Analysis of trace microplastics (MPs) in sediment samples
Part 2: Pyrogram and qualitative/quantitative results of sediment sample

[**Background**] In the previous note (PYA1-150E), sediment samples collected offshore were measured by pyrolysis (Py)-GC/MS using the F-Splitless injection method and it was found that the F-Splitless injection method was able to detect MPs with a higher sensitivity than the split injection method. In this note, qualitative and quantitative analysis of MPs in sediment samples are carried out using the F-Splitless injection method.

[**Experimental**] Sediment samples were provided by Dr. Scholz-Böttcher, ICBM, University of Oldenburg, and pretreated before measurements, as described in the previous note. 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 F-Splitless injection method at a pyrolysis temperature of 600 ºC using the analytical system and experimental conditions described in the previous note. Further, MP calibration standard[1], which is a polymer-silica mixture with known polymer contents, was used to make calibration curves of polymers. Qualitative and quantitative analysis of MPs were done using the F-Search MPs software[2].

[**Results**] A pyrogram of a sediment sample is shown in Fig. 1, where styrene monomer and linear hydrocarbons were mainly detected. The results of qualitative and quantitative analysis by F-Search MPs are shown in Table 1, where PE was the most abundant polymer. Other polymers such as PP, PMMA, PET, and N66 were also detected. In conclusion, MPs in the sediment samples were able to be qualitatively and quantitatively determined by F-Splitless injection method using the MP calibration standard and F-Search MPs.

Table 1 Qualitative/quantitative results of MPs in sediment sample (4.047 mg).

Polymer*	Prob. [%]	Qnty [µg]
PE	98.6	10.22
PP	90.4	0.23
PS	99.0	1.12
PMMA	99.9	0.15
PET	90.8	0.12
N66	87.1	0.25

Prob. : Percent matching score with the summation spectrum of the library
Qnty : Quantitative value
* PE : Polyethylene, PP: Polypropylene, PS: Polystyrene, PMMA: Polymethylmethacrylate, PET: Polyethylene terephthalate, N66: Nylon 6,6
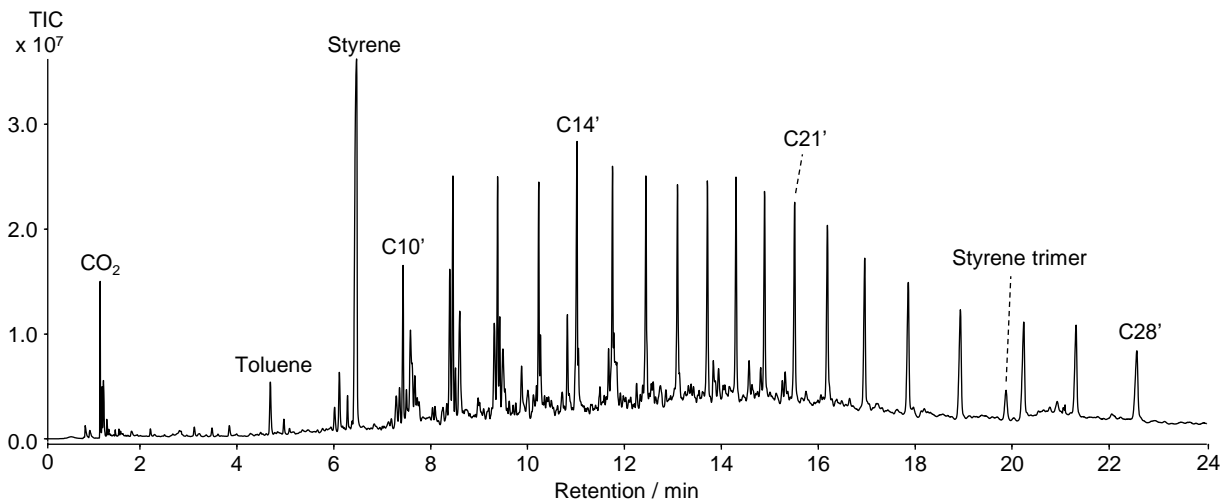

Fig. 1 Pyrogram of a sediment sample.

Furnace temp.: 600 ºC, ITF temp.: 300 ºC, GC Inj. Temp.: 300 ºC, Inj. Press.: 150 kPa (const. press.), F-Splitless injection method, 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 ºC/min - 280 ºC (10 min hold) - 40 ºC/min - 320 (15 min hold), Back flush time: 20~40 min, GC/MS ITF temp.: 300 ºC, MS scan range: *m/z* 29 - 350, MS scan rate: 4 scan/s, Sample amount: 4 mg.

1) M. Matsueda *et al., J. Anal. Appl. Pyrolysis* 154 (2021) 104993.
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 : PYA1-150E (Part 1), PYT-037E, PYT-038E, PYA1-143E, PYA1-144E, PYA1-145E

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

®: A registered trademark of Frontier Laboratories Ltd.