PlasticTrace
Tracing Micro and NanoPlastics in Food and Environment

EURAMET

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

Overview

The present project, PlasticTrace, aims to address the urgent need for the 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 inter-laboratory comparison validation studies. Novel and environmentally relevant SMP/NP reference materials will be developed within the project (e.g. Polyethylene terephthalate (PET), Polyethylene (PE)). International cooperation with key stakeholders such as EURAMET's European Metrology Networks (e.g. "Network for Safe and Sustainable Food" and "Pollution Monitoring"), standards developing organisations (e.g. ISO TC 229, ISO TC 61, and CEN TC 249) and end users (e.g. food and drink producers, environmental monitoring programmes and health experts) 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 (>100 µm) 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.

To improve this gap, 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) the 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 to 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.

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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.

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 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 Reference Materials (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 infrastructure capacity 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.

The decision for the first untreated and degraded polymer types was made based on the literature review and background exchange with previous projects (EU and JPI Oceans funded projects POLYRISK (ID: 964766), PlasticsFatE (ID: 965367) and ANDROMEDA). Polyethylene terephthalate (PET) and artificially UV-weathered Polyethylene (PE) were chosen. Both PET and PE are used in contact with food for beverages and packaging. Both are also found as waste in the environment. Their densities are lower and higher than 1 g/cm3, covering polymers that float or sediment in water. Bottle-grade PET granules and aged PE film were cryomilled in a centrifugal and ball mill, respectively, producing particles with polydisperse size distribution and irregular shape. The produced powder was sieved with a 100 µm stainless steel ring sieve to obtain particles < 100 µm.

The PET particles were mixed with a water-soluble matrix and pressed into a tablet with different concentrations. 1000 tablets were prepared with concentrations suitable for thermoanalytical and vibrational methods separately. Homogeneity control was performed by Thermal Extraction and Desorption GC-MS (TED-GC/MS). The tablets were distributed to the partners for homogeneity and stability control measurements including lab-to-lab uncertainties. To increase the comparability of results, the same filtration setup including

a 5-6 µm Si filter membrane was supplied to each partner. The filtration setup has been validated for mass and will need further work for particle number.

The project agreed to start the development of SMPs and NPs with Polypropylene (PP) particles < 1 µm based on available information in the literature. More than 800 glasses' containers were produced with 2 ml content. To speed up the production process and get a polydisperse distribution of the particles, filtration was done to remove large (> 1 µm) polymer fragments and not to reduce the polydispersity index. The particles are irregularly formed and have a mean size of 190 nm. These glasses' containers were provided to partners for the characterization of size distribution, particle number, shape and mass fraction by light scattering methods, AFM and electron microscopy and correlative/hyphenated approaches with spectroscopy techniques to provide chemical identification. First tests of Field flow fractionation techniques coupled with NTA analysis are running in parallel to separate and count particles < 100 nm.

All prepared SMP/NP RMs will be tested for homogeneity and stability control 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. The project will cover the application and harmonisation of procedures such as 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.

Four types of matrices, i.e. drinking water, milk, surface water and sewage sludge, were selected as relevant environmental and food matrices for MP contamination, exposure and human consumption, and are ready to be distributed to the partners in the consortium. A literature research on the current state of the art of sample preparation, and an exchange of existing Standard Operation Procedures (SOPs) with other projects (i.e. EUROqCHARM, POLYRISK, PlasticsFatE, CUSP) was sought. A preliminary recommendation plan on sample preparation for all these matrices was developed and distributed to the partners. Moreover, a good laboratory practices guideline for handling and spiking matrices was prepared with the aim to harmonise and define the basic requirements and quality control for SMPs/NPs analysis, in regard to sample handling and contamination reduction. These harmonized procedures will further support a survey on current calibration methodologies and analytical measurement techniques as a solid base for the organization of Inter-laboratory Comparisons (ILCs) during the course of the project.

As for plastic particles lower than 10 µm, the project will work on the selection of specific and innovative enrichment procedures suitable for each analytical approach (e.g. SPLITT down to 10 µm and continuous flow centrifugation down to 150 nm), the development of new silicon and/or aluminium oxide filters with pores in the sub-micron/nanometre range (from 100 µm down to 50 nm) for SMP/NP filtration, and the application, optimisation and comparison of different types of fractionation tools for pre-concentration/preparative steps and size separation, such as AF4 and CF3. This will provide an opportunity to further develop online and offline methods for particle size distribution analysis, stability evaluation, polymer identification by spectroscopic tools and mass fraction quantification by thermoanalytical techniques.

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

Given the challenge of characterising SMPs/NPs in complex matrices, the project will have two primary focus areas: (i) development and harmonisation of routine/established analytical methods (µFTIR, µRaman, EM, Pyr- GC/MS, TED-GC/MS), including sample preparation for the measurement of SMPs (100-10 µm) in food and environmental related samples, and (ii) development of innovative hyphenated, complementary and correlative analytical approaches for the measurement of SMPs/NPs in drinking water and milk samples.

In particular, the following beyond the state of the art 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), the project will characterise via ILC 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),. This will be considered a feasibility study directed 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

To create impact several dissemination and communication activities are were performed including a website (www.plastictrace.eu) and social media channels like LinkedIn (www.linkedin.com/company/92810943). A stakeholder advisory board consisting of 20 key stakeholders has been established and will support the project and further interaction with a larger group of stakeholders including a larger number of expert practitioners, standards community and metrology institutes through workshops. Joint EU project meetings to exchange knowledge and synchronize activities with on-going EU projects are integrated in two activities. Results from the different activities will be presented at different scientific international meetings and symposia and published in the international literature. Consortium participants are actively taking part in standards and technical committees and working groups to disseminate the outcomes from the different activities of the project. Training of technical staff is crucial and will be accomplice by capacity building workshops, an e-Learning program and a training network.

Already two stakeholder workshops were organised on the topic “Measurement needs for Microplastics” together at INRIM in connection with VAMAS Technical Working Area 47th SC Meeting, but also a Stakeholder Advisory Board meeting was organised in connection with the first project meeting. In addition, the stakeholders were asked in two questionnaires to advice on preferred reference materials and analytical methods. The project has also joined several EU projects meeting, such as EUROqCHARM and COST Action PRIORITY on nano and microplastic standardisations. The project has also organised special sessions with the NORMAN network and the general assembly, in addition with Plastics Europe in collaboration with CUSP and NORqCHARM to present the activities of the project on European harmonisation. The Project has also been active in several standardisation activities, to inform standards bodies and committees the development and results of the project to be incorporated into standards, such as with international standards development organisations (ISO, CEN, DIN) and metrology committees (BIPM and CIPM).

With the focus on dissemination and stakeholder interaction PlasticTrace has already presented the projects’ aim and results at 33 occasions including oral and poster communications for international, European and national conferences. Training sessions were provided for consortium members regarding “Use of Sartorius filtration adapter for microplastics” and “Optimization of filtration system procedure and background contamination control”.

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.

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 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 will represent major outcomes from the project, 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.

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 since 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 will 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 EU Framework Programme for Research and Innovation 2021–2027, particularly in Horizon Europe Cluster 6, and by the CEAP. These directives highlight 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 SMPs/NPs and their risk in the environment, drinking water and foods. Reliable SMPs/NPs analytical determination is a prerequisite to address major knowledge gaps, and to provide a framework for science-based risk assessment and 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.

List of publications
No publications are available yet.

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

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Project start date and duration:	01 October 2022, 36months	
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Internal Beneficiaries: 1. INRIM, Italy 2. BAM, Germany 3. DFM, Denmark 4. IHi, Portugal 5. IPQ, Portugal 6. LNE, France 7. NIVA, Norway 8. SMD, Belgium 9. SYKE, Finland 10. UBA, Germany	External Beneficiaries: 11. FC.ID, Portugal 12. FhG, Germany 13. Hereon, Germany 14. Postnova, Germany 15. Sciensano, Belgium 16. SINTEF, Norway 17. UDC, Spain 18. UNIPR, Italy 19. UNITO, Italy	Unfunded Beneficiaries: 20. HF, France 21. Nestlé Waters, France 22. SmartMembranes, Germany
Associated Partners: 23. LGC, United Kingdom		