

Soil and land research funding platform for Europe

# **Project MISSOURI**

Microplastics in soil and groundwater: sources, transfer, metrology and Impacts



*European interlaboratory study Report on microplastics in soil September 2021* 

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#### **SOILveR in brief**

The SOILveR platform strongly believes in the need for integrated soil and land research and knowledge exchange in Europe. We acknowledge the added value of coordinating, co-funding and disseminating crossborder soil and land management research. SOILveR is a self-financed platform. The platform members have a common interest in sharing and implementing integrated multidisciplinary research. SOILveR builds on the experiences from other funding networks such as SNOWMAN and address knowledge needs identified by e.g. the Horizon 2020 project INSPIRATION and other initiatives as well as those proposed by the members of SOILveR.

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## Abstract

MISSOURI organized, together with WEPAL-QUASIMEME, the first interlaboratory study (ILS) on the analysis of microplastics (MPs) in soil. Two soil materials (sand and a real soil), prepared by MISSOURI, were spiked with common plastic polymers, both as single (PE) as well as a mixture of different polymers (PE, PMMA, PS). In total five soil materials were prepared. In total 58 laboratories participated the ILS and 25 laboratories reported data. The results showed that the analysis of MPs in soil is difficult, but no more difficult than sediments. First indications were found that the soil matrix does not interfere with the MP analysis. Quantification of MPs in soil on mass basis had lower relative standard deviations (RSDs) (41-80%) than on particle basis (128-162%), but in both cases the RSDs are too high for reliable quantification. Similar RSDs were found for single spiked MPs or a mixture of different polymers, demonstrating that the analysis of a mixture was no more difficult than the analysis of single MPs. Indications were found that not all MP polymers behave equally in glass bottles, so it was recommended to make wet soil materials in next interlaboratory studies to generate more homogeneous samples. It was also recommended to perform additional soil and/or sediment ILS studies using lower concentrations of MPs.

## **Abbreviations and acronyms**

ADEME	Agence de l'Environnement et de la Maîtrise de l'Énergie (France)						
CV Coefficient of variation							
ILS	Interlaboratory study						
Ineris	Institut national de l'environnement industriel et des risques (France)						
ISSEP	Institut Scientifique de Service Public (Belgium)						
MP	Microplastics						
PMMA	poly methyl methacrylate						
PE	polethylene						
PS	polystyrene						
pyr-GC-MS	Pyrolyse Gazeous chromatography- Mass spectrometer						
RSD	Relative standard deviation						
VU	Vrije Universiteit of Amsterdam, department Environment & Health (The Netherlands)						

# 1. WP3 – European interlaboratory study

## **Overview and organization**

For the first time an interlaboratory study (ILS) on microplastics (MP) in soil was organized. MISSOURI, in cooperation with WEPAL-QUASIMEME and NORMAN, organized the ILS in order to maximize the number of participants. Two soil materials (sand and a real soil) were prepared by MISSOURI partners and spiked with common plastic polymers both as single (PE) as well as a mixtures of different polymers (PE, PMMA, PS). The consortium provided five MP-spiked soil samples to the second round of the **interlaboratory study on the Analysis of Microplastics in Environmental Matrices** organized by WEPAL-QUASIMEME. In the second round, in addition to soil, the focus was on the characterization of microplastics in other environmental matrices as well such as tablets, sediment and fish. These matrices were prepared by WEPAL-QUASIMEME.

This first interlaboratory study on MP in soil allowed the comparison of existing methods and provided a first estimation of the laboratory uncertainty associated to the quantification and identification of microplastics in soils. The comparison of existing analytical methods on common soil samples at an international scale is an **innovation** that will help to understand the differences between analysis protocols for fractions below 2 mm.

The aim of this study was to respond to the need of laboratories to control the quality of microplastics analysis. The ILS provides information on the suitability of analytical methods as well as providing preliminary guidance for the establishment of harmonized Standard Operational Procedures (SOP). It will also contribute to the identification of main knowledge gaps and provided information on follow-up research in the field of microplastics sampling and characterization.

# 2.1 Organisation and time frame of ILS

Preparation of the five soil samples was carried out by the MISSOURI partners in 2020. Registration for the round 2 ILS on MP analysis was organized by WEPAL-QUASIMEME/NORMAN, and the registration was open till September 2020. In October 2020 the samples were dispatched to the laboratories, and the deadline for returning the data was January 2021. The statistical analysis of the data was performed by QUASIMEME, and these were reported in May 2021. Interpretation of soil data was carried out by the Vrije Universiteit of Amsterdam (VU). The results were discussed at an international workshop on 20-21 May 2021.

## 2.2 Soil selection and preparation

The soil was spiked by ISSEP during summer 2020. Two types of soil were individually mixed with white MP microspheres to provide two levels of MPs in soil.

Soil A was a synthetic silica: technical Fontainebleau Sand was provided by Filter Service. This sand contains no organic matter or other natural compounds that would adsorb MP on their surface. Grains size was below 350 µm, which was three times larger than the microspheres. This material was used as a reference sample and was compared to the real soil, sample B. The characteristics of soil A are given in Table 1.

Table 1: Soil characteristics of soil A.

Formula Soil A : SiO₂ Molecular Weight: 60.08 g/mol Boiling Point: 2 230 °C (1013 hPa) CAS Number: 7631-86-9 EINECS: 238-878-4

• Soil B was a real sandy soil collected on an industrial Walloon site. Its physical and chemical parameters are given in Table 2, and the organic matter level is considered as medium to high. The sandy soil was sieved to deliver 2 fractions:

- 250  $\mu m$  , in order to simplify the MP separation.

- 2 mm, is the fraction usually analysed by laboratories for analyses of chemical compounds, and should be more difficult to handle for the MP separation.

#### Interlaboratory study MP in soil

Tahle	2: Soil	characteristics	of soil B.
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Parameter	Unity	Sandy Real Soil – Soil B
рН	-	9.65
Dry matter	%	85.5
Clay	%	< 0.1
Organic matter	%	4.6
Metals/ metalloids		
Cr(VI)	mg/kg d.w.	0.35
As	mg/kg d.w.	11.7
Cd	mg/kg d.w.	0.61
Cr	mg/kg d.w.	174
Cu	mg/kg d.w.	34
Hg	mg/kg d.w.	0.21
Ni	mg/kg d.w.	45
Pb	mg/kg d.w.	73
Zn	mg/kg d.w.	591
Polycyclic Aromatic Hydrocarbon	S	
Naphtalene	mg/kg d.w.	0.11
Acenaphtylene	mg/kg d.w.	< 0.01
Acenaphtene	mg/kg d.w.	0.02
Fluorene	mg/kg d.w.	0.03
Phenanthrene	mg/kg d.w.	0.90
Anthracene	mg/kg d.w.	0.09
Fluoranthene	mg/kg d.w.	1.35
Pyrene	mg/kg d.w.	1.21
Benzoanthracene	mg/kg d.w.	0.78
Chrysene	mg/kg d.w.	1.10
Benzo(b)fluoranthene	mg/kg d.w.	1.09
Benzo(k)fluoranthene	mg/kg d.w.	0.42
Benzo(a)pyrene	mg/kg d.w.	0.46
Dibenzoanthracene	mg/kg d.w.	0.10
Benzo(g,h,i)perylene	mg/kg d.w.	0.57
Indeno(1,2,3-c,d)pyrene	mg/kg d.w.	0.40
Sum 16 PAH	mg/kg d.w.	8.6
BTEX		
benzene	mg/kg d.w.	< 0.075
toluene	mg/kg d.w.	< 0.25
ethylbenzene	mg/kg d.w.	< 0.15
mp-xylene	mg/kg d.w.	< 0.4
o-xylene	mg/kg d.w.	< 0.13

d.w.: dry weight

#### Protocol of soil spiking

For the preparation of soil samples, a distributor Resch was used (Figure 1). First 200 g of soil was divided equally in 10 bottles. MP microspheres were added in bottles in the same way. Each bottle was mixed one hour in a flipper mixer (Figure 1).

#### Interlaboratory study MP in soil





Distributor Resh Flipper mixer Figure 1: Distributor Resh and flipper mixer used for preparation of the soil samples.

#### Spiked microplastic microspheres

Microspheres of polymethyl-methacrylate (PMMA), polystyrene (PS), and polyethylene (PE) were used for spiking. Microspheres were provided by Cosphere (USA) and details on their formula, main application, density, and size used for the ILS are given in Table 3. MP size were between 85 and 106  $\mu$ m which is the medium of MP size definition between 100 nm and 5 mm.

Microplastics	Type Resin code		MP size Pictures (electron microscopy- ISSEP)
PMMA – Polymethyl-methacrylate $CH_3$ $CH_2-C-D_n$ C=0 O $CH_3$		glasses (lenses), glazing, ruler, optical fiber, neon signs. Also called plexiglass	White microspheres 1.2g/cc . 90-106µm
Polystyrene PS +CH <sub>2</sub> -CH+	ê	CD cases, yogurt containers, cups, plates, cutlery, hinged takeout containers (clamshells), electronic housings, building isolation, medical products, packing, foamed coolers	1.07g/cc . <b>85-105µm</b>
Polyethylene PE + $CH_2$ - $CH_2$ +	Polyolefines for HDPE	LDPE (low density) : bottles for shampoo, bags, films HDPE (high density) : rigid storage containers	0.96g/сс. <b>90-106µm</b>

Polypropylene (PP) microspheres could not be obtained for the ILS as they could not be provided by MP providers (Cospherics LCC, Borealisgroup, Polysciences, Spherotech, Nexeo plastics). PP were not available in the microsphere-form with similar sizes as the PMMA, PS, and PE. The microspheres used for soil spiking were also used by QUASIMEME for spiking the other environmental samples for this ILS.

#### MP-spiked soil samples

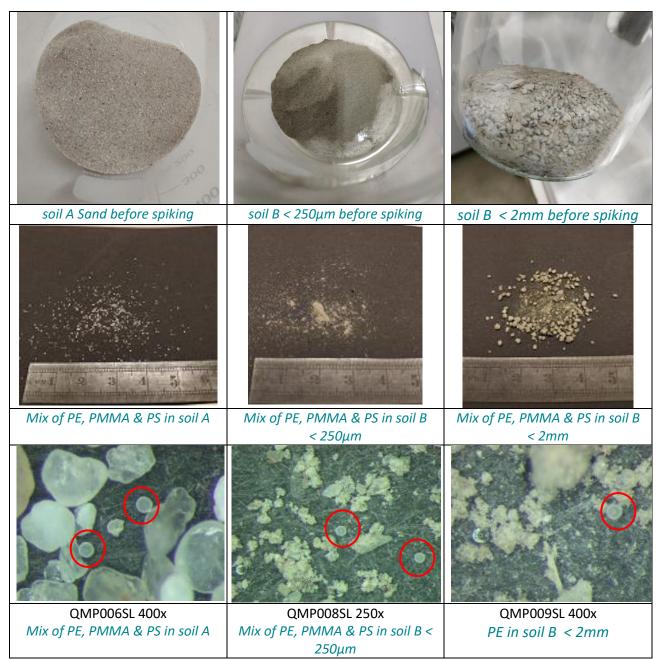
For each batch of soil (1 to 5), 90 bottles were prepared by ISSEP. A total of 450 bottles were sent in September 2020 to VU, which performed the homogeneity study, before sending the samples to QUASIMEME for distribution to the ILS laboratories.

Table 4: Spiked soil samples (1-5) used for the ILS study. Amount of soil, type of MP spiked and spiked MP quantity to each bottle.

Batch number	ILS name	Matrix	Amount of soil in each bottle	MP microspheres	Spiked quantity of MP in each bottle
1	QMP005SL	Sand (soil A)	20 g	PE	40 mg
	QMP006SL	Sand (soil A)	20 g	PE	10 mg
2				PMMA	15 mg
			PS	1.5 mg	
3	QMP007SL	Real sandy soil 250 µm (soil B)	20 g	PE	25 mg
4	4 QMP008SL Real sandy soil 250 μm (soil B)		20 g	PE	10 mg
				PMMA	15 mg
				PS	1.5 mg
5	QMP009SL	Real sandy soil (soil B) (25% 250 μm + 75% 2 mm)	20 g	PE	10 mg

### 2.3 MP-spiked soil samples

Soil samples were analysed by electron microscopy (Figure 1) showing the soils before and after spiking.



*Figure 1 : Soil A and B before and after spiking with microplastics microspheres. At the bottom three photos are taken by electron microscopy.* 

## 2.4 Homogeneity test

To determine if the spiked MP particles were homogeneously distributed in the soil samples, eight samples from each matrix were analysed by pyrolysis GC-MS (pyr-GC-MS) after extracting the MPs from the whole sample by Accelerated Solvent Extraction (ASE). This test was done by VU in December 2020.

The coefficient of variation (CV) of three samples are given in Figure 2. Firstly, the CVs for each MP are similar between the sand (soil A) and the real sandy soil (soil B) samples. Secondly, the homogeneity of the spiked PE and PS particles is satisfactory but seems high for the PMMA particles. At that time it was unclear why the CV of PMMA was much higher than the PE and PS as all MPs were spiked simultaneously. One option could be that PMMA particles were not equally distributed in the bottles anymore after transport, and are partly bound to the glass wall of the bottles as PMMA particles could have an electric charge. This needs further investigation, see below results of the ILS. Based on the data it was concluded that the spiking was successful and suitable for the ILS study.

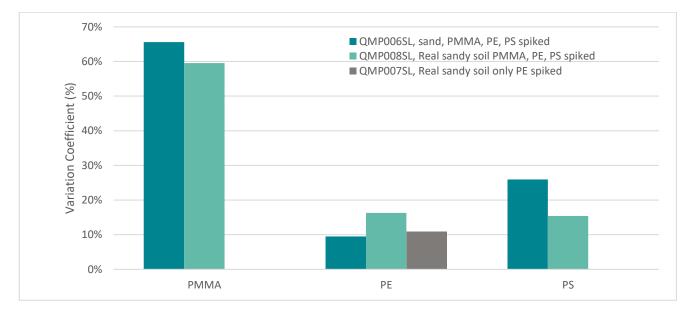
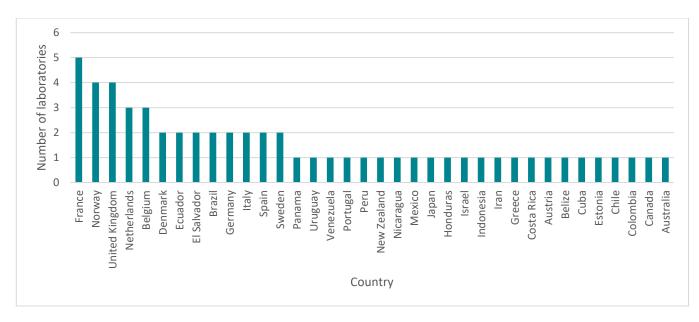


Figure 2 : Coefficient of variation (%) of the MP analysis in the spiked soil samples used for the interlaboratory study. Sample QMP006SL : soil A sample spiked with PMMA, PE and PS. Sample QMP008SL : soil B spiked with PMMA, PE, and PS. Sample QMP007SL : soil B spiked with PE particles.

### 2.5 Participating laboratories

In total 58 laboratories participated in the second round ILS of which 33 are from Europe, 19 from America, 4 from Asia, and 2 from Oceania. The number of laboratories per country are given in Figure 3, which range from 1 to 5. For the three funder countries involved in the MISSOURI project (France, Belgium and the Netherlands), there are 11 laboratories. In total, 25 laboratories reported data on MPs in soil.



*Figure 3 : Number of laboratories per country participating in the interlaboratory MP study (round 2).* 

## 2.6 Statistical analysis

Statistical analysis of the ILS data was performed by WEPAL-QUASIMEME. The statistical analysis consists of sophisticated methods to calculate the mean and the standard deviation based on Cofino et al. (2000) and Molenaar at al. (2018). As the participating laboratories reported single data no information is available about the underlying probability function. WEPAL-QUASIMEME use the so-called Normal Distribution Approximation (NDA) method. The NDA model is used to calculate the mean and standard deviation using all reported data when at least 4 results are left after removal of reported 'lower than' (<) and 0 (=zero) values. No outliers are removed. The NDA mean is centered around the highest density of values, and the assigned value represents the consensus value of *all* data. Although *all* data are included in the assessment, those values that lie some distance from the NDA mean contribute less to the mean than values which occur at or near the mean.

In some instances it is not possible to set an assigned value, and an indicative value is given. No assessment of laboratory performance is given where an indicative value is set. An overview of the assessment, with explanation, decision flowcharts and examples, is given in the paper *Assessment Rules for the evaluation of the QUASIMEME Laboratory Performance Studies Data*, available on the QUASIMEME website, <u>www.quasimeme.org</u>. The acronyms provided in the data reports given in the results and discussion section and Appendices are listed in Table 5.

Table 5: Acronyms used in the reporting of data in appendix 1 and appendix 2.

Total	Total number of particles						
Code	Sample filtration						
-	None						
QF	Quarz filter QM-A, 2.2 μm pore size						
SF	Filtration 0.7 μm						
Z	Others (Please inform quasimeme about details)						
Code	Density separation						
-	None						
ZN	ZnCl2 solution						
NA	NaCl (120 g/l)						
NL	Nal solution (density 1.8 g/ml)						

Z Others (Please inform quasimeme about details)

#### Code Clean-up technique

- None
- HO H2O2 (30%) at 60°C for 24 h
- HP H2O2 (30%) at 50 °C for max 5 days
- HN HNO3 (20%)
- ED Enzymatic digestion (protease, lipase, amylase)
- Z Others (please inform quasimeme about details)

#### Code Polymer identification and quantification

- μF μFTIR
- AF ATR-FTIR
- RA Raman
- PY Pyr-GC/MS
- μR μ-Raman
- MI Microscopy (manual counting)
- GR Gravimetric
- TD TED-GC-MS
- Z Others (please inform quasimeme about details)

#### Code Stdev and technical aspects

- Z Please inform quasimeme about stdev of the method used and about smallest detectable particle
- size

#### Total mass of plastic particles

#### Code Sample filtration

- None
- QF Quarz filter 2.2 µm pore size
- SF Filtration (0,7 µm)
- Z Others (please inform QUASIMEME about the details)

#### Code Density separation

- None
- ZN ZnCl2 solution in separation funnel
- NA NaCl (120 g/l)
- NL Nal solution (density 1.8 g/ml)
- Z Others (Please inform QUASIMEME about the details)

#### Code Clean-up technique

- None
- HO H2O2 (30%) at 60°C for 24 h
- HP H2O2 (30%) qat 50 °C for max 5 days
- HN HNO3 20%
- ED Enzymatic digestion (protease, lipase, amylase)
- Z Others (Please inform QUASIMEME about the details)

#### Code Polymer identification and quantification

- uF µFTIR, micro Fourier Transform infrared spectroscopy
- AF ATR-FTIR, Attenuated total reflection
- RA Raman spectroscopy Fourier Transform InfraRed
- PY Pyr-GC/MS, pyrolysis gas chromatography mass spectrometry
- μR μ-Raman spectroscopy
- MI Microscopy (Manual counting)

GR Gravimetric

- TD TED-GC-MS, Thermal Extraction and –Desorption gas chromatography mass spectrometry
- Z Others (Please inform QUASIMEME about the details)
- Code Stdev and technical aspects

```
Z Please inform quasimeme about stdev of the method used and about smallest detectable particle
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size
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# 3. Results and discussion

## **3.1 Participating laboratories and used methods**

In total 25 laboratories participated in the ILS on MPs in soil, of which 19 reported on particle number basis and 10 reported on mass basis. The laboratories used mainly density separation, filtration and pressurized liquid extraction (PLE) to separate or extract the MPs from the soil matrix. Some laboratories used a cleanup method, such as hydrogen peroxide to clean the sample extracts. Various techniques have been used to detect and identify the MPs in the soil samples. The main techniques used are  $\mu$ FTIR, pyrolysis gas chromatography mass spectrometry (pyr-GC/MS), and microscopy manual counting. Less used were gravimetric determination and ATR-FTIR.

## 3.2 MPs in soil

All raw reported data of MPs by the laboratories are provided in Appendix 1 on particle basis, and on mass basis in appendix 2, including the statistical analysis and graphical representations. An overview of the number of laboratories reported on particle basis and on mass basis, including the assigned values, robust standard deviation (SD), and robust relative standard deviation (RSD), are given in Tables 6 and 7, respectively.

Sample	Number of labs	Assigned Value	Robust SD of study	Robust RSD of study
Sand, QMP005SL	19	3.6E+05	5.6E+05	157%
Sand, QMP006SL	18	5.0E+05	6.5E+05	132%
Real soil, QMP007SL	19	8.0E+05	1.0E+06	128%
Real soil, QMP008SL	17	3.6E+05	5.9E+05	162%
Real soil, QMP009SL	17	7.5E+05	1.0E+06	135%

Table 6: Assigned value of **total number** of polymer particles (No. p/kg) found in the five spiked soil samples. SD: standard deviation, RSD: relative standard deviation.

Table 7: Assigned **concentration** (mg/kg) of polymers found in the five spiked soil samples. SD: standard deviation, RSD: relative standard deviation.

Sample	N labs	Assigned Value	Robust SD of study	Robust RSD of study	
Sand, QMP005SL	10	1281	707	55%	
Sand, QMP006SL	10	609	609 485		
Real soil, QMP007SL	10	1029	703	68%	
Real soil, QMP008SL	10	478	251	51%	
Real soil, QMP009SL	10	502	204	41%	

The RSDs of the total number of polymer particles in the five soils varied between 128% and 162%, but lower RSDs were found on mass basis (41% to 80%). The participants indicated that the high number of particles present in the samples made it more difficult to count the total number of particles. The RSD values of all samples are unsatisfactory and showed that the analysis of MPs is still difficult and is not at the same quality level as for some well-known contaminants such as PCBs. The RSD values of the soil samples were more or less comparable with the RSD values of the sediment samples reported in the second round, which showed that the analysis of soil in no more difficult than sediment.

For the individual polymers similar variations as for the total polymer particles were found, which is illustrated for a sand (QMP006SL) and a real soil sample (QMP008SL) that were spiked with the mixture of PE, PS, and PMMA (Figure 5); Appendix 3 shows the figures for the other three samples (QMP005SL, QPM007SL, QPM009SL). A large variation in MP levels is found between the laboratories for all samples. In general, the reported values per laboratory showed large deviations from the spiked values (solid lines in Figure 5). The results showed that the soil matrix component (real sandy soil vs clean sand) had no influence on the RSDs as these were similar between these two types of soil. This suggests that most of the matrix was efficiently removed before the analysis.

The mean values (assigned values) and spiked values are given in Figure 6. The mean values for PE and PS are a little lower than the spiked values (Figure 6), which is mainly due to the fact that some laboratories reported too low values and some reported too high values. This in contrast to PMMA were the reported values are much lower than the spiked values in all three samples. As PE, PS and PMMA were spiked together, it suggests that PMMA behaves differently than PE and PS in glass bottles, as was also indicated from the homogeneity study. Additional analysis showed that a large amount (ca. 20%) of PMMA particles were still present in the glass bottles after removing the sample from the glass bottle for analysis, while PE and PS particles were almost not present. This indicates less homogeneous distribution of PMMA microspheres in the bottles, which could be due to the transport of the samples. This observation needs further investigations.

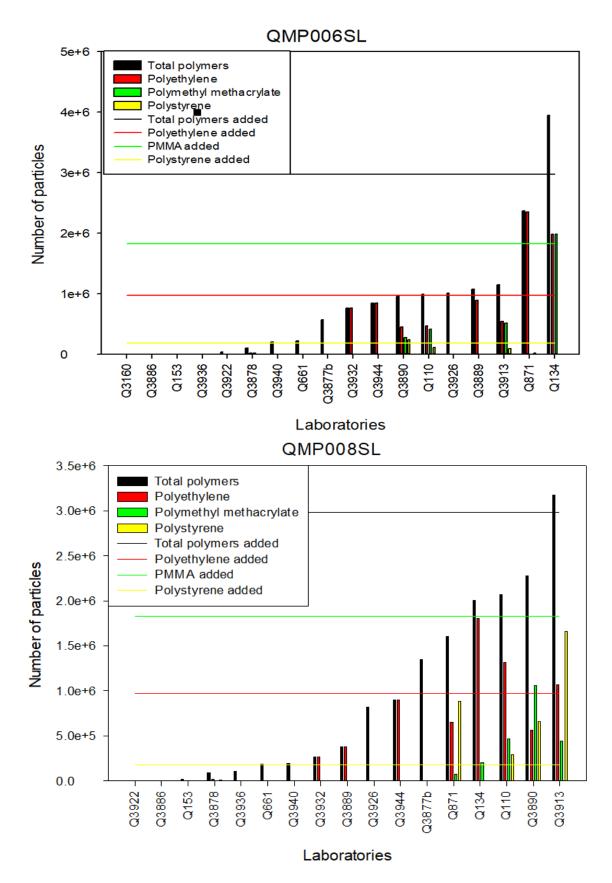


Figure 5: **Total number** of MPs reported in spiked sand (QMP006SL) and a real sandy soil (QMP008SL) samples. On the horizontal axis the laboratory codes are given and on the vertical axis the number of particles (No. p/kg) reported. Shown are the number of total polymers, PE, PPMA and PS. The spiked amounts of PE, PMMA, and PS are given as red, green and yellow lines.

#### Interlaboratory study MP in soil

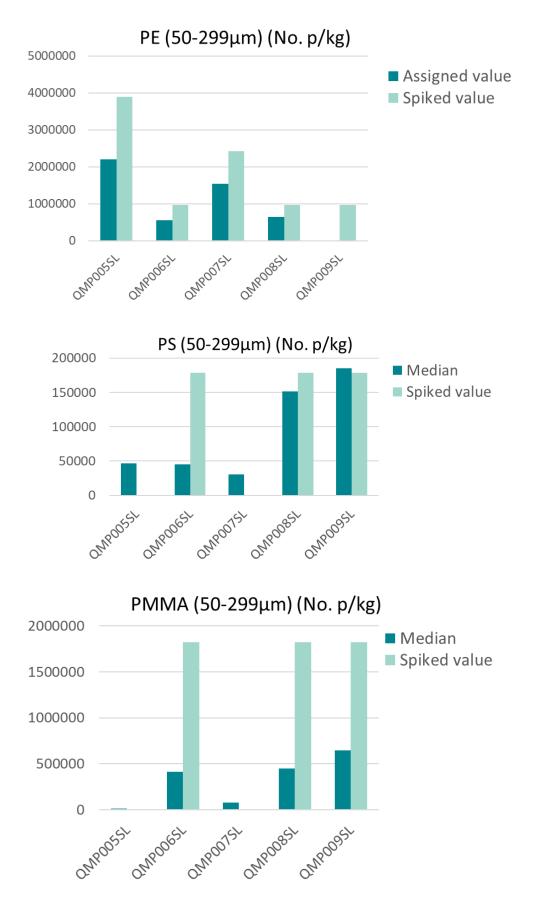


Figure 6: **Assigned** or **median values** and spiked levels of PE, PS, and PMMA on particle basis (No. *p/kg*) in the five soil samples. Note, sample QMP005SL and QMP007SL were not spiked with PS or PMMA only with PE.

## 5. Conclusions and recommendations

This first ILS of MPs in soil showed that the analysis is difficult but no more difficult than for instance sediments. First indications were found that the analysis methods remove the soil matrix and does not interfere with the MP analysis. Quantification of MPs in soil on mass basis had lower relative standard deviations (41-80%) than on particle basis (128-162%), but in both cases the RSDs are too high for a reliable quantification. Similar RSDs were found for single spiked MPs and mixtures of MPs, showing that the analysis a mixture of MPs was no more difficult than the analysis of single MPs. Indications were found that PMMA particles behave differently than PE and PS in glass bottles, resulting in less homogeneous samples, but this needs further confirmation.

It is highly recommended to set-up follow up ILS studies on soil and/or sediment. One recommendation for a new ILS study is to prepare materials at lower MP concentrations. The ILS workshop concluded that soil and sediment materials on dry samples are less homogeneous in bottles than on wet samples. Therefore, it was recommended to make wet soil and sediment samples for a next round of interlaboratory studies. Serious attempts should be made to reduce the within-homogenity of the test materials, and additional studies are needed to investigate the distribution of different MP polymer types in soil and sediment materials. The preparation of appropriated polymers in the micro and nano size is another recommendation to be able to spike samples with a wider range of polymers.

## References

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# 7. Appendices

# Appendix 1: Total number of particles found in the ILS soil samples.

Explanation of the acronyms given in the reported data are given in section 2.6, and the table below.

Abbreviation	Explanation						
MIC	method indicating code						
NDA mean	Consensus value						
MAD	median absolute deviation						
NDA st dev.	standard deviation						
Coeff Var	NDA relative standard deviation (%)						
-	not calculated						

Used abbreviations and symbols

#### Total number of particles Summary Statistics

Sample/ Determinand	Assigned Value	Units	Total Error	NDA st.dev	NDA rel. st.dev (%)	Nobs numerical	Nobs LCV	Median	MAD	Model Mean	Uncer- tainty
QMP005SL											
PE (50-299 μm)	2206838	(No. p/kg)		2603246	118.0	7	0	2164169	1887279	2206837.7	1229918.1
Polyethylene total (HD+LD)	1598262	(No. p/kg)		1946650	121.8	10	0	1643198	1410187	1598261.7	769480.8
Total polymers (50-299 μm)	139035	(No. p/kg)		392264	282.1	7	0	321400	320452	139035.1	185327.5
Total polymers	355062	(No. p/kg)		558094	157.2	19	0	398386	397651	355062.0	160044.4

Sample/ Determinand	Assigned Value	Units	Total Error	NDA st.dev	NDA rel. st.dev (%)	Nobs numerical	Nobs LCV	Median	MAD	Model Mean	Uncer- tainty
QMP006SL											
PE (50-299 μm)	555469	(No. p/kg)		301963	54.4	7	0	542563	213128	555468.7	142664.0
Polyethylene total (HD+LD)	607261	(No. p/kg)		393812	64.9	9	0	755691	288198	607261.1	164088.3
Total polymers (50-299 μm)	45269	(No. p/kg)		117332	259.2	9	0	93800	92738	45269.2	48888.2
Total polymers	496030	(No. p/kg)		652157	131.5	18	0	661599	473479	496030.2	192143.7

Sample/ Determinand	Assigned Value	Units	Total Error	NDA st.dev	NDA rel. st.dev (%)	Nobs numerical	Nobs LCV	Median	MAD	Model Mean	Uncer- tainty
QMP007SL											
PE (50-299 μm)	1539218	(No. p/kg)		1145872	74.4	7	0	1871412	795440	1539217.6	541373.5
Polyethylene total (HD+LD)	1374606	(No. p/kg)		1224676	89.1	10	0	1627948	914250	1374606.4	484095.6
Total polymers (50-299 μm)	85367	(No. p/kg)		144442	169.2	7	0	112985	112884	85367.1	68242.2
Total polymers	799504	(No. p/kg)		1023678	128.0	19	0	706184	695126	799503.9	293559.8

#### Total number of particles Summary Statistics

Sample/ Determinand	Assigned Value	Units	Total Error	NDA st.dev	NDA rel. st.dev (%)	Nobs numerical	Nobs LCV	Median	MAD	Model Mean	Uncer- tainty
QMP008SL											
PE (50-299 μm)	654384	(No. p/kg)		776650	118.7	7	0	559961	540502	654383.8	366932.8
Polyethylene total (HD+LD)	614109	(No. p/kg)		585119	95.3	10	0	605664	399730	614109.3	231288.8
Total polymers (50-299 μm)	55246	(No. p/kg)		91103	164.9	7	0	87838	72308	55245.9	43042.1
Total polymers	364489	(No. p/kg)		590781	162.1	17	0	378108	377658	364489.2	179106.8

Sample/ Determinand	Assigned Value	Units	Total Error	NDA st.dev	NDA rel. st.dev (%)	Nobs numerical	Nobs LCV	Median	MAD	Model Mean	Uncer- tainty
QMP009SL											
Polyethylene total (HD+LD)	801658	(No. p/kg)		363741	45.4	8	0	864144	264141	801657.8	160752.1
Total polymers	752362	(No. p/kg)		1014152	134.8	17	0	707567	695069	752361.8	307460.1

Sample	QMP005SL	QMP006SL	QMP007SL	QMP008SL	QMP009SL	МІС
ABS (50-299 μm)	((No. p/kg))					
Q3878	59350	4750	7900	17838	-	$ZA ZA  -  \mu F ZA$
ABS (300-5000 μι		467	758		150	
Q3160 Q3878	446 2000	407	758 500	- 811	152	ZA ZA ZA MI ZA ZA ZA  - µF ZA
03070	2000		500	011		
Acrylon.Butadi.St						
Q3160	446	467	758	-	152	
Q3878	61350	4750	8400	18649	-	ZA ZA  - µF ZA
PA (50-299 μm) (	(No. p/kg))					
Q3878	21650	16500	10600	7027	-	ZA ZA  - µF ZA
Q3886	-	151	-	-	-	$ZA ZA  -  \mu F ZA$
PA (300-5000 μm	$(N_0, n/k_0)$					
Q3878	500	100	-	541	-	ZA ZA  - µF ZA
20070		200		0.1		
Polyamide total (						
Q3878	22150	16600	10600	7568	-	ZA ZA  - µF ZA
Q3886	-	151	-	-	-	ZA ZA  - µF ZA
PC (50-299 μm) (	(No. p/kg))					
Q3878	1750	11350	450	-	-	ZA ZA  - µF ZA
DC (200 5000	\//NI=/I_=\\					
<b>PC (300-5000 μm</b> Q3878	) ((NO. p/kg))	_	50.0	_	_	77 77
03878			50.0			ZA ZA  - µF ZA
Polycarbonate to	tal ((No. p/kg))					
Q3878	1750	11350	500	-	-	ZA ZA  - µF ZA
PE (50-299 μm) ((	No. p/kg))					
Q110	2164169	467493	1910215	1317671	509805	ZA   ZA   μF   ZA
Q134	6733087	1975965	2492571	1805349	2165822	ZA ZA  -  ZA ZA
Q3878	68750	14700	29750	19459	-	ZA ZA  - µF ZA
Q3886	-	-	-	151	-	ZA ZA  - µF ZA
Q3887	4057296	-	2666852	-	-	ZB   ZB   ZB   ZA   ZB
Q3889	-	889747	-	-	-	- ZA µF
Q3890	1277344	448333	721212	559961	690202	ZC   ZB   ZA   ZA   ZC
Q3913	276890	542563	1871412	1068277	1277901	ZB ZB  -  ZB ZB
Q3932	3114517	755691	706184	268818	971017	ZA ZA  - AF ZA
	========	:===== S <sup>:</sup>	tatistical Results	5 ==========	======	
NDA mean	2206838	555469	1539218	654384		
NDA st dev	2603246	301963	1145872	776650	-	
Coeff Var (%)	118.0	54.4	74.4	118.7	-	
N	7	7	7	7	5	
Median	2164169	542563	1871412	559961	971017	
MAD	1887279	213128	795440	540502	306884	
Category	-	-	-	-	2	
Added Number	3896050	974000	2435050	974000	974000	
	=======			============	=====	

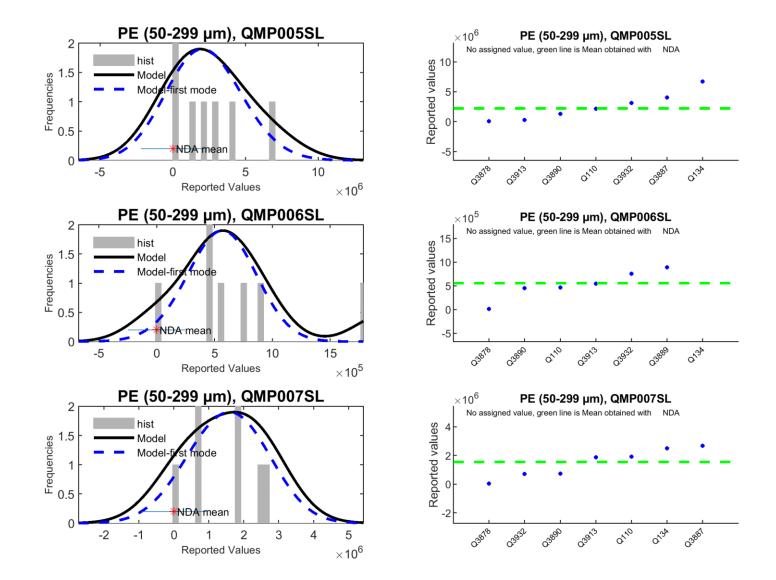
Sample	QMP005SL	QMP006SL	QMP007SL	QMP008SL	QMP009SL	
ΡΕ (300-5000 μm)	) ((No. n/kg))					
Q3878	14200	-	-	-	-	
Q3889	189133	-	668134	378108	757271	
Delivate la seta						
Polyethylene tota Q110	al (HD+LD) ((No. 2164169	. <b>р/кg))</b> 467493	1910215	1317671	509805	
Q134	6733087	1975965	2492571	1805349	2165822	
Q3878	82950	14700	29750	19459	-	
Q3886	-	-	-	151	-	
Q3887	4057296	-	2666852	-	-	
Q3889	189133	889747	668134	378108	757271	
Q3890 Q3913	1289063 276890	448333 542563	721212 1871412	559961 1068277	690202 1277901	
Q3932	3114517	755691	706184	268818	971017	
Q3944	1079010	838693	1384484	896683	698081	
Q871	1997333	2351675	153209698	651367	2379293	
		====== S	tatistical Results	5 =========		
NDA mean	1598262	607261	1374606	614109	801658	
NDA st dev	1946650	393812	1224676	585119	363741	
Coeff Var (%)	121.8	64.9	89.1	95.3	45.4	
N	10	9	10	10	8	
Median	1643198	755691	1627948	605664	864144	
MAD	1410187	288198	914250	399730	264141	
Added Number	3896050	974000	2435050	974000	974000	
	=======					
PET (50-299 μm)	((No. p/kg))					
Q3878	2550	1150	50.0	541	-	
PET (300-5000 μn	n) ((No. p/kg))					
Q3878	150	350	-	-	-	
Polyethylenetere						
Q3878	2700	1500	50.0	-	-	
PLA (50-299 μm)						
Q3878	14400	2100	1050	-	-	
PLA (300-5000 μn	n) ((No. p/kg))					
Q3878	-	400	-	-	-	
Polyactic Acid tot						
Q3878	14400	2500	1100	-	-	

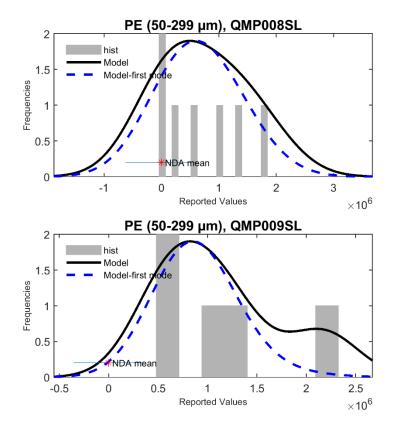
PMMA (50-299μm) ((Γ	No.p/kg))					
Q110	-	411698	-	463229	943433	ZA   ZA   ZA   µF   ZA
Q134	-	1975965	-	200594	352581	ZA   ZA   -   ZA   ZA
Q3878	15000	17400	450	14054	-	ZA ZA  - µF ZA
Q3890	-	267778	159091	1058994	1180115	ZC   ZB   ZA   ZA   ZC
Q3913	-	514500	-	445889	332254	ZB ZB  - ZB ZB
	========	==== Statistic	al Results (no N	NDA) ======		
Ν	1	5	2	5	4	
Median	15000	411698	79771	445889	648007	
MAD	-	143920	79321	245295	305590	
Category	3	2	3	2	2	
Added number	-	1826250	-	1826250	1826250	
	========				=====	
PMMA (300-5000μm)	((No p/kg))					
Q3878	((NO: P/ Kg)) -	250	_	-	_	ZA ZA  - µF ZA
03878		250				
Polymethylmethacryla	ate total ((No	. p/kg))				
Q110	-	411698	-	463229	943433	ΖΑ   ΖΑ   ΔΑ   μF   ΖΑ
Q134	-	1975965	-	200594	352581	ZA ZA  - ZA ZA
Q3878	15000	17650	450	-	-	$ZA ZA  -  \mu F ZA$
Q3890	-	267778	159091	1058994	1180115	ZC   ZB   ZA   ZA   ZC
Q3913	-	514500	-	445889	332254	ZB ZB  - ZB ZB
Q871	-	6856	-	76505	265782	
			al Results (no N			
N	1	6	2	5	5	
Median	15000	339738	79771	445889	352581	
MAD	-	248425 2	79321	245295	86799	
Category Added number	3	1826250	3	2 1826250	2 1826250	
Added Humber		=======================================	=======================================	=======================================	======	
PP (50-299 μm) ((No. ι	o/kg))					
Q3878	64000	4950	18650	12162	-	ZA ZA  - µF ZA
Q3886	-	-	-	-	49.8	$ZA   ZA   -   \mu F   ZA$
/	<i>a</i>					
PP (300-5000 μm) ((No						
Q3878	1700	-	-	-	-	ZA ZA  - µF ZA
Q871	-	3428	-	-	-	
Polypropylene total ((	No. p/kg))					
Q3878	65700	4950	18650	12162	-	ZA ZA  - µF ZA
Q3886	-	-	-	-	49.8	$ZA ZA  -  \mu F ZA$
Q871	-	3428	-	-	-	
	<i>k</i> . 11					
PS (50-299 μm) ((No. μ	o/kg))					
Q110	-	110425	-	291951	185175	ZA ZA ZA µF ZA
Q3878	46400	6750	14750	10270	-	ZA ZA  - µF ZA
Q3886	-	50.6	-	252	99.5	$ZA ZA  -  \mu F ZA$
Q3913	-	84191	45924	1662796	1243823	ZB ZB  - ZB ZB
		==== Statistic	al Results (no N	NDA) ======		
Ν	1	Statistic 4		4 (NDA)	3	
Median	46400	45471	30337	151111	185175	
MAD	+0+00	42070	15587	145849	185076	
Category	3	42070	15587	145845	3	
	5	2	5	4	5	

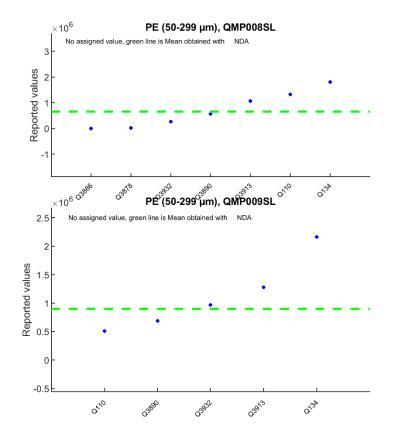
Added Value	-	178750	-	178750	178750	
Sample Q	MP005SL	QMP006SL	QMP007SL	QMP008SL	QMP009SL	МІС
<b>PS (300-5000 μm) ((N</b> Q3878	l <b>o. p/kg))</b> 3900	-	-	811	-	ZA ZA  - µF ZA
<b>Polystyrene total ((N</b> Q110	o. p/kg)) _	110425	-	291951	185175	ZA   ZA   UF   ZA
Q3878	50300	6750	14750	11081		$ZA ZA  -  \mu F ZA$
Q3886	-	50.6		252	99.5	$ZA ZA  - \mu F ZA$
Q3890	-	236111	154545	655706	164265	ZC   ZB   ZA   ZA   ZC
Q3913	-	84191	45924	1662796	1243823	ZB ZB  - ZB ZB
Q871	-	10284	-	880881	5461	
	=======	===== Statist	ical Results (no	NDA) ======	======	
Ν	1	6	3	6	5	
Median	50300	47238	45924	473829	164265	
MAD	-	43837	31174	434900	158804	
Category	3	2	3	2	2	
Added Value	-	178750	-	178750	178750	
PUR (50-299 μm) ((Ν						
Q3878	15200	8400	6000	5946	_	ZA ZA  - µF ZA
Q3886	98.8		-	-	-	$ZA ZA  =  \mu F ZA$
<b>PUR (300-5000 μm) (</b> Q3878	<b>(No. p/kg))</b> 1000					ZA ZA  - µF ZA
Polyurethane total ((						
Q3878	16200	8400	6000	5946	-	ZA ZA  - µF ZA
Q3886	98.8	-	-	-	-	$ZA ZA  - \mu F ZA$
PVC (50-299 μm) ((Να	n/kg))					
Q3878	12100	5750	-	541	-	ZA ZA  - µF ZA
Q3886	49.9	101	-	-	-	$ZA ZA  -  \mu F ZA$
PVC (300-5000 μm) ((						
Q3878	1250	50.0	-	-	-	ZA ZA  - µF ZA
Polyvinyl Chloride to	tal ((No. p/k	(g))				
Q3878	13350	5800	-	541	-	$ZA ZA  -  \mu F ZA$
Q3886	49.9	101	-	-	-	$ZA ZA  -  \mu F ZA$
Other shares (50.2						
Other polymers (50-2 Q3878	250 (199 <b>µm)</b>	·				« ⊂ التر ح ا ارج ح
Q3878 Q3886	250 799	- 759	- 101	- 404	- 99.5	ZA ZA  - µF ZA
43000	199	755	101	404	33.5	ZA ZA  - µF ZA

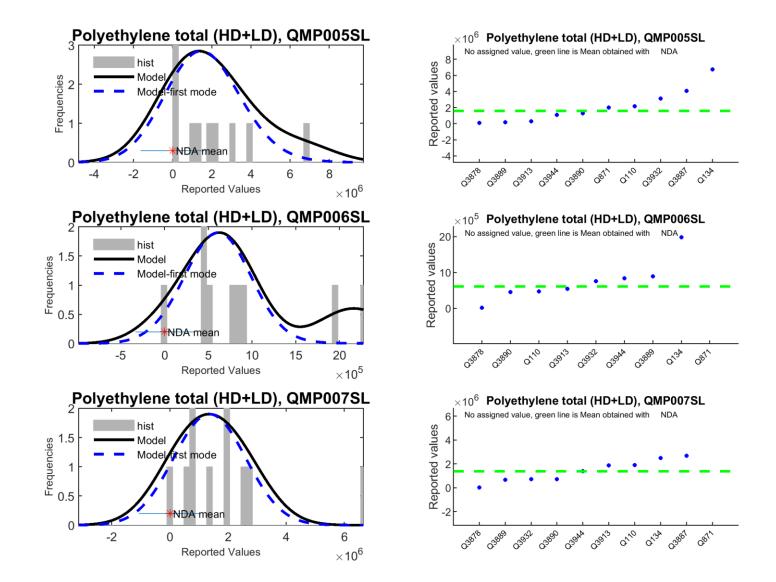
Q3887	1590	-	13039	-	-	ZB   ZB   ZB   ZA   ZB
<b>Other polymers (</b> Q3887	<b>300-5000μm) ((</b> 723	No. p/kg))	_	_	_	2B   2B   2B   2A   2B
Q3889	598	178319	313	-	-	
						-   ΖΑ   μF
Sample	QMP005SL	QMP006SL	QMP007SL	QMP008SL	QMP009SL	MIC
Other polymers t	otal ((No. p/kg)	)				
Q3878	250	-	-	-	-	ZA ZA  - µF ZA
Q3886	799	759	101	404	99.5	$ZA ZA  -  \mu F ZA$
Q3887	2313	-	13039	-	-	ZB ZB ZB ZA ZB
Q3889	598	178319	313	-	-	$ZC  -  ZA \mu F$
Q3940	14853	23131	20584	15309	7855	
Total polymers (5	50-299 μm) ((No	. p/kg))				
Q110	2164169	989616	1910215	2072851	1638413	ZA   ZA   ZA   µF   ZA
Q134	6733087	3951930	2492571	2005943	2518403	
Q153	3565	3588	11058	15530	12498	ZB ZB ZB µF ZB
Q3878	321400	93800	89650	87838	-	
Q3886	948	1062	101	807	249	ZA ZA  - µF ZA
Q3889	-	889747	-	-	-	
Q3922	-	25381	-	-	-	
Q3936	816	3767	112985	43643	52804	ZA   ZB   ZA   ZA   ZA
Q661	387876	197912	249143	140516	384205	ZA   ZA   ZA   MI   ZA
	=======		tatistical Result	5 =========	======	
NDA mean	139035	45269	85367	55246	-	
NDA st dev	392264	117332	144442	91103	-	
Coeff Var (%)	282.1	259.2	169.2	164.9	-	
N	7	9	7	7	6	
Median	321400	93800	112985	87838	218505	
MAD	320452	92738	112884	72308	212131	
Category		-	-		2	
Added number	3896050	2979000	2435050	2979000	2979000	
Total polymers (3	300-5000 μm) ((I	No. p/kg))				
Q3878	24700	1150	550	2162	-	
Q3889	189732	178319	668446	378108	757271	
Q3922	735	4975	705	450	1373	
Q3936	672	1406	42821	58652	40880	ZA   ZB   ZA   ZA   ZA
Q661	10509	13937	100363	47212	22351	ZA   ZA   ZA   MI   ZA
		===== Statist	ical Results (no	NDA) =====		
Ν	5	5	5	5	4	
Median	10509.0	4974.6	42821.0	47212.0	31615.5	
MAD	9837.0	3824.6	42271.0	45050.0	19753.3	
Category	2	2	2 ========	2	2	
Total polymers ((	No. p/kg))					
Q110	2164169	989616	1910215	2072851	1638413	ZA   ZA   ZA   µF   ZA
Q134	6733087	3951930	2492571	2005943	2518403	· · · · · · · · · · · · · · · · · · ·
Q153	3565	3588	11058	15530	12498	ZB ZB ZB µF ZB
Q3160	446	467	758		152	· · · · · · · · · · · · · · · · · · ·
Q3877b	672755	567506	1312064	1350159	1198191	ZB ZB ZB MI ZB
Q3878	346100	94950	90200	90000	-	
Q3886	948	1062	101	807	249	ZA ZA  - µF ZA
Q3887	4059609	-	2679891	-	-	
Q3889	189732	1068066	668446	378108	757271	
Q3890	1289063	952222	1034848	2274661	2034582	
Q3913	276890	1141254	1916706	3176962	2034382	
20010	2,0000	1171237	1910/00	51,0502	2000070	

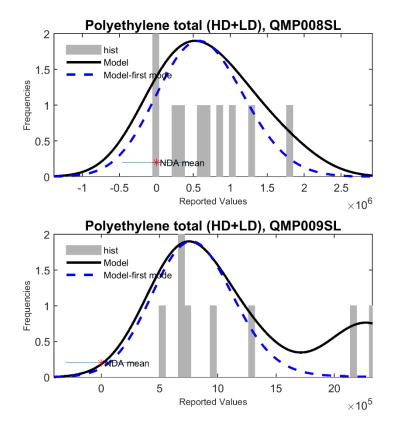
	========	========	Summary Statistics	========		
NDA mean	355062	496030	799504	364489	752362	
NDA st dev	558094	652157	1023678	590781	1014152	
Coeff Var (%)	157.2	131.5	128.0	162.1	134.8	
Ν	19	18	19	17	17	(cont.)
Added number	3896050	2979000	2435050	2979000	2979000	

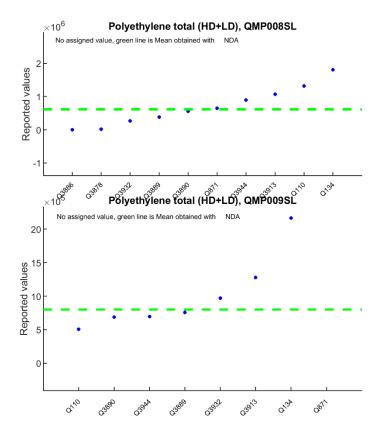


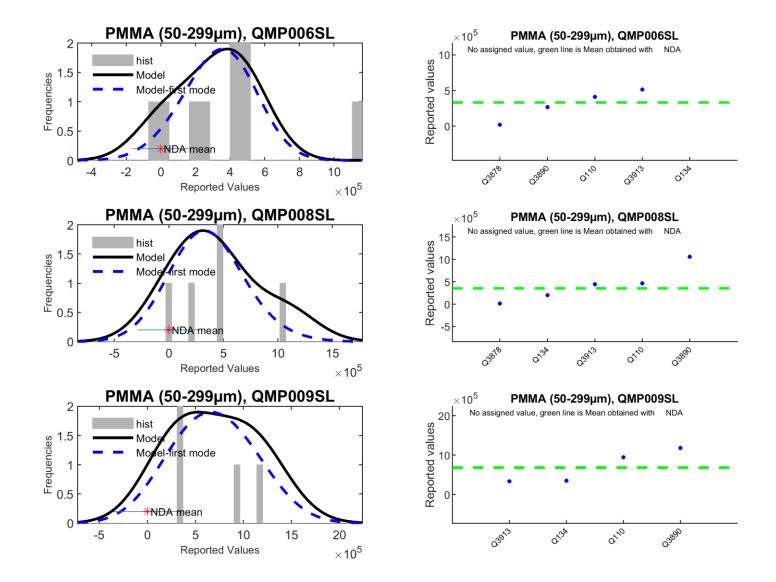


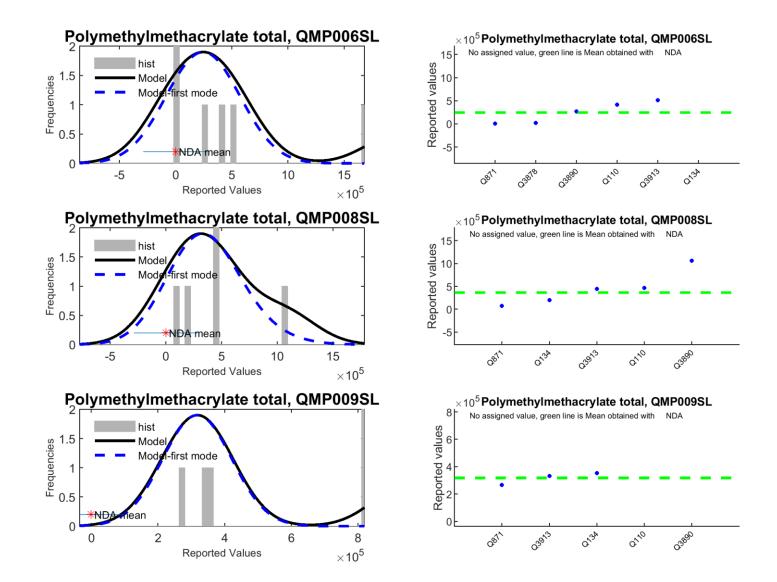


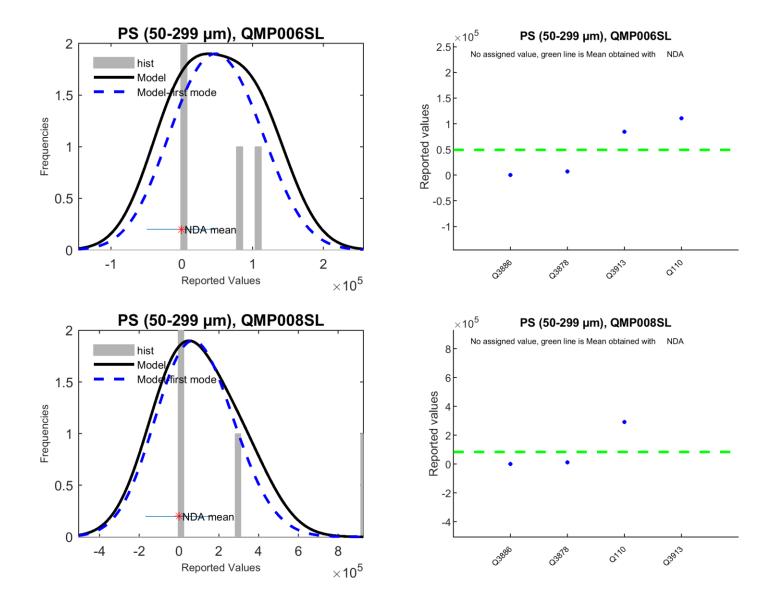


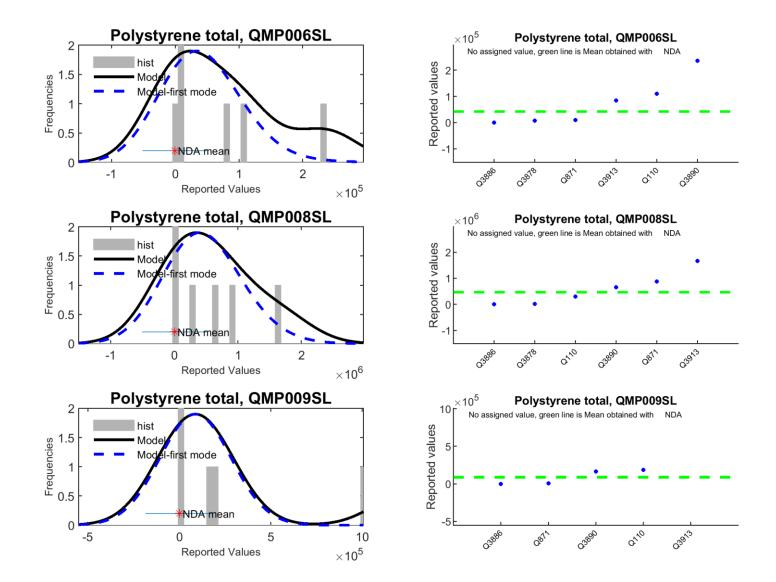


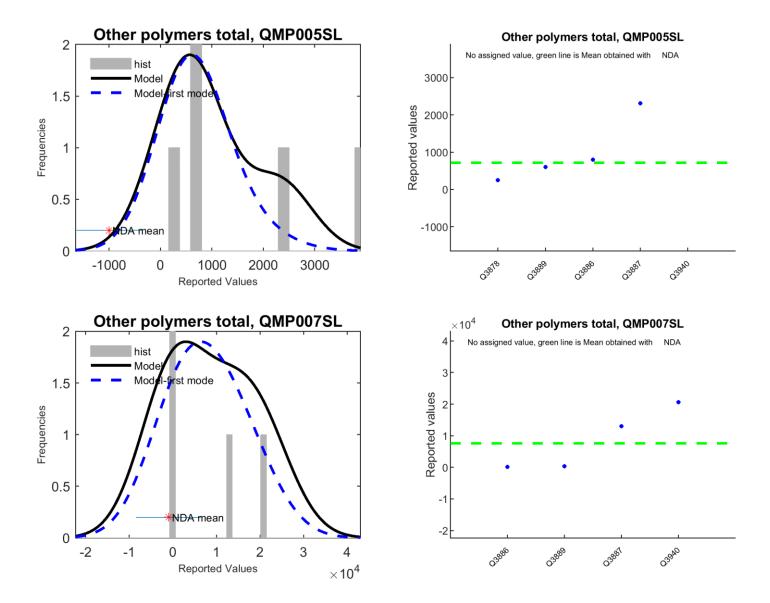


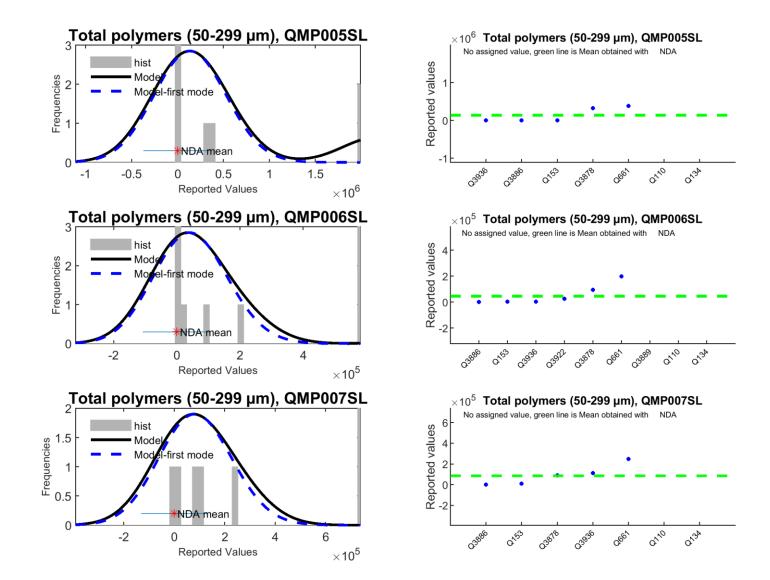


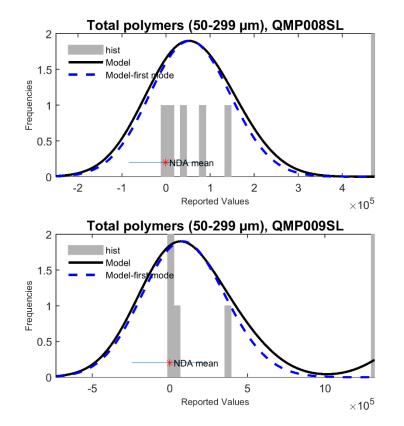


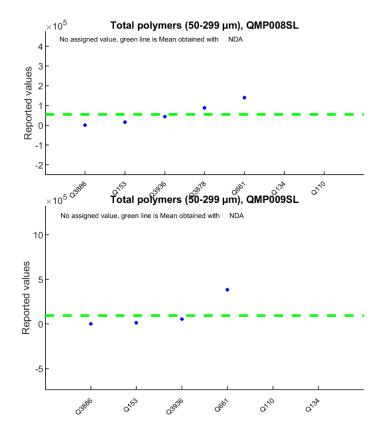


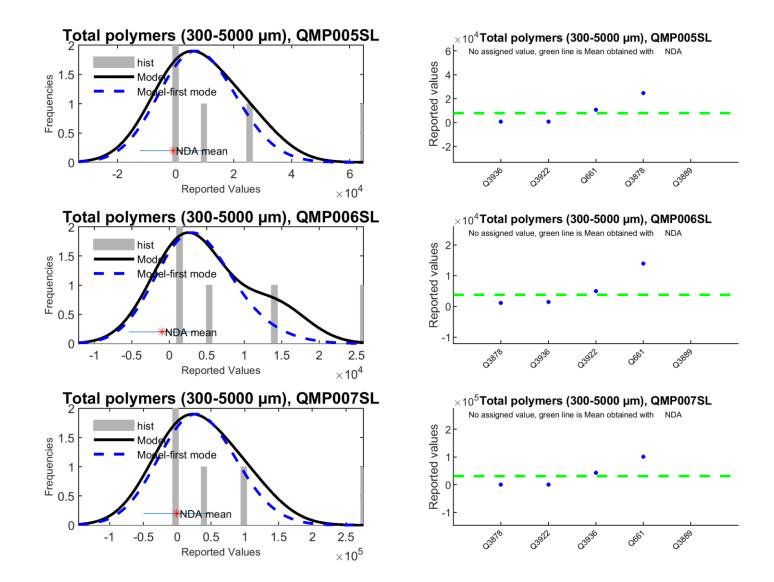


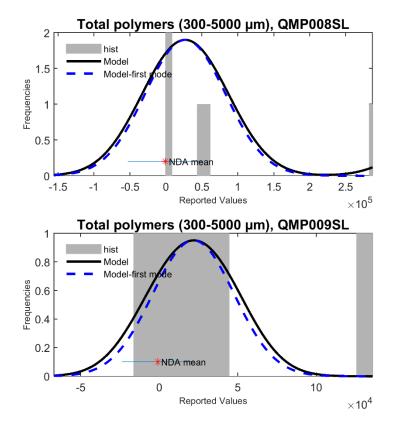


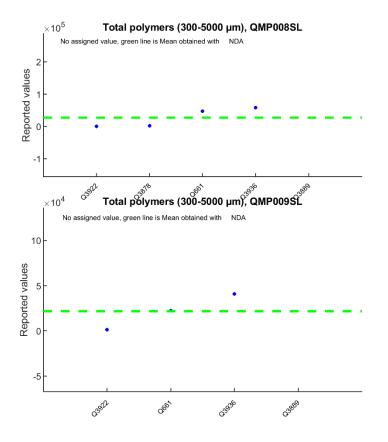


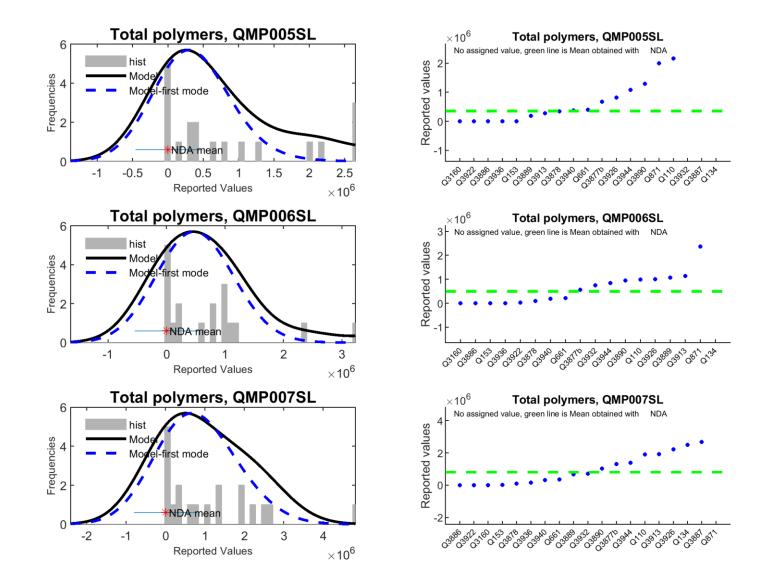


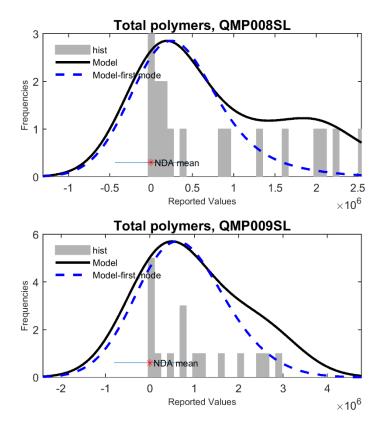


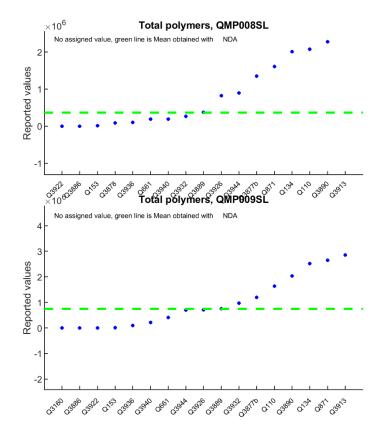












# **Appendix 2: Total number of particles found in the ILS soil samples.**

Explanation of the acronyms given in the reported data are given in section 2.6.

## Total mass of plastic particles Summary Statistics

Sample/ Determinand	Assigned Value	Units	Total Error	NDA st.dev	NDA rel. st.dev (%)	Nobs numerical	Nobs LCV	Median	MAD	Model Mean	Uncer- tainty
QMP005SL											
Total Polymers	1281	(mg/kg)		707	55.2	10	0	1464	484	1280.9	279.5

Sample/ Determinand	Assigned Value	Units	Total Error	NDA st.dev	NDA rel. st.dev (%)	Nobs numerical	Nobs LCV	Median	MAD	Model Mean	Uncer- tainty
QMP006SL											
Total Polymers	609	(mg/kg)		485	79.7	10	0	591	325	608.7	191.8

Sample/ Determinand	Assigned Value	Units	Total Error	NDA st.dev	NDA rel. st.dev (%)	Nobs numerical	Nobs LCV	Median	MAD	Model Mean	Uncer- tainty
QMP007SL											
Total Polymers	1029	(mg/kg)		703	68.3	10	0	1182	504	1029.0	277.9

Sample/ Determinand	Assigned Value	Units	Total Error	NDA st.dev	NDA rel. st.dev (%)	Nobs numerical	Nobs LCV	Median	MAD	Model Mean	Uncer- tainty
QMP008SL											
Total Polymers	478	(mg/kg)		251	52.5	10	0	559	194	478.1	99.2

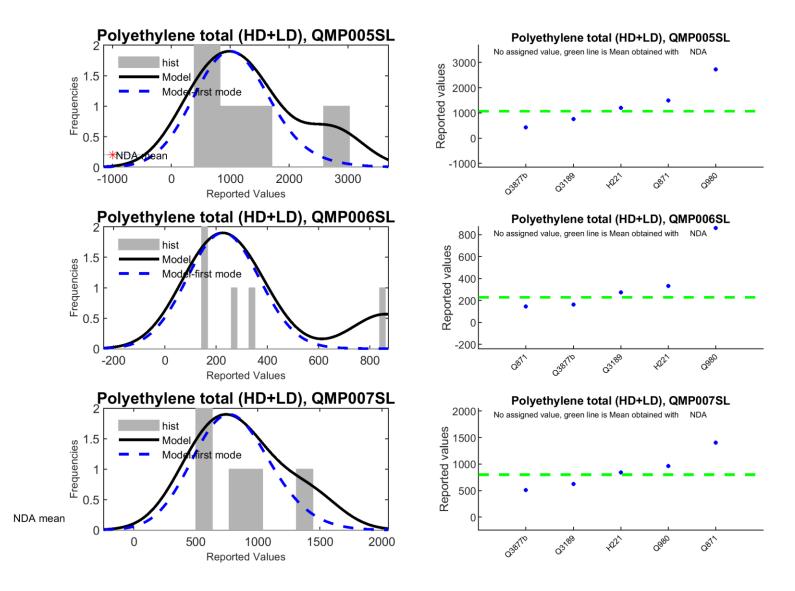
Sample/ Determinand	Assigned Value	Units	Total Error	NDA st.dev	NDA rel. st.dev (%)	Nobs numerical	Nobs LCV	Median	MAD	Model Mean	Uncer- tainty
QMP009SL											
Total Polymers	502	(mg/kg)		204	40.6	10	0	562	146	501.9	80.6

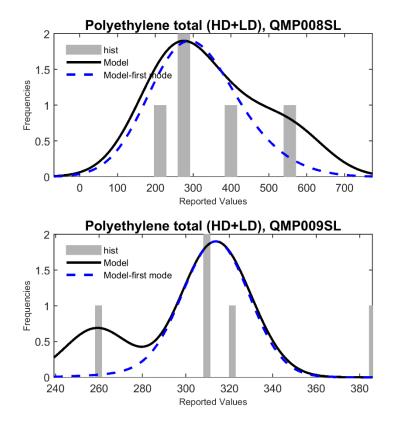
## Total mass of plastic particles Data and Statistics

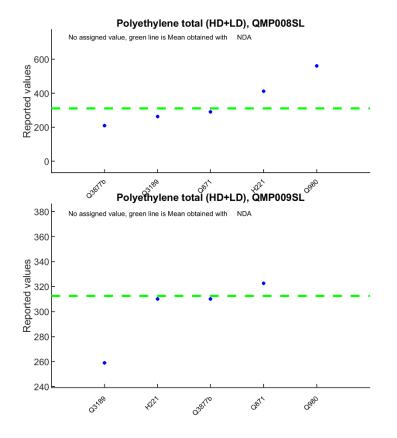
Sample	QMP005SL	QMP006SL	QMP007SL	QMP008SL	QMP009SL	МІС
Polycarbonate to	otal ((mg/kg))					
Q980	0.041	0.038	1.00 <	0.250	1.00 <	-  - ZA PY ZB
PE (50-299 μm) (	(mg/kg))					
Q3932	1432	641	1346	361	568	ZC ZC  - AF ZC
Polvethvlene to	tal (HD+LD) ((mg/	(kg))				
H221	1204	333	840	412	310	-  - ZA PY ZA
Q3189	751	274	623	263	259	ZB   ZB   ZB   PY   ZB
Q3877b	421	163	507	210	310	ZA   ZA   ZA   PY   ZA
Q871	1495	146	1405	290	323	
Q980	2730	860	960	560	590	-  - ZA PY ZB
Added mass	2000	500	1250	500	500	
	ephat. Total ((mg					
H221	3.00	0.010 <		0.010 <		
Q980	0.200	0.280	1.38	1.93	1.70	-  - ZA PY ZB
	nacrylate total ((n					
H221	0.010 <		0.010 <		40.0	-  - ZA PY ZA
Q3189	7.59	124	7.25	173	137	ZB   ZB   ZB   PY   ZB
Q980	1.00 <		0.600	23.0	210	-  - ZA PY ZB
Added mass	-	750	-	750	750	
Polypropylene to	otal ((mg/kg))					
Q871	-	-	1.38	-	-	
Q980	20.0	5.00 <	3.30	1.70	5.00 <	-  - ZA PY ZB
Polystyrene tota	ıl ((mg/kg))					
H221	39.0	17.0	22.0	43.0	23.0	-   -   ZA   PY   ZA
Q3189	-	7.64	-	16.8	17.9	ZB   ZB   ZB   PY   ZB
Q871	3.06	63.3	0.250	217	288	
Q980	4.10	0.900	0.600	3.00 <		-  - ZA PY ZB
Added mass	-	75	-	75	75	
	le total ((mg/kg))				-	
H221	250	68.0	155	60.0	57.0	-   -   ZA   PY   ZA
Q3189	-	7.35	-	6.35	5.95	ZB   ZB   ZB   PY   ZB
Q3877b	552	377	1131	1369	3068	ZA   ZA   ZA   PY   ZA
Q980	40.0	18.0	25.0	24.0	25.0	-  - ZA PY ZB
Total Dalama /	F0 200	(1.~))				
Total Polymers ( Q3934	50-299 μm) ((mg, 592	<b>/kg))</b> 655	593	483	238	
Q3934 Q3936	38.7	68.7	212	483	238 124	
43330	30.7	00.7	212	107	124	
Total Polymore (	300-5000 μm) ((n	ma/ka))				
Q3934	1001 1001	<b>пg/кg))</b> 896	3423	3326	317	
Q3934 Q3936	20.8	16.7	80.2	137	74.5	
45550	20.0	10.7	00.2	137	74.5	

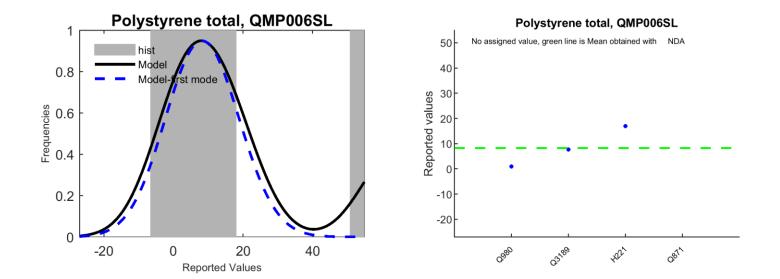
## Total mass of plastic particles Data and Statistics

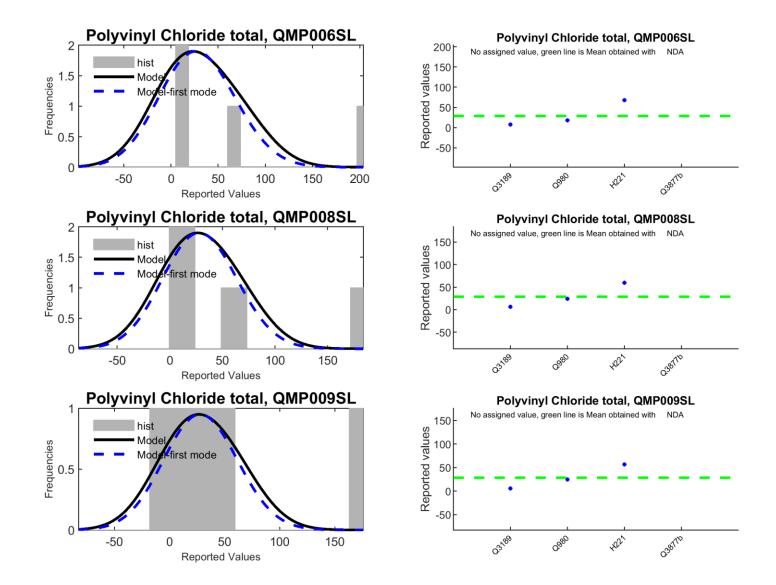
-   -   ZA   PY   ZA
ZB ZB ZB PY ZB
ZA   ZA   ZA   PY   ZA
ZA   ZA   ZA   ZA   ZA
ZA   ZA   ZA   GR   ZA

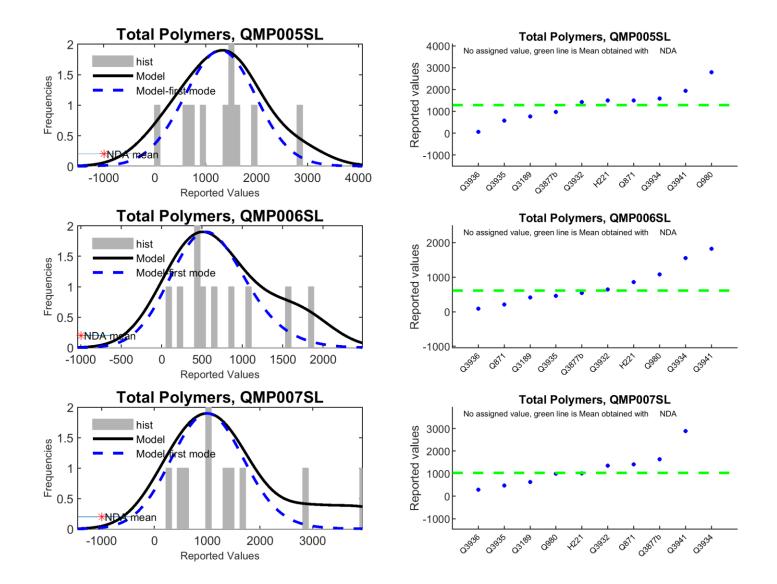


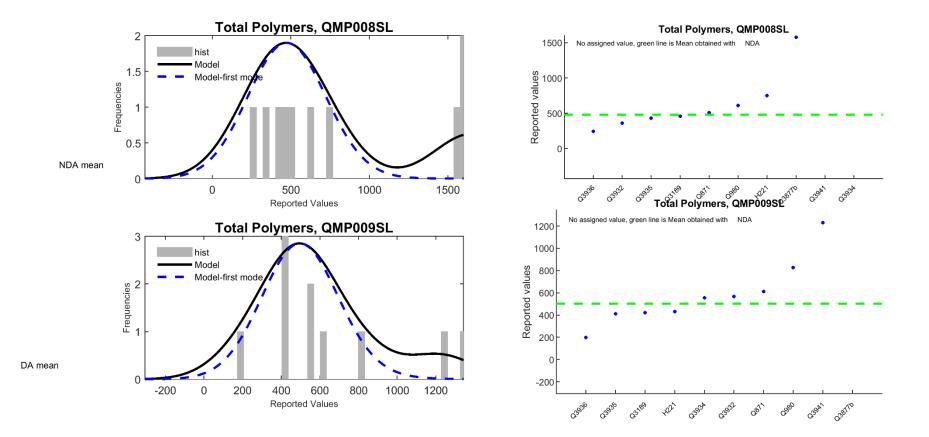






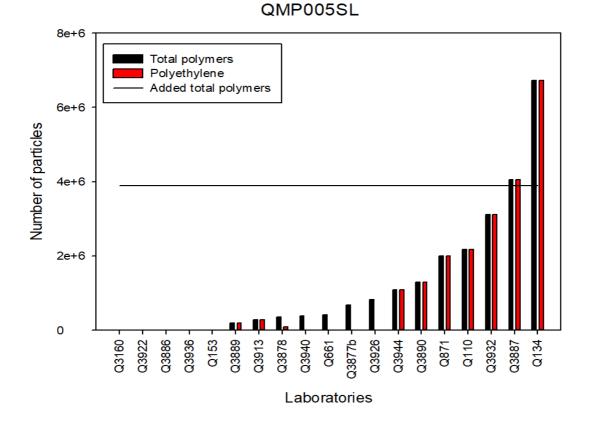






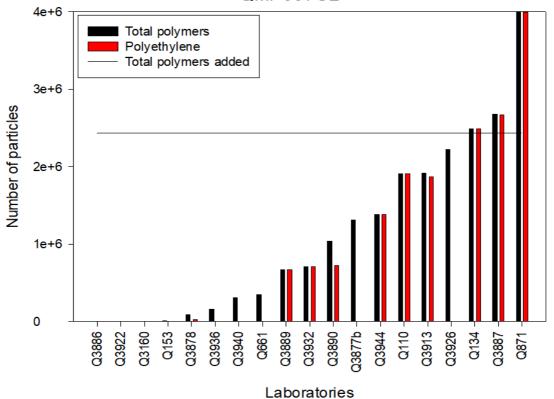
# **Appendix 3: Total number of particles**

Total number of MPs reported in spiked sand (QMP005L) and a real soil (QMP007SL and QMP009SL) samples. On the horizontal axis the laboratory codes are given and on the vertical axis the number of particles reported. Number of total polymers, or PE, PPMA and PS are given. The added amounts of total number of polymers, PE, PMMA, and PS are given as black, red, green and yellow lines, respectively.



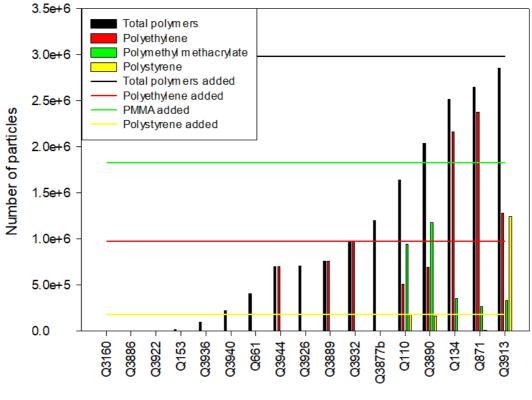


#### QMP007SL





QMP009SL



Laboratories

# Abstract

MISSOURI organized, together with WEPAL-QUASIMEME, the first interlaboratory study (ILS) on the analysis of microplastics (MPs) in soil. Two soil materials (sand and a real soil), prepared by MISSOURI, were spiked with common plastic polymers, both as single (PE) as well as a mixture of different polymers (PE, PMMA, PS). In total five soil materials were prepared. In total 58 laboratories participated the ILS and 25 laboratories reported data. The results showed that the analysis of MPs in soil is difficult, but no more difficult than sediments. First indications were found that the soil matrix does not interfere with the MP analysis. Quantification of MPs in soil on mass basis had lower relative standard deviations (RSDs) (41-80%) than on particle basis (128-162%), but in both cases the RSDs are too high for reliable quantification. Similar RSDs were found for single spiked MPs or a mixture of different polymers, demonstrating that the analysis of a mixture was no more difficult than the analysis of single MPs. Indications were found that not all MP polymers behave equally in glass bottles, so it was recommended to make wet soil materials in next interlaboratory studies to generate more homogeneous samples. It was also recommended to perform additional soil and/or sediment ILS studies using lower concentrations of MPs.



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