

Proposals for NORMAN Joint Programme of Activities 2023

Title	Sandwich Filters for the Comparison of Microplastic Measurements
Type of activity	WG 4 Reference material and standards for microplastic research
Leader	Eawag / NIVA
Topic / activities	<p>Background / Justification for the proposed activity:</p> <p>Despite considerable efforts to harmonize and standardize methods for the analysis of microplastic particles (MP), results from recent inter laboratory studies (ILS) still do not reach targeted quality criteria. Whereas the quality of results (z-scores) from thermal methods (e.g., pyrolysis gas chromatography - mass spectrometry (py GC-MS)) improved over time, the results from particle based methods (e.g. u-Fourier transform – infrared spectroscopy (u-FT-IR)) remain at a rather low level. Relative standard deviations (RSDs) between the laboratories were > 100% and only 60% of the participants having acceptable z-scores < 2 based on a RSD of 25% resulting in average z-scores ranging from 1.7 to 5.7 depending on the polymer type. In ILS, MP particles contained in matrices of different complexities ranging from water soluble tablets to dried sediments were sent to the participants for sample preparation and MP analysis. This approach does not allow a decoupling of the uncertainties related to sample preparation from the uncertainties related to the actual measurements.</p> <p>To disentangle the uncertainties related to sample preparation and measurement, we will fabricate filters loaded with different types of well-characterized MP that can be sent from lab to lab. We will enclose the MP-loaded filters between two IR transparent windows to immobilize the MP particles and we will construct a dedicated sample holder for our sandwich filter design.</p> <p>Description of the proposed activity and expected outcomes for 2023:</p> <p>Different types of well-characterized MP particles, such as polystyrene (PS) and Polymethylmethacrylat (PMMA) spheres, and polyvinylchloride (PVC) fragments, will be deposited on Anodisc filters (0.45 µm pore size, 25 mm diameter). The MP-loaded filters will be imaged using optical microscopy (VHX-7000, Keyence) and the deposited MP particles will be identified based on their characteristic size, shape and color. The filters will then be gently pressed between two IR-transparent calciumfluorid (CaF₂) windows and imaged again using both an optical microscope (VHX-7000, Keyence) and an automated focal plane array (FPA)-u-FT-IR system (64 x 64 pixel detector, Cary 670 FTIR instrument, Cary 610 IR microscope, Agilent). Initial experiments were conducted using CaF₂ window with a thickness of 1 mm. These windows provided sufficient stability and individual MP particle were successfully identified after the sandwich filter was carried around the Eawag campus (Figure 1). However, the sandwich design also reduced the signal intensity and thus the quality of the FT-IR spectra.</p>

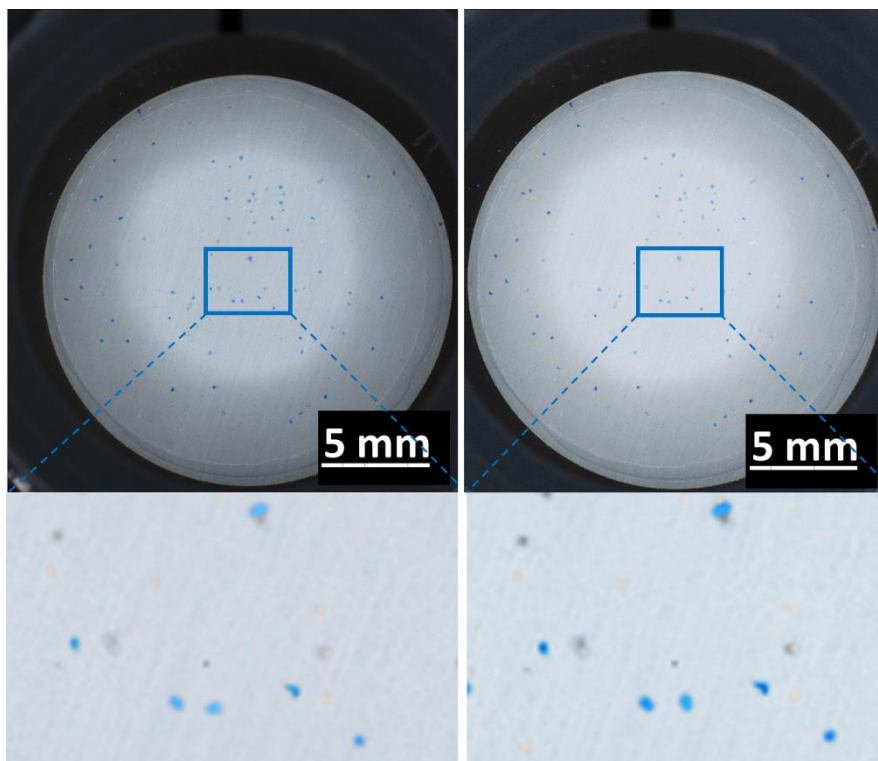


Figure 1: Left: Optical images after sample preparation; right: Optical images after carrying the sandwich filter around the Eawag campus.

However, the quality of the spectra still allowed an identification of the added polymer types (Figure 2). Nevertheless, we will assess the performance of thinner (0.5 mm) windows to further improve the quality of the IR spectra.

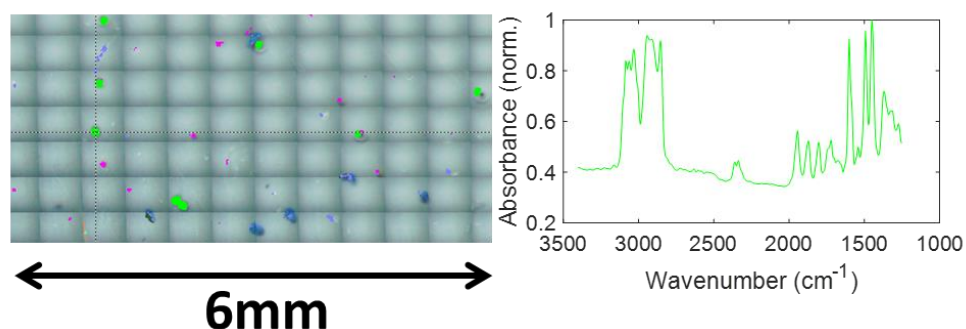


Figure 2: Optical microscopy images recorded on the focal plane array (FPA) - μ -FT-IR system, with identified polymer types (coloured). The green colour corresponds to polystyrene (PS) and the extracted spectrum of an identified PS sphere (100 μ m) is shown to the right (in green).

Our current sample holder for the sandwich filter essentially consists of two hollow plastic cylinders that are pressed together by means of a thread. However, the sample holder does not match the sample stage well, yet. We therefore aim for a modified and flatter design, exactly matching the stage dimensions of the FT-IR instrument.

The optimised design for the sandwich sample holder (adjusted to sample stage, possibly using 0.5mm IR transparent windows) will be evaluated at Eawag by performing measurements of MP loaded filters (with and without IR windows). In addition, sandwich filters will be imaged before sending them to and after receiving them back from other locations within Switzerland. Should this test be successful, sandwich filters will be sent to NIVA for FT-IR measurements and sent back again to Eawag for repeated FT-IR and optical microscopy measurements. Comparison of the results from these feasibility experiments will allow us to assess whether our sandwich filter design is robust enough for sending MP-loaded filters to other labs with the MP particles remaining in place. If this is the case, we will plan a next ILS study in 2024 using our sandwich filter design.

Added value / Link with other NORMAN activities and / or other projects

Separating the uncertainties related to sample preparation and experimental measurements will contribute to a better understanding of contrasting results from previous ILS studies and will contribute to the development of harmonised methods for particle-based microplastic analysis. Amongst others, the EU project EUROQCHARM will benefit from these activities.

Participants	Eawag, NIVA, VU-University, AWI, SINTEF, Agroscope.
Proposed in-kind contribution	The participants will provide well-characterized microplastic particles for the experiments. Furthermore, the participants will cover the costs for using the analytical facilities (μ -FT-IR system). Eawag will cover costs associated with the fabrication of the filter holder.
Contribution needed from NORMAN Association¹	In total, we ask for a contribution of 9000 Euros from NORMAN. The costs are distributed between materials (IR transparent windows and lab consumables) and person-months for designing the sandwich filter holder and assessing its suitability for future ILS studies. We estimate the material costs to roughly 1500 Euros, the remaining 7500 Euros will be assigned to work force (~0.75 person months).

¹ Please, provide here a transparent justification of the requested resources and of the in-kind contribution, thereby distinguishing between the costs associated with "person-months" for the organisation, the "travelling costs" for invited speakers and the costs for the logistics (e.g. meals, room rental etc.)