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Certificate of Preparation – Batch #067-4

12 polymers-$CaCO_3$ blend (50 µg/mg; 5% w/w)

This Certificate of Preparation (COP) provides information on the composition and preparation of the polymer reference material intended for use in the pyrolysis-gas chromatography–mass spectrometry (Py-GC-MS) applications.

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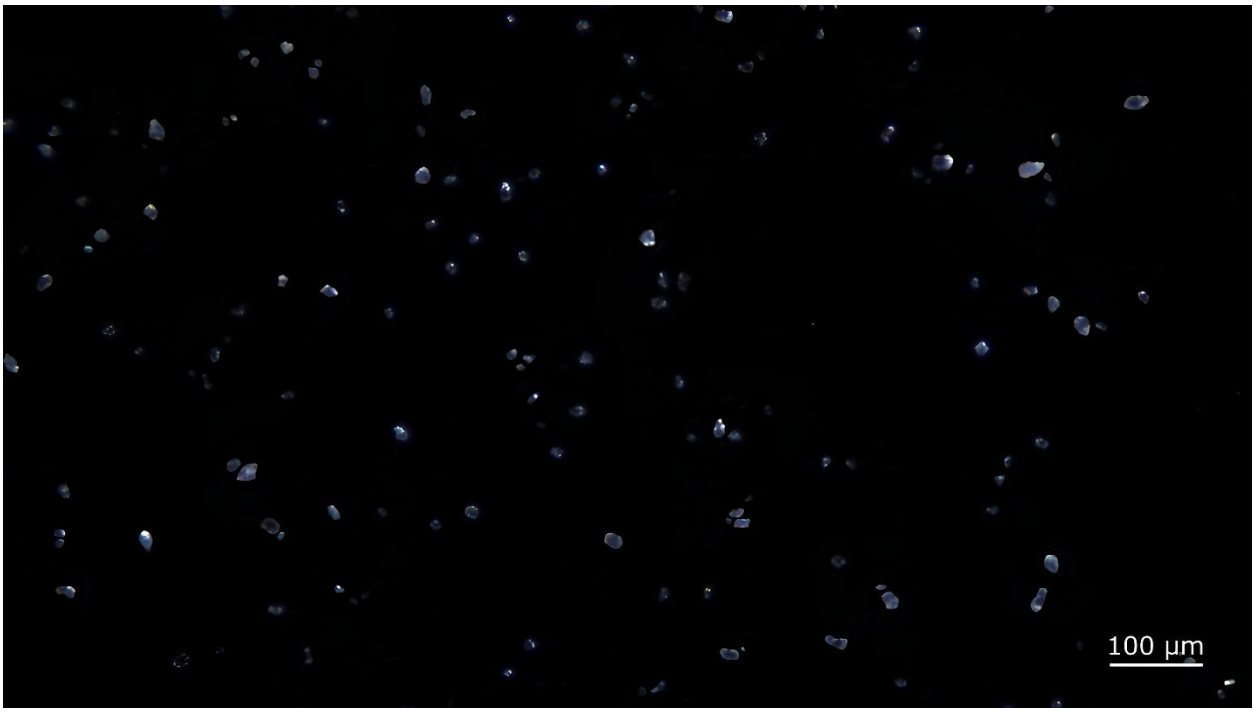
1. Composition and preparation

1.1. Polymers

Batch #067 corresponds to the Advanced kit consisting of 12 common polymers (PE, PP, PVC, PET, PS, PC, SBR, ABS, PMMA, PA6, PA66, and PU [MDI-based]). Each polymer type is provided in the form of microplastic (MP) fragments with a maximum diameter of approximately 50 µm, obtained by sieving through a 300-mesh grid (Fig. 1).

Fig. 1: Example: Photomicrograph captured under critical-angle darkfield illumination (CADLFI) of PE fragments sieved through a 300-mesh grid.



The MP fragments are weighed and dispersed in Calcium carbonate ($CaCO_3$) diluent, ensuring a traceable distribution of polymer mass within the matrix. Each MP fragment weighs less than 10 ng; therefore, 1 µg of MP powder typically consists of at least 100 particles (at the lowest available concentration of 1.0 µg/mg [0.1 % (w/w)]), ensuring reliable particles dispersion within

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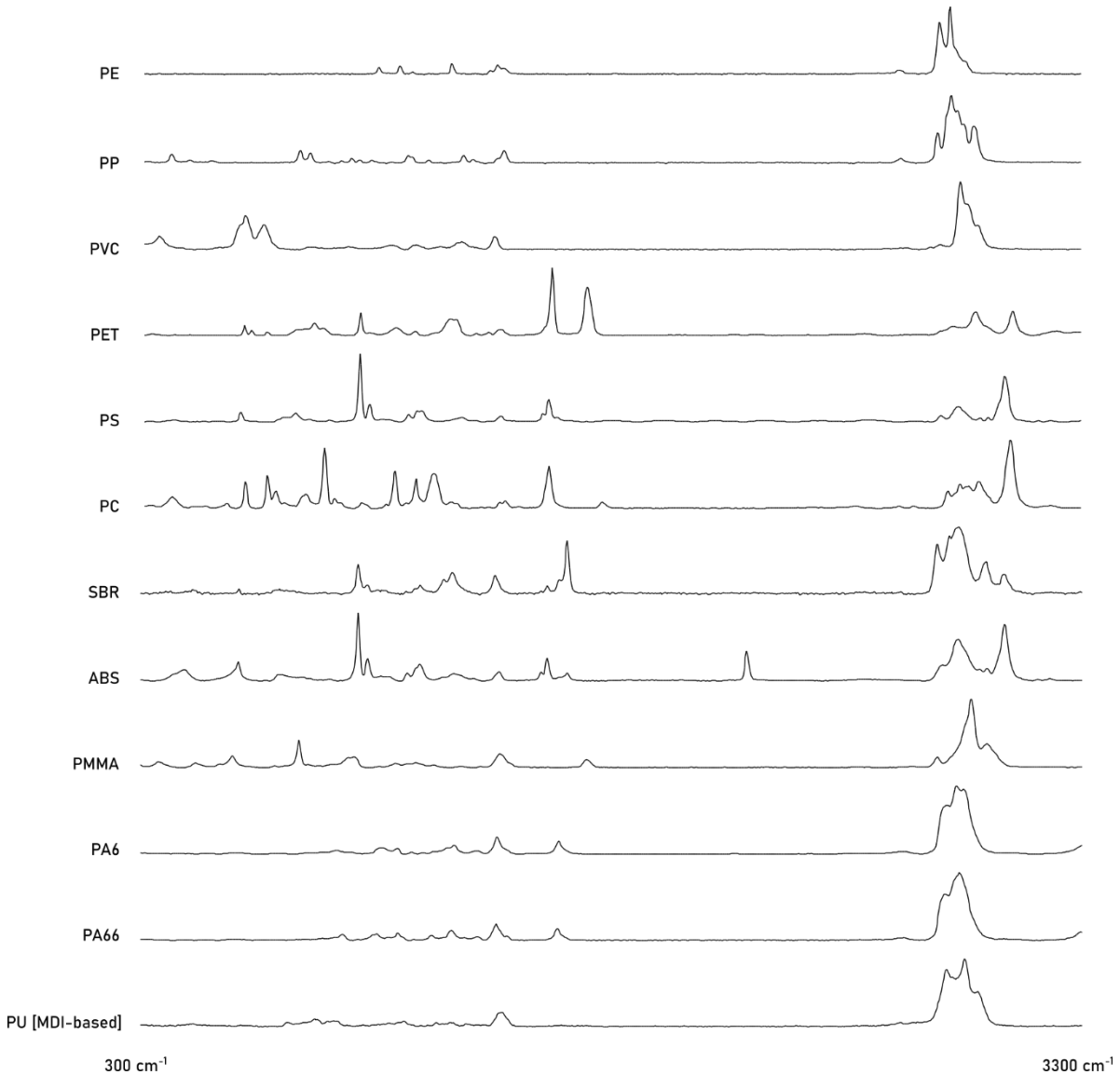
the $CaCO_3$ matrix. The nature and purity of each polymer were confirmed by Raman spectroscopy (Fig. 2).

Fig. 2: Raman spectra of the twelve polymers used in Batch #067. Raman analysis was conducted at a controlled room temperature (22°C) using a Horiba (Jobin Yvon, France) LabRAM Soleil equipped with a high stability air-cooled He–Cd 532 nm laser diode and Nikon LV-NUd5 100x objective. The laser power was set to 6.3% (5.7 mW). Spectra were collected in the 300–3300 cm^{-1} range using 600 grooves/cm grating with a 100 µm split. The spectra acquisition time was set to 3s with 3x accumulation.



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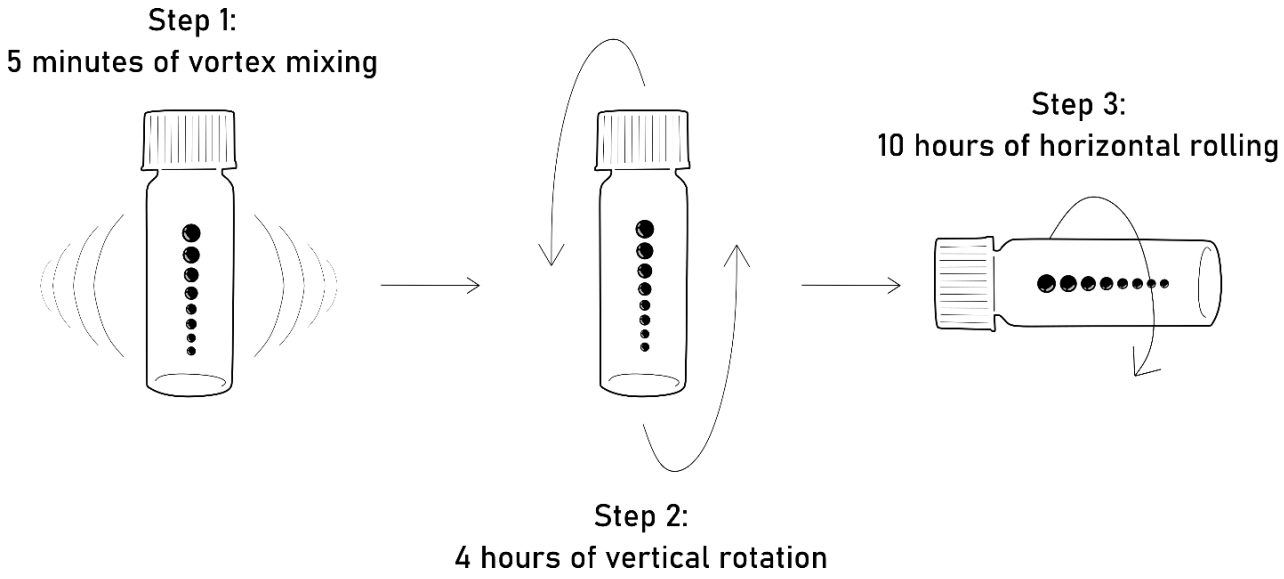
1.2. Diluent

$CaCO_3$ powder was calcined in a muffle kiln at 530°C prior to use to eliminate possible residual organic matter and ensure material purity.

1.3. Preparation of polymers-diluent blend

Twelve polymers were dispersed in $CaCO_3$ diluent at the concentration of 5% (w/w). To ensure complete homogeneity, polymer-diluent blend was mixed for 14 hours using continuous agitation, consisting of an initial 5 minutes of vortex mixing, followed by 4 hours of vertical rotation and 10 hours of horizontal rolling (Fig. 3). To enhance dispersion and facilitate the breakdown of potential aggregates, eight 304 stainless-steel spheres of different diameters (2.0 mm, 2.5 mm, 3.0 mm, 3.5 mm, 4.0 mm, 4.5 mm, 5.0 mm, and 5.5 mm) were added to the mixture.

Fig. 3: Scheme of polymer–$CaCO_3$ blend homogenization process.



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1.4. Py-GC-MS analysis of polymers-diluent blend

Py-GC-MS calibration standard samples were analyzed by CDS Analytical to assess homogeneity, linearity, and repeatability using a 6150 Pyroprobe coupled with Py-GC-MS. Calibration was performed over five concentration levels, with three replicates per level, using concentration ranges and target compounds for each polymer based on ASTM D8401-24 (Fig. 4). The analysis was conducted under the following conditions:

- Pyrolysis temperature: 600 °C (40 s)
- Inlet: Split injection mode, split ratio 1 :50
- Column flow : 1.25 mL/min
- Oven: 40 °C to 300 °C at 12 °C/min
- Pre-column: Rxi-17Sil MS, 2 m × 0.25 mm i.d., 0.25 µm film thickness
- Column: Rxi-5ms, 30 m × 0.25 mm i.d., 0.50 µm film thickness
- Scan: 29-400 amu.

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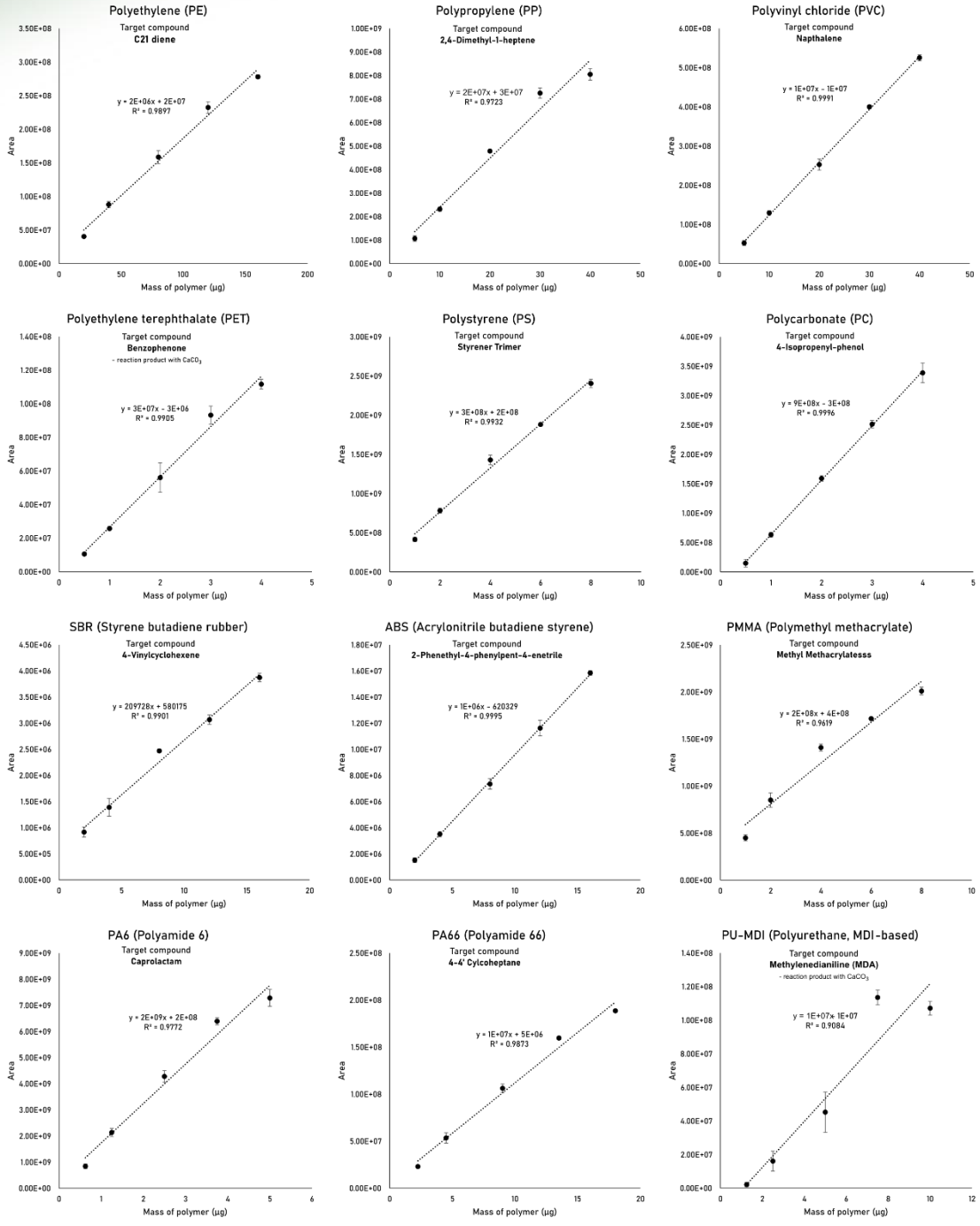
Fig. 4: Calibration curves for the 12 polymer types were obtained over five concentration levels, with three replicate measurements at each level.



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2. Sample details

2.1. Stock mixture

Stock mixture (Batch #067) with a total mass of 5.0 g was prepared, containing approximately 50 µg/mg (5.0% w/w) of each polymer. The specific weighed amounts are provided in Table 1.

Table 1: Polymer concentrations in the stock mixture used for the current sample.

Polymer type	Mass of polymer, (mg)	Mass of $CaCO_3$ diluent, (mg)	Total mass of stock mixture, (mg)	Polymer concentration, (µg/mg)	Polymer concentration, (%)
PE	250.0	2000.4	5003.0	50.0	5.0
PP	250.1			50.0	5.0
PVC	250.2			50.0	5.0
PET	250.2			50.0	5.0
PS	250.2			50.0	5.0
PC	250.4			50.0	5.0
SBR	250.2			50.0	5.0
ABS	250.2			50.0	5.0
PMMA	250.1			50.0	5.0
PA6	250.2			50.0	5.0
PA66	250.3			50.0	5.0
PU [MDI-based]	250.5			50.1	5.0
Mean				50.0	5.0

2.2. Batch #067-4

Batch #067-4 (mass = 1g) was subsampled directly from the homogenized stock mixtures without any dilution, preserving the original concentrations. Three 316L stainless-steel spheres with diameter of 3 mm were introduced into the mixture for further homogenization before use.

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3. Contamination control

All sample processing steps were carried out under contamination-controlled conditions. Operators were equipped with 100% cotton lab coats and nitrile gloves. All sampling tools were made of glass, metal or fluoropolymers (PTFE, PFA) to prevent contact with commodity plastics. Utensils (vials, stainless-steel spheres, etc.) were rinsed with abundant tap water, Milli-Q and ethanol. All glass tools were calcined in a muffle kiln for 2 hours at 530 °C.

4. Disclaimer

The information given in this COP is correct to the best of our knowledge at the time of issue. Microplastic Solution (MPS) makes no warranties, express or implied, and assumes no liability in connection with the use of this product.

4.1. Support

We are dedicated to helping researchers succeed. If you experience any issues with this material or require additional information, please reach out to us at contact@microplasticsolution.com. We are committed to supporting researchers in their micro- and nanoplastic analyses.

Approved by:

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Abbreviations

COP	Certificate of Preparation
Py-GC-MS	Pyrolysis-gas chromatography-mass spectrometry
MP	Microplastic
PE	Polyethylene
PP	Polypropylene
PVC	Polyvinyl chloride
PET	Polyethylene terephthalate
PS	Polystyrene
PC	Polycarbonate
SBR	Styrene butadiene rubber
ABS	Acrylonitrile butadiene styrene
PMMA	Polymethyl methacrylate
PA6	Polyamide 6
PA66	Polyamide 66
PU	Polyurethane
MDI	4,4′-diphenylmethane diisocyanate

Units

mg	Milligram
µg	Microgram
ng	Nanogram
cm	Centimeter
µm	Micrometer
w/w	Weight by weight
°C	Degree Celsius (temperature)