

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

Part 2 Styrene trimer/monomer peak area ratios (SSS/S) and reproducibilities by injection methods

[Background] In the previous note (PYA1-154E), the pyrograms obtained by different injection methods, i.e., the conventional split/splitless and new F-Splitless, were compared. Here, a comparison of the peak area ratios (SSS/S) of styrene trimer (SSS) to monomer (S) obtained using the different injection methods is reported. This serves as an indicator to evaluate the extent of secondary reactions that occur during the pyrolysis of polystyrene (PS).

[Experimental] Py-GC/MS of PS was carried out using the three injection methods described previously, and the peak areas were calculated based on the extracted ion chromatograms (EICs) for SSS (m/z 91) and S (m/z 104).

[Results] Plots of the peak area ratio (SSS/S) against GC total flow rate for F-Splitless, conventional splitless, and split methods are shown in Fig. 1. In the split method (split ratio 1/5), SSS/S increases significantly as the total flow rate increased from 8 to 29 mL/min, and gradually increases up to 203 mL/min. This result indicates that in the split method, the transformation of SSS to S through the secondary reaction is suppressed as the total flow rate increases. In the conventional splitless method, due to the low total flow rate, the decomposition of SSS is promoted. In contrast, in the F-Splitless method, its SSS/S is comparable to that of the split method under the same total flow rate, indicating that the secondary reaction (accelerated decomposition) is suppressed. The reproducibilities of the peak area values for three injection methods are shown in Fig. 2. The peak areas of S and SSS obtained with the split method and the F-Splitless method at flow rates above 13 mL/min exhibited excellent reproducibilities compared to the SSS reproducibility obtained with the conventional splitless method. In conclusion, the newly developed F-Splitless method is an analytical technique that allows for the quantification of PS with good reproducibilities while suppressing the secondary reactions.

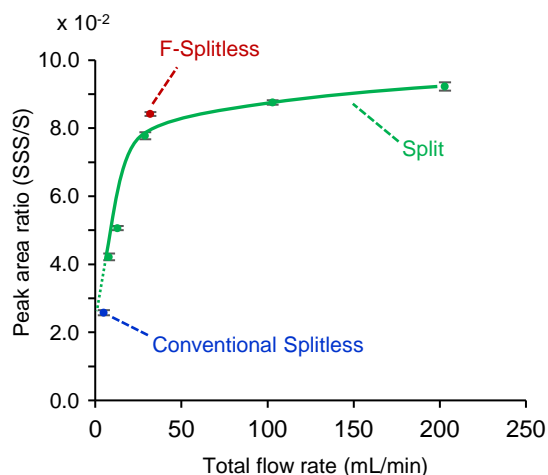


Fig. 1 Plots of peak area ratio (SSS/S) against total flow rate* of conventional split/splitless and F-Splitless methods.

* Total flow rate includes septum purge of 3 mL/min.

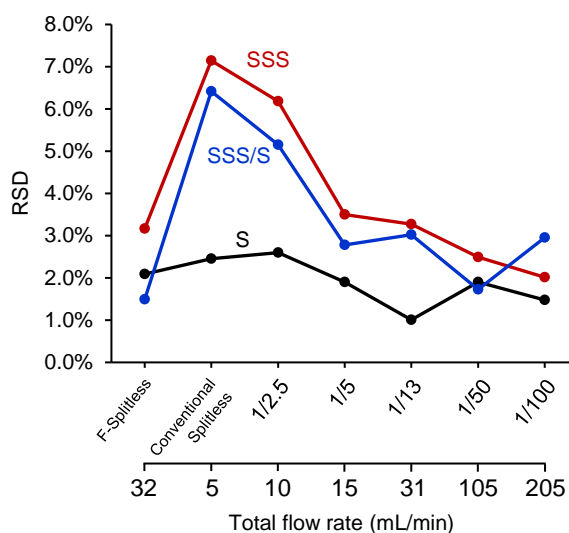


Fig. 2 Plots of RSD of S and SSS peak areas against total flow rate measured by the conventional split (split ratio 1/2.5 - 1/100)/splitless and F-Splitless methods ($n=5$).

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 : [PYA1-154E \(Part 1\)](#), [PYA1-156E \(Part 3\)](#), [PYA1-157E \(Part 4\)](#), [PYT-037E](#), [PYT-038E](#)

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