Report

Reference Material

BAM-P208

Polypropylene
(powder)

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Summary

This report describes the preparation and the analysis of polypropylene (PP) particles provided as powder and named as reference material BAM-P208.

The following equivalent particle diameter and standard deviations have been determined by laser diffraction:

Particle size distribution	**Equivalent particle diameter**[2] in µm	**Expanded uncertainty**[3] U in µm
Dv10[1]	123	8
Dv50[1]	248	10
Dv90[1]	340	4

[1] Equivalent particle diameter Dv10/Dv50/Dv90 of the measured volume-based particle size distribution, below which 10.3% / 50.3% / 90.3% of the cumulative particle volume is contained. The reported percentile values (10.3%, 50.3%, and 90.3%) follow the instrument manufacturer's definition of Dv10, Dv50, and Dv90.

[2] Mean values determined by laser diffraction under dry dispersion with two different devices using 18 values. For further information see the characterization study for the property of interest (section 3.2).

[3] Estimated expanded uncertainty $U = k \cdot u_c$ with a coverage factor $k = 2$, corresponding to a level of confidence of approximately 95%, calculated according to ISO 33405. The combined uncertainty u_c includes the standard uncertainty due to characterization, the contribution of variation between bottles and the long-term stability contribution.

This report contains physical-chemical information on the reference material as well as on homogeneity and stability investigations of the measurand (property of interest), the equivalent particle diameter, and on the analytical methods to determine the additional properties. The polymer type was determined by attenuated total reflection Fourier-transform infrared (ATR-FTIR) spectroscopy, the shape by scanning electron microscopy (SEM) and the phase morphology by differential scanning calorimetry (DSC). The values for particle size distribution are based on the results from laser diffraction measurements of two different devices with at least three replicate measurements of four randomly chosen units of the reference material BAM-P208.

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List of abbreviations

(If not explained elsewhere)

ATR-FTIR	attenuated total reflection Fourier-transform infrared
SEM	scanning electron microscopy
DSC	differential scanning calorimetry
ANOVA	analysis of variance
BAM	Bundesanstalt für Materialforschung und -prüfung, Berlin, Germany
Dv10	Dv10 value describes the equivalent particle diameter of the measured volume below which 10.3 % of the particles are below
Dv50	Dv50 value describes the equivalent particle diameter of the measured volume below which 50.3 % of the particles are below
Dv90	Dv90 value describes the equivalent particle diameter of the measured volume below which 90.3 % of the particles are below
k	coverage factor for expanded uncertainty U (k = 2)
PP	polypropylene
RM	reference material
rel.	relative
RSD	relative standard deviation
s	standard deviation
s_{bu}	standard uncertainty due to between-bottle variation
s_x	standard deviation of all measurements of a study
Std. Dev.	standard deviation
t_m	time interval between value assignment and the initial stability monitoring point
t_{cert}	period of validity of a certificate issued during that time
U	expanded standard uncertainty of a property value
u_{bu}	standard uncertainty associated with between-unit variability
u'_{bu}	maximum between bottle variation that could be masked by within bottle variation
u_{wu}	standard uncertainty associated with within-unit heterogeneity
u_c	combined standard uncertainty of a property value
u_{char}	standard uncertainty due to characterization
u_{hom}	standard uncertainty due to (in)homogeneity
u_{lts}	standard uncertainty due to long-term (in)stability
x_{RM}	property value of the reference material

$\overline{x}$	mean property value of material within a study
$\bar{x}_{hom}$	mean property value of material within the homogeneity study
y_{char}	value assigned to reference material in characterization study

1. Introduction

Polypropylene (PP) is one of the most abundant polymers used in industry. Especially PP packaging products can be found in the environment spread by accident due to insufficient collection. These plastic products may be degraded by environmental weathering and fragmented into small particles with irregular shapes and is called microplastics, when the particles size is between 1 µm and 5 mm. Smaller microplastics are more relevant to environmental issues and human health, because they can be air driven and partly inhalable.
The idea is to offer a reference material for the validation of methods for sampling, sample preparation and detection of microplastics in the size range of 1-500 µm, which contains irregular shaped particles The reference material can be portioned individually for higher concentration levels.

The evaluation of this reference material (RM) has been carried out based on ISO 17034 [2], ISO 33405 [3] and ISO 33401 [4].

2. Material

Polypropylene (Moplen RP320M, random copolymer) was kindly provided by LyondellBasell Industries Holdings B.V. The granulate was milled in a centrifugal mill (ZM200, Retsch, Haan, Germany) under pre-cooling with liquid nitrogen and a velocity of 18000 rpm. The mill was equipped with a stainless-steel ring sieve (500 µm) and a cyclone. The prepared fragments were sieved in 50 g batches using a sieving machine consisting of a sieve tower with a collecting pan and a 50 and 500 µm sieve (stainless-steal, 20 cm diameter, Retsch). The material retained on the 50 µm sieve was transferred into a 0.5 L brown glass bottle, homogenized in a tumble mixer (30 min), and subsequently aliquoted into 317 brown glass bottles with metal screw caps and silicone/polytetrafluoroethylene (PTFE) seals (1 g each).

3. Characterisation

3.1 Informative parameter for material characterization

The reference material was characterised by imaging the particle shape with scanning electron microscopy (SEM), by attenuated total reflection-fourier-transform infrared (ATR-FTIR) spectroscopy, and by determining the melting temperature with differential scanning calorimetry (DSC) (Table 1).

PP particles were analysed by SEM (Zeiss Supra 40, Oberkochen, Germany) with a Schottky field emitter and equipped with a high-resolution In-lens SE (secondary electrons) detector with an acceleration voltage of 10.0 kV and 5 mm working distance. An image of the PP particles is presented in Fig. 1.

ATR-FTIR spectroscopy was performed on BAM-P208 powder with a Nicolet Nexus 6700 FTIR spectrometer equipped with SmartOrbit Diamond module and DTGS detector. The spectra were recorded from 4000 to 500 cm^{-1} averaging 32 scans with a resolution of 4 cm^{-1}. The obtained data were processed with ATR correction (Diamond crystal) and automated baseline correction with the OMNIC 9 software (Thermo Fischer Scientific, Karlsruhe, Germany). Characteristic peaks were identified by

associated bands and additionally compared with the Hummel Polymer Sample Library from Thermo Scientific™. The results confirm that the material is composed of PP (Fig. 2).

DSC was carried out three times with a DSC 7020 (Seiko, THASS, Germany) from -150 to 200 °C at a constant heating rate of 10 K/min. A melting temperature T_m of 144.2 °C (Fig. 3 and Table 1) proves the PP material [5].

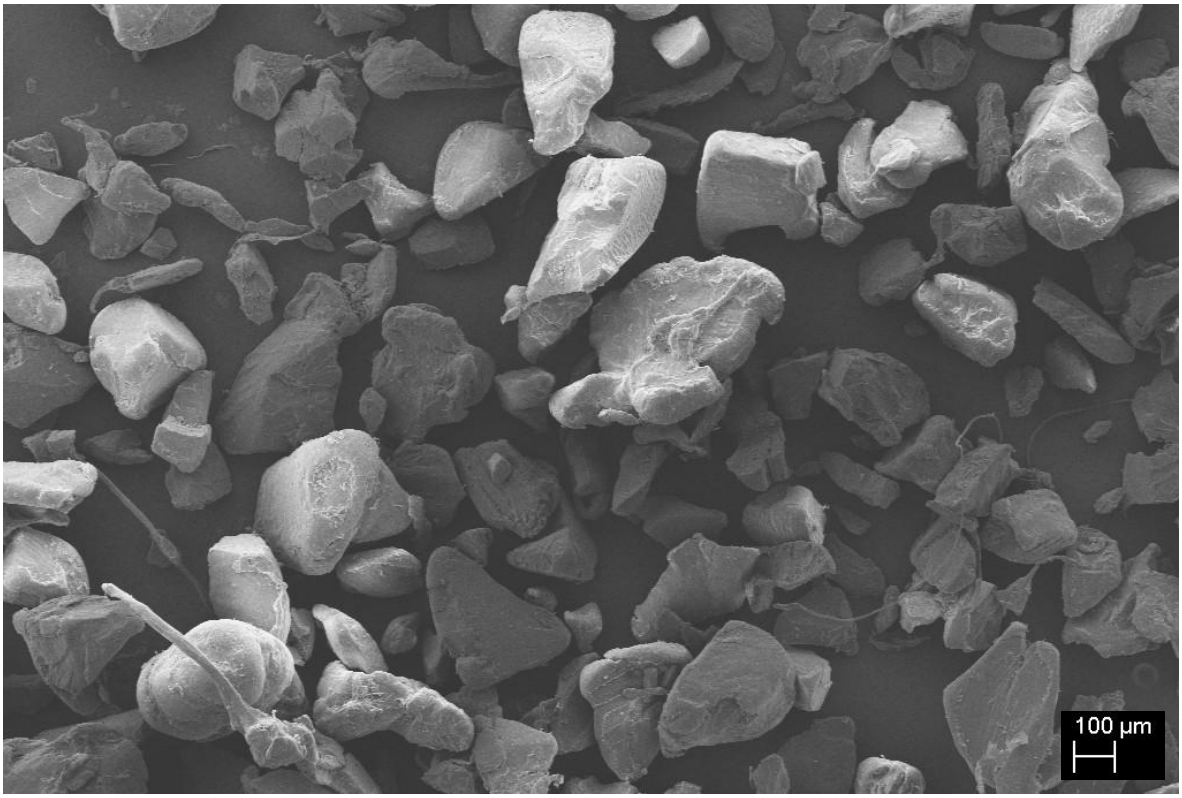

Fig. 1 SEM image of BAM-P208 particles.

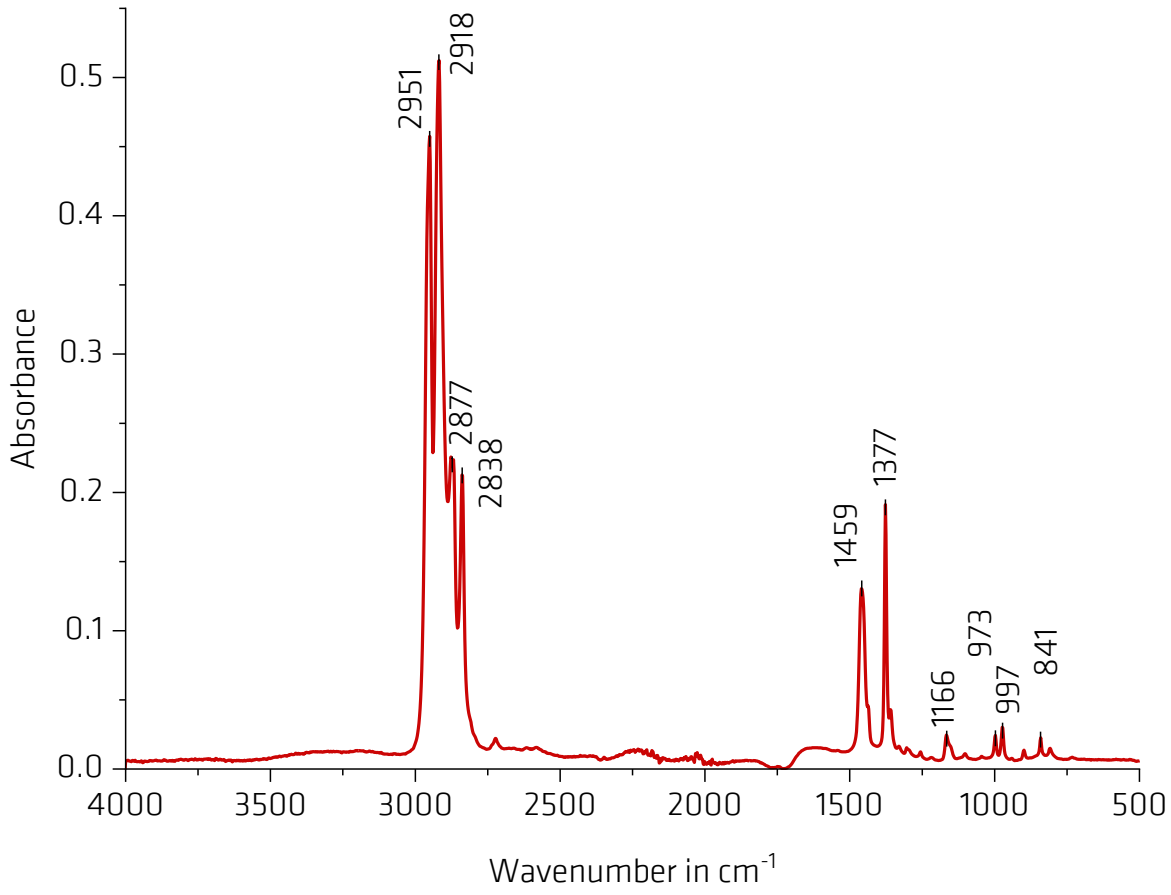

Fig. 2: ATR-FTIR spectrum of BAM-P208.

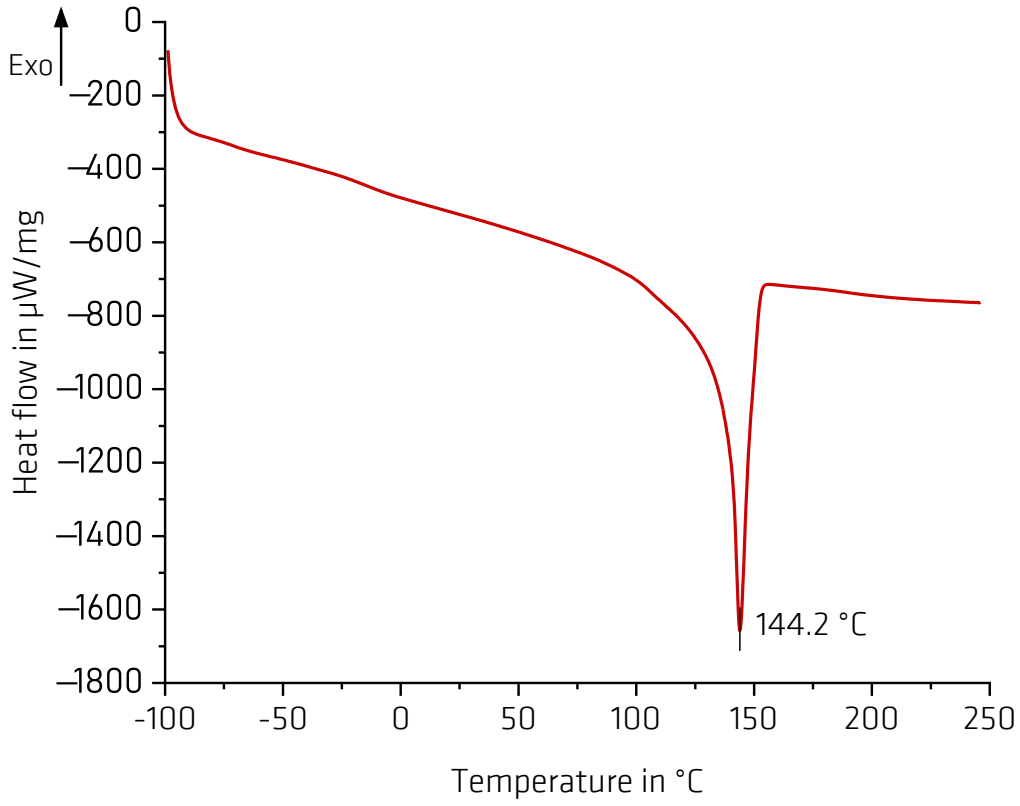

Fig. 3: DSC measurement of BAM-P208.

Table 1 Melting temperature T_m of BAM-P208 from DSC measurements. Mean value of three measurements.

Parameter	**Temperature** in °C	**Standard deviation** s in °C	**RSD** in %
Melting temperature T_m	144.2	0.2	0.2

3.2 Particle size distribution (measurand)

The particle size distribution of BAM-P208 has been characterized by laser diffraction in dry dispersion with two different devices.

1. HELOS/BR + RODOS/L + ASPIROS, (Sympatec, Germany, ISO 13320:2009 certified) at BAM as described in Chapter 3 for the homogeneity study.
2. HELOS/BR + RODOS/L + VIBRI, (Sympatec, Germany, ISO 13320:2009 certified) at BAM as described in Chapter 3 for the homogeneity study. (see Table S1.1)

The measurements in both laboratories were carried out under dry dispersion conditions using 100 mg of material per measurement at a dispersion pressure of 2.5 bar. Data evaluation was performed using the Fraunhofer approximation. Three samples were randomly selected and measured in triplicate using the BAM HELOS/BR+ASPIROS instrument in July 2026. In addition, three different samples were randomly selected and measured in triplicate using the BAM HELOS/BR+VIBRI instrument in May 2026. The two measurement setups were located in independent laboratory areas. Different sample feeding systems were used, and the measurements were performed by different operators. Therefore, the two measurement setups can be regarded as independent.

For each device the average particle size distribution values y_i have been obtained. The values are shown in Table 2.

Table 2 Average particle size distributions and standard deviation of all measurements obtained from laser diffraction in dry dispersion with different devices. The values are given in µm.

Device	BAM 6.5 HELOS/BR+ASPIROS	BAM 7.4 HELOS/BR+VIBRI
Mean value y_i Sample mean values Dv10 n=3	120.5	126.5
Std. Dev. of sample mean values Dv10	0.45	2.04
Mean value y_i of Sample mean values Dv50 n=3	244.1	252.6
Std. Dev. of sample mean values Dv50	1.22	2.53
Mean value y_i of Sample mean values Dv90 n=3	338.7	341.4
Std. Dev. of sample mean values Dv90	0.72	1.26

The assigned value y_{char} for the particle size distribution was calculated from the average values y_i obtained from the measurements of each device and the number of device p = 2 (Equation 1). u_{char} is then calculated according to Equation (2) [3].

$$y_{char} = \frac{\sum y_i}{p} \quad (1)$$

$$u_{char} = \frac{1}{\sqrt{p}} \sqrt{\frac{\sum (y_i - y_{char})^2}{p-1}} \quad (2)$$

Table 3 Assigned values y_{char} and uncertainty u_{char} from the characterization study.

particle size distribution	y_{char} in µm	u_{char} in µm	Rel. u_{char} in %
Dv10	123.5	3.0	2.5
Dv50	248.3	4.3	1.7
Dv90	340.0	1.4	0.4

4. Homogeneity

For testing the homogeneity, 10 individual bottles of BAM-P208 were randomly selected for particle size distribution measurements by laser diffraction with dry powder dispersion (HELOS/BR + RODOS/L + ASPIROS, Sympatec, Deutschland, ISO 13320:2009 certified). Three replicate measurements per bottle were carried out under repeatability conditions with approximately 100 mg each to detect the standard deviation between and within the bottles (see Appendix Table S2).

The results are shown as cumulative and density distribution curves in Figure 4 and are summarized as mean values ($\bar{x}_{hom}$) and corresponding standard deviations in Table 4.

Table 4 Mean values of particle size distribution and respective standard deviations from the homogeneity test of BAM-P208.

Particle size distribution	Equivalent particle diameter (Mean value of sample mean values, n=10) in µm	Standard deviation s of sample mean values, n=10 in µm	Relative standard deviation in %
Dv10	119.44	1.72	1.44
Dv50	242.38	1.41	0.58
Dv90	337.66	0.77	0.23

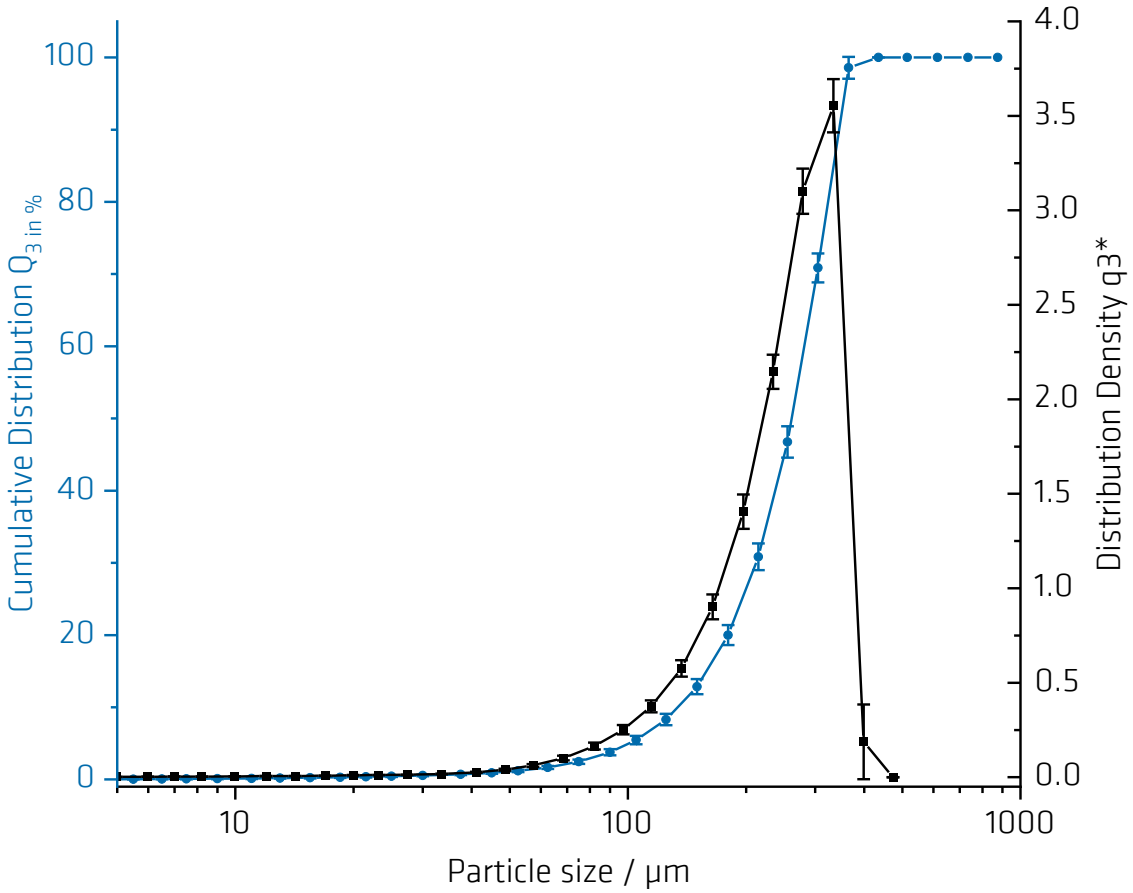

Fig. 4 Equivalent particle diameter with standard deviation of all 30 measurements of BAM-P208 measured by laser diffraction. The blue curve represents the cumulative distribution $Q_3(x)$ and shows the volume fraction of particles smaller than x in relation to the total volume. The black curve represents the distribution density $q_3(x)$ (1^{st} derivate of Q_3) and displays the probability of finding a particle with an equivalent particle diameter x in the population. 10 individual bottles of BAM-P208 were randomly selected and measure three times each. Table S2 shows the corresponding measured values.

The contribution of inhomogeneities to the total measurement uncertainty was calculated using a one way-ANOVA (Table 5 and Appendix Table S4).

Table 5 Analysis of variances calculated for particle size distribution analysis of BAM-P208.

particle size distribution	Source of variation	Mean sum of squares (MS)	Test value (F)	Critical F-value 95%
Dv10	Between Groups	8.922	3.736	2.393
	Within Groups	2.388		
Dv50	Between Groups	5.940	2.270	2.393
	Within Groups	2.617		
Dv90	Between Groups	1.787	2.376	2.393
	Within Groups	0.752		

The ANOVA results can be interpreted as follows. For Dv10, the F-value exceeded the critical value, indicating statistically significant between-unit variability. For Dv50, no significant between-unit variability was detected. The result for Dv90 was very close to the critical value and may be regarded as borderline.

The relatively high F-values observed for Dv10 and Dv90 may be partly attributed to the very good repeatability of the measurement method. As the within-unit variance is small, even minor between-unit differences lead to elevated F-ratios. Therefore, statistical significance does not necessarily imply a practically relevant contribution of inhomogeneity.

The estimated uncertainty associated with homogeneity was found to be within acceptable limits (<2%, see Table 6) and was therefore not considered to pose any significant practical limitation to the suitability of the material as a reference material.

The estimation of inhomogeneity contribution u_{hom} to be included into the total uncertainty budget was estimated from the results of the one-way ANOVA (Appendix Table S4), considering within and between unit variability. The calculations were performed using Equation (3) to (6) according to ISO 33405 [3], with $M_{between}$ mean of squared deviations between bottles, M_{within} mean of squared deviations within one bottle, n number of replicate measurements per bottle and N number of bottles selected for homogeneity study. The results are summarized in Table 6.

The within-unit standard deviation, u_{wu}, was estimated from the ANOVA mean square within units:

$$u_{wu} = \sqrt{M_{within}} \tag{3}$$

The term M_{within}includes both measurement repeatability and any potential variation within a unit. Therefore, u_{wu}represents a conservative estimate and an upper bound of the true within-unit variation.

- The uncertainty contribution due to between-unit variation, u_{bu}, was estimated as the larger of the between-unit standard deviation derived from the ANOVA

$$s_{\text{bu}} = \sqrt{\max(\frac{M_{between} - M_{within}}{n}, 0)} \tag{4}$$

and

- the maximum between-unit variation that could remain undetected due to measurement repeatability and within-unit variation

$$u'_{bu} = \sqrt{\frac{M_{within}}{n}} \sqrt[4]{\frac{2}{N(n-1)}} \tag{5}$$

The combined uncertainty associated with heterogeneity is now given by

$$u_{\text{hom}} = \sqrt{u_{wu}^2 + u_{bu}^2} = \sqrt{u_{wu}^2 + \max(s_{\text{bu}}, u'_{\text{bu}})^2} \tag{6}$$

Table 6 Results to determine the maximum inhomogeneity contribution u_{hom} of BAM-P208.

particle size distribution	Dv10	Dv50	Dv90
$\bar{x}_{hom}$ in µm	119.4	242.4	337.7
$M_{between}$	8.9	6.0	1.8
M_{within}	2.4	2.7	0.8
u_{wu} in µm	1.6	1.7	0.9
s_{bu} in µm	1.5	1.1	0.6
s_{bu} rel. in %	1.3	0.5	0.2
u'_{bu} in µm	0.5	0.6	0.3
u'_{bu} rel. in %	0.5	0.3	0.1
u_{hom}	2.2	2.0	1.1
u_{hom} rel. in %	1.8	0.9	0.4

5. Stability

A stability check of the bottled material was performed. Immediately after bottling selected units were stored at a temperature of 5 °C (the indicated temperature values imply a tolerance of ±3 °C). After a storage period of 0, 1, 3, 6, 12, 24, 36 and 48 months, 3 randomly selected bottles were tested in terms of particle size distribution. 3 measurements (100 mg each) per bottle were carried out (see Appendix Table S3). The results are summarized in Table 7 and presented in Figure 5.

Table 7 Average values of stability test for BAM-P208.

Storage period	Particle size distribution	Equivalent particle diameter in µm	Standard deviation of measurements s_x in µm	RSD in %
0 months	Dv10	119.4	1.8	1.48
	Dv50	242.4	2.0	0.79
	Dv90	337.7	1.1	0.31
1 months	Dv10	120.8	1.4	1.11
	Dv50	243.4	2.3	0.92
	Dv90	337.9	1.5	0.43
3 months	Dv10	118.5	1.2	0.99
	Dv50	241.2	1.1	0.46
	Dv90	337.2	0.8	0.23
6 months	Dv10	119.0	1.0	0.84
	Dv50	240.9	1.2	0.47
	Dv90	336.8	0.8	0.24
12 months	Dv10	120.7	1.1	0.85
	Dv50	245.1	2.0	0.81
	Dv90	339.1	1.2	0.34
24 months	Dv10	121.2	1.4	0.02
	Dv50	221.7	1.9	0.01
	Dv90	338.5	1.3	0.01
	Dv10	122.3	2.7	0.03

36 months	**Dv50**	243.5	2.6	0.01
	Dv90	337.5	1.6	0.01
	Dv10	118.4	5.7	0.01
48 months	**Dv50**	244.4	1.9	0.05
	Dv90	338.5	1.3	0.01

The stability was assessed according to ISO 33405:2024 [3], using linear regression (Fig. 5) followed by testing for statistically significant slopes different from zero. The t-test statistics for slopes were calculated using Equation (7) and compared with the critical values at the 95% level of confidence.

$$t\text{-statistic for slope b significance} \quad t_b = \frac{|b|}{s(b)} \tag{7}$$

Results of linear regression are presented in Figure 5 and values of t-statistics in Table 8. For all particle size distributions, the slopes are not significantly different from zero, and thus, no significant instability is detected.

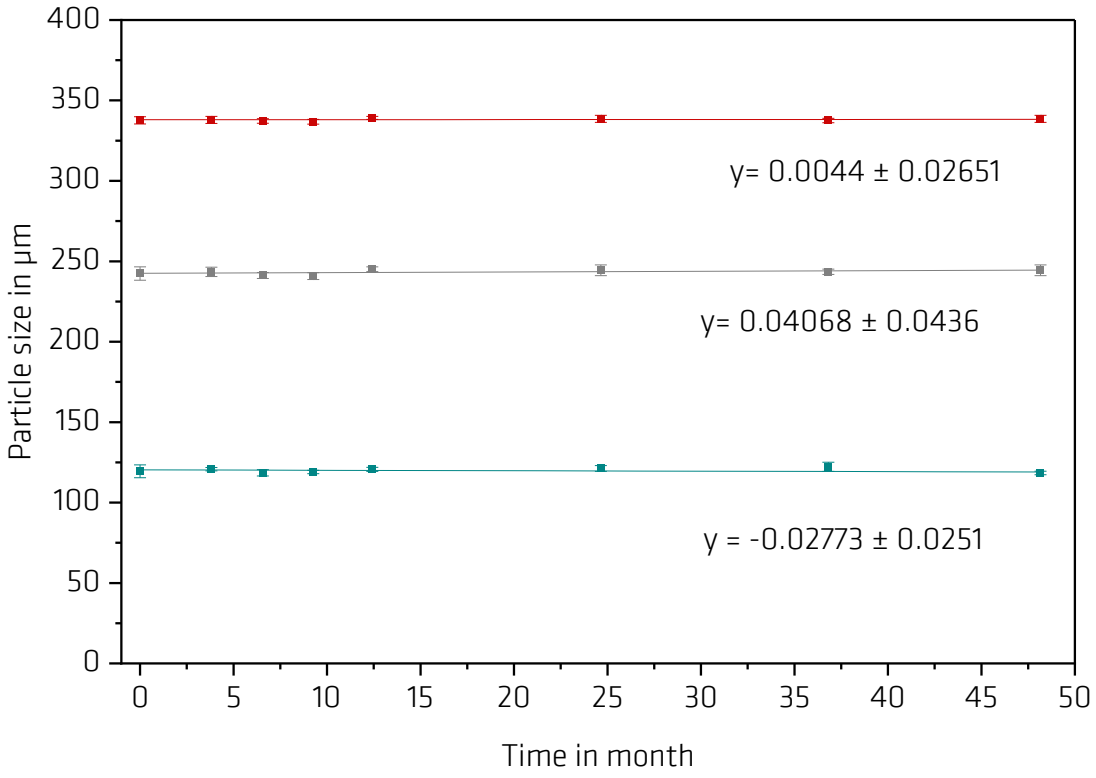

Fig. 5: Stability monitoring of particle size distribution by laser diffraction and linear regression analysis for statistical evaluation of stability of BAM-P208.

Table 8 Testing for significant instability. Values of slope from linear regression of stability data, calculated t-statistic, and critical value at the 95% level of confidence.

particle size distribution	Slope b ln µm/m	s (b) in µm/m	Intercept in µm	t-statistic (b)	Critical value at 95 %
Dv10	0.012	0.033	119.83	0.353	0.092
Dv50	0.046	0.032	242.33	1.458	0.124
Dv90	0.015	0.017	337.64	0.875	0.058

The results of the statistical evaluation of the stability data indicate that there is no instability and that BAM-P208 is stable regarding to particle size distribution over a 48-month period (see Figure 5, and Table 7, 8 and 9). However, the contribution of uncertainty of the long-term stability u_{lts} was calculated according to Equation (8), as in ISO 33405:2024 [3], with *s(b)* as standard error for estimated slope, t_m time interval between value assignment (12 month) and initial stability monitoring point, and t_{cert} period of validity issued (24 month) during that time. Results of uncertainty of the long-term stability are summarized in Table 9.

Contribution of standard uncertainty of long-term stability $u_{lts} = s(b)(t_m + t_{cert})$ (8)

Table 9 Results of stability data evaluation of BAM-P208 according to ISO 33405 [3].

particle size distribution	$\bar{x}_{stab}$ in µm	$s_{x,stab}$ in µm	Intercept in µm	Slope b in µm/m	s (b) in µm/m	u_{lts}(x) in µm	Rel. u_{lts}(x) in %
Dv10	120.27	1.39	119.83	0.012	0.033	1.18	0.98
Dv50	242.97	1.55	242.33	0.046	0.032	1.15	0.47
Dv90	337.82	0.77	337.64	0.015	0.017	0.63	0.18

6. Calculation of combined uncertainty

The combined uncertainty u_c was calculated according to Equation (9), using the numerical values summarized in Table 10. This equation is a combination of the standard uncertainty due to characterization, the contribution of the variation between the bottles and the long-term stability contribution. Furthermore, the reference value x_{RM} can be assigned as y_{char} since no between-unit variation or stability effects need to be regarded (Equation 10).

Combined uncertainty $u_c^2 = u_{\mathrm{char}}^2 + u_{\mathrm{hom}}^2 + u_{\mathrm{lts}}^2$ (9)

Certified value for RM $x_{RM} = y_{char}$ (10)

Table 10 Values of the uncertainty components for the particle size distribution of BAM-P208.

particle size distribution	x_{RM} in µm	u_{char} in µm	u_{hom} in µm	u_{lts} in µm	u_{c} in µm	U in µm
Dv10	123.48	3.00	2.14	1.18	3.87	7.74
Dv50	248.33	4.22	1.93	1.15	4.78	9.56
Dv90	340.03	1.35	1.05	0.63	1.82	3.64

The final attested values of the particle size distribution with a reasonable number of digits (rounded according to DIN 1333 [6]) and the respective expanded uncertainties U are summarized in Table 11.

Expanded uncertainties $U = k \cdot u_c$ with $k = 2$ (11)

Table 11 Final attested values for particle size distribution of BAM-P208.

particle size distribution	Equivalent particle diameter in µm	Expanded uncertainty U in µm
Dv10	123	8
Dv50	248	10
Dv90	340	4

7. Information on the proper use of the reference material

7.1 Recommended use

The present material is intended for the validation of sampling, sample preparation and detection of microplastics.

7.2 Transport, storage, and handling

Reference Material BAM-P208 can be shipped at ambient temperature. It should be stored under dark and dry conditions at a temperature of 5 ± 3 °C in its original tightly closed bottle. BAM cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened bottles. In case of further transport, the packaging should be free of plastic to avoid contamination.

7.3 Shelf life

The stability study after storage for 48 months of selected bottles at temperatures of 5 ± 3 °C did not reveal any statistically significant deterioration of the particle size distribution. However, starting with dispatch of the material from BAM the validity of the data sheet expires after 24 months. Post-certification measurements will be conducted in appropriate periods to keep this information up to date.

7.4 Safety information

The usual laboratory safety precautions must be applied. No hazardous effects are to be expected when the material is used under conditions commonly adopted for the analysis of environmental samples.

8. Information on purchase of the reference material

Reference material BAM-P208 is supplied by:

Bundesanstalt für Materialforschung und -prüfung (BAM)
Richard-Willstätter-Str. 11, D-12489 Berlin, Germany

Phone: +49 (0)30 - 8104 2061
Fax: +49 (0)30 - 8104 72061 https://www.bam.de
E-Mail: sales.crm@bam.de www.webshop.bam.de

Each bottle of BAM-P208 will be distributed together with a data sheet containing the mean values, standard deviations and uncertainties of all data sets and information on the analytical methods used.

9. References

[1] ISO/TR 21960, Plastics Environmental aspects - State of knowledge and methodologies. International Organization for Standardization, Geneva 2020.

[2] DIN EN ISO 17034, General requirements for the competence of reference material producers. International Organization for Standardization, Geneva 2016.

[3] ISO 33405:2024, Reference materials – Approaches for characterization and assessment of homogeneity and stability.
International Organization for Standardization, Geneva 2024.

[4] ISO 33401:2024, Reference materials - Contents of certificates, labels and accompanying documentation.
International Organization for Standardization, Geneva 2015.

[5] Li, Dawei & Zhou, Liwei & Wang, Xuan & He, Lijuan & Yang, Xiong. (2019). Effect of Crystallinity of Polyethylene with Different Densities on Breakdown Strength and Conductance Property. Materials. 12. 1746. 10.3390/ma12111746.

[6] DIN 1333, Zahlenangaben. DIN, Berlin 1992.

10. Appendix

Table S1.1 Results of characterization study in 3.2 four randomly selected samples with three replicate measurements (Rep. Meas.) from a single bottle measured with HELOS/BR+VIBRI at BAM.

Bottle ID	Rep. Meas.	Dv10 in µm	Dv10 average per bottle in µm, Std. Dev. in µm RSD in %	Dv50 in µm	Dv50 average per bottle in µm, Std. Dev. in µm RSD in %	Dv90 in µm	Dv90 average per bottle in µm, Std. Dev. in µm RSD in %
234	1	123.76	124.38	249.62	251.57	341.22	342.04
	2	124.23	0.71	252.61	1.69	342.59	0.72
	3	125.15	0.57	252.49	0.67	342.30	0.21
165	1	127.53	126.60	250.38	249.52	341.01	340.62
	2	125.69	0.92	246.22	2.97	338.75	1.71
	3	126.59	0.73	251.96	1.19	342.11	0.50
36	1	128.52	128.45	252.81	254.50	342.01	342.84
	2	127.96	0.46	255.63	1.49	343.36	0.72
	3	128.87	0.36	255.05	0.59	343.14	0.21

Table S2 Results of homogeneity test of 10 randomly selected samples with three replicate measurements (Rep. Meas.) from a single bottle.

Bottle ID	Rep. Meas.	Dv10 in µm	Dv10 average per bottle in µm, Std. Dev. in µm RSD in %	Dv50 in µm	Dv50 average per bottle in µm, Std. Dev. in µm RSD in %	Dv90 in µm	Dv90 average per bottle in µm, Std. Dev. in µm RSD in %
326	1	121.91	121.61	245.16	244.61	339.06	338.80
	2	122.42	1.00	246.47	2.19	339.77	1.12
	3	120.49	0.82	242.20	0.90	337.58	0.33
148	1	119.25	116.29	242.51	240.98	338.02	337.32
	2	114.57	2.58	239.90	1.36	336.86	0.61
	3	115.04	2.22	240.53	0.56	337.08	0.18
174	1	119.09	119.31	242.19	241.06	337.55	336.67
	2	119.42	0.19	241.23	1.23	336.67	0.88
	3	119.43	0.16	239.75	0.51	335.79	0.26
364	1	120.38	122.07	242.66	243.49	337.61	337.86
	2	122.23	1.62	243.48	0.83	337.75	0.32
	3	123.60	1.32	244.33	0.3	338.21	0.09
136	1	118.90	119.13	241.96	241.37	337.15	336.98
	2	119.00	0.32	241.64	0.77	337.37	0.50
	3	119.49	0.27	240.50	0.32	336.42	0.15
256	1	120.38	120.40	242.08	242.20	337.19	337.28
	2	120.69	0.28	244.26	2.01	338.65	1.33
	3	120.14	0.23	240.25	0.83	336.00	0.39
142	1	118.04	119.41	239.87	242.25	336.56	337.63
	2	118.95	1.65	241.83	2.61	337.38	1.22
	3	121.24	1.38	245.04	1.08	338.95	0.36
200	1	117.97	118.60	240.77	241.37	337.00	337.25
	2	118.62	0.62	241.14	0.74	337.13	0.33
	3	119.21	0.52	242.20	0.31	337.62	0.10
371	1	121.18	119.94	247.15	244.71	340.45	339.12
	2	120.38	1.51	243.71	2.12	338.21	1.17
	3	118.26	1.26	243.28	0.87	338.71	0.35
95	1	119.31	117.64	242.69	241.78	337.98	337.72
	2	119.24	2.83	241.82	0.93	337.67	0.24
	3	114.37	2.41	240.82	0.39	337.50	0.07

Table S3 Results of the stability test after storage times of 4, 6, 9, 12, 24, 36 and 48 months with three replicate measurements (Rep. Meas.) from a single bottle.

Storage Time	Bottle ID	Rep. Meas.	Dv10 in µm	Dv10 average per bottle in µm, Std. Dev. in µm Rel. error in %	Dv50 in µm	Dv50 average per bottle in µm, Std. Dev. in µm Rel. error in %	Dv90 in µm	Dv90 average per bottle in µm, Std. Dev. in µm Rel. error in %
4	225	1	121.99	122.21	244.80	245.42	338.59	339.08
		2	123.31	1.02	246.43	0.88	339.72	0.58
		3	121.32	0.83	245.03	0.36	338.94	0.17
4	119	1	120.34	120.70	242.86	243.27	337.49	337.66
		2	120.91	0.31	244.10	0.72	338.13	0.40
		3	120.84	0.26	242.85	0.29	337.37	0.12
4	75	1	119.56	119.37	244.47	241.55	339.27	336.95
		2	119.23	0.17	239.22	2.67	335.30	2.07
		3	119.33	0.14	240.97	1.11	336.30	0.61
6	214	1	119.49	119.24	241.27	241.17	337.19	337.13
		2	119.20	0.23	240.81	0.32	336.77	0.33
		3	119.03	0.19	241.43	0.13	337.43	0.10
6	118	1	116.76	117.77	240.82	240.47	337.61	336.99
		2	118.07	0.90	240.25	0.30	336.67	0.54
		3	118.50	0.77	240.35	0.13	336.68	0.16
6	354	1	119.23	118.58	243.11	241.82	338.59	337.53
		2	119.89	1.72	239.74	1.82	336.10	1.29
		3	116.63	1.45	242.62	0.75	337.89	0.38
9	116	1	118.68	118.40	241.31	241.03	337.19	337.07
		2	118.67	0.48	242.13	1.26	337.89	0.88
		3	117.84	0.41	239.66	0.52	336.14	0.26
9	304	1	119.86	120.11	241.95	241.12	337.34	336.78
		2	119.97	0.36	239.15	1.71	335.47	1.14
		3	120.52	0.30	242.26	0.71	337.54	0.34
9	248	1	117.89	118.47	240.63	240.39	336.62	336.50
		2	118.06	0.86	240.45	0.27	336.72	0.30
		3	119.46	0.73	240.10	0.11	336.15	0.09

Table S3 continued.

Storage Time	Bottle ID	Rep. Meas.	Dv10 in µm	Dv10 average per bottle in µm, Std. Dev. in µm Rel. error in %	Dv50 in µm	Dv50 average per bottle in µm, Std. Dev. in µm Rel. error in %	Dv90 in µm	Dv90 average per bottle in µm, Std. Dev. in µm Rel. error in %
12	369	1	118.67	120.06	245.28	245.98	339.64	339.81
		2	120.72	1.20	246.62	0.67	339.94	0.15
		3	120.78	1.00	246.04	0.27	339.86	0.05
12	356	1	121.52	121.75	247.28	246.56	340.14	339.77
		2	121.92	0.21	246.11	0.63	339.49	0.34
		3	121.81	0.17	246.28	0.26	339.67	0.10
12	194	1	120.40	120.25	243.18	242.65	338.10	337.75
		2	119.95	0.26	241.29	1.19	336.78	0.85
		3	120.41	0.22	243.48	0.49	338.37	0.25
24	259	1	118.90	119.90	243.70	245.43	338.49	339.41
		2	119.14	1.53	247.06	1.69	340.42	0.97
		3	121.66	1.27	245.53	0.69	339.33	0.28
24	215	1	122.21	121.85	244.71	244.21	338.58	338.33
		2	121.30	0.48	244.78	0.93	338.70	0.53
		3	122.02	0.39	243.14	0.38	337.73	0.16
24	207	1	122.53	121.91	246.44	243.59	339.92	337.87
		2	120.94	0.85	241.09	2.69	336.18	1.90
		4	122.26	0.70	243.22	1.11	337.50	0.56
36	330	1	124.87	125.18	246.63	245.96	339.32	338.82
		2	125.00	0.42	245.42	0.61	338.56	0.43
		3	125.65	0.33	245.84	0.25	338.57	0.13
36	150	1	120.03	121.25	243.42	244.07	337.60	338.02
		2	123.81	2.21	244.38	0.57	338.04	0.41
		3	119.92	1.82	244.43	0.23	338.43	0.12
36	68	1	118.35	120.34	241.79	240.54	336.82	335.70
		2	121.67	1.75	240.82	1.42	335.84	1.19
		3	120.99	1.46	239.00	0.59	334.45	0.35
48	241	1	110.65	110.97	236.75	245.43	335.52	339.41
		2	110.61	0.60	236.77	1.69	335.67	0.97
		3	111.67	0.55	236.26	0.69	334.41	0.28
48	380	1	122.16	122.37	247.59	244.21	340.10	338.33
		2	122.54	0.19	247.53	0.93	340.20	0.53
		3	122.41	0.16	244.51	0.38	338.30	0.16
48	61	1	124.31	121.91	246.72	243.59	339.53	337.87
		2	124.87	0.85	244.42	2.69	337.73	1.90
		3	126.25	0.70	245.61	1.11	337.86	0.56

Table S4 ANOVA results of homogeneity testing of 10 randomly selected bottles and their replicate measurements within the bottles.

	Source of Variation	Square sum (SS)	Degree of freedom (df)	Mean square sum (MS)	Test value (F)	p-Value	Critical F-Value
Dv10	Between groups	80.299	9	8.922	3.736	6.758E-03	2.393
	Within groups	47.766	20	2.388			
	Total	128.065	29				
Dv50	Between groups	53.459	9	5.940	2.270	6.088E-02	2.393
	Within groups	52.336	20	2.617			
	Total	105.794	29				
Dv90	Between groups	16.084	9	1.787	2.376	5.140E-02	2.393
	Within groups	15.046	20	0.752			
	Total	31.130	29				