Development of a Multi-Functional Splitless Sampler and its operating mechanism

Part 1: Operating mechanism of F-Splitless Py-GC/MS

[Background] In pyrolysis (Py)-GC/MS, the split injection method is normally used, while the splitless injection method could be used for high sensitivity analysis of trace samples. In the splitless injection, however, the total flow rate of carrier gas during pyrolysis is reduced to keep an optimum column flow rate, which may cause secondary reactions to take place inside the pyrolyzer due to the prolonged residence time of the pyrolyzates in the furnace. On the other hand, in recent microplastic analysis, there is a high demand for high sensitivity analysis of trace samples with small particle sizes. Therefore, a Multi-Functional Splitless Sampler (MFS) that allows splitless injection under a large total carrier gas flow rate with suppressed secondary reactions was developed.

In addition to high sensitivity analysis of trace samples, the new splitless Py-GC/MS (F-Splitless Py-GC/MS) allows back-flushing of high boiling compounds. This note describes the operating mechanism of F-Splitless Py-GC/MS, one of the features of MFS.

[Operating mechanism] The configuration of the system with the MFS is shown in Fig. 1, and the operating mechanism is shown in Fig. 2. All settings and controls are made through the installed software. (1) The GC is set to splitless mode by the GC software, and the split vent and septum purge are closed, and the GC is in ready state. (2) EGA/PY-3030D software automatically turns on the Cryotrap, and the suction pump of the MFS flow controller is also turned on to exhaust the carrier gas out of the system. (3) After the total flow rate of the carrier gas has stabilized at a certain value in the range of 20 to 30 mL/min (injector pressure 100 kPa), the sample is introduced into the pyrolyzer. All the pyrolyzates formed are cryo-trapped at the head of the pre-column by the Cryotrap. (4) After a predetermined time, the suction pump is turned off, the GC is switched to split mode, the split vent and septum purge are opened, and GC/MS analysis is started at the same time as the Cryotrap is turned off. Thus, the MFS allows efficient analysis of trace samples because all pyrolyzates are fed into the separation column while maintaining a relatively high carrier gas flow rate, which suppresses secondary reactions.

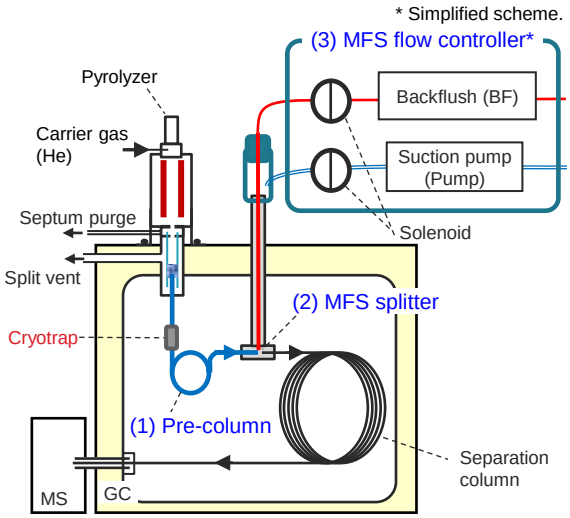
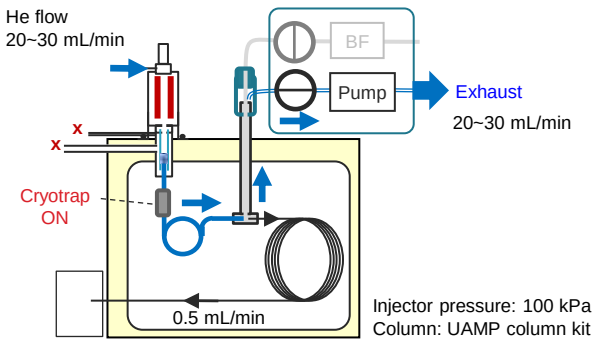


Fig.1 Py-GC/MS system equipped with Multi-Functional Splitless Sampler.

1. All pyrolyzates cryo-trapped at pre-column



2. Cryotrap turned off to start GC analysis

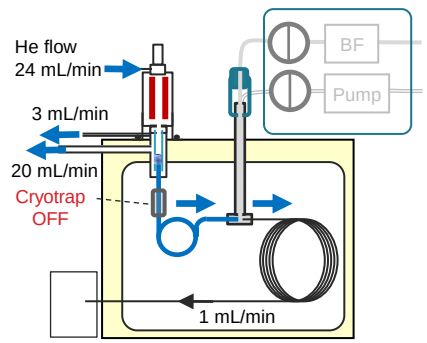


Fig. 2 Operating mechanism of F-splitless Py-GC/MS using MFS.

Reference: K. Tei *et al.*, *J. Anal. Appl. Pyrolysis* 168 (2022) 105707.

Keywords : Splitless analysis, High sensitivity analysis, Microplastic

Products used : Multi-Shot Pyrolyzer, Multi-Functional Splitless Sampler, MicroJet Cryo-Trap, UAMP column kit, Vent free GC/MS adapter

Applications : Microplastic analysis, Microanalysis, General polymer analysis

Related technical notes : PYT-038E (Part 2)

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