

Preparation and evaluation of calibration standard for microplastic (MP) analysis using SiO₂ as a diluent

Part 2: Pyrogram of the calibration standard for MP analysis

[Background] In the previous note (PYA1-143E), deactivated SiO₂ was selected as a diluent for the microplastic (MP) analysis of a mixed polymer sample. In this note, a pyrogram of the calibration standard (MPs-SiO₂) containing 12 types of polymers is shown, where deactivated SiO₂ was used as a diluent.

[Experimental] The MP calibration standard MPs-SiO₂ was prepared from a homogeneous mixture of deactivated SiO₂ and 12 polymers.¹⁾ The 12 polymers and their concentrations were determined considering the global production of plastics and their sensitivity in Py-GC/MS measurements. MPs-SiO₂ was analyzed by a Py-GC/MS system shown in Fig. 1.

[Results] A pyrogram of MPs-SiO₂ and the characteristic pyrolyzate and the indicator ion for each polymer are shown in Fig. 2. Table 1 summarizes the characteristic pyrolyzates. When the calibration standard sample using deactivated SiO₂ as a diluent was used, MDI, a pyrolyzate of PU, reacted with pyrolyzates of PET and nylon, so the MDI peak was not observed. Analysis of PU can be accomplished using calibration standard (MPs-CaCO₃) with CaCO₃ as a diluent (PYA1-146E ~ 148E). In the next note (PYA1-145E), calibration curves for each characteristic pyrolyzate are reported.

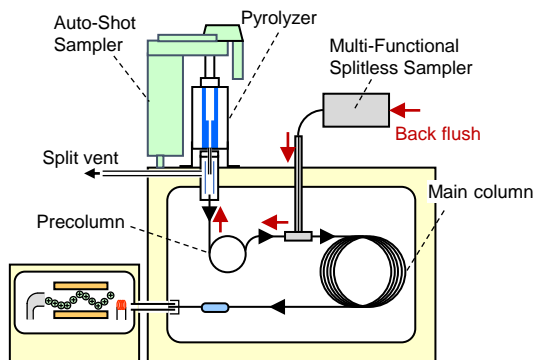


Fig.1 Analytical system for MPs analysis.

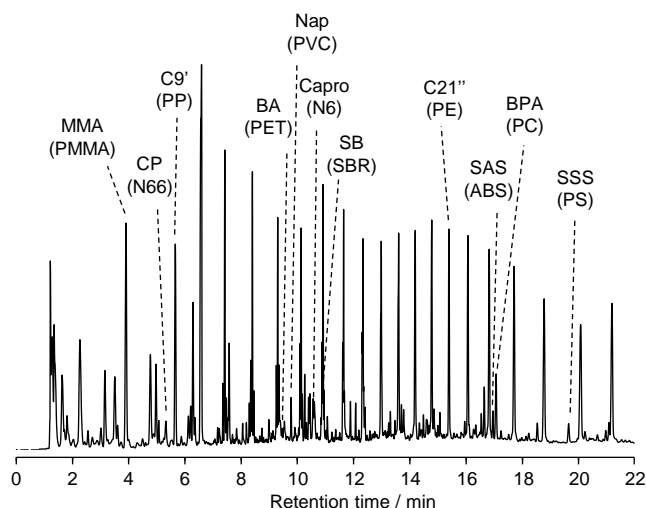


Fig. 2 Pyrogram of MPs-SiO₂.

Table 1 Characteristic pyrolyzate of each polymer and its indicator ion (*m/z*).

Polymer	Abbr.	Characteristic pyrolyzates	<i>m/z</i>
PE	C21''	1,20-Heneicosadiene	82
PP	C9'	2,4-Dimethyl-1-heptene	126
PS	SSS	2,4,6-Triphenyl-1-hexene	91
ABS	SAS	2-Phenethyl-4-phenylpent-4-enenitrile	170
SBR	SB	4-Phenylcyclohexene	104
PMMA	MMA	Methyl methacrylate	100
PC	BPA	Bisphenol A	213
PVC	Nap	Naphthalene	128
PU	MDI	4,4'-Diphenylmethane diisocyanate	250
PET	BA	Benzoic acid	122
N6	Capro	Caprolactam	113
N66	CP	Cyclopentanone	84
PU	MDI	4,4'-diphenylmethane diisocyanate	250

Furnace temp.: 600 °C, Furnace-interface temp.: 300 °C, GC Injector temp.: 300 °C, Injector press.: 150 kPa (constant press.), Split ratio: 1/50, Precolumn: UA*+50 (50 % diphenyl - 50 % dimethylpolysiloxane; L=2 m, i.d.=0.25 mm, df=1 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 (15 min hold) - 40 °C/min - 320 (10 min hold), Back flush time: 20 ~ 40 min, GC/MS interface temp.: 300 °C, 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.

Keywords : Microplastics, Calibration standard, Reference material, Diluent

Products used : Multi-Shot Pyrolyzer, Multi-Functional Splitless Sampler, Auto-Shot Sampler, MP calibration standard sample set (MPs-SiO₂), Eco-Cup LF, GC glass insert with filler, UAMP column kit, Vent-free GC/MS adapter, F-Search MPs

Applications : Environmental Analysis, Trace analysis, General polymer analysis

Related technical notes : PYA1-143E (Part 1), PYA1-145E (Part 3), PYA1-146E, PYA1-147E, PYA1-148E (MPs-CaCO₃)

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