

# MICROPLASTICS ANALYSIS WORKSHOP

## Report

### Second QUASIMEME-NORMAN Microplastics Interlaboratory Workshop

20 and 21 May 2021, online, Vrije Universiteit, Amsterdam, The Netherlands

Jacob de Boer<sup>1</sup>, Heather Leslie<sup>1</sup>, Louise van Mourik<sup>1</sup>, Bert van Bavel<sup>3,4</sup>, Steven Crum<sup>2</sup>, Wim Cofino<sup>2,4</sup>

<sup>1</sup> Vrije Universiteit, Dept. of Environment and Health, Amsterdam, The Netherlands

<sup>2</sup> QUASIMEME, Wageningen University, Wageningen, The Netherlands

<sup>3</sup> NIVA, Oslo, Norway

<sup>4</sup> NORMAN Network



VRIJE  
UNIVERSITEIT  
AMSTERDAM



NIVA  
Norwegian Institute for Water Research



**Table of Contents**

Synopsis ..... 3

Introduction .....3

Workshop goals ..... 4

Workshop outcomes..... 4

## **Synopsis**

A second workshop, coordinated by the Vrije Universiteit Amsterdam, was organized on line on 20 and 21 May 2021 to discuss the results of the first and second round of the QUASIMEME/NORMAN interlaboratory study (development exercise) on the analysis of microplastics. This workshop attracted a maximum of 66 participants on both days. Participants engaged in open discussions on the quality assurance/quality control (QA/QC) needs of analytical laboratories. Two presentations highlighted the results of the two rounds of a stepwise-designed development exercise that were organized in the period 2019-2021. Presentations of participants demonstrated the challenges that were met during the two rounds. A thorough discussion was held among all participants about the results, possible improvements in the organization and needs for the third round of the study. A brief on-line questionnaire was organized during the workshop on the needs for (certified) reference materials for micro-plastics. A third round of this interlaboratory study will be organized between the second half of 2021 and the first half year of 2022. This exercise will be open to any microplastics analysis laboratory with an interest of joining a community that is engaged in learning exercises and increasing their proficiency.

## **Introduction**

Following the Microplastics Analysis Workshop held in November 2018 in Amsterdam, the first two rounds of the interlaboratory study on microplastics were held between 2019 and 2021. The covid-19 situation caused a delay of the second round compared to the original planning. Closure or partial closure of their laboratories caused that some laboratories enlisted for the exercise could not deliver results. The first round was a simple study on the identification and quantification of preproduction pellets and microplastics in water using tablets to be dissolved in water to mimic real conditions. The simple set-up was chosen to allow as many participants as possible to join and not exclude any method. The second round included, in addition to a repeat of the tablet exercise, a spiked sediment and a fish sample.

## **Workshop goals**

The purpose of this workshop was to bring the participants of the first two rounds together and discuss the results and key challenges as to achieve a better harmonization of microplastics data being produced. In addition, the organization of a possible third round of this interlaboratory study was discussed.

## **Workshop Outcomes**

The first day of the workshop was devoted to two presentations on the results of the first and second round, both collectively given by Prof. van Bavel and Prof. de Boer. These presentations were followed by a thorough discussion on the results, the challenges of the various methods used and possible improvements in the organization of a next round. The lively discussion contained the following major elements.

Participants were satisfied with the use of the tablets.

There was some confusion on the lipid content of the fish sample distributed in the second round. The lipid content appeared to be higher than expected and had caused difficulties for digestion methods used by some participants. After double checking after the meeting it can be confirmed that the test material was freeze-dried blue whiting with a lipid content of 3.2% (0.8% before freeze-drying). This information will be added to the report.

It was suggested to add a table with densities of the various polymers that were used for spiking to the report.

The sample of the various methods intake was discussed. The methods based on counting particles generally used more than 20 g fish or sediment. The participants using pyrolysis GC /MS used less.

The homogeneity of the test materials was discussed. The organization should of course guarantee that each container/bottle or vial contains exactly the same amount of microplastics. On the other hand, participants need to re-homogenize the contents of a container/bottle/vial to make sure that segregation that might have occurred during transport is annulled.

All agreed that in particular the spike of the fish sample was extremely high. This caused especially difficulties for laboratories using counting methods.

Suggestions were made for adding training samples in the next round to allow participants to 'play' with the sample before doing the final analysis.

The second day started with two presentations by participants. Dr. Naesheim (Eurofins) presented results of his pyrolysis GC method. He also mentioned an overloading problem due to the high spikes of the fish and sediment. Dr. Rotander (Örebro University) presented results of her method based on ATR-FTIR. It was her first time to analyze microplastics in fish. She needed a digestion time of four weeks (!) due to the high lipid content and the advice to take the entire sample into analysis. Particle sizes <20 µm are a serious challenge for ATR-FTIR methods. She noticed a waxy character of the PE particles that were spiked. She asked for natural materials in a next round.

Dr. Leslie (Vrije Universiteit Amsterdam) spoke on the topic of laboratory needs for certified reference materials (CRMs) for microplastics in natural matrices, and organized a brief poll focused on what type of such materials participants think are needed in the future. Dr. Belz (JRC) mentioned that CRMs should not be used for interlab study materials but rather a golden standard to validate methods in the laboratory. Most votes were given to water, biota/fish and sediment as desired CRMs.

Prof. Leonards (Vrije Universiteit Amsterdam) presented the results of a sub-study (MISSOURI) in which six sediment samples were analyzed on plastics. The results were more or less comparable with those of the main study. The inhomogeneity of the test materials after transport was emphasized. Participants agreed that spiked round robin sediment samples should rather be wet than dry materials.

Finally, Dr. Leslie and Prof. De Boer (Vrije Universiteit Amsterdam) presented an outlook on the third round. The new European research project, EUROqCHARM, coordinated by Prof. van Bavel was introduced. There are good possibilities for collaboration with this project in the next round. This will offer reduced costs for participants and better statistics for the study. It may also assist in producing CRMs on the long term, in collaboration with the Joint Research Centre, Geel, Belgium.

The final discussion was lively and included the following elements.

Several participants asked for including colored particles in the next round.

Mussels was mentioned as a preferred biota sample.

Cryo-milling was recommended for preparing smaller plastic particles.

Self-spiking was suggested as an option.

ICP-MS was mentioned as an option to detect very small particles.

Several participants have an interest in analyzing fibers. However, the preparation of round robin materials with fibers is difficult and could, until now (in Round 1), only be done manually, which is very time-consuming.

Dr. Vorkamp emphasized to keep the link with monitoring programs such as OSPARCOM, HELCOM and AMAP programs. She also warned for too easy samples, that might provide good but unrealistic ILS results.

Dr. Suarez proposed to prepare a natural sand material as microplastics reference material. He also asked for using mussels in the next round, because they are strong bioaccumulators. Prof. Cofino emphasized to keep the link with South-America.

Dr. Kögel warned for calcified particles when analyzing mussels.

Dr. Strand emphasized the need for atmospheric samples in AMAP. AMAP will concentrate now on air, fulmars, water, sediment and beach plastic. He also suggested to avoid the use of round spherules since they can be more difficult to detect with  $\mu$ FTIR than fragments. Finally, he proposed to use PET strings, made of e.g. precise 100 fibers, and then one cut of a string using the same length of e.g. 500  $\mu$ m that will provide 100 identical fibers for each sample.

Prof. van Bavel emphasized the need to include fragments. Dr. Rotander suggested a mix of spheres, fragments and fibers.

Mr. Crum said that dry samples (fish and sediment) are less homogeneous in the bottle and therefore more difficult to sub-sample. He would prefer to provide wet fish tissue and sediment slurries because they would be easier to sub-sample.

The organizers will now concentrate on the preparation of the next round, which could include a repeat of the tablet study, a sediment and a mussels test material with low spikes. A serious attempt will be made to reduce the within-homogeneity of the test materials. Additional options, as mentioned in the discussion, such as color, fragments, fibers, an extra training sample, etc. will all be considered for their feasibility.

The recording of the workshop will remain available for the participants until 20 June 2021. The report on Round 2 will be adapted according to some remarks of the participants and will be distributed before the end of June.

Useful websites:

Website E UROqCHARM: <https://www.euroqcharm.eu/en/>

Food safety/EFSA: <https://www.efsa.europa.eu/en/events/event/update-scientific-colloquium-25-coordinated-approach-assess-human-health>

And: [https://www.efsa.europa.eu/sites/default/files/event/AF20190703-ax10a\\_MH.pdf](https://www.efsa.europa.eu/sites/default/files/event/AF20190703-ax10a_MH.pdf)

Plastics and human health: [www.cusp-research.eu](http://www.cusp-research.eu)