

Preparation and evaluation of calibration standard for microplastic (MP) analysis using CaCO₃ as a diluent

Part 3: Calibration curves for polymers in MPs-CaCO₃

[Background] Previous note (PYA1-147E) reported a pyrogram of MP calibration standard (MPs-CaCO₃) and characteristic pyrolyzate of each of the 12 polymers. In this note, calibration curves of the characteristic pyrolyzates and the limit of detection (LOD) for the 12 polymers are reported.

[Experimental] 0.4 mg, 2 mg, and 4 mg of MPs-CaCO₃ were each placed in an Eco-Cup LF. Measurements were done by the method described in the previous note, and the peak areas of the extracted ion chromatograms (EICs) of the ions listed in Table 1 were used to create calibration curves for the 12 polymers. The detection limit (LOD=3.3 s/a , where a is the slope of the linear calibration curve and s is the standard deviation at the lowest concentration) is summarized in Table 1 for each of 12 polymers. The GC injector pressure was 150 kPa (constant pressure mode) with a split ratio of 1/50. The mass spectrometer was operated in a scan mode.

[Results] Fig. 1 shows the calibration curves for the 12 polymers in MPs-CaCO₃, which showed good linearity with a determination coefficient of $R^2 > 0.99$, except for PS. The calibration curve for PS was found to be quadratic. For PET, which showed a poor linearity with MPs-SiO₂, the linearity was improved by the conversion of benzoic acid, a pyrolyzate, to BP by the catalytic reaction with CaCO₃.

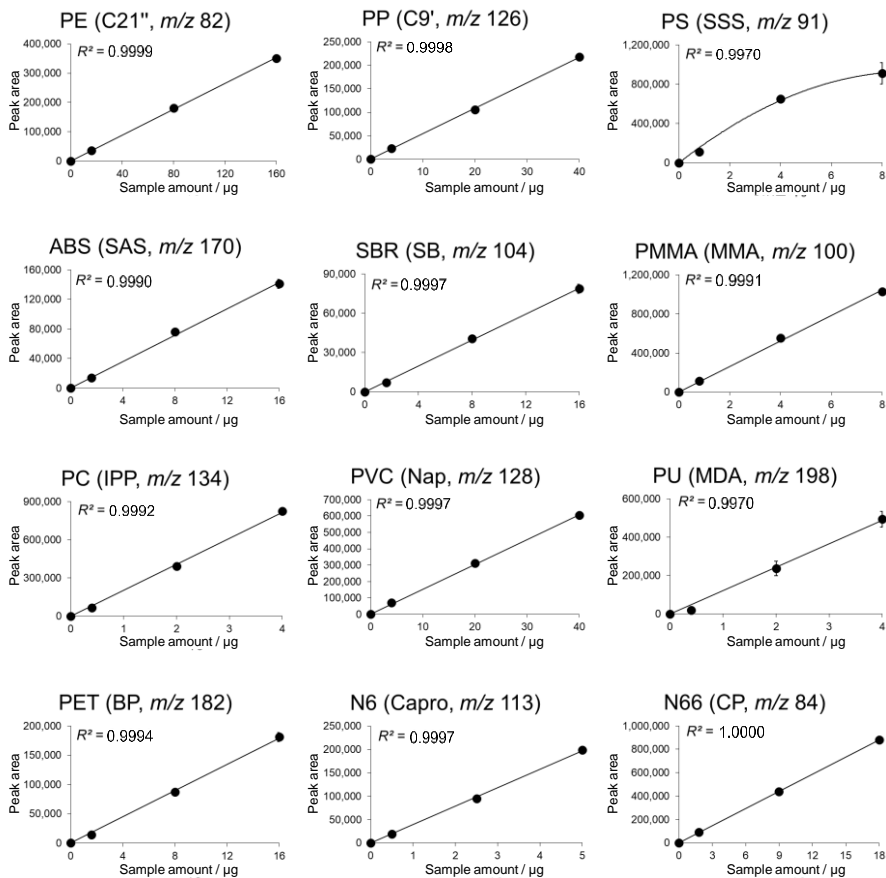


Table 1. Characteristic pyrolyzate for 12 polymers and LOD.

Polymer	Characteristic pyrolyzate*	m/z	LOD [µg]
PE	C21"	82	1.63
PP	C9'	126	0.54
PS	SSS	91	0.23
ABS	SAS	170	0.21
SBR	SB	104	0.38
PMMA	MMA	100	0.09
PC	IPP	134	0.06
PVC	Nap	128	0.51
PU	MDA	198	0.18
PET	BP	182	0.18
N6	Capro	113	0.10
N66	CP	84	0.35

* See previous note (PYA1-147E) for abbreviation.

Fig. 1 Calibration curves for 12 polymers created using MPs-CaCO₃.

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), PYA1-147E (Part 2), PYA1-143E, PYA1-144E, PYA1-145E (MPs-SiO₂)

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