

Evolved Gas Analysis (EGA)-MS in Air Atmosphere

Part 6: Thermo-oxidative decomposition of polypropylene

[Background] In the previous note (PYA3-033E), a new flow system that solves some problems in EGA-MS in air atmosphere was developed. In this note, the new flow system was applied to the EGA-MS analysis of isotactic polypropylene (*iso*-PP) and the thermo-oxidative degradation behavior of *iso*-PP was investigated.

[Experimental] The surface of *iso*-PP pellets was scraped off with a cutter knife and about 0.2 mg of *iso*-PP was put in a sample cup. EGA-MS measurements were carried out in He atmosphere (flow rate: 50 mL/min) and air atmosphere (flow rate: 10 mL/min), using the conventional flow system (PYA3-033E Fig. 1 (a)) and the new flow system (PYA3-033E Fig. 1 (b)), respectively.

[Results] The EGA thermograms of *iso*-PP measured in He and air are respectively shown in Figs. 1 (a) and (b). Both peaks in Zones A and B originate from the thermal decomposition of *iso*-PP, but the peak top temperature in air (Fig. 1 (b)) is 193 °C lower than that in He (Fig. 1 (a)), suggesting a thermo-oxidative decomposition in air atmosphere. The averaged mass spectra are shown at the bottom of Fig. 1. Zones A and B show different mass spectra. Carbon dioxide was detected in Zone C. Peaks in Zone B are likely to contain oxidized products such as aldehydes, while peaks in Zone A contain only unoxidized pyrolyzates of *iso*-PP. However, oxidized products and the fragment ions derived from hydrocarbons often share the same *m/z* values and cannot be differentiated from the mass resolution of a quadrupole mass spectrometer. The identification of products in each zone will be possible by using a high-resolution mass spectrometer such as a time-of-flight mass spectrometer.

(a) He: 60 mL/min (Conventional flow system)

(b) Air: 10 mL/min, additional He: 50 mL/min (New flow system)

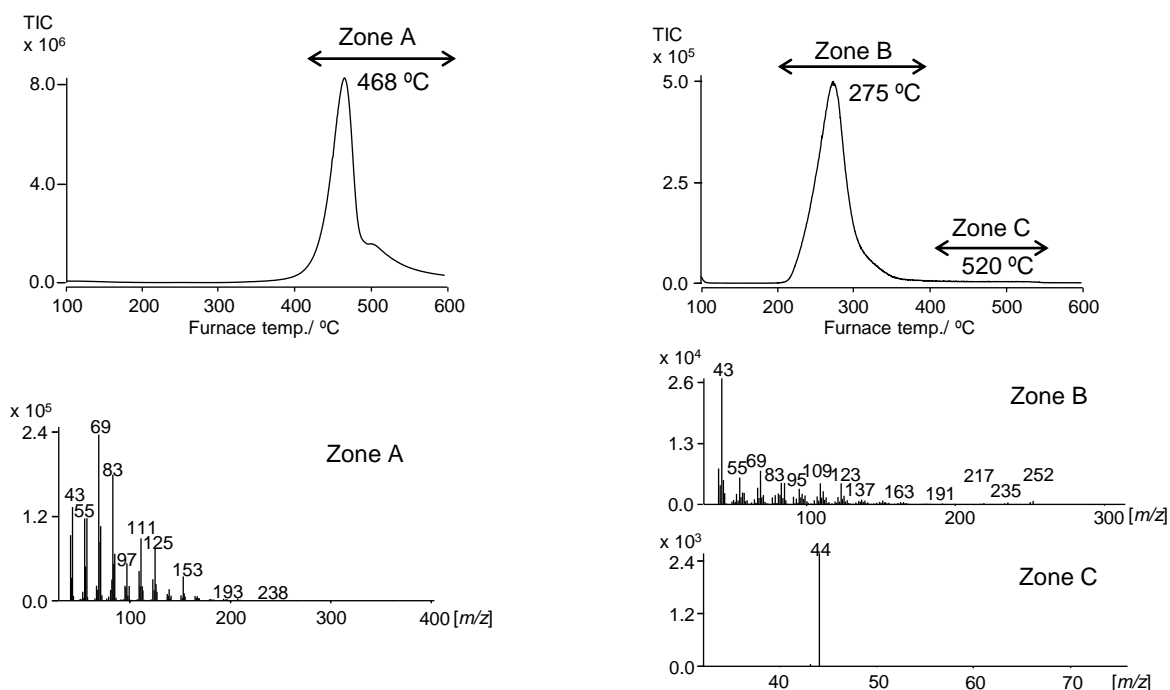


Fig. 1 EGA thermograms and averaged mass spectra of *iso*-PP obtained using two different atmospheric gases

Furnace temp.: 100 - 600 °C (20 °C/min), EGA tube: UADTM-2.5N (L=2.5 m, i.d.=0.15 mm),

[Conventional flow system] He flow rate: 60 mL/min, [New flow system] Airflow rate: 10 mL/min, additional He flow rate: 50 mL/min,

Split ratio: 1/60, Tube flow rate: 1 mL/min, GC oven temp.: 300 °C, MS scan range: *m/z* 41 - 400, MS scan rate: 1 scan/s, Sample amount: 0.2 mg

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Keywords : Air atmosphere, Thermal oxidative degradation, EGA-MS, Evolved gas analysis

Products used : Multi-Shot pyrolyzer, Auto-Shot Sampler, UADTM-2.5N, Eco-Cup LF, Vent-free GC/MS adapter

Applications : General polymer analysis, Degradation evaluation, Material analysis

Related technical notes : PYA4-002E, PYA3-033E, PYA3-034E, PYA3-035E, PYA3-036E, PYA3-037E

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