

High-sensitivity detection of polystyrene by F-Splitless Py-GC/MS using Multi-Functional Splitless Sampler

Part 1 Features of pyrograms obtained with different injection methods

[Background] In pyrolysis (Py)-GC/MS, the split injection method is commonly employed. On the other hand, for trace amounts of analytes, splitless injection can be considered. However, this method may lead to secondary reactions, such as accelerated decomposition, if pyrolyzates are not rapidly moved from the heated furnace. To address this issue, we developed a new F-Splitless injection method which involves the forced venting of the carrier gas and cryo-trapping of pyrolyzates. This note presents the pyrograms of polystyrene (PS) obtained through both the conventional split/splitless and the F-Splitless methods.

[Experimental] 1 mg of PS was dissolved in 10 mL of dichloromethane and subsequently diluted according to the split ratio so that 10 ng of PS is introduced into the separation column. The sample was put in a sample cup, followed by evaporation of the solvent. Py-GC/MS measurements were carried out at a furnace temperature of 550 °C using the system shown in Fig. 1 with the total flow rate given in the caption of Fig. 2.

[Results] The pyrograms of PS obtained by different injection methods are shown in Fig. 2. Styrene monomer (S), dimer (SS), and trimer (SSS) peaks are observed with variations in the peak area ratio of SSS/S. With the conventional splitless method, the low total carrier gas flow rate promotes the decomposition of SSS and results in the reduced peak areas and alters the pyrolysis behavior differed from that observed with the split method (split ratio: 1/13). On the other hand, the F-Splitless method suppresses the decomposition of SSS, resulting in a similar pyrolysis behavior to that of the split method. The next note (PYA1-155E) will provide the comparison of SSS/S, an indicator of the secondary reaction in the PS pyrolysis.

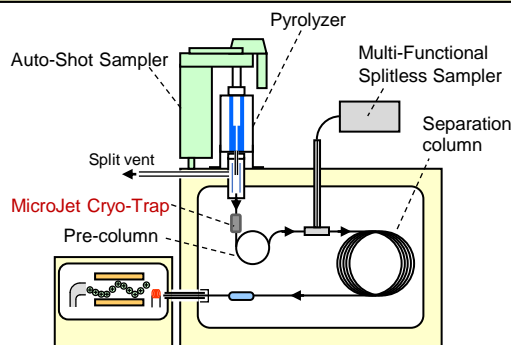


Fig. 1 System configuration of F-Splitless Py-GC/MS.

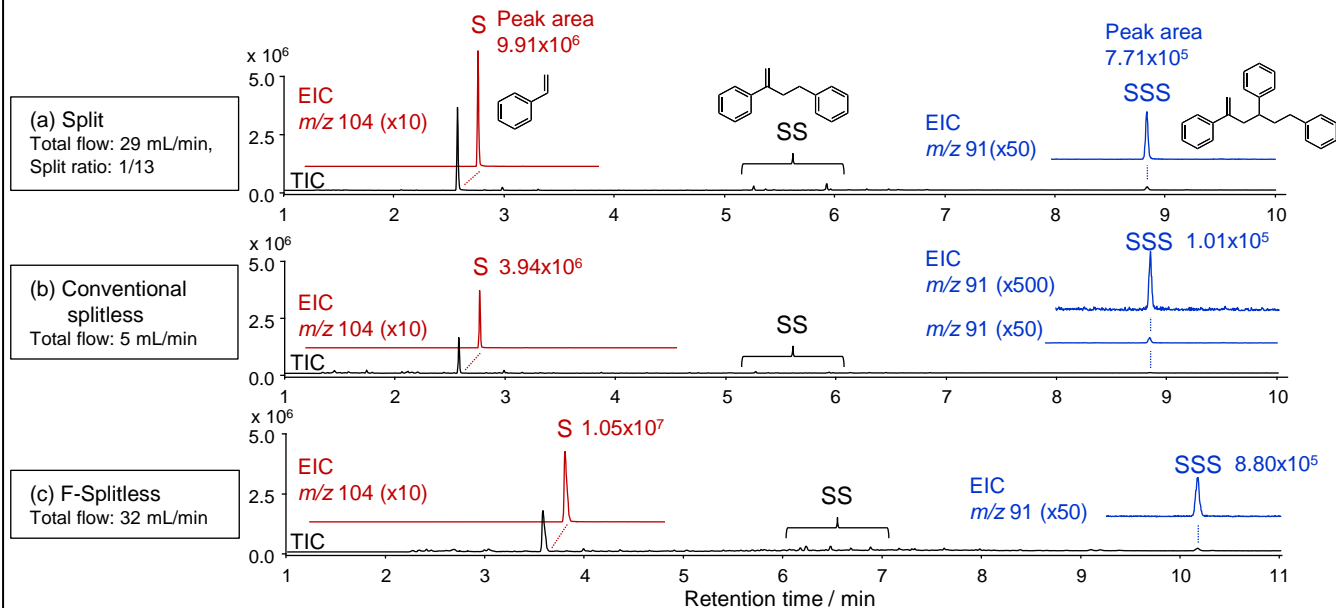


Fig. 2 Pyrograms of PS obtained by different injection methods.

Furnace temp.: 550 °C, Furnace-interface temp.: 300 °C, GC injector temp.: 300 °C, Injector press.: 150 kPa (constant press.), Initial column flow rate: 2 mL/min, Pre-column: UA*-50 (50 % diphenyl 50 % dimethylpolysiloxane; L=2 m, i.d.=0.25 mm, df=1 µm), Main column: UA*-5 (5 % diphenyl 95 % dimethylpolysiloxane; L=30 m, i.d.=0.25 mm, df=0.5 µm), GC oven temp.: 40 (5 min hold) - 20 °C/min - 280 °C (13 min hold), GC/MS interface temp.: 300 °C, MS scan range: m/z 29 - 350, MS scan rate: 4.4 scan/s, Carrier gas flow rate and sample amount: (a) Total flow rate: 29 mL/min, Split ratio: 1/13, PS 130 ng, (b) Total flow rate: 5 mL/min, PS 10 ng, (c) Total flow rate: 32 mL/min, PS 10 ng.

Reference: [K. Tei et al., J. Anal. Appl. Pyrolysis 168 \(2022\) 105707.](#)

Keywords : F-Splitless, Secondary reaction, Splitless analysis, High-sensitivity analysis, Microplastics

Products used : Multi-Shot Pyrolyzer, Multi-Functional Splitless Sampler, Auto-Shot Sampler, Eco-Cup LF, Packed GC glass insert, UAMP column kit, Vent-free GC/MS adapter, F-Search MPs 2.1

Applications : Environmental analysis, Trace analysis, General polymer analysis

Related technical notes : PYA1-155E (Part 2), PYA1-156E (Part 3), PYA1-157E (Part 4), PYT-037E, PYT-038E

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