

# Certificate of Preparation - Batch #0003 and Sub-batch #0003(1) (HDPE-CaCO<sub>3</sub> blend)

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 preparations

## 1.1. Polymer Used

Batch #0003 consists of HDPE, provided in the form of microplastic (MP) fragments with a maximum diameter of approximately 50  $\mu$ m, obtained by sieving through a 300-mesh grid (Fig. 1).

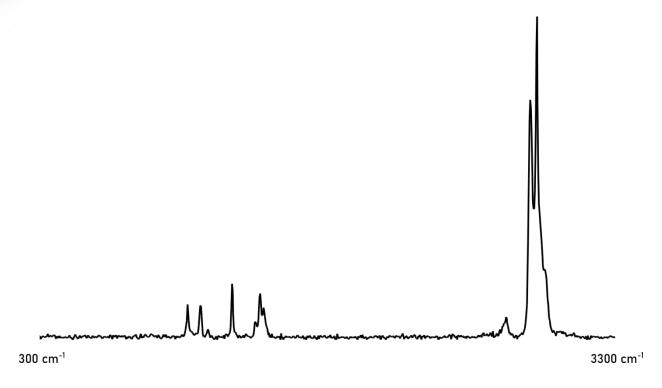
Fig. 1: Example: Photomicrograph captured under critical-angle darkfield illumination (CADLFI) of LDPE fragments sieved through a 300-mesh grid.



The MP fragments are weighed and dispersed in  $CaCO_3$  diluent, ensuring a traceable distribution of polymer mass within the matrix. Each MP fragment weighs less than 10 ng; therefore, 1  $\mu$ g of MP powder typically consists of at least 100 particles (at the lowest available concentration of 1.0  $\mu$ g/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).



Fig. 2: Raman spectrum of the HDPE fragments used in Batch 0003. 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  $300-3300 \, \text{cm}^{-1}$  range using 600 grooves/cm grating with a 100  $\mu$ m split. The spectra acquisition time was set to 3s with 3x accumulation.



#### 1.2. Diluent

Calcium carbonate (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.

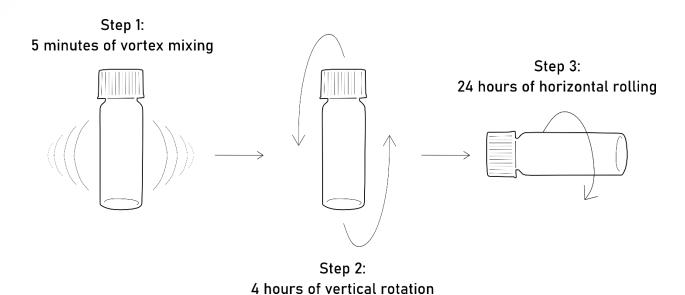
## 1.3. Preparation of polymer-diluent blend

HDPE fragments were dispersed in CaCO<sub>3</sub> 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).

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





## 2. Sample contents

#### 2.1. Stock blend

Stock blend (Batch #0003) with a total mass of 5.0 g was prepared, containing approximately  $50 \mu g/mg$  (5.0% w/w) of HDPE. The specific weighed amounts are provided in Table 1.

Table 1: Polymer concentration of stock mixture used in 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, (%)
HDPE	251.3	4751	5002	50.2	5.0

## 2.2. Batch #0003(1)

Batch #0003(1) was subsampled directly from the homogenized stock mixtures without any dilution, preserving the original concentrations.

#### 3. 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.

### 3.1. 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 <a href="mailto:contact@microplasticsolution.com">contact@microplasticsolution.com</a>. 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
HDPE	High-density polyethylene

## Units

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