Evolved Gas Analysis (EGA)-MS in Air Atmosphere

Part 4: Relationship between airflow rate and S/N

[Background] In the previous note (PYA3-033E), a new flow system that solves some problems with EGA-MS in air atmosphere was developed. In this note, EGA-MS measurements were carried out by varying the airflow rate in the range of 10 to 50 mL/min using the new flow system, and the relationship between the airflow rate and S/N was investigated.

[Experimental] About 25 mg of polystyrene (PS) was dissolved in 1 mL of dichloromethane, and 5 µL of this solution was put in a sample cup. A thin film of about 0.125 mg was obtained by evaporating the solvent. EGA-MS measurements of PS were carried out using the new flow system with an additional He flow rate of 50 mL/min and airflow rates of 10, 20, 30, and 50 mL/min, and the peak height (S) and noise (N) were determined., S/N was calculated as described in the previous note (PYA3-034E). The flow rate of the gas in the EGA tube was set to 1 mL/min by adjusting the split ratio.

[Results] The EGA thermograms of PS obtained at airflow rates of 10, 30, and 50 mL/min (Fig. 1) showed similar peak top temperatures, but the S and N varied with the airflow rate. Fig. 2 shows plots of S, N, and S/N against the airflow rate. S decreases monotonically with increasing the airflow rate, but the decrement is larger than the expectation considering the dilution effect caused by increased airflow rates with the split ratios of 1/60 to 1/100, suggesting that the significant decrease in S is ascribed to the decrease in the ionization efficiency caused by the increased N_2 concentration in the ion source of the mass spectrometer. Likewise, N increases gradually as the airflow rate increases, which is also ascribed to the increased N_2 concentration in the ion source. As a result, the S/N decreased with increasing airflow rate. Since an airflow rate of less than 10 mL/min leads to unexpected secondary reactions during pyrolysis, 10 mL/min of air was chosen as the optimum airflow rate.

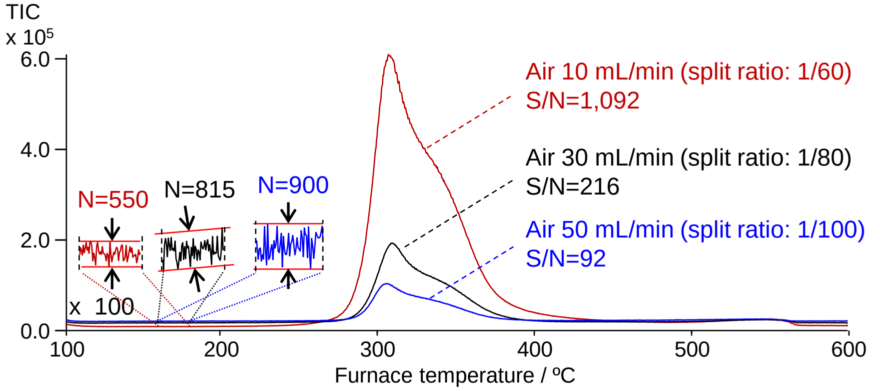

Fig. 1 EGA thermograms of PS at airflow rates of 10, 30, 50 mL/min (additional He flow rate : 50 mL/min)

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 - 50 mL/min, Additional He flow rate: 50 mL/min, Split ratio: 1/60 - 1/100, Tube flow rate: 1 mL/min, GC oven temp.: 300 ºC, MS scan range: *m/z* 41 - 400, MS scan rate: 1 scan/s, Sample amount: 0.125 mg

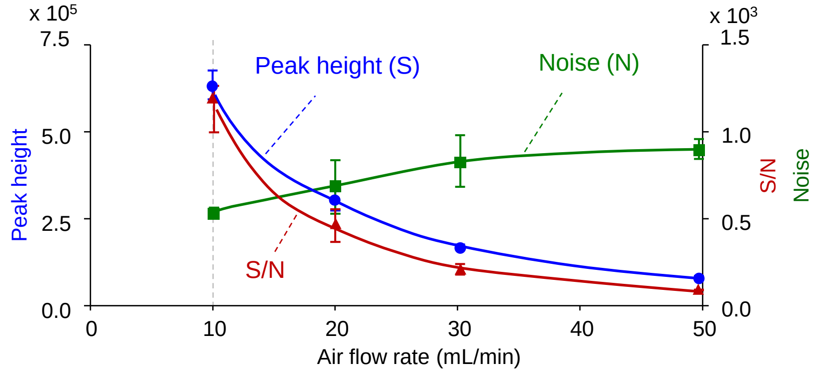

Fig. 2 Plots of peak height, noise, and S/N against airflow rate (additional He flow rate : 50 mL/min)

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

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, PYA3-037E, PYA3-038E

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