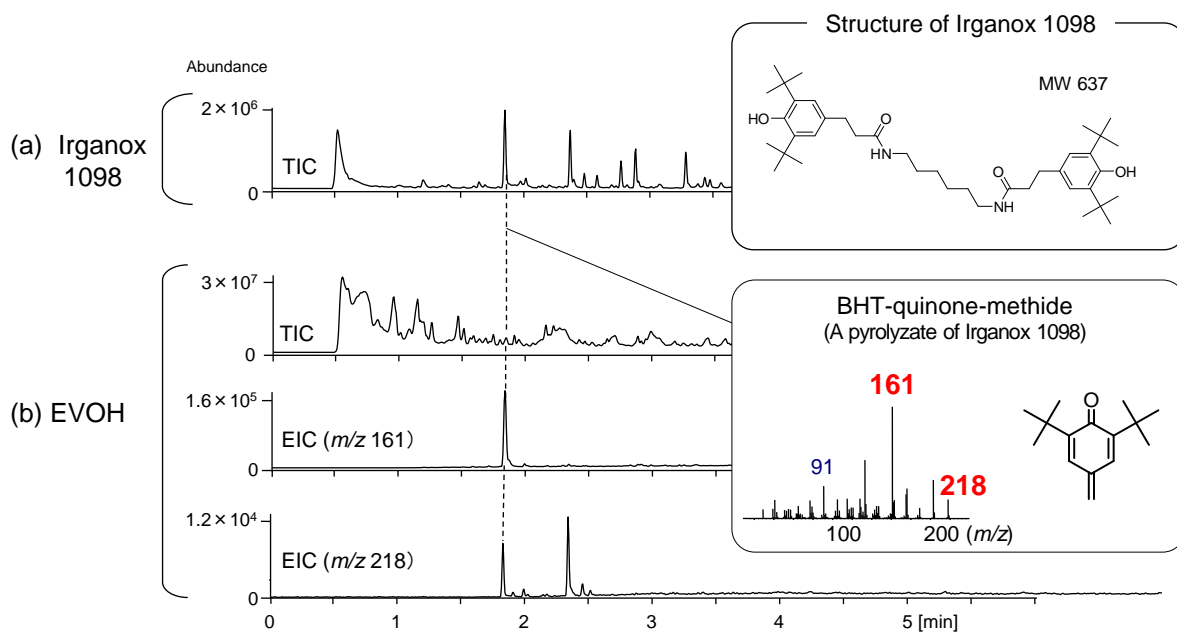


Fig. 1 (a) Pyrogram (TIC) of Irganox 1098 and (b) pyrograms (TIC and EICs) of EVOH

Furnace temp.: 600°C, column flow rate: 1.2 mL/min, split ratio: 1/50,
 GC oven temp.: 40 - 350°C (20 °C/min, 4 min hold), scan range: 29 - 650 (m/z),
 Separation column: UA⁺-1 (dimethyl polysiloxane, L=15 m, i.d.=0.25 mm, df.=0.1 μm),
 Sample weight : ca. 1.5 μg (Irganox 1098); ca. 200 μg (EVOH, spiked with ca. 5000 ppm of Irganox 1098)

Keywords : Ethylene-vinyl alcohol copolymer, antioxidant, Irganox 1098, quantitative analysis, Py-GC/MS

Product used : Multi-functional pyrolyzer, UA⁺-1, Eco-Cup LF

Applications : Polymer analysis, additive analysis, quality assurance

Related technical notes : [PYA1-080E](#), [PYA1-081E](#), [PYA2-017E](#)

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