

Trace analysis of acrylic copolymers using pyrolysis (Py)-GC/MS with F-Splitless injection Part 3: Calibration curves and reproducibilities of peak areas of monomers

[Background] In the previous note (PYA1-161E), Py-GC/MS measurements of acrylic copolymer samples of 0.1 and 1 µg using different injection methods were reported, and it was found that the F-Splitless injection method gave a high detection sensitivity. In this note, the calibration curves of monomers were made using different injection methods. Additionally, the relative standard deviation (RSD) values of the EIC peak areas of monomers were obtained by three repeated measurements and the F-Splitless injection method was found to offer better results.

[Experimental] The same acrylic copolymer as in the previous note (PYA1-160E) was dissolved in dichloromethane and put in a sample cup (sample amount: 0.1, 0.5, and 1 µg), and the solvent was evaporated. The sample was then pyrolyzed at 600 °C using the Py-GC/MS system (see Fig. 1 and the analytical conditions given in the caption of Fig. 2 in PYA1-160E).

[Results] Calibration curves for each monomer (except HEA and HEMA) are shown in Fig. 1, where the EIC peak area was plotted against the sample amount and fairly good linearity was obtained for all injection methods. Furthermore, the slope of the calibration curve obtained with the F-Splitless injection method is larger than those obtained with the other injection methods, indicating its high sensitivity. Table 1 summarizes the range of RSD values of the peak area of monomers for a sample amount of 0.5 µg, and the F-Splitless Injection method shows the lowest RSD value i.e. the best reproducibility. HEA was not detected at sample amounts below 1 µg, and HEMA was not detected at 0.1 µg. Their detection will be explored in the future. In conclusion, highly sensitive detection of acrylic monomers was achieved by the F-Splitless injection method and the trace analysis of acrylic monomers with a sample amount of 1 µg or less could be made.

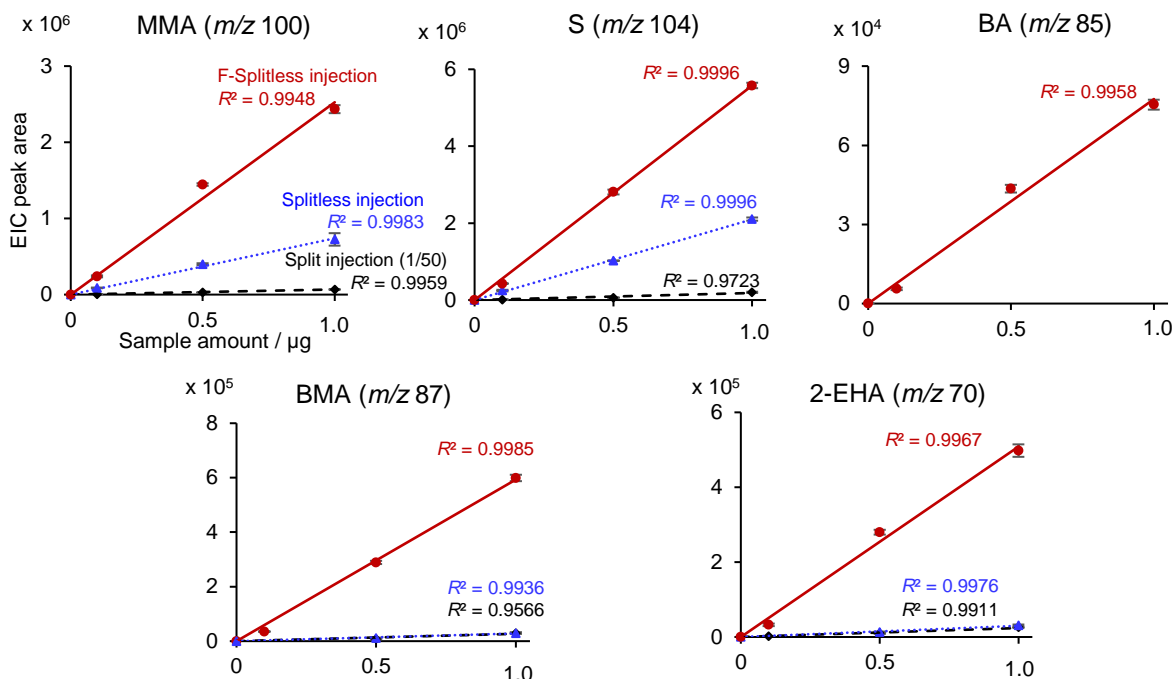


Fig. 1 Calibration curves obtained by different injection methods (sample amount: 0.1 - 1 µg)

Table 1 Ranges of RSD (sample amount: 0.5 µg, n=3)

RSD / %	Split injection (1/50)	Splitless injection	F-Splitless Injection
	3.8 - 9.4	1.1 - 8.6	1.2 - 4.4

Keywords : Acrylic resin, Trace analysis, F-Splitless injection, High sensitivity analysis, Flash Pyrolysis (Py)-GC/MS

Products used : Multi-Shot Pyrolyzer, Multi-Functional Splitless Sampler, MicroJet Cryo-Trap, Auto-Shot Sampler, UA⁺-5, Eco-Cup LF, F-Search, Vent-free GC/MS adapter

Applications : General polymer analysis, Quality assurance, Material analysis, Foreign materials analysis

Related technical notes : PYA1-160E (Part 1), PYA1-161E (Part 2), PYT-037E, PYA1-154E

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