Evolved Gas Analysis (EGA)-MS in Air Atmosphere

Part 2: EGA thermograms of PS obtained in varied atmospheric gases

[Background] In the previous note (PYA3-033E), a new flow system that solves various issues in EGA-MS in the air atmosphere was described. In this note, EGA-MS analysis of polystyrene (PS) was performed using each of the flow systems described in the previous note, and the EGA thermogram profile, averaged mass spectrum, and S/N were compared. In addition, the concentration of oxygen flowing into the mass detector (MSD) was calculated in the analysis under the oxidative atmosphere for each flow system.

[Experimental] 25 mg of PS was dissolved in 1 mL of dichloromethane, and 5 µL of the PS solution was put in a sample cup. Then, the solvent was evaporated to form a thin film of 0.125 mg PS. Using the flow systems described in the previous note, EGA-MS analysis was performed under various atmospheres, and peak height (S), noise (N), and S/N were calculated. S is the difference between the intensity and the baseline at the peak temperature, and N is the peak-to-peak intensity of the baseline in the range of 160 to 180ºC where the baseline is relatively flat.

[Results] EGA thermograms obtained under different flow systems and atmospheric gases are shown in Figs. 1 (a)-(d). The peak temperature was 429 ºC in He (a), while it shifted to 302-315 ºC in the oxidative atmosphere (b)-(d). In the averaged mass spectrum, ions characteristic of PS oligomers were detected in (a), whereas ions characteristic of benzaldehyde formed by thermo-oxidative decomposition of PS (*m/z* 77, 105) were detected in (b)-(d). In the conventional flow system, the S/N under air (b) was 12.3, which was much lower than that under He (a), and the S/N under O_2/He synthetic air (c) becomes to be high, due to the replacement of N_2 with He. In the new flow system (d), S/N = 909, 2.6 times higher than that in (c). The oxygen concentration in the total carrier gas flowing into the MSD was 3.3 % in (d), whereas it was 20 % in (b) and (c). In conclusion, the dilution of air by additional He in the new flow system was found to be effective in improving the S/N and suppressing oxidation of the ion source filament of the MSD.

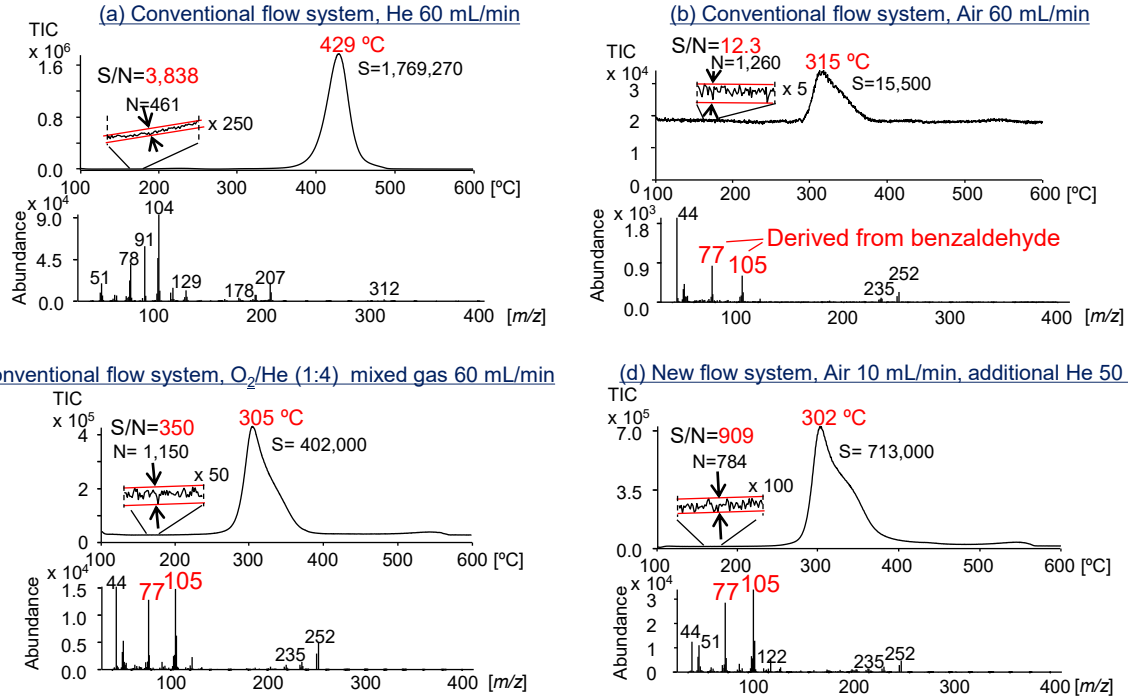

Fig. 1 EGA thermograms of polystyrene obtained using two flow systems in varied atmospheric gases.

Pyrol. furnace temp.: 100 - 600 ºC (20 ºC/min), EGA tube: UADTM-2.5N (*L*=2.5 m, i.d.=0.15 mm), Tube flow rate: 1 mL/min, Split ratio: 1/60, GC oven: 300 ºC, MS scan range: *m/z* 41 - 400, MS scan rate: ca. 1 scan/s, Sample amount: ca. 0.125 mg.

Reference: A. Shiono *et al.*, *J. Anal. Appl. Pyrol.*, 156 (2021) 105122.

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

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

Applications : General polymer analysis, Degradation evaluating, Material analysis

Related technical notes : PYA4-001E, PYA4-002E, PYA3-033E, PYA3-035E, PYA3-036E, PYA3-037E, PYA3-038E

Please forward your inquiries via our web page or send us a fax message.

R&D and manufactured by :
Frontier Laboratories Ltd.
Phone: (81)24-935-5100 Fax: (81)24-935-5102
www.frontier-lab.com

®: A registered trademark of Frontier Laboratories Ltd.