

Planned European Method Evaluation Study - ILC, QA/QC

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Summary

- What is it?
- Why do we need it?
- Who should participate?
- How are we going to do it?

WHAT:

Interlaboratory Study on the Analysis of Microplastics in Environmental Matrices

A Microplastics Analysis Workshop was held in November 2018 in Amsterdam and was dedicated to the topic of microplastics analysis in environmental matrices. Based on the outcome of that workshop QUASIMEME announced the first phase of an international microplastics interlaboratory study.

Reference materials

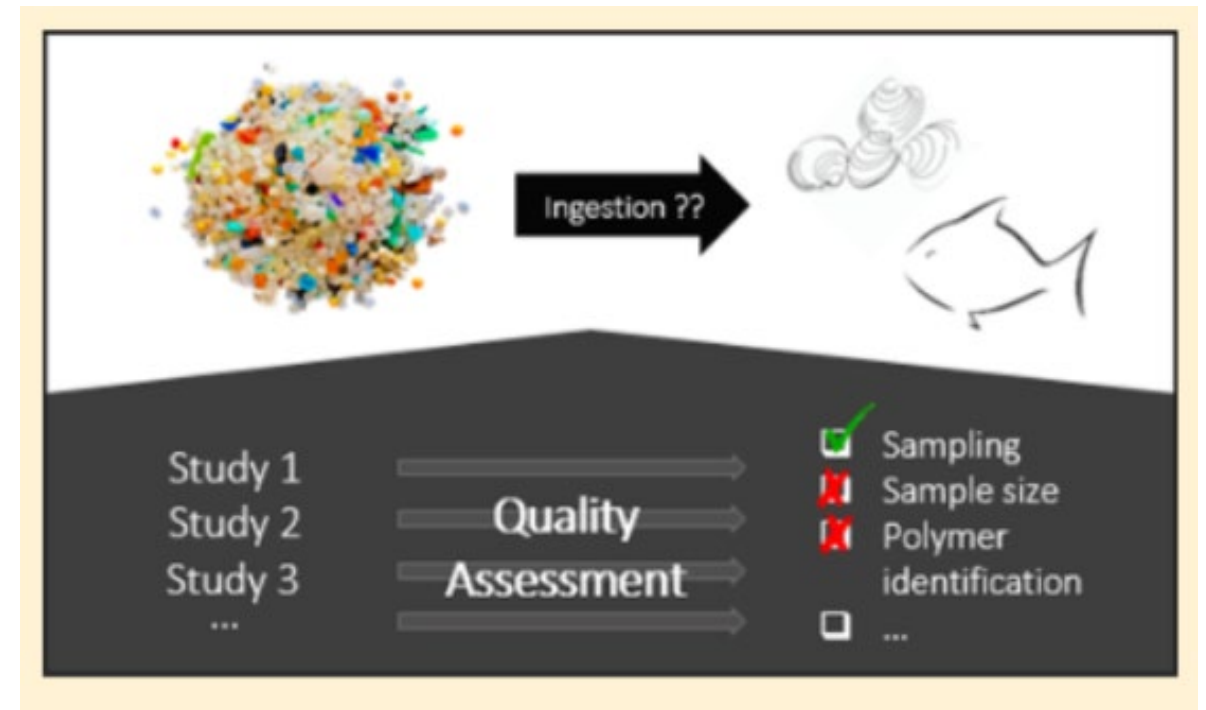
Interlab comparison of methods

QA/QC

WHY: Lack of QA/QC during Microplastic Analysis

Current studies portray a wide spread in results on the occurrence of MPs, highlighting a lack of comparability of results.

The results of this quality assessment show a dire need for stricter quality assurance in MP in studies.



(Hermsen et al. ES&T 2018)

WHY: Lack of QA/QC during Microplastic Analysis

Table 1. Scoring of the Reviewed Articles in the Current Quality Assessment^a

study	year	criterion										accumulated score
		1	2	3	4	5	6	7	8	9	10	
		sampling methods	sample size	sample processing and storage	laboratory preparation	clean air conditions	negative control	positive control	target component	sample treatment	polymer identification	
Lusher et al. ⁴⁸	2016	2	2	2	2	1	2	0	2	2	0	15
Tanaka and Takada ⁷⁸	2016	2	2	2	0	0	1	0	2	2	2	13
Davidson and Dudas ⁵⁹	2016	1	1	2	2	0	2	2	2	0	0	12
Rummel et al. ⁵⁸	2016	2	2	0	2	1	2	0	0	1	2	12
Courten-Jones et al. ⁴⁹	2017	0	0	2	2	0	1	0	2	2	2	11
Devriese et al. ⁵⁶	2015	2	1	2	0	2	2	0	2	0	0	11
Mathalon and Hill ⁸	2014	1	1	2	1	1	2	1	2	0	0	11
Wesch et al. ⁷⁹	2016	0	2	2	0	2	0	0	2	2	1	11
Cannon et al. ⁴³	2016	0	2	2	0	2	0	0	2	0	2	10
Desforges and Galbraith ⁵⁰	2015	2	2	2	0	0	2	0	2	0	0	10
Li et al. ⁸⁰	2016	2	2	0	0	0	0	2	0	0	2	10
Murphy et al. ⁸¹	2017	2	1	0	2	0	1	0	2	0	2	10
Vandemeersch et al. ²⁷	2015	1	1	2	0	0	2	0	2	0	0	10
Davison and Asch ⁴¹	2011	2	2	2	0	0	1	2	0	0	0	9
Foekema et al. ^{6,b}	2013	2	2	1	0	0	0	0	2	2	0	9
Karlsson et al. ⁵³	2017	1	1	2	1	0	2	0	2	0	0	9
Nadal et al. ⁸²	2016	2	2	2	0	0	1	0	0	0	0	9
Torre et al. ⁵⁴	2016	0	2	2	2	1	2	0	0	0	0	9
Bellas et al. ⁴⁷	2016	2	1	1	1	0	1	0	0	1	0	8
Jabeen et al. ⁴⁴	2016	0	2	0	1	0	1	0	2	0	2	8
Lusher et al. ⁵	2013	2	2	2	0	0	0	0	0	0	2	8
Van Cauwenbergh et al. ⁶⁰	2014	1	0	1	0	2	2	0	2	0	0	8
Brate et al. ⁸³	2016	0	2	0	2	0	1	0	0	0	2	7
Anastasopoulou et al. ⁸⁴	2013	2	2	2	0	0	0	0	2	0	0	6
Besseling et al. ^{16,b}	2015	2	0	0	0	0	0	0	0	2	2	6
Jantz et al. ⁸⁵	2013	1	2	2	0	0	0	0	0	1	0	6
Murray and Cowie ⁵¹	2011	2	2	2	0	0	0	0	0	0	0	6
Peters et al. ⁷⁰	2017	1	2	2	0	0	1	0	0	0	0	6
Vendel et al. ⁸⁶	2017	2	2	1	0	0	0	0	0	0	0	5
Boerger et al. ⁵²	2010	2	2	0	0	0	0	0	0	0	0	4
Liboiron et al. ⁵⁵	2016	0	2	0	0	0	0	0	2	0	0	4
Neves et al. ⁷	2015	0	0	1	0	0	0	0	0	0	2	3
Wojcik-Fudalewska et al. ⁸⁷	2016	0	1	2	0	0	0	0	0	0	0	3
Romeo et al. ⁹	2015	1	1	0	0	0	0	0	0	0	0	2
Miranda and de Carvalho-Souza ⁸⁸	2016	0	0	0	0	0	0	0	0	0	0	0
Av all-study score (n = 35)		1.14	1.46	1.31	0.57	0.40	0.86	0.17	1.03	0.43	0.66	8.0

^aScores of 0–2 were assigned to each publication in each of the 10 categories. The publications are sorted from high to low based on the “accumulated score”. The overall reliability score was 0 for all studies and is not indicated. ^bStudies with involvement of 1 or more of the authors of the present paper.

Klimisch score

- Criteria for Reporting and Evaluating Ecotoxicity Data (CRED)

11 QA Criteria

- sampling method and strategy
- sample size
- sample processing and storage
- laboratory preparation
- clean air conditions
- negative controls
- positive controls
- target component
- sample (pre)treatment
- polymer identification

WHY: Lack of QA/QC during Microplastic Analysis

On average, studies scored:

8/20 “completeness of information”

0 for “reliability”

Negative control

- *10 out of 35*

Positive control

- *2 out of 35*

Accuracy?

Uncertainty?

LoD?

WHO:

Laboratories performing microplastics analyses
in abiotic or biotic environmental matrices,
food or biological tissues.

Currently no limit to the number of participating labs.



HOW:

1. Participants register for participation in different rounds.
2. Samples are prepared by organizing labs and sent to participants.
3. Complexity of samples increases as rounds progress.
4. Participants analyze samples at own lab and report their results.
5. Organizers compile data received and compare results per laboratory with assigned values, calculate z scores.
6. Labs get feedback on their method used: (“learning exercise”).
7. z-scores communicate to labs how their reported data was evaluated (satisfactory score or outside) 😊😐😞
8. Results are discussed and follow-up rounds are organized.
9. A second workshop is organized to discuss results and tips.

Study Design: Round 1

- Participants will receive “pills” to spike own samples and test recover rates
- Participants are free to use any analytical methods

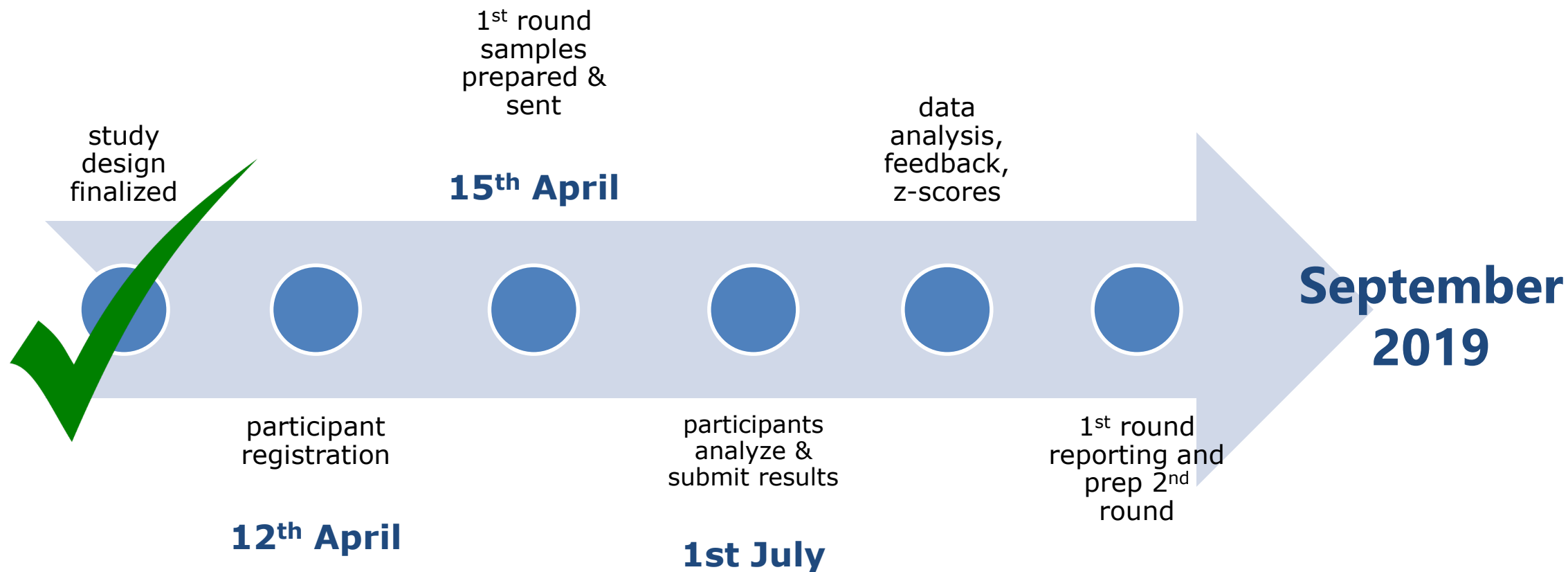
Particle size:

- A) 1 mm to 300 um (all methods including optical methods *(most labs)*)
- B) Samples that include the <300 um range for labs with these capabilities *(fewer labs)*

Content of pills:

1. Pre-production pellets, different sizes and one blank (10 pills)
main objective: (counting particles)
2. Fibers (2 pills) (main objective: identification of plastic)

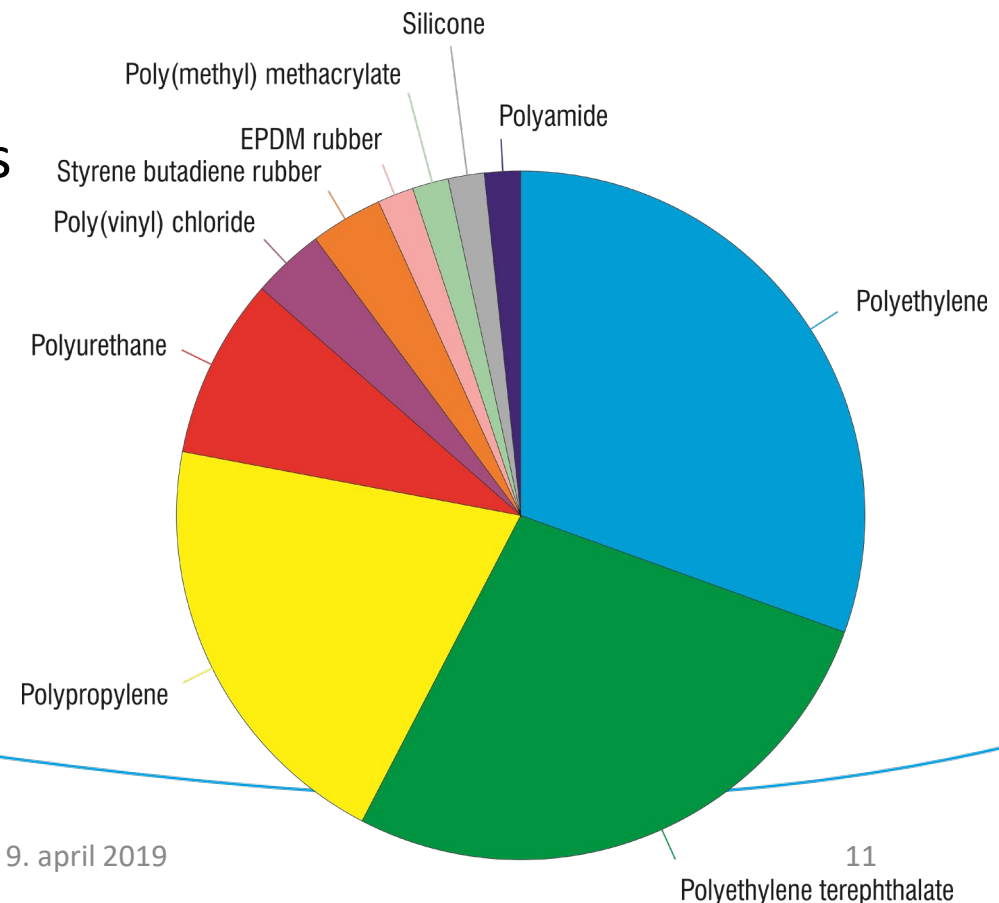
HOW: Study Design (Round 1)



HOW: Standard reference material

- Reference standard materials of common, relatively high-production volume (co)polymers that are likely to be components of real-world MPs-contaminated samples (fragments, spheres, fibers)
- Weathered MPs in an uncleaned extract (contains potential interferences)
- MPs commonly found in environmental matrices (*e.g.* sediment, biota)

Lusher et al., NIVA Report, 2017



Standard reference material for analytical QA/QC



3 materials

- *Synthetic fibers*
- *Different polymer fragments*
- *Car tire dust*

Procedure tablet production

A powder mixture consisting of sodium hydrogen carbonate (NaHCO_3) and citric acid ($\text{C}_6\text{H}_8\text{O}_7$)

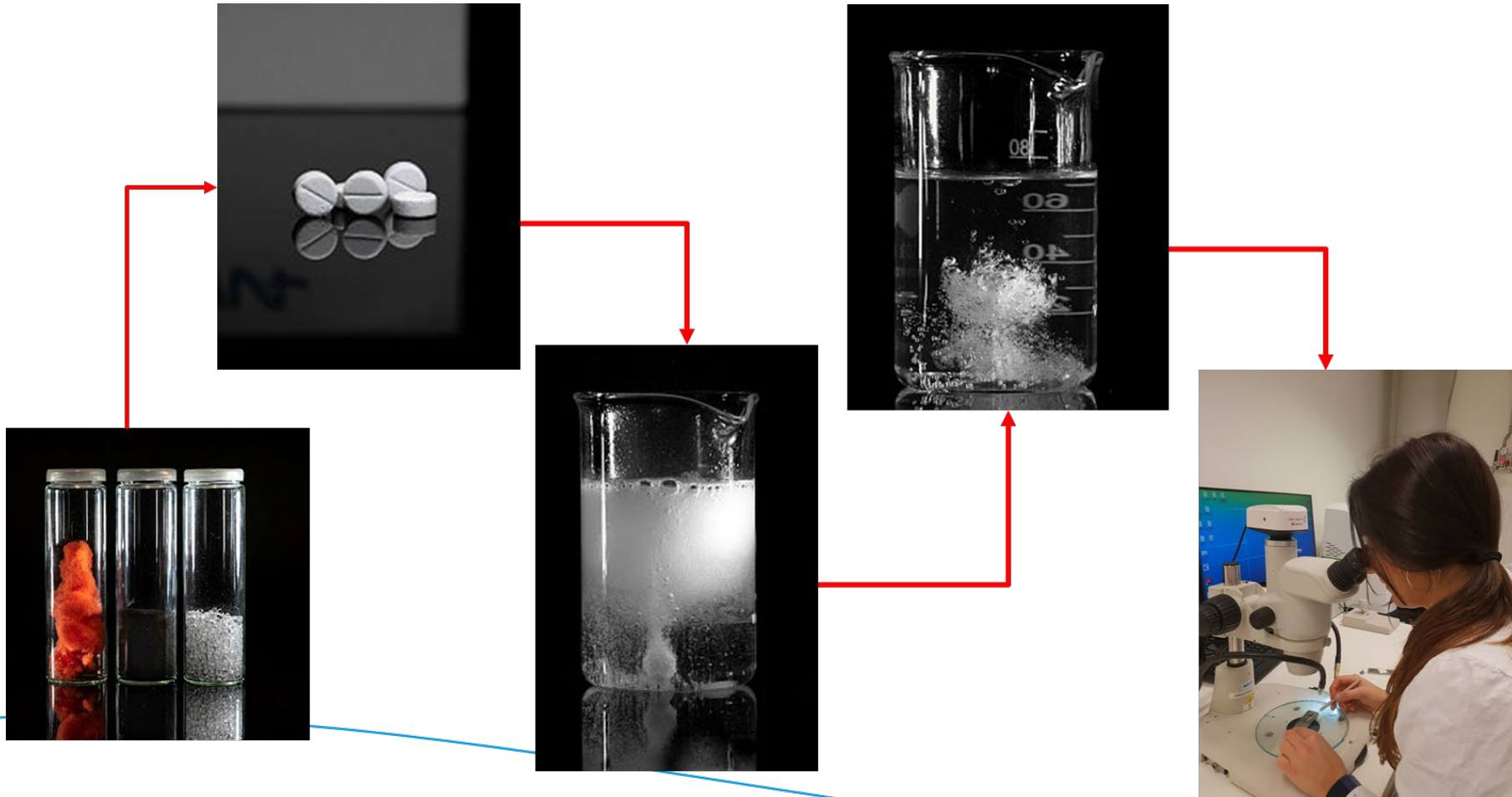
Both tyre dust and polymer fragments were encapsulated in the tablets.

Polyester fibres were added and counted manually under a microscope.

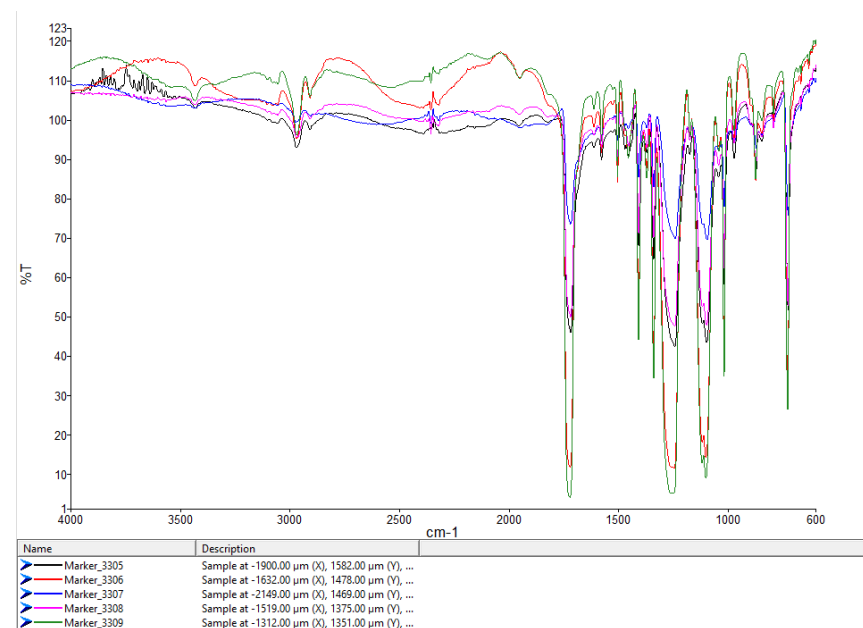
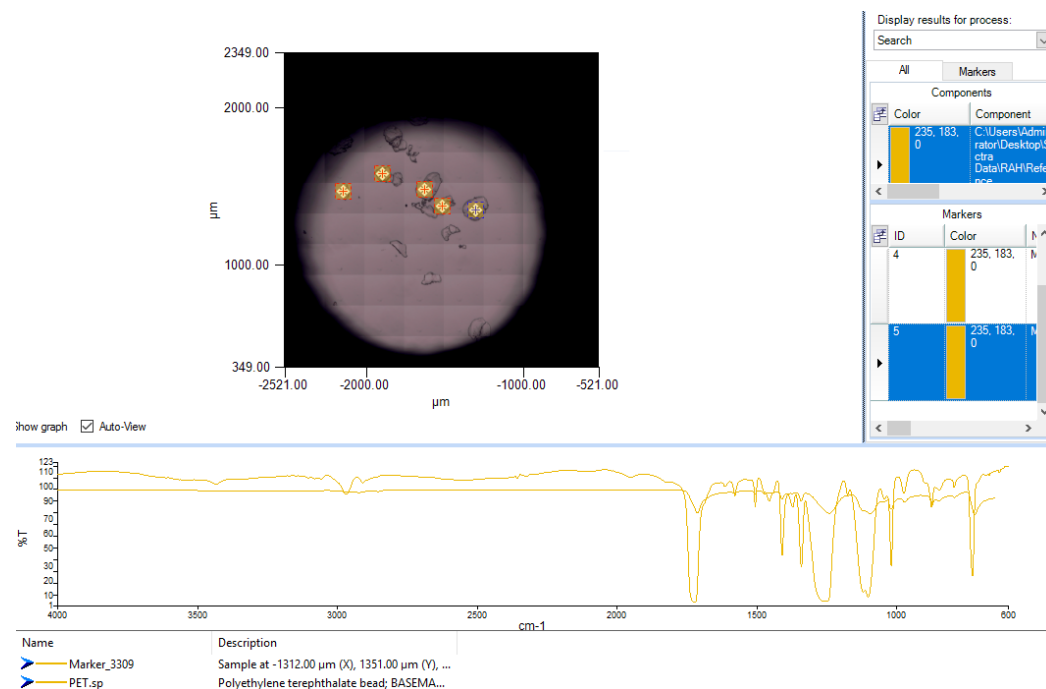
Blank tablets were produced with every batch.



How the pills work:

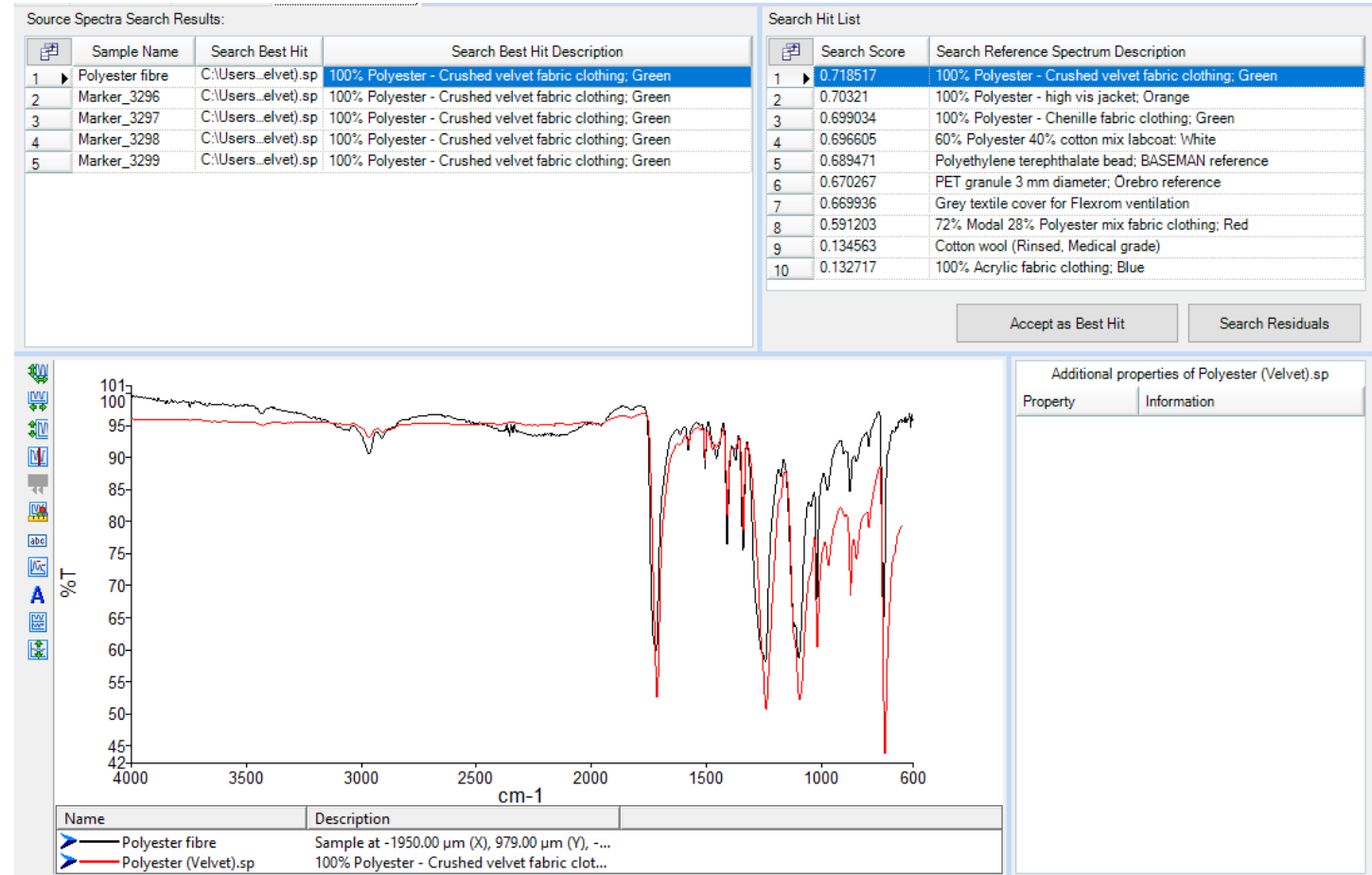
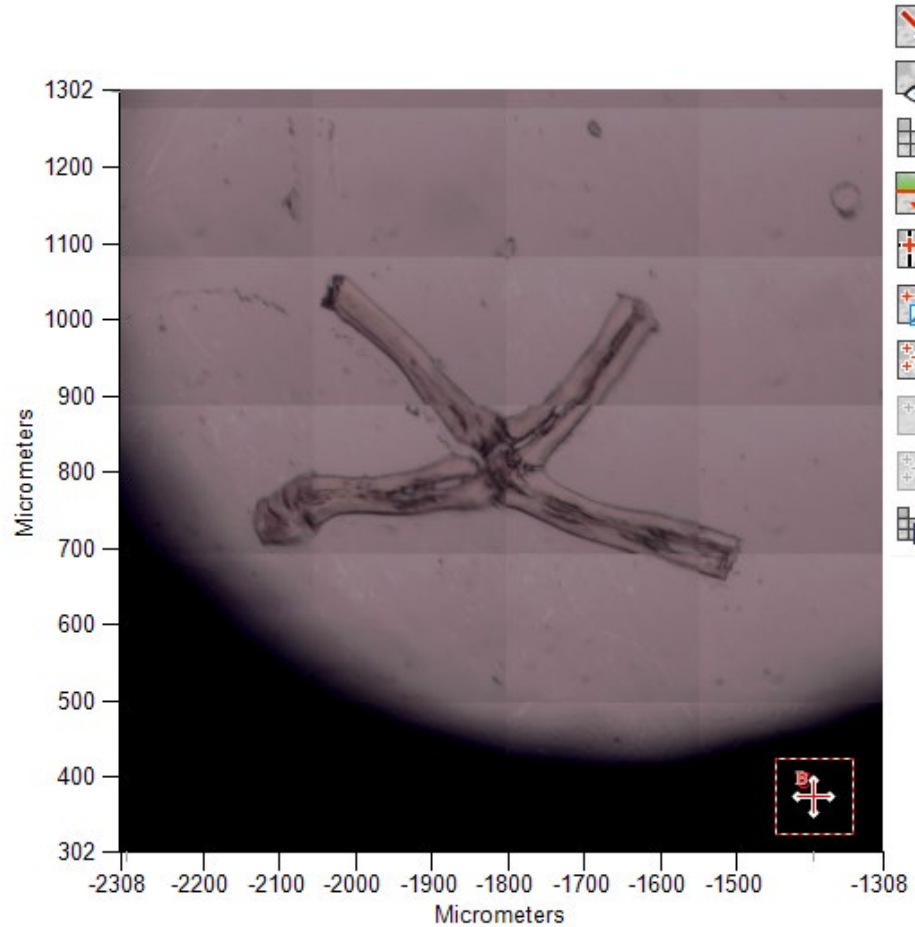


Polymer fragments 50-150 μm



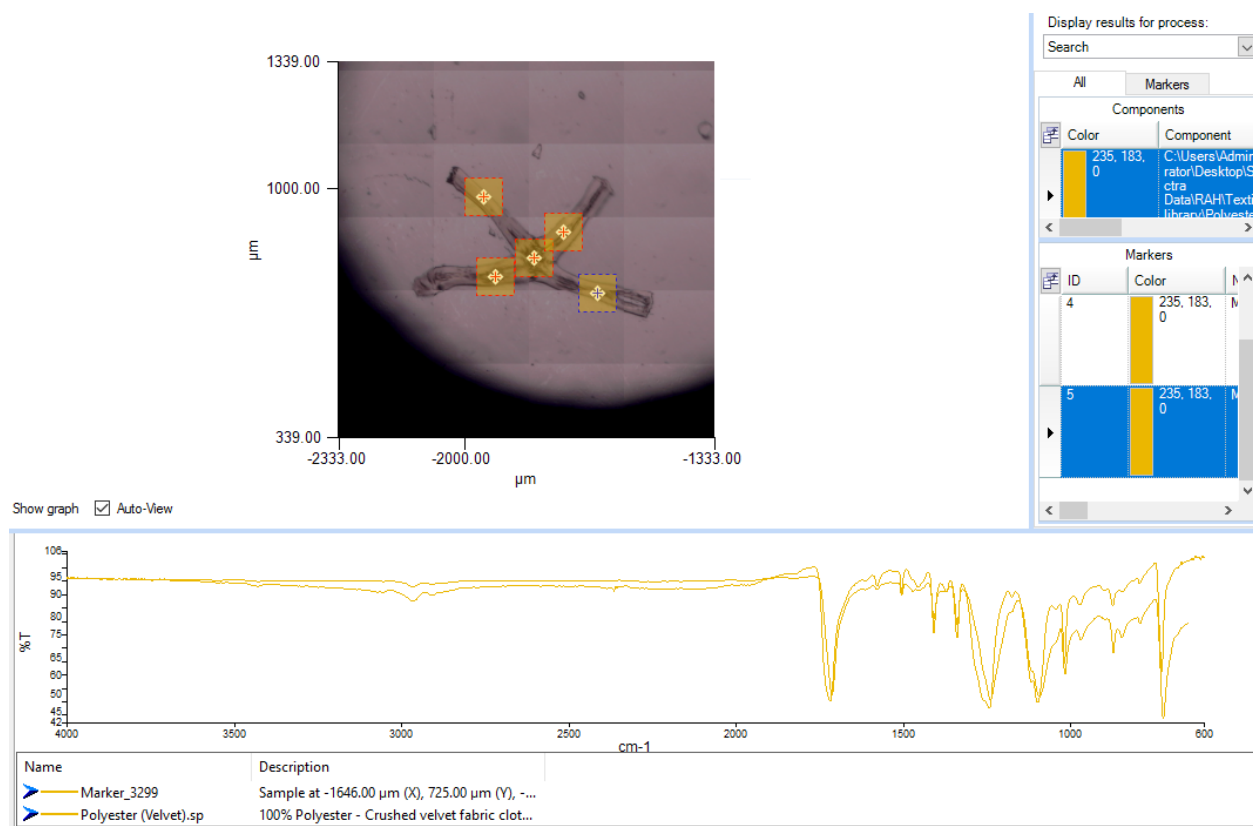
Fibers 250-500 μm

Stage View



Fibers 50-150 μm

RDS: 9.7% by
hand, <3% with
machine



Pill number	IN	OUT
9	30	29
10	28	24
11	31	29
12	30	24
13	30	30
14	30	24
15	30	27
16	30	24
17	30	30
18	30	28
X	29.9	26.9
SD	0.73	2.64
RSD	2.4 %	9.8 %

Pills for QA/QC

Calibration curves

Easy to handle

Suitable for several methods

- *Visual*
- *FTIR*
- *Raman*
- *Pyr-GC/MS*

Might be useful for QA/QC studies



Logistics for the European method evaluation study

- Labs can sign up when interlab registration opens on www.quasimeme.org
- Stay in touch via the QUASIMEME microplastics interlab mailing list quasimeme@wur.nl
- Each round has a small registration fee.

THANKS

ILC TEAM



REFERENCE MATERIAL TEAM



Bert van Bavel



Rachel Hurley



Luca Nizzetto



Nina T.
Buenaventura



Elena Martinez-
Frances



James D. Berg

This study will be coordinated by Dr. Louise van Mourik and Prof. Jacob de Boer, Vrije Universiteit, Amsterdam, The Netherlands. Both of them are highly experienced in the organization of large, international interlaboratory studies. Materials will be provided by the research group of Prof. Bert van Bavel, NILU, Oslo, Norway. Data management and statistics for this exercise will be developed and provided by QUASIMEME (Quality Assurance of Information in Marine Environmental Monitoring in Europe) (Wim Cofino, Steven Crum and Esther van de Brug). QUASIMEME operates Proficiency Testing Studies for institutes making chemical measurements in the aquatic environment worldwide. As part of the improvement programme, QUASIMEME co-operates with centers of excellence to provide workshops for discussion, and “hands on” experience to complement the development programmes in the Laboratory Performance Studies.

Participation Fee

The fee for participation in this study will be 750 Euro per round. In case a pre-payment is made for all three rounds, the fee will be 2000 Euro in total for all three rounds. The samples will be dispatched after receipt of the fee.

Registration

Participants should register before 12th April 2019. To register, please return the 2019 application form DE-17 Microplastics, containing all details necessary, by email to the Quasimeme office (quasimeme@wur.nl) Suggestions with regard to the design of the study and the type of test materials are also welcome and could be added to your email. Upon receipt of your email you will receive a confirmation of your participation and an invoice