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MICROPLASTIC solution

Certificate of Preparation – Batch #025-1-1

PVC-$CaCO_3$ blend (1.0 µg/mg; 0.1% 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.

Table of contents

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1. Composition and preparation

1.1. Polymer

Batch #025 consists of PVC, 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 PVC 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 1000 particles (at the lowest available concentration of 1.0 µg/mg [0.1 % (w/w)]), ensuring reliable particles dispersion within the $CaCO_3$ matrix. The nature and purity of the polymer were confirmed by Raman spectroscopy (Fig. 2).

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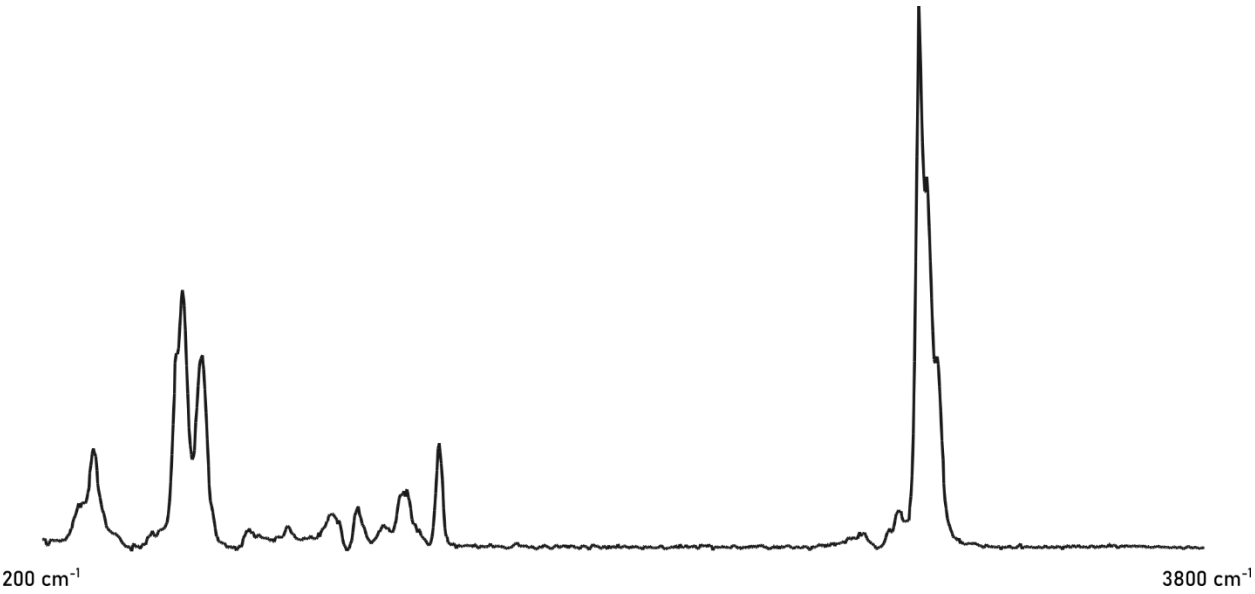
Fig. 2: Raman spectrum of the PVC fragments used in Batch #025. Raman analysis was conducted at a controlled room temperature (22°C) using a Horiba (Jobin Yvon, France) LabRAM Soleil equipped with a high stability air-cooled He–Cd 532 nm laser diode and Nikon LV-NUd5 100x objective. The laser power was set to 6.3% (5.7 mW). Spectra were collected in the 200–3800 cm^{-1} 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

$CaCO_3$ 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 polymer-diluent blend

PVC fragments were dispersed in $CaCO_3$ diluent at the concentration of 5% (w/w). To ensure complete homogeneity, polymer-diluent blend was mixed for 28 hours using continuous agitation, consisting of an initial 5 minutes of vortex mixing, followed by 4 hours of vertical rotation and 24 hours of horizontal rolling (Fig. 3). To enhance dispersion and facilitate the

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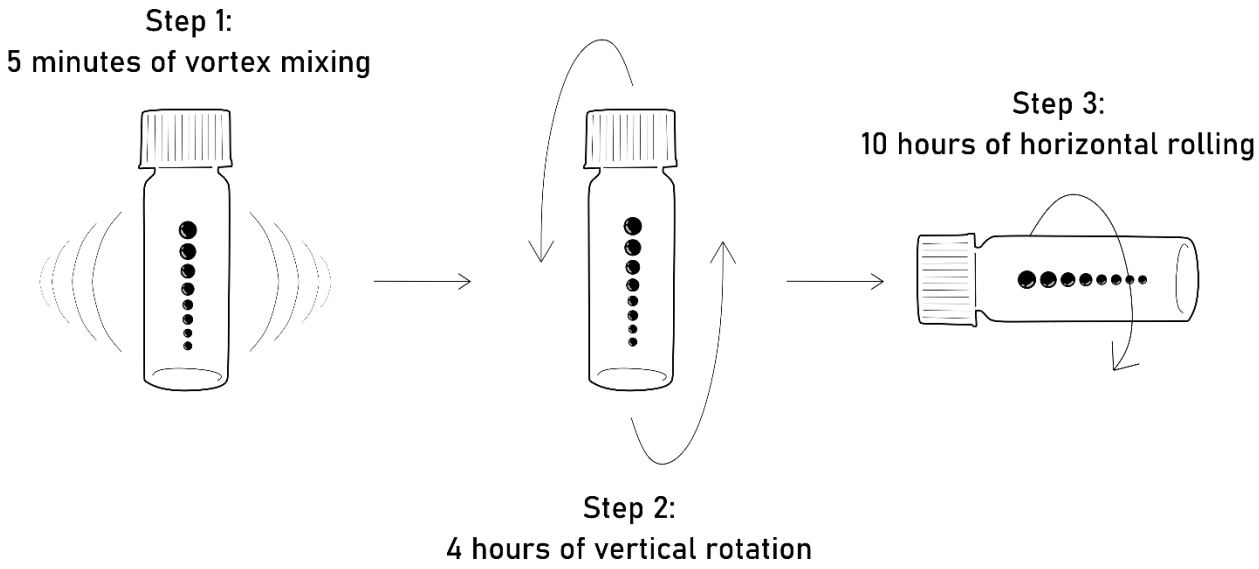
breakdown of potential aggregates, eight stainless-steel spheres of different diameters (2.0 mm, 2.5 mm, 3.0 mm, 3.5 mm, 4.0 mm, 4.5 mm, 5.0 mm, and 5.5 mm) were added to the mixture.

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



2. Sample contents

2.1. Stock blend

Stock blend (Batch #025) with a total mass of 5.0 g was prepared, containing approximately 50 µg/mg (5.0% w/w) of PVC. The specific weighed amounts are provided in Table 1.

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Table 1: Polymer concentration of stock mixture used in 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, (%)
PVC	250.3	4750.2	5000.5	50.1	5.0

2.2. Batch #025-1-1

Following preparation of the stock mixture, Batch 025-1-1 was produced through a two-step dilution. Details of the dilution volumes and resulting concentrations are provided in Table 2.

Table 2: Polymer concentration of diluted mixture (Batch #025-1-1).

Dilution step / Resulting Batch	Stock Batch used	Mass of Stock Batch, (mg)	Mass of $CaCO_3$ diluent, (mg)	Total mass, (mg)	Polymer concentration, (µg/mg)	Polymer concentration, (%)
1 – Batch #025-1	#025	200.5	800.3	1000.8	10.0	1.0
2 – Batch #025-1-1	#025-1	100.2	900.4	1000.6	1.0	0.1

For each dilution step, three 3 mm stainless-steel spheres were introduced into the mixture, and the samples were homogenized following the same procedure applied to the stock solution (Fig. 3).

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.

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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.

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Abbreviations

COP	Certificate of Preparation
Py-GC-MS	Pyrolysis-gas chromatography-mass spectrometry
MP	Microplastic
PVC	Polyvinyl chloride

Units

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