# Interlaboratory Study on Microplastics Analysis Development Exercise – DE 17

## Round 2 (2020)

### Final Report

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19 July 2021

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This report can be cited as: van Mourik, L.M., Crum ,S.J.H., van Bavel, B., Martinez-Frances, E., Leslie, H.A., de Boer, J., Cofino, W.P. 2020 Interlaboratory Study on Microplastics Analysis, Quasimeme Development Exercise DE 17 Round 2, May 2021.

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## Abbreviations and acronyms

| | |
|---|---|
| ATR | Attenuated total reflection |
| AV | Assigned value |
| DE | Development exercise |
| EPS | Expanded polystyrene |
| µFTIR | Microscopy and Fourier transform infrared spectroscopy |
| ILS | Interlaboratory study |
| LDPE | Low-density polyethylene |
| Max | Maximum |
| Min | Minimum |
| *n* | Number |
| NIAS | Non-intentionally added substances |
| NIVA | Norwegian Research Institute for Water Research |
| no. | Number |
| PC | Polycarbonate |
| PET | Polyethylene terephthalate |
| PE | Polyethylene |
| PMMA | Polymethyl methacrylate |
| PP | Polypropylene |
| PS | Polystyrene |
| PVC | Polyvinyl chloride |
| Pyr-GC-MS | Pyrolysis gas chromatography-mass spectrometry |
| QAQC | Quality assurance and quality control |
| RSD | Relative standard deviation |
| SD | Standard deviation |
| TED-GC-MS | Thermal extraction and desorption gas chromatography-mass spectrometry |
| VU | Vrije Universiteit Amsterdam |

**Summary**

The second round of the interlaboratory study on microplastics consisted of three types of test materials: a standard-like material (i.e. tablet), a spiked sediment and a spiked fish sample. Participants were requested to identify the polymer type as well as the number of particles and/or mass of particles (in total and per polymer type) in these test materials.

Compared to the previous round (n = 35 of which 30 submitted results), a larger number of laboratories participated in this round (n=59), of which 35 laboratories submitted results before the deadline (completion rate of 59%). The main reason of laboratories being unable to submit data before the deadline was the COVID-19 situation, despite the extension of the deadline. Almost all laboratories reported results for the tablet (QMP002SW: n=33), followed by the sediment (QMP003MS: n=28). The least results were reported for fish (QMP004BT), but results were still reported by the majority of laboratories (n=24). While most laboratories still reported the number of particles (n=16-25), an increasing number of laboratories (n=10-11) reported the mass of plastic (i.e. mg/kg by GC-MS) compared to previous round (n=2).

The added polymers were the most frequently reported polymers in the tablets and environmental samples, with the majority of laboratories (61-64%) identifying the added polymers. A large variation was found in reported number of particles in the tablet QMP002SW (relative standard deviation 46-83%), while for the environmental samples this was larger still (85-231%). The variation found in the tablet was comparable to the previous interlaboratory round which was 29-91%, and which had higher number of participants.

With regard to the preparation of materials, it is clear that different techniques are required to ensure homogeneous test materials that represent the various environmental compartments.

# 1 Introduction

Microplastics are present in every environmental compartment and have gained recent interest as an environmental pollutant. 'Plastic' is not a well-defined analyte, but rather a set of materials that encompass a wide range of high molecular weight synthetic polymers such as thermoplastics and thermosets. 'Microplastics' are plastic particles spanning 6 orders of magnitude in particle size (low nanometre to 5 mm) and a large variety of chemical compositions: (co)polymers, chemical additives, residual monomers, fillers, catalysts, non-intentionally added substances (NIAS) etc.

The diversity of this analyte class gave rise to a search for fit for purpose methodologies to answer the burning questions in microplastic research and to support plastic pollution monitoring and mitigation policies under consideration by state and non-state actors. To date there are no validated standard methods available for the analysis of microplastic and various number of analytical protocols, methods and techniques are used. The analysis of microplastics is difficult due to the large number of different polymers, size fractions and shape. Furthermore, as there is still not consensus on the reporting format, microplastic are reported as number of particles, fibres or mass of different size fractions. There is an obvious need to validate and harmonize various methods. Another challenge analytical scientists face with microplastics analysis is how to check and demonstrate analytical proficiency. Open interlaboratory studies (ILSs) are very limited in number, and there is still a total absence of certified reference materials with which to investigate analytical proficiencies.

Participation in ILS studies increases confidence in the data produced, both for the analytical laboratories and the data users. For accreditation, regular proficiency testing will eventually be required. ILS studies will also present a 'state of the art' of the analytical procedures used for polymer identification and quantification and are a useful tool for method development and further standardisation. Each participating laboratory receives a confidential laboratory code and an anonymized study report presenting the overall results at the end of the round. Participants benefit from follow-up workshops in which the study results of the testing rounds are discussed with regard to analytical performance of different methods used.

The Vrije Universiteit Amsterdam (VUA), the Norwegian Research Institute for Water Research (NIVA) and WEPAL-QUASIMEME Laboratory Performance Studies (Wageningen Environmental Research) have taken the initiative to organise an interlaboratory study on microplastics. The study has been supported by the NORMAN workgroup nano-and micro scale particulate contaminants, which has recognized microplastics as an emerging issue. The four institutions have joined forces to set up a program to address the quality of microplastic analyses.

As a first step, a workshop on microplastics was organised in Amsterdam, the Netherlands, in November 2018. During this workshop (ca. 110 participants) it was generally agreed that an ILS on microplastics was needed, preferably designed in a step-wise way. Because this ILS focuses on a new and not yet standardized analysis, this study was classified as a 'Development Exercise' (DE), and coded DE-17. In 2019 the first round was held, with the analysis of 'standard' like test samples. The objective of this round was to assess the ability to determine the polymer type and number or mass of polymer particles in 12 samples prepared specifically for this exercise, i.e. six samples containing of pre-production pellets, five dissolvable soda tablets containing different (smaller) polymer particles and one blank soda tablet. In total, 34 laboratories participated with their own method of choice, of which 30 submitted data. A variety of identification and quantification methods (n=7) was used. The majority of the laboratories correctly identified the types of polymers in all pre-production pellets with the exception of LDPE, which was identified as HDPE by some participants, indicating satisfactory performance. The analysis of the smaller particles in the soda tablets varied however considerably between laboratories.

This report describes the design and the results of the second ILS round DE-17, in which complexity and difficulty were increased with regard to the first round. The objective of this second round was the same as the first round, i.e. assess the ability to determine the polymer type of plastic particles, as well as the number or mass of plastic particles, but this time also spiked environmental samples were included.

### *1.1 Confidentiality of results*

The confidentiality of the results is extremely important in the WEPAL-QUASIMEME programs. In the report only the laboratory codes are mentioned in the data reporting and therefore, no list of participants is included in this report. When an accreditation body or a regulatory authority requests the proficiency test results to be provided by WEPAL-QUASIMEME, the participants shall be notified and asked for permission first.
Participants may not use or report individual data from other laboratories. Assigned values, means and standard deviations of the interlaboratory studies published in this report may be used.

## 2 Materials and methods

### *2.1 Study design*

This study was setup in agreement between WEPAL-QUASIMEME, VUA and NIVA. WEPAL-QUASIMEME is a leading expert in the organisation of interlaboratory studies with a focus on e.g. the marine environment. It is accredited for organizing proficiency tests for several determinands and matrices. WEPAL-QUASIMEME handled the logistics and analysed the data. VUA has an extensive experience in environmental analysis and in the organization of interlaboratory studies, many in collaboration with WEPAL-QUASIMEME, and is actively involved in the field of microplastics. VUA organised the preparatory workshop, coordinated the project and reporting, and gave input to the data interpretation. NIVA is Norway's leading institute concerning the aquatic environment. NIVA is involved in several quality assurance and quality control (QA/QC) studies and develops certified reference materials for different contaminants including microplastics. NIVA has prepared a number of microplastic standard and test materials.

The developing exercise was designed in a step-wise manner, consisting of a number of rounds of sending out samples for analysis to participants, collecting the participants' data, and then analysing and reporting the data back to participants. The first step (i.e. the first round), started with the analysis of 'standard' like test samples. This creates a basis for laboratories to check their performance in both identifying and quantifying polymers in samples in the absence of a (complex) matrix. This round included both the standard like samples as well as fortified environmental samples. After the three rounds of the developmental exercise, the analytical methodologies for microplastics are expected to be better comparable and will be included in the routine proficiency testing scheme of WEPAL-QUASIMEME.

National reference, governmental, research, academic and commercial laboratories as well as other research facilities world-wide were invited to participate. The analytical work of ILS DE-17 was performed between May 2020 and August 2020. Participants were asked to identify and quantify, i.e. count particles (integer) and/or determine the mass of particles (mg or µg) and polymer types in six preproduction pellets and six tablets, using their own method of choice. In addition to the results, information was requested about the participants' analysis methods for a more in-depth analysis of the submitted data as well as performance characteristics. All the requested data was filled in and submitted by Excel report forms. The laboratory code of the participating laboratories is kept confidential and will not be revealed to other participants.

### *2.2 Material preparation*

#### 2.2.1 Standard-like test materials

The tablets were prepared by NIVA. They consisted of a mixture of sodium hydrogen carbonate ($NaHCO_3$) citric acid ($C_6H_8O_7$), and a binder (lactose) which were not expected to interfere during the analysis as the tablets completely dissolve in water. The tablets were made by hand by combining the ingredients and the different polymers into the mixture. The mixture was poured into a metal form in which the tablets were moulded by applying pressure. Blank tablet (QMP001SW) consisted only of the ingredients without

the addition of polymers. No lubricants were added to the mixture as most of them are not completely soluble in water. The tablets were sealed in an aluminium strip before shipment.

The following polymers: polyethylene (PE), polyethylene terephthalate (PET), polyvinylchloride (PVC) and polystyrene (PS) in powder form, were sieved using a vibratory sieve shaker, resulting in fractions of 500 μm to 50 μm, but only the fractions of 125–150 μm, 100–50 μm, 50–150 μm and 100–150 μm, respectively, were added to the tablets.

Homogeneity of the tablets samples was verified by analysing 10 tablets from the batch QMP002SW. The total number of particles found was 71 ± 6 (RSD 8%) for QMP002SW. This was considered good in relation to the expected variation between the laboratories. The number and mass of the polymers are given in Table 2-1.

### 2.2.2 Environmental samples

Two environmental test materials were prepared by VU. One jar labelled as QMP003MS contained ca. 20 g dry marine sediment (Westerscheldt, the Netherlands). A second jar, labelled as QMP004BT contained ca. 5 g freeze dried blue whiting (North Sea) with a fat content of 3.2% (0.8% fat before freeze-drying). Both fish and sediment were tested for presence of microplastic particles by microscopy. The total number of microplastic particles in 100 mg freeze-dried fish remained under the detection limit (<1.2 particles, or <60 particles per jar, or 12,000 particles/kg). As 8-10 particles were detected in 5 g sediment, the sediment was heated up to 600 °C overnight. After this treatment no particles were detected by microscopy.

The different polymer fragments were purchased from Cospheric (Santa Barbara, CA, USA). i.e. polymethyl methacrylate (PMMA) in fractions of 63-75 μm and 90-106 μm, PE in fractions of 53-63 μm and 63-75 μm, and PS in a fraction of 85-105 μm. The ranges in sizes in each fraction were confirmed by microscopy determination.

After homogenization, glass jars were filled with either ca. 20 g sediment or 5 g fish. Because we wanted to obtain microplastics levels above the detection limit of Pyr-GC-MS analysis (7-100 mg/kg, dependent of method), it was decided to have a spiked concentration of ca. 5-10 particles/mg of each polymer type. An overview of added polymer types, size fraction and number is given in Table 2-1. Based on the recorded weight of the sample, particles were added by weight, with a RSD values between jars below 3% for sediment and below 7% for fish.

## *2.3 Analytes of interest*

For the tablet (QMP002SW) the results on the *polymer type of plastic particles present*, and the *number* of plastic particles *per tablet (no. P/tablet)* and/or the *mass* of the plastic particles *per tablet (mg/per tablet)* had to be reported. For the environmental samples (QMP003MS and QMP004BT) the results on the *polymer type of plastic particles present*, and the *number* of plastic particles *per kg (no. P/kg)* and/or the *mass* of the plastic particles *per kg (mg/kg)* had to be reported. A blank tablet with no added plastic particles (QMP001SW) was to be analysed and reported with the same procedural steps as the other tablet. This sample was used as a quality control sample only and was not validated statistically in terms of standard deviation or z-scores.

Table 2-1 shows the characteristics of the tablet and environmental samples, of which the data for the tablets have been obtained by homogeneity studies carried out by NIVA. For the environmental samples, the number of the added polymers particles for each batch are given. These are based on the weight of sample added and the density of the average particle size added.

**Table 2-1 Characteristics of the samples distributed**

| QMP002SW | µg added to tablet | (*n* particles) | particle size (µm) |
|---|---|---|---|
| Polyethylene terephthalate (PET)* | 14.6 | 30 | 100 - 150 |
| Polyvinylchloride (PVC) | 17.3 | 16 | 50 - 150 |
| Polystyrene (PS) | 15.1 | 15 | 100 - 150 |
| Polyethylene (PE) | 21.7 | 15 | 125 – 150 |
| Total Microplastics | 68.7 | 76 | 50 - 150 |
| **QMP003MS** | **mg/kg added**** | **(*n*/kg particles)** | **particle size (µm)** |
| Polymethyl methacrylate (PMMA) | 2052 | $5.00 * 10^6$ | 90 - 106 |
| Polyethylene (PE) | 900 | $5.00 * 10^6$ | 63 - 75 |
| Total Microplastics | 2952 | $10.00 * 10^6$ | 63 - 106 |
| **QMP004BT** | **mg/kg added**** | **(*n*/kg particles)** | **particle size (µm)** |
| Polymethyl methacrylate (PMMA) | 1406 | $10.04 * 10^6$ | 63 - 75 |
| Polyethylene (PE) | 1129 | $10.26 * 10^6$ | 53 – 63 |
| Polystyrene (PS) | 2074 | $4.94 * 10^6$ | 85 – 105 |
| Total Microplastics | 4609 | $25.24 * 10^6$ | 63 - 105 |

* Densities of added polymers: PET: 1.40 g/cm$^3$, PVC: 1.40 g/cm$^3$, PS: 1.07 g/cm$^3$, PE: 0.92 g/cm$^3$, PMMA: 1.18 g/cm$^3$ (http://polymeracademy). ** Concentration for QMP003MS and QMP004BT estimated based on mass added and density of average particle size.

## *2.4 Methods applied*

Figure 2-1 shows an overview of methods applied, while Appendix B provides full details of reported methods per laboratory. It was noted that most participants reported the filtration, separation and clean-up methods used as additional information rather than selecting the options available. This is something to consider for the third round. An effort was made to sort the information into main categories. Six participants did not report any data on applied methods. Overall, the applied methods varied largely between the 29 laboratories that submitted data.

For the filtration methods four laboratories reported sieving with sieves ranging from 25 to 5000 µm. Most participants (n=24) reported to have used filtration, of which one after sieving, while two participants reported having applied no filtration. Nine different types of filters were used by the participants that used filtration (Figure 2-1A).

The majority of laboratories (n=19) had also applied density separation. Five different types of salts were used (Figure 2-1B), with many different densities (Appendix B). For clean-up 19 participants reported to have used digestion, while two participants used pressurized liquid extraction. Digestion was done by hydrogen peroxide (n=12) or potassium hydroxide (n=9, Figure 2-1C), and sometimes both (Appendix B).

In total eight different identification methods were reported, of which Fourier transform infrared spectroscopy in combination with microscopy (µFTIR) was the most commonly applied (43%). Compared to first round, the use of µFTIR doubled (n=16 vs. 7), while the use of attenuated total reflection FTIR (ATR-FTIR) decreased significantly (n=2 vs. 14). The use of GC-MS (i.e. pyrolysis and gas chromatography-mass spectrometry (pyr-GC-MS) and thermal extraction and desorption GC-MS (TED-GC-MS)) also increased to n=5 vs. 2 in the first round, while the number of submitted datasets reporting on mass (i.e. mg/kg, Figure 3-1) is even higher (n=11), possibly due to not reporting method information by some labs and including gravimetric determinations. This makes the frequency of using

GC-MS in this round almost equal to that of the use of FTIR. Two participants reported using Nile red staining, of which one in combination with µFTIR. Three participants reported having used microscopy only (without identification method), while two participants reported the mass after a gravimetric determination.

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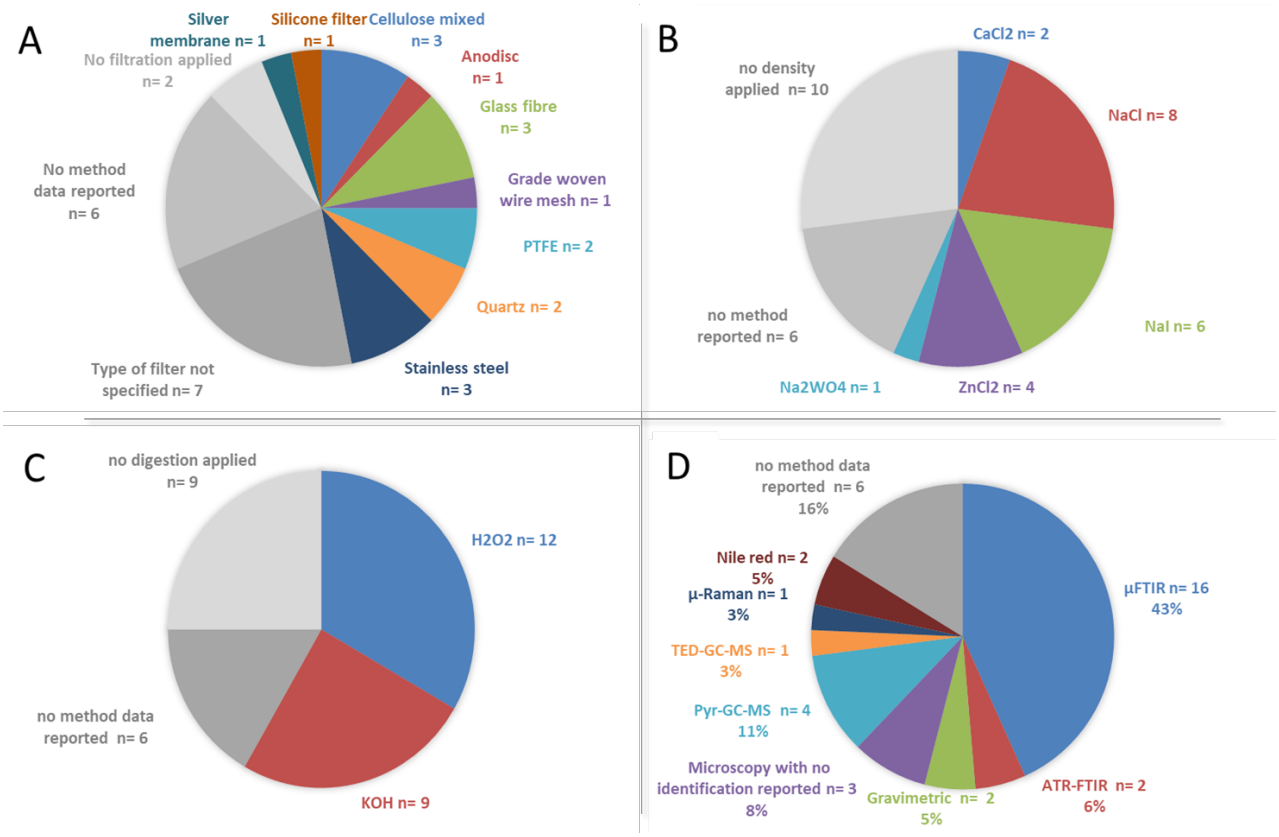


***Figure 2-1*** Summary of data on reported sample preparation and determination methods for microplastics determination in tablets and/or environmental samples, with A) type of filter used, B), type of salt used, C) type of digestion used and D) type of determination method used. Detailed information per laboratory can be found in Appendix B.

## *2.5 Data assessment*

The evaluation of the data reported for the tablets (QMP001SW and QMP002SW) and environmental samples (QMP003MS and QMP004BT) focused on the identification and quantification, both mass and number of particles of the polymers.

The data assessment was carried out according to the principles of data assessment employed by the WEPAL-QUASIMEME proficiency testing organisation (www.WEPAL.nl). All data received from the participants were entered into an excel database and assessed using a robust method (NDA statistics, Molenaar et al. 2018) enabling direct comparison between participants. See appendix C for further details.

## *2.6 Z-score assessment*

In this report, z-scores are presented for the polymers that were added to the tablet QMP002SW. Please be aware that these z-scores are given to enhance the insights deduced from the ILS and as a support to improvements of methodology. The z-scores are in this case not intended to be used in evaluating the

performance of laboratories. No z-scores were calculated for the other samples as the variability in the results was too big.

Z-scores are calculated as

$$z_i = \frac{(\bar{x} - x_{pt})}{\sigma_{pt}}$$

In this formula, $z_i$ is the z-score of laboratory i, $x_{pt}$ is the assigned value (AV, i.e. consensus value of the dataset), and $\sigma_{pt}$ is the standard deviation for proficiency assessment. The z-score $z_i$ represents how far the result of laboratory i is from the assigned value in terms of the standard deviation $\sigma_{pt}$.
In this study, $\sigma_{pt}$ is set to be 12.5% of the assigned value. This approach differentiates the dataset in three zones:

- Zone I: results that are within 25% of the assigned value, in proficiency tests denoted as 'satisfactory results';
- Zone II: results with 2<|z|<3, thus that differ in absolute sense between 25% and 37% from the assigned value, in proficiency tests referred to as 'questionable results';
- Zone III: results that differ 37.5% or more from the assigned value, indicated in proficiency tests as 'unsatisfactory results'.

The three zones of z-scores are illustrated in Figure 2.4.

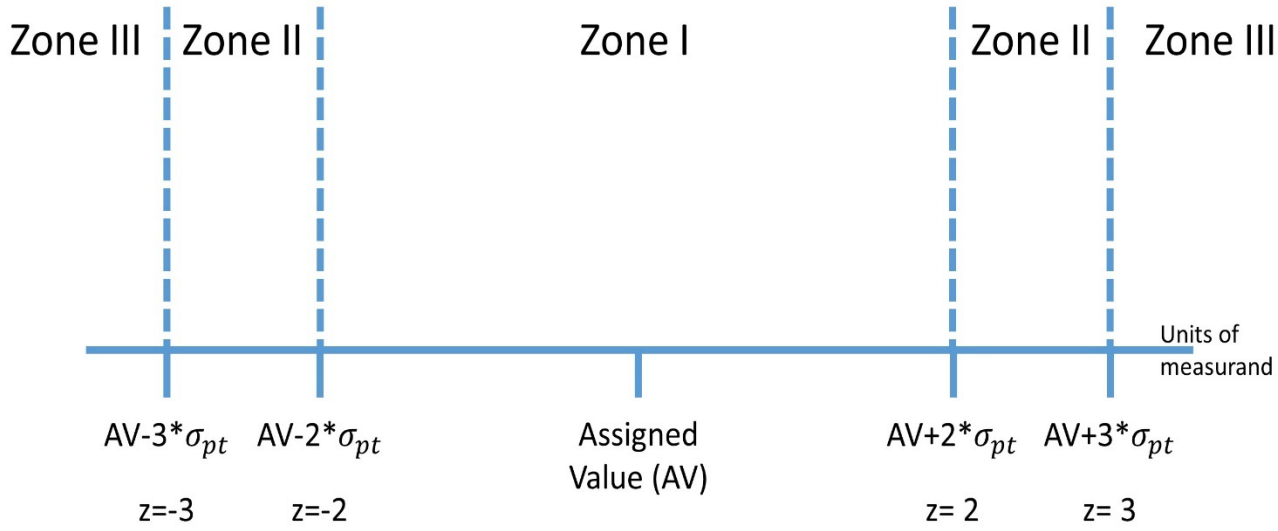


***Figure 2-2*** Illustration of z-score zones in relation to the assigned value.

The data have been analysed with a robust method described by Cofino *et al* (2000). The mathematical basis of the method has been strengthened in Molenaar et al. (2018).

The robust mean of the datasets is used as the assigned value $x_{pt}$. In proficiency tests, the standard uncertainty $u(x_{pt})$ of the assigned value is incorporated in the calculation of the z-score (giving rise to the z'- score, ISO 13528 (2016)). The term $u(x_{pt})$ is given by

$$u(x_{pt}) = 1{,}25 * \frac{s^*}{\sqrt{p}}$$

where $s^*$ is the robust standard deviation of the exercise and p the number of data analysed.

In the calculations presented, the uncertainty in the assigned value is not taken into consideration as it hampers a consistent interpretation of the z-scores for the purpose of this study. The standard uncertainty of the assigned value is, however, given to illustrate its magnitude.

Z-scores have been calculated for the individual polymers that were added to the tablet (QMP002SW) and for the total number of particles in these tablets. The assigned values, the robust standard deviations, the standard uncertainties and the standard deviation used to assess the data is given in Table 3-3 and Table 3-5. These tables show that the standard deviation used to assess the data are relatively high and illustrate that z-scores in this study should not be used to judge the performance of laboratories.

# 3 Results

An overview of participating laboratories and submitted data is given in Figure 3-1. In total, 59 laboratories participated of which 35 laboratories were able to submit the results before the deadline. COVID-19 was the main reason that laboratories were unable to submit data before the deadline, even after extension. Compared to the previous round, an increasing number of laboratories participated (59 vs 35) and submitted data (35 vs 27). All 35 laboratories submitted data for the tablet, whereas 24 for the blank tablet. The majority of the laboratories also submitted data for the sediment (32) and fish sample (26).

Most laboratories still reported the number of particles (62-71%). Compared to the last round (n = 2), an increasing number of laboratories (n = 9-11) reported the mass of plastic (i.e. mg/kg by pyr- or TED-GC-MS).

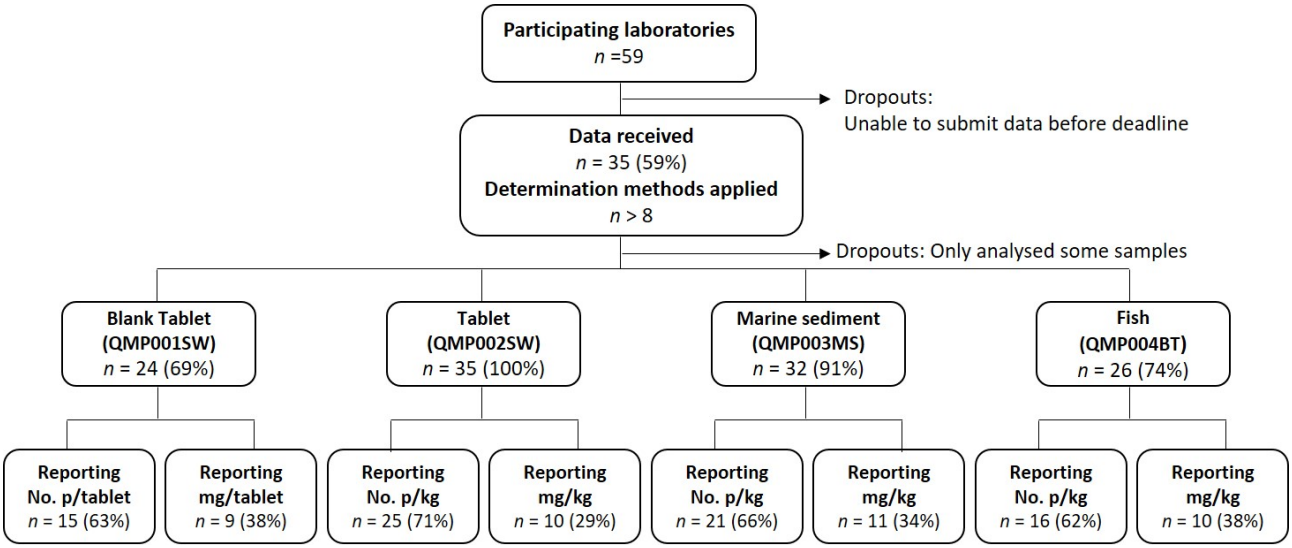


**Figure 3-1** *Flow diagram showing number of participants and number of submitted results.*

## *3.1 Polymer type identification*

Table 3-1 shows the number of laboratories that reported qualitatively, in total, mass and particles. The added polymers were the most abundant reported polymers in the tablets and environmental samples. The majority of laboratories identified the added polymers. Of all added polymers types, polyethylene was found most often (n=15-20), and PMMA least (n=11-13). The majority also identified the correct size fraction (50-299 µm). In all sample types, one to three laboratories occasionally identified the correct polymer type but incorrect size fraction (300-5000 µm).

***Table 3-1 Number of laboratories that correctly identified the polymer types***

| | Polymer added | N labs reporting polymer type | N labs that correctly identified polymer type added | % labs that correctly identified polymer type added |
|---|---|---|---|---|
| QMP002SW | Polyethylene | 23 | 20 | 87% |
| | Polyvinylchloride | | 17 | 74% |
| | Polystyrene | | 19 | 83% |
| | Polyethylene terephthalate | | 16 | 70% |
| QMP003MS | Polymethyl methacrylate | 19 | 13 | 68% |
| | Polyethylene | | 17 | 89% |
| QMP004BT | Polymethyl methacrylate | 17 | 11 | 65% |
| | Polyethylene | | 15 | 88% |
| | Polystyrene | | 12 | 71% |

## *3.2 Determination of plastic particles by numbers*

An overview of inter-comparability of reported size fractions of particles (of added polymers and total) between laboratories is given in Table 3-2. Large differences were found in reported numbers of particles in the tablet (RSD 46-83%), while even larger differences were found for the environmental samples (RSD 85-231%). The differences found in the tablet were comparable to those reported in the previous round, 29-91%.

**Table 3-2 Inter-comparability of reported number of particles (of added polymers and total) between laboratories**

| | Polymer added | RSD (%) - size fraction that was added (50-299 µm) | RSD (%) - all sizes |
|---|---|---|---|
| QMP002SW | Polyethylene | 58 | 57 |
| | Polyvinylchloride | 83 | 55 |
| | Polystyrene | 50 | 49 |
| | Polyethylene terephthalate | 78 | 73 |
| | Total polymers | 46 | 62 |
| QMP003MS | Polymethyl methacrylate | 89 | 103 |
| | Polyethylene | 92 | 97 |
| | Total polymers | 160 | 155 |
| QMP004BT | Polymethyl methacrylate | 96 | 85 |
| | Polyethylene | 125 | 129 |
| | Polystyrene | 117 | 107 |
| | Total polymers | 171 | 231 |

### 3.2.1 Tablet (QMP002SW)

Table 3-3 summarizes the results obtained for the *number of particles* for the spiked tablet (QMP002SW), while Table 3-4 and Figure 3-2 show the *number of particles* in the tablet reported per laboratory. Appendix A provides full details of reported types and number of plastic particles per laboratory. Assigned values for the number of total polymer particles (40/per tablet, Table 3-3) was a factor of 2 lower than the spiked value (76/per tablet, Table 2-1). The assigned values for the number of added polymer particles (8-9 /per tablet) were a factor of 2 or, in case of PE (11 vs 30 /per tablet), even more (factor of 3) lower than the spiked value (15-16 /per tablet). Interestingly, one laboratory (Q101) was close to the number of total polymers added to the tablet, but did not identify the correct polymers (Figure 3-2).

**Table 3-3 Summary statistics for number of added polymers and total number of particles in tablet (QMP002SW)**

| Polymer type | N labs | Assigned value | Robust SD of study | Standard uncertainty | Added number of particles | SD used to calculate z-scores (12,5% of AV + uncertainty) |
|---|---|---|---|---|---|---|
| Polyethylene | 16 | 11 | 6.4 | 2.0 | 15 | 2.5 |
| Polyethylene terephthalate | 12 | 9.3 | 6.8 | 2.5 | 30 | 2.7 |
| Polystyrene | 14 | 9.2 | 4.5 | 1.5 | 15 | 1.9 |
| Polyvinylchloride | 13 | 8.6 | 4.8 | 1.7 | 16 | 2.0 |
| Total polymers | 25 | 40 | 25 | 6.3 | 76 | 8.0 |

**Table 3-4 Reported number of particles per tablet (QMP002SW) for the polymers added, total number of polymers and other polymers**

| Labcode | Polyvinyl chloride total | Polyethylene total (HD+LD) | Polyethylene terephthalate Total | Polystyrene total | Calculated totals for added polymers | Reported totals for polymers | Totals for other polymers |
|---|---|---|---|---|---|---|---|
| Q101 | | | | | | 75 | |
| Q104 | | 9 | 5 | 7 | 21 | 30 | 9 |
| Q110 | 5 | 12 | 11 | 10 | 38 | 40 | 2 |
| Q122 | 7 | 16 | 11 | 12 | 46 | 47 | 1 |
| Q134 | 20 | 17 | 2 | 7 | 46 | 46 | |
| Q3160 | | | | | | 6016 | 6016 |
| Q3231 | 6 | 15 | 4 | 1 | 26 | 56 | 30 |
| Q3877b | | | | | | 137 | |
| Q3878 | 20 | 20 | 10 | 10 | 60 | 192 | 132 |
| Q3882 | 6 | 1 | | 6 | 13 | 17 | 4 |
| Q3886 | | | | | | 7 | |
| Q3887 | 14 | 7 | 18 | 17 | 56 | 57 | 1 |
| Q3888b | 7 | 15 | 4 | 11 | 37 | 46 | 9 |
| Q3889 | 12 | | | | 12 | 31 | 19 |
| Q3890 | 15 | 6 | 18 | 10 | 49 | 50 | 1 |
| Q3911 | 1 | 9 | 8 | 13 | 31 | 38 | 7 |
| Q3913 | 8 | 9 | 15 | 17 | 49 | 49 | |
| Q3917 | 10 | 15 | 9 | 6 | 40 | 42 | 2 |
| Q3926 | | | | | | 49 | |
| Q3932 | | 1634 | | | 1634 | 1634 | |
| Q3936 | | | | | | 19 | |
| Q3940 | | | | | | 255 | |
| Q3943 | | 3 | | 3 | 6 | 6 | |
| Q661 | | | | | | 247 | |
| Q871 | | 2224 | | | 2224 | 2224 | |
| Added | 16 | 15 | 30 | 15 | 76 | 76 | 0 |

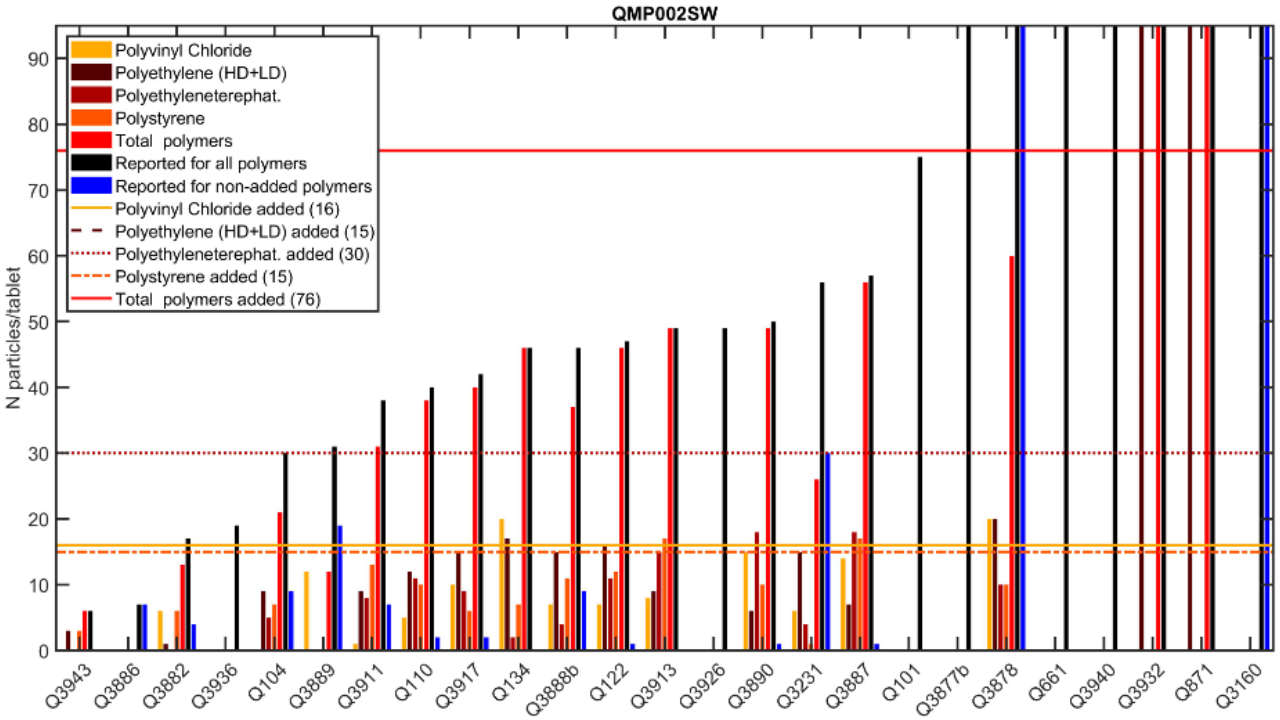


***Figure 3-2*** Reported number of particles for the individual polymers spiked and the total number of polymers for the tablet sample QMP002SW.

### 3.2.2 Environmental samples (QMP003MS and QMP004BT)

Table 3-5 and Figure 3-3 show the number of particles reported per laboratory for the spiked sediment (QMP003MS), and Table 3-6 and Figure 3-4 for spiked fish (QMP004BT). Appendix A provides full details of reported types and number of plastic particles per laboratory. While assigned values are given in Appendix A, z-scores are not calculated due the large differences found in results (Tables 3-2, 3-5 and 3-6). It is clear from Tables 3-5 and 3-6 and Figures 3-3 and 3-4 that most of the reported results were ca. a factor of 10 lower than the added value.

**Table 3-5 Reported number of particles for the polymers added to the sediment sample (QMP003MS), total number of polymers and other polymers**

| Labcode | Polyethyelene | Polymethyl methacrylate | Totals for added polymers | Reported totals for polymers | Totals for other polymers |
|---|---|---|---|---|---|
| Q104 | 2.147.663 | 2.937.695 | 5.085.358 | 5.085.358 | |
| Q110 | 707.921 | 903.210 | 1.611.131 | 1.611.131 | |
| Q122 | 1.558.470 | 1.363.661 | 2.922.131 | 2.922.131 | |
| Q134 | 4.168.421 | 2.344.737 | 6.513.158 | 6.513.158 | |
| Q153 | | | | 54.488 | |
| Q3160 | | | | 5.611.434 | 5.611.434 |
| Q3231 | 507.401 | 428.905 | 936.306 | 936.639 | 333 |
| Q3877b | | | | 3.073.149 | |
| Q3878 | 15.512 | 24.049 | 39.561 | 79.268 | 39.707 |
| Q3886 | | | | 306 | |
| Q3887 | 4.030.190 | 2.997.392 | 7.027.582 | 7.027.582 | |
| Q3889 | 777.989 | | 777.989 | 777.989 | |
| Q3890 | 807.007 | 608.795 | 1.415.802 | 1.415.802 | |
| Q3913 | 1.490.142 | 2.074.736 | 3.564.878 | 3.564.878 | |
| Q3922 | | | | 4.722 | |
| Q3926 | | | | 156.882 | |
| Q3932 | 2.903.587 | | 2.903.587 | 2.903.587 | |
| Q3936 | | | | 133.526 | |
| Q3940 | | | | 20.054 | |
| Q661 | | | | 310.296 | |
| Q871 | 455.515.445 | 192.166.942 | 647.682.387 | 647.682.387 | |
| Added | 4.998.903 | 5.003.367 | 10.002.270 | 10.002.270 | |

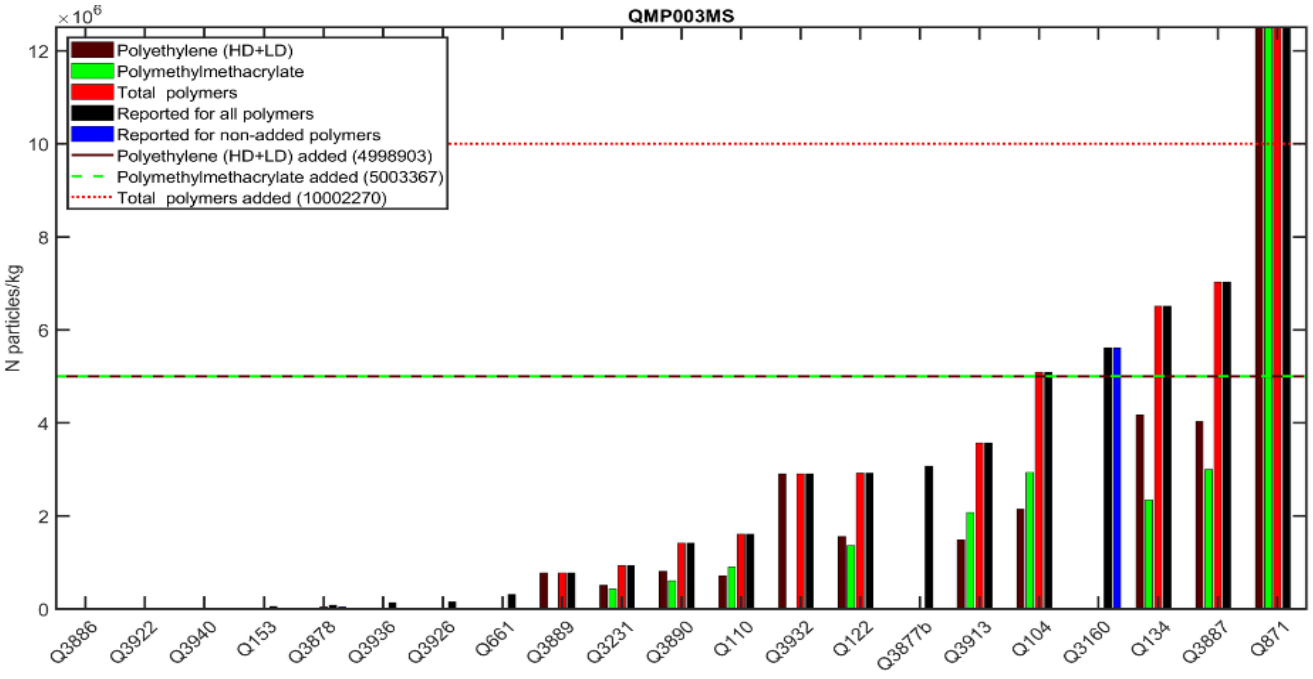


***Figure 3-3*** Total number of particles reported for the polymers added, other polymers and for all polymers in the sediment sample QMP003MS

**Table 3-6 Reported number of particles for the polymers added to the fish sample (QMP004BT), total number of polymers and other polymers**

| Labcode | Polymethyl methacrylate total | Polyethylene total (HD+LD) | Polystyrene total | Calculated totals for added polymers | Reported totals for polymers | Totals for other polymers |
|---|---|---|---|---|---|---|
| Q110 | 3.840.361 | 3.341.114 | 5.440.512 | 12.621.987 | 12.621.987 | |
| Q122 | 2.864.815 | 1.762.963 | 4.187.037 | 8.814.815 | 8.814.815 | |
| Q153 | | | | | 1.050.769 | |
| Q3160 | | | | | 5.382 | 5.382 |
| Q3231 | 409.406 | 1.426.976 | 333.383 | 2.169.765 | 2.174.182 | 4.417 |
| Q3877b | | | | | 2.097.999 | |
| Q3878 | 18.996 | 66.487 | 49.390 | 134.873 | 1.052.391 | 917.518 |
| Q3886 | | 186 | | 186 | 2.050 | 16.864 |
| Q3887 | 1.457.866 | 4.157.385 | 2.646.801 | 8.262.052 | 8.262.053 | |
| Q3889 | | 539.502 | | 539.502 | 545.110 | 5.608 |
| Q3890 | 4.299.162 | 5.080.593 | 2.390.716 | 11.770.471 | 11.770.471 | |
| Q3922 | | | | | 1.538 | |
| Q3926 | | | | | 305.217 | |
| Q3943 | 9.871.725 | 15.794.760 | 740.379 | 26.406.864 | 26.406.864 | |
| Q661 | | | | | 1.300.289 | |
| Q871 | 3.040.850 | 6.976.788 | 1.226.149 | 11.243.787 | 11.243.787 | |
| Added | 10.040.421 | 10.264.701 | 4.939.222 | 25.244.344 | 25.244.344 | |

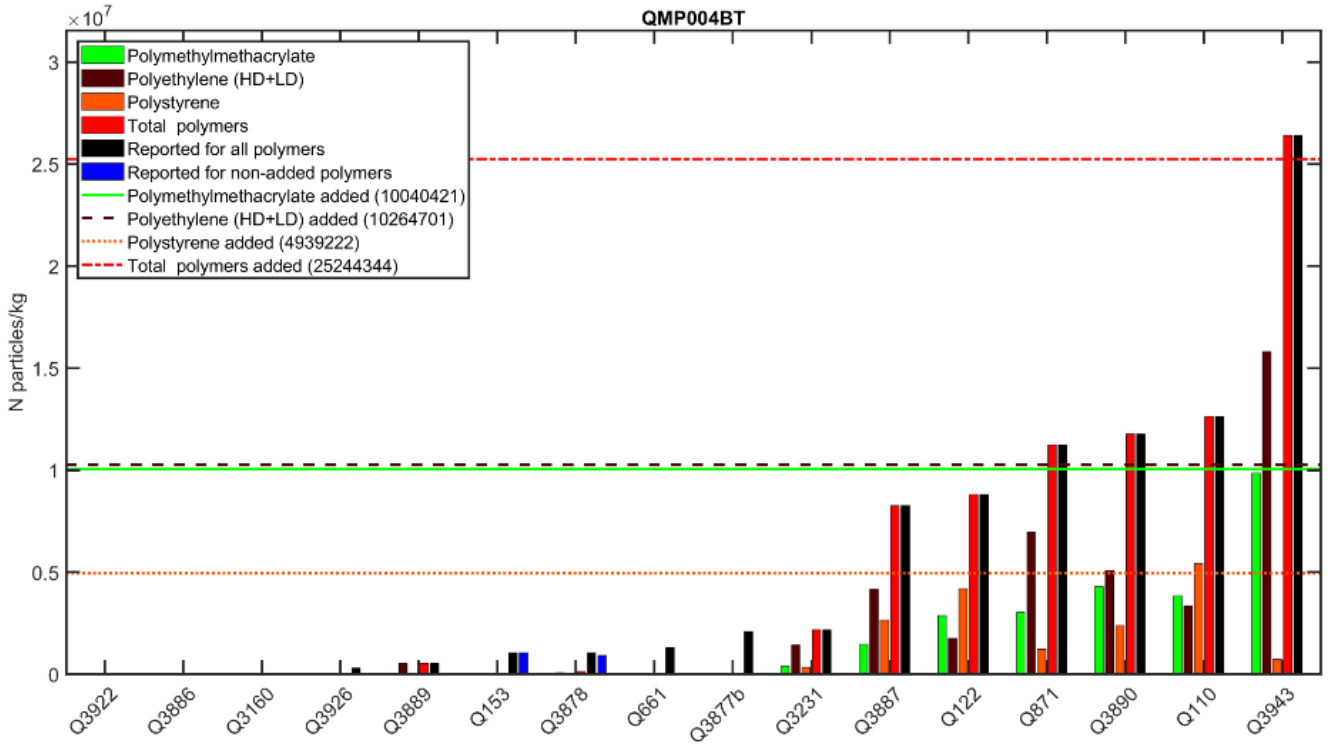


***Figure 3-4*** Total number of particles reported for the polymers added, other polymers and for all polymers in the biota sample QMP004BT.

### *3.3 Determination of plastic particles by mass*

The variation in reported *mass* of total polymers was large, with RSDs of 50, 93 and 120% for the tablet, sediment and fish, respectively. Appendix A provides full details of reported types and mass of particles per laboratory.

Table 3-7 summarizes the results of total polymers by mass obtained for the tablet (QMP002SW), spiked sediment (QMP003MS), and spiked fish (QMP004BT). Table 3-8 and Figure 3-5 show the mass of particles reported per laboratory for the tablet, and Table 3-9 and Figure 3-6 for the environmental samples.

The variations in the reported mass of total polymers, expressed as RSD were 50% for the tablet, 93% for the sediment and 120% for the fish. We have decided not to calculate z-scores, due to the low number of data entries in combination with the high variation in the reported results.

Just like the reported number of particles, the assigned value for the mass of total particles added to the tablet (0.045 mg/tablet) was about half of the spiked mass (0.069 mg/tablet, Table 2-1). The assigned value for the mass of total particles added to the sediment sample (1333 mg/kg) was closer (i.e. factor of 2.2) to the spiked mass (2952 mg/kg) compared to the reported number of particles (factor of 10). The assigned value for the mass of total particles added to the fish sample (6361 mg/kg) was also closer (factor of 1.3) but higher than the spiked value, i.e. 6361 mg/kg versus 4609 mg/kg.

**Table 3-7 Overview of the summary statistics of the reported weights of the total of microplastic polymers found in the spiked samples by pyrolysis GC-MS.**

| Sample | N labs | Units | Assigned Value | Robust SD of study | Robust RSD of study | Standard uncertainty | SD used to calculate z-scores (12.5% of AV + uncertainty) |
|---|---|---|---|---|---|---|---|
| QMP002SW | 9 | mg/tablet | 0.045 | 0.023 | 50% | 0.009 | 0.011 |
| QMP003MS | 11 | mg/kg | 1333 | 1245 | 93% | 469 | - |
| QMP004BT | 10 | mg/kg | 6361 | 7610 | 120% | 3008 | - |

**Table 3-8 Overview reported weights (µg/tablet) of microplastics found in the tablets (QMP001SW and QMP002SW)**

| Sample | Lab code | Poly-amide | Poly-carbonate | Poly-ethylene | Polyethylene terephthalate | Polymethyl methacrylate | Poly-propylene | Poly-styrene | Polyvinyl chloride | Total polymers |
|---|---|---|---|---|---|---|---|---|---|---|
| QMP001SW | H221 | | | | | < 0.1 | | | 0.9 | 0.9 |
| | Q871 | | | | | | | 390 | | 390 |
| | Q980 | | < 7 | < 160 | < 20 | < 5 | < 30 | < 16 | 18 | 18 |
| | Q3175 | 80 | | 60 | 380 | | < 20 | < 10 | < 100 | 520 |
| | Q3929 | | | | | | | | | < 2000 |
| | Q3934 | | | | | | | | | 5700 |
| | Q3935 | | | | | | | | | 50 |
| | Q3936 | | | | | | | | | 23 |
| | Q3941 | | | | | | | | | 160 |
| | | | | | | | | | | |
| QMP002SW | H221 | | | 1.1 | 1.0 | < 0.1 | | 12 | 19 | 33 |
| | Q871 | | | | | | | | | 47 |
| | Q980 | | < 7 | < 160 | < 20 | < 5 | < 30 | 6 | 42 | 48 |
| | Q3175 | 80 | | 50 | 440 | | < 20 | 50 | < 100 | 620 |
| | Q3189 | | | 2 | 9 | | | 2 | 38 | 52 |
| | Q3929 | | | | | | | | | < 2000 |
| | Q3934 | | | | | | | | | 22700 |
| | Q3935 | | | | | | | | | 1010 |
| | Q3936 | | | | | | | | | 45 |
| | Q3941 | | | | | | | | | 180 |
| | Added | | | 21.7 | 14.6 | | | 15.1 | 17.3 | 68.7 |

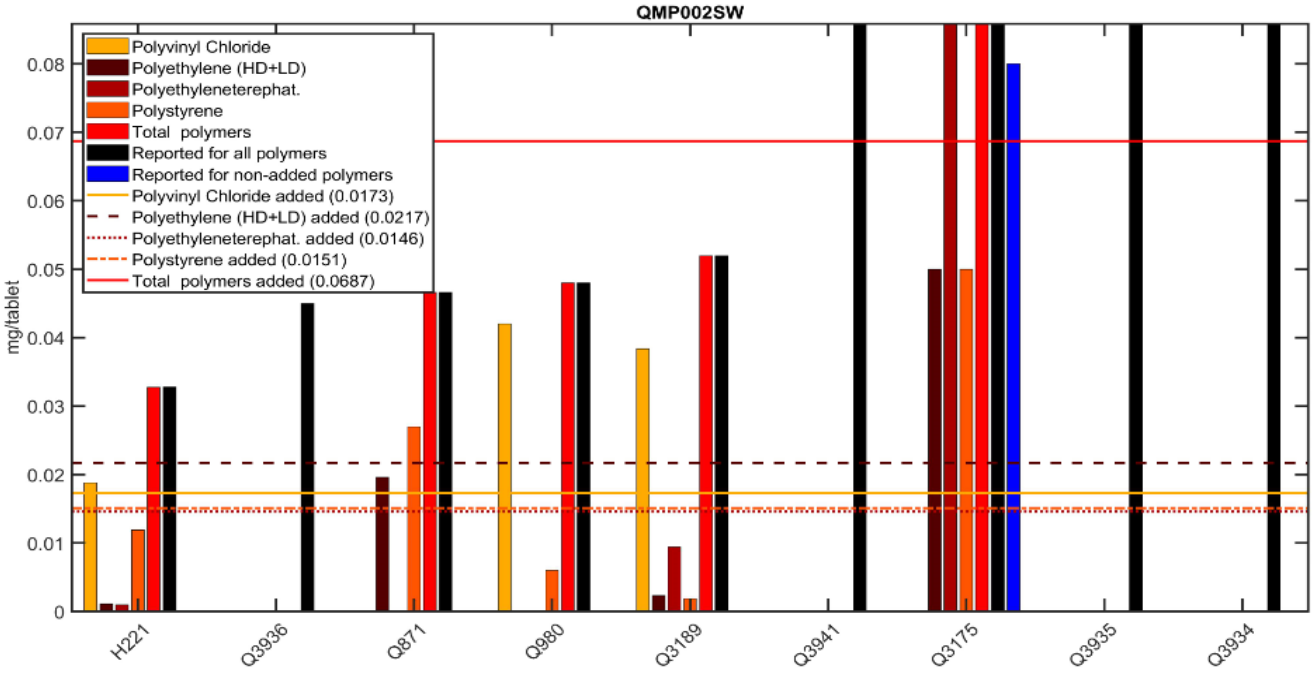


***Figure 3-5*** Reported mass for the added polymers and for the total mass of polymers in the tablet sample QMP002SW.

**Table 3-9 Overview reported weights (mg/kg) of microplastics found in sediment (QMP003MS) and fish (QMP004BT) sample**

| Sample | Labcode | Poly-amide | Poly-carbonate | Poly-ethylene | Polyethyelene terephathalate | Polymethyl methacrylate | Poly-propylene | Poly-styrene | Poly-vinyl chloride | Total polymers |
|---|---|---|---|---|---|---|---|---|---|---|
| QMP003MS | H221 | | | 522 | | 1140 | | < 34.2 | 78 | 1740 |
| | Q871 | | | 2133 | | | | 8.07 | | 2141 |
| | Q980 | | < 1 | 1310 | < 5 | 1330 | < 5 | < 3 | 60 | 2700 |
| | Q3175 | 20.5 | | 152 | 71.9 | | < 0.02 | < 0.01 | < 0.1 | 244 |
| | Q3189 | | | 300 | 1.56 | 185 | | | 7.41 | 494 |
| | Q3877b | | | 936 | | | | | 434 | 1370 |
| | Q3932 | | | 846 | | | | | | 846 |
| | Q3934 | | | | | | | | | 3561 |
| | Q3935 | | | | | | | | | 486 |
| | Q3936 | | | | | | | | | 250 |
| | Q3941 | | | | | | | | | 2161 |
| | Added | | | 900 | | 2052 | | | | 2952 |
| | | | | | | | | | | |
| QMP004BT | H221 | | | 1093 | | 1176 | | 1189 | 398 | 3856 |
| | Q871 | | | 325 | | | | 3222 | | 3547 |
| | Q980 | | < 1 | 4030 | 170 | 1170 | < 5 | 60 | 8900 | 14330 |
| | Q3189 | | | 307 | | 286 | | 345 | | 938 |
| | Q3877b | | | 1356 | | | | 1223 | 8437 | 11016 |
| | Q3929 | | | | | | | | | 11629 |
| | Q3932 | | | 33050 | | | | | | 33050 |
| | Q3934 | | | | | | | | | 60.6 |
| | Q3935 | | | | | | | | | 377 |
| | Q3941 | | | | | | | | | 11844 |
| | Added | | | 1129 | | 1406 | | 2074 | | 4609 |

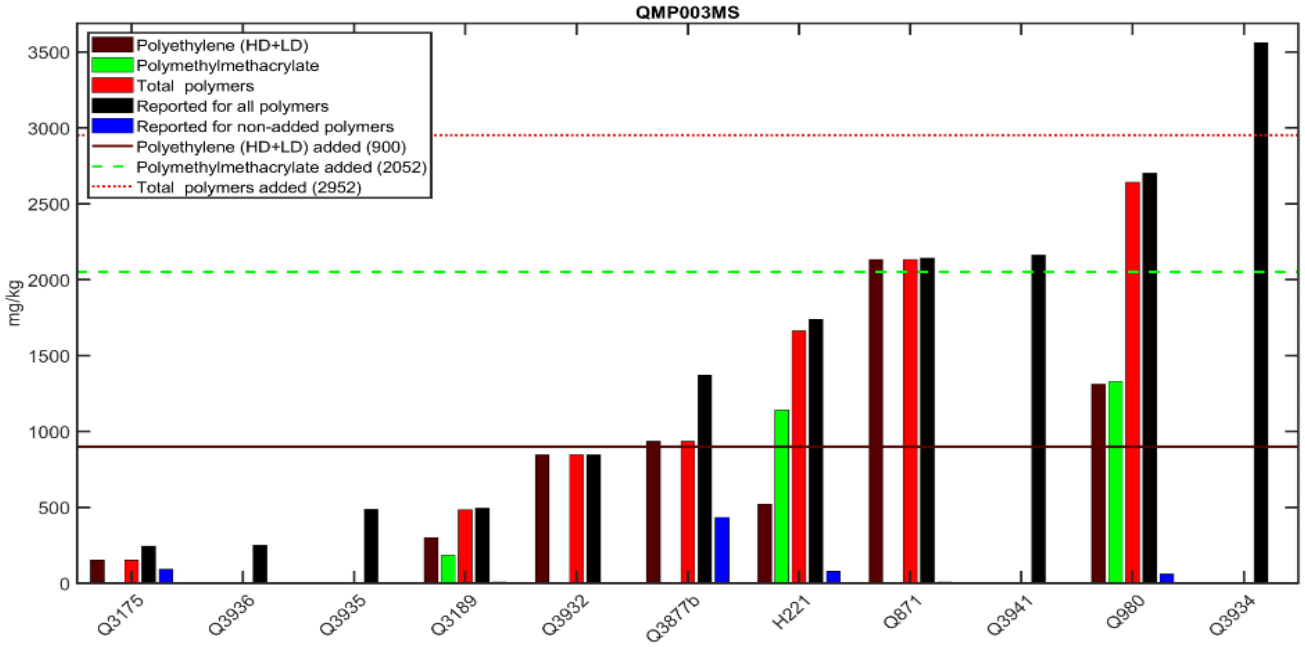


***Figure 3-6*** Total mass reported for the polymers added, other polymers and for all polymers in the sediment sample QMP003MS.

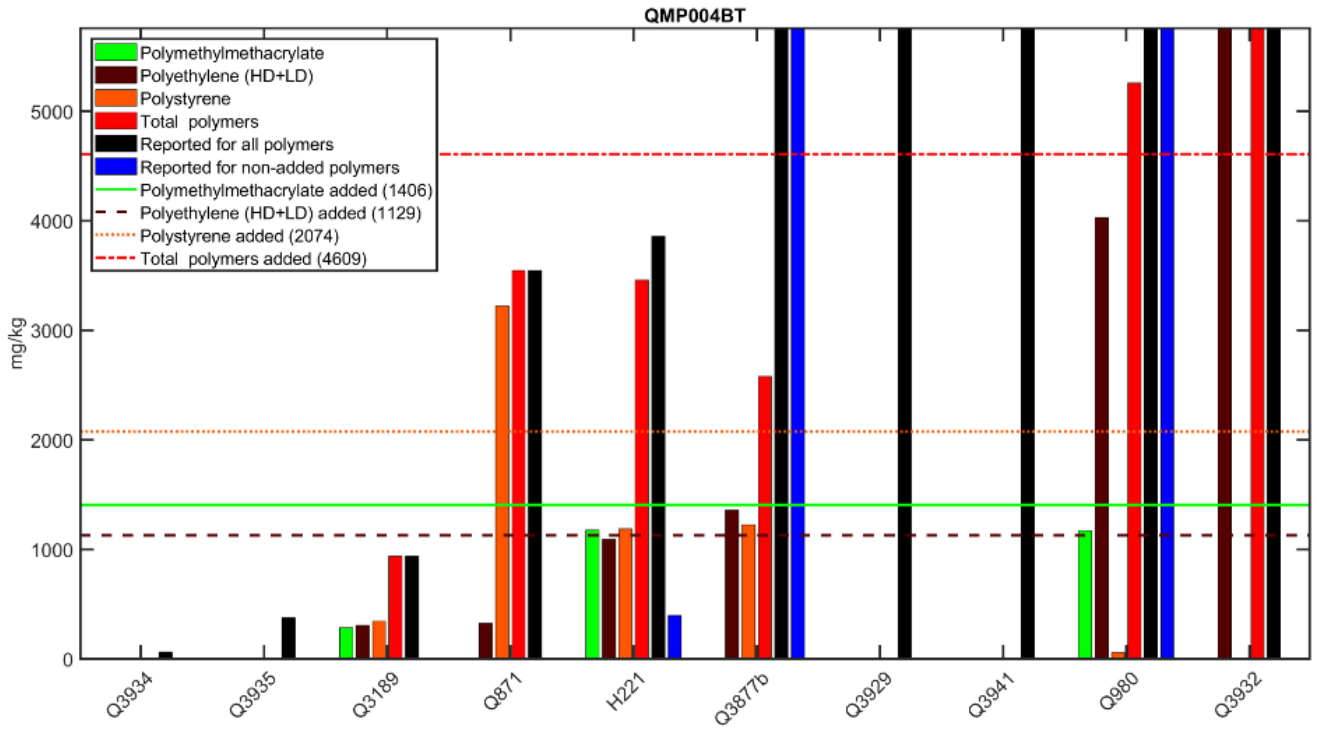


***Figure 3-7*** Total mass reported for the polymers added, other polymers and for all polymers in the fish sample QMP004BT

### *3.4 Z-Scores*

Z-scores for the reported number of particles in tablet are presented in Table 3-10. Z-scores for the biota and sediment samples were not calculated because of the variation in the results uploaded.

**Table 3-10 Z-scores calculated for the polymers added and for the total of polymers added to the tablet sample QMP002SW**

| Laboratory code | Polyethylene | Polyethylene-terephthalate | Polystyrene | Polyvinyl chloride | Total polymers |
|---|---|---|---|---|---|
| Q101 | | | | | 4.3 |
| Q104 | -0.9 | -1.6 | -1.1 | | -1.3 |
| Q110 | 0.3 | 0.6 | 0.4 | -1.9 | 0.0 |
| Q122 | 1.9 | 0.6 | 1.5 | -0.8 | 0.8 |
| Q134 | 2.3 | -2.7 | -1.1 | 5.8 | 0.7 |
| Q3160 | | | | | 745.1 |
| Q3231 | 1.5 | -1.9 | -4.3 | -1.3 | 2.0 |
| Q3877b | | | | | 12.1 |
| Q3878 | 3.6 | 0.3 | 0.4 | 5.8 | 18.9 |
| Q3882 | -4.2 | | -1.7 | -1.3 | -2.9 |
| Q3886 | | | | | -4.1 |
| Q3887 | -1.7 | 3.2 | 4.1 | 2.7 | 2.1 |
| Q3888b | 1.5 | -1.9 | | -0.8 | 0.7 |
| Q3889 | -2.2 | | 1.0 | 1.7 | -1.1 |
| Q3890 | -0.9 | 3.2 | 0.4 | 3.2 | 1.2 |
| Q3911 | -0.9 | -0.5 | 2.0 | -3.9 | -0.3 |
| Q3913 | 1.5 | 2.1 | 4.1 | -0.3 | 1.1 |
| Q3917 | | -0.1 | -1.7 | 0.7 | 0.2 |
| Q3926 | | | | | 1.1 |
| Q3932 | 662.3 | | | | 198.7 |
| Q3936 | | | | | -2.6 |
| Q3940 | | | | | 26.8 |
| Q3943 | -3.4 | | -3.3 | | -4.3 |
| Q661 | | | | | 25.8 |
| Q871 | 903.2 | | | | 272.3 |

# 4 Discussion

## *4.1 Tablets (QMP002SW)*

## *4.2 Marine sediment sample (QMP003MS)*

Several reports describe the amounts of microplastics (MPs) that are present in the aquatic environment. A review by Van Cauwenberghe (2015) shows ranges of MPs in marine and estuarine sediments and on beaches from all over the world. A realistic estimate that emerges from this review for the North Sea shows numbers around 100 particles per kg dry sediment with a size range of 38 μm - 1 mm for both harbors and the central North Sea. In the same review MP data from Florida, USA, are reported in the same range. The intake of a GC/MS pyrolysis cup is ca. 75 μg. However, there are several steps before the sample arrives in the pyrolysis cup. Normally, 5 g of a dry sediment sample is taken for extraction. After dilution, due to extraction (e.g. in 40 mL), sub-sampling into the pyrolysis cup (ca. 75 μg), and a split injection of 1:100, the corresponding amount of dry sediment that is fully pyrolized and brought on the GC column is ca. 0.1 mg. The sensitivity of the pyrolysis analysis is ca. 10 ng absolute. This means that a concentration of 10 ng/100 μg can be detected, or 100 mg/kg. Dierkes et al. (2019) report an LOQ of 7 mg/kg, using the same technique. They concentrated the extracts after extraction. The weight of one PET particle of 100 μm diameter, assuming it has a spherical shape, is ca. 722 ng. The detection limit of of 7 mg/kg of the pyr GC/MS corresponds for this size of PET particle to ca. 140,000 particles per kg. For particles with a diameter of 10 μm this is 140 million particles/kg. For 1 mm diameter particles this will be 140 particles per kg, which comes close to the level of 100 particles per kg dry marine and estuarine North Sea sediment reported by Van Cauwenberghe (2015). For other polymer types the result is somewhat different, dependent of the specific density of the polymer. For wet sediment, about a factor 10 higher sample intake would be needed. These numbers show the difficulty and complexity of the MP analysis. The size of the particles is of utmost importance for the detection limit of the pyrolysis technique. Dierkes et al. (2019) analysed sediment from the river Rhine and found 30 mg/kg for PP and PE. This corresponds roughly with 40,000 particles/kg wet sediment if the diameter would be 100 μm, or ca. 400,000 particles /kg dry sediment.

To enable labs reporting data for this interlab study with different techniques, we have spiked the sediment and fish test materials up to 50,000 – 100,000 particles/kg per polymer with two polymer types in sediment and three in the fish material. The size ranges were ca. 50-100 μm. According to the above calculations, this corresponds with a levels of ca. 72 mg/kg dry sediment, ca. 10-fold higher than the detection limit reported by Dierkes et al. (2019), which seems realistic, although on the low side, given the reported PE and PP levels in river Rhine sediment of ca. 30 mg/kg wet sediment or 300 mg/kg dry sediment (Dierkes et al. (2019).

Unfortunately, because of difficulties encountered in homogenizing the fish and sediment materials, we had to decide to add a fixed number (weight) of MPs to each jar of both test materials. The particles were not homogenously distributed over the samples. This meant that the participants had to take to entire contents of the jars into analysis. The consequence was that participants who used counting techniques, had to count very high numbers of particles. This is obviously a learning element for a next study. Another preparation technique for sediment (and fish) samples is required. These observations also suggest that the sensitivity of the pyrolysis technique is still critical. It works fine for larger particles (>1 mm) but less good for smaller particles. Results from an microplastics interlaboratory study by Becker et al. (2020) for thermographic methods, including seven labs using pyrolysis GC/MS, show good results for concentrations of 5-20 μg/mg or 5-20 g/kg. This is interesting, but it is immediately clear that the microplastics level of that study is 1,000-fold higher than observed in real world samples. Dierkes et al. (2019) reported that levels of ca. 30 mg/kg are found in river Rhine sediment. Therefore, the good results of the study of Becker et al. (2020) do not mean much for analyzing realistic samples. Our spiking level of sediment was more realistic that of Becker et al. (2020), although it created counting difficulties due to the advice to take the entire sample into analysis. The poorer RSD values obtained in the present study, may therefore mainly be caused by the lower MP levels spiked. Table 3.7 shows that when only

considering the labs that used pyrolysis GC/MS, a large RSD value (93%) was found for the total mass of particles. Table 3.9 shows that only for polyethylene reasonable results were reported: 855 mg/kg, range 153-2133 mg/kg, whereas 900 mg/kg was added.

As regards the number of particles in the sediment test material, Table 3-5 shows that results are extremely widely distributed. The high numbers of particles, due to the need to take the entire sample into analysis, have obviously played a role in creating this wide spread in the data, but other factors can certainly not be excluded.

### *4.3 Fish sample (QMP004BT)*

Wang et al. (2020) report a range of microplastic particle numbers in fish, including and probably mainly in the intestine track of 0.34 – 19 per individual fish for the Mediterranean and Rio de la Plata, resp. Jovanovic (2017) reports 0.01-2.2 particles in the gastro intestinal tract of various fishes worldwide with 1-6 particles per fish in which microplastic particles were detected (in several fishes no particles were detected). The size range reported is 26 μm – 5 mm. Clearly, for this exercise the numbers of particles spiked for each polymer, ca. 10,000,000 per jar of 5 g, were much higher than occurs in nature. Mass-based techniques would have great difficulty to find one or two particles in a fish, even for the larger particles. Although the absolute detection limit of pyr GC/MS is ca. 10 ng and one 100 μm particle of e.g. PET weighs 722 ng, the issue is how to get that one particle in the final pyrolysis cup. Sub-sampling, concentration and splitting ratios are all critical. Mussels contain higher numbers of particles. Reports from the UK and Norway give a range of 1-6 and 1.5 (±2.3) particles per individual mussel (*Mytilus edulis*), respectively (Li et al., 2018, Bråte et al., 2018). However, a large part of that (83% according to Bråte et al. (2018)) are fibers. According to Qu et al. (2018), fibers accounted for > 60% of the microplastics in field investigations. It means that also for mussels mass-based techniques may have difficulties to quantify the mass of these low numbers of particles. Large sample intakes and thorough concentration will be needed. Qu et al. showed that the number of microplastic particles present in individual mussels (*Mytilus edulis* and *Perna viridis*) in Chinese waters is roughly the same as the number of microplastic particles per L seawater, with fibers being the dominant microplastics. The sizes of microplastics in the mussels were smaller than those in the water.
In the present study, the results for mass, determined with pyr GC/MS show an RSD value of 120%. The high standard deviation may be caused by the high variation that occurs when analyzing the small cups. Several cups will be needed to exclude the inhomogeneity that plays an important role in sub-sampling 75 μg. Dierkes et al. (2019) recommend cryo-milling of environmental samples to improve the homogeneity of the samples and a better distribution of the MP particles over the sample. The results for the numbers of particles in Table 3-6 widely vary between 186 and 15.7 million, the last one being 'only' 50% off. Also for this fish materials, the particles were not homogenously distributed over the samples and we had to ask the participants to take into analysis the entire content of the jar, which was, for those that used counting methods, not a good idea. Z-scores were not given as they would be meaningless. It confirms that the spiking of the fish tissue was unrealistic and especially counting techniques were not prepared for analyzing the entire fish content of the jar.

## Conclusions

The second round of this microplastics interlaboratory study has delivered valuable information on the current state-of-the-art of laboratories analysing microplastics. The increased number of participants, even in spite of the covid-19 crisis, was reassuring and showed the high interest worldwide. The results show a mixed picture. The identification of the polymer types in the tablets provided was generally good. The quantification in the tablets, and certainly also in the two environmental test materials was far from what is needed. Pyrolysis GC/MS was used more frequently now, but results were not better than in the first round of this exercise. With regard to the preparation of materials, it is clear that different techniques are required to ensure homogeneous test materials that represent the various environmental compartments. For fish and/or shellfish it is a challenge to supply test materials with realistic levels of microplastics, and the same is true for test materials containing fibers.

The results and challenges of the first and second round will be presented and in the upcoming workshop (20-21 May 2021). Final conclusions can then be drawn. During this workshop, the study design of the third round will also be discussed.

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# Appendices

A. Reported data and graphical output

B. Additional method information

D. NDA statistics

Appendix A

# Total number of particles Analysis DE17 2020.1

# 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 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **QMP001SW** | | | | | | | | | | | |
| Total polymers | 6.38 | (No. p/tablet) | | 9.3 | 146 | 14 | 1 | 9.50 | 7.50 | 6.38 | 3.11 |

| Sample/ Determinand | Assigned Value | Units | Total Error | NDA st.dev | NDA rel. st.dev (%) | Nobs numerical | Nobs LCV | Median | MAD | Model Mean | Uncer-tainty |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **QMP002SW** | | | | | | | | | | | |
| PE (50-299 µm) | 10.6 | (No. p/tablet) | | 6.1 | 57.5 | 14 | 0 | 10.5 | 4.50 | 10.61 | 2.04 |
| Polyethylene total (HD+LD) | 11.3 | (No. p/tablet) | | 6.4 | 57.0 | 16 | 0 | 13.5 | 4.50 | 11.27 | 2.01 |
| PET (50-299 µm) | 8.34 | (No. p/tablet) | | 6.52 | 78.1 | 12 | 0 | 8.50 | 4.50 | 8.344 | 2.35 |
| Polyethyleneterephat. Total | 9.30 | (No. p/tablet) | | 6.82 | 73.3 | 12 | 0 | 9.50 | 5.00 | 9.296 | 2.46 |
| PS (50-299 µm) | 8.94 | (No. p/tablet) | | 4.46 | 49.9 | 12 | 0 | 9.00 | 3.00 | 8.943 | 1.61 |
| Polystyrene total | 9.17 | (No. p/tablet) | | 4.50 | 49.1 | 14 | 0 | 10.00 | 3.00 | 9.174 | 1.50 |
| PVC (50-299 µm) | 9.35 | (No. p/tablet) | | 7.76 | 83.1 | 12 | 0 | 9.00 | 5.50 | 9.349 | 2.80 |
| Polyvinyl Chloride total | 8.65 | (No. p/tablet) | | 4.76 | 55.1 | 13 | 0 | 8.00 | 3.00 | 8.646 | 1.65 |
| Total polymers (50-299 µm) | 39.9 | (No. p/tablet) | | 18.4 | 46.1 | 16 | 0 | 44.0 | 13.0 | 39.86 | 5.74 |
| Total polymers | 40.2 | (No. p/tablet) | | 25.0 | 62.2 | 25 | 0 | 49.0 | 19.0 | 40.20 | 6.25 |

| Sample/ Determinand | Assigned Value | Units | Total Error | NDA st.dev | NDA rel. st.dev (%) | Nobs numerical | Nobs LCV | Median | MAD | Model Mean | Uncer-tainty |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **QMP003MS** | | | | | | | | | | | |
| PE (50-299 µm) | 1299420 | (No. p/kg) | | 1196400 | 92.1 | 11 | 0 | 1490142 | 782221 | 1299420 | 450910 |
| Polyethylene total (HD+LD) | 1395786 | (No. p/kg) | | 1355065 | 97.1 | 12 | 0 | 1524306 | 916645 | 1395786 | 488967 |
| PMMA (50-299µm) | 1489059 | (No. p/kg) | | 1327153 | 89.1 | 9 | 0 | 1363661 | 934756 | 1489059 | 552980 |
| Polymethylmethacrylate total | 1501040 | (No. p/kg) | | 1546763 | 103.0 | 10 | 0 | 1719199 | 1164450 | 1501040 | 611418 |
| Total polymers (50-299 µm) | 1011523 | (No. p/kg) | | 1614963 | 159.7 | 14 | 0 | 1176221 | 1109343 | 1011523 | 539521 |
| Total polymers | 1344449 | (No. p/kg) | | 2086266 | 155.2 | 21 | 0 | 1415802 | 1411080 | 1344449 | 569076 |

# 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 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **QMP004BT** | | | | | | | | | | | |
| PE (50-299 µm) | 1883359 | (No. p/kg) | | 2358360 | 125.2 | 9 | 0 | 1762963 | 1702175 | 1883359 | 982650 |
| Polyethylene total (HD+LD) | 2379800 | (No. p/kg) | | 3069872 | 129.0 | 10 | 0 | 2552039 | 2249044 | 2379800 | 1213473 |
| PMMA (50-299µm) | 2192190 | (No. p/kg) | | 2104095 | 96.0 | 7 | 0 | 2864815 | 1434347 | 2192190 | 994091 |
| Polymethylmethacrylate total | 2392705 | (No. p/kg) | | 2024222 | 84.6 | 8 | 0 | 2952833 | 1420648 | 2392705 | 894588 |
| PS (50-299 µm) | 2107184 | (No. p/kg) | | 2473047 | 117.4 | 7 | 0 | 2390716 | 1796321 | 2107184 | 1168405 |
| Polystyrene total | 1769320 | (No. p/kg) | | 1890362 | 106.8 | 8 | 0 | 1808433 | 1271552 | 1769320 | 835430 |
| Total polymers (50-299 µm) | 2068174 | (No. p/kg) | | 3539966 | 171.2 | 11 | 0 | 2174182 | 2172132 | 2068174 | 1334175 |
| Total polymers | 941297 | (No. p/kg) | | 2173988 | 231.0 | 16 | 0 | 1699144 | 1695428 | 941297 | 679371 |

# Total number of particles Data and Statistics

| Sample | QMP001SW | QMP002SW | QMP003MS | QMP004BT | MIC |
|---|---|---|---|---|---|
| **ABS (50-299 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q3160 | 6726 | 2005 | 5611434 | - | ZA\|ZA\|ZA\|MI\|ZA |
| Q3878 | - | 5.00 | 439 | 58128 | ZA\|ZA\| -\|µF |
| Q3886 | - | 1.00 | - | 186 | ZA\|ZA\| -\|µF\|ZA |
| **ABS (300-5000 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q3160 | 8968 | 4011 | - | 5382 | ZB\|ZB\|ZB\|MI\|ZB |
| Q3878 | - | - | - | 2280 | ZA\|ZA\| -\|µF |
| **Acrylon.Butadi.Styrene total** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q3160 | 15694 | 6016 | 5611434 | 5382 | ZA\|ZA\|ZA\|MI\|ZA |
| Q3878 | - | 5.00 | 439 | 60408 | ZA\|ZA\| -\|µF |
| Q3886 | - | 1.00 | - | 186 | ZA\|ZA\| -\|µF\|ZA |
| **PA (50-299 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q122 | - | 1.00 | - | - | ZA\| -\| -\|µF\|ZA |
| Q153 | 9.00 | - | - | - | ZC\|ZA\|ZA\|ZB\|ZA |
| Q3231 | 1.00 | - | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 10.0 | 97.6 | 241442 | ZA\|ZA\| -\|µF |
| Q3886 | - | - | - | 186 | ZA\|ZA\| -\|µF\|ZA |
| **PA (300-5000 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q3231 | - | 2.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | - | - | 7788 | ZA\|ZA\| -\|µF |
| Q3887 | 2.00 | - | - | - | |
| **Polyamide total** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q122 | - | 1.00 | - | - | ZA\| -\| -\|µF\|ZA |
| Q153 | 9.00 | - | - | - | ZC\|ZA\|ZA\|ZB\|ZA |
| Q3231 | 1.00 | 2.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 10.0 | 97.6 | 249231 | ZA\|ZA\| -\|µF |
| Q3886 | - | - | - | 186 | ZA\|ZA\| -\|µF\|ZA |
| Q3887 | 2.00 | - | - | - | |
| **PC (50-299 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q3878 | - | 10.0 | - | 73136 | ZA\|ZA\| -\|µF |
| Q3886 | - | - | - | 186 | ZA\|ZA\| -\|µF\|ZA |
| **PC (300-5000 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q3878 | - | 1.00 | - | - | ZA\|ZA\| -\|µF |
| **Polycarbonate total** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q3878 | - | 11.0 | - | 73136 | ZA\|ZA\| -\|µF |
| Q3886 | - | - | - | 186 | ZA\|ZA\| -\|µF\|ZA |

# Total number of particles Data and Statistics

| Sample | QMP001SW | QMP002SW | QMP003MS | QMP004BT | MIC |
|---|---|---|---|---|---|
| **PE (50-299 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q104 | - | 9.00 | 2147613 | - | |
| Q110 | - | 12.0 | 707921 | 3341114 | ZA\|ZA\|ZA\|AF\|ZA |
| Q122 | - | 15.0 | 1558470 | 1762963 | -\|ZB\| -\|µF\|ZB |
| Q134 | - | 17.0 | 4168421 | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3231 | 1.00 | 5.00 | 507401 | 1426976 | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 15.0 | 15512 | 60788 | ZA\|ZA\| -\|µF |
| Q3886 | - | - | - | 186 | ZA\|ZA\| -\|µF\|ZA |
| Q3887 | - | 7.00 | 4030190 | 4157385 | |
| Q3888b | 8.00 | 15.0 | - | - | ZA\| -\| -\|µF |
| Q3889 | - | - | 777989 | 539502 | ZC\| -\|ZA\|µF |
| Q3890 | - | 6.00 | 807007 | 5080593 | ZC\| -\|ZB\|ZA\|ZC |
| Q3911 | - | 9.00 | - | - | ZB\| -\|ZB\|µF\|ZB |
| Q3913 | - | 9.00 | 1490142 | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3917 | - | 15.0 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3932 | - | 1634 | 2903587 | - | ZB\|ZB\|ZB\|AF\|ZB |
| Q3943 | - | 3.00 | - | 15794760 | ZA\| -\|ZB\|ZB\|ZB |
| | ========== | Statistical Results | ========== | | |
| NDA mean | - | 10.61 | 1299420 | 1883359 | |
| NDA st dev | - | 6.10 | 1196400 | 2358360 | |
| Coeff Var (%) | - | 57.5 | 92.1 | 125.2 | |
| N | 2 | 14 | 11 | 9 | |
| Median | 4.5 | 10.5 | 1490142 | 1762963 | |
| MAD | 3.5 | 4.5 | 782221 | 1702175 | |
| Added Number | - | 15 | 4998903 | 10264701 | |
| | ========== | | | | |

| PE (300-5000 µm) | (No. p/tablet) | (No. p/tablet) | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|
| Q104 | - | - | 50.0 | - | |
| Q122 | - | 1.00 | - | - | ZA\| -\| -\|µF\|ZA |
| Q3231 | - | 10.0 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 5.00 | - | 5699 | ZA\|ZA\| -\|µF |

| Polyethylene total (HD+LD) | (No. p/tablet) | (No. p/tablet) | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|
| Q104 | - | 9.00 | 2147663 | - | |
| Q110 | - | 12.0 | 707921 | 3341114 | ZA\|ZA\|ZA\|AF\|ZA |
| Q122 | - | 16.0 | 1558470 | 1762963 | ZA\| -\| -\|µF\|ZA |
| Q134 | - | 17.0 | 4168421 | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3231 | 1.00 | 15.0 | 507401 | 1426976 | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 20.0 | 15512 | 66487 | ZA\|ZA\| -\|µF |
| Q3882 | - | 1.00 | - | - | ZA\|ZA\| -\|µF\|ZA |
| Q3886 | - | - | - | 186 | ZA\|ZA\| -\|µF\|ZA |
| Q3887 | - | 7.00 | 4030190 | 4157385 | |
| Q3888b | 8.00 | 15.0 | - | - | ZA\| -\| -\|µF |
| Q3889 | - | - | 777989 | 539502 | ZC\| -\|ZA\|µF |
| Q3890 | - | 6.00 | 807007 | 5080593 | ZB\|ZA\|ZA\|µF\|ZB |
| Q3911 | - | 9.00 | - | - | ZB\| -\|ZB\|µF\|ZB |
| Q3913 | - | 9.00 | 1490142 | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3917 | - | 15.0 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3932 | - | 1634 | 2903587 | - | |
| Q3943 | - | 3.00 | - | 15794760 | |
| Q871 | 567 | 2224 | 455515445 | 6976788 | |
| | ========== | Statistical Results | ========== | | |
| NDA mean | - | 11.27 | 1395786 | 2379800 | |
| NDA st dev | - | 6.42 | 1355065 | 3069872 | |
| Coeff Var (%) | - | 57.0 | 97.1 | 129.0 | |
| N | 3 | 16 | 12 | 10 | |
| Median | 8.0 | 13.5 | 1524306 | 2552039 | |
| MAD | 7.0 | 4.5 | 916645 | 2249044 | |
| Added Number | - | 15 | 4998903 | 10264701 | |
| | ========== | | | | |

# Total number of particles Data and Statistics

| Sample | QMP001SW | QMP002SW | QMP003MS | QMP004BT | MIC |
|---|---|---|---|---|---|
| **PET (50-299 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q104 | - | 5.00 | - | - | |
| Q110 | - | 11.0 | - | - | ZA\|ZA\|ZA\|AF\|ZA |
| Q122 | - | 11.0 | - | - | ZA\| -\| -\|µF\|ZA |
| Q134 | - | 2.00 | - | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3231 | - | 2.00 | - | 2250 | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 5.00 | 683 | 82824 | ZA\|ZA\| -\|µF |
| Q3887 | - | 18.0 | - | - | |
| Q3888b | - | 3.00 | - | - | ZA\| -\| -\|µF |
| Q3890 | - | 18.0 | - | - | ZA\| -\| -\|µF\|ZA |
| Q3911 | - | 8.00 | - | - | ZB\| -\|ZB\|µF\|ZB |
| Q3913 | - | 15.0 | - | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3917 | - | 9.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| | ========== | Statistical Results | ========== | | |
| NDA mean | - | 8.34 | - | - | |
| NDA st dev | - | 6.52 | - | - | |
| Coeff Var (%) | - | 78.1 | - | - | |
| N | - | 12 | 1 | 2 | |
| Median | - | 8.5 | 683 | 42537 | |
| MAD | - | 4.5 | - | 40287 | |
| Added Number | - | 30 | - | - | |
| | ========== | ========== | ========== | ========== | |

| PET (300-5000 µm) | (No. p/tablet) | (No. p/tablet) | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|
| Q3231 | - | 2.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | 1.00 | 5.00 | - | 3799 | ZA\|ZA\| -\|µF |
| Q3888b | - | 1.00 | - | - | ZA\| -\| -\|µF |

| Polyethyleneterephat. Total | (No. p/tablet) | (No. p/tablet) | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|
| Q104 | - | 5.00 | - | - | |
| Q110 | - | 11.0 | - | - | ZA\|ZA\|ZA\|AF\|ZA |
| Q122 | - | 11.0 | - | - | ZA\| -\| -\|µF\|ZA |
| Q134 | - | 2.00 | - | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3231 | - | 4.00 | - | 2250 | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | 1.00 | 10.0 | 683 | 86623 | ZA\|ZA\| -\|µF |
| Q3887 | - | 18.0 | - | - | |
| Q3888b | - | 4.00 | - | - | ZA\| -\| -\|µF |
| Q3890 | - | 18.0 | - | - | |
| Q3911 | - | 8.00 | - | - | ZB\| -\|ZB\|µF\|ZB |
| Q3913 | - | 15.0 | - | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3917 | - | 9.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q871 | 3.00 | - | - | - | |
| | ========== | Statistical Results | ========== | | |
| NDA mean | - | 9.30 | - | - | |
| NDA st dev | - | 6.82 | - | - | |
| Coeff Var (%) | - | 73.3 | - | - | |
| N | 2 | 12 | 1 | 2 | |
| Median | 2.000 | 9.5 | 683 | 44436 | |
| MAD | 1.000 | 5.0 | - | 42186 | |
| Category | - | 30 | - | - | |
| | ========== | ========== | ========== | ========== | |

# Total number of particles Data and Statistics

| Sample | QMP001SW | QMP002SW | QMP003MS | QMP004BT | MIC |
|---|---|---|---|---|---|
| **PLA (50-299 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q3878 | - | 5.00 | 48.8 | 221306 | ZA\|ZA\| -\|µF |
| **PLA (300-5000 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q3878 | - | - | - | 190 | ZA\|ZA\| -\|µF |
| **Polyactic Acid total** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q3878 | - | 5.00 | 48.8 | 221496 | ZA\|ZA\| -\|µF |
| **PMMA (50-299µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q104 | - | - | 2937695 | - | |
| Q110 | - | - | 903210 | 3840361 | ZA\|ZA\|ZA\|AF\|ZA |
| Q122 | - | - | 1363661 | 2864815 | -\|ZB\| -\|µF\|ZB |
| Q134 | - | - | 2344737 | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3231 | 4.00 | 1.00 | 428905 | 409406 | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 60.0 | 24049 | 17097 | ZA\|ZA\| -\|µF |
| Q3887 | - | - | 2997392 | 1457866 | |
| Q3890 | - | - | 608795 | 4299162 | ZC\| -\|ZB\|ZA\|ZC |
| Q3913 | - | - | 2074736 | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3943 | - | - | - | 9871725 | ZA\| -\|ZB\|ZB\|ZB |
| | ========== | Statistical Results | ========== | | |
| NDA mean | - | - | 1489059 | 2192190 | |
| NDA st dev | - | - | 1327153 | 2104095 | |
| Coeff Var (%) | - | - | 89.1 | 96.0 | |
| N | 1 | 2 | 9 | 7 | |
| Median | 4.0 | 30.5 | 1363661 | 2864815 | |
| MAD | - | 29.5 | 934756 | 1434347 | |
| Added Number | | - | 5003367 | 10040421 | |
| | ========== | | | | |
| **PMMA (300-5000µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q3231 | - | 2.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 3.00 | - | 1897 | ZA\|ZA\| -\|µF |
| **Polymethylmethacrylate total** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q104 | - | - | 2937695 | - | |
| Q110 | - | - | 903210 | 3840361 | ZA\|ZA\|ZA\|AF\|ZA |
| Q122 | - | - | 1363661 | 2864815 | ZA\| -\| -\|µF\|ZA |
| Q134 | - | - | 2344737 | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3231 | 4.00 | 3.00 | 428905 | 409406 | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 63.0 | 24049 | 18996 | ZA\|ZA\| -\|µF |
| Q3887 | - | - | 2997392 | 1457866 | |
| Q3890 | - | - | 608795 | 4299162 | |
| Q3913 | - | - | 2074736 | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3943 | - | - | - | 9871725 | |
| Q871 | 5.00 | - | 192166942 | 3040850 | |
| | ========== | Statistical Results | ========== | | |
| NDA mean | - | - | 1501040 | 2392705 | |
| NDA st dev | - | - | 1546763 | 2024222 | |
| Coeff Var (%) | - | - | 103.0 | 84.6 | |
| N | 2 | 2 | 10 | 8 | |
| Median | 4.5 | 33.0 | 1719199 | 2952833 | |
| MAD | 0.5 | 30.0 | 1164450 | 1420648 | |
| Added number | - | - | 5003367 | 10040421 | |
| | ========== | | | | |

# Total number of particles Data and Statistics

| Sample QMP001SW | QMP002SW | QMP003MS | QMP004BT | MIC | |
|---|---|---|---|---|---|
| **PP (50-299 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q104 | 5.00 | 9.00 | - | - | |
| Q3231 | 1.00 | 14.0 | 333 | 2167 | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 3.00 | 16000 | 43501 | ZA\|ZA\| -\|µF |
| Q3888b | 4.00 | 2.00 | - | - | ZA\| -\| -\|µF |
| Q3890 | - | 1.00 | - | - | ZA\| -\| -\|µF\|ZA |
| Q3911 | - | 7.00 | - | - | ZB\| -\|ZB\|µF\|ZB |
| **PP (300-5000 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q104 | 2.00 | - | - | - | |
| Q153 | - | - | - | 1.00 | ZC\|ZA\|ZA\|ZB\|ZA |
| Q3231 | - | 11.0 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | - | - | 3799 | ZA\|ZA\| -\|µF |
| **Polypropylene total** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q104 | 7.00 | 9.00 | - | - | |
| Q3231 | 1.00 | 25.0 | 333 | 2167 | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 3.00 | 16000 | 47301 | ZA\|ZA\| -\|µF |
| Q3888b | 4.00 | 2.00 | - | - | ZA\| -\| -\|µF |
| Q3890 | - | 1.00 | - | - | |
| Q3911 | - | 7.00 | - | - | ZB\| -\|ZB\|µF\|ZB |
| Q871 | 3.00 | - | - | - | |
| **PS (50-299 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q104 | - | 7.00 | - | - | |
| Q110 | - | 10.0 | - | 5440512 | ZA\|ZA\|ZA\|AF\|ZA |
| Q122 | - | 12.0 | - | 4187037 | ZA\| -\| -\|µF\|ZA |
| Q134 | - | 7.00 | - | - | ZA\|ZA\| -\|ZA\|ZA |
| Q153 | 1.00 | - | - | - | ZC\|ZA\|ZA\|ZB\|ZA |
| Q3231 | 1.00 | - | - | 333383 | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 5.00 | 17317 | 49390 | ZA\|ZA\| -\|µF |
| Q3887 | - | 17.0 | - | 2646801 | ZA\|ZA\|ZA\|AF\|ZA |
| Q3888b | 8.00 | 8.00 | - | - | ZA\| -\| -\|µF |
| Q3890 | - | 10.0 | - | 2390716 | ZC\| -\|ZB\|ZA\|ZC |
| Q3911 | 2.00 | 13.0 | - | - | ZB\| -\|ZB\|µF\|ZB |
| Q3913 | - | 17.0 | - | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3917 | - | 6.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3943 | - | 3.00 | - | 740379 | ZA\| -\|ZB\|ZB\|ZB |
| | ========== | Statistical Results | ========== | | |
| NDA mean | - | 8.94 | - | 2107184 | |
| NDA st dev | - | 4.46 | - | 2473047 | |
| Coeff Var (%) | - | 49.9 | - | 117.4 | |
| N | 4 | 12 | 1 | 7 | |
| Median | 1.5 | 9.0 | 17317 | 2390716 | |
| MAD | 0.5 | 3.0 | - | 1796321 | |
| Added number | - | 15 | - | 4939222 | |
| | ========== | | | | |
| **PS (300-5000 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q3231 | - | 1.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 5.00 | - | - | ZA\|ZA\| -\|µF |
| Q3887 | 1.00 | - | - | - | |
| Q3888b | 1.00 | 3.00 | - | - | ZA\| -\| -\|µF |
| Q3911 | 2.00 | - | - | - | ZB\| -\|ZB\|µF\|ZB |

# Total number of particles Data and Statistics

| Sample | QMP001SW | QMP002SW | QMP003MS | QMP004BT | MIC |
|---|---|---|---|---|---|
| **Polystyrene total** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q104 | - | 7.00 | - | - | |
| Q110 | - | 10.0 | - | 5440512 | ZA\|ZA\|ZA\|AF\|ZA |
| Q122 | - | 12.0 | - | 4187037 | ZA\| -\| -\|µF\|ZA |
| Q134 | - | 7.00 | - | - | ZA\|ZA\| -\|ZA\|ZA |
| Q153 | 1.00 | - | - | - | ZC\|ZA\|ZA\|ZB\|ZA |
| Q3231 | 1.00 | 1.00 | - | 333383 | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 10.0 | 17317 | 49390 | ZA\|ZA\| -\|µF |
| Q3882 | - | 6.00 | - | - | ZA\|ZA\| -\|µF\|ZA |
| Q3887 | 1.00 | 17.0 | - | 2646801 | ZA\|ZA\|ZA\|AF\|ZA |
| Q3888b | 9.00 | 11.0 | - | - | ZA\| -\| -\|µF |
| Q3890 | - | 10.0 | - | 2390716 | |
| Q3911 | 4.00 | 13.0 | - | - | ZB\| -\|ZB\|µF\|ZB |
| Q3913 | - | 17.0 | - | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3917 | - | 6.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3943 | - | 3.00 | - | 740379 | |
| Q871 | 1.00 | - | - | 1226149 | |
| | ========== | Statistical Results | ========== | | |
| NDA mean | - | 9.17 | - | 1769320 | |
| NDA st dev | - | 4.50 | - | 1890362 | |
| Coeff Var (%) | - | 49.1 | - | 106.8 | |
| N | 6 | 14 | 1 | 8 | |
| Median | 1.000 | 10.0 | 17317 | 1808433 | |
| MAD | - | 3.0 | - | 1271552 | |
| Added number | - | 15 | - | 4939222 | |
| | ========== | ========== | ========== | ========== | |

| PUR (50-299 µm) | (No. p/tablet) | (No. p/tablet) | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|
| Q110 | - | 2.00 | - | - | ZA\|ZA\|ZA\|AF\|ZA |
| Q3878 | - | 20.0 | 244 | 123096 | ZA\|ZA\| -\|µF |
| Q3886 | - | - | 51.0 | - | ZA\|ZA\| -\|µF\|ZA |
| Q3888b | - | 3.00 | - | - | ZA\| -\| -\|µF |

| PUR (300-5000 µm) | (No. p/tablet) | (No. p/tablet) | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|
| Q3878 | - | 3.00 | - | 20136 | ZA\|ZA\| -\|µF |
| Q3888b | - | 1.00 | - | - | ZA\| -\| -\|µF |

| Polyurethane total | (No. p/tablet) | (No. p/tablet) | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|
| Q110 | - | 2.00 | - | - | ZA\|ZA\|ZA\|AF\|ZA |
| Q3878 | - | 23.0 | 244 | 143232 | ZA\|ZA\| -\|µF |
| Q3886 | - | - | 51.0 | - | ZA\|ZA\| -\|µF\|ZA |
| Q3888b | - | 4.00 | - | - | ZA\| -\| -\|µF |
| Q3889 | - | 8.00 | - | - | ZC\| -\|ZA\|µF |

# Total number of particles Data and Statistics

| Sample | QMP001SW | QMP002SW | QMP003MS | QMP004BT | MIC |
|---|---|---|---|---|---|
| **PVC (50-299 µm)** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q110 | - | 5.00 | - | - | ZA\|ZA\|ZA\|AF\|ZA |
| Q122 | - | 7.00 | - | - | ZA\| -\| -\|µF\|ZA |
| Q134 | - | 20.0 | - | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3231 | 1.00 | 2.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 20.0 | 4878 | 36093 | ZA\|ZA\| -\|µF |
| Q3887 | - | 14.0 | - | - | |
| Q3888b | - | 3.00 | - | - | ZA\| -\| -\|µF |
| Q3889 | - | 12.0 | - | - | ZC\| -\|ZA\|µF |
| Q3890 | - | 15.0 | - | - | ZA\| -\| -\|µF\|ZA |
| Q3911 | - | 1.00 | - | - | |
| Q3913 | - | 8.00 | - | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3917 | - | 10.0 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| | ========== | Statistical Results | ========== | | |
| NDA mean | - | 9.35 | - | - | |
| NDA st dev | - | 7.77 | - | - | |
| Coeff Var (%) | - | 83.1 | - | - | |
| N | 1 | 12 | 1 | 1 | |
| Median | 1.0 | 9.0 | 4878 | 36093 | |
| MAD | - | 5.5 | - | - | |
| Added number | - | 16 | - | - | |
| | ========== | | | | |

| PVC (300-5000 µm) | (No. p/tablet) | (No. p/tablet) | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|
| Q3231 | - | 4.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3888b | - | 4.00 | - | - | ZA\| -\| -\|µF |

| Polyvinyl Chloride total | (No. p/tablet) | (No. p/tablet) | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|
| Q110 | - | 5.00 | - | - | ZA\|ZA\|ZA\|AF\|ZA |
| Q122 | - | 7.00 | - | - | ZA\| -\| -\|µF\|ZA |
| Q134 | - | 20.0 | - | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3231 | 1.00 | 6.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | 20.0 | 4878 | 36093 | ZA\|ZA\| -\|µF |
| Q3882 | - | 6.00 | - | - | ZA\|ZA\| -\|µF\|ZA |
| Q3887 | - | 14.0 | - | - | |
| Q3888b | - | 7.00 | - | - | ZA\| -\| -\|µF |
| Q3889 | - | 12.0 | - | - | ZC\| -\|ZA\|µF |
| Q3890 | - | 15.0 | - | - | |
| Q3911 | - | 1.00 | - | - | |
| Q3913 | - | 8.00 | - | - | ZA\|ZA\| -\|ZA\|ZA |
| Q3917 | - | 10.0 | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| | ========== | Statistical Results | ========== | | |
| NDA mean | - | 8.65 | - | - | |
| NDA st dev | - | 4.76 | - | - | |
| Coeff Var (%) | - | 55.1 | - | - | |
| N | 1 | 13 | 1 | 1 | |
| Median | 1.0 | 8.0 | 4878 | 36092 | |
| MAD | - | 3.0 | - | - | |
| Added number | - | 16 | - | - | |
| | ========== | | | | |

# Total number of particles Data and Statistics

| Other polymers (50-299µm) | (No. p/tablet) | | (No. p/tablet) | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|---|
| Q3878 | - | | 12.0 | - | - | ZA\|ZA\| -\|µF |
| Q3886 | - | | 6.00 | 255 | 1305 | ZA\|ZA\| -\|µF\|ZA |
| Q3888b | 1.00 | | 3.00 | - | - | ZA\| -\| -\|µF |
| Q3889 | 3.00 | | 11.0 | - | 5607 | |
| Q3917 | - | | 2.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |

| Other polymers total | (No. p/tablet) | | (No. p/tablet) | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|---|
| Q3878 | - | | 12.0 | - | - | ZA\|ZA\| -\|µF |
| Q3882 | - | | 4.00 | - | - | ZA\|ZA\| -\|µF\|ZA |
| Q3886 | - | | 6.00 | 255 | 1305 | ZA\|ZA\| -\|µF\|ZA |
| Q3888b | 1.00 | | 3.00 | - | - | ZA\| -\| -\|µF |
| Q3889 | 3.00 | | 11.0 | - | 5607 | ZC\| -\|ZA\|µF |
| Q3917 | - | | 2.00 | - | - | ZA\|ZA\|ZA\|µF\|ZA |

| Total polymers (50-299 µm) | (No. p/tablet) | | (No. p/tablet) | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|---|
| Q101 | 2.00 | < | 75.0 | - | - | ZA\| -\|ZA\|ZA\|ZA |
| Q104 | 5.00 | | 30.0 | 5085308 | - | |
| Q110 | - | | 40.0 | 1611131 | 12621987 | |
| Q122 | - | | 46.0 | 2922131 | 8814815 | ZA\| -\| -\|µF\|ZA |
| Q134 | - | | 46.0 | 6513158 | - | |
| Q153 | 10.0 | | - | 54488 | 1050768 | ZC\|ZA\|ZA\|ZB\|ZA |
| Q3231 | 9.00 | | 24.0 | 936639 | 2174182 | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | - | | 170 | 79268 | 1006801 | |
| Q3886 | - | | 7.00 | 306 | 2050 | ZA\|ZA\| -\|µF\|ZA |
| Q3887 | - | | 57.0 | 7027582 | 8262053 | |
| Q3889 | 3.00 | | 31.0 | 777989 | 545110 | ZC\| -\|ZA\|µF |
| Q3890 | - | | 50.0 | 1415802 | 11770471 | |
| Q3911 | 2.00 | | 38.0 | - | - | |
| Q3913 | - | | 49.0 | 3564878 | - | |
| Q3917 | - | | 42.0 | - | - | |
| Q3936 | - | | - | 129845 | - | ZA\|ZA\|ZA\|ZA\|ZA |
| Q3943 | - | | 6.00 | - | 26406864 | |
| Q661 | 58.0 | | 150 | 306208 | 1240116 | ZA\|ZA\|ZA\|MI\|ZA |
| | ========== | | Statistical Results | | ========== | |
| NDA mean | - | | 39.86 | 1011523 | 2068174 | |
| NDA st dev | - | | 18.36 | 1614963 | 3539966 | |
| Coeff Var (%) | - | | 46.1 | 159.7 | 171.2 | |
| N | 6 | | 16 | 14 | 11 | |
| Median | 7.0 | | 44.00 | 1176221 | 2174182 | |
| MAD | 3.5 | | 13.00 | 1109343 | 2172132 | |
| Added number | - | | 76 | 10002270 | 25244344 | |

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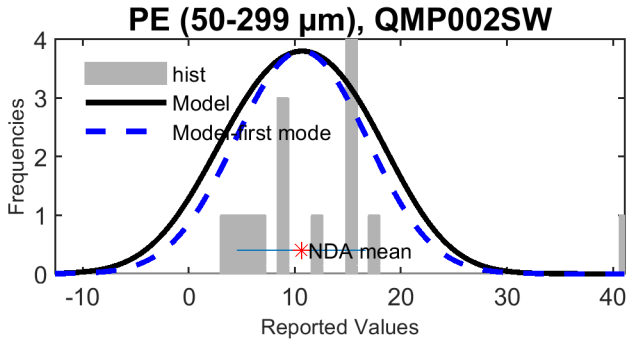
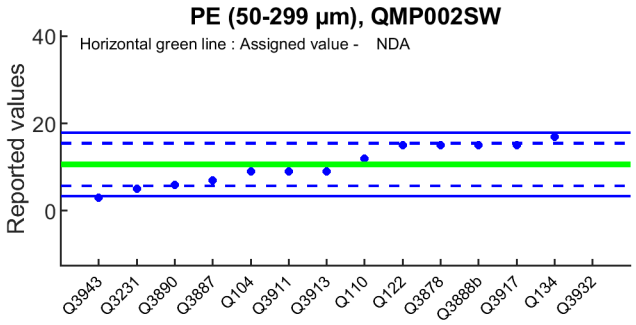
| Total polymers (300-5000 µm) | (No. p/tablet) | | (No. p/tablet) | | (No. p/kg) | (No. p/kg) | |
|---|---|---|---|---|---|---|---|
| Q101 | 2.00 | < | 2.00 | < | - | - | |
| Q104 | 2.00 | | - | | 50.0 | - | |
| Q122 | - | | 1.00 | | - | - | ZA\| -\| -\|µF\|ZA |
| Q153 | - | | - | | - | 1.00 | ZC\|ZA\|ZA\|ZB\|ZA |
| Q3231 | - | | 32.0 | | - | - | ZA\|ZA\|ZA\|µF\|ZA |
| Q3878 | 1.00 | | 22.0 | | - | 45588 | |
| Q3887 | 3.00 | | - | | - | - | |
| Q3911 | 2.00 | | - | | - | - | |
| Q3922 | - | | - | | 4722 | 1539 | |
| Q3936 | - | | - | | 3681 | - | ZA\|ZA\|ZA\|ZA\|ZA |
| Q661 | 222 | | 97.0 | | 4086 | 60174 | ZA\|ZA\|ZA\|MI\|ZA |

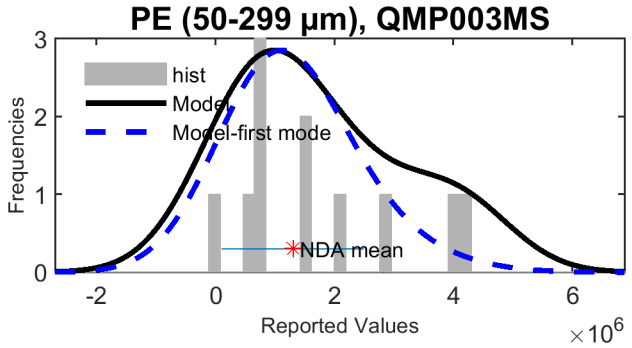
# Total number of particles Data and Statistics

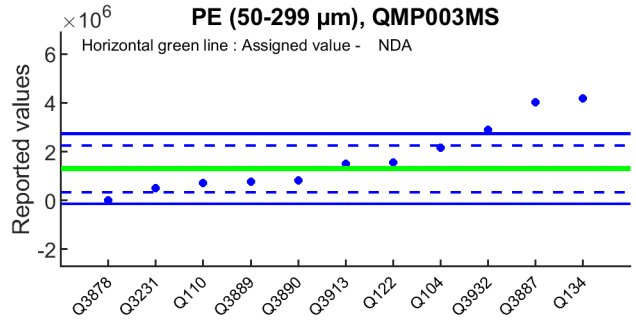
| Sample | QMP001SW | QMP002SW | QMP003MS | QMP004BT | MIC |
|---|---|---|---|---|---|
| **Total polymers** | **(No. p/tablet)** | **(No. p/tablet)** | **(No. p/kg)** | **(No. p/kg)** | |
| Q101 | 2.00 | < l 75.0 | - | - | ZA\| -\|ZA\|ZA\|ZA |
| Q104 | 7.00 | 30.0 | 5085358 | - | |
| Q110 | - | 40.0 | 1611131 | 12621987 | |
| Q122 | - | 47.0 | 2922131 | 8814815 | ZA\| -\| -\|µF\|ZA |
| Q134 | - | 46.0 | 6513158 | - | |
| Q153 | 10.0 | - | 54488 | 1050769 | ZC\|ZA\|ZA\|ZB\|ZA |
| Q3160 | 15694 | 6016 | 5611434 | 5382 | |
| Q3231 | 9.00 | 56.0 | 936639 | 2174182 | ZA\|ZA\|ZA\|µF\|ZA |
| Q3877b | 393 | 137 | 3073149 | 2097999 | ZB\|ZB\|ZB\|MI\|ZB |
| Q3878 | 1.00 | 192 | 79268 | 1052391 | |
| Q3882 | - | 17.0 | - | - | ZA\|ZA\| -\|µF\|ZA |
| Q3886 | - | 7.00 | 306 | 2050 | ZA\|ZA\| -\|µF\|ZA |
| Q3887 | 3.00 | 57.0 | 7027582 | 8262053 | |
| Q3888b | 22.0 | 46.0 | - | - | |
| Q3889 | 3.00 | 31.0 | 777989 | 545110 | ZC\| -\|ZA\|µF |
| Q3890 | - | 50.0 | 1415802 | 11770471 | |
| Q3911 | 4.00 | 38.0 | - | - | |
| Q3913 | - | 49.0 | 3564878 | - | |
| Q3917 | - | 42.0 | - | - | |
| Q3922 | - | - | 4722 | 1539 | |
| Q3926 | - | 49.0 | 156882 | 305217 | ZA\|ZA\|ZA\|µF |
| Q3932 | - | 1634 | 2903587 | - | |
| Q3936 | 7.00 | 19.0 | 133526 | - | ZA\|ZA\|ZA\|ZA\|ZA |
| Q3940 | 277 | 255 | 20054 | - | |
| Q3943 | - | 6.00 | - | 26406864 | |
| Q661 | 280 | 247 | 310296 | 1300289 | ZA\|ZA\|ZA\|MI\|ZA |
| Q871 | 579 | 2224 | 647682387 | 11243787 | |
| | ========== | Statistical Results | ========== | | |
| NDA mean | 6.38 | 40.2 | 1344449 | 941297 | |
| NDA st dev | 9.31 | 25.0 | 2086266 | 2173988 | |
| Coeff Var (%) | 145.7 | 62.2 | 155.2 | 231.0 | |
| N | 14 | 25 | 21 | 16 | |
| Median | 9.5 | 49.0 | 1415802 | 1699144 | |
| MAD | 7.5 | 19.0 | 1411080 | 1695428 | |
| Added number | - | 76 | 10002270 | 25244344 | |
| | ========== | | | | |

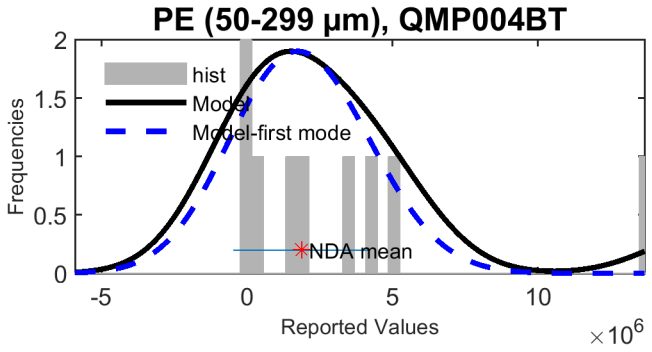
# Total number of particles Histogram+PDFs and Ranked overview

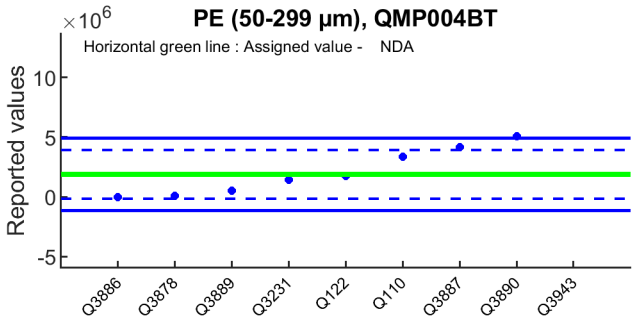


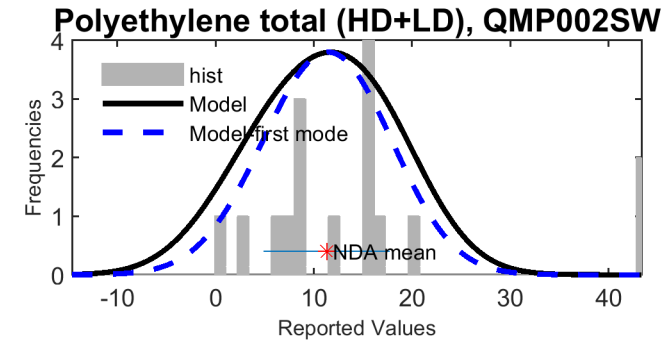



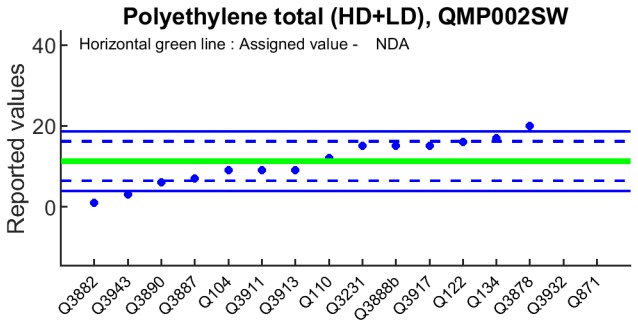



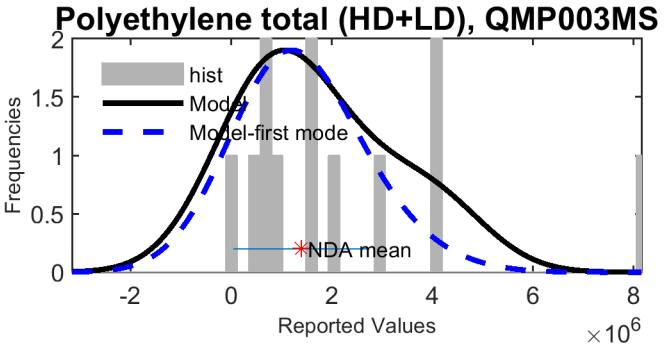



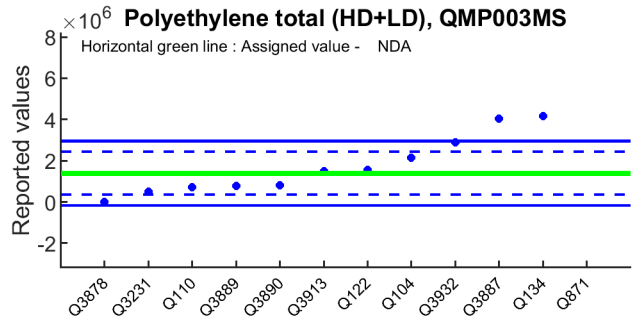



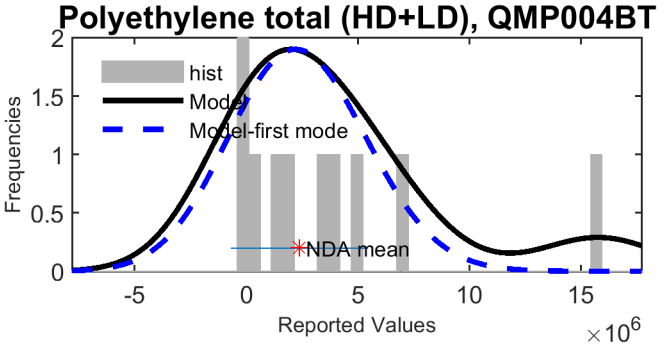
# Total number of particles Histogram+PDFs and Ranked overview

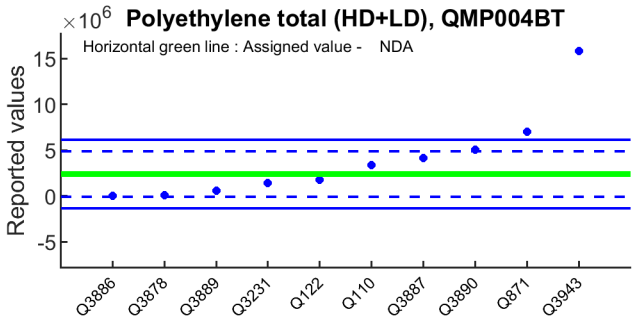


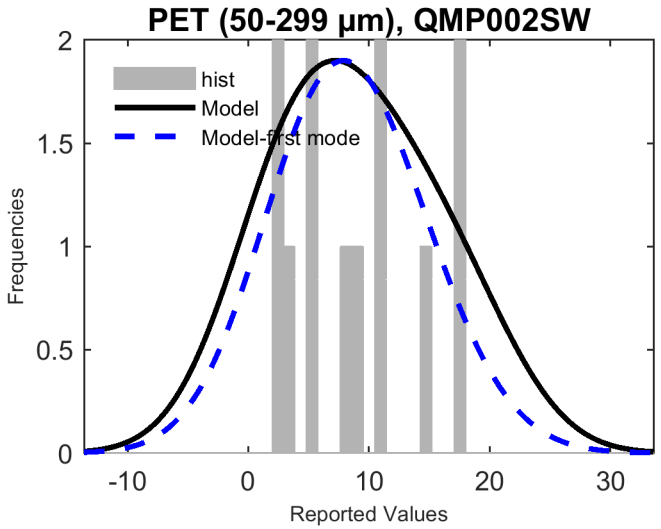



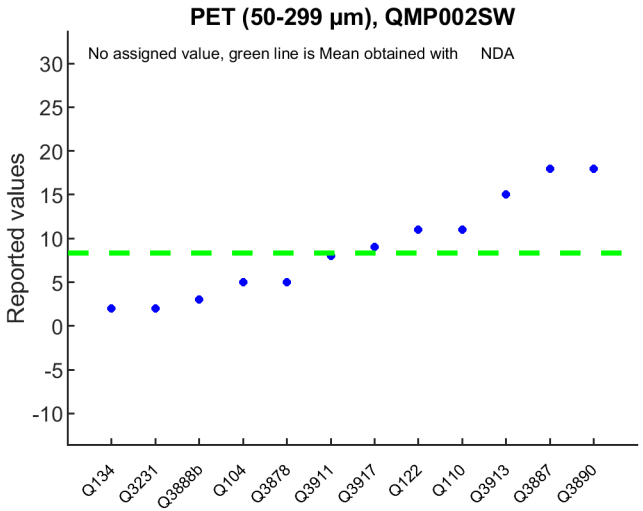



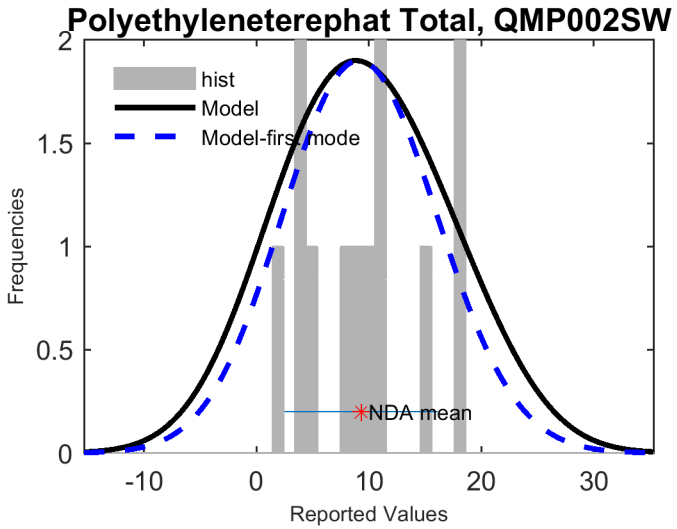



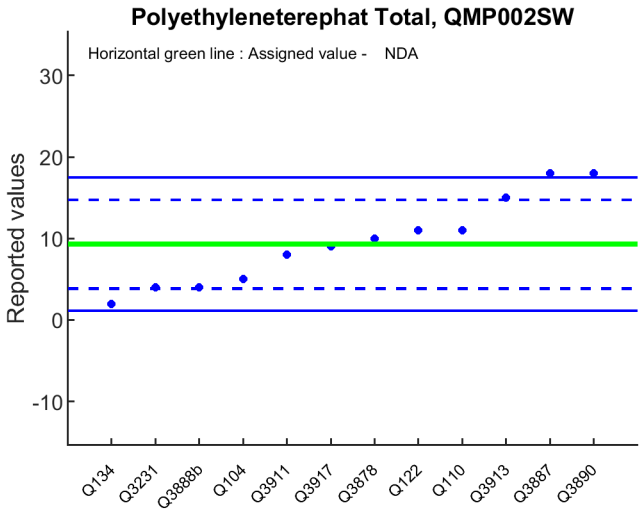
# Total number of particles Histogram+PDFs and Ranked overview



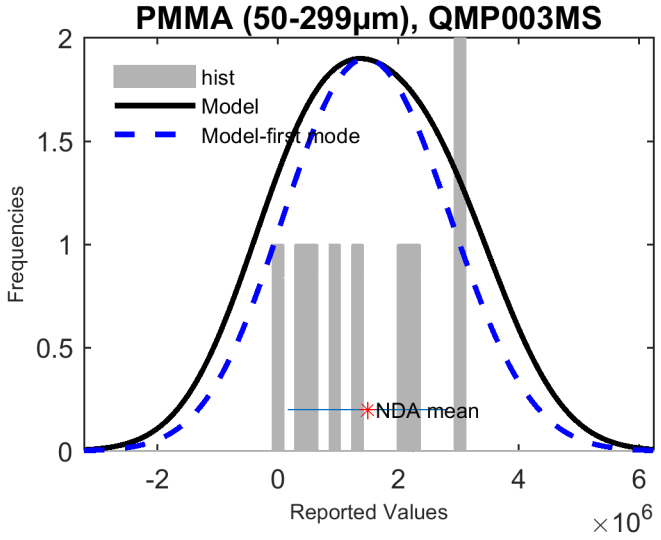



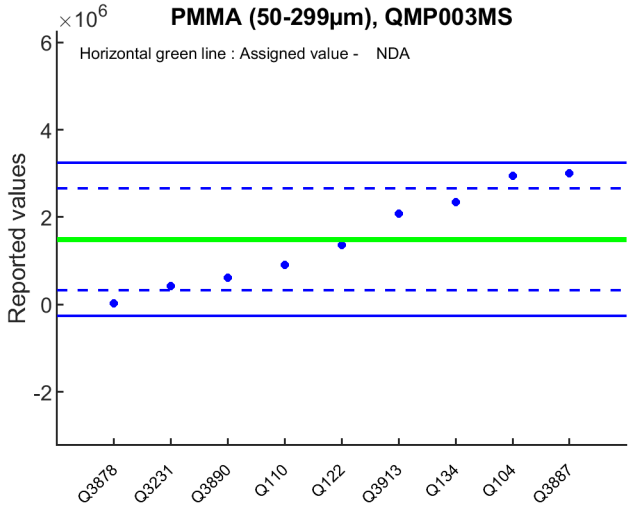
# Total number of particles Histogram+PDFs and Ranked overview

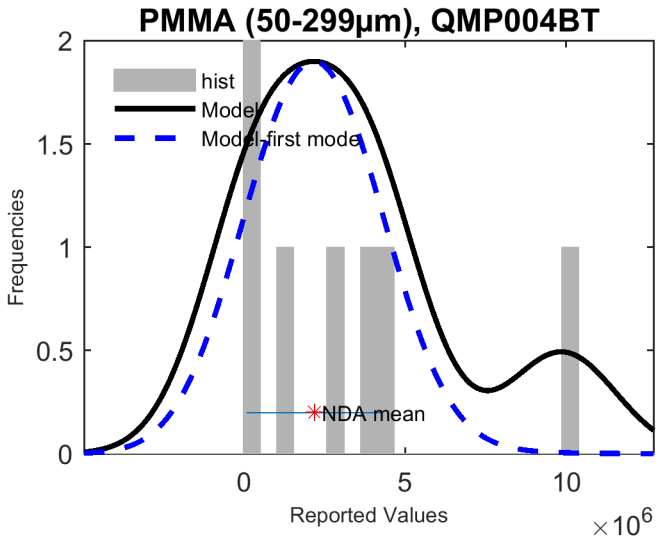


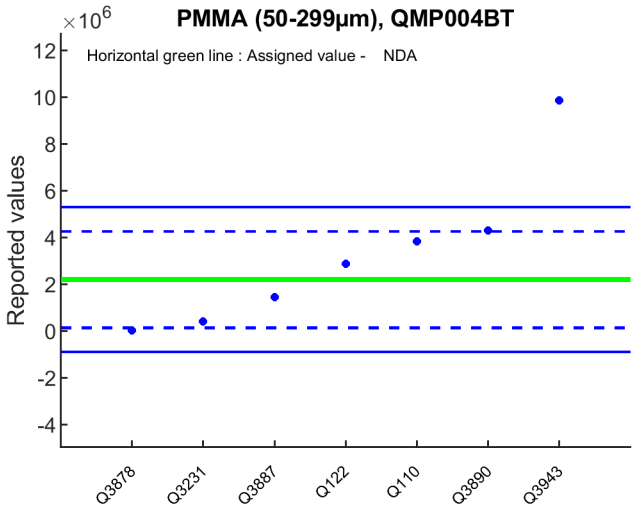



# Total number of particles Histogram+PDFs and Ranked overview

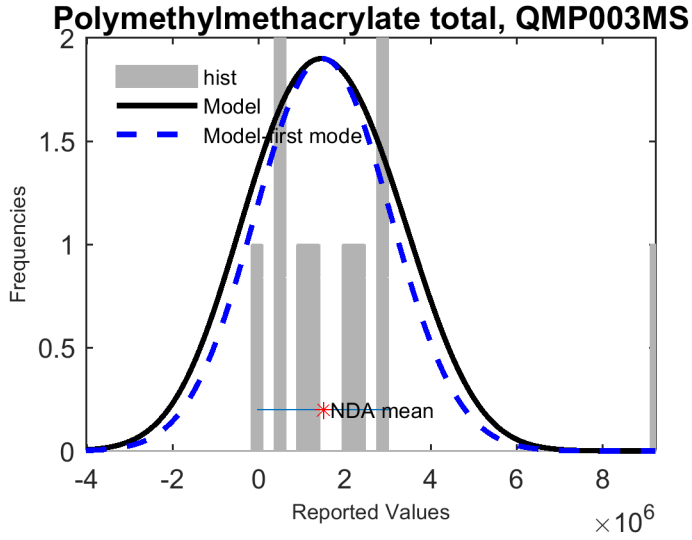


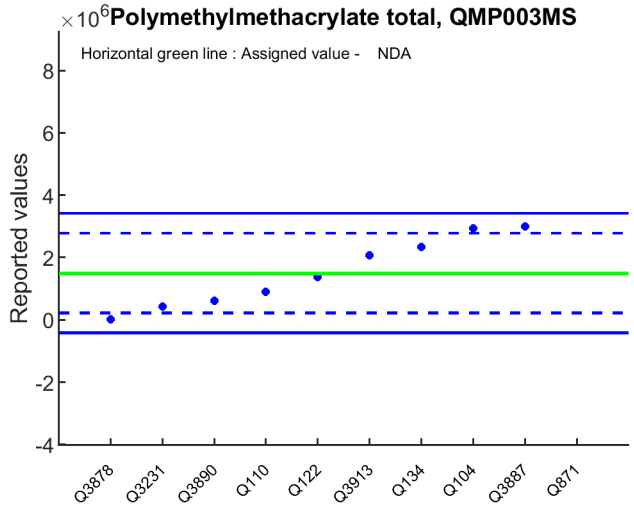



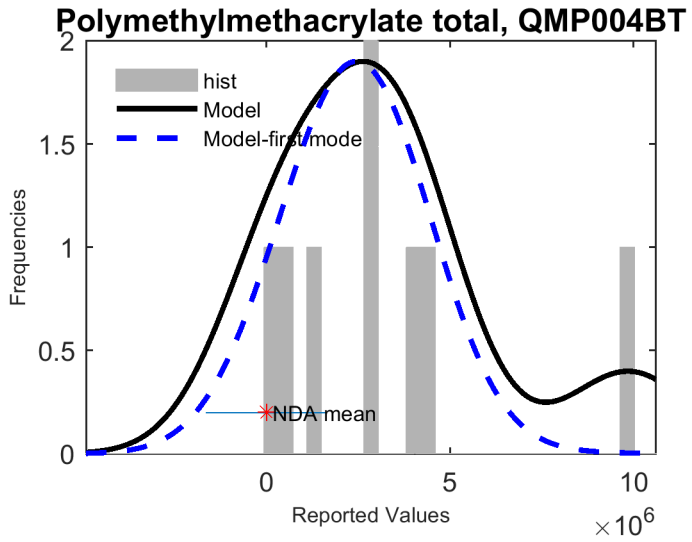



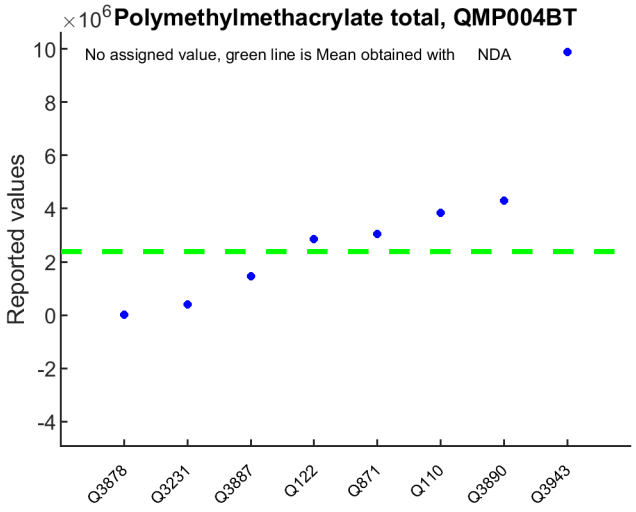



# Total number of particles Histogram+PDFs and Ranked overview

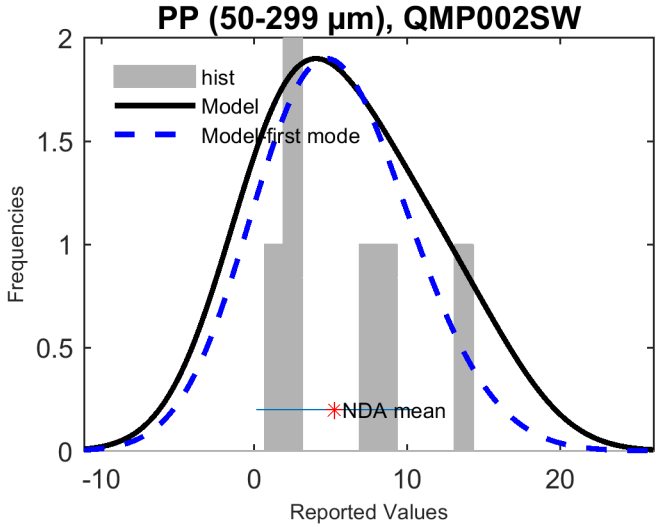


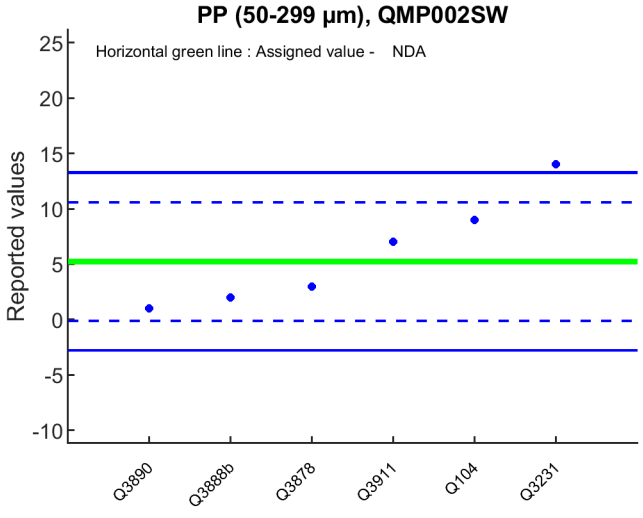



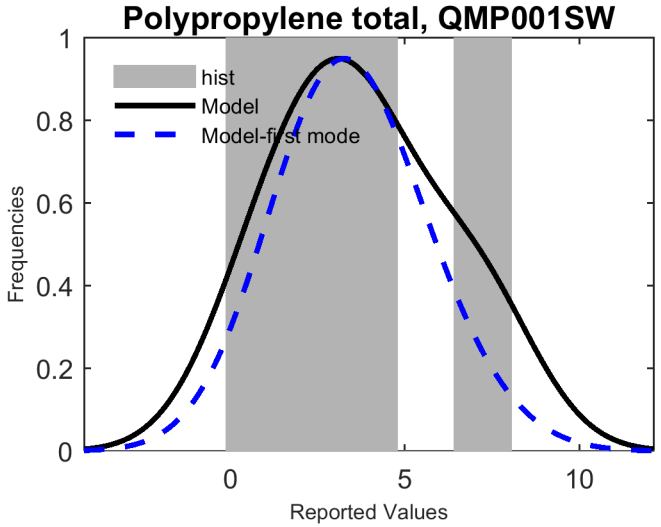



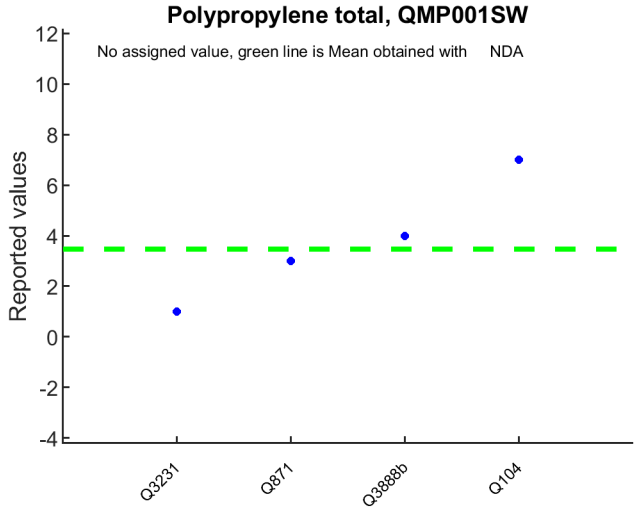
# Total number of particles Histogram+PDFs and Ranked overview

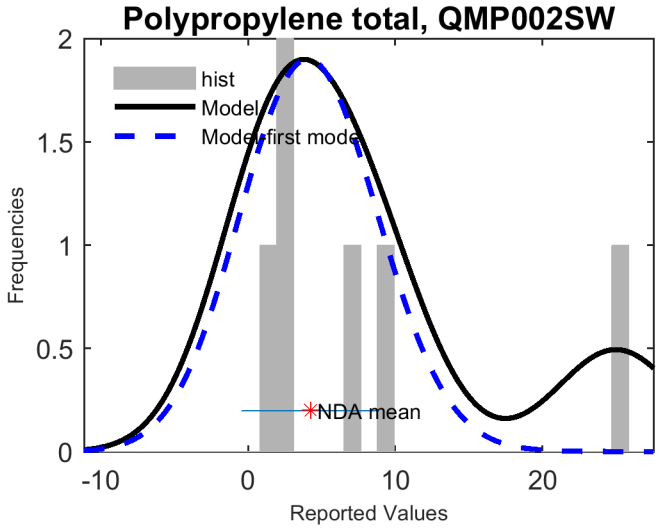


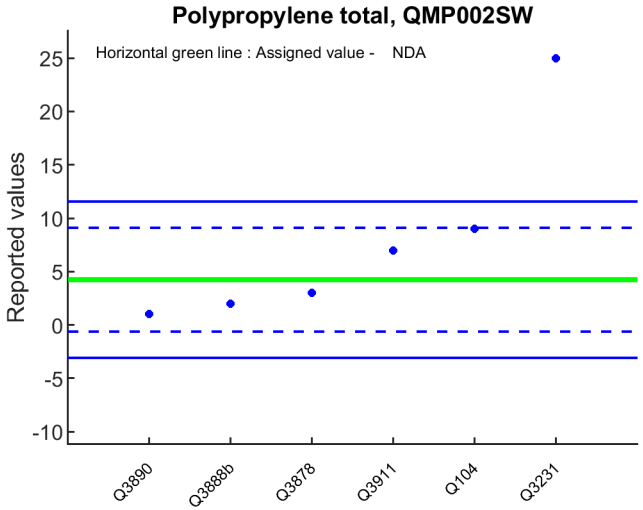



# Total number of particles Histogram+PDFs and Ranked overview

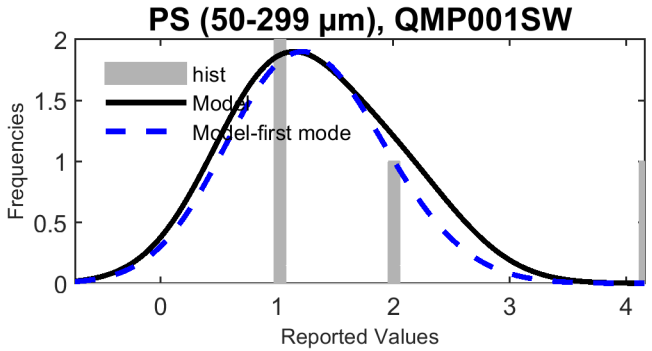


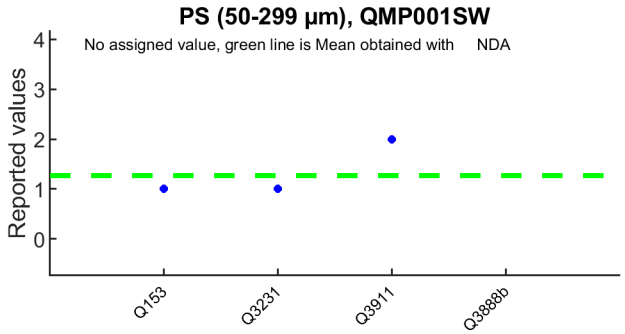



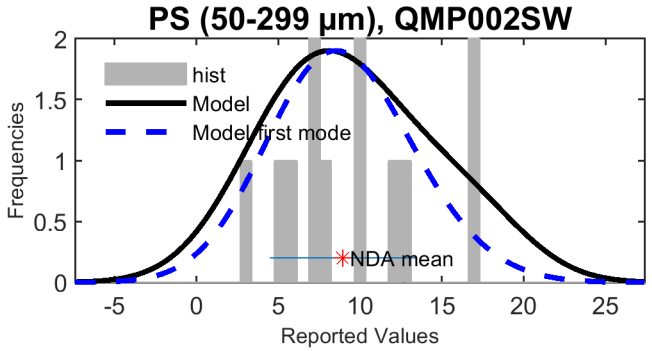



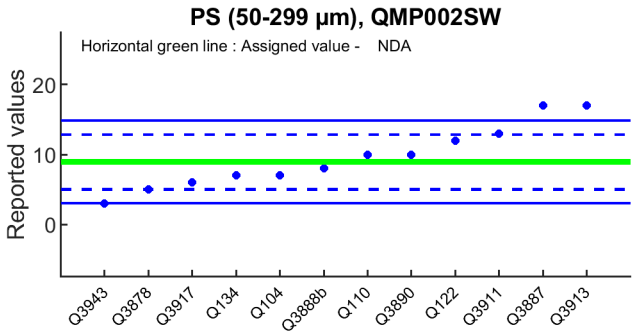



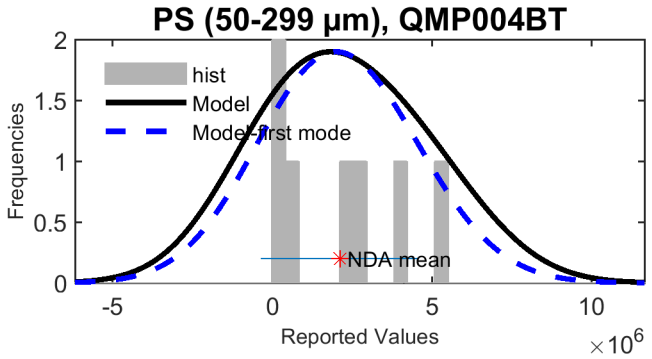
# Total number of particles Histogram+PDFs and Ranked overview

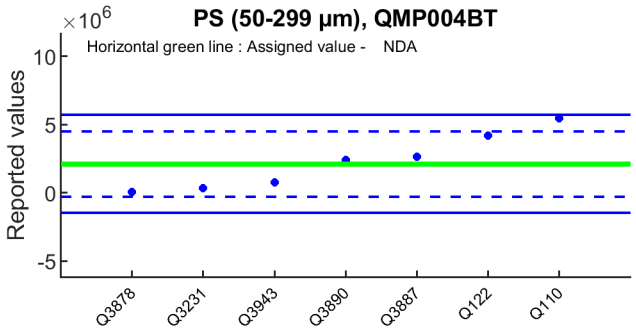


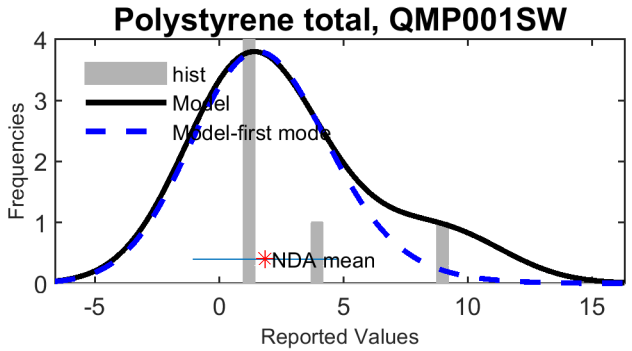



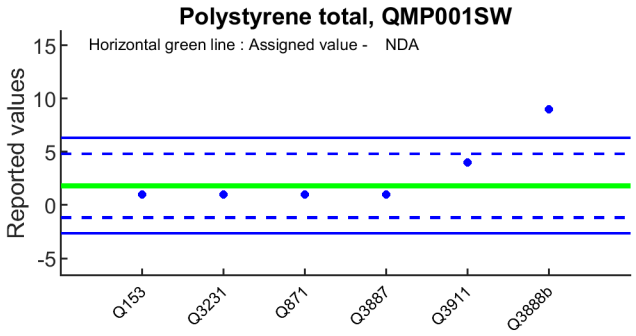



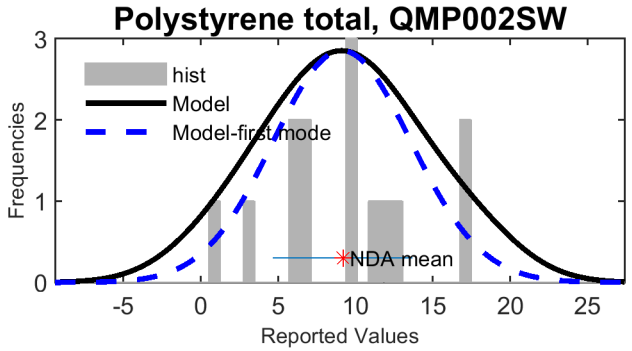



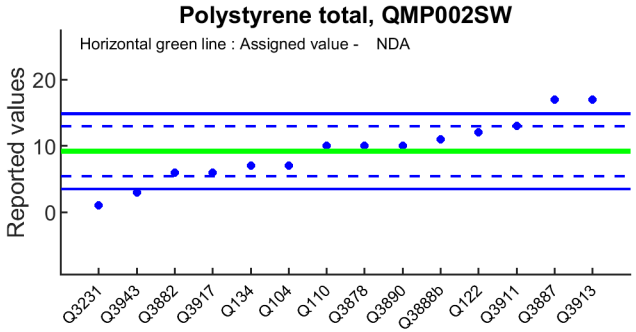



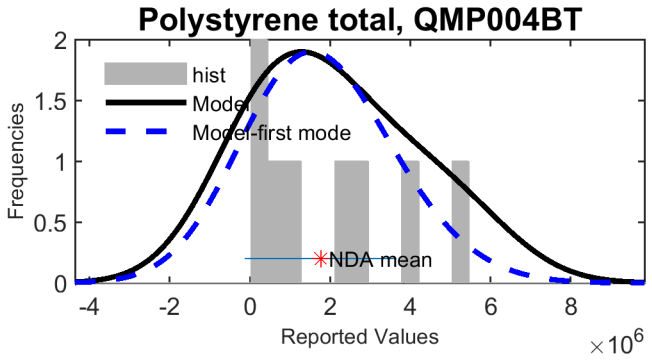
# Total number of particles Histogram+PDFs and Ranked overview

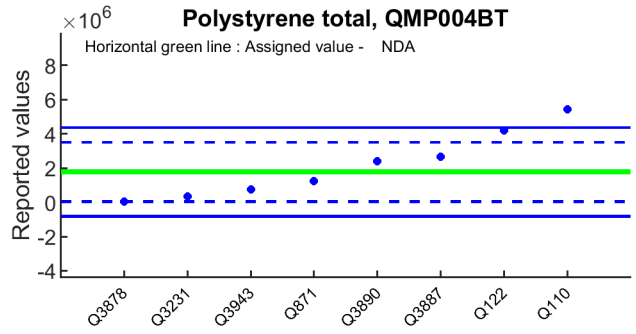


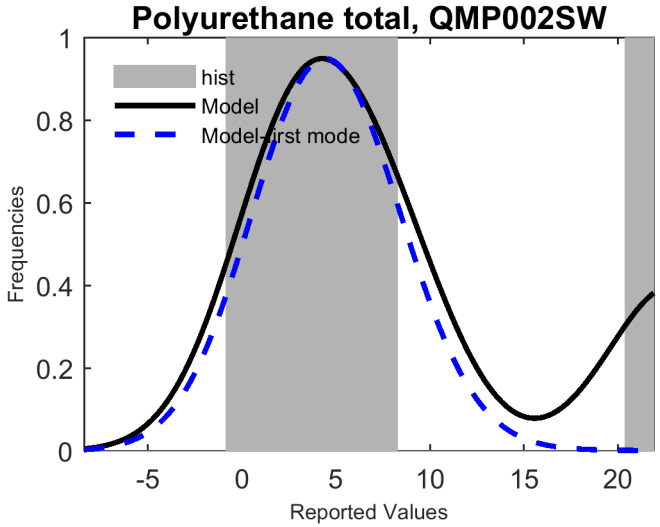



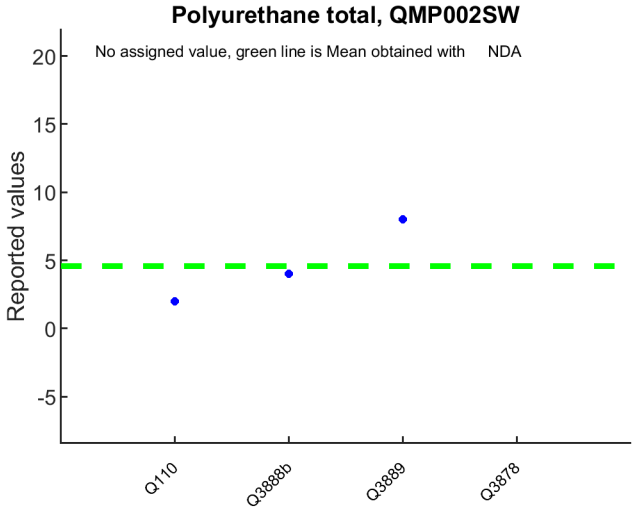



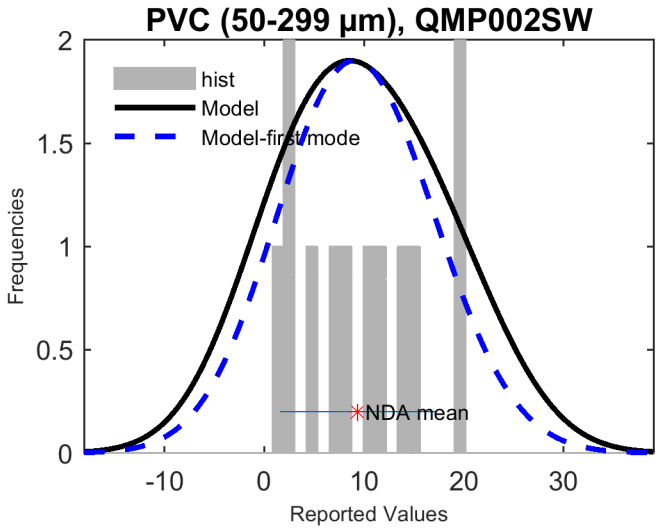



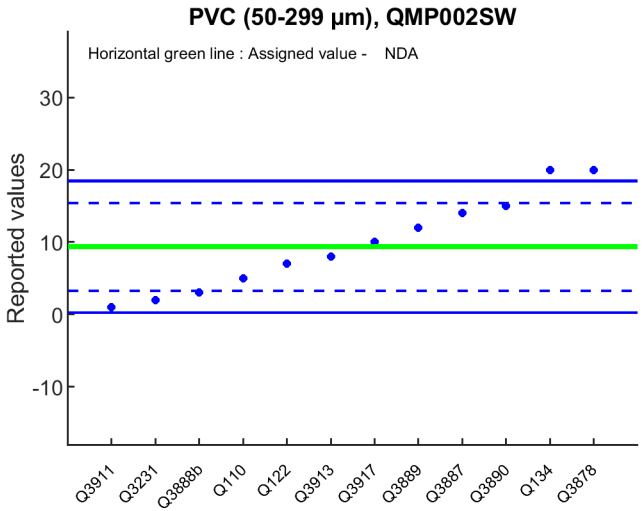
# Total number of particles Histogram+PDFs and Ranked overview



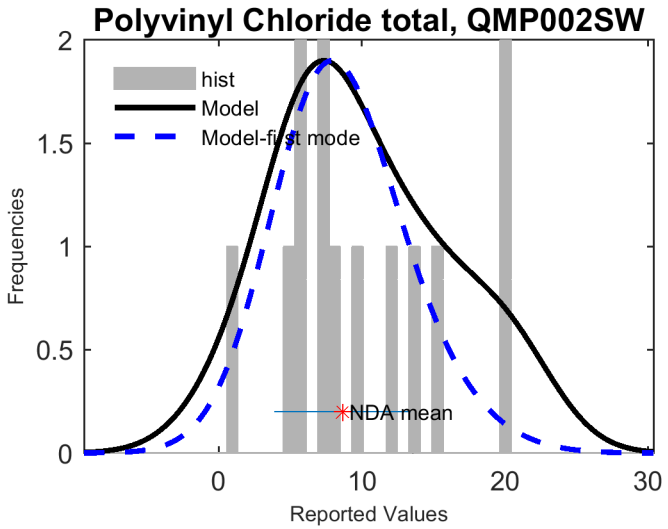



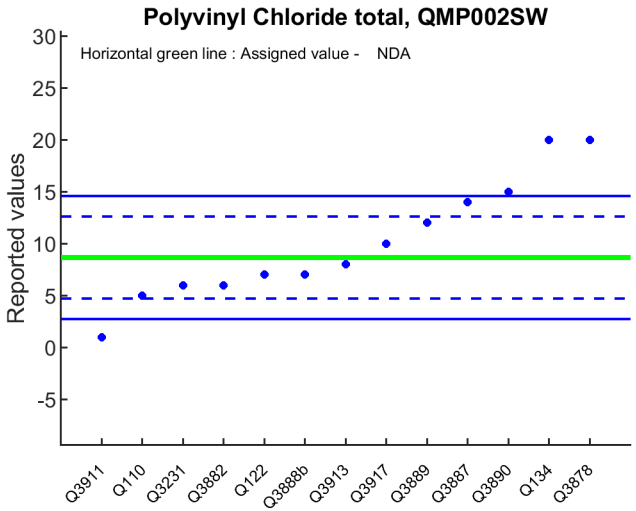
# Total number of particles Histogram+PDFs and Ranked overview



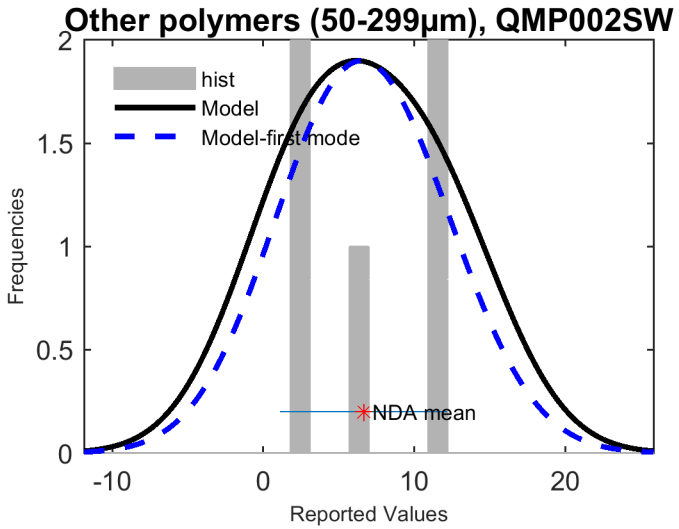



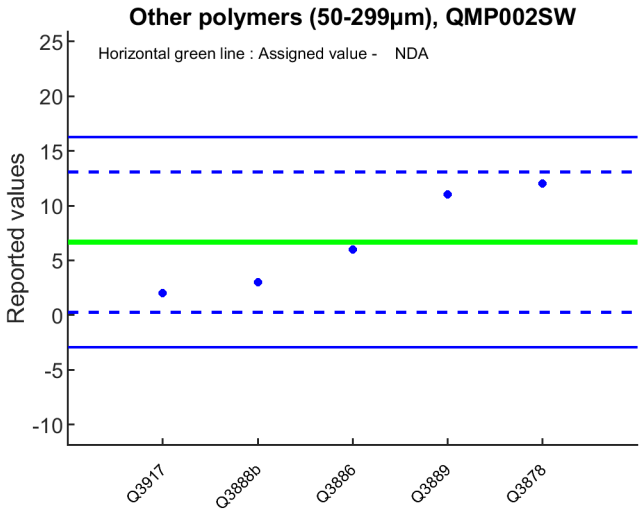
# Total number of particles Histogram+PDFs and Ranked overview



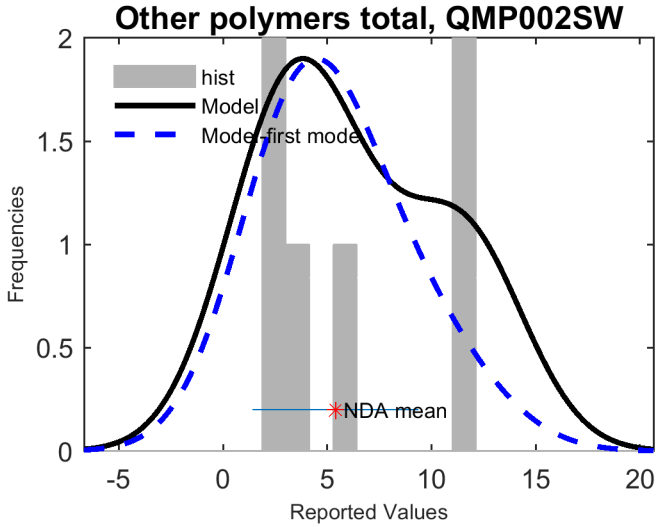



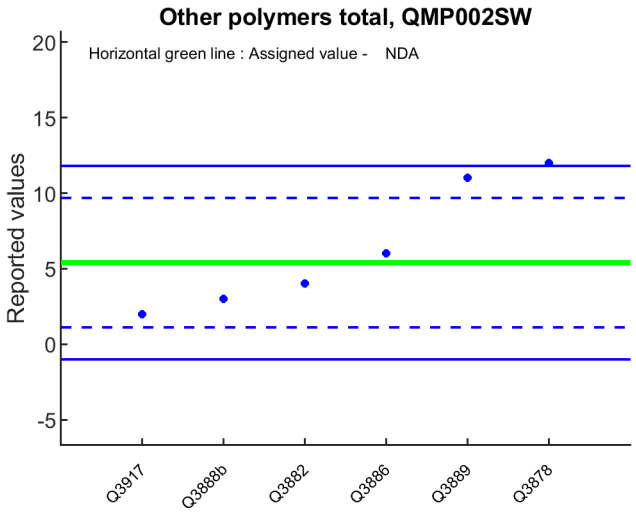
# Total number of particles Histogram+PDFs and Ranked overview



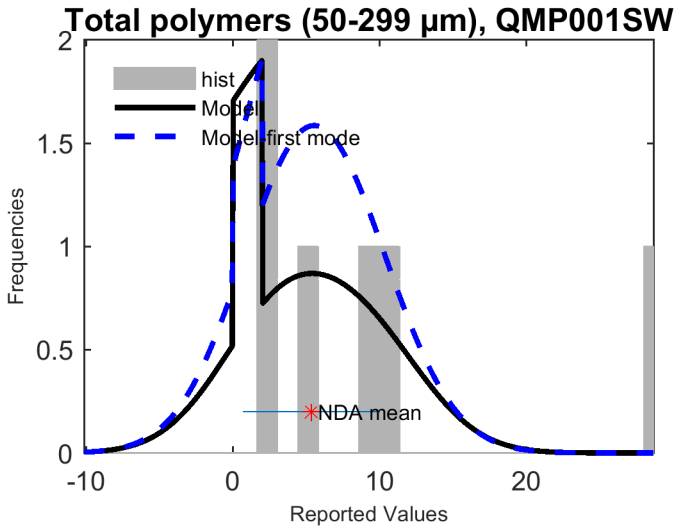



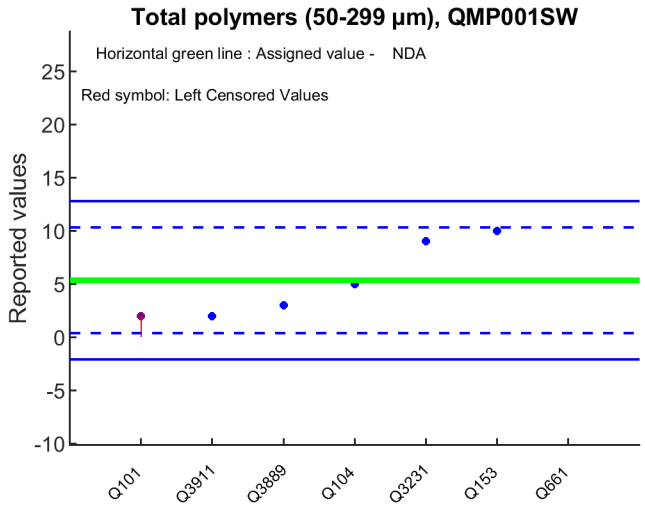
# Total number of particles Histogram+PDFs and Ranked overview

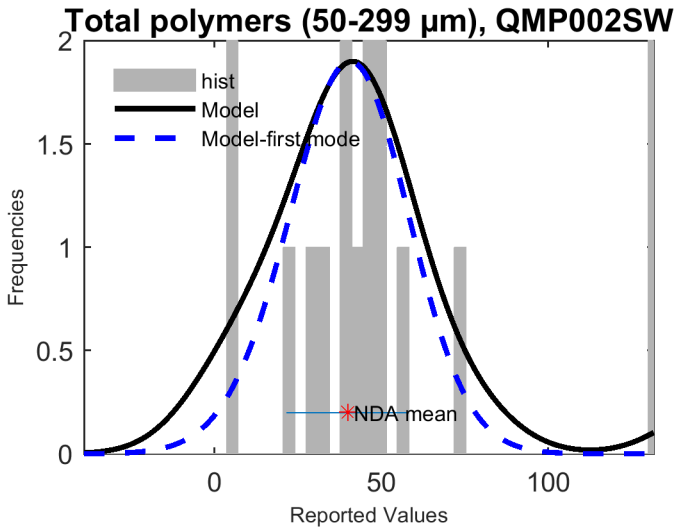


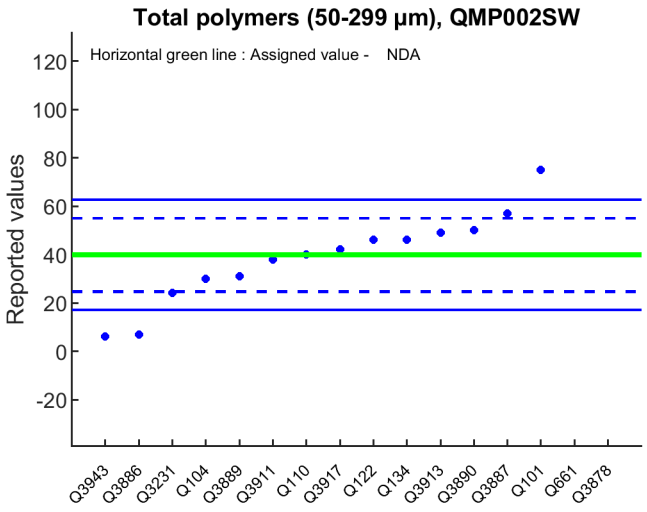



# Total number of particles Histogram+PDFs and Ranked overview

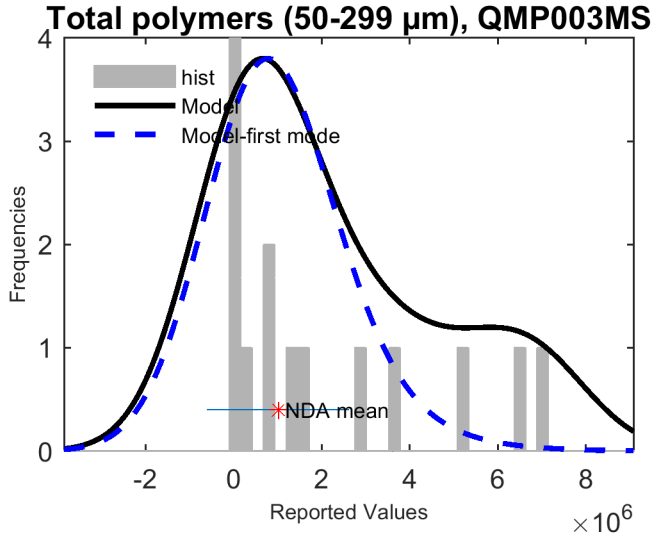


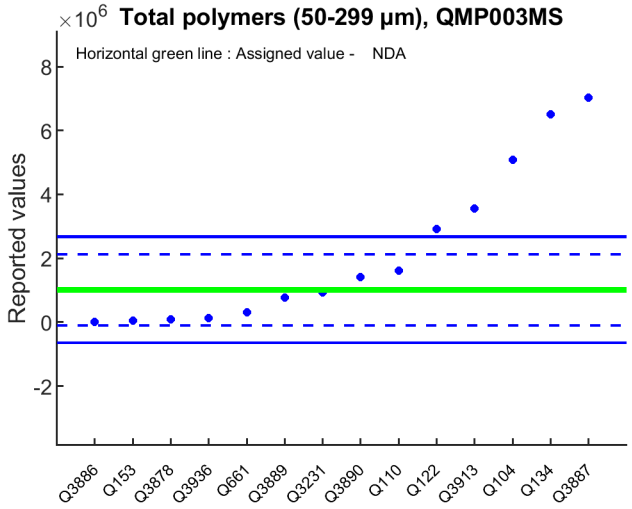



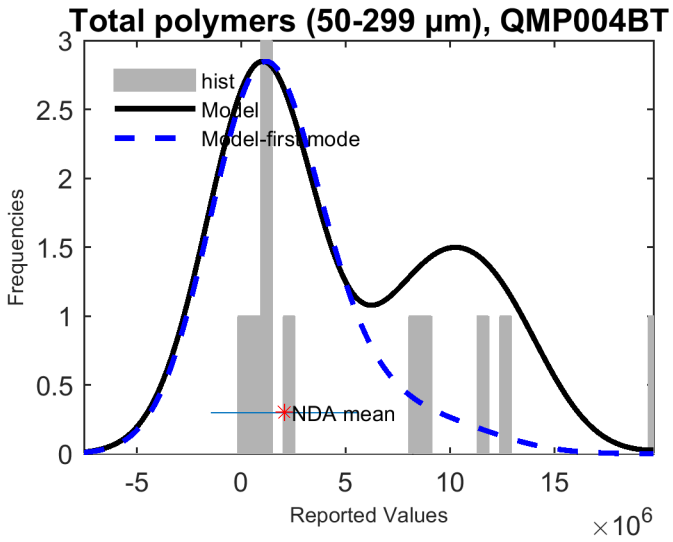



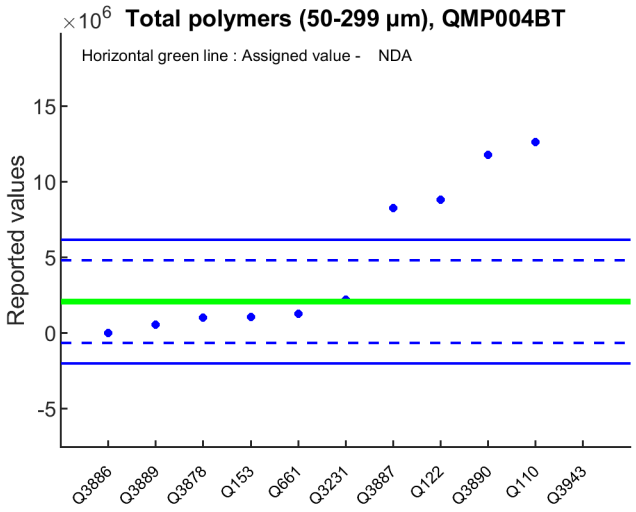



# Total number of particles Histogram+PDFs and Ranked overview

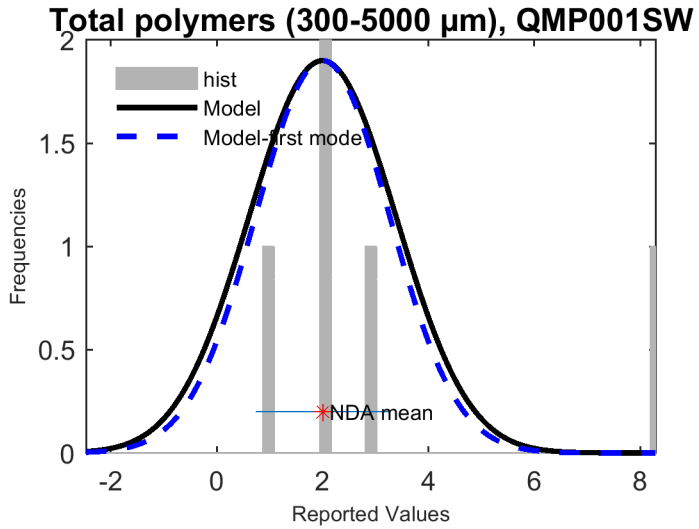


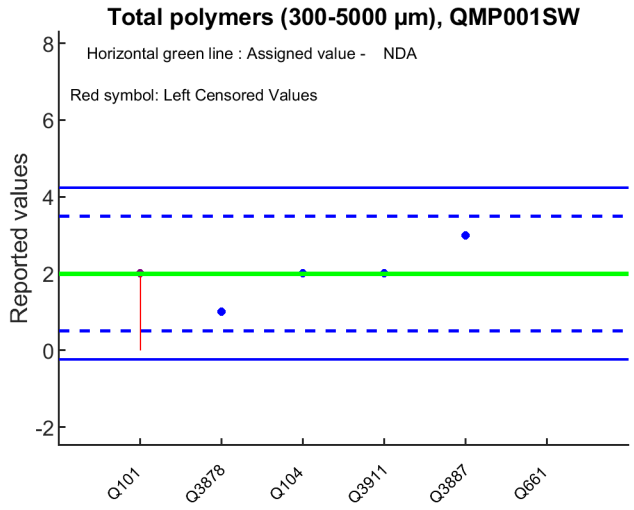



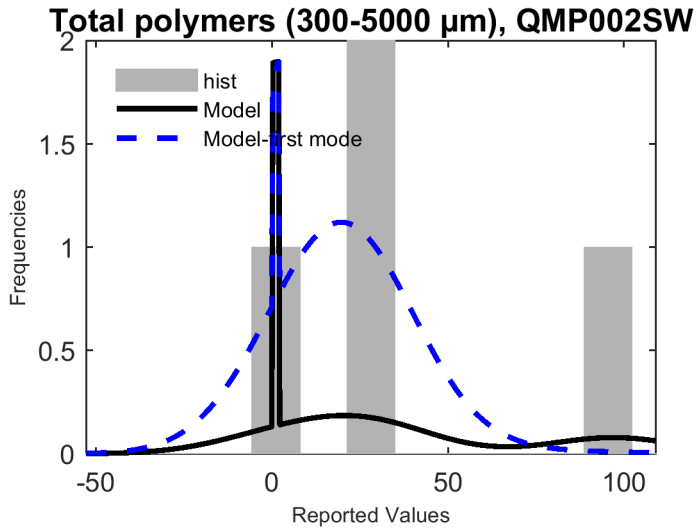



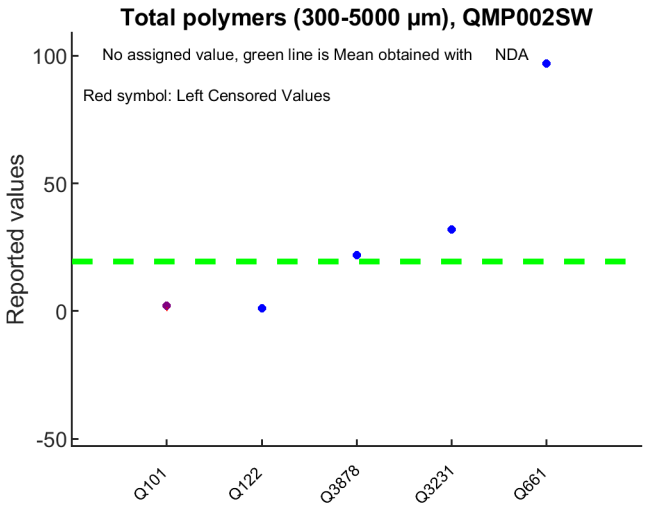



# Total number of particles Histogram+PDFs and Ranked overview



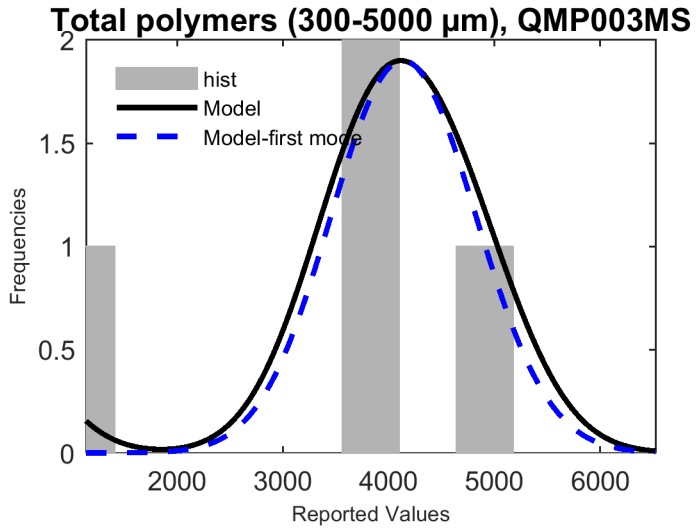



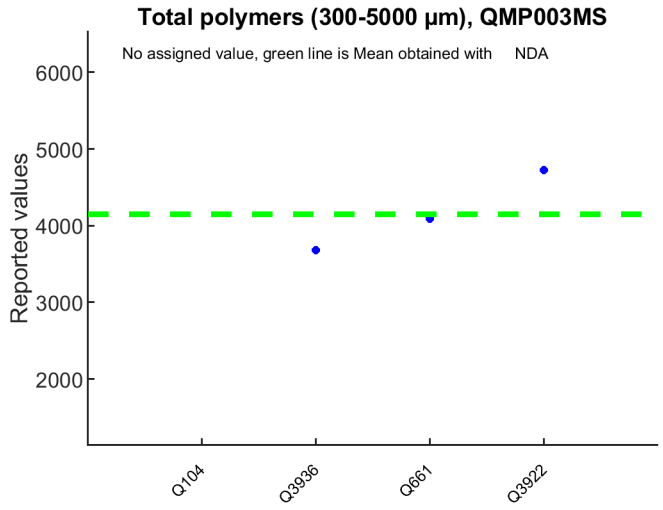



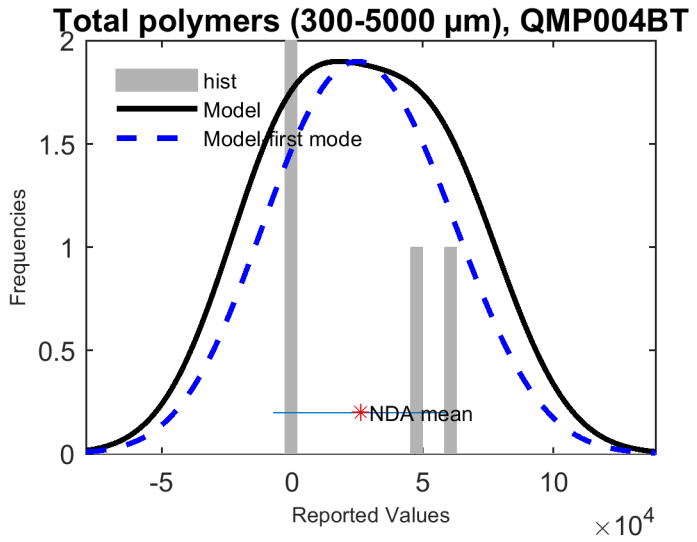



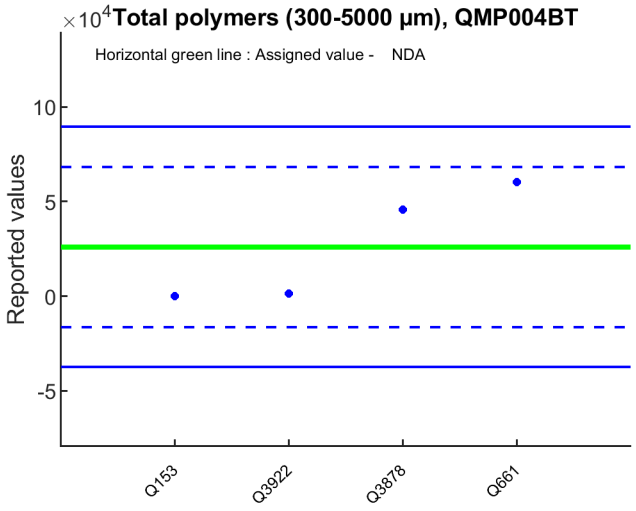
# Total number of particles Histogram+PDFs and Ranked overview

NDA mean

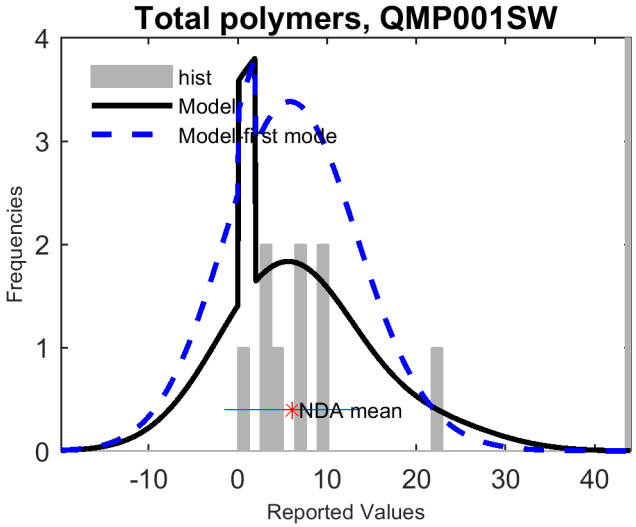


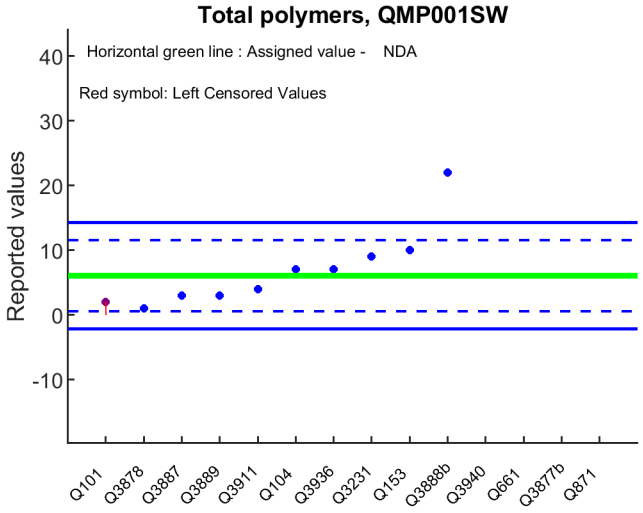



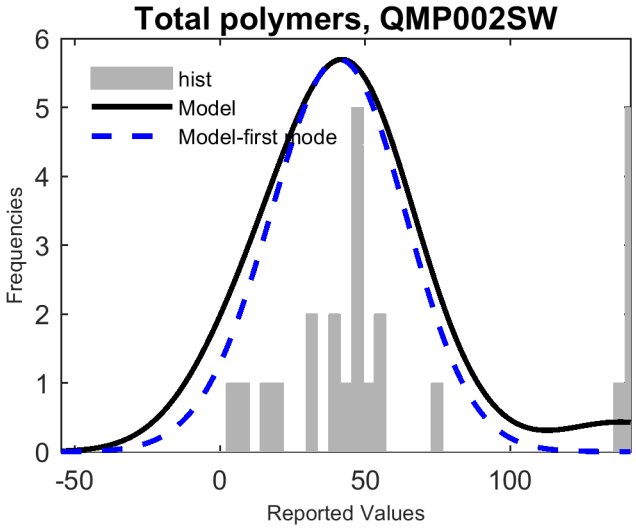



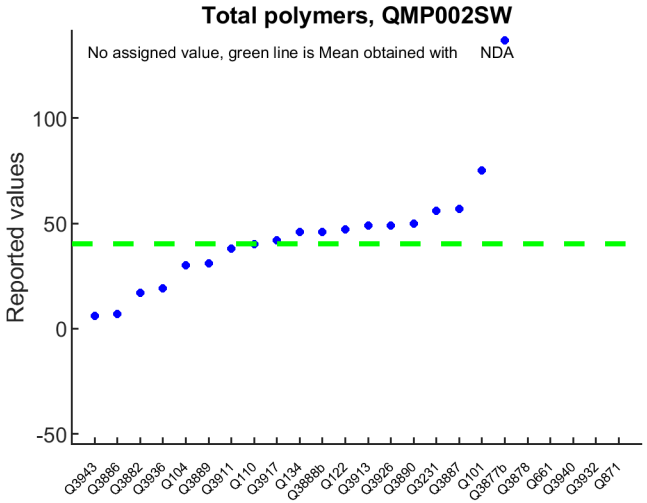



# Total number of particles Histogram+PDFs and Ranked overview

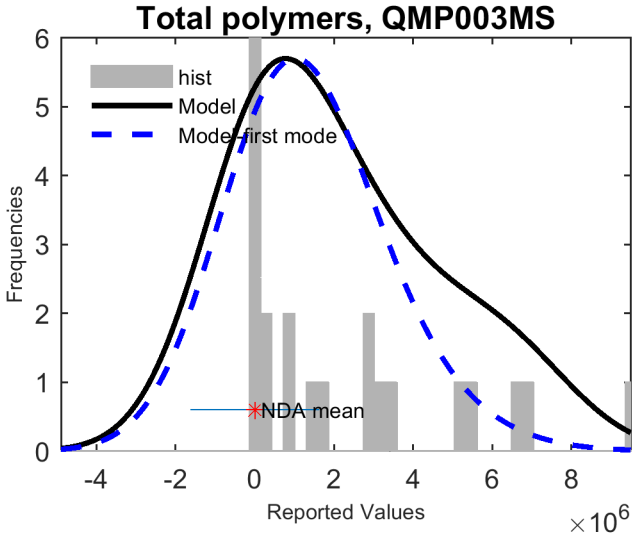


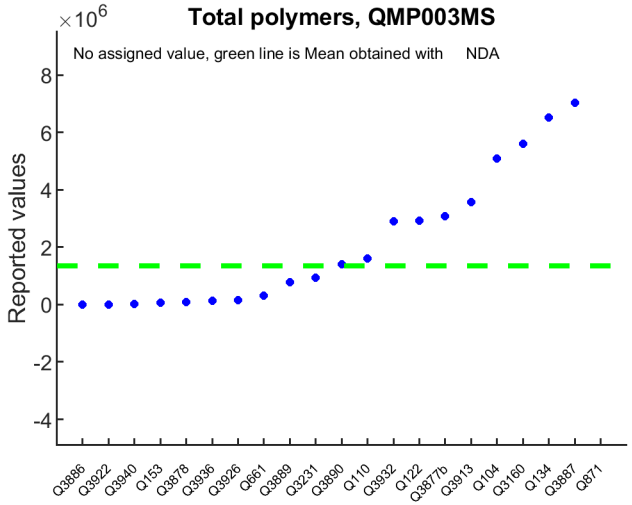



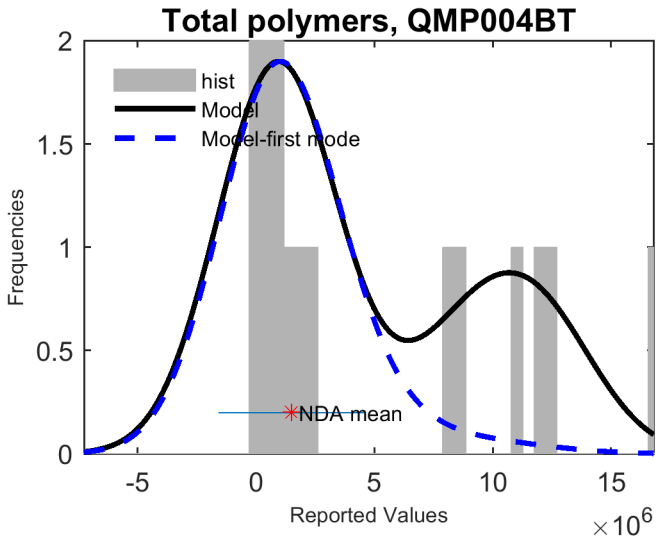



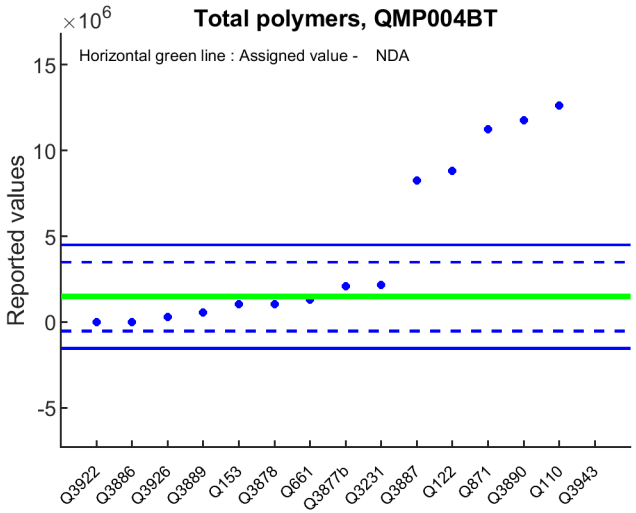



# Total number of particles Histogram+PDFs and Ranked overview



# Total mass of plastic particles Analysis DE17  2020.1

# 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 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **QMP001SW** | | | | | | | | | | | |
| Total Polymers | 0.067 | (mg/kg) | | 0.137 | 203.5 | 8 | 1 | 0.105 | 0.096 | 0.0673 | 0.0605 |

| Sample/ Determinand | Assigned Value | Units | Total Error | NDA st.dev | NDA rel. st.dev (%) | Nobs numerical | Nobs LCV | Median | MAD | Model Mean | Uncer-tainty |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **QMP002SW** | | | | | | | | | | | |
| Total Polymers | 0.045 | (mg/kg) | | 0.023 | 50.3 | 9 | 1 | 0.052 | 0.019 | 0.0450 | 0.0094 |

| Sample/ Determinand | Assigned Value | Units | Total Error | NDA st.dev | NDA rel. st.dev (%) | Nobs numerical | Nobs LCV | Median | MAD | Model Mean | Uncer-tainty |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **QMP003MS** | | | | | | | | | | | |
| Polyethylene total (HD+LD) | 755 | (mg/kg) | | 655 | 86.8 | 7 | 0 | 846 | 464 | 755 | 310 |
| Total Polymers | 1333 | (mg/kg) | | 1245 | 93.4 | 11 | 0 | 1370 | 876 | 1333 | 469 |

| Sample/ Determinand | Assigned Value | Units | Total Error | NDA st.dev | NDA rel. st.dev (%) | Nobs numerical | Nobs LCV | Median | MAD | Model Mean | Uncer-Tainty |
|---|---|---|---|---|---|---|---|---|---|---|---|
| **QMP004BT** | | | | | | | | | | | |
| Total Polymers | 6361 | (mg/kg) | | 7610 | 119.6 | 10 | 0 | 7436 | 5453 | 6361 | 3008 |

# Total mass of plastic particles Data and Statistics

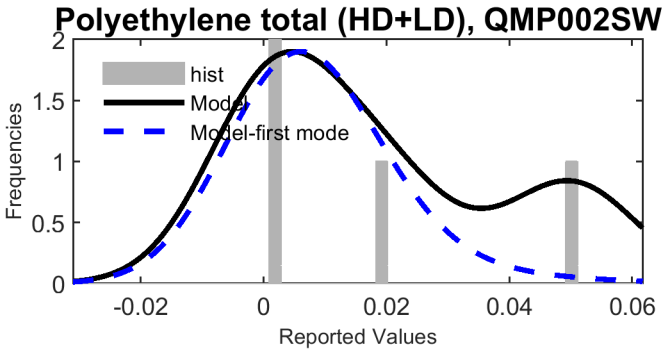
| Sample | QMP001SW | | QMP002SW | | QMP003MS | | QMP004BT | | MIC |
|---|---|---|---|---|---|---|---|---|---|
| **Polyamide total** | **(mg/tablet)** | | **(mg/tablet)** | | **(mg/kg)** | | **(mg/kg)** | | |
| Q3175 | 0.080 | | 0.080 | | 20.5 | | - | | QM\|ZN\|HO\|TD\|ZB |
| **Polycarbonate total** | **(mg/tablet)** | | **(mg/tablet)** | | **(mg/kg)** | | **(mg/kg)** | | |
| Q980 | 0.007 | < | 0.007 | < | 1.00 | < | 1.00 | < | -\| -\|ZC\|PY\|ZA |
| **PE (50-299 µm)** | **(mg/tablet)** | | **(mg/tablet)** | | **(mg/kg)** | | **(mg/kg)** | | |
| Q3932 | - | | - | | 846 | | 33050 | | ZA\|ZA\|ZA\|AF\|ZA |
| **Polyethylene total (HD+LD)** | **(mg/tablet)** | | **(mg/tablet)** | | **(mg/kg)** | | **(mg/kg)** | | |
| H221 | - | | 0.001 | | 522 | | 1093 | | -\| -\|ZA\|PY\|ZA |
| Q3175 | 0.060 | | 0.050 | | 152 | | - | | QM\|ZN\|HO\|TD\|ZB |
| Q3189 | - | | 0.002 | | 300 | | 307 | | ZB\|ZB\|ZB\|PY\|ZB |
| Q3877b | - | | - | | 936 | | 1356 | | ZA\|ZA\|ZA\|PY\|ZA |
| Q3932 | - | | - | | 846 | | 33050 | | |
| Q871 | - | | 0.020 | | 2133 | | 325 | | |
| Q980 | 0.160 | < | 0.160 | < | 1310 | | 4030 | | -\| -\|ZC\|PY\|ZA |
| | ========== | | Statistical Results | | ========== | | | | |
| NDA mean | - | | - | | 754.9 | | - | | |
| NDA st dev | - | | - | | 655.4 | | - | | |
| Coeff Var (%) | - | | - | | 86.8 | | - | | |
| N | 1 | | 4 | | 7 | | 6 | | |
| Median | 0.0600 | | 0.0110 | | 846.0 | | 1224.5 | | |
| MAD | - | | 0.0093 | | 464.0 | | 908.5 | | |
| Added mass | - | | 0.0217 | | 900 | | 1129 | | |
| | ========== | | | | | | | | |
| **Polyethyleneterephat. Total** | **(mg/tablet)** | | **(mg/tablet)** | | **(mg/kg)** | | **(mg/kg)** | | |
| H221 | - | | 0.001 | | - | | - | | -\| -\|ZA\|PY\|ZA |
| Q3175 | 0.380 | | 0.440 | | 71.9 | | - | | QM\|ZN\|HO\|TD\|ZB |
| Q3189 | - | | 0.009 | | 1.56 | | - | | ZB\|ZB\|ZB\|PY\|ZB |
| Q980 | 0.020 | < | 0.020 | < | 5.00 | < | 170 | | -\| -\|ZC\|PY\|ZA |
| **Polymethylmethacrylate total** | **(mg/tablet)** | | **(mg/tablet)** | | **(mg/kg)** | | **(mg/kg)** | | |
| H221 | 0.000 | | 0.000 | | 1140 | | 1176 | | -\| -\|ZA\|PY\|ZA |
| Q3189 | - | | - | | 185 | | 286 | | ZB\|ZB\|ZB\|PY\|ZB |
| Q980 | 0.005 | < | 0.005 | < | 1330 | | 1170 | | -\| -\|ZC\|PY\|ZA |
| Added mass | - | | - | | 2052 | | 1406 | | |
| **Polypropylene total** | **(mg/tablet)** | | **(mg/tablet)** | | **(mg/kg)** | | **(mg/kg)** | | |
| Q3175 | 0.020 | < | 0.020 | < | 0.020 | < | - | | QM\|ZN\|HO\|TD\|ZB |
| Q980 | 0.030 | < | 0.030 | < | 5.00 | < | 5.00 | < | -\| -\|ZC\|PY\|ZA |
| **Polystyrene total** | **(mg/tablet)** | | **(mg/tablet)** | | **(mg/kg)** | | **(mg/kg)** | | |
| H221 | - | | 0.012 | | 34.2 | < | 1189 | | -\| -\|ZA\|PY\|ZA |
| Q3175 | 0.010 | < | 0.050 | | 0.010 | < | - | | QM\|ZN\|HO\|TD\|ZB |
| Q3189 | - | | 0.002 | | - | | 345 | | ZB\|ZB\|ZB\|PY\|ZB |
| Q3877b | - | | - | | - | | 1223 | | ZA\|ZA\|ZA\|PY\|ZA |
| Q871 | 0.390 | | 0.027 | | 8.07 | | 3222 | | |
| Q980 | 0.016 | < | 0.006 | | 3.00 | < | 60.0 | | -\| -\|ZC\|PY\|ZA |
| Added mass | - | | 0.0151 | | - | | 2074 | | |
| | ========== | | | | | | | | |

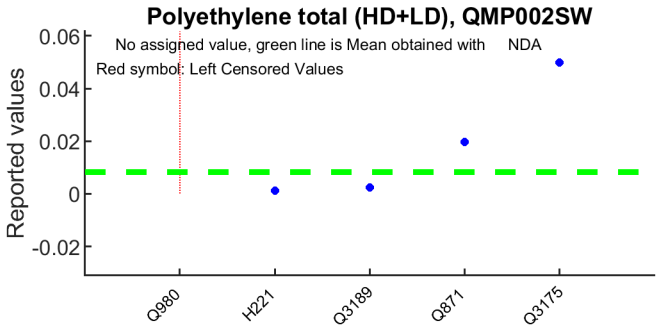
# Total mass of plastic particles Data and Statistics

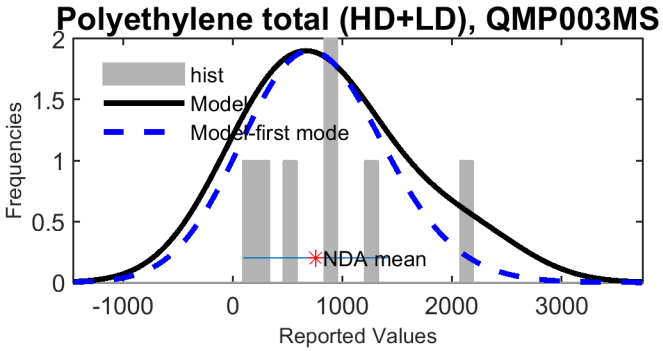
| Sample | QMP001SW | | QMP002SW | | QMP003MS | | QMP004BT | MIC |
|---|---|---|---|---|---|---|---|---|
| **Polyvinyl Chloride total** | **(mg/tablet)** | | **(mg/tablet)** | | **(mg/kg)** | | **(mg/kg)** | |
| H221 | 0.001 | | 0.019 | | 78.0 | | 398 | -\| -\|ZA\|PY\|ZA |
| Q3175 | 0.100 | < | 0.100 | < | 0.100 | < | - | QM\|ZN\|HO\|TD\|ZB |
| Q3189 | - | | 0.038 | | 7.41 | | - | ZB\|ZB\|ZB\|PY\|ZB |
| Q3877b | - | | - | | 434 | | 8437 | ZA\|ZA\|ZA\|PY\|ZA |
| Q980 | 0.018 | | 0.042 | | 60.0 | | 8900 | -\| -\|ZC\|PY\|ZA |
| Added mass | - | | 0.0173 | | - | | - | |
| **Total Polymers (50-299 µm)** | **(mg/tablet)** | | **(mg/tablet)** | | **(mg/kg)** | | **(mg/kg)** | |
| Q3934 | 5.70 | | 22.7 | | 2253 | | 60.6 | |
| Q3936 | - | | - | | 243 | | - | |
| **Total Polymers (300-5000 µm)** | **(mg/tablet)** | | **(mg/tablet)** | | **(mg/kg)** | | **(mg/kg)** | |
| Q3934 | - | | - | | 1308 | | - | |
| Q3936 | - | | - | | 6.92 | | - | |
| **Total Polymers** | **(mg/tablet)** | | **(mg/tablet)** | | **(mg/kg)** | | **(mg/kg)** | |
| H221 | 0.001 | | 0.033 | | 1740 | | 3856 | -\| -\|ZA\|PY\|ZA |
| Q3175 | 0.520 | | 0.620 | | 244 | | - | QM\|ZN\|HO\|TD\|ZB |
| Q3189 | - | | 0.052 | | 494 | | 938 | ZB\|ZB\|ZB\|PY\|ZB |
| Q3877b | - | | - | | 1370 | | 11016 | ZA\|ZA\|ZA\|PY\|ZA |
| Q3929 | 2.00 | < I | 2.00 | < I | - | | 11629 | -\| -\|ZB\|GR\|ZA |
| Q3932 | - | | - | | 846 | | 33050 | |
| Q3934 | 5.70 | | 22.7 | | 3561 | | 60.6 | |
| Q3935 | 0.050 | | 1.01 | | 486 | | 377 | ZA\|ZA\|ZA\|ZA\|ZA |
| Q3936 | 0.023 | | 0.045 | | 250 | | - | |
| Q3941 | 0.160 | | 0.180 | | 2161 | | 11844 | ZA\|ZA\|ZA\|GR\|ZA |
| Q871 | 0.390 | | 0.047 | | 2141 | | 3547 | |
| Q980 | 0.018 | | 0.048 | | 2700 | | 14330 | |
| | ========== | | Statistical Results | | ========== | | ========== | |
| NDA mean | 0.0673 | | 0.0450 | | 1333 | | 6361 | |
| NDA st dev | 0.1370 | | 0.0227 | | 1245 | | 7610 | |
| Coeff Var (%) | 203.5 | | 50.3 | | 93.4 | | 119.6 | |
| N | 8 | | 9 | | 11 | | 10 | |
| Median | 0.1051 | | 0.0520 | | 1370 | | 7436 | |
| MAD | 0.0957 | | 0.0192 | | 876 | | 5453 | |
| Added mass | - | | 0.0687 | | 2952 | | 4609 | |

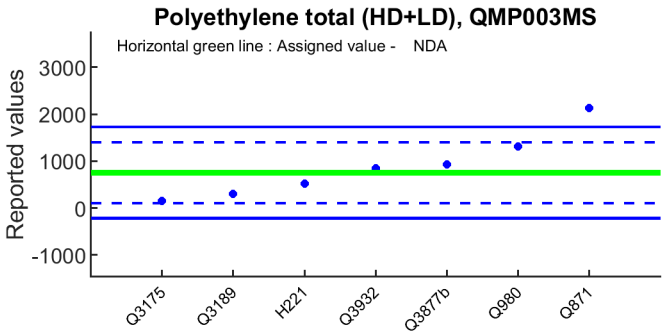
==========

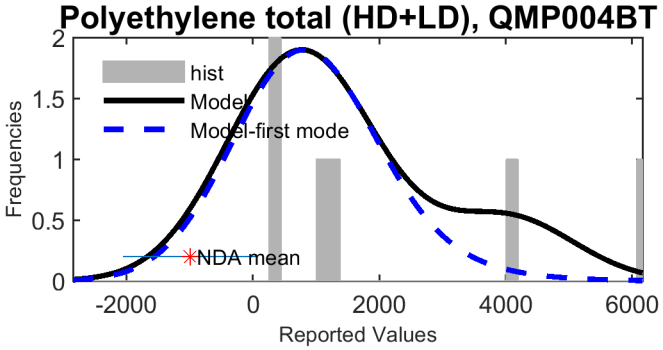
# Total mass of plastic particles Histogram+PDFs and Ranked overview

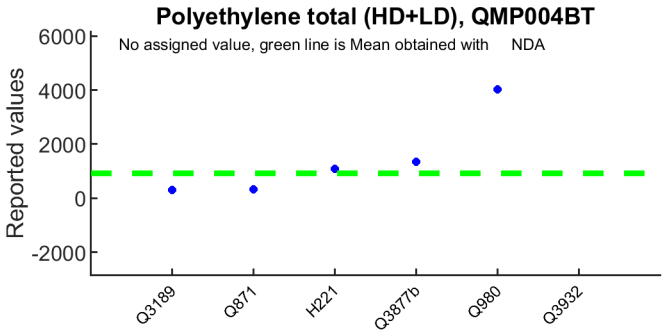


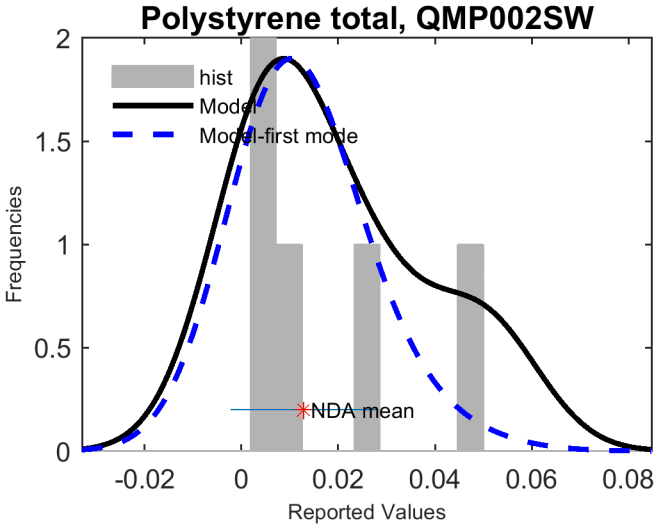



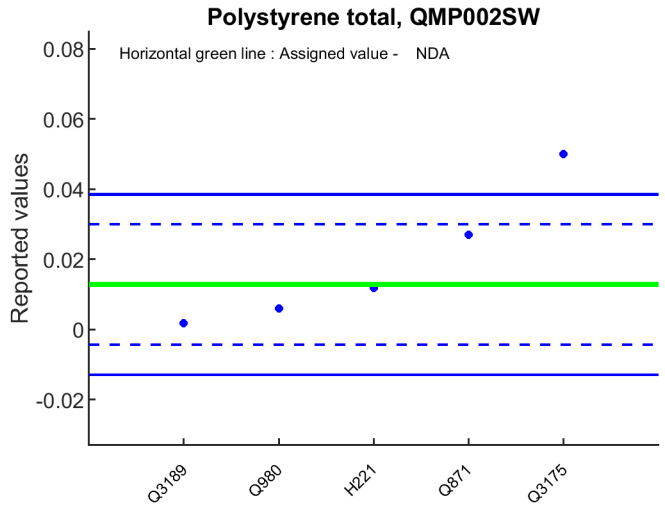



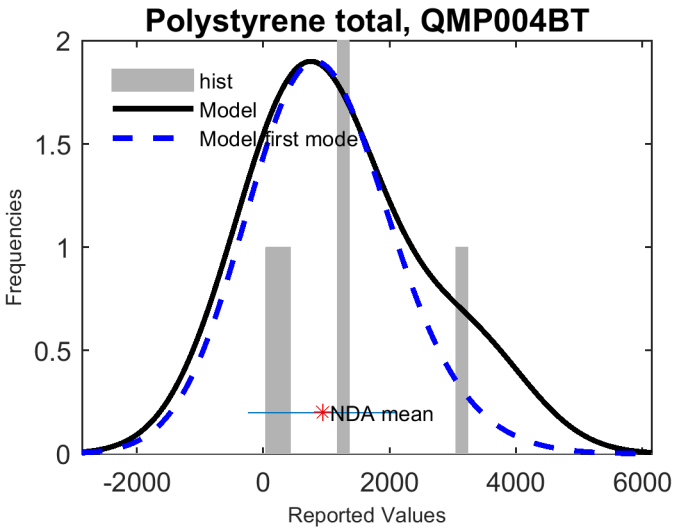



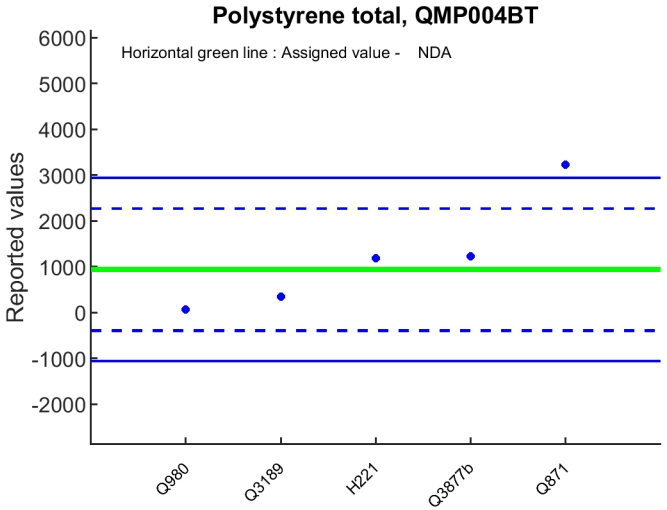



# Total mass of plastic particles Histogram+PDFs and Ranked overview

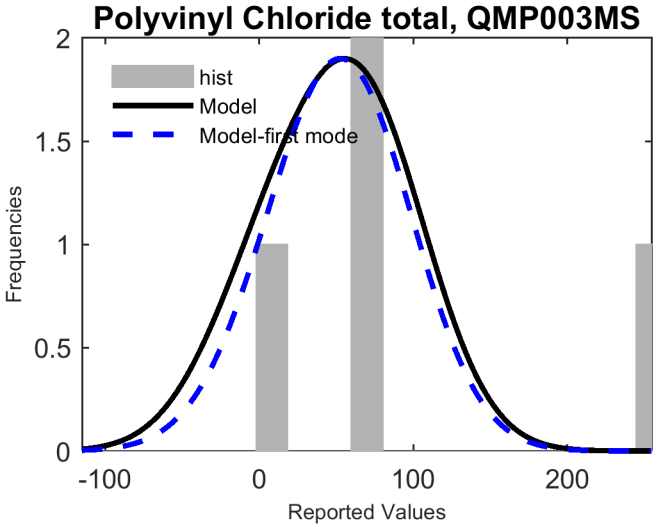


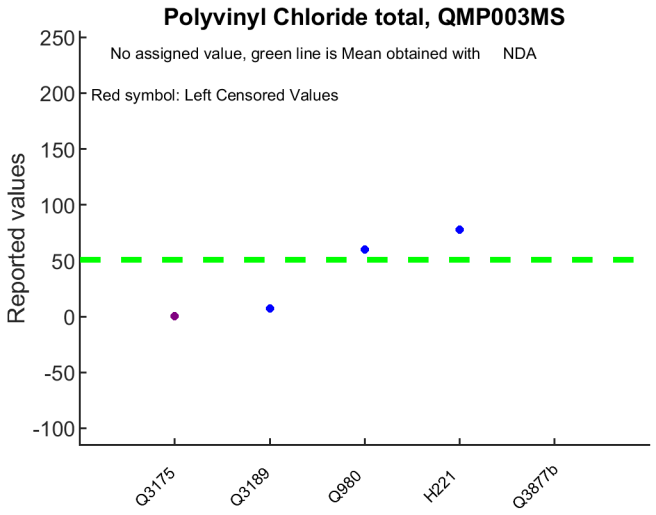



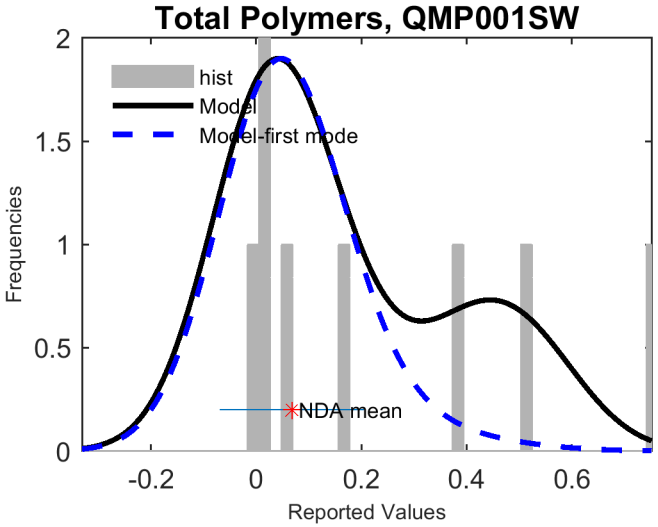



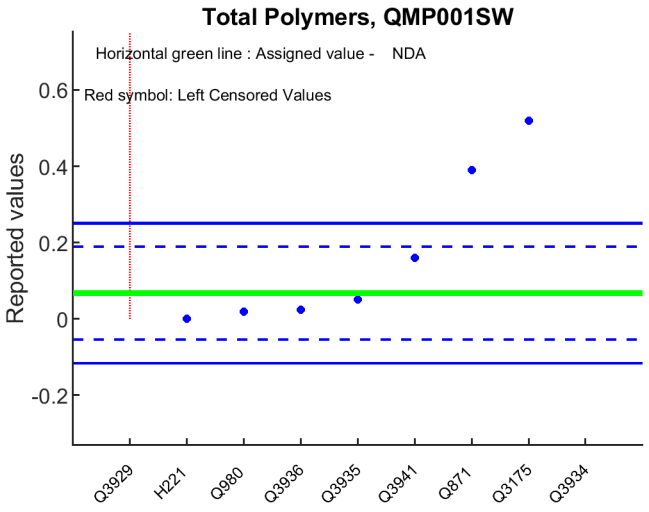
# Total mass of plastic particles Histogram+PDFs and Ranked overview

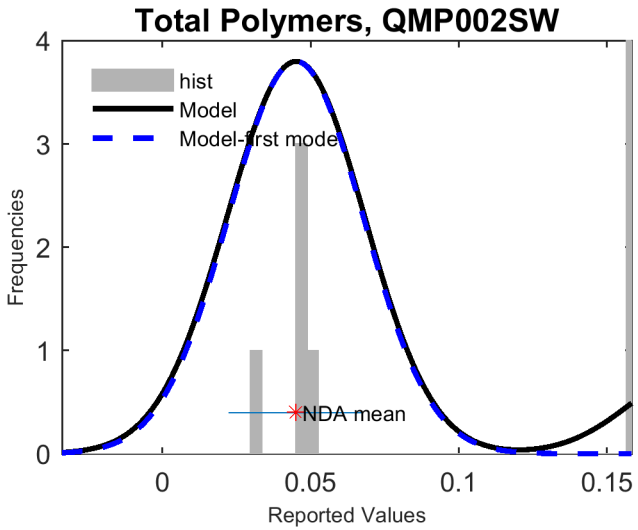


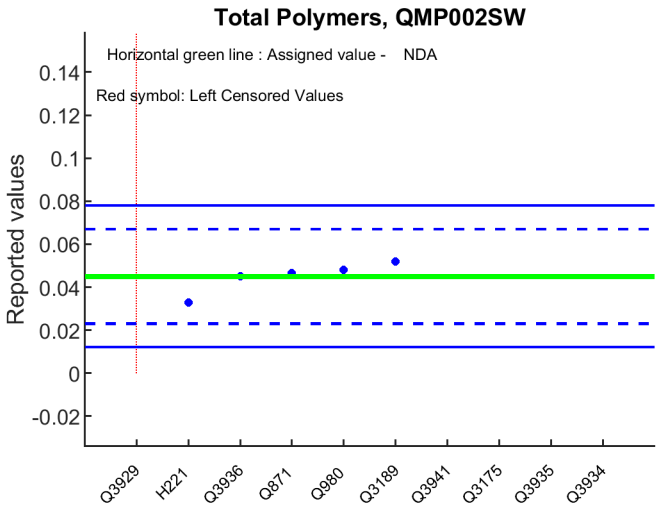



# Total mass of plastic particles Histogram+PDFs and Ranked overview

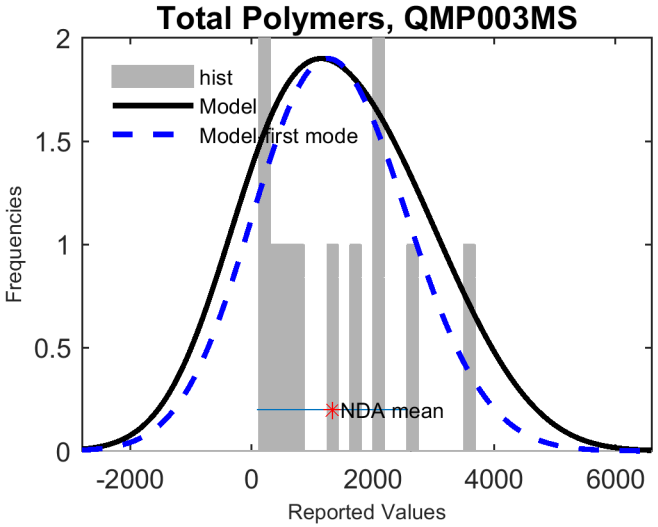


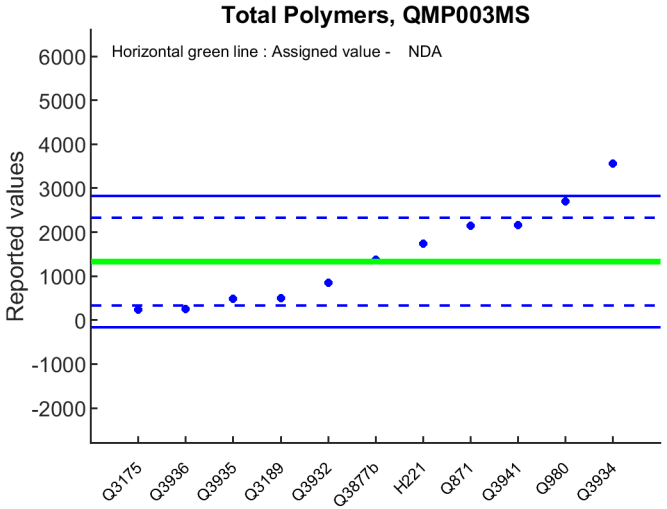



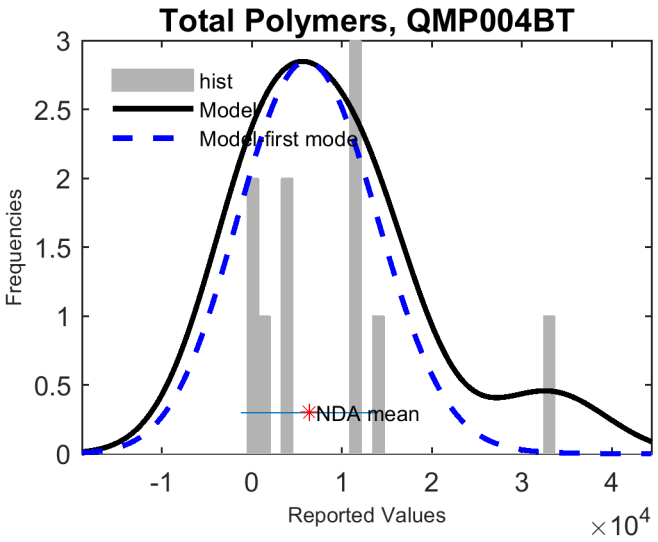



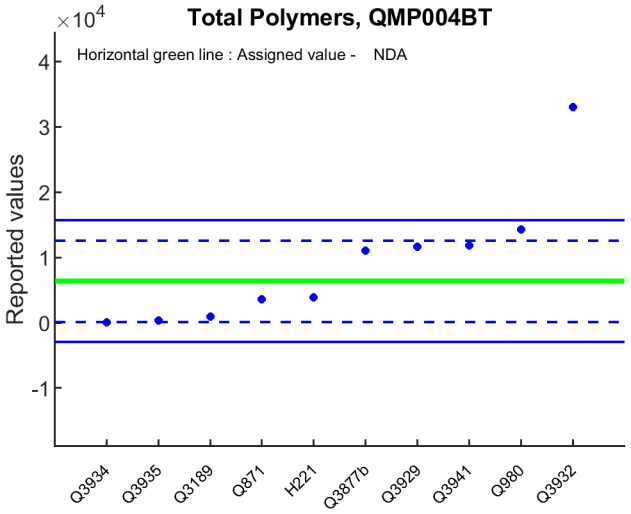



# Total mass of plastic particles Histogram+PDFs and Ranked overview



# Appendix B Additional method information

**Reported filtration methods**

| Lab code | Applied method | Comments |
|---|---|---|
| H221 | - | |
| Q101 | ZA | 1st step stainless steel filter - 2nd step Nile Red PTFE filter |
| Q110 | ZA | Filtration on cellulose nitrate filter (12µm) |
| Q122 | ZA | For the biota sample, after oxygene peroxide digestion, samples were vacuum filtered |
| Q134 | ZA | Vacuum filtration |
| Q153 | ZC | 0.2 micron filters for tablet and sediment. 2.7 micron for fish |
| Q3160 | ZA | filtering through a glass fiber filter (e.g. GF/F 0.7µm/pore, Whatman) using glass filtration system |
| Q3175 | Quartz filter QM-A, 2.2 pore size | |
| Q3189 | ZB | Vacuum filtration on 27µm mesh steel filters in custom stainless steel filtration apparatus |
| Q3231 | ZA | 25 µm stainless steel mesh sieve |
| Q3877b | ZB | Filtration of the supernatant after density separation on a 47mm quartz filter calcined at 450°C. |
| Q3878 | ZA | Filtered on Si filter with 50um pore size |
| Q3882 | ZA | 40-45µm filter, The Mesh Company #300 Mesh SS316 Grade Woven Wire Mesh |
| Q3886 | ZA | Whatman 540, pore size diameter 8 micron |
| Q3887 | ZA | 20 um stainless steel filter, prior to analysis: 0.45 um gridded filter |
| Q3888b | ZA | Filtration of 1L of spiked ultra-pure water |
| Q3889 | ZA | Tablets: 1) 50 ml H2O closed with aluminium foil; 2) blister inside and closed for 30 min; 3) filtration 0.7 |
| Q3889 | ZB | Sediment: 100 ml KOH 10% 6h/60ºC; 2)Overnight room temp; 3)Centrifugation 4000rpm/10min; 4) Filtration 0.7 um; 5) 40 ml HNO3 20% 1h; 6) filtration; 7) Overnight 60ºC |
| Q3889 | ZC | Biota: 400 ml NaCl (120 g/L) shaking for 30 min; 2) left to rest overnight; 3) Filtration 0.7 um; 4) 40 ml HNO3 20% 1h; 5) Filtration; 6) Overnight 60ºC |
| Q3890 | ZC | Tablets: anodisc 25mm 0.1um pore size |
| Q3890 | ZC | Sediment & biota: nylon net 20µm 47mm, then anodisc 0.1µm 25mm |
| Q3911 | ZB | Filtration through 5.0µm silver membrane |
| Q3913 | ZA | vacuum pump, PTFE filters d = 4.5 cm & pore = 10 um |
| Q3917 | ZA | Collected solids were passed through PTFE filters (pore size 0.2 µm). |
| Q3926 | ZA | 0.45 um filter |
| Q3935 | ZA | Vacuum filtration with stainless steel funnel and were concentrated on a cellulose acetate filter |
| Q3936 | ZA | Metallic mesh (63 and 250 µm) |
| Q3941 | ZA | For QM001 and QM002 filtration system and Celullose mixed filters 0.8 um |
| Q3943 | ZA | Filters of 300 and 40 microns |
| Q661 | ZA | Sieves for 5000, 300, 50 um |
| Q980 | - | The tablets were dissolved in MilliQ water, filtered through a 1um GFF, then extracted with ASE (pressurised liquid extraction). The extract was then pyrolyzed for analysis (Pyr-GC-MS). |
| Q980 | - | For the soil, sediment, fish, an aliquot was directly ASE extracted with the extract then pyrolyzed for analysis. |

ZA, ZB or ZC means that the laboratory has use another filtration method than in options available, which is then described in the column comments.

**Reported separation methods**

| Lab code | Applied method | Comments |
|---|---|---|
| Q110 | ZA | Density separation by KOH 10% and sodium tungstate 700g/L |
| Q122 | ZB | For sediment sample density separation was acheived with NaCl (one time) and NaI (twice) |
| Q134 | ZA | Sodium chloride saturated solution |
| Q153 | ZA | Only for sediment. Zinc chloride (1.5 g/mL density) |
| Q3160 | ZA | Sodium chloride solution (NaCl – 1.2 g cm-3) to soil sample volume in a 1:3 ratio |
| Q3175 | ZnCl2 solution | - |
| Q3189 | ZB | CaCl2 solution (1.45g/cm3). Sample stirred then 4-12h settling prior to decantation, 4 repetitions. |
| Q3231 | ZA | NaI 1.8 g/mL on sample QMP003MS (sediment) |
| Q3877b | ZB | With CaCl2 1,4 density after H202 digestion (soil and sediment) or KOH (fish) |
| Q3878 | ZA | With NaI solution, repeated three times |
| Q3882 | ZA | Density separation with zinc chloride of soil samples. |
| Q3886 | ZA | 250 mL NaCl (1.2g/mL) for QMP001SW, QMP002SW and QMP004BT. For QMP003SW, 250 mL 4.4M NaI was used. |
| Q3886 | ZA | For all spoil samples, 250 mL 4.4M NaI was used in density separation. The process was repeated 2x. |
| Q3887 | ZA | Soils and sediment were separated using ZnCl2 approx. 1.6 g/mL according to Coppock et al. 2017 |
| Q3913 | ZA | NaI, centrifugation 1000 rpm (216 x g-force) for 5 min |
| Q3917 | ZA | Density separation with 6.7 M sodium iodine (NaI) solution was conducted with lid. |
| Q3926 | ZA | With NaCl |
| Q3935 | ZA | Particle suspension using a supersaturated NaCl solution with an approximate density of 1.2 g/mL |
| Q3941 | ZA | 3 extractions w. saturated NaCl; centrifugation 15 minutes, 4,000 RPM. only supernatant |
| Q661 | ZA | Water, NaCl (density 1.2 g/mL ), NaI (density 1,6 g/mL) |

ZA, ZB or ZC means that the laboratory has used another filtration method than in options available, which is then described in the column comments.

**Reported clean-up methods**

| Lab code | Applied method | Comments |
|---|---|---|
| H221 | ZA | ASE-extraction including a clean-up with methanol and extraction with dichloromethane |
| Q101 | ZA | first step KOH 10% / 2nd step: H2O2 15% |
| Q110 | ZA | H2O2 and KOH digestion and sifting |
| Q122 | ZA | H2O2 (30%) digestion at 50 °C for max 5 days |
| Q153 | ZA | Chemical digestion (KOH:NaClO) for sediment and fish |
| Q3160 | ZA | Fish:digestion of soft tissue with Hydrogen peroxide 30%. 1:1 ratio with sample Wg. oven at 40ºC 48h |
| Q3175 | H2O2 30% @ 40°C | - |
| Q3189 | ZB | Sample solved in 10% KOH solution agitated at 60C for 48h. |
| Q3231 | ZA | 6.66 % KOH (m/m) with surfactant |
| Q3877b | ZB | H202 digestion (soil and sediment) or KOH (fish) |
| Q3878 | ZA | Enzymatic digestion (protease, lipase, amylase) for fish |
| Q3887 | ZA | Hydrogen peroxide at 40 degrees in oscillating incubator |
| Q3889 | | 1)100 ml KOH 10% 6h/60ºC;2)Overnight 20ºC;3)Centrif.4000rpm/10min;4)0.7um;5)40mlHNO3 20%1h;6)60ºC |
| Q3889 | | 400 ml NaCl (120 g/L) shaking for 30 min; 2) quiet 30 min; 3) Filtration 0.7 um; 4) Overnight 60ºC |
| Q3890 | ZA | 15mL H2O2 30% v/v 40°C, |
| Q3890 | ZB | 90mL H2O2 30% v/v 40°C, SDS 5% w/v |
| Q3911 | ZB | Decon % + Ethanol + MQ water |
| Q3917 | ZA | 30% H2O2 with 0.05 M Iron (II) Sulfate solution was added to digest natural organic matters. |
| Q3926 | ZA | Hydrogen peroxide 20 % and KOH |
| Q3929 | ZB | Microwave assisted digestion using SRC technology and diluted HNO3, employing the UltraWAVE system. |
| Q3935 | ZA | Were concentrated on the cellulose acetate filter using a dosing bottle with ultra pure water MilliQ |
| Q3936 | ZA | Digestion of biogenic material with 30% H2O2, 45 °C, 72h |
| Q3941 | ZA | Filters washed with destilled water |
| Q3943 | ZB | KOH digestion for fish sample |
| Q661 | ZA | Hydrogen Peroxyde (10%) |

ZA, ZB or ZC means that the laboratory has use another filtration method than in options available, which is then described in the column comments.

**Reported determination methods**

| Lab code | Applied method | Comments |
|---|---|---|
| H221 | Pyr-GC-MS | |
| Q101 | ZA | µFTIR + manual count on FI-microscope |
| Q110 | µ-FTIR | |
| Q122 | µFTIR | |
| Q134 | ZA | Manual counting with stereomicroscope and polymer identification with µ-FTIR |
| Q153 | ZB | Micro-FTIR FPA for tablet. micro-FTIR for sediment and fish |
| Q3160 | Microscopy | |
| Q3175 | TED-GC-MS | |
| Q3189 | Pyr-GC-MS | |
| Q3231 | µFTIR | |
| Q3877b | Microscopy & Pyr-GC-MS | |
| Q3878 | µFTIR | |
| Q3882 | µFTIR | |
| Q3886 | µFTIR | |
| Q3887 | Microscopy & ATR-FTIR | |
| Q3888b | µFTIR | |
| Q3889 | µFTIR | |
| Q3890 | µFTIR | [µF], [RA], [MI] |
| Q3911 | µFTIR | |
| Q3913 | ZA | uFTIR, Nile Red and manual microscope counting under fluorescence |
| Q3917 | µFTIR | |
| Q3926 | µFTIR | |
| Q3929 | Gravimetric | |
| Q3932 | ATR-FTIR | |
| Q3935 | ZA | Tests are being carried out for the identification analysis, but will not be ready by the deadline |
| Q3936 | ZA | Fluorescence microsopy (40X magnification) using the dye Nile red |
| Q3941 | Gravimetric | |
| Q3943 | ZB | Polarized optical microscopy (MOLP and image analysis) and micro-Raman confirmation |
| Q661 | Microscopy | |
| Q980 | Pyr-GC-MS | |

ZA, ZB or ZC means that the laboratory has use another filtration method than in options available, which is then described in the column comments.

# Appendix C NDA statistics

## Normal Distribution Approximation (NDA)

Interlaboratory studies like those of WEPAL-QUASIMEME frequently give rise to datasets that have complex distributions including excessive tailing and multiple modes. Consequently, sophisticated statistical methods are required to obtain meaningful assessments. A methodology is needed that does not rely on arbitrary outlier removal or subjective manual interpretations. The model that is chosen calculates population characteristics (mean and standard deviation) from experimental datasets as described by Cofino et al. (2000) and Molenaar et al. (2018).
The statistical principles of the model that we use to assess the data are outlined in two steps. Firstly, the full model is described, thereafter a description is given of the way the model is implemented for the assessment of the data in WEPAL and Quasimeme.

We assume that each laboratory i submits a result given by a probability density function $q_i$. We start thus from a set of probability density function $q_i$. i=1,....,N. We set ourselves to establish the average probably density function $\bar{q}$ that best describes the set.

It is insightful to make at this point an analogy with the calculation of the arithmetic mean of a set of data $a_i, i = 1, \dots, N$. The average $\bar{a}$ can be defined as the point that minimises the sum of the squared Euclidean distances $d(\bar{a}, a_i)$ to the given data. This can be accomplished by equating the first derivative of $\sum_{i=1}^{N} d^2(\bar{a}, a_i) = \sum_{i=1}^{N} (\bar{a} - a_i)^2$ with respect to $\bar{a}$ to zero. One readily finds the well known expression $\bar{a} = \frac{1}{N}\sum_{i=1}^{N} a_i$

In a similar manner, we construct the average probability density function $\bar{q}$ of the set of probability density functions $q_i, i = 1, \dots, N$. We define a measure $d(p, q)$ for the distance between two probability density functions $p$ and $q$. We obtain $\bar{q}$ by minimising the sum of the square distances from each probability density function $q_i$ to $\bar{q}$,thus by equating the first derivative of $\sum_{i=1}^{n} d(\bar{q}, q_i)^2$ with respect to $\bar{q}$ to zero. The calculation itself is extensive and not given here. The mean and standard deviation of the population are calculated using the first and second moments of the probability density function $\bar{q}$ . The variance obtained from the second moment comprises both a within-laboratory and between-laboratory component.

In WEPAL and Quasimeme, laboratories report single data, we have no information about the underlying probability function. To cope with this problem we use a specific implementation of the model: the so-called Normal Distribution Approximation (NDA). The NDA approach is parametrised to reproduce the population characteristics of truly normal distributions, and is a robust method to evaluate interlaboratory studies.

The NDA approach has been devised using a set of normal distributions $q_i = N(\mu_i, \sigma), i = 1, \dots, N$. We assume thus that all normal distributions have the same standard deviation $\sigma$. The expected values $\mu_i$ are also taken to be normally distributed: $\mu_i = N(\bar{\mu}, S)$. It appears that the mean $\bar{\mu}$ and the standard deviation $S$ of the normal distribution describing the population can be exactly reproduced when $\sigma = 0.78 * S$. In the NDA method, the standard deviation $S$ is calculated directly from the total variance, no distinction between within-laboratory and between-laboratory components is made.

In practice, we have N laboratories each reporting a single value. This gives rise to a dataset $x_i, i = 1, \dots, N$. We calculate the population standard deviation from this dataset using the robust estimate *S=1.4826*MAD* (MAD: median of absolute standard deviations). The normal distributions associated with the data $x_i$ are estimated by $q_i = N(x_i, 0.78S) = N(x_i, 1.156 * MAD)$. We calculate the average probability density function $\bar{q}$ of the set of normal distributions qi as described above. The mean

and standard deviation of the interlaboratory study are obtained using the first and second moments of the average probability density function $\bar{q}$ .

## The NDA-mean (assigned value)

The NDA mean is centered around the highest density of values. Unless otherwise stated, 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.
With the NDA model, mean and standard deviation are calculated (under special conditions, see indicative values) 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