



## Certificate of Preparation – Batch #071-4

### 12 polymer-CaCO<sub>3</sub> blend (ASTM D8401-24 kit)

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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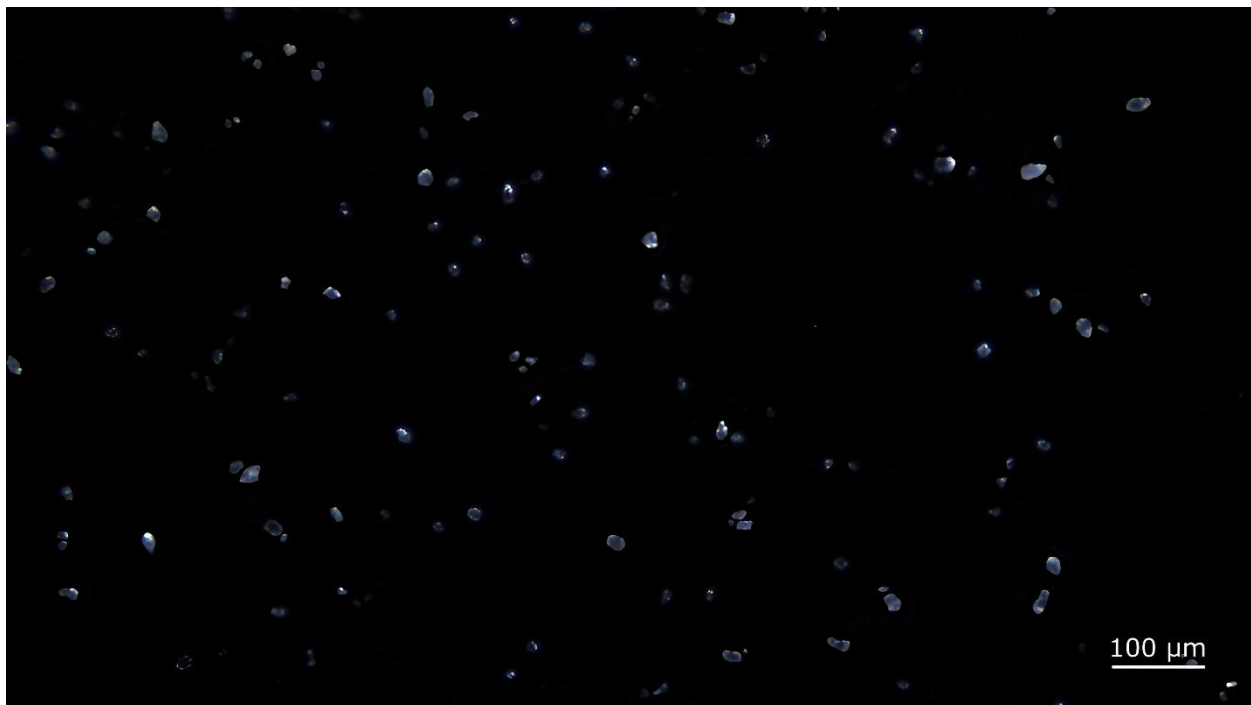


## 1. Composition and preparation

### 1.1. Polymers

Batch #071 corresponds to the ASTM D8401-24 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 an average 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.

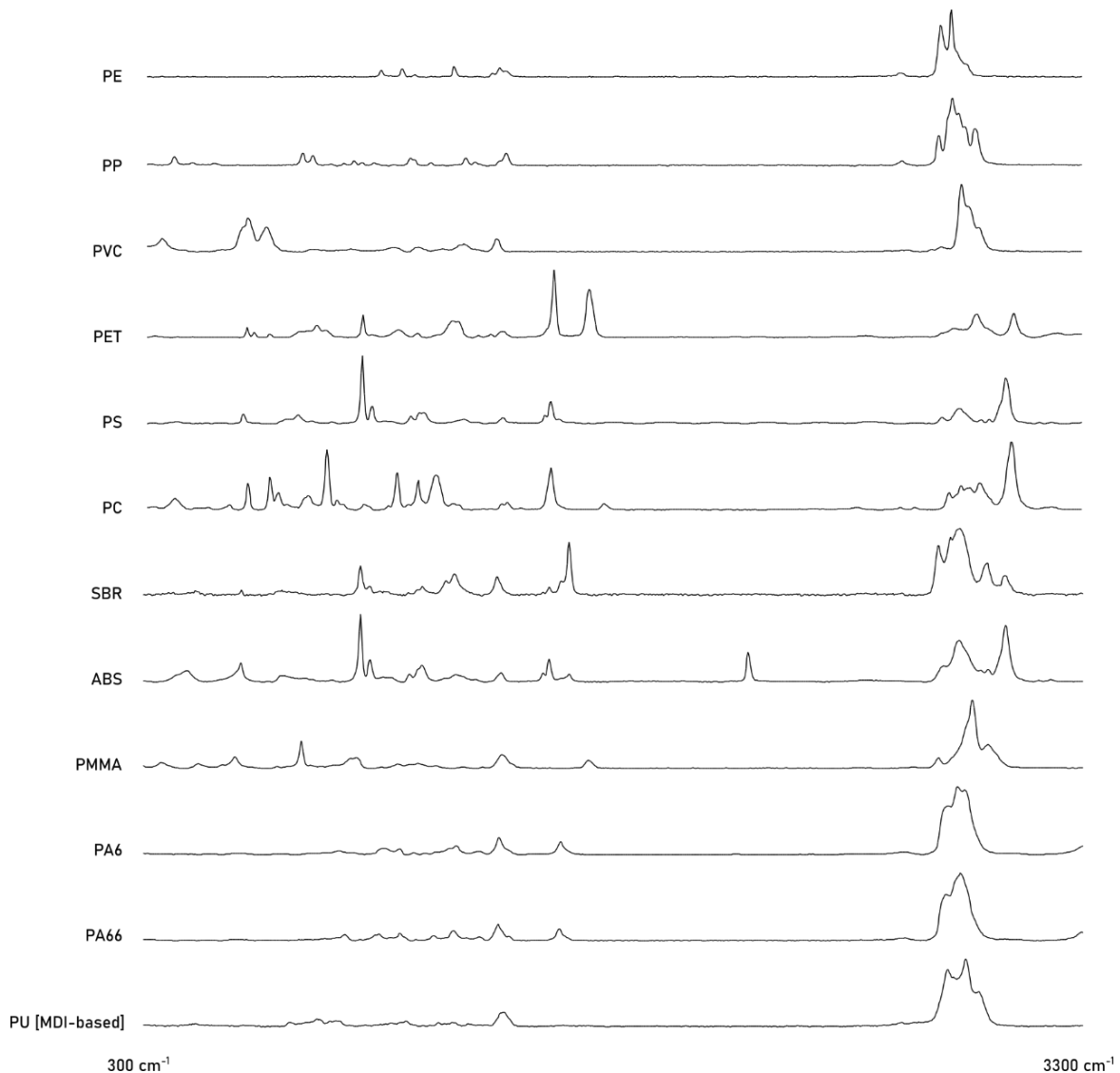


The MP fragments are weighed and dispersed in Calcium carbonate (CaCO<sub>3</sub>) diluent, ensuring a traceable distribution of polymer mass within the matrix. Each MP fragment weighs less than 10 ng; therefore, 1 µg of MP powder typically consists of at least 100 particles (at the lowest available concentration of 1.0 µg/mg [0.1 % (w/w)]), ensuring reliable particles dispersion within



the CaCO<sub>3</sub> 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 #071. 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<sup>-1</sup> range using 600 grooves/cm grating with a 100 μm split. The spectra acquisition time was set to 3s with 3x accumulation.





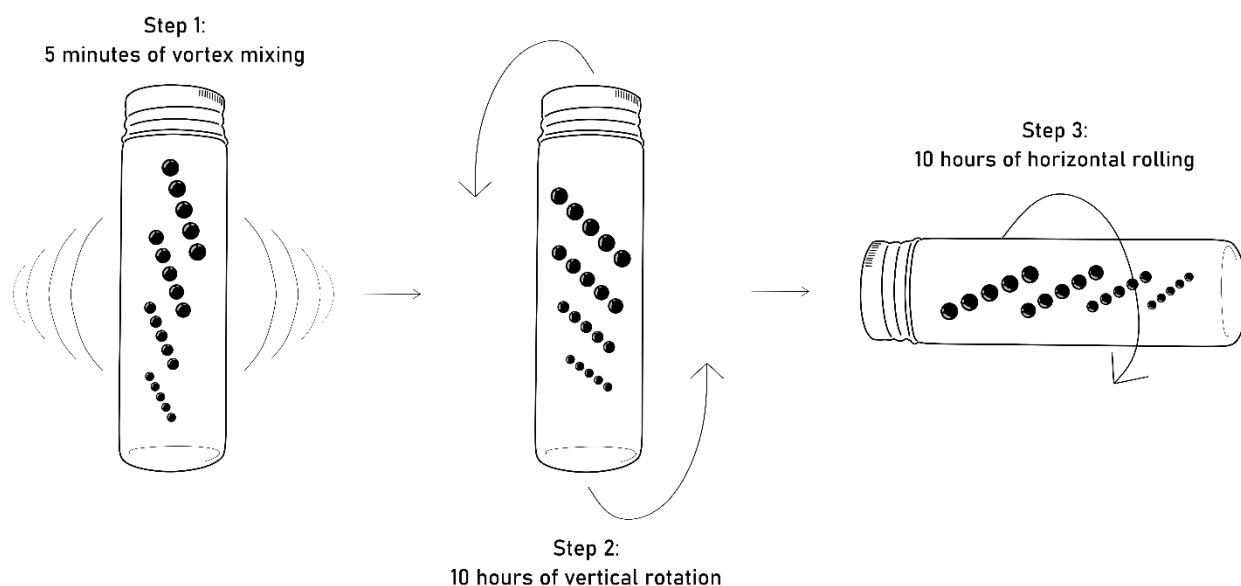
## 1.2. Diluent

CaCO<sub>3</sub> 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<sub>3</sub> diluent at the concentrations in accordance with ASTM D8401-24. To ensure complete homogeneity, polymer–diluent blend was mixed for 20 hours using continuous agitation, consisting of an initial 5 minutes of vortex mixing, followed by 10 hours of vertical rotation and 10 hours of horizontal rolling (Fig. 3). To enhance dispersion and facilitate the breakdown of potential aggregates, twenty 304 stainless–steel spheres of different diameters (3.0 mm, 4.0 mm, 5.0 mm, and 6.0 mm) were added to the mixture.

Fig. 3: Scheme of polymer–CaCO<sub>3</sub> blend homogenization process.





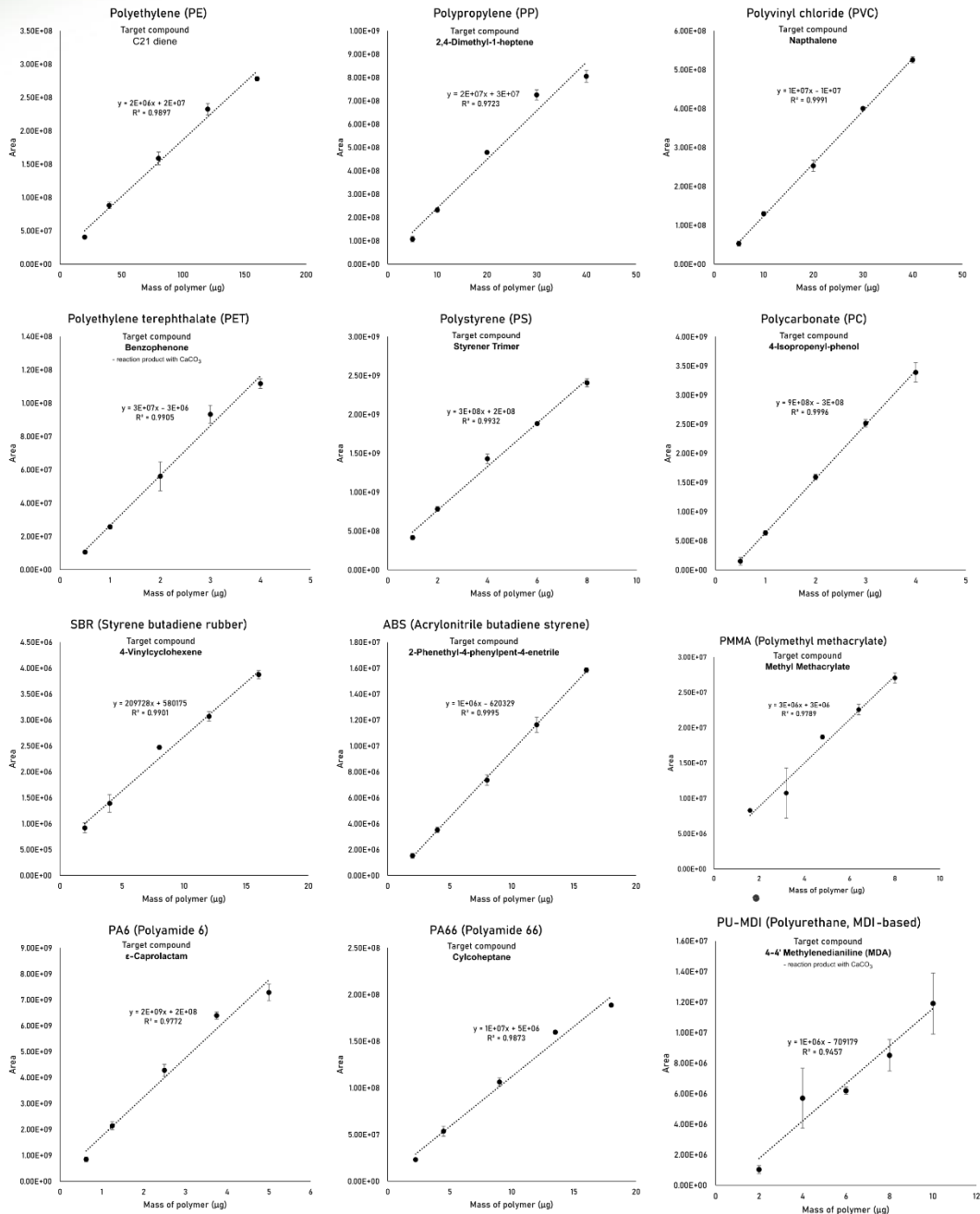
#### 1.4. Py-GC-MS analysis of ASTM D8401-24 kit

ASTM D8401-24 standard sample 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.



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





## 2. Sample details

### 2.1. Stock mixture

Stock mixture (Batch #071) with a total mass of 20.0 g was prepared, with polymer concentrations specified by ASTM D8401-24. The specific weighed amounts concentrations 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 <sub>3</sub> diluent, (mg)	Total mass of stock mixture, (mg)	Polymer concentration, (µg/mg)	Polymer concentration, (%)
PE	800.1	18295.3	20001.7	40.0	4.0
PP	200.1			10.0	1.0
PVC	200.3			10.0	1.0
PET	80.1			4.0	0.4
PS	40.1			2.0	0.2
PC	20.0			1.0	0.1
SBR	80.3			4.0	0.4
ABS	80.1			4.0	0.4
PMMA	40.2			2.0	0.2
PA6	25.0			1.25	0.125
PA66	90.1			4.5	0.5
PU	50.0			2.5	0.2

### 2.2. Batch #071-4

Batch #071-4 (mass = 1.0 g) 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.

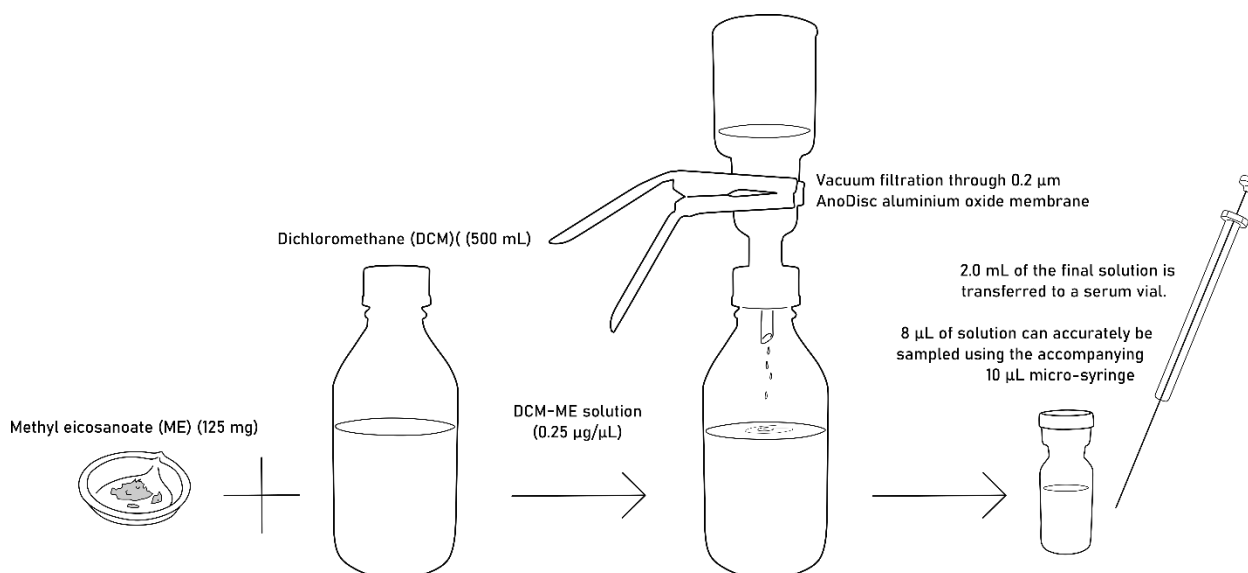


## 2.3. Methyl Eicosanoate in Dichloromethane (ME-DCM) solution (0.25 µg/µL)

### 2.3.1. Preparation of ME-DCM reference solution

The ME-DCM reference solution was prepared by dissolving 125 mg of methyl eicosanoate (ME) in 500 mL of dichloromethane (DCM), resulting in a final concentration of 0.25 µg/µL. The solution was subsequently filtered through a 0.2 µm Anodisc aluminum oxide membrane using vacuum filtration to remove any particulate contaminants and ensure a clean, homogeneous reference solution suitable for trace-level analytical applications. Following filtration, 2.0 mL of the final solution is transferred into a separate vial for routine laboratory use (Fig. 4).

**Fig. 4:** Preparation workflow of the ME-DCM reference solution. Methyl eicosanoate (ME) (125 mg) is dissolved in 500 mL dichloromethane (DCM) to obtain a solution with a concentration of 0.25 µg/µL. The solution is filtered through a 0.2 µm Anodisc membrane using vacuum filtration to remove particulate contamination. A 2.0 mL aliquot is then transferred to a dedicated vial for routine use. Immediately prior to Py-GC-MS analysis, 8 µL of the ME-DCM solution is dosed into the pyrolysis cup using a microsyringe, corresponding to 2.0 µg of methyl eicosanoate per analysis.





### 2.3.2. Intended use

The ME-DCM solution is intended to be added to the samples as an analytical check standard. ME serves as an indicator of the analytical performance for each injection. It is stable at typical pyrolysis temperatures and does not react with polymer pyrolysis products. Therefore, it is used to monitor the performance and consistency of the GC-MS system during analysis.

### 2.3.3. Aliquoting and dosing procedure

Prior to analysis, the solution must be dosed as follows:

- Using the supplied 10  $\mu\text{L}$  microsyringe, withdraw exactly 8  $\mu\text{L}$  of the ME-DCM solution.
- Carefully dispense the entire volume into the bottom of the pyrolysis sample cup and allow the solvent to evaporate completely.
- After evaporation, add the sample powder to the cup.

At a concentration of 0.25  $\mu\text{g}/\mu\text{L}$ , dosing 8  $\mu\text{L}$  delivers exactly 2.0  $\mu\text{g}$  of ME into the sample cup, ensuring consistent and traceable internal standard for quantitative GC-MS analysis.

## 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](mailto:contact@microplasticsolution.com). We are committed to supporting researchers in their micro- and nanoplastic analyses.

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A handwritten signature in black ink, appearing to be 'OH'.

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A handwritten signature in blue ink, appearing to be 'N. Yakovenko'.



## 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
DCM	Dichloromethane
ME	Methyl eicosanoate

## Units

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