Bundesanstalt für Materialforschung und -prüfung

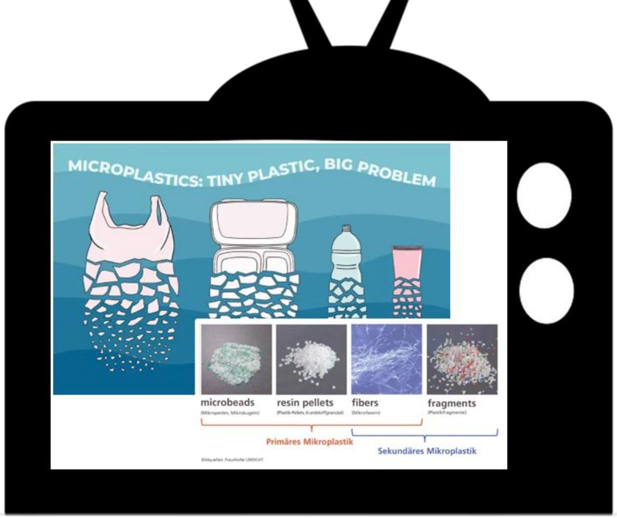
Sicherheit in Technik und Chemie

16th Oct 2025

CHALLENGES IN MICROPLASTICS STANDARDISATION

Korinna Altmann, Dmitri Ciornii, Dan Hodoroaba

Motivation



Nepali researcher says people can consume up to five grams of plastic particles every week

A lecturer at the University of Technology Sydney (UTS) studying the negative health impacts of microplastics claims that people,...

vor 1 Monat

Nature

Bioaccumulation of microplastics in decedent human brains

The majority of MNPs found in tissues consist of PE and appear to be nanoplastic shards or flakes. MNP concentrations in normal decedent brain...

03.02.2025

Source: Fraunhofer UMSICHT; m.malinika – stock.adobe.com; pixabay

Consequences

2018

Regulation for Producers

Regulation on Plastic products

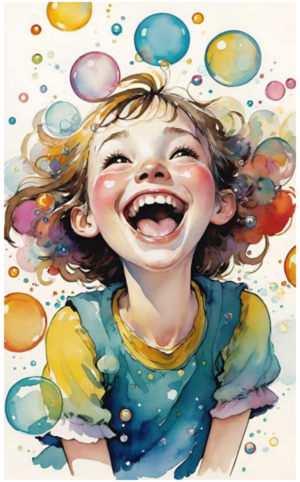
Regulation as a precautionary principle for maintaining health

Solution

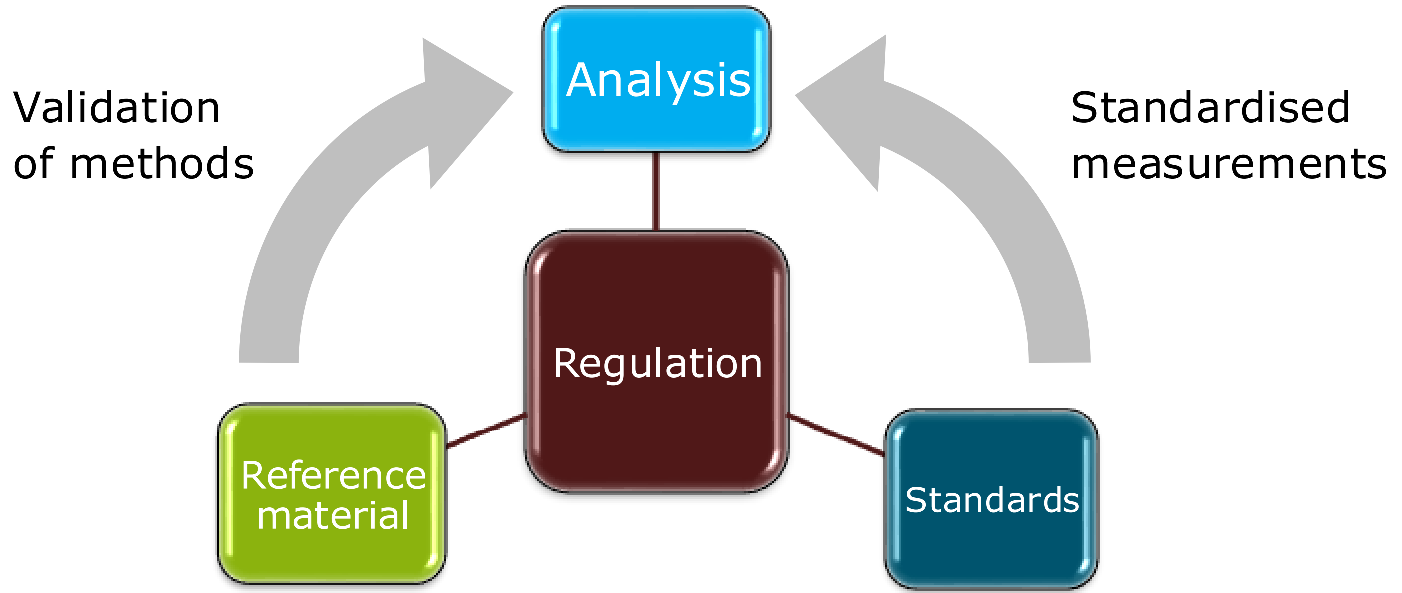
Reliable and Accurate Measurements

What is the true exposure in:

- Air
- Water
- Soil
- Biota
- Human body ?

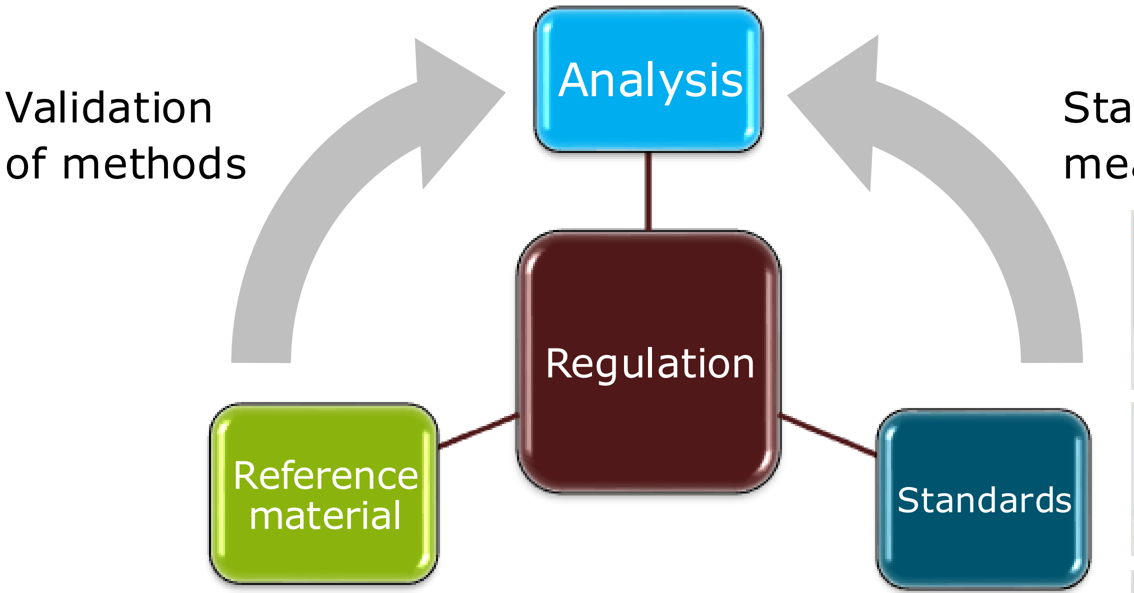


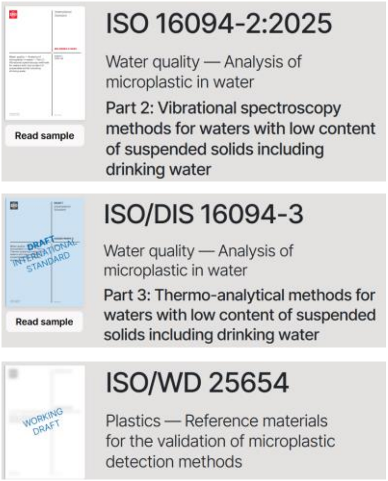
Challenges



Validation of methods
Analysis
Standardised measurements
Regulation
Reference material
Standards

Challenges




ISO 16094-2:2025

Water quality — Analysis of microplastic in water

Part 2: Vibrational spectroscopy methods for waters with low content of suspended solids including drinking water

Read sample

ISO/DIS 16094-3

Water quality — Analysis of microplastic in water

Part 3: Thermo-analytical methods for waters with low content of suspended solids including drinking water

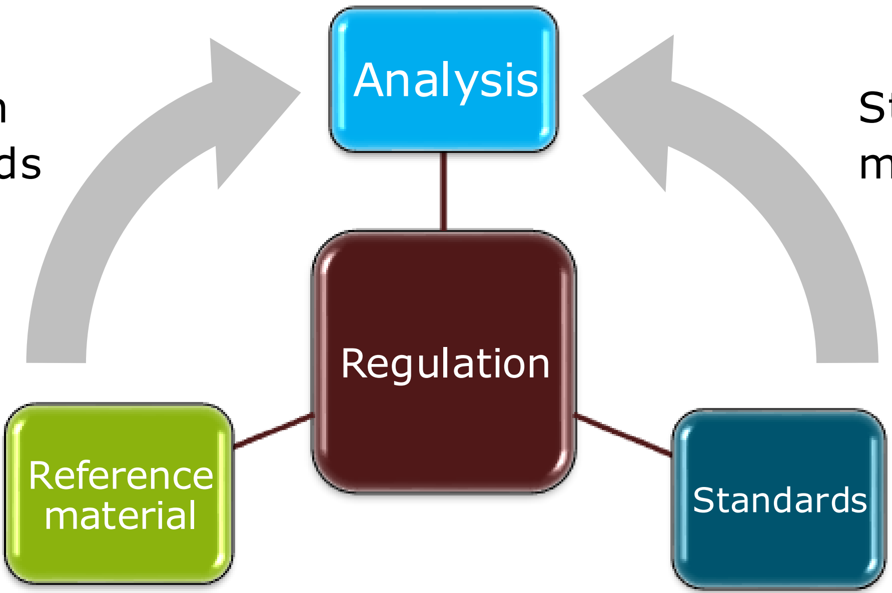
Read sample

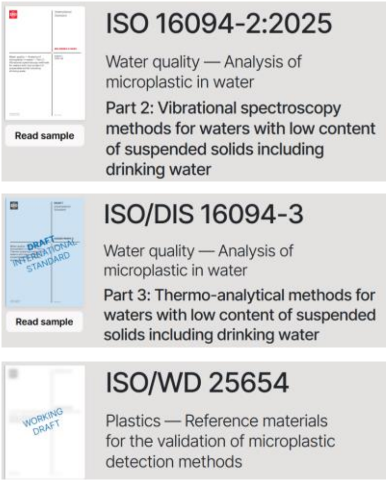
ISO/WD 25654

Plastics — Reference materials for the validation of microplastic detection methods

Challenges

- µ-FTIR, µ-Raman, QCL
- Py-GC-MS, TED-GC/MS

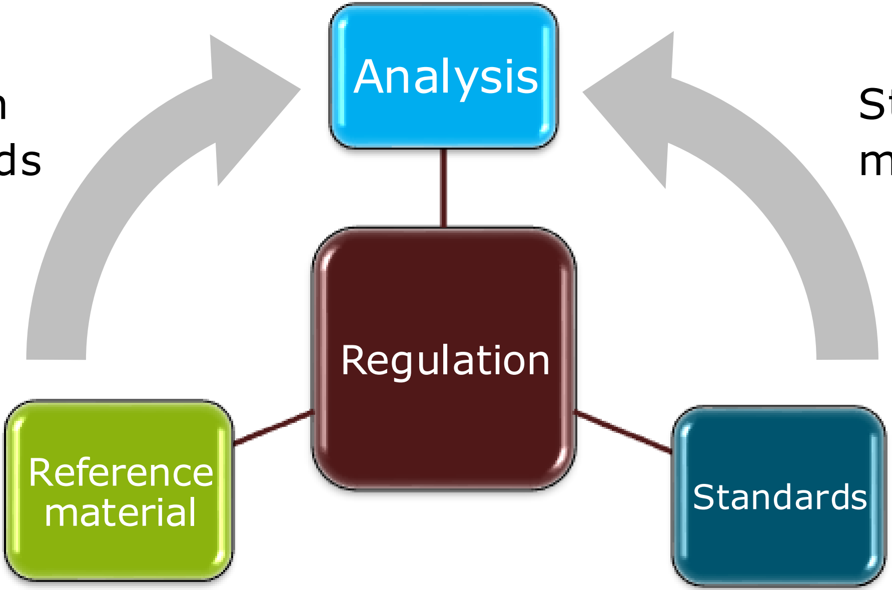




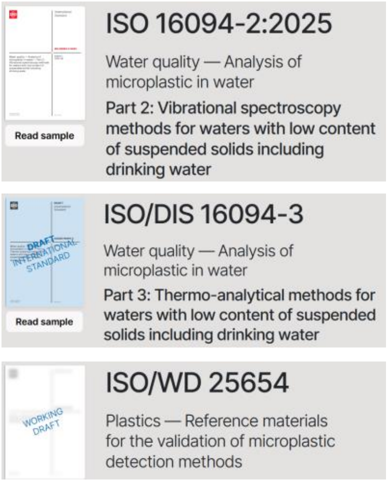
Challenges

- µ-FTIR, µ-Raman, QCL
- Py-GC-MS, TED-GC/MS

Validation of methods



Standardised measurements


ISO 16094-2:2025

Water quality — Analysis of microplastic in water

Part 2: Vibrational spectroscopy methods for waters with low content of suspended solids including drinking water

Read sample

ISO/DIS 16094-3

Water quality — Analysis of microplastic in water

Part 3: Thermo-analytical methods for waters with low content of suspended solids including drinking water

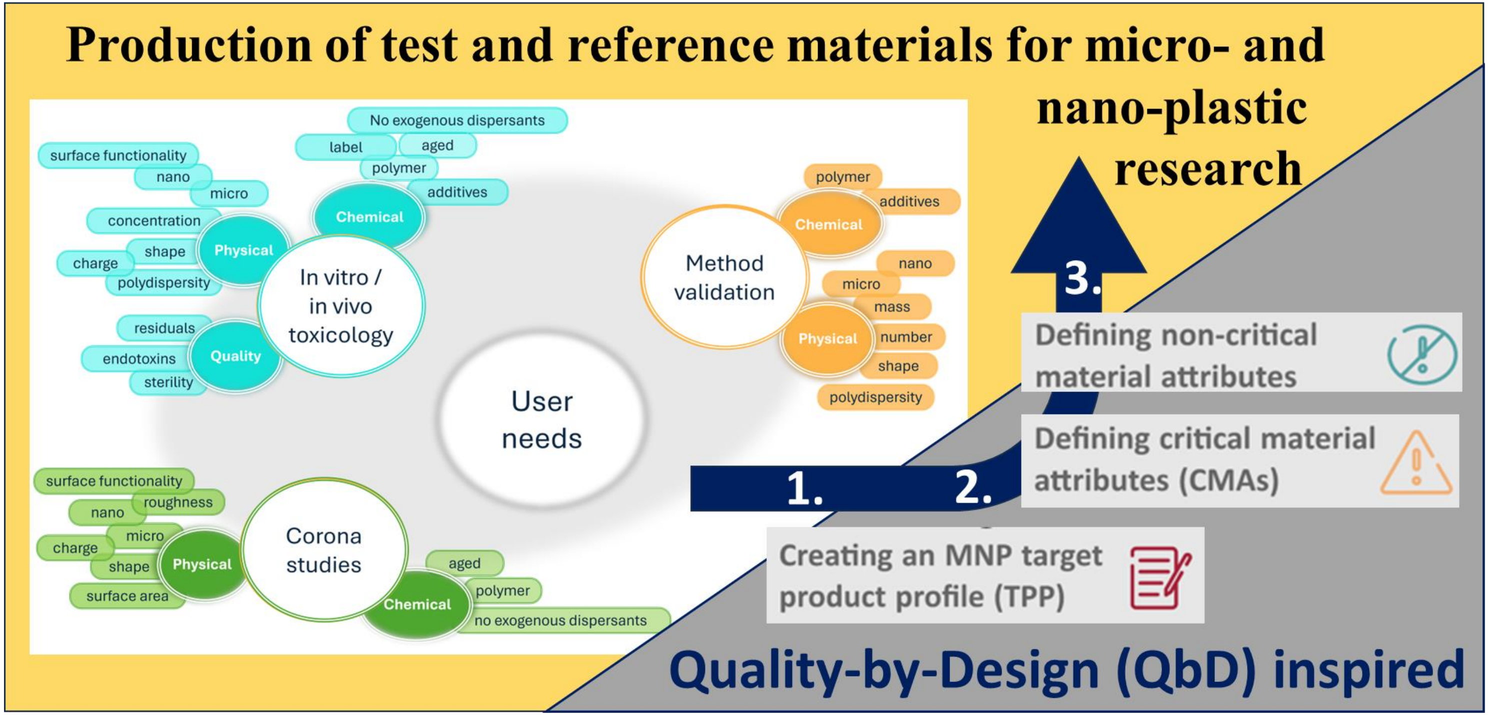
Read sample

ISO/WD 25654

Plastics — Reference materials for the validation of microplastic detection methods

???

User needs



Journal of Hazardous Materials 497 (2025) 139595, https://doi.org/10.1016/j.jhazmat.2025.139595

Intended use

- ➢ Standardized reference materials for **instrument calibration**
- ➢ Standardized reference materials for **instrument development**
- ➢ Standardized reference materials for **sample preparation**

Which instruments?

- µ-IR, µ-Raman = quantification of particle number
- TED-GC/MS and Py-GC/MS = quantification of particle mass

What are mandatory and desired properties?

BAM

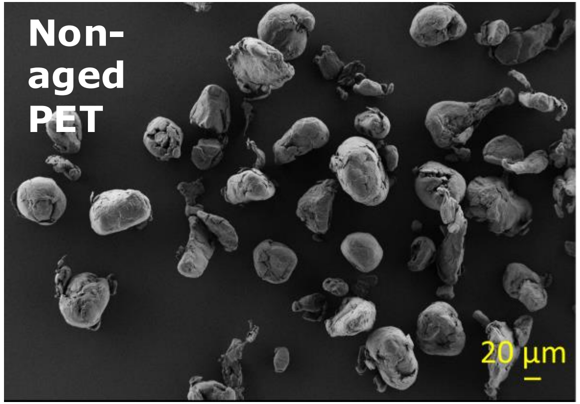
Mandatory attributes
Morphology must reflect MPs in the environment (i.e., **irregular and polydisperse**)
Must contain 50% of particles in the **specified size class** based on laser diffraction
Must not contain exogenous dispersing agents in the product and **<1% residual contaminants**, such as metals from the milling procedure
Each unit from a single batch must conform to **homogeneity and stability** criteria set by ISO Guides 31 and 35

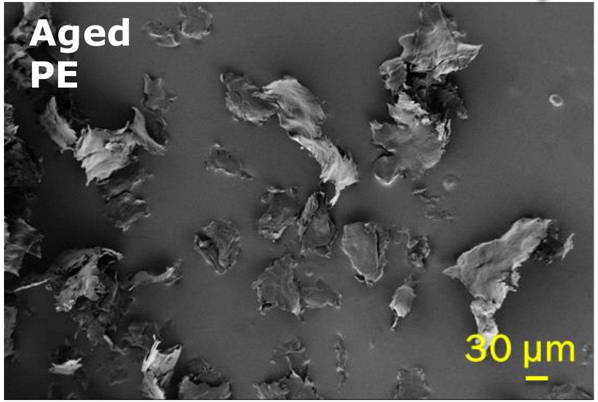
Desired attributes
Pristine test materials should have a **surface chemistry** that reflects the pristine polymer
Aged test materials should have a surface chemistry that reflects environmentally relevant MPs
Should maintain PSD **stability over at least one year** shelf life and in-use life span
Should have an acceptable product **yield** per batch and manufacturing time

Journal of Hazardous Materials 497 (2025) 139595, https://doi.org/10.1016/j.jhazmat.2025.139595

How are they made?

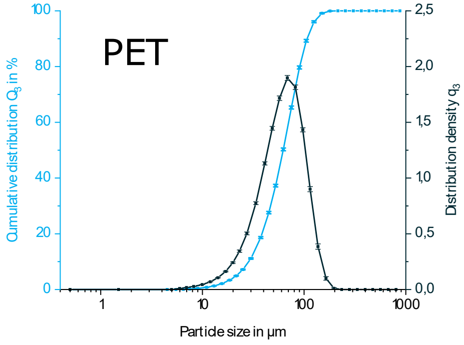
1000 µm 10 µm

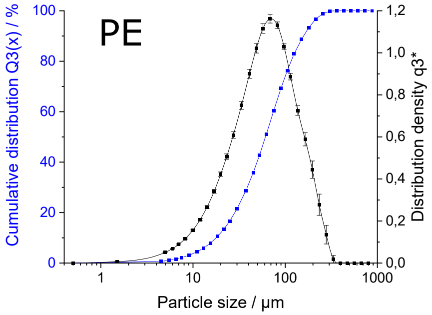




Cryomilling

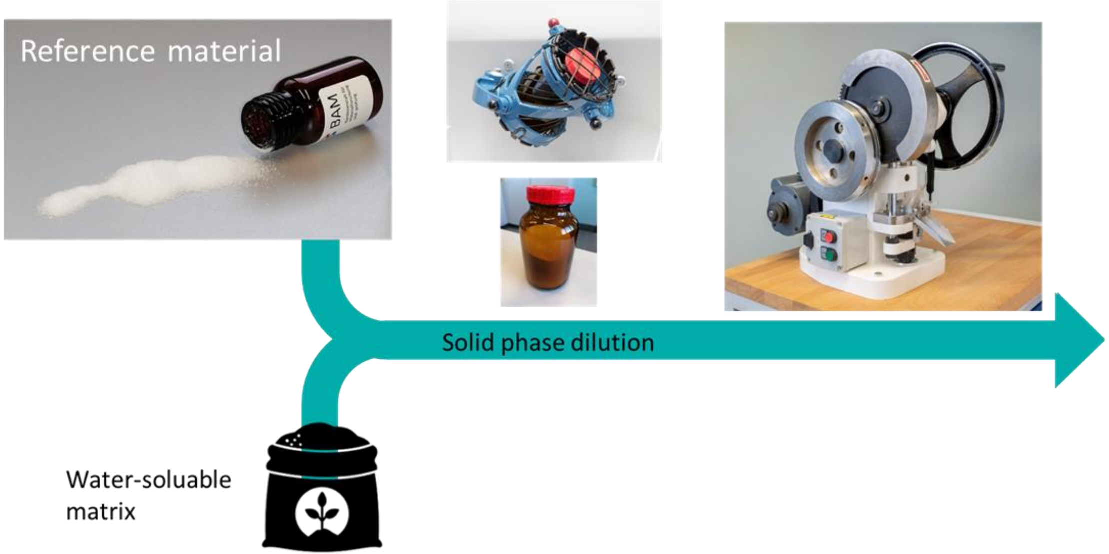
Fragments with irregular shape
Non-aged material slightly rounder and more compact
Size selection via sieving
Yield: g - mg





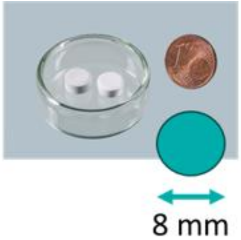
https://webshop.bam.de/
as BAM-P201 and BAM-P206

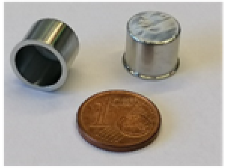
Solid phase dilution and tabletting



Why tablets?

- Easy, uniform handling
- quantitative transport and storage
- very low mass
- particle number (> 10 particles)





How are the tablets used?

Crucible filled with MilliQ water

Wait 4 min

Vacuum filtration

Add 30 mL MilliQ water

Vacuum filtration

Add 120 mL MilliQ water

Vacuum filtration

TED-GC/MS


Fraunhofer

BAM

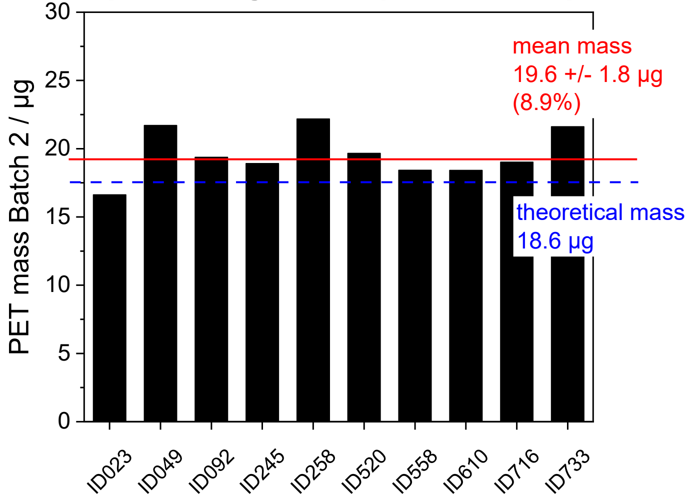
SOP
FILTRATION OF NANO- AND MICROPLASTIC PARTICLES FROM LIQUID SOLUTION

Miclea, P.-T., Wiesner, Y., & Altmann, K. (2025). FILTRATION OF NANO- AND MICROPLASTIC PARTICLES FROM LIQUID SOLUTION (Version v1). Zenodo. https://doi.org/10.5281/zenodo.14850536

14

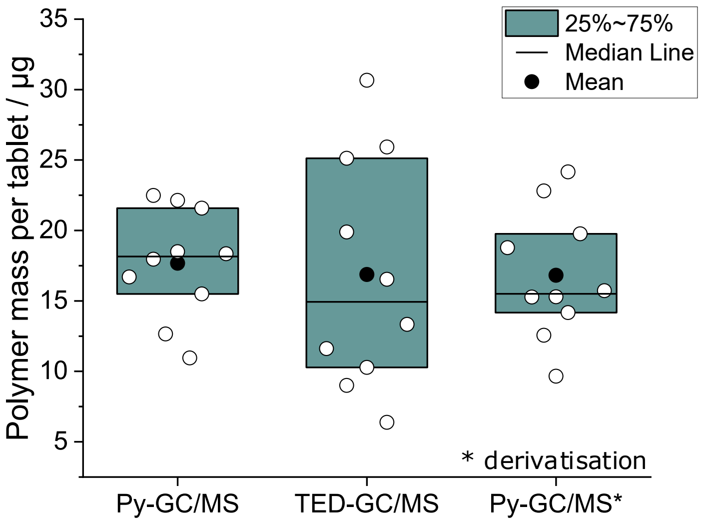
Particle mass of PET in tablet

Thermogravimetric analysis (Homogeneity control)



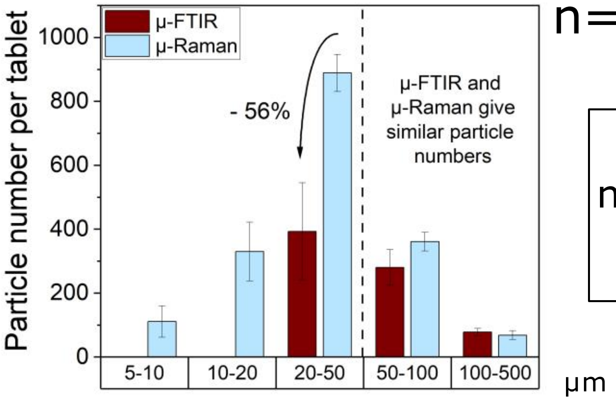
Theoretical: 18.6 µg
Measured: 19.6 +/- 1.8 µg (9.2%)

Py-GC-MS and TED-GC/MS



Theoretical: 18.6 µg
Measured: 16.9 +/- 6.3 µg (37.1%)

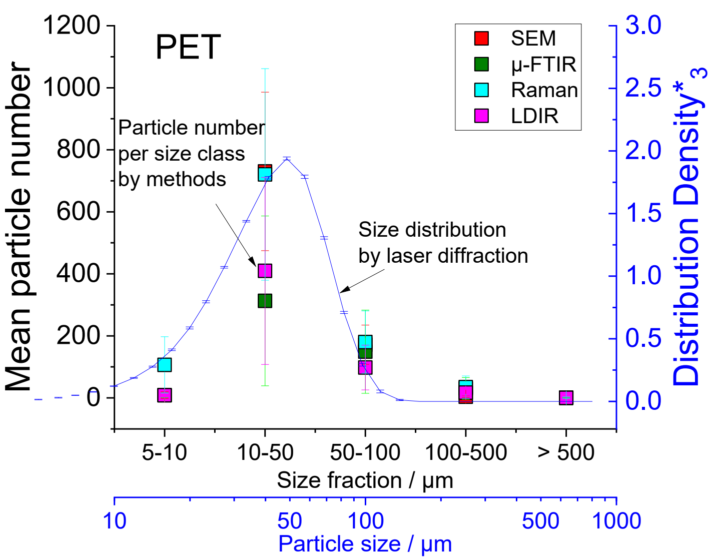
Particle numbers of PET in tablet



n=10

> 50 µm similar numbers for µ-FTIR and µ-Raman

Number vs. Laser diffraction



µ-FTIR

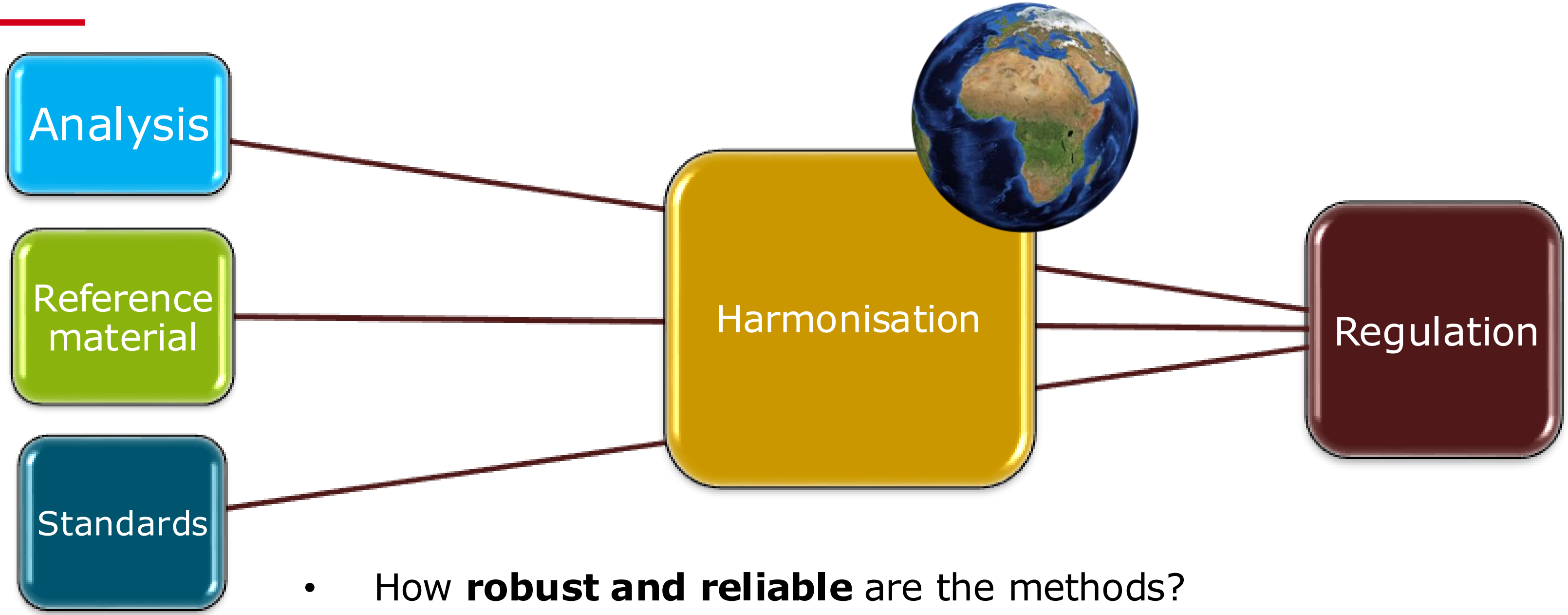
	5-10 µm	10-20 µm	20-50 µm	50-100 µm	100-500 µm	Sum
Mean value	-	-	393	280	78	751
STD	-	-	144	53	10	208
Rel STD / %	-	-	36.7	19.1	13.4	27.7

µ-Raman

	5-10 µm	10-20 µm	20-50 µm	50-100 µm	100-500 µm	Sum
Mean value	163	336	878	331	57	1764
STD	65	60	75	53	22	155
Rel STD / %	39.6	17.9	8.5	15.9	38.7	8.8

µ-Raman similar to laser diffraction

Reliable and Accurate Measurements



- How **robust and reliable** are the methods?
- How **accurate** are the particle numbers and masses determined?
- How **reproducible** are the results?
- Do colleagues measure the same? How **comparable** are our results?

Interlaboratory comparisons

2019 „Plastics in the environment", national

2020 JRC/BAM

2021 -2023 Quasimene/NORMAN
https://doi.org/10.1016/j.scitotenv.2021.145071

2023 -2024 VAMAS TWA45 – microplastics (PlasticsFatE/PlasticTrace)
https://doi.org/10.1021/acs.analchem.4c05403

2025 VAMAS TWA45 – nanoplastics (PlasticTrace) in progress

Interlaboratory comparisons

2019 „Plastics in the environment", national

2020 JRC/BAM

2021 -2023 Quasimene/NORMAN
https://doi.org/10.1016/j.scitotenv.2021.145071

2023 -2024 VAMAS TWA45 – microplastics (PlasticsFatE/PlasticTrace)
https://doi.org/10.1021/acs.analchem.4c05403

2025 VAMAS TWA45 – nanoplastics (PlasticTrace) in progress

Study design


Standard Operating Procedure

SOP – Sample preparation and measurement

TWA 45 – Micro- and Nanoplastics

Collaboration with VAMAS

VAMAS

Versailles Project on Advanced Materials and Standards (VAMAS). http://www.vamas.org/

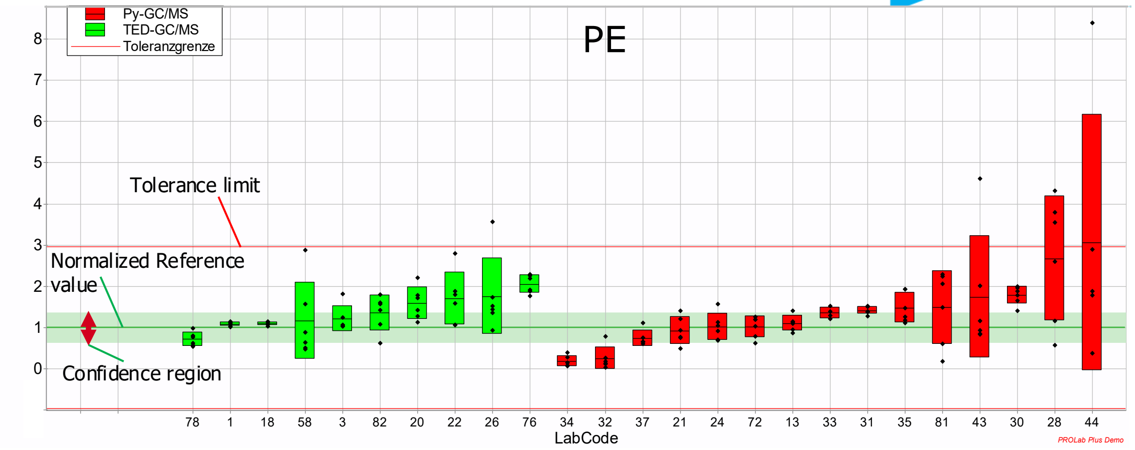
- µ-FTIR, µ-Raman (particle number)
- TED-GC/MS, pyr-GC/MS (particle mass)
- 85 participants all over the world
- Different instruments
- 6 samples (each 2 tablets, PE+PET) + blank samples
- Individual concentrations for vibrational and thermoanalytical methods

Precision and accuracy of plastic particles number and mass of the individual labs and lab-to-lab variation

Mass

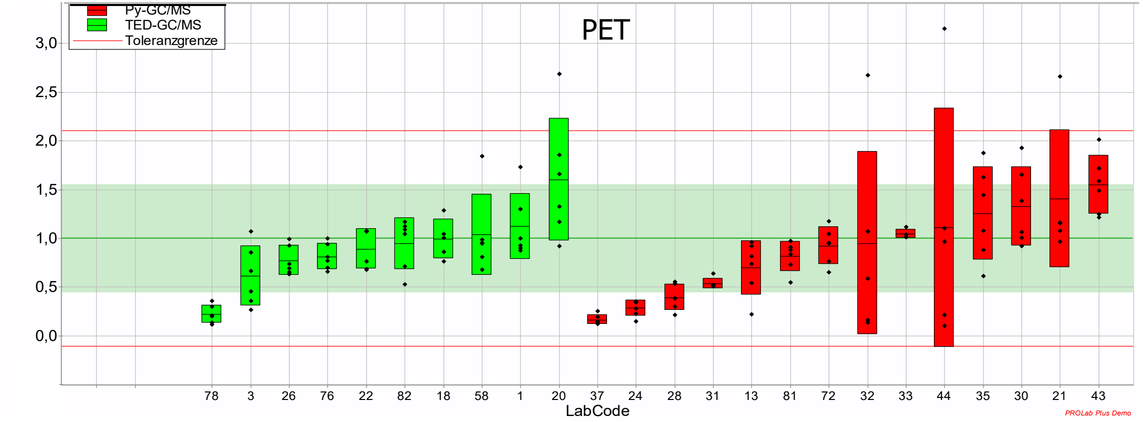
Theoretically (gravimetric) → 1.06 µg/mg

Average mass
TED-GC/MS = 1.13 ± 0.33 µg/mg
Py-GC/MS = 1.11 ± 0.64 µg/mg



Theoretically (gravimetric) → 2.22 µg/mg

Average mass
TED-GC/MS = 1.69 ± 0.67 µg/mg
Py-GC/MS = 1.67 ± 0.80 µg/mg



Higher variation for aged PE film for some labs, but in general lab-to-lab variation is similar for PE and PET

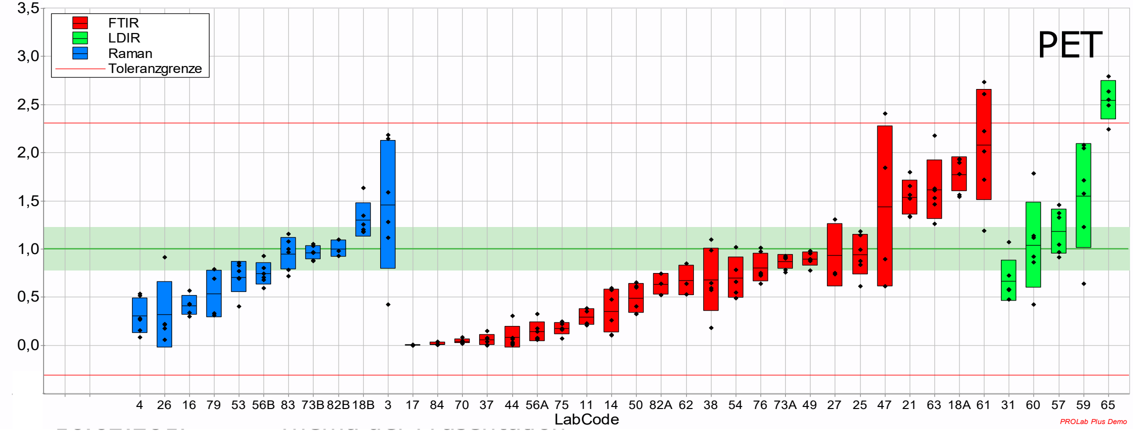
Total particle numbers

Reference value µ-Raman 1557 ±632

Reference value µ-FTIR 336 ± 94

Average number (µ-Raman) =1750+/-1944
Average number (µ-FTIR) = 416+/-372
Average number (LDIR) = 526+/-364

Higher particle number for µ-Raman than for µ-FTIR



Reference value µ-Raman 1295 ±152

Reference value µ-FTIR 550 ±79

Average number (µ-Raman) =1024+/-497
Average number (µ-FTIR) = 408+/-345
Average number (LDIR) = 767+/-393

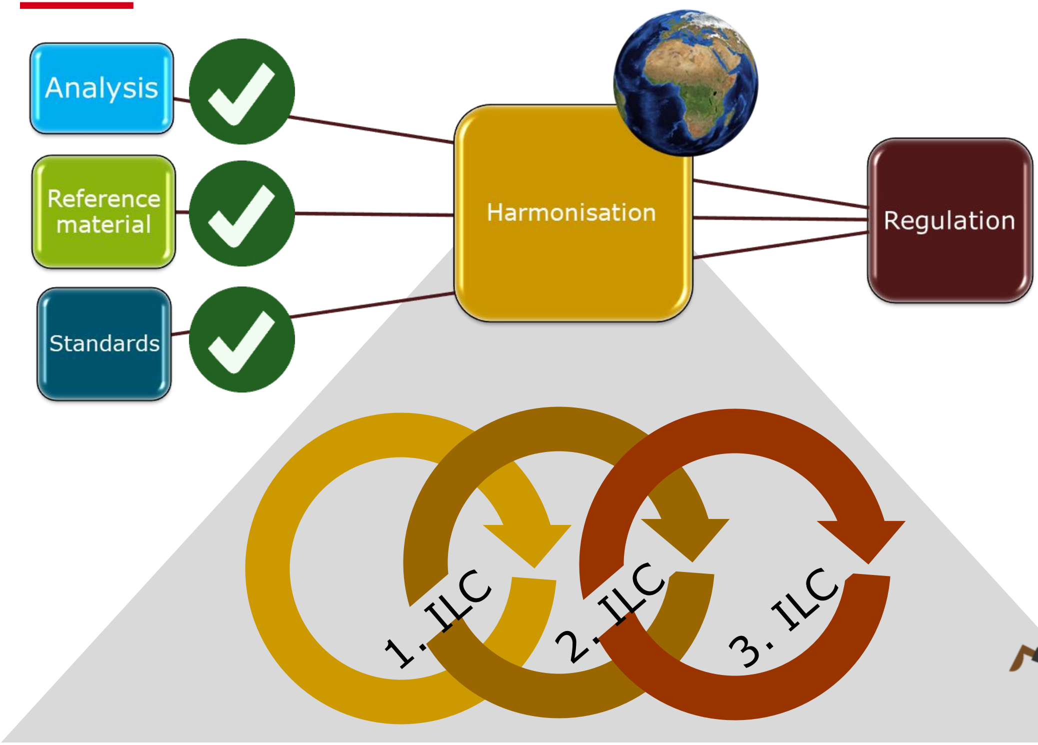
Much more scattering in particle number than in mass

Lessons learnt

Source		Uncertainty	Comment
Test sample	Tablet mass	Low	*Tablet mass variation ± 5 mg (äq. ± 2 %)*
	Background (blank samples)	Low	*Presence of MPs even in blank samples (negative control). Estimated < 1-6 %*
	Sample homogeneity	High	*MP content RSD = 30-40 % (particle Nr), RSD = 16-27% (mass fraction)*
Sample preparation	Filtration	High	*Loss of particles during filtration (assumption: minus 10-30 %).*
Detection	Method sensitivity	Low-Middle	*Determines whether smallest particles are counted or not.*
	Instrument calibration	Middle-High	*Calibration plays a role in trueness/bias of the measurement. Not provided by the labs*
	Measurement repeatability	Middle-High	*The repeatability of the results based on 6 samples of the same material*
	Instrument measurement uncertainty	Low	*Uncertainty of the instrument at best calibration and best settings*
	Instrumental settings	Low-Middle	*Software parameter settings, like region of interest, aperture, focus etc.*
Evaluation	Extrapolation of results	Middle-High	*e.g. measuring only 25% of the sample and extrapolating results to 100%. Could skew the real particle Nr.*
	Precision of the software	Middle	*When using automated particle counting, exact uncertainty unknown*
Operator’s effect	Operator’s effect	Middle-High	*Untraceable. Explains how accurate the sample is handled in the experiment.*

Summary and Outlook

- Reliable and accurate measurements
- We will have answers for the regulators in the future.



Reliable and Accurate Measurements

What is the true exposure in:
- Air
- Water
- Soil
- Biota
- Human body ?

Thank you for your attention.

Funded by the Horizon 2020 Framework Programme of the European Union

Contact:

Dr. Korinna Altmann
Scientist

Phone: +49 30 8104-4305
Email: korinna.altmann@bam.de

www.bam.de