

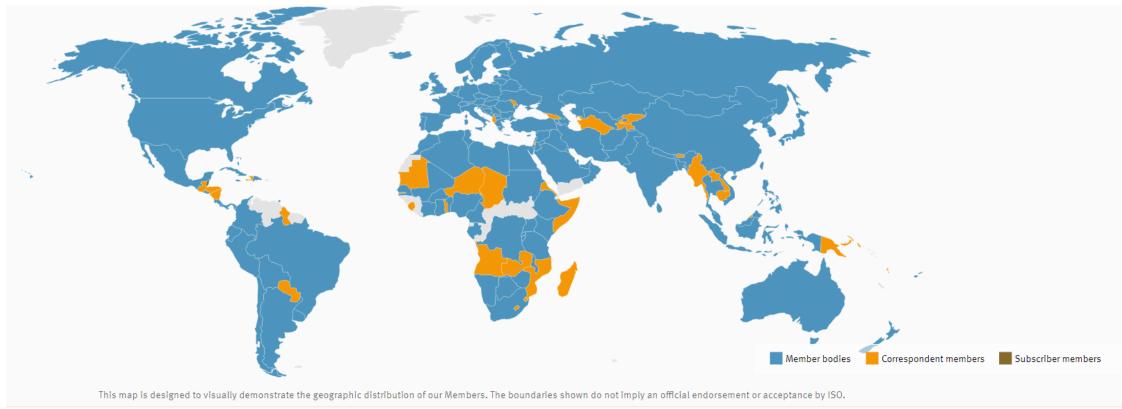
Regulation and Standardisation of Microplastic Analytics

Ulrike Braun, German Environment Agency (UBA)
Nizar Benismail, Nestlé Quality Assurance Center Vittel, France



International Standardisation Organization (ISO)

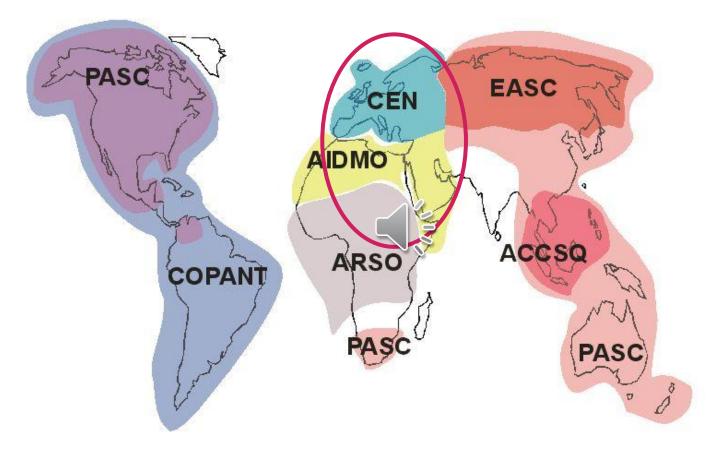
https://www.iso.org/members.html?m=MB



- ISO includes 167 Members
- ISO consists of 810 Technical Committees (TC) and Subcommittees (SC)



Regional Standards Organizations



- CEN (Comité Européen de Normalisation) includes 34 members
- CEN consists of more than 317 Technical Committees (TC)



Vienna Agreement

Vienna Agreement Experts Experts Standards Standards Germany Membership standardization committee afnor ANSI DS **Interested Parties Experts**

France standardization committee

ISO and CEN accept their standardization work to avoid double work



Identification of ISO technical committees

ISO/TC 8	Ships and marine technology		
ISO/TC 20	Aircraft and space vehicles		
ISO/TC 22	Road vehicles	ISO /TC 404	
ISO/TC 23	Tractors and machinery for agriculture and forestry	ISO/TC 181	Safety of toys
ISO/TC 24	Particle characterization including sieving	ISO/TC 188	Small craft
ISO/TC 31	Tyres, rims and valves	ISO/TC 190	Soil quality
ISO/TC 35	Paints and varnishes	ISO/TC 205	Building environment design
ISO/TC 38	Textiles	ISO/TC 207	Environmental management
ISO/TC 39	Machine tools	ISO/TC 216	Footwear
ISO/TC 45	Rubber and rubber products	ISO/TC 217	Cosmetics
ISO/TC 61	Plastics	ISO/TC 219	Floor coverings
ISO/TC 83	Sports and other recreational facilities and equipment	ISO/TC 221	Geosynthetics
ISO/TC 98	Bases for design of structures	ISO/TC 224	Drinking water, wastewater and stormwater systems and services
ISO/TC 122	Packaging	ISO/TC 229	Nanotechnologies
ISO/TC 126	Tobacco and tobacco products	ISO/TC 234	Fisheries and aquaculture
ISO/TC 134	Fertilizers, soil conditioners and beneficial substance	SISO/TC 238	Solid biofuels
ISO/TC 135	Non-destructive testing	100/10200	Pigments, dyestuffs and extenders
ISO/TC 137	Footwear sizing designations and marking systems	ISO/TC 261	Additive manufacturing
ISO/TC 138	Plastics pipes, fittings and valves for the transport of	ISO/TC 262	Risk management
ISO/TC 146	Air quality	150/10 200	Sustainable cities and communities
ISO/TC 147	Water quality	ISO/TC 269	Railway applications
ISO/TC 148	Sewing machines	ISO/TC 270	Plastics and rubber machines
ISO/TC 149	Cycles	ISO/TC 275	Sludge recovery, recycling, treatment and disposal
ISO/TC 150	Implants for surgery	ISO/TC 282	Water reuse
ISO/TC 162	Doors, windows and curtain walling	ISO/TC 297	Waste collection and transportation management
ISO/TC 180	Solar Energy	ISO/TC 300	Solid recovered materials, including solid recovered fuels
		ISO/TC 313	Packaging machinery
		<u>ISO/TC 334</u>	Reference materials



Documents of ISO/TC61/SC14/WG 4

ISO/TC 61/SC 14/WG 4:
Plastics – Environmental aspects –
State of knowledge and
methodologies
CEN ISO/TR 21960: 2020

- => Finalised and available
- => State of scientific knowledge ~ 2018 More than 200 cited articles, 41 pages

ISO/TC 61/SC 14/WG 4: Principles for plastic and microplastic analysis present in the environment ISO/FDIS 24187: 2023

- => Nearly finalised
- => General aspects as basis for specific norms Links to 32 ISO norms, 24 pages



CEN ISO/TR 21960:2020 - First terms and definitions

nanoplastic

plastic particles smaller than 1 µm

Note 1 to entry: According to OECD nanoparticles are up to 100 nm.

microplastic

any solid plastic particle insoluble in water with any dimension between 1 μm and 1 000 μm (=1 mm)

Note 1 to entry: This term relates to plastic materials within the scope of ISO/TC 61. Rubber, fibres, cosmetic means, etc. are not within the scope.

Note 2 to entry: Typically, a microplastic object represents a particle intentionally added to end-user products, such as cosmetic means, coatings, paints, etc. A microplastic object can also result as a fragment of the respective article.

Note 3 to entry: Microplastics may show various shapes.

Note 4 to entry: The defined dimension is related to the longest distance of the particle.

large microplastic

any solid plastic particle insoluble in water with any dimension between 1 mm and 5 mm

Note 1 to entry: *Microplastics* (3.9) may show various shapes.

Note 2 to entry: Typically, a large microplastic object represents an article consisting of plastic or a part of an end-user product or a fragment of the respective article.

Note 3 to entry: Microplastics in this size range are, for example, plastic pellets as intermediates for further down-stream processing such as moulding, extrusion, etc. resulting to semi-finished products which are not final end-user products.

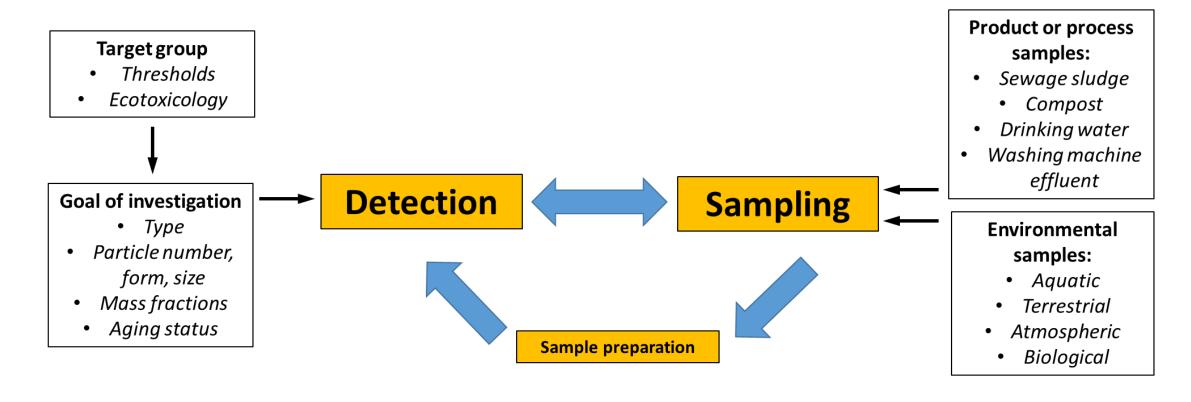
macroplastic

any solid plastic particle or object insoluble in water with any dimension above 5 mm

Note 1 to entry: Typically, a macroplastic object represents an article consisting of plastic or a part of an end-user product or a fragment of the respective article, such as cups, cup covers. Note 2 to entry: The defined dimension is related to the longest distance of the particle.



ISO/FDIS 24187: 2023 - Identification system for suitable analytical tool





Identification of ISO technical committees

ISO/TC 8	Ships and marine technology		
ISO/TC 20	Aircraft and space vehicles		
ISO/TC 22	Road vehicles	ISO /TC 4.04	
ISO/TC 23	Tractors and machinery for agriculture and forestry	ISO/TC 181	Safety of toys
ISO/TC 24	Particle characterization including sieving	ISO/TC 188	Small craft
ISO/TC 31	Tyres, rims and valves	ISO/TC 190	Soil quality
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ISO/TC 147 - SC2 / JWG 1

Title: Plastics (including microplastics) in waters and related matrices

Convenor: Nizar Benismail; Nestle Waters (previous Convenor: C.G. Bannick; German Environment Agency)

Scope: Standardization of methods for the characterization and quantification of plastics including microplastics and related polymers in water ... NOTE: The JWG 1 offers to other interested technical committees to cooperate in the development and application of methods and methodologies elaborated in this group.

Series of Standards: Water quality - Analysis of microplastics -

Part 1: General and sampling

Part 2: Vibrational spectroscopy

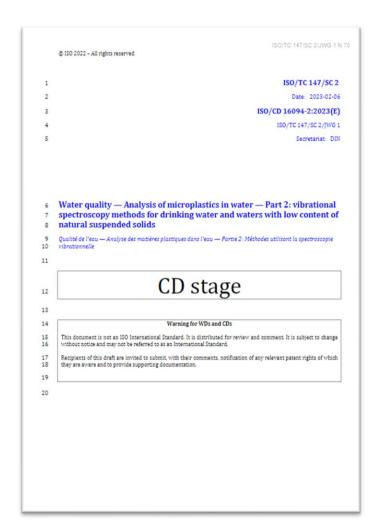
Part 3: Thermoanalytical methods

Part 4: Sample preparation methods

Part 5: Ecotoxicological methods (in preparation)

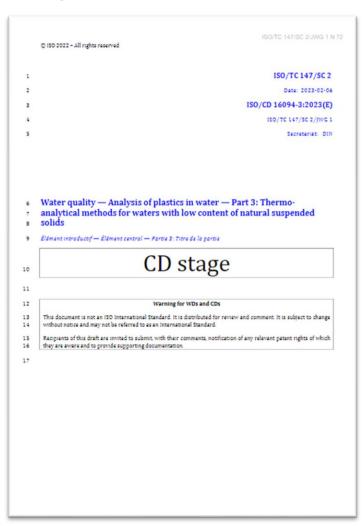


Water quality — Analysis of microplastics -Part 2: Vibrational spectroscopy methods for drinking water and water with low content of natural suspended solids ISO/CD 16094-2: 2023



- analysis of micro prastics by vibrational spectroscopy methods (Raman, micro-InfraRed instruments): generate information about particle size, particle type and particle numbers
- detection of microplastics in waters with low content of natural suspended solids, e.g. drinking water, groundwater or laboratory water.
- very specific technical description for harmonizing all the analytical steps: choices of filters, precautions to minimize cross contamination, calculation of the blank and limit of reporting of the laboratories, statistical models to deliver comparable results, data treatment, interpretation and reporting
- Description of mandatory controls / validation procedures

Water quality — Analysis of microplastics -Part 3: Thermoanalytical methods for waters with low content of natural suspended solids ISO/CD 16094-3: 2023



- general recommendations for the analysis of microplastics by thermoanalytical methods coupled with analysis of decomposition gases: determination of mass fractions and polymer type.
- detection of microplastics in waters with low/moderate content of natural suspended solids.
- general aspects for sample preparation and the application of thermoanalytical methods, and also includes related terms and definitions.
- very detailed technical description for the investigation of water filtrates
 using thermal extraction desorption gas chromatography/mass
 spectrometry (TED-GC/MS) and pyrolysis gas chromatography/mass
 spectrometry (Py-GC/MS) and investigation of isolated particles using CC/MS.

Water quality — Analysis of microplastics – Part 2, 3



Standard	Part 2:		Part 3:		
	Vibrational :	spectroscopy	Thermoana	ls*	
Technique	Raman Infrared micro-spectroscopy spectroscopy		Thermal Extraction Desorption associated with Gas Chromatography - Mass Spectrometry	Pyrolysis associated with Gas Chromatography - Mass Spectrometry	
Sort of Sample	Water filtrate residues				
hemical composition of he polymer Yes					
Information	Functional production				
Results expression	Polymer type number of particles, six and appe		Polymer type, mass		Polymer type
Minimum measurable size of particles	1 μm to 10 μm	15 μm to 20 μm	Undefined	Undefined	Visual identify- cation
Minimum mass subject to measurement after preparation	Undefined		0,1 – 2 μg (absolute)	0,01 – 1 μg (absolute)	
Number of particles examined/measurement	1 to 20 000		Complete mass of particles		

Thermoanalytical methods include all methods, using thermal treatment on sample and subsequent analysis of decomposition roducts by use of gas chromatography - mass spectrometry. Alternative detection methods are also possible and not shown in this able, but also addressed in the document.

- Harmonization of scope, normative references, terms and definitions
- Where possible: harmonization of materials, equipment, lab working conditions, ...
- Next meeting of JWG, including sub groups: 17.4.2023, Rovaniemi (FIN)

All known activities ... challenge in communication

ISO/TC 38 Textiles and CEN/TC 248 Textiles and textile products

- ISO/DIS 5157, Textiles Environmental aspects Vocabulary
- EN ISO 4484-1:2023, Textiles and textile products –
 Microplastics from textile sources Part 1: Determination of material loss from fabrics during washing
- ISO/DIS 4484-2, Textiles and textile products Microplastics from textile sources – Part 2: Qualitative and quantitative evaluation of microplastics
- ISO/FDIS 4484-3, Textiles and textile products –
 Microplastics from textile sources Part 3: Measurement of
 collected material mass released from textile end products
 by domestic washing method

ISO/TC 61 Plastics/SC 14 Environmental aspects and CEN/TC 249 Plastics and

- EN ISO 17422:2018, Plastics Evironmental aspects –
 General guidelines for their inclusion in standards
- EN 17615:2022, Plastics Environmental Aspects Vocabulary

ISO/TC 147 Water quality and CEN/TC 230 Water analysis

ISO/CD 5667-27, Water quality – Sampling – Part 27:
 Sampling for microplastic particles and fibres in water

DIN standardization committee Food

 DIN TS 10068:2022, Food – Determination of microplastics – Analytical methods (Text in English)

CEN/TC 444 Environmental characterization

 Up to 2023 establishment task group on "Microplastics" for the preparation Technical Specification on sampling and sample pretreatment: "Soil quality — Sludge, treated biowaste, soil and waste — Sampling, pretreatment and analysis of microplastics"

... ?



Conclusions

- International standards for the analysis of plastics in environmental matrices are needed (comparability of data).
- A fundamental challenge in the sectoral system of standardization is the handling of overarching horizontal topics.
- First documents are available.
- The primary goal must be to avoid contradictions at the technical level (precision of the same methods) in the standards (CEN/ISO).



Welcome for participation in JWG!

Thank you for listening!

