Supporting Information

Interlaboratory comparison reveals state-of-the-art in microplastic detection and quantification methods

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Table S1. Overview of interlaboratory comparisons related to the analysis of microplastic.

Parameters	Polymers	Particle size (μm)	Matrix	Methods	Nr. labs	Drawbacks	Reference
Particle number, PSD	PP, HDPE, LDPE	400-5700	Seawater	FTIR, μ-FTIR, Raman	12	Polymer identity missing, only large particles	Isobe *et al.*[1]
Mass fraction	PE, PET, PS, PP	145-174	Freshwater sediment	Py-Gc/MS, TED-GC/MS, TGA-FTIR, TGA-MS, DSC,	16	Pristine polymers, narrow particle size range	Becker *et al.*[2]
Particle number, polymer type, or particle mass	PE, PVC, PMMA, PET, PS,	8-140	Ultrapure water	Microscopy, μ-FTIR, μ-Raman spectroscopy, TED-GC/MS, SEM	17	Pristine MP, test material RSD 26-85%, ultra-pure water	Müller *et al.*[3]
Polymer type, particle number, polymer mass	PC, PS, PP, PET, LDPE, EPS	150-300 and 2000-4000	Soda tablets	Microscopy, gravimetric, ATR-FTIR, μFTIR, Py-GC–MS, Raman, μ-Raman spectroscopy.	34	Only large particles	WEPAL-QUASIMEME/NORMAN *et al.*[4]
Polymer type, particle number	PVC, PET, PE, EPS, PS	1-500	Drinking water + gelatine	FTIR, Raman	12		Munno *et al.*[5]
Polymer type, particle number, particle size	PE, PS, PVC, PET	1-500	Drinking water	OM, μ-FTIR, μ-Raman spectroscopy	22	Clean water as matrix	De Frond *et al.*[6]
Particle number, size, mass fraction	PET in water	30-200	Clean water	μ-FTIR, μ-Raman spectroscopy, Py-GC/MS, fluorescence microscopy, TGA, LDIR, NMR, HPLC	98	Clean water as matrix	European Commision[7]
Polymer type, polymer mass	PE, PS, PVC	125-355	Soda tablets,	μ-FTIR, Py-GC/MS, microscopy, μ-Raman spectroscopy, LDIR	7	Narrow particle size range	INOPOL/SINOPLAST[8]
Polymer identity, PSD, particle number	PE, PET, PS, PC, PP, PVC	50-300	Soda tablets, sediments, sand samples	OM, gravimetric, ATR-FTIR, μFTIR, μFTIR-FPA, Py-GC–MS, Raman, μ-Raman spectroscopy, LDIR, Fluorescent	90		EUROqCHARM[9]

				microscopy, stereomicroscopy, SPERO-QT			
Polymer identity, particle count, total weight	HDPE, PET, PC, PP, PVC	300-5000	Marine sediment and sea water	ATR-FTIR, FTIR	5	Large MPs, few laboratories, only two methods	Cadiou et al.[10]

Table S2. Mass fraction (as µg polymer per mg tablet) (n=19) in the homogeneity study

	PET	SD	RSD	PE	SD	RSD
TGA	2.2	0.3	14.1	0.9	0.1	16.0
TED-GC/MS	1.9	0.5	27.5	0.8	0.1	18.0

Table S3. Particle number for both polymers measured with µ-FTIR (n=10) NQAC Vittel

µ-FTIR	PET (n=10)	SD	RSD	PE (n=10)	SD	RSD
>500 µm	0.7	1.6	228.5	3.5	4.9	140.0
100-500 µm	136.5	40.4	29.6	137.8	46.2	33.5
50-100 µm	214.3	45.8	21.3	121.5	38.2	31.4
*20-50 µm	198.4	55.5	27.9	72.9	45.5	62.4
Total	549.9	78.4	14.2	335.7	94.1	28.0

* > 20 µm - lowest limit of confident detection for µ-FTIR

Table S4. Particle number for both polymers measured with µ-Raman (n=10) NQAC Vittel

µ-Raman	PET (n=10)	SD	RSD	PE (n=10)	SD	RSD
> 500 µm	1.0	0.8	80.0	2.2	2.3	104.5
100-500 µm	107.5	15.1	14.0	129.3	25.2	19.4
50-100 µm	265.6	38.4	14.4	210.4	82.8	39.3
20-50 µm	492.6	73.4	14.9	496.9	228.2	45.9
10-20 µm	275.2	33.2	12.0	450.9	193.1	42.8
5-10 µm	152.3	36.7	24.0	266.9	140.1	52.5
Total	1294.2	151.9	11.7	1556.6	631.2	40.5

Table S5. Particle number concentration (n=20) per tablet measured by manual counting from SEM micrographs

	PET (n=10)	SD	RSD	PE (n=10)	SD	RSD
>500 µm	0.0	0.0	0.0	0.1	0.3	244.2
100-500 µm	3.9	2.5	65.4	81.7	30.5	37.4
50-100 µm	171.5	63.2	36.8	298.9	97.8	32.7
10-50 µm	730.3	255.5	34.9	494.7	279.8	56.5
Total	905.7	295.7	32.2	876.8	357.1	40.7

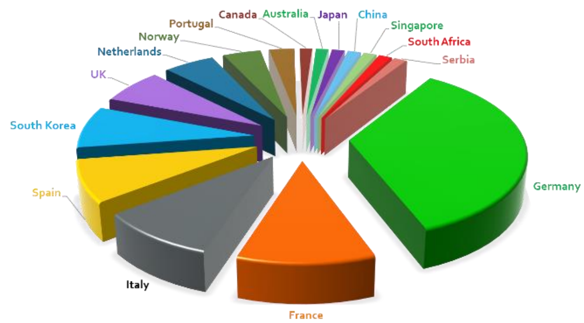

Figure S1. Overview of participants by country

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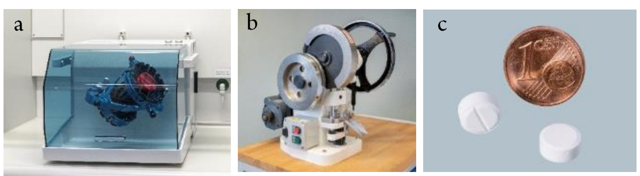
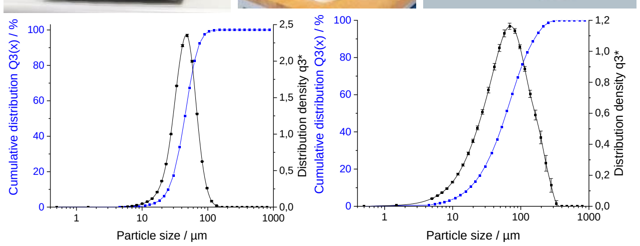




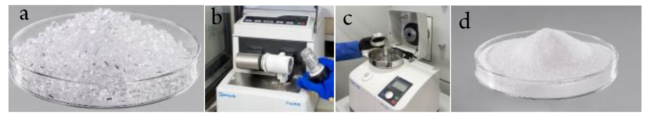


Figure S2. Particle size distribution curves of microplastic polymers as determined with laser diffraction method: a) PET and b) aged PE.

Figure S3. Production process of microplastic powder from commercial plastic granulate: a) plastic granulate, b) cryo-milling, c) sieving, d) microplastic powder (1-100 µm).

Figure S4. Preparation of the tablets from microplastic powder: a) homogenization, b) pressing step with press machine, c) pressed tablets (about 250 mg).

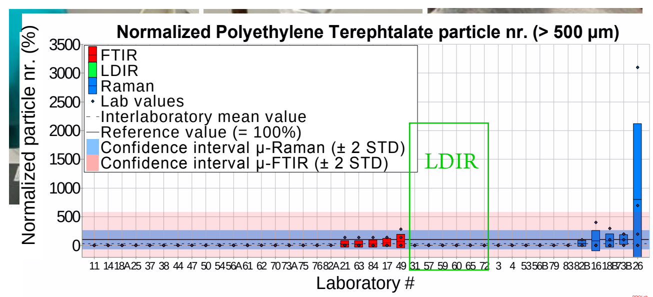


Figure S5. Filtration for thermo-analytical experiments with use of a crucible. a) placing the crucible; b) MilliQ water dropping on the tablet; c) tablet rests (presumably microplastic) after completing the filtration process.

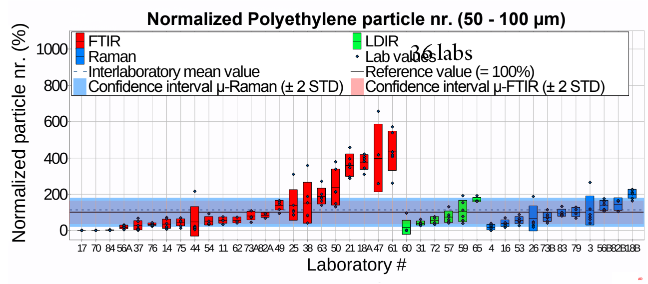
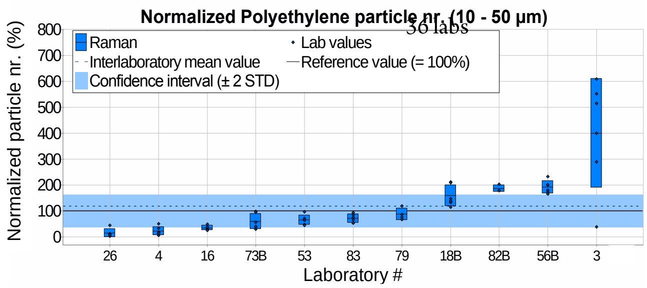
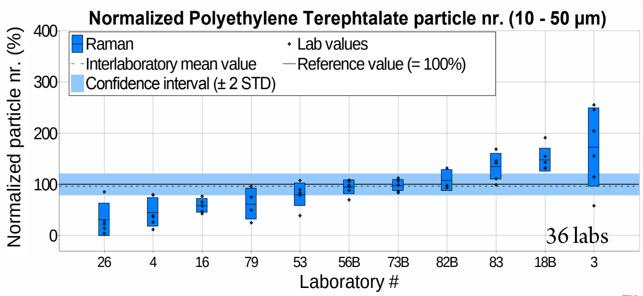


Figure S6. Normalized aged PE particle numbers (> 500 µm) per tablet derived from µ-FTIR, µ-Raman and LDIR measurements. Red, green and blue bars – standard deviations of intra-laboratory results (repeatability).





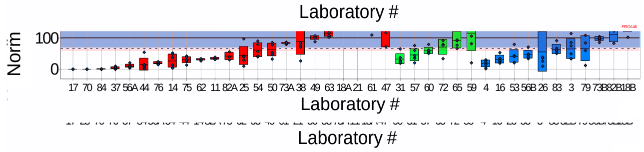


Figure S7. Normalized PET particle numbers (> 500 µm) per tablet derived from µ-FTIR, µ-Raman and LDIR measurements. Red, green and blue bars – standard deviations of intra-laboratory results (repeatability).

Figure S8. Normalized aged PE particle numbers (100 - 500 µm) per tablet derived from µ-FTIR, µ-Raman and LDIR measurements. Red, light green and blue bars – standard deviations of intra-laboratory results (repeatability).

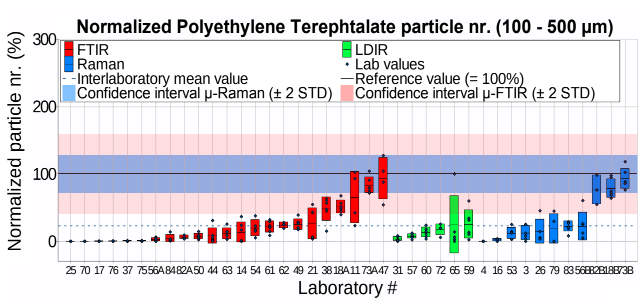
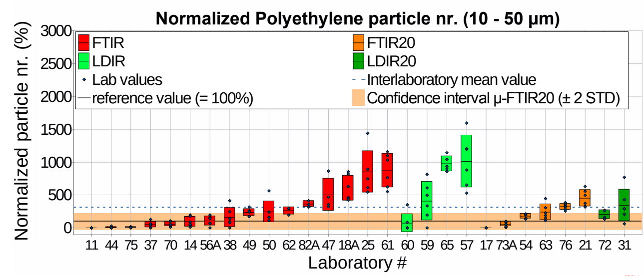


Figure S9. Normalized PET particle numbers (100 - 500 µm) per tablet derived from µ-FTIR, µ-Raman and LDIR measurements. Red, light



green and blue bars – standard deviations of intra-laboratory results (repeatability).

Figure S10. Normalized aged PE particle numbers (50 - 100 µm) per tablet derived from µ-FTIR, µ-Raman and LDIR measurements. Red, light green and blue bars – standard deviations of intra-laboratory results (repeatability).

36 labs

Figure S11. Normalized PET particle numbers (50 - 100 µm) per tablet derived from µ-FTIR, µ-Raman and LDIR measurements. Red, light green and blue bars – standard deviations of intra-laboratory results (repeatability).

Figure S12. Normalized aged PE particle numbers (10 – 50 µm) per tablet derived from µ-Raman measurements. Blue bars – standard deviations of intra-laboratory results (repeatability).

Figure S13. Normalized PET particle numbers (10 – 50 µm) per tablet derived from µ-Raman measurements. Blue bars – standard deviations of intra-laboratory results (repeatability).

11 labs
36 labs

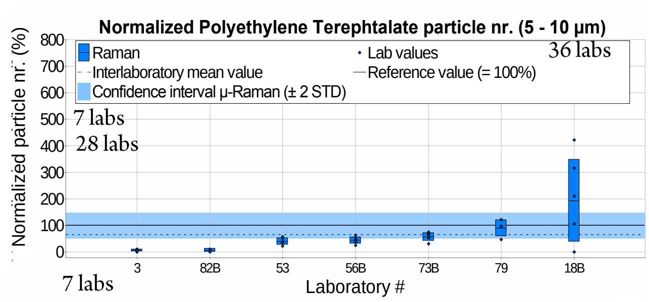
11 labs

Figure S14. Normalized aged PE particle numbers (10 – 50 µm and 20 - 50 µm) per tablet derived from µ-FTIR and LDIR measurements. Red, light green, dark green and orange bars – standard deviations of intra-laboratory results (repeatability).

28 labs

Figure S15. Normalized PET particle numbers (10 – 50 µm and 20 - 50 µm) per tablet derived from µ-FTIR and LDIR measurements. Red, light green, dark green and orange bars – standard deviations of intra-laboratory results (repeatability).

Figure S16. Normalized aged PE particle numbers (5 – 10 µm) per tablet derived from µ-Raman measurements. Blue bars – standard deviations of intra-laboratory results (repeatability).



7 labs

Figure S17. Normalized PET particle numbers (5 – 10 µm) per tablet derived from µ-Raman measurements. Blue bars – standard deviations of intra-laboratory results (repeatability).

Table S6. Main sources of uncertainty

Source	Uncertainty	Comment
Sample homogeneity	High	MP content: RSD particle number = 12-40 %, RSD mass fraction = 18-27%
Tablet mass	Low	Tablet mass variation ±5 mg, corresponding to ±2%
Sample preparation (Filtration)	High	Loss of particles during filtration: assumption of minus 10-30%
Method sensitivity	Low-Middle	Determines whether the smallest particles are counted or not
Instrument calibration	Middle-High	Calibration plays a role in trueness/bias of the measurement result. Not provided by the labs
Spectral library used	Middle	There may be differences in matches between a curated vs laboratory generated library
Measurement repeatability	Middle-High	The repeatability of the results based on 6 samples of the same material
Instrument measurement uncertainty	Low	Measurement uncertainty of the measurand measured with the instrument at best calibration and best settings
Instrumental settings	Middle-High	Software parameter settings, like threshold size, threshold to positively identify spectra by comparison against spectral databases, region of interest, aperture, focus, acquisition time, number of pixels, settings stability during measurement, etc.
Background (laboratory and blank samples)	Low	Presence of MPs even in blank samples (negative control). Estimated <1-6%. Laboratory background contamination not assessed
Operator's effect	Middle-High	Untraceable. Explains how accurate the sample is handled and how is the measurement carried out
Extrapolation of results	Middle-High	Analysing only X% of the filter surface (sometimes as low as just 1% of the filter) and extrapolating the result to 100% could alter the particle number.
Accuracy of the counting software	Middle	When using automated particle counting, exact uncertainty unknown, particularly for agglomerated and/or small particles with complex shapes

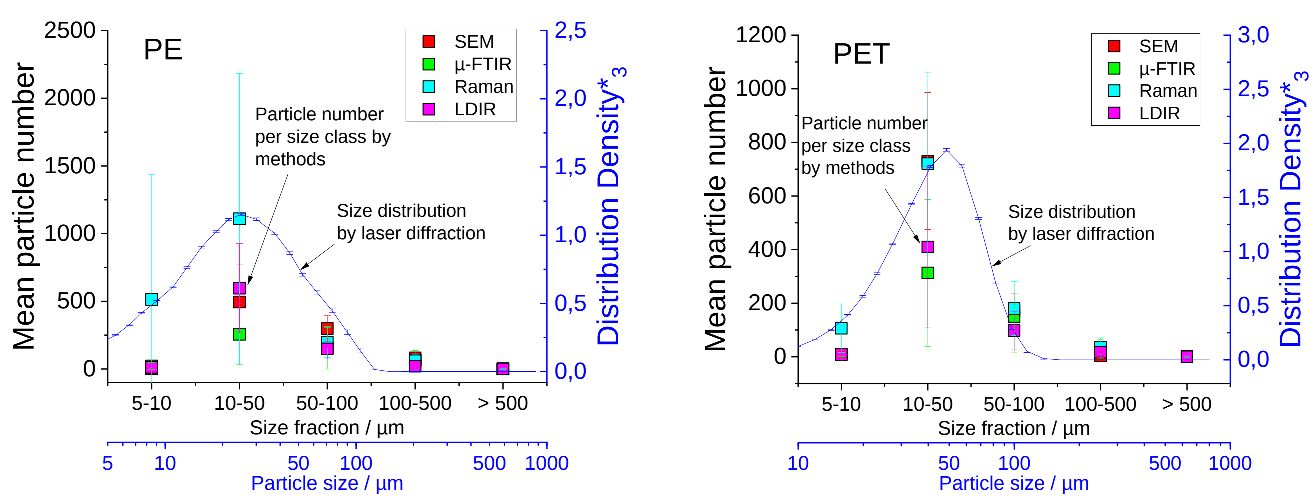


Figure S18. Particle size distribution for aged PE and PET calculated from results derived from SEM, µ-Raman, µ-FTIR, LDIR measurements.

Table S7. Type of particle counting (automated *vs* manual)

Lab Nr.	Method	Automated	Semi-automated	Manually
56	FTIR		YES	
21	FTIR			YES
18	FTIR	YES		
17	FTIR			YES
50	FTIR	YES		
73	FTIR	YES		
3	FTIR	YES		
38	FTIR	YES		
14	FTIR	YES		
82	FTIR	YES		
75	FTIR	YES		
54	FTIR	YES		
47	FTIR	YES		
44	FTIR	YES		
63	FTIR			YES
27	FTIR	YES		
49	FTIR	YES		
84	FTIR	YES		
31	LDIR	YES		
59	LDIR	YES		
72	LDIR	YES		
56	Raman	YES		
18	Raman	YES		
83	Raman	YES		
73	Raman	YES		
3	Raman	YES		
16	Raman			YES
26	Raman	YES		
82	Raman	YES		

Recommendations for best practices:

1. **Blank samples**

We recommend investigating background contamination in the laboratories as lab blank prior to analysis of the microplastic particles. Specific measures should be undertaken to minimize the contribution of such background contamination to ensure as clean working environment as possible.

2. **Filtration of tablets**

The filtration step represents a critical step. We recommend here to use the same filter material and pore size as the reference material provider used for determining the reference value. We recommend placing the tablet directly on the filter and rinsing water over the tablet during filtration. If this is not feasible, we recommend dissolving tablets in warm water with ultra sonic bath as long as necessary, until the tablet is dissolved completely before the solution is applied to the filter. We also recommend rinsing with MilliQ water all the walls of the filtration funnel/beaker etc. to transfer the particles quantitively on the filter. Loaded filters should be dried before measurement.

3. **Instrumental setting**

We noticed that different laboratories used different instrumental settings, probably driven by the experience of individual experts with the own instrument. However, it would be helpful, if all users of the same instrument type (either μ-FTIR or μ-Raman) use the same instrumental settings, to minimize uncertainty contributions derived from different settings. Reference material providers should give detailed setting instructions, e.g. HQI threshold. We recommend use of the same library of spectral lines. This might be discussed in a prior online meeting with participants towards agreement between all the participants. Use of different marker libraries might skew results. We recommend using the same evaluation software; however, the assumption is that the contribution to overall uncertainty due to different software used is rather small.

4. **Instrumental calibration**

We recommend calibration of the instruments with certified reference materials prior to measurements on microplastic samples.

5. **Measurement repeatability**

We recommend measurement of tablets possibly on the same day, independent of method. If the material is to be used as RM for method validation, then we would recommend, for example, always taking 3 measurements for each control measurement, e.g. once a month (or once per week).

6. **Operators' degree of expertise**

We recommend employing most experienced researchers for carrying out experiments. The person carrying out the work should be regularly informed and sensitized about the possibility of contamination.

7. **Extrapolation of results**

This is probably one of the largest sources of the erroneous measurements. We strongly recommend measuring the whole filter and to not extrapolate results. For example, if 1-2% of the filter is measured, assuming a homogeneous distribution of particles on the whole filter and extrapolating the particles number to 100% of the filter surface area might skew results tremendously. If measuring of the 100% area is not feasible, we recommend taking at least 10 smaller areas at different locations, possibly comprising areas with fewer particles and those with higher particle numbers, so that the overall result is as close as possible to the real particle number.

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