Trace analysis of acrylic copolymers using pyrolysis (Py)-GC/MS with F-Splitless injection
Part 1: Comparison of pyrograms obtained with various injection methods

[Background] Pyrolysis (Py)-GC/MS usually uses a split injection method with a minimum sample amount of about 1 µg. In recent years, the demand for trace analysis has led to the need for the analysis of a more smaller sample amount. In response to this demand, we have developed the F-Splitless injection method[1)], which enables highly sensitive detection of pyrolyzates by splitless injection using a Multi-Functional Splitless Sampler (MFS). In this report, as a basic study on the trace analysis of acrylic copolymers by Py-GC/MS using the F-Splitless injection method, pyrograms obtained by various injection methods were compared.

[Methods] A specified amount of a dichloromethane solution of acrylic copolymers consisting of seven monomers (Table 1) was put in a sample cup and the solvent was allowed to evaporate. Then, measurements were done using a Py-GC/MS system shown in Fig. 1.

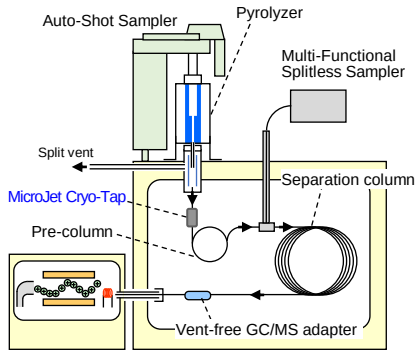

Fig. 1 Py-GC/MS system

[Results] The pyrograms obtained by different injection methods are shown in Fig. 2. In the split injection method, peaks of all seven monomers were detected, while in the splitless injection method, the pyrogram profile was very different from that obtained by the split injection method. This was due to the secondary reactions occurring under a slow carrier gas flow. On the other hand, the F-Splitless injection method afforded a pyrogram that was similar to that obtained by the split injection.
This is because in the F-Splitless injection method, the total flow rate is increased to about 30 mL/min by operating the MFS during pyrolysis, thereby suppressing the secondary reactions. In the next note (PYA1-161E), the high sensitivity of the F-Splitless injection method is described.

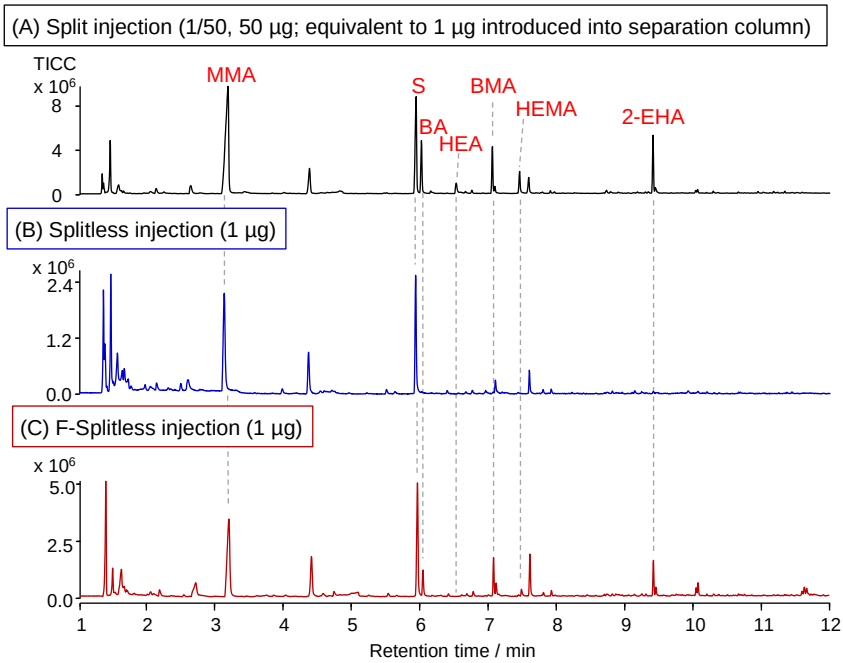

Fig. 2 Pyrograms of acrylic copolymers obtained by different injection methods.

Table 1. Monomers constituting the acrylic copolymer and the mass number used to calculate the EIC peak area.

Name	Abbreviation	*m/z*
Methyl methacrylate	MMA	100
Styrene	S	104
Butyl acrylate	BA	85
2-Hydroxyethyl acrylate	HEA	86
Butyl methacrylate	BMA	87
2-Hydroxyethyl methacrylate	HEMA	87
2-Ethylhexyl acrylate	2-EHA	70

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.

1) K. Tei *et al.*, *J. Anal. Appl. Pyrolysis*, 2022, *168*, 105707.

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 : PYA1-161E (Part 2), PYA1-162E (Part 3), PYT-037E, PYA1-154E

Please forward your inquiries via our web page or send us a fax message.

R&D and manufactured by :
Frontier Laboratories Ltd.
Phone: (81)24-935-5100 Fax: (81)24-935-5102
www.frontier-lab.com