eurofins | Environment Testing

Microplastics and rubber particles

All you need to know

ABOUT MICROPLASTICS

What are microplastics?

Microplastics - plastic particles with a size range of 1 µm to 5 mm - are nowadays commonly found in various environmental elements. Microplastics can be classified as primary or secondary microplastics. This is based on whether the microscopic particles are manufactured (primary) or whether they are the result of environmentally occurring fragmentation processes (secondary).

Risks associated with microplastics

Microplastics, generated from different artificial polymers are poorly biodegradable in the environment, however through mechanical, chemical or UV-induced fragmentation they can enter several environmental compartments such as surface waters and soils. These persistent particles pose significant ecological and health concerns: can be ingested and accumulated by different organisms, possibly affecting human health and constituting an almost unimaginable risk to our future. Further to their physical occurrence their capability to accumulate and transport toxic chemicals and pathogens is considerable.

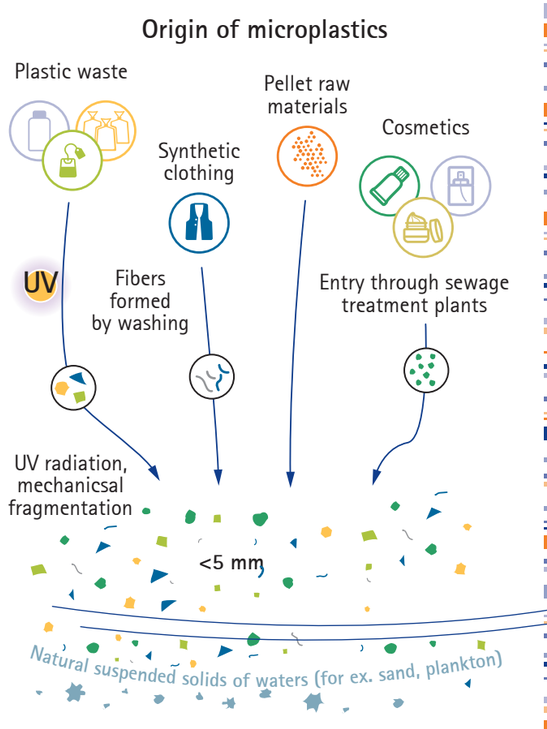
From food packaging to household products and industrial components, plastics are ubiquitous in everyday life. As a result, their increasing prevalence in the environment is becoming a global concern.

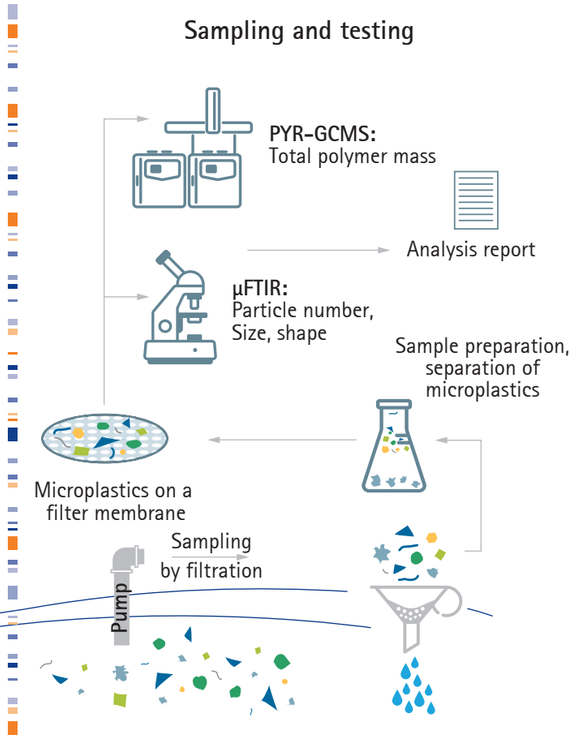
About Eurofins

Eurofins Environment Testing Hungary is part of Eurofins Scientific and your partner for environmental testing. Our goal is to help you achieve your objectives with the support of our efficient and qualitative analysis techniques. Our customer service stands at your disposal with specialized knowledge and extensive experience.

Sources and affected industries

There are numerous sources of microplastics because plastics are widely used in our everyday life and in different industries. Households may release microplastics in wastewater with the washing of synthetic clothing, abrasion of household products, paints and primary microplastics originating from rinse-off cosmetics and glitters. This may be a source for not only microplastics, but tire and road wear particles (TRWP) as well. As microplastics are present in the air as well, bunch of industries may be affected with the issue either during their production processes, wastes or the product itself. Because of the numerous sources and the various industrial processes and environmental compartments affected, microplastic testing are relevant for a wide range of stakeholders, not only actors in the water and environmental industry, but the chemical-, food- and light industry as well.





MICROPLASTIC ANALYSIS TECHNIQUES

Sample preparation

The analysis of microplastics is particularly challenging. One reason is that no single automated method currently covers the entire size range with the information needed, such as particle numbers, size, morphology, mass and polymer type. All analysis need a proper sample preparation, a set of separation techniques to enrich and extract the microplastic particles. This varies depending on the sample matrix type. Generally, inorganic material is removed after flotation, degradation of organic material is performed by enzymatic treatment and oxidation before the final filtration. The more complex the sample matrix, the more sample preparation steps are needed. Complexity of matrix limits the sample mass/volume that can be prepared for analysis.

Analysis

To obtain a proper chemical analysis and characterisation of microplastics two main techniques are used:

Vibrational spectroscopy

Multiple vibrational spectroscopy methods such as

- FTIR (Fourier-transformed infrared),
- Raman or
- LDIR (laser direct infrared)

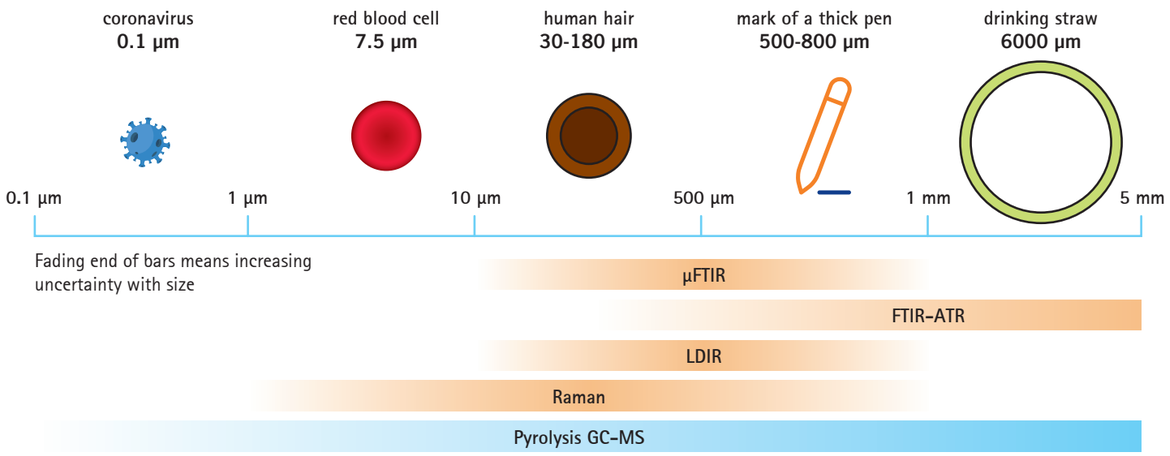
microscopy can provide appropriate information on microplastic particle numbers, chemical identity and morphological parameters (dimensions, shape). All the spectra obtained from the sample will be compared and confirmed with established plastics spectra libraries for identification and counted for abundance analysis.

Thermoanalytical methods

Thermoanalytical methods such as

- PYR-GCMS (pyrolysis gas chromatography-mass spectrometry),
- TED-GCMS (thermal extraction-desorption gas chromatography-mass spectrometry),

can provide information on mass concentration of the polymers. Samples are exposed to high temperatures in the absence of oxygen (thermal extraction, i.e. pyrolysis) to break down the polymer into signature organic compounds then introduced to the GCMS system. Here quantitative analysis is based on selected characteristic compounds and their ions for the selected polymers.



The world how our colleageus see this.
Environmental issues from laboratory perspective – Eurofins Foto Competition 2023

A PET bottle on Lake Tisza from the perspective of a yellow floating heart
Photo: Bence Prikler I Award: 1st place

WHAT WE OFFER

Accredited analysis for several matrices and polymer types with both techniques

MATRICES ANALYSED

Water samples
drinking water, bottled water, groundwater, surface water, treated wastewater, process water, raw wastewater

Solid samples
soil, sludge, sand, sediment, compost

Air
indoor, environmental, workplace

Cosmetics*

Food*
salt, honey, soft drinks, milk

*not accredited

POLYMERS ANALYSED

Polymer types analysed	FTIR	PYR-GCMS
PE | Polyethylene	•	•
PP | Polypropylene	•	•
PET | Poly(ethylene-terephthalate)	•	•
PA | Polyamide	•	•
PMMA | Poly(methyl methacrylate)	•	•
PVC | Poly(vinyl-chloride)	•	•
PU | Polyurethane	•	
PS | Polystyrene	•	•
PC | Polycarbonate	•	•
POM | Polyoximethylene	•	
PLA | Polylactic acid	•	
ABS | Acrylonitrile-butadiene-styrol		•
PIP | Polyisoprene (natural rubber)		•
BR+SBR | Butadiene rubber + styrol-butadiene rubber		•
Other polymers on request*	•	•

Speciality services: microplastic sampling

MP sampling can be challenging. During the past years we have developed an on-site pressurised cascade filtration system designed for microplastic sampling in drinking water plants, surface waters and treated wastewaters. This helps to concentrate up to 1000 L water before analysis, that improves the confidentiality of the results.

The plastic free system can take samples down to 20 um particle size and as such is necessary to comply with regulation on the field of drinking water.

What results do you get?

FTIR:
- Total microplastic number
- MP number per polymer type
- Results in size classes
- Particle shape (fibre, fragment)

PYR-GCMS:
- Total mass of polymers
- Mass per polymer type

Your benefit

The results of our investigation will support your organisation to get comprehensive knowledge on microplastics and comply with relevant standards and regulations to take the appropriate decisions.

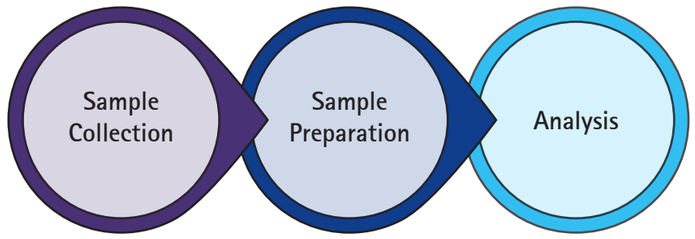
Sample containers and necessary sample volume/mass

Water samples	2x1 L in glass jars, covered with aluminium foil under the plastic lid
Solid samples	200 g in glass jars, covered with aluminium foil under the plastic lid
Air (indoor, environmental, workplace)	Sample in glass container, covered with aluminium foil under the plastic lid (as per air sampling method, e.g. on filter or liquid suspension)
Prepared samples directly for analysis	as per result of sample preparation (e.g. small volume vials, filters in glass Petri-dishes)
Consumer products	3-5 individual packed product per sample

General turnaround time is 4 weeks

OUR ANALYTICAL METHODS: FTIR MICROSCOPY AND PYROLYSIS GC-MS

With our thorough experience in different sample preparation techniques and with our state-of-the-art instrumentation we can support you to obtain all of the most necessary information you might need in microplastic analysis. With our instruments we can determine both the number and the mass ratio of microplastics.



We provide you **accredited analysis** with both µFTIR and PYR-GCMS for a wide range of matrices. These two techniques are complementary, their parallel use can provide you the most information on MPs in the samples.

Our **FTIR microscopes**, also referred to as µFTIR systems are infrared spectroscopy imaging systems used at our laboratory in Budapest. These instruments makes it possible to count the microplastic particles in the samples, identify their material types, shapes and measure their sizes, down to a size of 10 µm. The imaging system enables to cover a large area during analysis, so the whole sample after sample preparation can be analysed, that enables to avoid subsampling-driven biases that might occur in microplastic analysis. This is important because of the significant versatility of the MP particles and their heterogeneous distribution in the sample matrix. Our FTIR-ATR devices are suitable for examining larger pieces of waste, packaging materials and even surface contaminants. FTIR techniques are non-destructive, so the samples can be further investigated with other methods, e.g., PYR-GCMS.

µFTIR results

- **10–1000 µm size range**
- Polymer type
- Particle number
- Particle size
- Shape (fiber, fragment)

FTIR-ATR results

- (500) **1000–5000 µm size range**
- Polymer type
- Particle number

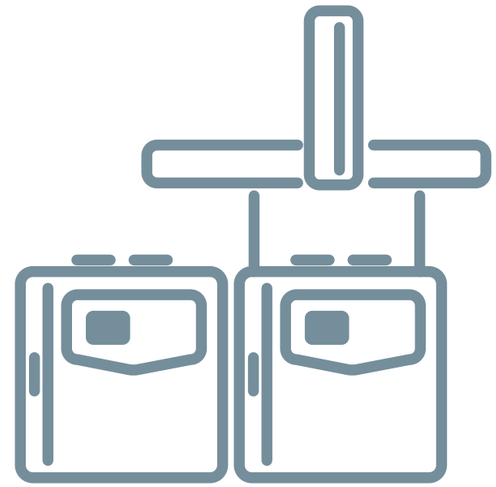
Our **PYR-GC-MS device**, a pyrolysis–gas chromatography–mass spectrometry instrument enables us to identify the various polymer types and their total masses in the samples. One advantage of this method is that it operates without a lower limit regarding particle size so we can use it for the analysis of particles smaller than 10 µm as well. Although it does not enable us to obtain a particle count, but it provides exact information on the total mass of the particles.
Earlier, in the developing field of microplastics, FTIR analysis was used more frequently then thermoanalytical methods. FTIR usually can provide information on larger variety of polymers due to the broad reference spectra libraries available. However, the use of PYR-GCMS is more and more common, as it tackles smaller size ranges and also those dark, black particles, that are not possible to be analysed by FTIR, e.g. tire and road wear particles (TRWP).

Tire and road wear particles (TRWP)

These tiny particles are considered nowadays as a significant source of environmentally occurring microparticles and can be analysed by PYR-GCMS.

PYR-GCMS results

- 1-1000 (5000) µm size range
- Polymer type
- Total polymer mass

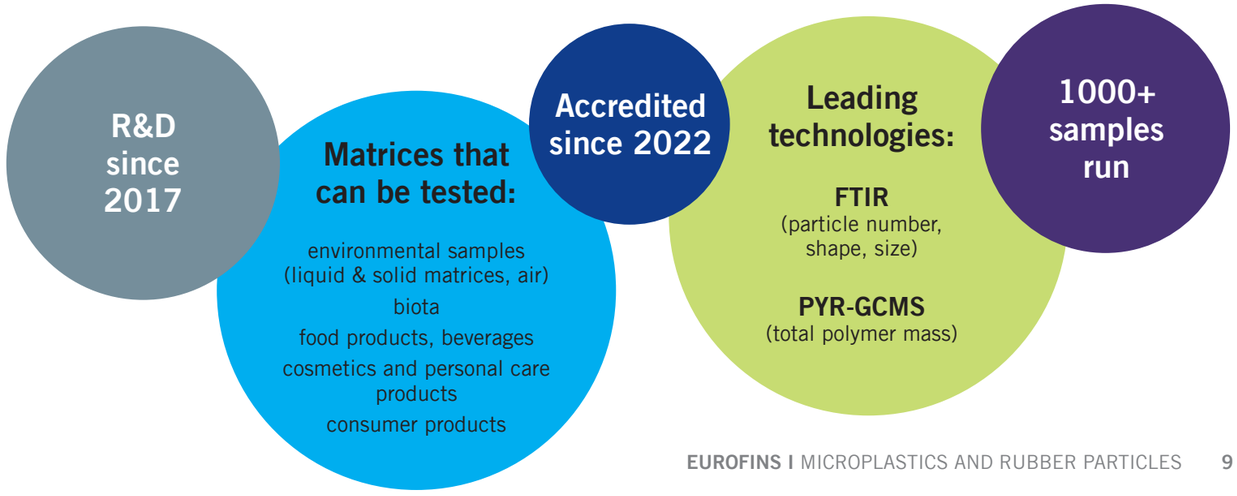


Quality assurance

To minimize sample contamination, standard laboratory precautions are implemented. Cotton lab coats are worn and sample preparation is conducted under a laminar flow hood to reduce airborne contamination. Deionized water used for washing and working solutions are pre-filtered through 0.7 µm glass fiber filters. Regular laboratory blanks are analysed to monitor and maintain high-standard working conditions.

Our instrumentation:

- Bruker Lumos II FPA imaging FTIR microscope with ATR
- Thermo Nicolet iN10MX imaging FTIR microscope with ATR
- ThermoFischer Trace 1610 gas chromatograph coupled with an ISQ7610 mass spectrometer and Frontier EGA/PY-3030D pyrolyzer



OUR ANALYTICAL WORKFLOW

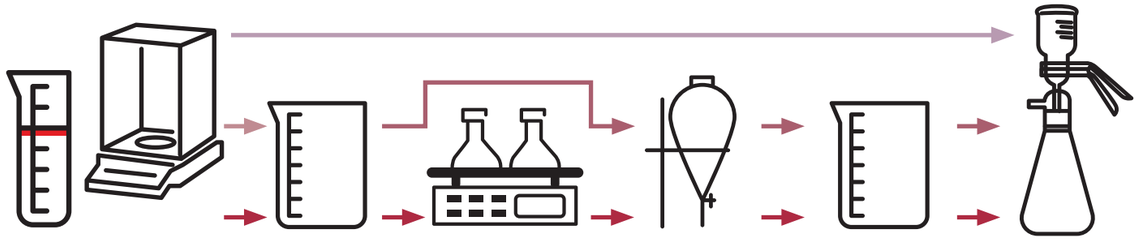
Sample type

Liquids with **low suspended solid** content
(drinking water, bottled water, groundwater, soft drinks

Liquids with **medium suspended solid** content
(surface water, treated wastewater, process water)

Liquids with **high suspended solid content** (raw wastewater)
Solid samples (soil, sludge, sand, sediment, compost)

Sample preparation

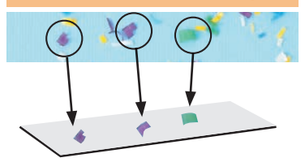


Weighting / volume measurement | Pre-oxidation | Enzymatic treatment | Density separation | Fenton-oxidation | Final filtration

Sample after preparation steps

FTIR

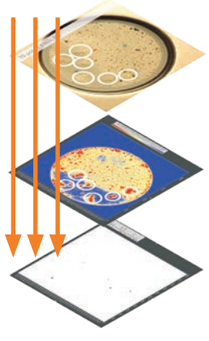
µFTIR (10–1000 µm)



FTIR-ATR (1000–5000 µm)

FTIR microscope

IR beam (transmission analysis)



Visual image of the sample

Absorbance map after FTIR imaging of the whole sample

FTIR results compared to polymer spectra database to map microplastics

Report: chemical identity., size, shape, number/type

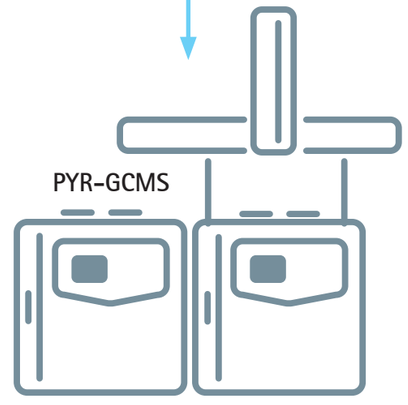
Total number of particles

Chemical identification on a subsample

Report:
chemical identity

PYR-GCMS

Homogenisation (CryoMill) or subsampling



PYR-GCMS

Report:
chemical identity, total mass/type

REGULATION IN THE EUROPEAN UNION AND BEYOND

We help you to comply with the regulatory testing needs with our analysis methods that are based on available and in-development standards such as own validated methods.

2019

Single Use Plastic (SUP) directive (EU 2019/904)

Ban on certain single-use products, that are wholly or partly made of plastic like ear buds and ballon sticks, cutlery, plates, straws, stirrers and food or liquid containers made of expanded polystyrene.

Our FTIR and PYR-GCMS analysis provide information to investigate if your product is plastic free.

2020

EU 2020/2184 Drinking Water Directive (DWD)

Tackling pollutants of concern, such microplastics. Microplastics should be monitored on a watch list between 2024-2029 to carry out a risk assessment.

2021

California Senate Bill No.1422 first in world requiring MP testing in drinking water

2022

Restriction on primary microplastics in products under REACH regulation (EU 2023/2055)

Prohibits the placing on the market of synthetic polymer microparticles (SMP) when placed on the market on their own or in mixtures if they are present in a concentration of at least 0.01% by weight in the mixture (product).

Synthetic Polymer Microparticles (SMP):
– particles between 0,1 µm and 5 mm in all dimensions
– fibre-like particles between 0,3 µm and 15 mm in length

With PYR-GCMS we analyse the compliance of your product for SMPs

2023

(EU) 2024/1441 Commission Delegated Decision supplementing EU DWD

Describes a methodology to measure microplastics in drinking water in a harmonised way:

- **sampling** protocol of 1000 L water
- analysis of **10 prority polymers** with vibrational spectroscopy (e.g. **FTIR**)
- reporting per **size categories, fibres and fragments** separately

strict **QA/QC** policy

Our water sampling methods and FTIR analysis complies the regulation (EU) 2024/1441

Directive EU 2024/3019 concerning urban wastewater treatment

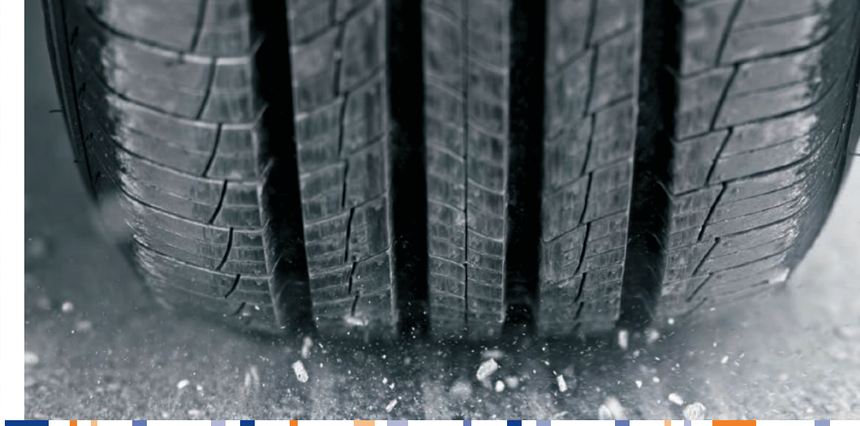
Microplastics should be systematically monitored

- when **sludge** is reused in agriculture
- in **storm water overflow** discharges and in discharges of **urban runoff** from separate systems
- at the **inlets and outlets** of urban wastewater treatment plants at agglomerations of 10 000 p.e. and above.

We help you with our expertise to analyse microplastic with FTIR and/or PYR-GCMS in wastewater and sludge.

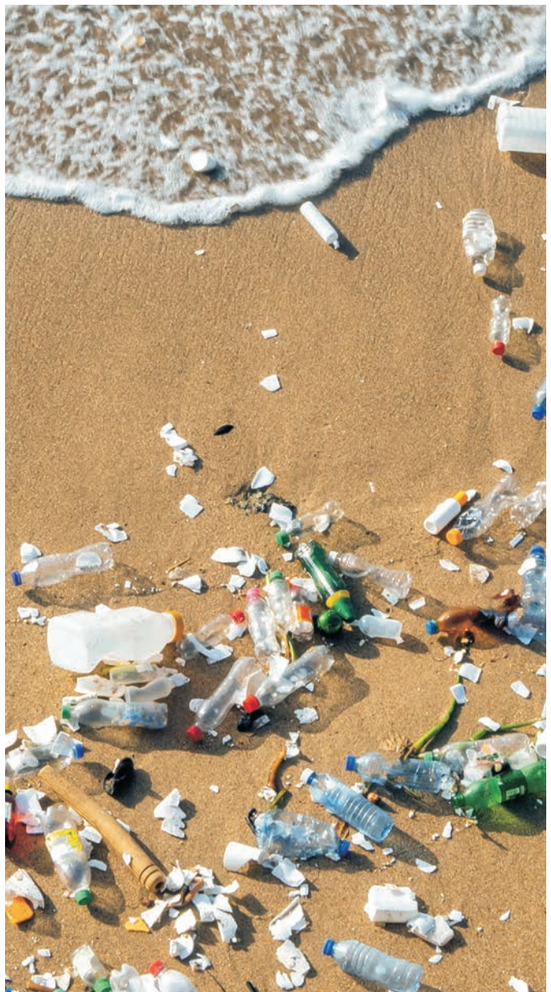
2024

Check out more details on regulations for microplastics here



FROM R&D TO STANDARDISATION

Microplastic research has started in the early 2000s and intensively developing since the mid 2010s. As a completely new topic, different research groups applied different methodologies in sampling and analysis. This resulted in, that the datasets published were hardly comparable and as such, could not support accurate evaluation of the presence of microplastics to investigate weather these particles having harmful effects and posing environmental, ecological and human health risks. Harmonised data and these evaluations are needed to support decision making, policy and legislation. This need was strongly identified and in the past years MP research are strongly focusing on harmonisation and to establish appropriate quality assurance and quality control measure.



At Eurofins Environment Testing Hungary, we have led and participated in several national, regional and EU wide projects that helped us to gather experience and knowledge, develop appropriate sampling and analysis techniques for MPs.

As a result, on one hand our experts have released significant publications in high-impact journals* and became a well-respected part of the scientific scene. On the other hand, these developments help us to provide adequate services to our customers: the development is continuous, further to R&D activities our experts are involved in the work of standardisation committees on this field to improve the framework of microplastics sampling and analysis generally.

Check out our publication list

Current and future standards for MP analysis

Different standardisation bodies, such as ISO, CEN and ASTM are releasing standards defining not only principles and guidance, but microplastics sampling and analysis from different matrices in details. Among available standards, multiple documents are under development to cover this broad topic.

Analysis of microplastic in **water**	ISO/CD 16094-1 ISO/DIS 16094-2 ISO/DIS 16094-3
Sampling for microplastics	ISO/DIS 5667-27
Microplastics from **textile** sources	ISO 4484-1:2023
Environmental characterization of solid **matrices**, task group microplastics	CEN/TC 444/WG 6

Standards to analyse TRWP

Multiple ISO norms are available at the moment that are dealing with tire and road wear particles (TRWP) and rubbers. These particles can be analysed with our PYR-GCMS method.

Ambient air — Determination of the mass concentration of tire and road wear particles (TRWP) — Pyrolysis-GC-MS method	ISO/TS 20593:2017
Rubber — Determination of mass concentration of tire and road wear particles (TRWP) in **soil and sediments** — Pyrolysis-GC/MS method	ISO/TS 21396:2017
Rubber — **Identification of polymers** — Pyrolytic gas-chromatographic method using mass-spectrometric detection	ISO 17257:2020

Your benefit

Our microplastic laboratory is using not only released standards, but our experts are actively participating in standardisation initiatives, ensuring access to lates developments on the field.

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QUESTIONS AND SHIPMENT

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