

Preparation and evaluation of calibration standard for microplastic (MP) analysis using CaCO₃ as a diluent Part 2: Pyrogram of MPs-CaCO₃

[Background] In the previous note (PYA1-146E), when CaCO₃ was used as a diluent and catalyst, conversion of pyrolyzates of PET, PC and PU due to catalytic reactions were observed. In this report, pyrograms of the MP calibration standard (MPs-CaCO₃) using CaCO₃ as a diluent are described.

[Experimental] The MP calibration standard MPs-CaCO₃ was prepared by homogeneously mixing CaCO₃ and 12 polymers.¹⁾ The 12 polymer species and their contents were chosen in consideration of the global plastic production and their sensitivity in Py-GC/MS. Pyrograms of MPs-CaCO₃ were measured by the Py-GC/MS system shown in Fig. 1.

[Results] A pyrogram of MPs-CaCO₃ together with characteristic pyrolyzates for 12 polymers is shown in Fig. 2., Indicator ions for characteristic pyrolyzates are summarized in Table 1. While MDI, a pyrolyzate of PU, was not observable with MPs-SiO₂, it can now be detectable as MDA. In the next note (PYA1-148E), calibration curves of characteristic pyrolyzates for 12 polymers are described.

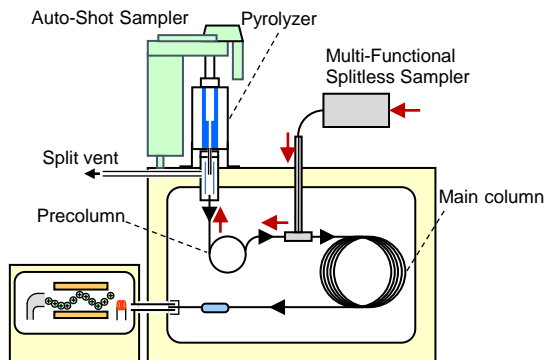


Fig. 1 Analytical system for MP analysis.

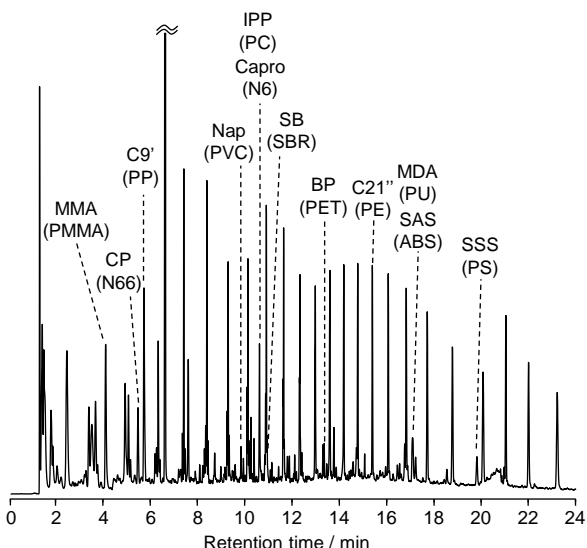


Fig. 2 Pyrogram of MPs-CaCO₃.

Table 1 Characteristic pyrolyzate of each of 12 polymers and its indicator ion (*m/z*).

Polymer	Characteristic pyrolyzate	<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	IPP 4-Isopropenylphenol	134
PVC	Nap Naphthalene	128
PU	MDA 4,4'-Methylenedianiline	198
PET	BP Benzophenone	182
N6	Capro Caprolactam	113
N66	CP Cyclopentanone	84

Py furnace temp.: 600 °C, 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), Main 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) T. Ishimura *et al.*, *J. Anal. Appl. Pyrolysis* 157 (2021) 105188.

Keywords : Microplastic, Calibration standard material, Reference material, Diluent

Products used : Multi-Shot Pyrolyzer, Multi-Functional Splitless Sampler, Auto-Shot Sampler, MP calibration standard set (MPs-CaCO₃), Eco-Cup LF, GC glass insert with filler, UAMP column kit, Vent-free GC/MS adapter, F-Search MPs 2.0

Applications : Environmental analysis, Trace analysis, General polymer analysis

Related technical notes : [PYA1-146E \(Part 1\)](#), [PYA1-148E \(Part 3\)](#), [PYA1-143E](#), [PYA1-144E](#), [PYA1-145E \(MPs-SiO₂\)](#)

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Frontier Laboratories Ltd.

Phone: (81)24-935-5100 Fax: (81)24-935-5102
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