Publishable Summary for 21GRD07 PlasticTrace
Metrological traceability of measurement data from nano- to small-microplastics for a greener environment and food safety

Overview

PlasticTrace aims to address the urgent need for development and harmonisation of methods for the chemical identification, physical characterisation and quantification of released small micro/nanoplastics (SMPs/NPs) in drinking water, food and environmental matrices, as required by the EU's Circular Economy Action Plan (CEAP). In this context, hyphenated and complementary analytical approaches will be developed, optimised, compared and harmonised, leading to the establishment of metrological traceability of measurements through robust validation studies. Novel and environmentally relevant SMP/NP reference materials will be developed within the project. International cooperation with key stakeholders globally will be considered as the basis for a European Metrology network.

Need

Plastic pollution is recognised as a severe anthropogenic issue globally, where complex physico-chemical transformation processes (such as aging, degradation and fragmentation) producing MPs and, subsequently, NPs. These processes occur during production, consumer use, waste processing, as well as through environmental process after vehicles/industrial emissions. Several studies have reported the occurrence, analytical methods and toxicity of larger MPs in the environment and food matrices. However, MPs (< 100 µm SMPs) and NPs (< 0.1 µm) in natural systems have been overlooked, primarily due to significant methodological challenges associated with their micro- and nano-specific properties.

In this respect, the European Commission (EC) commissioned a study focused on the potential ecotoxicological impacts of smaller plastic particles (SMPs/NPs), encouraging research aimed at a more accurate characterisation of both materials and exposure conditions. The need for efficient and reliable measurement infrastructure is required in support of (i) European Chemicals Agency (ECHA)'s proposed restriction targeting intentionally added MPs in consumer products, (ii) the Marine Strategy Framework Directive (MSFD) which requires specific thresholds for litter types after harmonisation of the methodology, (iii) the new Drinking Water Directive (DWD) that mentions MPs explicitly, and (iv) the new Circular Economy Action Plan (CEAP) adopted in March 2020. In particular, the CEAP promotes circular economy processes, encourages sustainable consumption, and aims to ensure that waste is prevented. However, to support the CEAP and reduce plastic contamination, methods for SMP/NP identification in food and environmental matrices are needed. These methods need to be metrologically validated using appropriate reference materials, so that Europe can establish harmonised and traceable measurements of SMPs and NPs.

End users of the procedures to be developed include public organisations / non-governmental organisations (NGOs) dealing with environmental and food monitoring, regulatory bodies responsible for the control of environmental pollution and food safety, as well as industries potentially responsible (directly or indirectly) for MP emissions and disposal into the environment or the human food chain.

Objectives

The overall aim of this project is to develop international metrological capacity that enables the traceable measurement and characterisation of SMPs and NPs in environmental and food samples and the production of suitable reference materials, according to the metrological requirements.

Report Status:
PU – Public, fully open

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European Partnership Co-funded by the European Union

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The specific objectives are:

1. **To develop pristine SI-traceable reference materials for SMPs (0.1 µm - 100 µm) and NPs (< 0.1 µm),** representative of partially degraded/naturally aged samples in complex food and environmental matrices. Realistic polydisperse size distributions and irregular shapes will be investigated.
2. **To develop accurate and efficient sample preparation methods for the measurement of SMPs and NPs in complex food and environmental matrices** (drinking and surface water, sewage sludge and milk). Such methods will include (i) enrichment prior to analysis, (ii) selective removal of natural background organic/inorganic matter, (iii) size fractionation/isolation, and (iv) homogenisation and partition steps. The sample preparation methods will be optimised to demonstrate a negligible effect on the particle characteristics and polymer compositions of samples
3. **To develop accurate and robust methods** for the (i) characterisation of chemical identity of the SMPs/NPs polymer type; (ii) physical particle characterisation and quantification, size distribution and particle morphologies; and (iii) quantification of the mass fraction in complex matrices.
4. **To demonstrate the validity and applicability of the methods and reference materials developed in Objectives 1-3 via an inter-laboratory comparison**. As part of the comparison, best practice guidance on the traceable measurement and characterisation of SMPs and NPs in food and environmental matrices will be developed.
5. **To facilitate the take up of the technology and measurement infrastructure** developed in in the project by the measurement supply chain, appropriate EURAMET's European Metrology Networks, relevant associations outside of Europe (e.g. National Nanotechnology Initiative USA), standards developing organisations (e.g. ISO TC 229, ISO TC 61, CEN TC 249 and those associated with the Urban Waste Water Treatment Directive (91/271/EU), the Marine Strategy Framework Directive (2008/56/EC) and the Drinking Water Directive (EU) 2020/2184) and end users (e.g. food and drink producers, environmental monitoring programmes and health experts).

Progress beyond the state of the art and results

Development of SI-traceable reference materials for SMPs (0.1 µm - 100 µm) and NPs (< 0.1 µm)

SMP/NP reference materials representative of partially degraded/naturally aged samples are currently not available and will be developed and provided by PlasticTrace. Candidate SMP/NP RMs representative of real plastic particles found in food and the environment will be developed in accordance with their relevant types, shapes, sizes and ageing status. Particle sizes are produced according to the possibilities of production and the toxicological relevance with two primary categories: (i) 100 - 10 µm and (ii) < 10 µm. To increase environmental relevance, some of the RMs will be aged. All prepared SMP/NP RMs will be tested for homogeneity and stability control (at least 3-month) according to ISO GUIDE 35:2017(E).

Development of accurate and efficient sample preparation methods for the measurement of SMPs and NPs in complex food and environmental matrices

The identification and the analysis of SMPs/NPs in complex media is very challenging due to the inability to readily distinguish SMPs/NPs from other types of particles in the same size range (dissolved and particulate organic matter) and due to the need for pre-concentrating samples to meet the detection limits for their identification. The existing procedures for sample preparation are often critical in terms of the stability of very small and aged particles, as well as very time consuming. PlasticTrace will cover the application and harmonise these procedures, including the application of state of the art digestion protocols for complex organic media, the selection of specific enrichment procedures suitable for each analytical approach, the development of filters in the sub-micron/nanometre range for SMPs/NPs filtration, and the application of different types of innovative fractionation steps for size separation.

Development of accurate and robust methods for the characterisation of SMPs/NPs

Given the challenge of characterising SMPs/NPs in complex matrices, PlasticTrace will primarily focus on the development and harmonisation of routine/established analytical methods for the measurement of SMPs (100-10 µm) and on the development of innovative hyphenated, complementary and correlative analytical approaches for the measurement of SMPs/NPs (<10 µm) in complex samples.

In particular, the following beyond state measurement capabilities will be developed and optimised:

(i) Light scattering methods for the characterisation of size distribution and particle number with hyphenated approaches based on fractionation techniques;
(ii) Innovative micro-spectroscopy methods for fast automation and data processing for large scale plastic particle monitoring and a new online hyphenated prototype based on fractionation-sizing-quantification and chemical characterisation;
(iii) Mass spectrometry-based methods for the characterisation of mass concentration, number and chemical identification with hyphenated approaches based on fractionation techniques;
(iv) High-resolution, correlative and automated microscopy methods for the characterisation of size distribution, shape and chemical identification performed on fractionated or filtered samples.

Validity and applicability of the developed methods and reference materials

To establish a reference value for critical MP/NP measurands (size, particle number concentration, particle mass fraction), at least two selected and more representative pristine SMPs with different polymeric composition and one selected NPs sample material prepared in this project, whose homogeneity and stability has been thoroughly evaluated in accordance with ISO GUIDE 35:2017(E), will be characterised via ILC. This will be considered a feasibility study driving to the certification process of the reference materials. Moreover, another ILC test will be organised with the involvement of the consortium and a wide external laboratory community in close cooperation with regulatory bodies. This will address the characterisation of a selected food and/or environmental sample matrix spiked with SMPs and NPs, in order to support the validation and harmonisation of the developed methods, as well as to provide reliable quantitative data that contribute directly to the development and implementation of future management strategies.

Outcomes and impact

The project will impact many different sectors including plastics industries, national / international food and environmental agencies, globally leading instrument producers, commercial and accredited laboratories, and leading food producers. It will also improve the reliability and accuracy of SMPs/NPs characterisation in food and environmental media.

Outcomes for industrial and other user communities

The project will provide application-oriented analysis tools, as well as an infrastructure for SMPs/NPs measurement across various fields. The establishment of a traceable measurement chain, with the provision of new reference materials and associated methods, will improve the reliability and accuracy of SMPs/NPs characterisation, and thus supporting utilisation of the results by end users (accredited commercial laboratories, national environmental institutes and monitoring agencies). Together with tools for quality control and proficiency testing, the traceability will guarantee standardisation and comparability of the results, currently lacking for SMPs and especially NPs. The outcome of the project will enable comparable and traceable monitoring to support decision-making and effective assessment of mitigation measures implemented by the EU's Plastic Strategy.

The research results from PlasticTrace will benefit the plastics and food industries, international/national food and environmental monitoring agencies, instrument producers, institutional and commercial laboratories, due to the production of certified reference materials for SMPs/NPs, the publication of metrological traceable analytical methods for SMPs/NPs in (drinking) water and food samples, and the development of new innovative and cost-effective technologies to measure SMPs/NPs. The project outcomes will directly benefit industry and monitoring agencies, through the involvement (as project participants, collaborators and stakeholders) of key actors, from SMEs (Postnova, SmartMembranes), to globally leading instrument producers (ThermoScientific, HF and Agilent), commercial laboratories (EUROFINS), leading food producers (Nestlé Waters, Barilla and Parmalat) and national / international environmental agencies (Norwegian Environment Agency, AMAP, UNESCO).

Outcomes for the metrology and scientific communities

PlasticTrace will provide validated SI-traceable measurement capabilities for the integral quantification of SMPs/NPs derived from the most common polymers, which is currently not available. It will also provide efficient sample preparation SOPs (Standard Operation Procedures) for relevant complex environmental and

food samples, which will support both the measurement infrastructure (aimed at routine laboratories) and the academic scientific community. New technological developments in innovative hyphenated, complementary and correlative analytical approaches and their standardisation represent major outcomes for PlasticTrace, to be quickly adopted into common use by metrological, research and scientific communities. These new and innovative methods and technologies not only have a significant potential for high-impact publications in high ranking scientific journals in both the environmental, food and metrological fields, but also direct implementation of the standardised methods to measure SMPs/NPs within ongoing and future scientific and research projects. Metrological outcomes related to food and nutrition will be communicated to METROFOOD-RI, the European Metrological Network for Safe and Sustainable Food, the European Metrological Network for Pollution Monitoring, and the NORMAN network WG micro and nano plastics, assuring a solid anchoring in the European research community for a rapid uptake of the new developed methodologies and their harmonisation. To further promote the use of the developed SMPs/NPs reference materials and the associated analytical validated tools, the consortium will target interactions with the European Commission's General Directorate for Health, Consumers & Reference Materials, the Consumer Products Safety Unit within the Joint Research Centre (JRC) and the European Research Executive Agency (REA). This will also ensure a solid connection and uptake at the international level due to the close connection with several international experts from China, Australia, USA, South Africa, Korea, Japan and Brazil, who have expressed their interest to be part of the PlasticTrace Stakeholder Advisory Board.

Outcomes for relevant standards

Several project participants are represented internationally in several ISO/CEN committees and working groups in VAMAS and BIPM, as well as national standardisation organisations (DIN, AFNOR, SFS, Standard Norway). This will guarantee the implementation of the project results in standardised methods both at a national and international level. In turn, this will build capacity for European environmental, food and drinking water monitoring programmes on SMPs/NPs. In line with the EU strategy and action plans, several EU directives are currently being revised or updated to include SMPs/NPs. This includes (i) the Urban Waste Water Treatment Directive (UWWTD), in which the MPs are currently not included and which is under revision in 2022, (ii) the Sewage Sludge Directive (SSD) which has signalled similar needs, (iii) the Environmental Quality Standards Directive (EQSD) where inclusion of MPs is currently being discussed, and (iv) the Marine Strategy Framework Directive (MSFD) which is currently discussing threshold values that need to be measured and controlled. Concerning food, no regulation on SMP/NPs is currently being considered due to the lack of harmonised analytical methods, but the European Commission sees a critical need in this field. In addition, the recast of the Drinking Water Directive (DWD) will adopt a methodology for measuring MPs in drinking water by adopting a legal binding delegated act(s) by January 2024. The results of PlasticTrace represent a crucial contribution towards achieving the objectives of these standards and directives on the restriction and especially control of SMP/NPs.

Longer-term economic, social and environmental impacts

PlasticTrace will address major societal needs defined in the Priority Themes of the EU Framework Programme for Research and Innovation 2021–2027, particularly in Horizon Europe Cluster 6, and by the CEAP, which highlights the severe impact of chemical pollutants and MPs on the health of water bodies and the need to develop harmonised measurement methods for unintentionally released of SMPs/NPs to close existing gaps in the scientific knowledge related to the risk occurrence of SMPs/NPs in the environment, drinking water and foods. Achieving reliable SMPs/NPs analytical determination is a prerequisite for addressing these major knowledge gaps, for providing a framework for science-based risk assessment and for consequent adoption of measures tackling plastic particle distribution and accumulation in the environment and food with potential long-range public health, economic and social impacts.

In order to monitor mitigation measures and emission control, efficient metrological traceability methods and technologies for SMPs/NPs are needed to support the EU's Green Deal, including the EU's Plastic Strategy and the Zero Pollution Action Plan for air, water and soil. Without the availability of traceable and quality assured analytical methods and reference materials, the goals of several EU directives on waste water treatment, sewage sludge, environment quality, marine framework and drinking water (UWWTD, SSD, EQSD, MFSD, and DWD) to reduce SMP/NP emissions to the environment cannot be achieved. By delivering cutting-edge outputs, including innovative measuring technologies combined with traceable QA/QC tools and environmentally relevant polymer reference materials, PlasticTrace will contribute directly to the EU Plastic Strategy objective to reduce the impact of SMPs/NPs to the environment.

Moreover, the development of novel analytical methodologies for the identification and quantification of SMPs/NPs in food matrices will facilitate the generation of critical exposure, ingestion and uptake data, thereby laying the basis for dietary exposure assessment. PlasticTrace has the ambition to provide the analytical tools necessary to facilitate a solid evidence-base for any regulatory action at national, EU and international level aimed at increasing the sustainability of the plastic industry and addressing the challenges posed by plastics throughout their entire life-cycle. The standardisation of methods will help to facilitate the assessment of the relevance, origin and fate of SMPs/NPs at European and international level and thus contribute to the creation of efficient strategies to reduce plastic inputs into the environment and food chain. This will also be in support of EFSA evaluation for risk assessment and toxicity of plastic materials along the food chain. In the long term, PlasticTrace is expected to directly contribute to new regulations for nanoplastics.

List of publications
n/a

This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/

<table>
<tr><td colspan="2">Project start date and duration:</td><td>01 October 2022, 36months</td></tr>
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6. LNE, France
7. NIVA, Norway
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11. FC.ID, Portugal
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