Trace analysis of acrylic copolymers using pyrolysis (Py)-GC/MS with F-Splitless injection
Part 2: Enhanced sensitivity by F-Splitless injection method

[Background] In the previous note (PYA1-160E), the pyrograms of acrylic copolymer obtained by Py-GC/MS using the split, splitless and F-Splitless injection methods were compared. In this note, high sensitivity of F-Splitless injection method is demonstrated by comparing the EIC peak areas of acrylic monomers of pyrolyzates obtained by the split, splitless and F-Splitless injection methods.

[Experimental] The same acrylic copolymer as in the previous note (see PYA1-160E for the constituent monomers and the mass number used to calculate the EIC peak area) was dissolved in dichloromethane, put in a sample cup, and the solvent was evaporated. Py-GC/MS measurements were done at a pyrolysis temperature of 600 ºC using the system shown in Fig. 1 of the previous note.

[Results] The pyrograms of 1 µg and 0.1 µg of the acrylic copolymer sample obtained by different injection methods are shown in Figs. 1 and 2, respectively. The EIC peak areas of each monomer measured by different injection methods were compared. The enhancement factor was calculated based on the EIC peak area obtained by the split injection method, and the calculated factors are given for the monomer peaks in Figs. 1 and 2. With a sample amount of 1 µg, the F-Splitless injection method showed 26 to 45-fold enhancement with respect to the split injection method, while only 1 to 19-fold enhancement was obtained for the splitless method (Fig. 1). With 0.1 µg of sample, the F-Splitless injection method improved the sensitivity by a factor of 19 to 39, while the splitless injection only improved by a factor of around 10 (Fig. 2). Further, HEA was not detected by any of the injection methods. In summary, it was possible to detect each monomer with high sensitivities by F-Splitless injection method for acrylic copolymers with sample amounts of 0.1 µg to 1 µg. In the next report (PYA1-162E), the calibration curve and reproducibility in the range of 0.1 - 1 µg are reported.

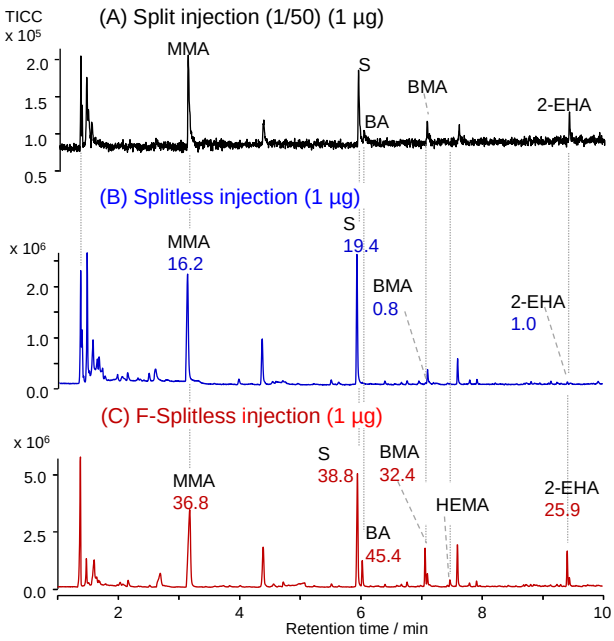

Fig. 1 Pyrograms of acrylic copolymer (1 µg) obtained by different injection methods and the enhancement factors of the EIC peak areas for monomers.

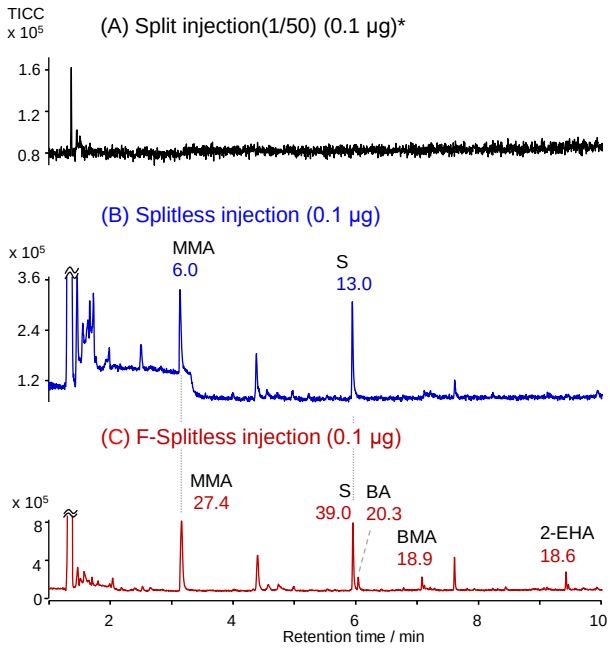

Fig. 2 Pyrograms of acrylic copolymer (0.1 µg) obtained by different injection methods and the enhancement factor of the EIC peak area for each monomer

* Since no monomer peaks could be recognized in Fig. 2 (A), data given in Fig. 2 (A) in the previous note, PYA1-160E, were used as a reference.

Furnace temp.: 600 ºC, Injector press.: 150 kPa, Pre-column: UA⁺-5 (5 % diphenyl 95 % dimethylpolysiloxane, *L*=2 m, i.d.=0.25 mm, df=0.25 µm), Separation column: UA⁺-5 (5 % diphenyl 95 % dimethylpolysiloxane, *L*=30 m, i.d.=0.25 mm, df=0.25 µm), GC oven: 40 ºC (3 min hole) - 320 ºC (20 ºC/min, 3 min hold), MS scan range: *m/z* 29 - 550, MS scan rate: ca. 3 scan/s, Sample amount/injection method: see main text.

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), PYA1-162E (Part 3), PYT-037E, PYA1-154E

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