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Lactose Tablets spiked with PET as suitable Microplastics reference material to support validation studies and quality control

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ABSTRACT

Method validation in the field of microplastics analysis is challenging due to a lack of suitable reference materials describing the property of interest. The EU-funded PlasticTrace project aims to overcome that gap by developing water-soluble tablets containing defined numbers and masses of polyethylene terephthalate (PET) particles. Tablets were produced by solid-phase dilution following a strict preparation protocol with additional quality control. Different batches containing different particle numbers and masses were prepared and tested down to the detection limits of the analytical methods: theoretical mass (determined number by μ-Raman): 0.2 μg (151), 18.5 μg (1764) and 400 μg (not measured). PET particles had a mean size of approximately 40 μm, ranging from 1 to 100 μm, and an irregular shape determined by laser diffraction and scanning electron microscopy. All measurements were performed in accordance with ISO 16094–2 and ISO/FDIS 16,094–3. The most accepted microplastics detection methods were applied for characterisation: μ-FTIR, μ-Raman, Py-GC/MS and TED-GC/MS. Homogeneity and stability tests were assessed in terms of mass by TGA. The PET particles in the tablets show homogeneity with a relative standard deviation of <20%. No significant changes were observed during 6 months of storage. The developed tablet production method was also applied for polyethylene microplastics to assess its transferability to another polymer type. These reference materials can help to fulfill the criteria of EU Commission for MP monitoring in drinking water and wastewater.

1. Introduction

The increased awareness of the potential environmental and human health risks associated with microplastic (MP) particles (1–1000 μm, ISO/TR 21960:2020) (ISO/TR, 2020), combined with their ubiquitous occurrence globally, has prompted the European Union (EU) to adopt the precautionary principle and initiate regulatory actions. In recent years, several EU directives, such as the Drinking Water Directive (Directive (EU) 2020/2184) (Directive, 2020) and the Urban Wastewater Treatment Directive (Directive (EU) 2024/3019) (Directive,

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2024), have introduced requirements for the monitoring of MP in water systems. Such monitoring is essential to improve the understanding of MP occurrence and transport pathways and to provide scientific basis for developing targeted measures to reduce MP contamination in the wider environment.

Accurate and comparable MP measurements require validated, harmonised and standardised analytical techniques, including sampling, sample preparation and detection. The International Organization for Standardization (ISO) ISO/TC 147/SC 2/JWG 1 recently published ISO 16094-2:2025 (ISO, 2025) for the number-based identification and quantification of MP in water using vibrational spectroscopy methods, and ISO/FDIS 16094-3 (ISO/FDIS, 2026) for mass-based detection and quantification of MP in water using thermo-analytical methods. Number-based approaches commonly include Fourier–transform infrared microscopy (μ-FTIR) and Raman microscopy(μ-Raman), whereas mass-based approaches include pyrolysis-gas chromatography-mass spectrometry (Py-GC/MS) and thermal extraction desorption-GC-MS (TED-GC/MS) (ISO/FDIS, 2024; Huang et al., 2024; Ciornii et al., 2025). The implementation and harmonisation of these analytical approaches, both number- and mass-based, require appropriate reference materials to support the assessment of their performance. A reference material is a material sufficiently homogenous and stable with respect to one or more specified properties, which has been established to be fit for its intended use in a measurement process (e.g., calibration of a measurement system, assessment of a measurement procedure or quality control) (ISO, 2026). The terminology and definitions, production, and approaches for the characterization of reference materials are addressed respectively by ISO 33400:2026 (ISO, 2026), ISO 17034:2016 (ISO, 2016) and ISO 33405:2024 (ISO, 2024). Since MPs occur in a wide range of matrices and exhibit different particle characteristics, reference material samples intended to support MP monitoring must closely reflect the characteristics of the real MP particles found in the target matrices. Particle sizes are particularly relevant because smaller particles (<50 μm) are generally more challenging to identify and quantify, especially in complex matrices, while their environmental and biological relevance has raised increasing concern (Xu et al., 2024; Corami et al., 2022). MP reference material samples should ideally include irregularly shaped fragments with sharp edges and cover size ranges relevant to the target application. Because environmental MPs originate often from the weathering and fragmentation of plastic products, they commonly exhibit oxidized surface functionalities and altered surface properties (Baby et al., 2025). Depending on the analytical approach used for MP detection, this aging process may affect the accuracy of both particle number- and mass-based measurements. However, the impact on mass quantification is generally less pronounced, as the oxidized surface layer represents only a small fraction of the total particle volume for micrometer-sized particles, whereas surface-related alterations play a much greater role in microscopic analyses. MP reference material samples should also cover MP concentration levels relevant to the target matrices and analytical applications. As MP concentrations can vary substantially between matrices with relatively high concentrations found in environmental samples (e.g., wastewater) (Altmann et al., 2023) and lower concentrations in food products or biota (e.g., milk products) (Hayder et al., 2026), MP reference material samples at different concentration levels may be required to address these different applications.

Some MP reference material samples are currently available. Most of them, including BAM particles such as BAM-P201 (aged polyethylene, PE), BAM-P202 (polystyrene, PS), BAM-P206 (polyethylene terephthalate, PET), BAM-P208 (polypropylene, PP) or BAM-P210 (low-density polyethylene, LD-PE) (BAM-webshop, 2026), are provided as fragmented particles that can be individually weighed according to the require amount. This format is suitable when high polymer masses are required. Other formats are also available, including MP particles incorporated into a salt-based matrix, such as EURM-060 recently made available by the European Commission's Joint Research Centre (JRC) (Centre, 2025), as well as soda tablets and capsules designed for the detection of common polymers (Microplastic Reference Materials | Buy Microplastic Testing Supplies - ZeptoMetrix, 2026; Martínez-Francés et al., 2023). However, the particle size range of many of these materials is generally between 50 and 300 μm, resulting in limited coverage of the smaller size fractions that are of increasing interest for health and environmental studies. MP reference material samples containing low polymer masses are also available, but their availability remains limited in terms of polymer types and particle size ranges. Such materials should contain total polymer masses in the μg range, which can be challenging to achieve and accurately weigh even when using analytical balances. Furthermore, these low masses must be quantitively transferred during handling and transport to ensure that all end users receive comparable amount of the MP reference material. These challenges can be addressed by using a tablet-based format, in which MP particles, with different shapes and polymer type, are embedded within a solid matrix. For most polymer types, MP particles below 100 μm can be readily produced by cryogenic milling of plastic granulate feedstock, followed, where necessary, by additional fragmentation and subsequent sieving to obtain the desired particle size range for the planned reference materials (Abdolahpur Monikh et al., 2023; Eitzen et al., 2019; Sørensen et al., 2024; Oster et al., 2024).

The aim of this study was to produce and characterize MP reference materials in the tablet-based format to support method performance assessment, quality control and laboratory-to-laboratory harmonisation, contributing to the standardisation of MP measurements.

Pressed tablets containing PET particles with a size range of 1–100 μm, irregular morphology and sharp edges were developed. PET was selected as the main polymer because of its relevance to food-contact applications, particularly beverages packaged in PET, and to the monitoring requirements introduced by the Drinking Water Directive (Directive, 2020). The potential exposure to PET-derived MP through food and beverages, together with the still limited knowledge of their potential toxicological effects, further highlights the relevance of PET for MP studies. LDPE particles were also investigated because of its widespread use in packaging and its frequent occurrence in environmental samples. The overall mass incorporated into the tablets was varied to obtain three different concentrations levels targeting different analytical applications: (I) an environmental-like concentration for mass-based thermo-analytical methods (Batch 1); (II) a food-related concentration intended to be detectable for both mass- and number-based analysis methods (Batch 2); and (III) a very low mass approaching the limit of reporting for number-based spectroscopic methods (Batch 3) (ISO, 2025). The design of the reference material candidate was guided by their intended analytical applications and the specific characteristics required for their use, including polymer type, particle size, particle number and polymer mass, following a fit-for-purpose approach consistent with the principles of Quality-by-Design (QbD), which emphasize defining the intended use and the critical characteristics required to achieve it (Altmann et al., 2025).

PET particles were characterized in terms of particle size and shape using laser diffraction (LD) and scanning electron microscopy (SEM), while polymer identity was verified by FTIR.

The homogeneity and stability controls according to mass were assessed by thermogravimetric analysis (TGA), using at least 10 individual tablets, according to ISO 33400:2026 (ISO, 2026) and ISO 33405:2024 (ISO, 2024). Complementary characterization was performed using number-based approaches (μ-FTIR and μ-Raman spectroscopy) and other mass-based instruments (Py-GC/MS and TED-GC/MS). The lowest PET concentration (B3) was characterised only by μ-FTIR and μ-Raman spectroscopy since the mass is too low for accurate measurements.

The overall experiment workflow is presented in Fig. 1.

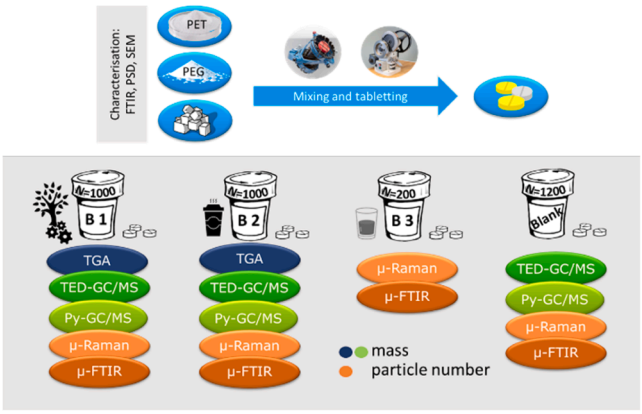


Fig. 1. Overall experimental summary of the number of tablets per batch with varying concentrations and characterization methods used for the different batches (**Batch** 1 – related to environmental MP concentration, **Batch** 2 – related to food MP concentration, **Batch** 3 – related to drinking water MP concentration) of tablets containing the different PET MP concentrations and for Blanks containing the tablet matrix only.

2. Experimental section

2.1. Materials

Directly compressible lactose (FlowLac90, D50: 138.2 ± 3.1 µm) was provided by MEGGLE GmbH & Co. KG (Germany) and used as the tablet matrix. Polyethylene glycol 6000 (PEG, D50: 98.5 ± 2.2 µm) was purchased at Merck. PET powder was provided by BAM with an irregular particle shape and a broad size distribution (from 1 to 100 µm, D50: 42.4 ± 0.2 µm). The starting material for the PET was bottle-grade PET granulate, kindly provided by PlasticsEurope. Low-density polyethylene (LDPE), most probably produced by polymer emulsion, was kindly provided by Schaetti AG, Wallisellen, Switzerland (D50: 18.0 ± 0.7 µm). The particle size distribution measurements were determined by laser diffraction in dry dispersion (Instrumental equipment: HELOS/BR+RHODOS/L+ASPIROS, Sympatec, Clausthal-Zellerfeld, Germany) with a mass of 100 mg.

Different filters are used for sample preparation of the tablets. Silicon (Si) filters used are produced by chemical etching (Smart membranes, Germany) with 5 µm round pores (Fig. 2**, left**). The pitch of the filters is 12 µm, the depth 270 µm and the roughness of the top surface of the filter is about 10 nm. The filters are cut by laser dicing to the desired diameter of 9 mm. To enable more efficient filtration and analysis by the thermo-analytical instruments, dedicated filter-crucibles for TGA and TED-GC/MS measurements were designed (Fig. 2**, right**). The filter-crucible (GKD - Gebr. Kufferath AG, Düren, Germany, https://www.gkd-group.com/de-de/) has a height of 13 mm and a diameter of 9 mm, where the tablet fit in ideally. The filter material is welded at the bottom to prevent particle loss during filtration (Braun et al., 2021).

2.2. Tableting PET and LDPE MP test materials

To circumvent the difficulties associated with the weighing of very small masses and their quantitative transportation, the MP particles were embedded in lactose and a lubricant (PEG). Tablets were pressed with a conventional tablet press used for producing pharmaceutical tablets. Lactose is commonly used in pharmaceutical applications as a cost-effective matrix for ingredients or drugs. It is easy to handle in the laboratory, can be readily produced under clean conditions, and can be directly pressed into tablets. This helps minimize contamination from transport packaging in the final product, which is often made of plastic. Furthermore, the resulting tablets are both water-soluble and non-toxic, making them easy to use in the laboratory. To produce the tablets, the water-soluble lactose, PET particle test material and PEG were mixed together in equal amounts. The PEG is added to act as a lubricant during the tablet pressing process. The first batch of tablets (Batch 1) started with 1.05 g of each of the three ingredients being mixed together with a tumble mixer (LM-TM100, Laarmann, Roermond, Netherlands) for 10 mins (Table 1). After this time, an additional 10.5 g of lactose and PEG were added to the sample to make the first dilution step and a further 10 min of mixing applied. Subsequent dilutions of the PET with additional lactose and PEG are outlined in Table 1, with mixing applied at each dilution step. Once the final dilution and mixing step (20 min) had been completed, resulting in a PET concentration of 2.2 mg/g, small aliquots of the mixed powder were pressed into tablets using a TDP 5 Desktop Tablet Press (LFA Tablet Press, Bicester, United Kingdom). The stainless-steel punch had a diameter of 8 mm. Each tablet has a mass of

Table 1
Sample preparation procedure for PET Batch 1 tablets (Batch 2 and 3 in brackets).

	Tablet ingredients				
	PET (g)	Lactose (g)	PEG (g)	PET Concentration (mg/g of matrix)	Mixing time (min)
Batch 1 (Batch 2/3)	1.05 (0.03/0.01)	1.05	1.05	333 (14.1/4.7)	10
1st dilution step	-	10.5	10.5	43.5 (1.3/0.4)	10
2nd dilution step	-	19.2	19.2	16.8 (0.48/0.16)	10
3rd dilution step	-	38.5	-	10.4 (0.3/0.1)	10
4th dilution step	-	115.5	-	4.8 (0.14/0.05)	10
5th dilution step	-	255.1	-	2.2 (0.06/0.02)	20

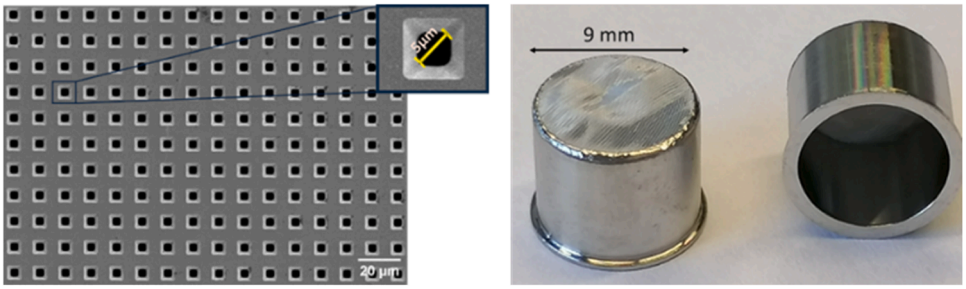


Fig. 2. Top view of the 5 µm pore size Si filters. The inset shows a selected pore (left); Smart filter crucible for TGA and TED-GC/MS measurements with metal fabric with 5–6 µm size (right).

250 ± 5 mg. An additional two batches of tablets (Batch 2 and Batch 3) were produced using the same dilution and mixing procedure as described for Batch 1, but with starting amounts of PET test material of 0.03 and 0.01 g, respectively. Finally, a batch of blank tablets was produced using the same method, but without the addition of PET particles.

Owing to the different starting amounts of PET test material added to the mixture in each of the batches, the resulting tablets contained different particle numbers and masses. This provided a range of MP reference materials that were suitable for assessing the detection limits of the individual thermo-analytical and spectroscopic analysis techniques. The different batches contain various theoretical masses of PET per tablet as follows:

- Batch 1: 400 µg
- Batch 2: 18.5 µg
- Batch 3: 0.2 µg
- Blanks: 0 µg (matrix only)

A total of 1000 tablets were pressed from each of the Batch 1 and Batch 2 mixtures, while 200 tablets were prepared from the Batch 3 mixture and the blank matrix mixture.

LDPE tablets were produced by the same principle as the tablets containing PET with a concentration of Batch 2.

2.3. Characterisation

To produce useful reference materials in the field of MP, a range of parameters must be tested and verified for the reference material to be considered suitable for the validation of the thermo-analytical and spectroscopic detection methods (Table 2). These parameters were used for characterisation of the PET particles and define the analytical detection methods. A detailed description of the methods is presented in the Supplementary Information.

2.4. Processing of tablets for PET analysis

Sample preparation involves dissolution and filtration of the tablet to isolate the PET, where the filtration is carried out according to a jointly developed standard operating procedure (Putzu et al., 2026). The ultrapure water required for dissolution is provided in-house at each user laboratory but should always be filtered through a 200 nm filter prior to use. The Si filters are reusable and can be cleaned by washing with ethanol and acetone in an ultrasonic bath. Each filtration unit was validated in-house prior to use with the PET RM tablets.

2.5. Homogeneity and stability control studies

Owing to the labor-intensive filtration procedures involved in sample preparation, which are necessary to minimize particle loss and prevent potential contamination, we have assessed the homogeneity of the PET spiked tablets by a total of 10 tablets (minimum requirement of units according to ISO 33405) (ISO, 2024) from each of Batch 1 and Batch 2 that were filtered over a 5 µm pore sized filter crucible (stainless steel, 9 mm in diameter) and analysed by TGA to determine the mass of PET polymer in each individual tablet. A small mass loss between 25 and 200 °C typically indicates the loss of any water present in the sample, while biological compounds decompose at higher temperatures (200–400 °C). The mass loss of polymers typically occurs between 350 and 500 °C. Although individual polymer types can give different decomposition temperatures, TGA and similar methods are not feasible for identifying different polymer types within one sample, nor for distinguishing their quantitative mass loss as the decomposition temperature areas can interfere with each other. TGA only works effectively for simple systems/mixtures where all compounds or components are known, and the individual decomposition temperatures are sufficiently separated from each other. Stability control was performed on Batch 1 and Batch 2 tablets after 6 and 12 months, respectively.

Table 2
Parameters for the fit-for-purpose design of a reference material for validation of detection methods.

	Measurand	Methods
Homogeneity and stability control (monitored parameter in µg/tablet)	Mass fraction of PET in lactose tablets	TGA
Concentration values (in number/tablet or µg/tablet)	Particle number of PET in lactose tablets Mass fraction of PET in lactose tablets	µ-FTIR, µ-Raman Py-GC/MS, TED-GC/MS
Identification and dimensional characterization	Size and size distribution Shape Polymer type	Laser diffraction SEM FTIR

2.6. Quality assurance/quality control

First quality control occurred by measuring gravimetrically the mass of at least 20% of the tablets from each batch using a Mettler Toledo AT DeltaRange® analytical balance. Tablets with a mass outside the acceptable tolerance range of 250 ± 5 mg were discarded. This quality control step assessed the total tablet mass and was not intended to assess PET homogeneity between the tablets, which was evaluated separately by TGA.

All labs followed the ISO 16094-2:2025 and ISO/FDIS 16094-3 norms (ISO, 2025; ISO/FDIS, 2026) regarding quality assurance and quality control, including the management of reagents, taking relevant precautions with laboratory environment, and the use of analytical control blanks. Careful attention was given to ensuring all the reagents used in the sample/filtration process, including the ultrapure water and chemicals (ethanol, surfactant), were pre-filtered before use with a non-polymeric membrane with a pore size ≤ 5 µm to remove any contaminant MP. The ubiquitous nature of MP in the working environment was considered by taking contamination prevention measures, including not wearing gloves, washing hands and washing the outside of all containers. Laboratory personnel wore cotton lab coats and did not wear face masks or cosmetics. Most work was conducted under a laminar flow hood that was regularly cleaned. The use of plastic-based equipment was avoided to prevent cross-contamination.

The establishment of analytical control blanks is mandatory to measure the impact of the different steps (sampling and filtration) and ensure the quality and significance of the analysis is in line with ISO 12141:2024 (ISO, 2024). For that purpose, an analytical control blank comprising particle-free water of known quality was produced for each analysis in series. This blank followed the same protocol as the corresponding sample and was analysed first to ensure that no contamination has been introduced during the sampling and filtration process. This is most important for number-based quantification methods, which detect each individual particle. Although the same blank controls were used for mass-based quantification methods, the mass of any possible contamination is often below the limit of detection (LOD). The number or mass of PET particles found in the blank samples were subtracted from the results of the main samples as part of accurate measurements.

2.7. Results and discussion

2.7.1. Characterisation of PET particles

The PET polymer type was confirmed by Attenuated Total Reflectance-FTIR (ATR-FTIR) measurements of the pristine PET powder (**Fig. S1**). SEM images showed that the pristine PET particles were irregular shaped with sharp edges, and that a range of particle sizes was evident (Fig. 3). Laser diffraction analysis confirmed that the pristine

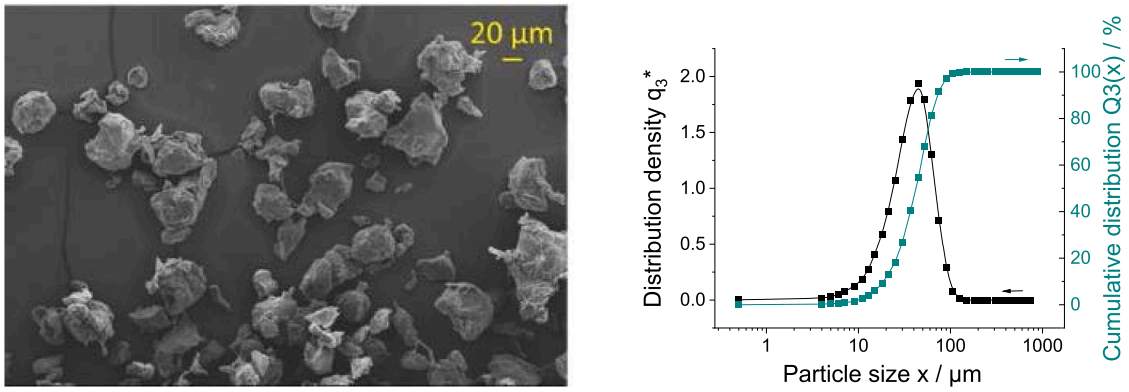


Fig. 3. SEM image (left) and particle size distribution of PET particles (right).

PET particles had a broad size distribution, with distribution density values of D10=19.1 ± 0.2 µm (1.0%), D50=42.4 ± 0.2 µm (0.4%) and D90=72.9 ± 0.3 µm (0.5%), where D10/D50/D90 is the equivalent particle diameter of the measured volume below which 10.3/50.3/90.3% of the particles are. It should be noted that 93.5% of all particles are in the size range between 20 and 100 µm with an approximately equal number distributed across the size fractions 20–50 µm (49.1%) and 50–100 µm (44.4%) (Table 3).

2.7.2. *Homogeneity and stability control studies*

The PET spiked tablets consist of three components, and TGA of each component revealed different distinct derivative thermogravimetric (DTG) peaks: lactose (DTG = 301.4 °C), PEG (DTG = 361.7 °C) and PET (DTG = 430.9 °C) (Fig. 4). The DTG is the peak maxima of the first-time derivative of the mass loss curve determined in TGA. The mass loss rate curves indicated that there is a slight interference between the mass loss of PEG and PET between 330 and 400 °C. Since PEG is easily soluble in water, as is lactose, it is expected to be removed during the filtration process and therefore should no longer be present on the filter after the PET particles have been isolated. Thus, the total mass loss observed in the temperature range 330–480 °C is assumed to represent only PET, allowing a direct mass correlation (m (mass loss,330–480 °C) = m (PET in tablet)). Hence, one tablet was used for each filtration, and the resulting dry residue was analysed by TGA. Ten randomly selected tablets were tested to assess the homogeneity of PET particles.

The first indication of PET homogeneity in the tablets is presented in Fig. 5 **(see Table S1 and S2).**

As quality check, the theoretical PET mass of 400 µg (Batch 1) and 18.6 µg (Batch 2) match well with the calculated mean masses (n = 10) by TGA measurements, where m(Batch 1) =398.0 ± 47.3 µg and m (Batch 2) =19.6 ± 1.8 µg. The relative standard deviation (RSD) for both batches is approximately 8–12%. It should be noted that the uncertainty of the TGA itself is 2 µg according to the manufacturer. Further TGA measurements done in similar experimental setup but with another TGA of the same model and brand indicate comparable results (n = 3). Hence, m(Batch 1) =398.0 ± 47.3 µg and m(Batch 2) =19.6 ± 1.8 µg will be used as reference values in this work.

2.7.3. *Mass-based thermo-analytical methods*

A well-characterised reference material is required to support the assessment of the performance of MP detection methods. In accordance with the ISO/FDIS 16094-3 (ISO/FDIS, 2026), other mass-based methods were used to measure the amount of PET present in the Batch 1 and Batch 2 tablets, providing supportive information for the TGA-based homogeneity assessment and reference values for the selected mass-based detection methods. A total of 17 PET RM tablets from Batch 1 were analyzed by Py-GC/MS and TED-GC/MS using direct sample preparation, in which the PET particles retained on the filters were directly introduced into the respective analytical system. Further measurements were carried out with 10 tablets using Py-GC/MS after derivatization of the PET (Fig. 6, see Table S1 and S2). Details of the two sample preparation procedures are provided in the Supplementary Information. The mass-based results for Batch 3 PET spiked tablet are not shown, however, one laboratory detected 0.27±0.05 µg of PET (RSD: 19.7%; n = 10).

All 44 measurements of Batch 1 are in good correlation to each other, with a mean mass value of 422.3 µg, a mean standard deviation (SD) of 88.7 µg, and a mean RSD of 21.0%. Importantly, this mean PET mass value is fully consistent with the reference value of 398.0 ± 47.3 µg determined in the homogeneity control by TGA. Direct Py-GC/MS and TED-GC/MS show quite comparable mean values of 448.9 µg and 431.3 µg, respectively, with both slightly overestimating the mass of PET relative to the TGA reference value. The SD of TED-GC/MS (55.7 µg) is lower than that of Py-GC/MS (105.1 µg). The derivatized PET particles detected by Py-GC/MS gave a slight underestimation compared to the reference value, indicating not all of the PET particles were fully derivatized. Non-derivatized PET will not be detected when using dimethyl terephthalate as the quantifier.

The mass of PET particles in the Batch 2 tablets is diluted by a factor of 30 compared to the Batch 1 tablets. This dilution was conducted to achieve a PET concentration that is more realistic according to the MP mass/numbers found in food products. For Batch 2, 10 tablets were directly analysed by Py-GC/MS and 10 tablets were directly analysed by TED-GC/MS. A further 10 tablets were analysed by Py-GC/MS using derivatization. All three methods show similar mean values with an average of 17.1 ± 5.7 µg (33.1%) that corresponds well to the TGA reference value of 19.8 ± 1.8 µg (8.9%). The direct and derivatized Py-GC/MS measurements are very similar in mass (17.7 and 16.8 µg) and SD (3.9 and 4.5 µg), while the SD and RSD of the TED-GC/MS measurements is quite high with values of 8.2 µg and 48.6%, respectively.

The Py-GC/MS and TED-GC/MS analyses show generally similar masses for both Batch 1 and Batch 2 tablets as those determined by TGA.

Table 3
Comparison of integrated areas of distinct size fractions determined with laser diffraction to mean values of all partners of particle numbers per fraction and 28 tablets measured.

	Size fraction	µm	5–10	10–20	20–50	50–100	100–500
Laser diffraction	Ratio integrated area	%	0.5	4.4	49.1	44.4	1.7
µ-Raman	Ratio integrated area (mean all labs)	%	9.3	19.0	49.7	18.8	3.2

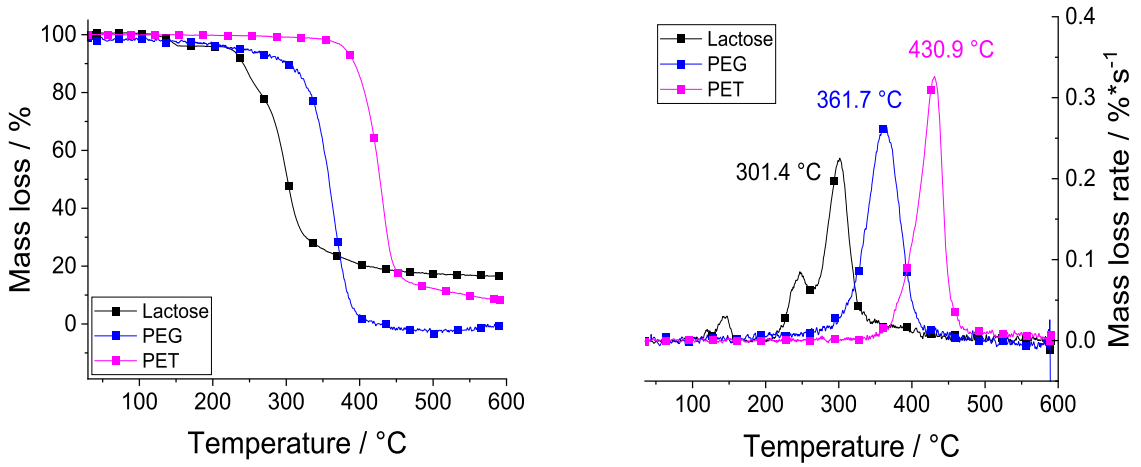


Fig. 4. Mass loss (left) and mass loss rate (right) of lactose, PEG and PET of TGA measurements.

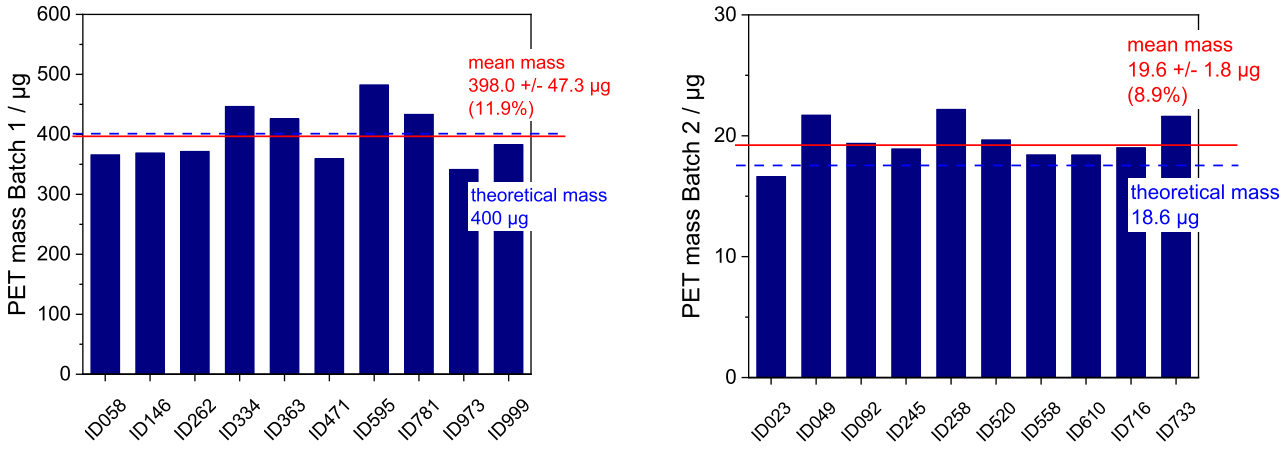
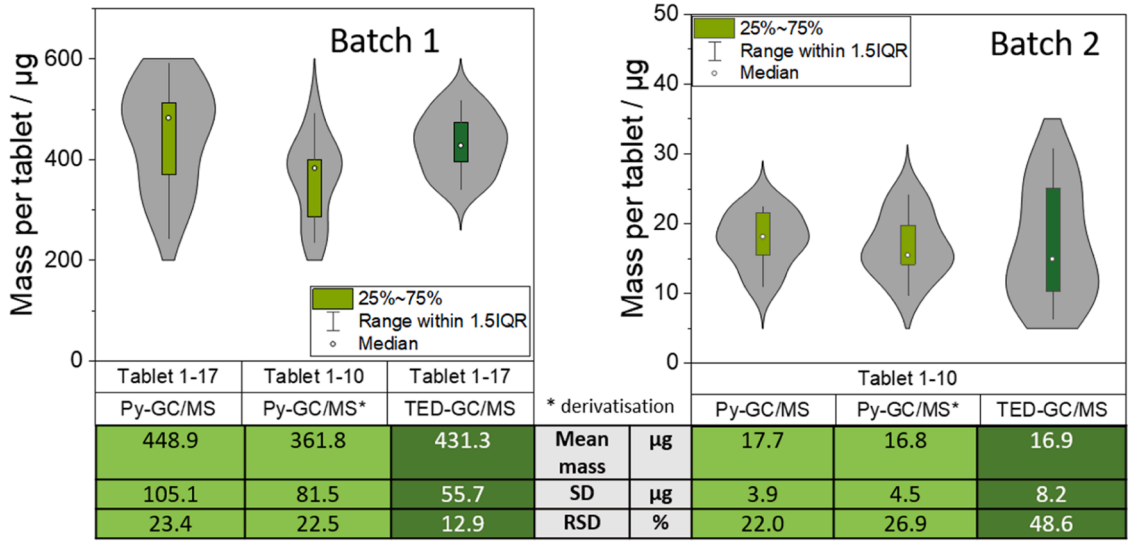


Fig. 5. PET masses per tablet determined by TGA for Batch 1 (left) and Batch 2 (right) as a homogeneity control; n = 10.



Tablet 1-17	Tablet 1-10	Tablet 1-17			Tablet 1-10		
Py-GC/MS	Py-GC/MS*	TED-GC/MS	* derivatisation		Py-GC/MS	Py-GC/MS*	TED-GC/MS
448.9	361.8	431.3	Mean mass	µg	17.7	16.8	16.9
105.1	81.5	55.7	SD	µg	3.9	4.5	8.2
23.4	22.5	12.9	RSD	%	22.0	26.9	48.6

Fig. 6. PET masses per tablet detected with direct Py-GC/MS analysis, direct TED-GC/MS analysis, and with derivatization Py-GC/MS analysis. The grey part of the violine plot marks the width that indicates the frequency or density of the data. The table gives mean values, SD and relative RSD in numbers. The results for Batch 1 PET spiked tablets are presented on the left side of the figure and the results for Batch 2 PET spiked tablets are presented on the right side of the figure.

The higher PET masses present in the Batch 1 tablets are slightly overestimated compared to the TGA, while the lower PET masses in the Batch 2 tablets, which are near the instrumental LODs, are within the TGA reference value or are slightly underestimated. Overall, the masses

and the SDs are in the same range for both batches of PET spiked tablets and correspond well with theoretical values and the TGA reference values. This makes the PET spiked tablets a valuable product for use in method validation for MP identification and quantification methods, supporting EU directives and monitoring campaigns and enabling reliable comparison of analytical results.

Stability control of PET spiked tablets was assessed after 6 months for Batch 1 and after 12 months for Batch 2, using 2 to 5 tablets per batch (Fig. 7, see Table S7, S8 and S9). No significant changes in PET mass were observed for either batch by TGA, Py-GC/MS and TED-GC/MS compared with the values determined at month 0. These results indicate that PET spiked tablets, irrespective of the PET concentration, remained stable over these storage periods.

2.7.4. Spectroscopic methods

PET spiked tablets were also analysed for particle numbers and size distributions as this is an important metric for supplementing the mass, especially as MP monitoring typically requires particle number to be reported. The Batch 1 tablets were not analysed using the spectroscopic techniques as the high concentration of PET particles would result in too many particles being collected on the filters. This has the double challenge of overloading the filters and making it difficult for the image-based techniques to identify and analyse individual particles, while a higher number of particles requires much more time to complete a full analysis of the filter. Therefore, Batch 2 and Batch 3 tablets with a lower theoretical PET particle concentration were analysed with the spectroscopic techniques.

Three different laboratories measured Batch 2 tablets to determine the number of PET particles numbers and their respective sizes. One laboratory analysed 8 tablets (Lab 2) by μ-Raman, another laboratory analysed 10 tablets by μ-Raman (Lab 3), while a third laboratory analysed 10 tablets by both μ-Raman and μ-FTIR (Lab 1). A comparison of the resulting PET spiked tablet particle numbers, which are counted and sorted for the size fractions 1–5, 5–10, 10–20, 20–50, 50–100 and 100–500 μm, is shown in Fig. 8 **(see Table S3).** In addition, the values are compared to those determined by laser diffraction for the pristine PET particles. The particle numbers in the individual size classes match well within and between the laboratories. The mean total particle number (n = 28) in Batch 2 tablets with a size between 20 and 500 μm is 1764 ± 155 (8.8%). The sizes correspond well with the results from the laser diffraction.

A comparison of the particle size distributions obtained by μ-Raman spectroscopy and laser diffraction revealed a generally similar overall trend (Table 3). However, it should be noted that the two techniques are based on fundamentally different measurement principles and data representations. Laser diffraction provides a volume-based particle size distribution, in which larger particles contribute disproportionately to the measured signal, whereas μ-Raman spectroscopy generates a particle-number-based size distribution, where each detected particle contributes equally regardless of its size. Consequently, a direct quantitative comparison between the integrated laser diffraction signal and the particle counts obtained by μ-Raman is inherently limited due to fundamentally different weighting of particle populations and methodological aspects associated with the respective measurement principles.

Despite these limitations, both methods indicated that a substantial proportion of the particles were present in the size range of 20–50 μm. Compared to laser diffraction, μ-Raman spectroscopy detected a higher proportion of particles in the smaller size classes. Next to the different measurement principles, one possible explanation is particle fragmentation during the tableting process, which was performed at compression forces of up to approximately 50 kN (manufacturer's specification). However, no direct evidence of fragmentation was obtained in the present study, and this explanation should therefore be regarded as hypothetical.

Furthermore, laser diffraction data can be evaluated using either the Fraunhofer or the Mie theory model, which are generally more suitable for larger or smaller particles, respectively. As the investigated material exhibited a broad particle size distribution, the Fraunhofer approach was selected as a compromise, potentially favoring the representation of larger particles. Accordingly, while laser diffraction indicated that 44% of the PET particles were present in the 50–100 μm size range, only 19% of the particles detected by μ-Raman fell into this size class. In contrast, μ-Raman spectroscopy identified higher proportions of PET particles in the smaller size classes of 10–20 and 5–10 μm. These differences highlight the challenges associated with directly comparing results obtained by the two analytical techniques.

The particle number counted with μ-Raman for Batch 2 were also compared to particle number values determined with μ-FTIR. A similar comparison was conducted for Batch 3 tablets containing the lowest concentration of PET particles. The results are presented in Fig. 9 **(see Table S3, S4 and S5).** Although the total number of particles was reduced from 1759 (n = 10, μ-Raman, 5–500 μm) for Batch 2 to 151 particles for Batch 3 (n = 10, μ-Raman, 5–500 μm) it is clear that the size distribution is similar. PET particle numbers counted with μ-Raman and μ-FTIR for both Batch 2 and Batch 3 tablets were similar for particle sizes larger than 50 μm. In contrast, μ-FTIR detects 56% less (Batch 2) and 55% less (Batch 3) PET particles than μ-Raman in the 20–50 μm size category. The observed difference is likely related to the different detection capabilities of the two techniques, particularly for smaller particles. The particle-size detection limit of μ-FTIR is higher than that of μ-Raman, with μ-FTIR showing limitations for particles below approximately 20 μm, according to ISO 16094-2:2025 (ISO, 2025). This

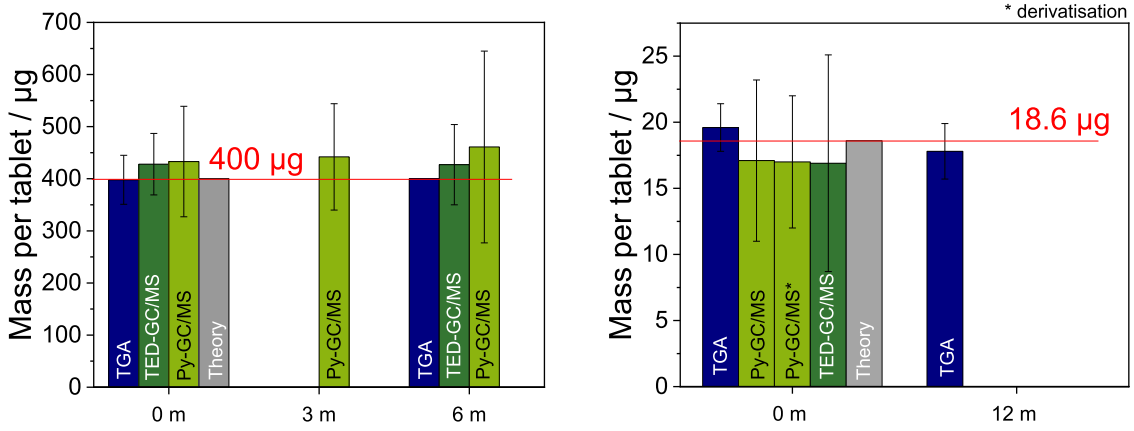


Fig. 7. PET masses per tablet determined by mass-based methods for Batch 1 (left) and Batch 2 (right) as stability control assessment; n = 2–5 (The red line indicates the theoretical mass).

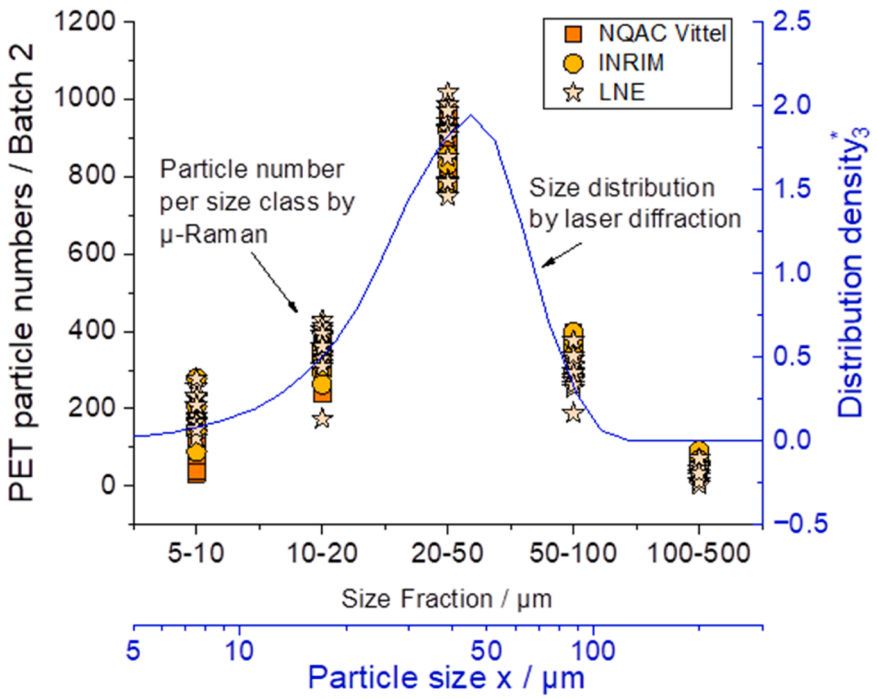
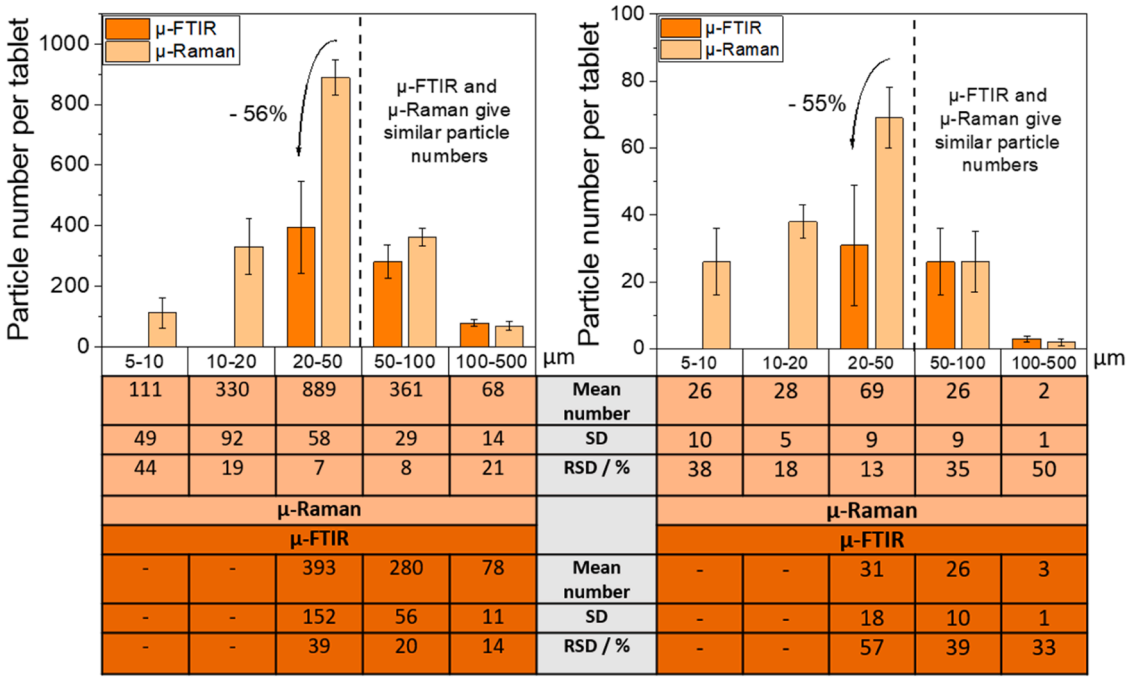


Fig. 8. Lab-to-lab comparison of PET particle numbers per tablet detected by µ-Raman of three different partner for Batch 2.



5-10	10-20	20-50	50-100	100-500	µm	5-10	10-20	20-50	50-100	100-500
111	330	889	361	68	Mean number	26	28	69	26	2
49	92	58	29	14	SD	10	5	9	9	1
44	19	7	8	21	RSD / %	38	18	13	35	50
µ-Raman						µ-Raman				
µ-FTIR						µ-FTIR				
-	-	393	280	78	Mean number	-	-	31	26	3
-	-	152	56	11	SD	-	-	18	10	1
-	-	39	20	14	RSD / %	-	-	57	39	33

Fig. 9. Comparison of particle numbers of Batch 2 (left) and Batch 3 (right) detected with µ-FTIR and µ-Raman, n = 10, all measurements are performed in the same laboratory (Lab 1).

difference is consistent with the higher spatial resolution generally achievable by µ-Raman compared with µ-FTIR, which facilitates the detection and chemical identification of smaller particles. Nevertheless, the present results show that differences in particle detection between µ-Raman and µ-FTIR were already evident within the 20–50 µm size fraction. This indicates that differences in practical analytical performance may arise at particle sizes above the nominal lower size limit considered for regulatory monitoring, consistent with the critical size threshold of 50 µm identified in a recently published interlaboratory study of IR-based methods (Putzu et al., 2026). For the µ-Raman, all size distributions for Batch 2 and Batch 3 tablets determined by microscopy showed the highest RSD for smallest and largest size categories (5–10 and 100–500 µm), while the lowest RSD were recorded for the size fraction 20–50 µm. This is probably related to the number of PET particles per size class. From a statistical point of view, it is easier to distribute a larger number of particles of the same size homogeneously from tablet to tablet than a smaller particle number. The presence or absence of individual particles in a tablet has a greater effect on the RSD

when the total number of particles in a size class is very low. For the μ-FTIR data, the RSDs were notably higher across all measured size categories compared to the equivalent μ-Raman values. The highest RSD for both Batch 2 and Batch 3 were observed for the smallest size category measurable by μ-FTIR (20–50 μm). This suggests that particles in the 20–50 μm size range, particularly those close to 20 μm, are more challenging to detect consistently by μ-FTIR under the applied analytical conditions. As discussed above, the higher spatial resolution generally achievable by μ-Raman facilitates the detection and chemical identification of smaller particles. This is reflected in the μ-Raman results, which showed that the 20–50 μm size range contained the highest number of PET particles in all tablets tested. Under the analytical conditions investigated in this study, μ-FTIR provided comparable PET particle numbers to μ-Raman for particles larger than 50 μm, whereas μ-Raman enabled the detection and quantification of PET particles down to at least 20 μm.

2.7.5. *Matrix contamination*

Although blank control samples need to be conducted for determination of accurate particle mass and number measurements as part of QA/QC, it is equally important to analyse the matrix (lactose and PEG) as possible source for contamination. Therefore, matrix blank tablets were produced containing lactose and PEG but with no addition of PET particles. These tablets were tested in the participating different laboratories for particle number and mass after running through the entire filtration process in the same way as for the PET containing tablets. Analysis of the resulting data revealed that no thermoplastic polymers were detected in the matrix material using thermo-analytical methods. This indicates that no thermoplastic particles were present or, more likely, that the mass of any polymer contamination is below the LODs of the instruments.

In contrast to the thermo-analytical analysis of the matrix blank tablets, six different types of polymer particle were detected in the number-based spectroscopic approaches. A summary of the results is provided in Fig. 10. Polyethylene (PE), polypropylene (PP), polystyrene (PS), PET, polyamide (PA) and polytetrafluoroethylene (PTFE) particles were found across 8 tablets analysed by μ-Raman and 7 tablets analysed by μ-FTIR. For μ-Raman, 6 of the 8 matrix blank tablets contained polymer particles, ranging in number from 1 particle to 51 particles. PS particles dominate 3 of the tablets (27–32 particles), while PET (1–6 particles) was found in 5 of the matrix blank tablets. For μ-FTIR, all 7 of the matrix blank tablets contained polymer particles, ranging in number from 2 particles to 36 particles. PET particles were found in 5 of the 7 the matrix blank tablets, ranging from 1–16 particles.

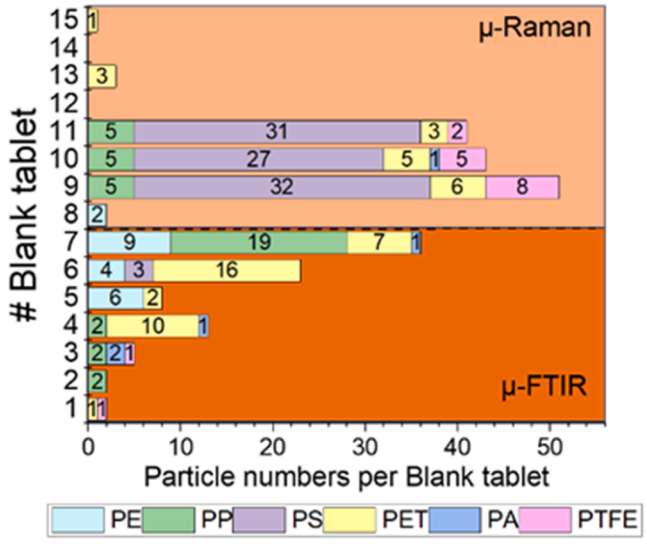


Fig. 10. Plastic contamination found in matrix blank tablets as determined by μ-Raman and μ-FTIR (n = 8/n = 7).

The presence of PET particles in matrix blank samples is of particular concern because the target measurand of the reference material candidate is the mass of PET particles. Even low numbers of PET particles introduced through contamination may therefore bias the interpretation of the results and affect the assessment of method performance, especially for tablets with very low particle numbers. Furthermore, the source of such contamination cannot be unambiguously identified, as PET particles may originate from numerous environmental sources, including airborne particles in the laboratory environment, laboratory consumables, equipment, or synthetic fibers released from clothing worn by personnel during sample handling. Consequently, the occurrence of PET particles in blank samples highlights the importance of stringent contamination control and the careful interpretation of low particle counts close to background levels. Future reference material processing must work as free of contamination as possible.

Overall, the number of contaminant particles detected was very low (>3%) compared to the total mean number of PET particles present in the Batch 2 tablets (1759 particles). In the case of the Batch 2 tablets, the matrix is considered to be sufficiently clean for it to be used to produce reference materials containing this concentration of PET particles. It should be noted that the number of polymer particles identified by the spectroscopic methods in the matrix blank tablets includes both matrix contamination as well as any possible contamination occurring during the sample production, storage, transportation, sample preparation (filtration) and measurement of the filter itself. When comparing the two analysis techniques, μ-Raman detects more particles than μ-FTIR. This is expected due the lower particle size detection limit of Raman microscopes and may explain why PS particles were not found in μ-FTIR if they are smaller than 20 μm. PTFE particle contamination was also only be detected by μ-Raman, which indicates that the particles are again <20 μm and that somewhere in the sample preparation process a seal made of PTFE was used. It should also be noted that PS and PTFE contamination was only found in the same 3 tablets analysed by μ-Raman and not the other 5 tablets, indicating the contamination was not consistent.

2.7.6. *Tablets with varying polymer types and smaller particle sizes as outlook*

Recognizing that tablet production works reasonably well for the PET particles utilized in the current study and knowing that reference materials comprising more polymer types are needed to monitor microplastics, we tried to press cryomilled PP and commercially available LDPE particles into 'Batch 2' type tablets with the same lactose and PEG matrix. The results of PP are published by Putzu et al. (Putzu et al., 2026), whereas first result of the very small LDPE can be found in **Table S10.** These LDPE particles proved to be much more challenging than the PET particles due to the mean particle size being D50: 18.0 ± 0.7 μm. This size distribution mainly covers the most challenging region of <50 μm for the number-based μ-FTIR and μ-Raman spectroscopic methods. The homogeneity control determined by TGA showed a mean LDPE mass of 17.8 ± 1.6 μg, which perfectly matches the theoretical mass (17.9 μg) **(Fig. S2, Table S11).**

Lab 1 and Lab 3 determined the total particle number by μ-Raman with mean total numbers of LDPE particles being 6205 ± 469 and 5460 ± 1069, respectively **(Fig. S3, Table S12, S13 and S14).** As with the PET spiked tablets, the mean total number of LDPE particles detected by μ-FTIR (1589 ± 206, Lab 1) was lower compared to μ-Raman. This result is expected given the small average size of the LDPE particles (18 μm) incorporated into the tablets. As a result, a high proportion of the LDPE particles are below the limit of reporting for μ-FTIR. The total particle number determined by μ-Raman differs for the smallest size category (5–10 μm) between Lab 1 and Lab 3. Similarly large differences between the total particle numbers derived by the two laboratories are observed for the 20–50 μm and the 100–500 μm size categories. As one

lab is not consistently reporting higher particle numbers across all size categories further investigation is needed into why this is the case. Although, μ-IR and μ-Raman techniques have undergone substantial development in recent years, the reliable detection, identification, and quantification of microplastic particles in the size range below 50 μm remains challenging and is still an active area of methodological research. At these sizes, particles approach the physical and instrumental limits of optical detection. For μ-FTIR, the spatial resolution is constrained by the diffraction limit of infrared radiation, resulting in a substantial proportion of particles being below the limit of reliable detection and identification. While μ-Raman microscopy offers higher spatial resolution, the analysis of very small particles is still hampered by weak signal intensities, fluorescence interference, particle orientation effects, and differences in image processing and particle recognition algorithms. Consequently, small variations in instrumental settings, measurement strategies, and data evaluation procedures can significantly influence the reported particle numbers, particularly in the smallest size classes. These challenges likely contribute to the observed variability between laboratories and highlight the need for continued method development and standardisation for the analysis of microplastics below 50 μm.

3. Conclusion

The tablet-based production approach can be used to prepare candidate reference materials with different polymer type, polymer mass and particle number levels, while providing sufficient consistency among individual tablets for their intended use in MP method assessment. The results demonstrate that PET mass and/or particle number should be considered as distinct measurands, as the analytical methods investigated showed different performance characteristics for their determination. For the measured parameter "mass," the PET contents determined by TGA agreed well with the theoretical values. In contrast, Py-GC/MS and TED-GC/MS (reported as informative values) generally yielded comparable mass values but exhibited greater variability, particularly in the lower PET mass range (Batch 2), suggesting that the development of a robust GC/MS-based method for microplastic mass determination still requires further work. Despite these variations, the measured masses and SDs remained within a comparable range for both batches and corresponded well with the theoretical and TGA reference values. For the particle number measurand, the PET spiked tablets enabled the comparison of μ-Raman and μ-FTIR measurements between laboratories, showing comparable particle numbers and size distributions. At the same time, the comparison with μ-FTIR highlighted a pronounced difference in particle detection in the 20–50 μm size range, consistent with the different detection capabilities of the two techniques and highlighting the importance of including small particle sizes when assessing method performance and comparability. The detection of polymer particles in the matrix blanks further highlighted the importance of rigorous contamination control throughout the entire sample preparation and analysis workflow, particularly when working with materials containing low particle numbers.

Overall, the developed PET spiked tablets provide a useful test material for assessing method performance across different PET mass and particle number levels, enabling the identification of method-dependent differences and supporting the comparison and harmonization of analytical procedures. Preliminary results obtained with LDPE suggest that the tablet-based production approach could be extended to other polymers, although further investigation is needed to evaluate its broader applicability.

Associated content

Supporting information

Supporting Information contains detailed information on measurement procedures for the individual analytical techniques as well as tables with the individual PET or LDPE particle numbers and masses determined for homogeneity and stability control. The dilution scheme of LDPE production is also presented.

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CRediT authorship contribution statement

Korinna Altmann: Writing – original draft, Supervision, Conceptualization. **Yosri Wiesner:** Writing – review & editing, Investigation. **Petra Fengler:** Investigation. **Christiane Weimann:** Investigation. **Claudia Drago:** Writing – review & editing, Investigation. **Ulrike Braun:** Writing – review & editing, Investigation, Conceptualization. **Marta Fadda:** Writing – review & editing, Investigation. **Mara Putzu:** Writing – review & editing, Investigation. **Alessio Sacco:** Investigation. **Andrea Mario Rossi:** Supervision, Funding acquisition. **Soledad Muniategui Lorenzo:** Writing – review & editing, Supervision. **Carmen Moscoso-Pérez:** Investigation. **Verónica Fernández-González:** Investigation. **Paul-Tiberiu Miclea:** Writing – review & editing, Conceptualization. **Nizar Benismail:** Writing – review & editing, Supervision. **Laureen Coic:** Writing – review & editing, Investigation. **Alina Maltseva:** Investigation. **Bert van Bavel:** Conceptualization. **Vilde Kloster Snekkevik:** Conceptualization. **Andy M. Booth:** Writing – review & editing, Supervision. **Lisbet Sørensen:** Investigation. **Enrica Alasonati:** Supervision. **Carine Chivas-Joly:** Writing – review & editing, Investigation. **Andrea Mario Giovannozzi:** Writing – review & editing, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests.

Korinna Altmann reports financial support was provided by European Partnership on Metrology. Korinna Altmann reports financial support was provided by European Union. Korinna Altmann has patent #Method for detecting microplastic particles or fibers in a sample, Reference Material, Tablet or Capsule, Manufacturing method and Use thereof, and Biological assessment test (35891P-DE) pending to Licensee. The work is funded by the project 21GRD07 PlasticTrace (Funder ID: 10.13039/100019599) which has received funding from the European Partnership on Metrology, co-financed from the European Union's Horizon Europe Research and Innovation Programme and by the Participating States" and received additional funding from the European Un-ion's Horizon 2020 research and innovation programme under grant agreement of Polyrisk (No 964766) and PlasticsFate (No 965367).

Korinna Altmann, Ulrike Braun and Yosri Wiesner submitted a patent with the title "Method for detecting microplastic particles or fibers in a sample, Reference Material, Tablet or Capsule, Manufacturing method and Use thereof, and Biological assessment test " (35891P-DE)

If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

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Supplementary materials

Supplementary material associated with this article can be found, in the online version, at doi:10.1016/j.hazadv.2026.101686.

Data availability

Data will be made available on request.

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