

Analysis of trace microplastics (MPs) in sediment samples

Part 1: F-Splitless Py-GC/MS using Multi-Functional Splitless Sampler

[Background] In the analysis of microplastics (MPs) using pyrolysis (Py)-GC/MS, the F-splitless Py-GC/MS method (F-Splitless method), which uses a Multi-Functional Splitless Sampler, can be used to boost the measurement sensitivity¹⁾. In this note, the F-Splitless method is used for the analysis of microplastics in sediment samples collected from environment and its validity is demonstrated.

[Experimental] Sediment samples, provided by Dr. Scholz-Böttcher, ICBM, University of Oldenburg, were collected from the seafloor surface off the Bay of Biscay in the North Atlantic Ocean. The samples were pretreated with acid digestion and density separation. About 4 mg of the sediment sample was placed in an Eco-Cup LF and covered with quartz wool, then Py-GC/MS measurements were done by the Split or F-Splitless method at a pyrolysis temperature of 600 °C using the analytical system shown in Fig. 1.

[Results] The pyrograms of a sediment sample obtained by the Split and F-Splitless methods are shown in Fig. 2. As clearly seen, the use of the F-Splitless method allows peak intensities to be significantly increased. As an example, the peak area of 1-tridecene, one of the pyrolyzates of polyethylene, increased about 48-fold compared to that by the split method (split ratio: 1/50). In conclusion, it was demonstrated that the F-Splitless method increased the sensitivity of the MPs measurement. In the next note (PYA1-151E), the identification and quantitation of MPs in the sample using the F-Search MPs software²⁾ are presented.

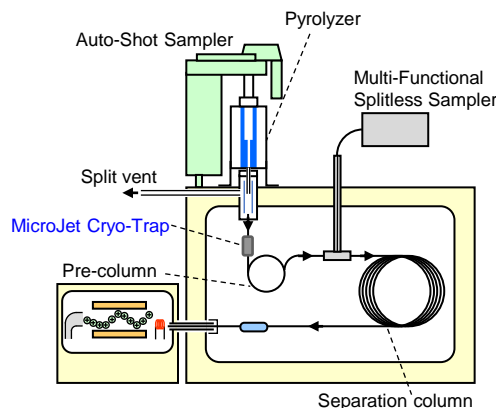


Fig.1 Analytical system for MPs analysis.

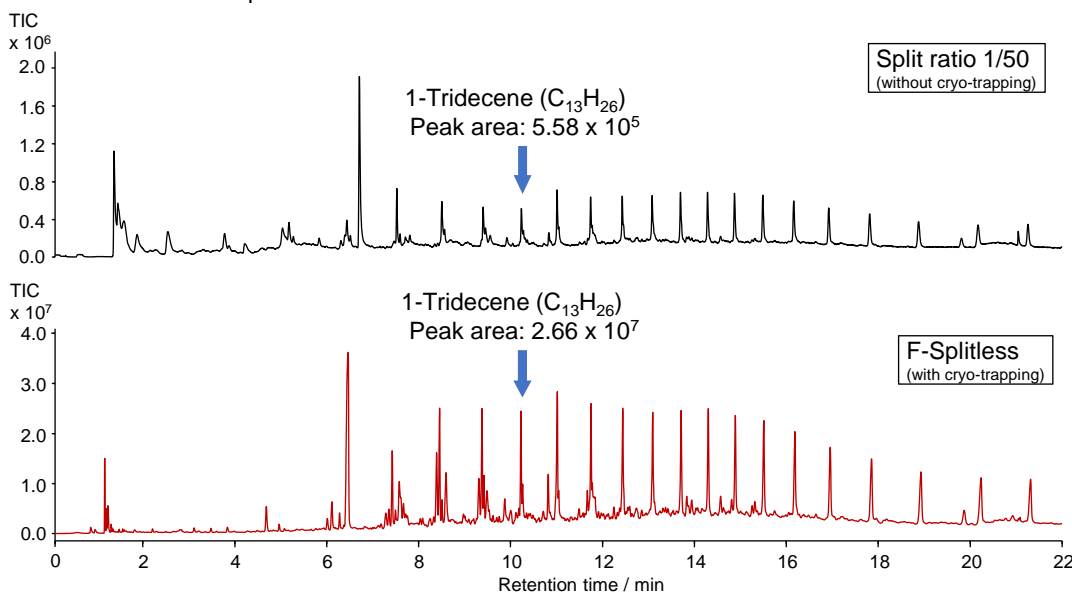


Fig. 2 Pyrograms of a sediment samples (Top: Split method, Bottom: F-Splitless method).

Furnace temp.: 600 °C, ITF temp.: 300 °C, GC inj. temp.: 300 °C, Inj. press.: 150 kPa (const. press.), Split ratio: 1/50, Pre-column: UA+50 (50 % diphenyl - 50 % dimethylpolysiloxane; L=2 m, i.d.=0.25 mm, df=1.0 µm), Separation column: UA+5 (5 % diphenyl - 95 % dimethylpolysiloxane; L=30 m, i.d.=0.25 mm, df=0.5 µm), GC oven: 40 (2 min hold) - 20 °C/min - 280 °C (10 min hold) - 40 °C/min - 320 °C (15 min hold, Back flush time: 20-40 min, GC/MS ITF temp.: 300 °C, MS scan range: m/z 29 - 350, MS scan rate: 4 scans/s, Sample amount: 4 mg.

1) K. Tei et al., *J. Anal. Appl. Pyrolysis* 168 (2022) 105707. 2) K. Matsui et al., *J. Anal. Appl. Pyrolysis* 149 (2020) 104834.

Keywords : Microplastics, Environmental sample, Sediment sample, Seabed sample

Products used : Multi-Shot Pyrolyzer, Multi-Functional Splitless Sampler, Auto-Shot Sampler, MicroJet Cryo-Trap, Eco-Cup LF, GC glass insert with filler, UAMP column kit, Vent-free GC/MS adapter, F-Search MPs 2.1

Applications : Environmental analysis, Trace analysis, General polymer analysis

Related technical notes : PYA1-151E (Part 2), PYT-037E, PYT-038E

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