Microplastic Calibration Using EasyPyCal™ Standards

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Application Note
Microplastics

Abstract
This application note demonstrates the use of a Pyroprobe 6150 with a commercially available mixed microplastics standard to build microplastic calibration curves

Introduction
ASTM D8401-24 defines a calibration-based method for the quantification of microplastics in water, in which polymer concentrations are set so their markers can be reliably quantified with minimal interference between polymers. However, preparing a mixed microplastic standard that contains all 12 microplastics specified is a time-consuming endeavor. It involves preparing 4 solutions of 10 soluble polymers, as well as a solid standard of the insoluble polymers, polypropylene and polyethylene, to be in a solid mineral diluent, which must be homogeneous. It is time-consuming, as well as not environmentally-friendly.

To ease the burden, LabTech CDS Analytical supplies the EasyPyCal™ Py-GC-MS calibration standard: ASTM D8401-24 kit (CDS p/n 6203-5095) developed by Microplastic Solution to support qualitative and quantitative analysis, allowing for easy calibration curve development. Having the 12 specified polymers evenly dispersed in $CaCO_3$ or SiO_2 helps for easy weighing milligram quantities on a semi-microbalance, in which the polymer themselves are in microgram amounts.

Experiment Setup
The vial containing EasyPyCal™ standard in $CaCO_3$ and 4 stainless steel balls was placed on a vortex blender for 3 minutes. Sample amounts 0.4 to 4 mg (exact weights recorded) were added to DISC tubes in triplicate for each level of 5 calibration levels.

Pyrolysis

Pyroprobe 6150 Autosampler

Pyrolysis: 600°C 40 sec

Interface: 300°C
Transfer Line: 325°C
Valve Oven: 300°C

GC-MS

Pre Column:
Rxi-17Sil MS (2m x 0.25mm x 0.25µm)
Column:
5% phenyl (30m x 0.25mm x 0.50µm)
Carrier:
He 1.25mL/min, 50:1 spl
Injector: 360°C
Oven: 40°C for 2 minutes
12°C/min to 320°C (20min)
Ion Source: 230°C
Mass Range: 35-400amu

Results and Discussion
The EasyPyCal™ standard contains Poly(ethylene) PE, poly(propylene) PP, poly(vinyl chloride) PVC, poly(ethylene terephthalate) PET, poly(styrene) PS, poly(carbonate) PC, styrene-butadiene rubber SBR, acrylonitrile-butadiene-styrene ABS, poly(methyl methacrylate) PMMA, Nylon 6 PA6, Nylon 6.6 PA6.6, and an MDI-based polyurethane PU in calcium carbonate, each one at concentrations specified in specified in ASTM Method D8401-24 designed to identify and quantify microplastic particles and fibers in water by Py-GC/MS.

Each polymer was identified and quantified using the an m/z of its specified target compound, provided in Table 1.

Table 1. Polymers and Target Ions.

Microplastic	Abbr.	Target Compound	m/z	R.T.
Polymethyl methacrylate	PMMA	Methyl Methacrylate	100	5.70
Nylon 6.6	N66	Cyclopentanone	84	7.14
Polypropylene	PP	2,4-Dimethyl-1- heptene	126	7.79
Styrene-Butadiene Rubber	SBR	4-Vinylcyclohexene	54	7.78
Polyvinylchloride	PVC	Naphthalene	128	13.58
Nylon 6	N6	Caprolactam	113	14.71
Polycarbonate	PC	4-Isopropenyl phenol	134	14.93
Polyethylene terephthalate	PET	Benzophenone	182	19.07
Polyethylene	PE	1,20-Heneicosadiene	82	22.68
MDI Polyurethane	PU	4-4'-Methylenedianiline	198	24.24
Acrilonitrile butadiene styrene copolymer	ABS	2-Phenyethyl-4-phenylpent-4-enenitrile	170	24.47
Polystyrene	PS	Styrene Trimer	91	28.10

Figure 1 provides Extracted Ion Chromatograms (EICs) of all the Target Ions.

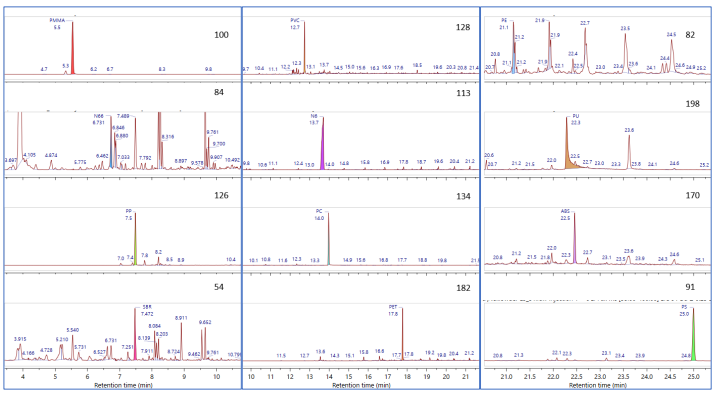

Figure 1. Extracted Ion Chromatograms of Target Ions.

The average calibrations yielded r^2 of 0.99 for every microplastic Target Ion.

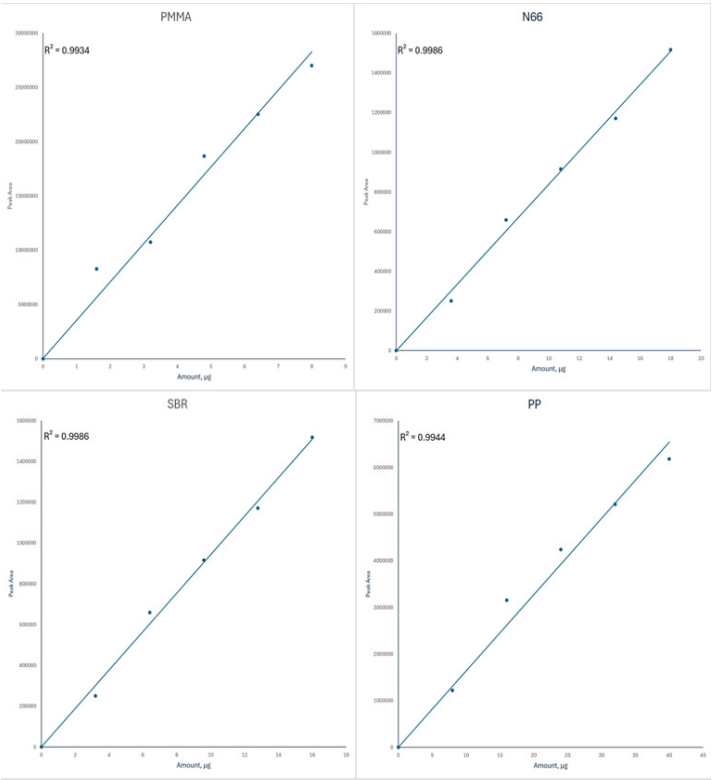

Figure 2. Target Ion Calibration Curves.

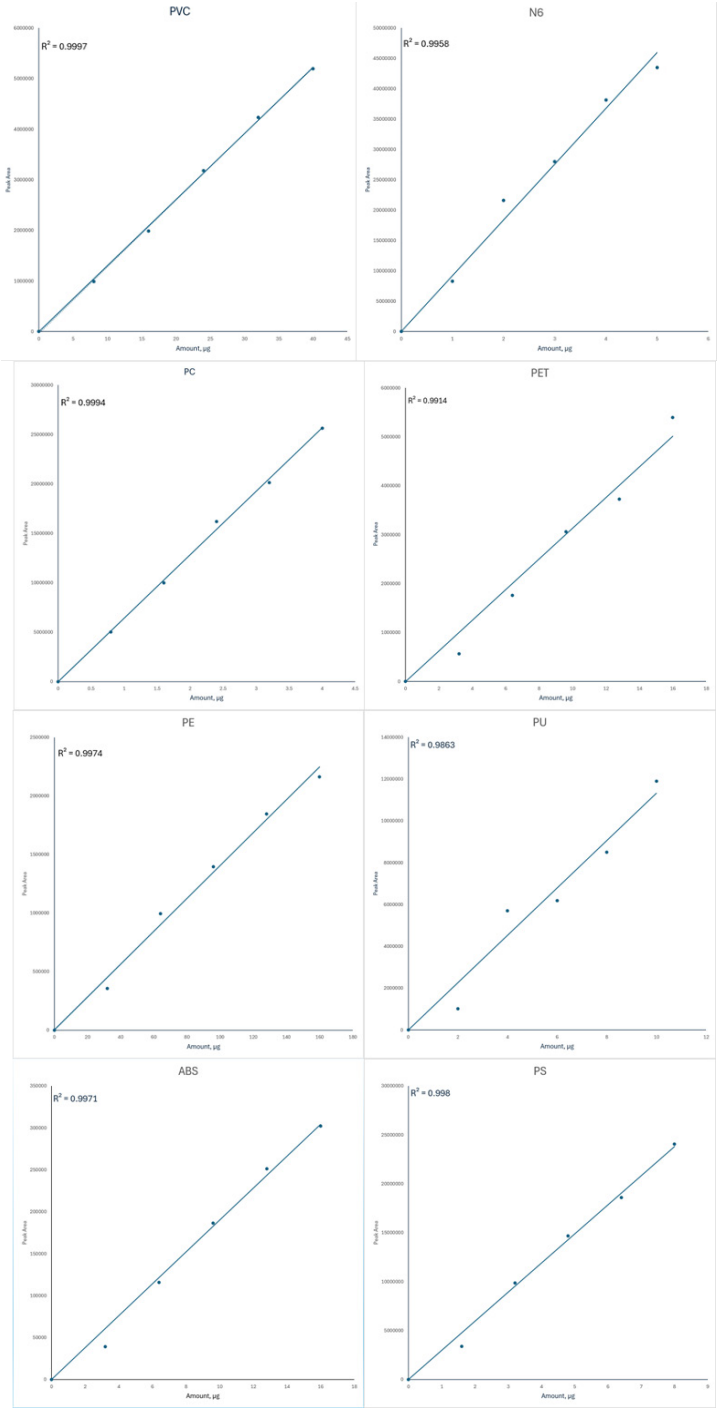

Figure 2 (cont'd). Target Ion Calibration Curves.

Conclusion

The powerful combination of an EasyPyCal™ Py-GC-MS micro-plastic calibration standard and a Pyroprobe from LabTech CDS Analytical assists productivity for quantitative microplastic analysis.