Evolved Gas Analysis (EGA)-MS in Air Atmosphere

Part 3: Optimum flow rate of additional helium

[Background] In the previous note (PYA3-033E), a new flow system to solve various issues involved in EGA-MS analysis in the air atmosphere was described. In this note, EGA-MS analysis was performed by varying the additional He flow rate in the range of 5 - 300 mL/min in the new flow system with a fixed flow rate of an air carrier gas at 10 mL/min, and the optimum flow rate of He was determined from the study on the relationship between the additional He flow rate and S/N.

[Experimental] 25 mg of polystyrene (PS) was dissolved in 1 mL of dichloromethane, and 5 µL of this solution was put in a sample cup to form a thin film of 0.125 mg by solvent evaporation. EGA-MS analysis of PS was performed using the new flow system, and the peak height (S), noise (N), and S/N were calculated as in the previous note (PYA3-034E). The flow rate of the EGA tube was set to 1 mL/min by adjusting the split ratio.

[Results] EGA thermograms at additional He flow rates of 5, 50, and 300 mL/min (Fig. 1) showed similar peak temperatures for all EGA thermograms, but S and N varied with additional He flow rate. Plots of S, N, and S/N against additional He flow rate are shown in Fig. 2. As the additional He flow rate increases, the N_2 concentration at the ion source of a mass detector decreases, resulting in the monotonous decrease in N. Whereas, S increases initially with the increase in the He flow rate (0-25 mL/min), due to the recovery from the reduced ionization efficiency by N_2. After 25 mL/min, the effect of dilution of the evolved gases by additional He becomes to be prominent, resulting in the monotonous decrease in S. As a result, the S/N reaches the maximum at 50 mL/min of additional He flow rate which is an optimum condition for thermo-oxidative experiments in EGA-MS using the new flow system.

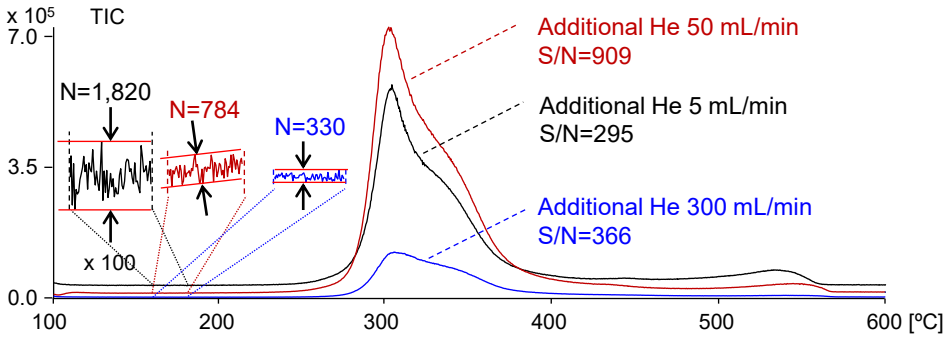

Fig. 1 EGA thermograms of PS with additional He flow rates of 5, 50, and 300 mL/min (air flow rate: 10 mL/min).

Pyrol. Furnace temp.: 100 - 600 ºC (20 ºC/min), EGA tube: UADTM-2.5N (*L*=2.5 m, i.d.=0.15 mm), Air flow rate: 10 mL/min, Additional He flow rate: 5 - 300 mL/min, Split ratio: 1/15 - 1/310, Tube flow rate: 1 mL/min, GC oven: 300 ºC, MS scan range: *m/z* 41 - 400, MS scan rate: *ca.* 1 scan/s, Sample amount: *ca.* 0.125 mg

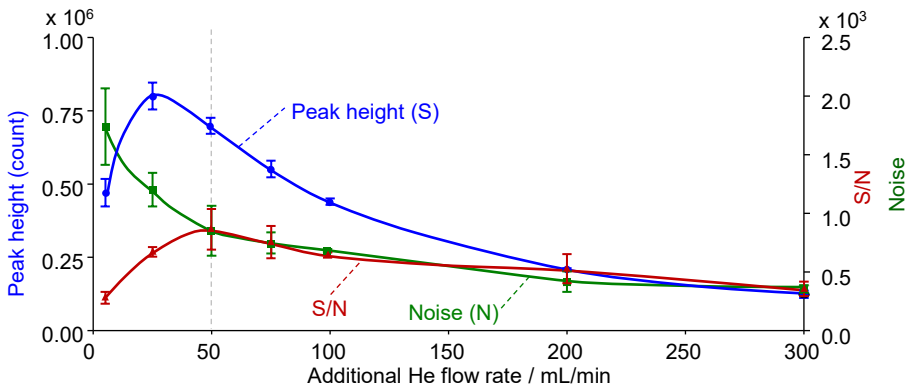

Fig. 2 Peak height, noise and S/N vs. additional He flow rate (air flow rate: 10 mL/min).

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-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, PYA3-036E, 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