Analysis of gases evolved during the molding process of polyethersulfone (PES) and identification of unknown peaks using a time-of-flight mass spectrometer
Part 3: Analysis of unknown peak by TD-GC/TOFMS

[**Background**] In the previous study (PYA1-176E), using a quadrupole mass spectrometer (QMS) as a detector, thermal desorption (TD)-GC/QMS measurements of a PES sample were conducted under air and helium (He) atmospheres. In this note, identification of the unknown peak, which could not be identified by QMS, was accomplished using a time-of-flight mass spectrometer (TOFMS) with two different ionization methods.

[**Experimental**] All measurements were done using a GC/TOFMS system with a Multi-Shot Pyrolyzer (EGA/PY-3030D) directly interfaced to the GC inlet. A UA$^+$-5 column was used as a separation column. Approximately 20 mg of a PES sample was put in a sample cup (Eco-Cup LF). Since the TD chromatograms obtained in air and He were identical to each other in the previous study (PYA1-176E), measurements were done in He in this study. The volatile gases evolved from PES by TD were cryo-trapped using a MicroJet Cryo-Trap. The trapped components were then detected by TOFMS (JEOL Ltd.) using ionization methods of electron ionization (EI), a hard ionization method, and field ionization(FI), a soft ionization method. Structural analysis was done using msFineAnalysis AI software (JEOL Ltd.).

[**Results**] TD chromatograms of the PES sample obtained by TOFMS using two different ionization methods are shown in Fig. 1, and the mass spectra of the unknown peak are shown in Fig. 2. The mass spectrum obtained by EI was identical between QMS and TOFMS. The composition formula was estimated from the accurate mass of the molecular ion obtained by FI, and the possible structures of the fragment ions obtained by EI were predicted using msFineAnalysis AI software. The predicted molecular structure is shown in Fig. 3. The unknown peak in Fig. 1 was predicted to be due to CPMPS, which shares common moieties of the compounds corresponding to the peaks before and after it (marked with dots in Fig. 1).

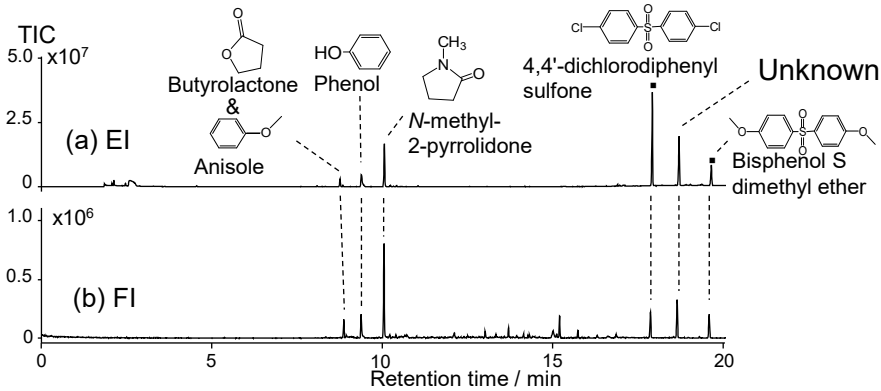

Fig. 1 TD chromatograms obtained by TOFMS of PES
(a) EI method, (b) FI method.

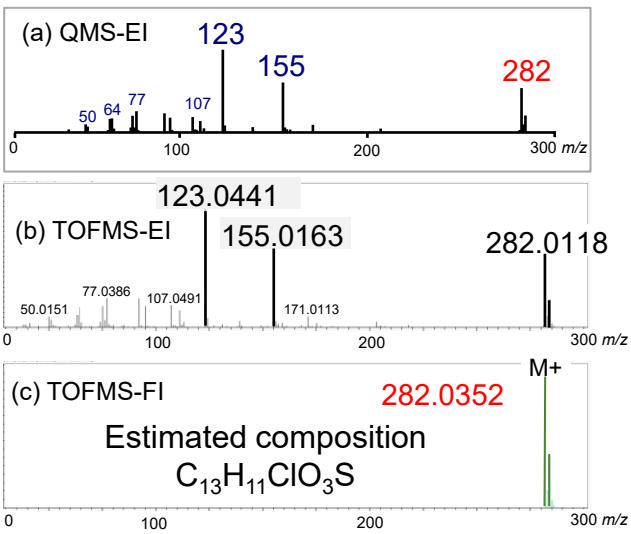

Fig. 2 Mass spectra of the unknown peak obtained by
(a) QMS-EI, (b) TOFMS-EI, and (c) TOFMS-FI methods.

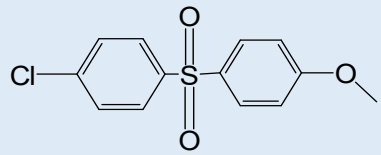

Fig. 3 Molecular structure of the compound, corresponding to the unknown peak, predicted by msFineAnalysis AI software
4-Chlorophenyl-4'-methoxyphenylsulfone (CPMPS).

TD temp.: 400 ºC (10 min hold), GC inj. temp.: 300 ºC,
GC oven: 40 (2 min hold) - 320 ºC (20 ºC/min, 6 min hold),
Separation column: UA$^+$-5 (5 % diphenyl 95 % dimethyl-polysiloxane), *L*=30 m, i.d.=0.25 mm, df=1.0 µm,
Split ratio: 1/10, Column flow rate: 1.0 mL/min,
MS scan range: *m/z* 29 - 600, Sample amount: ca. 20 mg
MS ionization: QMS・EI : 70 eV
TOFMS・EI : 70 eV, 300 µA
TOFMS・FI : -10 vK, 40 mA/30 msec.

Keywords : Polyethersulfone, Air atmosphere, Thermal desorption analysis, Time-of-flight mass spectrometer

Products used : Multi-Functional Pyrolyzer, Auto-Shot Sampler, Vent-free GC/MS adapter, Carrier gas selector, Selective sampler Selective Sampler, MicroJet Cryo-Trap, F-Search

Applications : General polymer analysis, Electric and Electronic industry

Related technical notes : PYA3-033E, PYA3-042E, PYA1-149E, PYA3-049E (Part-1), PYA1-176E (Part-2)

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