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Determination of PBT cyclic oligomers in and migrated from food contact materials

FCM-19/01 Proficiency Testing Report

E. Tsochatzis, P. Dehouck, J. Alberto Lopes,
H. Emteborg, P. Robouch, E. Hoekstra

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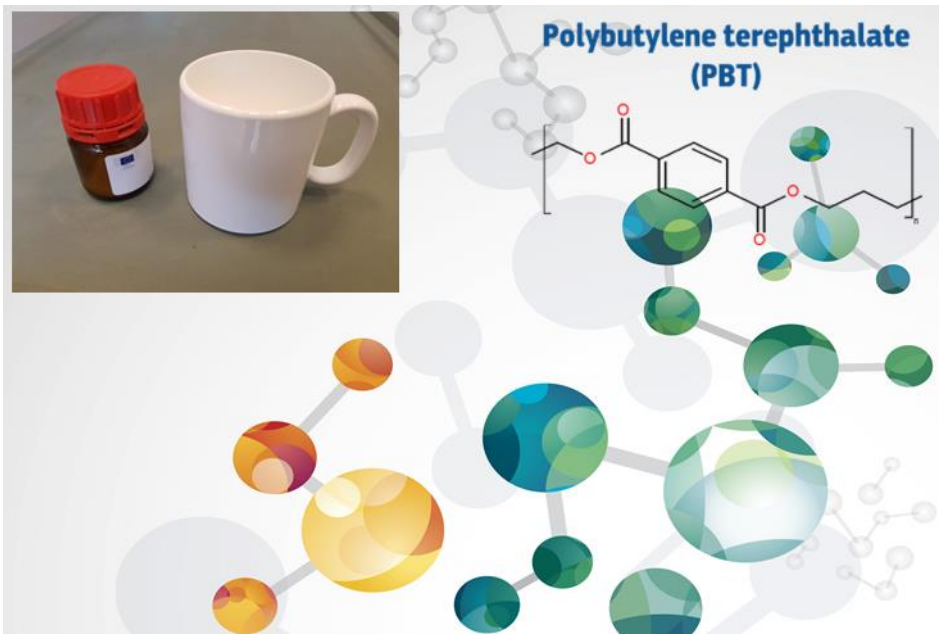

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FCM-19/01 Proficiency Test Report

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P. Robouch, E. Hoekstra

268-PT Accredited by the
Belgian Accreditation Body (BELAC)

Table of contents

Executive summary

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM) organised a proficiency test (FCM-19/01) for the determination of four butylene terephthalate cyclic oligomers (dimer, trimer, tetramer and pentamer) in (i) a ground food contact material and (ii) after migration from polybutylene terephthalate (PBT) cups at 70 °C for 2 h, using food simulant D1 (ethanol/water 50 % v/v) to support Commission Regulation (EU) No 10/2011 on plastic materials and articles intended to come into contact with food. This proficiency test was open to National Reference Laboratories (NRLs) and Official Control Laboratories (OCLs).

Two test items were provided: a ground PBT food contact material for an extraction experiment, and PBT cups for a migration experiment at 70 °C for 2 h, using food simulant D1. The homogeneity and stability of the test items were evaluated and the assigned values were derived from the results obtained by the EURL-FCM.

Twenty two NRL and six OCLs reported results for the 8 measurands.

Results reported for Test item 1 were rated using D % performance, while the results reported for Test item 2 were rated using *z* and zeta (ζ) scores in accordance with ISO 13528:2015. A relative standard deviation for proficiency assessment (σ_{pt}) of 20 % of the respective assigned values was set for the four oligomers from Test item 2, based on the perception of experts.

A significant scatter of reported results was observed in the frame of the extraction experiment for Test item 1. This may be attributed to the different extraction techniques applied by the participants (ranging from a mild solid-liquid extraction up to the full dissolution of the plastic powder). This indicates that an harmonised extraction protocol may need to be defined in order to ensure comparable results in the frame of Regulation (EU) No 10/2011.

Regarding the migration experiment performed with Test item 2, ca. 60 % of the participating laboratories performed satisfactorily (according to the z score) for the determination of the PBT cyclic dimer and trimer. However, the satisfaction rate dropped to ca. 30 % for the determination of the PBT cyclic tetra and pentamer.

Most of the laboratories (over 60 %) reported realistic measurement uncertainties. However, several laboratories applied not yet validated analytical methods and estimated their measurement uncertainty from replicate analyses only.

The extraction and migration experiments indicate that further studies are needed before one could demonstrate the ability of NRLs in monitoring PBT oligomers in the frame of Commission Regulation (EU) No 10/2011.

List of abbreviations and symbols

DG SANTE	Directorate General for Health and Food Safety
EURL-FCM	European Union Reference Laboratory for Food Contact Materials
GUM	Guide for the Expression of Uncertainty in Measurement
HPLC	High Performance Liquid Chromatography (with ultraviolet or Diode Array Detection: HPLC-UV and HPLC-DAD, respectively)
HFIP	1,1,1,3,3,3-hexafluoro-2-propanol
ISO	International Organization for Standardization
JRC	Joint Research Centre
LC-MS/MS	Liquid Chromatography-tandem mass spectrometry
LOD	Limit of Detection
MAE	Microwave Assisted Extraction
NIAS	Non-Intentionally Added Substances
NRL	National Reference Laboratory
OCL	Official Control Laboratory
PBT	Polybutylene terephthalate
PET	Polyethylene terephthalate
PT	Proficiency Test
RM	Reference Material
k	coverage factor
σ_{pt}	standard deviation for proficiency test assessment
$u(x_i)$	standard measurement uncertainty reported by participant "i"
$u(x_{pt})$	standard uncertainty of the assigned value
$u_{rel}(x_i)$	relative standard measurement uncertainty reported by participant "i"
$U_{rel}(x_{pt})$	relative expanded uncertainty of the assigned value
u_{char}	(standard) uncertainty contribution due to characterisation
u_{hom}	(standard) uncertainty contribution due to homogeneity
u_{st}	(standard) uncertainty contribution due to stability
$U(x_i)$	expanded uncertainty reported by participant "i"
$U(x_{pt})$	expanded uncertainty of the assigned value
x_i	mean value reported by participant "i"
x_{pt}	assigned value
z	z score
ζ	zeta score
D_i %	Difference performance score

1 Introduction

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM), hosted by the Joint Research Centre of the European Commission, organised a proficiency test (PT) for the determination of four cyclic polybutylene terephthalate (PBT) oligomers in and migrated from food contact materials, to support Commission Regulation (EC) No 10/2011 The four cyclic PBT oligomers are regulated as FCM substance No 885 [1, 2].

This PT was agreed with the Directorate General for Health and Food Safety (DG SANTE) as part of the EURL-FCM annual work programme 2019, thus complying with the mandate set in Regulation (EU) 2017/625 [3]. The PT was open to National Reference Laboratories (NRLs) and Official Control Laboratories (OCLs) willing to participate.

This PT implies two different experiments:

i. The migration of oligomers from PBT cups applying the experimental conditions set by the legislation - to be considered as the follow-up of the previous PT (FCM-18-01) [4]; and

ii. The extraction of the abovementioned oligomers from ground PBT cups – first PT of this kind organised by the EURL-FCM.

This report summarises the outcome of the PT.

2 Scope

The present PT aims to assess the performance of NRLs and OCLs in the determination of the mass fractions of butylene terephthalate cyclic dimer (PBT-di), butylene terephthalate cyclic trimer (PBT-tri), butylene terephthalate cyclic tetramer (PBT-tetra) and butylene terephthalate cyclic pentamer (PBT-penta) in two test items: (i) a PBT ground material and (ii) a solution resulting from a migration test with a PBT cup.

This PT, organised in line with ISO 17043:2010 [5], is identified as "FCM-19/01".

3 Set up of the exercise

3.1 Quality assurance

The JRC Unit hosting the EURL-FCM is accredited according to:

- ISO/IEC 17025:2005 (certificate number: BELAC 268-TEST); and
- ISO/IEC 17043:2010 (certificate number: BELAC 268-PT, proficiency test provider)

The reported results were evaluated following the relevant administrative and logistic procedures.

3.2 Confidentiality

The procedures used for the organisation of PTs guarantee that the identity of the participants and the information provided by them is treated as confidential. The participants in this PT received a unique laboratory code used throughout this report. However, the laboratory codes of NRLs appointed in line with Regulation (EU) 2017/625 [3] may be disclosed to DG SANTE upon request for the purpose of an assessment of their (long-term) performance. Similarly laboratory codes of appointed OCLs may be disclosed to their respective NRL upon request.

3.3 Time frame

The organisation of the PT FCM-19/01 round was announced by invitation letters to NRLs and OCLs on April 02, 2019 (Annex 1). The registration deadline was set to April 30, 2019. Samples were sent to participants on May 13, 2019. The deadline for reporting of results was set to June 30, 2019. This deadline was extended till July 5, 2019 triggered by the request of certain laboratories that submitted well justified reasoning.

3.4 Distribution

Each participant received:

- One bottle containing 10 g of ground PBT beverage cups, with an average particle size of 266.3 µm, for the extraction experiment (Test item 1);
- Three PBT cups to be used for migration experiment (Test item 2);
- One vial of the calibration solution - mixture containing of four target analytes (PBT cyclic dimer to pentamer) at 20 µg mL^{-1} each, containing approx. 1 mL of HFIP/ethanol;
- The "Test item accompanying letter" (Annex 2); and
- The "Confirmation of receipt form" to be sent back to the PT coordinator after receipt of the test item (Annex 3).

3.5 Instructions to participants

Detailed instructions were given to participants in the "Test item accompanying letter" mentioned above (Annex 2).

The measurands were defined as

- "the mass fractions (mg kg^{-1}) of butylene terephthalate (PBT) cyclic dimer, trimer, tetramer and pentamer extracted from ground PBT cups";
- "the concentrations (mg L^{-1}) of butylene terephthalate (PBT) cyclic dimer, trimer, tetramer and pentamer in the migration solution".

Participants were asked to check whether the test items and vial were undamaged after transport, and to report if necessary using the "Confirmation of receipt form" (Annex 3).

Participants were asked to perform two or three independent measurements and to report their calculated mean (x_i) and the associated expanded measurement uncertainty ($U(x_i)$) together with the coverage factor (k) and the analytical technique used for analysis.

Results had to be reported in the same format (e.g. number of significant figures) as normally reported to customers. The homogeneity study was performed with intakes of 500 mg for test item 1 and 50 µL for test item 2, with recommended minimum sample intakes of 500 mg and 50 µL, respectively.

Participants were informed that the procedure used for the analysis should resemble as closely as possible their routine procedures for this type of matrix/analytes and mass fraction or concentration levels.

Participants received an individual code to access the on-line reporting interface, to report their measurement results and to complete the related questionnaire. The latter was designed to gather additional information related to measurements and laboratories (Annex 4).

Random laboratory codes were attributed and communicated to participants by e-mail.

4 Test item

4.1 Preparation

4.1.1 Test item 1

PBT cups were supplied by Wako GmbH & Co. KG (Vlotho, Germany), covered with PE, shipped on cardboard base, and stored at room temperature (max 25 °C). These cups were used for the preparation of the ground material to be used for the extraction experiment.

The EURL-FCM and the Reference Material Unit of the JRC (Unit F.6) prepared the material. The cups were first cut into pieces and crushed manually. The pieces were then pre-cooled and cryo-milled using a Palla VM-KT vibrating mill, to obtain particle sizes below 500 µm, with an average particle size of 266.3 µm. Ten grams portions were filled in Schott bottles and properly labelled.

4.1.2 Test item 2

PBT cups were used as such for the migration experiment to be performed at 70 °C for 2 h with food simulant D1 (solution of water with ethanol 50 % v/v), as prescribed by Annex III of Commission Regulation (EU) No 10/2011 [1].

4.1.3 Calibration solution

A stock solution (20 µg mL^{-1}) containing the four cyclic oligomers in HFIP/EtOH was prepared gravimetrically. Portions of 1 mL were manually transferred to 5 mL amber vials, and stored at -18 °C. Each vial was identified with a unique number and the PT identifier.

4.2 Analytical approaches for samples characterisation

4.2.1 Extraction experiment

The following Soxhlet extraction protocol was applied for the determination of the mass fraction of the four oligomers of interest contained in Test item 1: 500 mg (± 1 mg) of PBT material was accurately weighed and placed into the Soxhlet extraction thimbles (33 x 80 mm), with 100 mL of dichloromethane (DCM) as the extraction solvent. The system was operated for 48 h. No PBT cyclic oligomer was detected after performing a second Soxhlet. The solvent was then quantitatively transferred into a 100 ml volumetric flask that was filled to volume. An aliquot was then diluted (1:100 v/v) with food simulant D1 to obtain the solution to be analysed.

4.2.2 Migration experiment

The migration experiment was performed according to Annex V of Regulation (EC) 10/2011, CEN 13130-1:2004 and ISO 1186-9:2002 [1, 6, 7]. An in-house developed oven method was adapted for this specific test item [6, 8, 9]. Each cup was filled, up to 5 mm from the top, with 285 mL (± 5 mL) of pre-heated food simulant D1 and covered

with a convex clock glass to minimise evaporation during the experiment. The oven was rigorously monitored with calibrated and certified dataloggers to ensure that the temperature of the food simulant was kept at 70 ± 2 °C throughout the entire migration experiment [6, 8, 9]. No pre-heating of the test item prior to the experiment was applied. At the end of the migration experiment the food simulant was carefully transferred and the volume was measured to determine any potential losses. The solution was then analysed directly.

4.2.3 Analytical method

The concentrations of the four oligomers of interest (after extraction or migration) were determined by the EURL-FCM using an in-house validated method. This method was based on High Performance Liquid Chromatography with Diode Array Detection (HPLC-DAD, at λ = 240 nm). An Agilent Zorbax Eclipse XDB-C18 column (150 x 4.6 mm, 5 µm) thermostated at 40 ± 1 °C was selected. The mobile phase consisted of acetonitrile (solvent A) and water (solvent B). The applied elution profile is described in Annex 4. The injection volume was set to 50 µL.

4.3 Homogeneity and stability

Measurements for the homogeneity and stability studies and the statistical treatment of data were performed by the EURL-FCM.

The assessment of homogeneity was performed after the preparation of the test items and before distribution to participants. For each item, ten units were randomly selected and analysed in duplicate.

Results were evaluated according to ISO 13528:2015 [10]. Both test items proved to be adequately homogeneous for the target analytes (Annex 5.1). The contribution from homogeneity (u_{hom}) to the standard uncertainty of the assigned value ($u(x_{pt})$) was calculated using SoftCRM [11].

Two additional samples of each test item were analysed in duplicate after the reporting deadline. Results were then compared to those obtained from the homogeneity study. This stability study confirms that the two test items are adequately stable (i) at 20 °C over the whole period of time of the PT (9 weeks, from the value assignment till the deadline for reporting results), (ii) for 1 week at 40 °C (simulating extreme conditions which may occur during transport). Hence, the uncertainty contribution due to stability was set to zero (u_{st} = 0) for all the investigated analytes (Annex 5.2).

5 Assigned values and corresponding uncertainties

5.1 Assigned values

Table 1 presents the assigned value (x_{pt}) of the mass fractions (mg kg^{-1}) of the PBT cyclic dimer, trimer, tetramer and pentamer determined after the Soxhlet extraction (Test item 1, cf. Section 4.2.1). Similarly, Table 2 presents the assigned values of concentrations (mg L^{-1}) of the four cyclic oligomers after migration with food simulant D1, at 70 °C for 2 h.

5.2 Associated uncertainties

The associated standard uncertainties of the assigned values ($u(x_{pt})$) were calculated following the law of uncertainty propagation, combining the standard measurement uncertainty of the characterization (u_{char}) with the standard uncertainty contributions from homogeneity (u_{hom}) and stability (u_{st}), in compliance with ISO 13528:2015 [10]:

$$u(x_{pt}) = \sqrt{u_{char}^2 + u_{hom}^2 + u_{st}^2} \qquad \text{Eq. 1}$$

where u_{char} was derived from the intermediate precision obtained in the frame of the method validation study, according to ISO 5725 [12].

5.3 Standard deviation for proficiency assessment for test items

Relative standard deviations for PT assessment (σ_{pt}) of 20 % was set for all measurands based on expert judgment for Test item 2.

Tables 1 and 2 present the relevant parameters needed for scoring: the assigned value (x_{pt}), its associated expanded uncertainty ($U(x_{pt})$) calculated with a coverage factor (k=2), and the standard deviation for the PT assessment (σ_{pt}) [for Test item 2, only].

Table 1: Assigned values related to the determination of extracted oligomers from Test item 1.

Oligomers	$x_{pt} \pm U(x_{pt})$ mg kg^{-1}
PBT-dimer	3595 ± 201
PBT-trimer	1423 ± 74
PBT-tetramer	670 ± 47
PBT-pentamer	408 ± 26

Table 2: Assigned values (x_{pt}), uncertainty contributions, corresponding expanded uncertainty ($U(x_{pt})$, k=2) and standard deviation for the PT assessment (σ_{pt}) related to the determination of migrated oligomers from Test item 2.

	Unit	**PBT cyclic dimer**	**PBT cyclic trimer**	**PBTcyclic tetramer**	**PBT cyclic pentamer**
x_{pt}	mg L^{-1}	2.23	0.353	0.0370	0.0245
u_{char}	mg L^{-1}	0.021	0.0032	0.0005	0.0002
u_{hom}	mg L^{-1}	0.079	0.0093	0.0007	0.0006
u_{stab}	mg L^{-1}	0	0	0	0
$u(x_{pt})$	mg L^{-1}	0.081	0.010	0.0009	0.0006
$U(x_{pt})$, k=2	mg L^{-1}	0.16	0.020	0.0018	0.0012
σ_{pt}	mg L^{-1} (%)	0.45 (20 %)	0.071 (20 %)	0.0074 (20 %)	0.0049 (20 %)
$u(x_{pt})/\sigma_{pt}$		0.2	0.1	0.1	0.1

6 Evaluation of results

6.1 Scores and evaluation criteria

Since the EURL-FCM addresses extraction experiments for the first time in this interlaboratory comparison, the individual performance for Test item 1 is expressed in terms of the difference $D_{\%}$ performance score according to ISO 13528:2015 [10]:

$$D_{i\,\%} = 100\,\frac{x_i - x_{pt}}{x_{pt}} \qquad \text{Eq. 2}$$

where: x_i is the measurement result reported by a participant; and

x_{pt} is the assigned value.

For the migration experiment, Table 2 shows that the uncertainties of the assigned values $u(x_{pt})$ were smaller than the corresponding "0.3 σ_{pt}" for the four measurands investigated. Hence, the individual laboratory performances for Test item 2 are expressed in terms of z and ζ scores in accordance with ISO 13238:2015 [10], as in the previous PT (FCM-18-01, [4]):

$$z = \frac{x_i - x_{pt}}{\sigma_{pt}} \qquad \text{Eq. 3}$$

$$\zeta = \frac{x_i - x_{pt}}{\sqrt{u^2(x_i) + u^2(x_{pt})}} \qquad \text{Eq. 4}$$

Where: $u(x_i)$ is the standard measurement uncertainty reported by a participant;

$u(x_{pt})$ is the standard measurement uncertainty of the assigned value;

σ_{pt} is the standard deviation for proficiency test assessment.

The interpretation of the *z* and *ζ* performance scores is done according ISO 13528:2015 [10]:

|score| ≤ 2	satisfactory performance	(green in Annexes 7 - 14)
2 < |score| < 3	questionable performance	(yellow in Annexes 7 - 14)
|score| ≥ 3	unsatisfactory performance	(red in Annexes 7 - 14)

The *z* scores compare the participant's deviation from the assigned value with the standard deviation for proficiency test assessment (σ_{pt}) used as common quality criterion.

The *ζ* scores state whether the laboratory's result agrees with the assigned value within the respective uncertainty. The denominator is the combined uncertainty of the assigned value $u(x_{pt})$ and the measurement uncertainty as stated by the laboratory $u(x_i)$. The *ζ* score includes all parts of a measurement result, namely the expected value (assigned value), its measurement uncertainty in the unit of the result as well as the uncertainty of the reported values. An unsatisfactory *ζ* score can either be caused by an inappropriate estimation of the concentration, or of its measurement uncertainty, or both.

Standard measurement uncertainty of the laboratory $u(x_i)$ was obtained by dividing the reported expanded measurement uncertainty by the reported coverage factor, *k*. When no uncertainty was reported, it was set to zero ($u(x_i) = 0$) by the PT coordinator. When *k* was not specified, the reported expanded measurement uncertainty was considered by the PT coordinator as the half-width of a rectangular distribution; $u(x_i)$ was then calculated by dividing this half-width by √3, as recommended by Eurachem [13].

Uncertainty estimation is not trivial, therefore an additional assessment was provided to each laboratory reporting measurement uncertainty, indicating how reasonable has been their measurement uncertainty estimation. Relative standard measurement uncertainty was calculated based on the absolute values for either the assigned values [$u_{rel}(x_{pt}) = (u(x_{pt})/ x_{pt)}$ x 100] and of the reported values [$u_{rel}(x_i) = (u(x_i)/ x_i)$ x 100].

The relative standard measurement uncertainty from the laboratory $u_{rel}(x_i)$ is most likely to fall in a range between a minimum and a maximum allowed uncertainty (case "a": $u_{min,rel} \leq u_{rel}(x_i) \leq u_{max,rel}$). $u_{min,rel}$ is set to the relative standard uncertainties of the assigned values $u_{rel}(x_{pt})$. It is unlikely that a laboratory carrying out the analysis on a routine basis would determine the measurand with a smaller measurement uncertainty than the expert laboratories chosen to establish the assigned value. $u_{max,rel}$ is set to the standard deviation accepted for the PT assessment (σ_{pt}). Consequently, case "a" becomes: $u_{rel}(x_{pt}) \leq u_{rel}(x_i) \leq \sigma_{pt}$.

If $u_{rel}(x_i)$ is smaller than $u_{rel}(x_{pt})$ (case "b") the laboratory may have underestimated its measurement uncertainty. Such a statement has to be taken with care as each laboratory reported only measurement uncertainty, whereas the measurement uncertainty associated with the assigned value also includes contributions for homogeneity and stability of the test item. If those are large, relative measurement uncertainties smaller than $u_{rel}(x_{pt})$ are possible and plausible.

If $u_{rel}(x_i)$ is larger than σ_{pt} (case "c") the laboratory may have overestimated its measurement uncertainty. An evaluation of this statement can be made when looking at the difference between the reported value and the assigned value: if the difference is smaller than the expanded uncertainty $U(x_{pt})$ then overestimation is likely. If the difference is larger but x_i agrees with x_{pt} within their respective expanded measurement uncertainties, then the measurement uncertainty is properly assessed resulting in a satisfactory performance expressed as a ζ score, though the corresponding performance, expressed as a z score, may be questionable or unsatisfactory.

It should be pointed out that "$u_{max,rel}$" is a normative criterion when set by legislation.

6.2 General observations

Twenty six NRLs from twenty four Member States and Switzerland, and eight OCLs (from Germany, Portugal and Italy) registered to the exercise. Four NRLs and two OCL did not report results. Twenty eight laboratories, representing most of the EU Member States (except Netherlands, Sweden, Latvia, Malta and Romania), reported results for the 8 measurands. One laboratory did not report any results for Test item 1, while several laboratories reported "less than" values for the migration experiment (Test item 2).

Most of the participants used HPLC-UV (89 %) or LC-MS/MS (11 %). The experimental details are provided in Annexes 6 to 13 along with all the method parameters and performance characteristics (Annex 14).

6.3 Laboratory results and scorings

6.3.1 Performances

Annexes 6 to 13 present the reported results as tables and figures for the Test item 1 (extraction experiment) and Test item 2 (migration experiment). National Reference Laboratories and Official Control Laboratories are denoted as N-xx and O-xx, respectively.

The corresponding Kernel density plots have been obtained by using the software available from the Statistical Subcommittee of the Analytical Methods Committee of the UK Royal Society of Chemistry [14].

Test item 1 – extraction experiment

Three main analytical techniques have been applied by the laboratories for the extraction of Test item 1: solid-liquid extraction; dissolution with organic solvent; or microwave assisted extraction.

A large scatter of reported results is observed for the four oligomers, ranging from 0 % up to 150 %. The Kernel distribution plots indicate the presence of two (cf. dimer) to three (tri, tetra and pentamer) modes (cf. Annexes 6 to 9). All experimental details related to the extraction with Test item 1 are presented in Annex 15.

- Several laboratories (N-07, N-11, N-13, N-24, N-29, N-31, O-12, O-22 and O-28) reported results leading to D% ranging from – 50 % to + 50 % for the four oligomers.
- Assuming that there were no mistakes in the reporting unit in the calculations of the reported mass fractions, laboratories (e.g. N-02, N-04, N-06, N-08, N-27) having used **mild extraction conditions** (short contact time, ethanol solvent

and/or temperatures below 70 °C) reported very low results, much lower than the assigned value presented in Table 1.

- Significantly higher results for the trimer, tetramer and pentamer were reported by laboratories having performed full dissolution of the test item.

Test item 2 – migration experiment

Figure 1 presents the laboratory performances for the concentrations (mg L^{-1}) of the four measurands investigated in Test item 2. Most of the participants reported results with satisfactory z scores for the PBT cyclic dimer (64 %) and PBT trimer (57 %), while the scores for the PBT cyclic tetramer and pentamer were below 50 %. In all cases, ζ scores were significantly lower compared to z scores.

Many participants (10 out of 28) did not control the temperature during the migration experiment, while 9 out of 28 preheated the FCM cups in different time-temperature combinations. All migration testing parameters are given in Annex 16.

Annexes 10-13 show that most of the reported results are below the assigned values for the four oligomers investigated (see negative z scores). The information extracted from the questionnaire indicates that the oven temperatures used by participants could not guarantee systematically the proper temperature range in the food simulant (70 ± 2 °C) throughout the migration experiment [6,7]. This may explain the lower values observed.

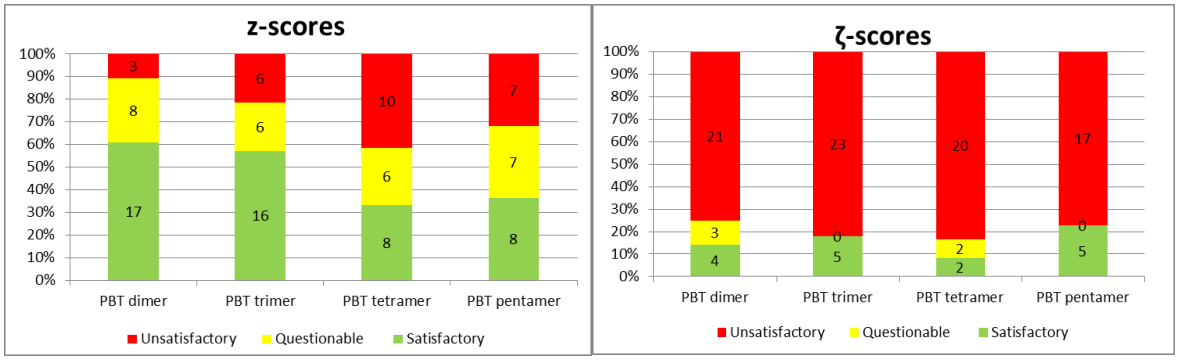

Figure 1: Overview on laboratory performance per measurand according to *z* and ζ scores in Test item 2. Corresponding number of laboratories included in the graph. Satisfactory, questionable and unsatisfactory performances indicated in green, yellow and red, respectively.

6.3.2 Truncated values

"Less than" values were reported by several laboratories for concentrations of the PBT cyclic tetramer and pentamer in migrated solutions from Test item 2. These values corresponded to their limit of detection (LOD) or quantification (LOQ) and could not be included in the data evaluation. However, they were compared with the corresponding "$x_{pt} - U(x_{pt})$". Acceptable "less than" values were reported by laboratories N-03, N-04, N-05 and N-18. Only N-29 and N-02 reported values below the assigned range and these statements are considered as incorrect. The latter two laboratories should have detected the PBT cyclic tetramer and/or pentamer in Test item 2.

6.3.3 Measurement uncertainties

Most of the participants (22 out of 28) routinely report uncertainties for this type of analysis to their customers. Several approaches were used to estimate measurement uncertainties (Table 2). Most of the laboratories derived their uncertainty estimates from measurement replicates.

The majority of laboratories having reported quantitative results provided expanded measurement uncertainties and coverage factors. Figure 2 presents the measurement uncertainty evaluation. Most of the participants reported reasonable measurement uncertainties (Case "a" (green): $u_{rel}(x_{pt}) < u_{rel}(x_i)$).

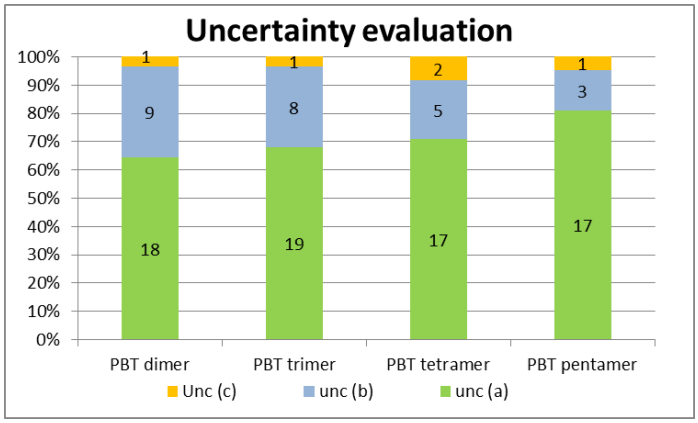

Figure 2: Review of uncertainties reported per measurand result. Corresponding number of laboratories indicated in the graph. Case "a" (green): $u_{rel}(x_{pt}) \leq u_{rel}(x_i) \leq \sigma_{pt}$; Case "b" (blue): $u_{rel}(x_i) < u_{rel}(x_{pt})$; Case "c" (orange): $u_{rel}(x_i) > \sigma_{pt}$

Table 2: Overview of the approaches used to estimate measurement uncertainties (multiple selections were possible).

Approach	Nr of labs
According to ISO-GUM	1
From known uncertainty of a standard method	1
Derived from a single-laboratory validation study	3
Measurement of replicates	15
Estimation based on judgment	4
From Horwitz	3
From Nordtest	1

6.3.4 Additional information extracted from the questionnaire

The questionnaire was answered by all participants and provides valuable information on the laboratories, their way of working and their analytical methods. Annexes 14 presents the analytical technique used and the limits of detection (LOD).

All participants, except one, stated that they have an ISO/IEC 17025 accreditation.

Half of the participants (14 out of 28) did not have experience with the analysis of the four PBT cyclic oligomers investigated. However, all of them stated that they had experience with the analysis of another type of oligomers.

The experimental details presented in Annexes 15 to 16 show technical details of the applied methods for both extraction and migration from FCM, and do not indicate a direct correlation between the applied methods and the performance in the PT study.

The participants were asked to report results in both concentration (mg/L) and in mass fractions (mg/kg). The EURL-FCM derived the densities used by the participants for each measurand (Annex 17). Most of the laboratories used identical densities - the small variations (RSDs) may be attributed to rounding. Several laboratories used a density of 1.0 kg/L. It is likely that laboratory N-09 may have inverted the reported results for the PBT pentamer. A significantly higher density value was obtained for laboratory N-24 (1.36 kg/L).

7 Conclusion

The proficiency test PT-19/01 was organised to assess the analytical capabilities of EU NRLs and OCLs to determine the mass fractions and concentrations of PBT cyclic oligomers (FCM 885) in ground FCM and migrated from FCM at 70 ^{o}C for 2 h, respectively.

A significant scatter of reported results was observed in the frame of the extraction experiment for Test item 1. This may be attributed to the different extraction techniques applied by the participants (ranging from a mild liquid-solid extraction up to the full dissolution of the plastic powder). This indicates that an harmonised extraction protocol may need to be defined in order to ensure comparable results in the frame of Regulation (EC) 10/2011.

Despite the lack of experience in this type of migration analysis for most of the participants, the overall performance of the participants in the determination of these oligomers was satisfactory for the PBT cyclic dimer and trimer, while it was less satisfactory for the PBT cyclic tetramer and pentamer.

Acknowledgements

The twenty eight laboratories listed hereafter are kindly acknowledged for their participation in the PT.

Organisation	Country
AGES Austrian Agency for Health & Food Safety	Austria
Sciensano	Belgium
National Center of Public Health and Analyses	Bulgaria
Croatian Institute of Public Health	Croatia
State General Laboratory, Cyprus	Cyprus
National Institute of Public Health	Czech Republic
National Food Institute, Technical University of Denmark	Denmark
Health Board	Estonia
Finnish Customs Laboratory	Finland
SCL Service Commun des Laboratoires	France
German Federal Institute for Risk Assessment	Germany
Landesbetrieb Hessisches Landeslabor (LHL)	Germany
CVUA-MEL	Germany
Thueringer Landesamt fuer Verbraucherschutz	Germany
Chemisches und Veterinäruntersuchungsamt Stuttgart (CVUA Stuttgart)	Germany
Landesuntersuchungsanstalt für das Gesundheits- und Veterinärwesen Sachsen (LUA)	Germany
General Chemical State Laboratory	Greece
National Food Chain Safety Office Food Chain Safety Laboratory Directorate	Hungary
Public Analyst's Laboratory	Ireland
Istituto zooprofilattico sperimentale Lombardia Emilia Romagna	Italy
National Public Health Surveillance Laboratory	Lithuania
Laboratoire National de Santé	Luxembourg
National Institute of Public Health - National Institute of Hygiene	Poland
Escola Superior de Biotecnologia - Universidade Católica Portuguesa	Portugal
Regional Public Health Authority	Slovakia
National Laboratory of Health, Environment and Food	Slovenia
Centro Nacional Alimentacion (CNA) - AECOSAN	Spain
Kantonales Labor Zürich; NRL-CH	Switzerland

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Annex 1: Invitation letter

EUROPEAN COMMISSION
Joint Research Centre
Directorate F – Health, Consumers & Reference Materials
European Union Reference Laboratory for Food Contact Materials

Ref. Ares(2019)2416374 - 05/04/2019

Ispra, 2 April 2019

(sent by e-mail)

Subject: Invitation to participate in Proficiency Test FCM-19/01 PT round

Dear National Reference Laboratory representative,

On behalf of the EURL for Food Contact Materials (EURL-FCM), we would like to invite you to participate in the Proficiency Test round FCM-19/01 "Determination of PBT cyclic oligomers in and migrated from food contact materials".

The PT fulfils the EURL-FCM mandate under Regulation (EU) 2017/625.

According to Regulation (EU) 2017/625 it is your duty as NRL to participate in PTs organised by the EURL-FCM.

Your participation is free of charge.

Please register electronically by using the link below and following the instructions on screen.

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=2241

Once you have submitted your registration electronically, you will have to:

- Print your registration form, as indicated on screen
- Sign it, date it and send it to us by e-mail (JRC-EURL-FCM@ec.europa.eu)

Please register by Monday the 30th of April 2019.

Please forward this invitation to the Official Control Laboratories (OCLs) in your network that would be interested in participating. They should also register electronically by using the same link above

Samples will be dispatched on the 13th of May 2019.

The deadline for submission of results is the 30th of June 2019.

Do not hesitate to contact us if you have any further questions.

Kind regards,

/signed electronically in Ares/
Dr. E.D. Tsochatzis
FCM-19/01 PT Coordinator

/signed electronically in Ares/
Dr. E.J. Hoekstra
Operating Manager EURL-FCM

Cc: Prof. Dr. H. Emons (Head of Unit, Food & Feed Compliance, F.5)

Annex 2: Test item accompanying letter

EUROPEAN COMMISSION
DIRECTORATE-GENERAL
JOINT RESEARCH CENTRE
Directorate F - Health, Consumers & Reference Materials (Geel/Ispra)
European Union Reference Laboratory for Food Contact Materials

Ispra, 6th May 2019
JRC.F.5/ET/sf/ARES(2019)19-027/3068174

Attn.: «Title» «Firstname» «Surname»
«Organisation»
«Department»
«Address»
«Address2»
«Zip» «Town»
«Country»

Subject: Participation in FCM-19/01 - Determination of PBT cyclic oligomers in and migrated from food contact materials

Dear «Title» «Surname»,

Thank you for participating in the FCM-19/01 proficiency test (PT) for the **"Determination of PBT cyclic oligomers in and migrated from food contact materials".** This PT is organised in support to *Regulation (EU) No. 10/2011 on plastic materials and articles intended to come into contact with food*.

The measurands are mass fractions (mg kg^{-1}) or concentrations (mg L^{-1}) of butylene terephthalate (PBT) cyclic dimer, trimer, tetramer and pentamer.

Please keep this letter. You will need it to report your results.

The parcel you received contains, in addition to this letter:

- test item 1: one bottle containing ground PBT beverage cups for the extraction experiment.
- test item 2: Three (3) PBT cups to be used for migration experiment with food simulant D1 (50 % v/v ethanol:H_2O) at 70 °C for 2 h. These items must be considered as single use FCM. Please follow the General recommendations on the migration protocol reported in EURL-FCM-02-2016 Proficiency Test.
- a calibration solution mixture containing the four target analytes (PBT cyclic dimer to pentamer) at 20 µg mL^{-1} each, in a vial of approx. 1 mL of hexafluoro-2-propanol/ethanol.
- the "Confirmation of receipt" form.

Upon arrival of this parcel, please check whether the bottles and vial are undamaged after transport.

The calibration solution mixture should be stored until analysis in a dark place at a temperature of -18 °C, while test item 1 and 2 can be stored at 20 °C (± 2 °C).

European Commission, Via Enrico Fermi 2749, I-21027 Ispra (Varese) - Italy. Telephone: (39)0332-78-9111.
e-mail: jrc-eurl-fcm@ec.europa.eu
URL: https://ec.europa.eu/jrc/en/eurl/food-contact-materials

Send us or email the "Confirmation of receipt" form within 3 days after receipt of the samples.

The procedure used for the analyses should resemble as closely as possible the one you use in routine analyses.

Please report separately for each item, the following:

1. For test item 1:

- the **mean** of your two or three measurements results (in mg kg^{-1});
- the associated expanded **uncertainty** (in mg kg^{-1});
- the **coverage factor;** and
- the analytical technique used.

2. For test item 2:

- the **mean** of your two or three measurements results (in mg kg^{-1} and mg L^{-1});
- the associated expanded **uncertainty** (in mg kg^{-1} and mg L^{-1});
- the **coverage factor**; and
- the analytical technique used.

The results should be reported in the same format (e.g. number of significant figures) as you normally report to customers.

The homogeneity study was performed with a sample intake of 0.5 g for test item 1 and 100 µL for test item 2 and therefore 0.5 g and 100 µL are the recommended minimum sample intakes for test items 1 and 2 respectively.

The reporting website is https://web.jrc.ec.europa.eu/ilcReportingWeb

To access the webpage you need the following personal password key: **«Part_key»**.

The system will guide you through the reporting procedure. Then complete the corresponding questionnaire. **Do not forget to submit and confirm when required.**

Directly after submitting your results and the questionnaire online, you will be requested to print the completed report form. Please check carefully this report form. In the case mistakes are detected contact the PT coordinator as soon as possible before the reporting deadline.

The deadline for submission of results is **30/06/2019**.

The procedures used for the organisation of PTs are accredited according to ISO/IEC 17043:2010 and guarantee that the identity of the participants and the information provided by them is treated as confidential. However, lab codes of National Reference Laboratories appointed in line with Regulation (EU) 2017/625, will be disclosed to DG SANTE upon request for (long-term) performance assessment. Lab codes of appointed Official Control Laboratories may be disclosed to their National Reference Laboratory upon request.

European Commission, Via Enrico Fermi 2749, I-21027 Ispra (Varese) - Italy. Telephone: (39)0332-78-9111.
e-mail: jrc-eurl-fcm@ec.europa.eu
URL: https://ec.europa.eu/jrc/en/eurl/food-contact-materials

Remember that collusion is contrary to professional scientific conduct and serves only to nullify the benefits of proficiency tests to customers, accreditation bodies and analysts alike.

Your participation in this project is greatly appreciated.

Do not hesitate to contact me for further information.

With kind regards,

/signed electronically in Ares/

Dr. Emmanouil Tsochatzis
FCM-19/01 Coordinator

Cc: H. Emons (Head of Unit, Food & Feed Compliance, F.5),
E. Hoekstra (Operating Manager EURL-FCM)
P. Dehouck (FCM-19/01 Deputy PT Coordinator)

European Commission, Via Enrico Fermi 2749, I-21027 Ispra (Varese) - Italy. Telephone: (39)0332-78-9111.
e-mail: jrc-eurl-fcm@ec.europa.eu
URL: https://ec.europa.eu/jrc/en/eurl/food-contact-materials

Annex 3: Confirmation of receipt form

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Directorate F – Health, Consumers and Reference Materials
European Union Reference Laboratory for Food Contact Materials

Ispra, 06th May 2019
JRC.F.5/ET/sf/ARES(2019)19-026/3063060

Attn.: «Title» «Firstname» «Surname»
«Organisation»
«Department»
«Address»
«Zip» «Town»
«Country»

Subject: "Confirmation receipt" form - FCM-19/01 - Determination of PBT cyclic oligomers in and migrated from food contact materials

Please return this form at your earliest convenience, to confirm that the package arrived well to your laboratory. If samples are damaged, please mention it below and contact us as soon as possible.

Date of package arrival _____

Were the samples damaged? ☐ YES ☐ NO

Remarks _____
_____

Signature

Thank you for returning this form by email to:

/signed electronically in Ares/

Dr. Emmanouil Tsochatzis
FCM-19/01 Coordinator
e-mail : jrc-eurl-fcm@ec.europa.eu

European Commission, Via Enrico Fermi 2749, I-21027 Ispra (Varese) - Italy. Telephone: (39)0332-78-9111.
e-mail: jrc-eurl-fcm@ec.europa.eu
URL: https://ec.europa.eu/jrc/en/eurl/food-contact-materials

Annex 4: Questionnaire

Milc questionnaire

Comparison for FCM-19/01

Submission Form

1. **Are you a National Reference Laboratory?**

- ○ A) Yes
- ○ B) No

2. **Does your laboratory have a quality management system?**

- ○ A) Yes
- ○ B) No

3. **If yes based on which standard?**

- ☐ ISO 17025
- ☐ ISO 9001
- ☐ other

3.1. If "other" specify which one.

4. **What was the basis for your measurement uncertainty evaluation?**

See table **Measurement uncertainty evaluation** at bottom

5. **Do you provide an uncertainty statement to your customers?**

- ◯ A) Yes
- ◯ B) No

6. **Please specify the laboratory experience in the analysis of oligomers (samples/year).**

See table **Laboratory experience (samples / year)** at bottom

7. **Test item 1**

7.1. Is your laboratory experienced in the quantification of mass fractions resulting from the extraction of food contact materials?

- ◯ A) Yes
- ◯ B) No

7.1.1. If yes, please identify the food contact materials and extracted analytes. *

7.2. Please provide details of the used analytical method.

See table **Analytical method details** at bottom

7.3. Was this analytical method a (thick what is relevant):

- ☐ 1) Standard method
- ☐ 2) Validated method
- ☐ 3) Accredited method

7.4. Specify the limit of detection (LOD) for the measured analytes.

See table **Limit of Detection (LOD)** at bottom

7.5. How did you extract the ground material?

- ☐ 1) Liquid-solid extraction
- ☐ 2) Soxhlet
- ☐ 3) Accelerated Solvent Extraction/Pressurised Liquid Extraction (ASE/PLE)
- ☐ 4) Microwave Assisted Extraction (MAE)
- ☐ 5) Other

7.6. Please provide the extraction conditions that you applied.

See table **Extraction conditions** at bottom

8. **Test item 2**

8.1. Was the analytical method used for test item 2 the same as for test item 1?

- ○ a) Yes
- ○ b) No

8.1.1. If no, please provide the details of the analytical method used for test item 2. *

See table **Analytical method details test item 2** at bottom

8.2. What was the volume (in mL) of Simulant D1 used for the migration experiment?

8.3. Please provide the temperature (in °C) of the oven used for the migration experiment.

8.4. Did you preheat the PBT cups before the migration?

- a) Yes
- b) No

8.4.1. If yes, please provide us with the exact preheating temperature and time. *

8.5. Did you control the Simulant D1 temperature during migration?

- a) Yes
- b) No

8.5.1. If yes, please specify how you controlled the temperature. *

- 1) Calibrated thermometer
- 2) Calibrated datalogger
- 3) Non-calibrated thermometer
- 4) Non-calibrated datalogger
- 5) Other

8.5.1.1. If other, please specify.

8.6. Did you measure the Simulant D1 losses after migration?

- a) Yes
- b) No

8.6.1. If yes, please specify the measured volume. *

8.7. What conversion factor did you use for the conversion from mg/kg to mg/L?

Analytical method details

Questions/Response table	*stationary phase*	*mobile phase*	*injection volume*	*detection*
Give the method details				

Analytical method details test item 2

Questions/Response table	*stationary phase*	*mobile phase*	*injection volume*	*detection*
Give the method details				

Extraction conditions

Questions/Response table	*Time*	*Temperature*	*Solvents*
Extraction conditions			

Laboratory experience (samples / year)

Questions/Response table	*1) None (0)*	*2) 1-50*	*3) 51-250*	*4) 251-1000*	*5) > 1000*
Indicate experience with "X"					

Limit of Detection (LOD)

Questions/Response table	*LOD*
1) butylene terephthalate cyclic dimer	
2) butylene terephthalate cyclic trimer	
3) butylene terephthalate cyclic tetramer	
4) butylene terephthalate cyclic pentamer	

Measurement uncertainty evaluation

Questions/Response table	*Indicate with "X"*
a) Uncertainty budget (ISO GUM)	
b) Known uncertainty of standard method (ISO 21748)	
c) From in-house method validation	
d) Measurement of replicates (precision)	

Questions/Response table	*Indicate with "X"*
e) Evaluation based on judgment	
f) From interlaboratory comparison	
g) Other (please specify)	

Annex 5: Homogeneity and stability results

5.1 Homogeneity study for test items

Test item 1 (all values in mg kg^{-1})								
Bottle ID	**PBT Dimer**		**PBT Trimer**		**PBT Tetramer**		**PBT Trimer**	
	R_1	**R_2**	**R_1**	**R_2**	**R_1**	**R_2**	**R_1**	**R_2**
1	3548	3519	1393	1397	686	681	407	403
2	3460	3480	1370	1374	655	651	399	387
3	3714	3690	1466	1462	677	677	412	420
4	3533	3543	1401	1389	655	664	387	405
5	3710	3737	1466	1478	695	686	416	424
6	3505	3491	1381	1389	655	651	411	403
7	3733	3737	1451	1475	699	686	411	415
8	3557	3538	1421	1401	651	655	407	403
9	3605	3586	1436	1424	673	681	406	399
10	3618	3605	1451	1428	668	655	407	407
Mean	3596		1422		670		407	
s_x	95		36		16		8	
s_w	13		10		6		7	
s_s	95		35		15		7	
σ_{pt} (10 %)	360		142		67		41	
0.3 * σ_{pt}	108		43		20		12	
$s_s \leq 0.3 * \sigma_{pt}$	passed		passed		passed		passed	
Assessment	Homogeneous		Homogeneous		Homogeneous		Homogeneous	

Test item 2 (all values in mg L^{-1})								
Bottle ID	**PET Dimer**		**PBT Dimer**		**PET Trimer**		**PBT Trimer**	
	R_1	**R_2**	**R_1**	**R_2**	**R_1**	**R_2**	**R_1**	**R_2**
1	2.39	2.39	0.367	0.367	0.038	0.038	0.025	0.025
2	2.27	2.29	0.362	0.360	0.039	0.039	0.025	0.025
3	2.20	2.19	0.358	0.345	0.036	0.037	0.025	0.025
4	2.29	2.32	0.362	0.366	0.037	0.037	0.025	0.025
5	2.19	2.22	0.350	0.349	0.037	0.036	0.025	0.025
6	2.15	2.12	0.336	0.341	0.036	0.036	0.023	0.024
7	2.27	2.25	0.357	0.354	0.037	0.038	0.025	0.025
8	2.23	2.24	0.355	0.353	0.038	0.037	0.025	0.025
9	2.22	2.23	0.352	0.348	0.037	0.037	0.025	0.025
10	2.11	2.12	0.338	0.339	0.036	0.037	0.024	0.023
Mean	2.24		0.353		0.0370		0.0245	
s_x	0.08		0.010		0.0009		0.0006	
s_w	0.01		0.003		0.0006		0.0002	
s_s	0.08		0.009		0.0007		0.0006	
σ_{pt} (20 %)	0.45		0.071		0.0074		0.0049	
0.3 * σ_{pt}	0.13		0.021		0.0022		0.0015	
$s_s \leq 0.3 * \sigma_{pt}$	passed		passed		passed		passed	
Assessment	Homogeneous		Homogeneous		Homogeneous		Homogeneous	

Where: σ_{pt} is the standard deviation for the PT assessment,
s_x is the standard deviation of the sample averages,
s_w is the within-sample standard deviation,
s_s is the between-sample standard deviation,

5.2 Stability study (at 20 °C, time in weeks (w)

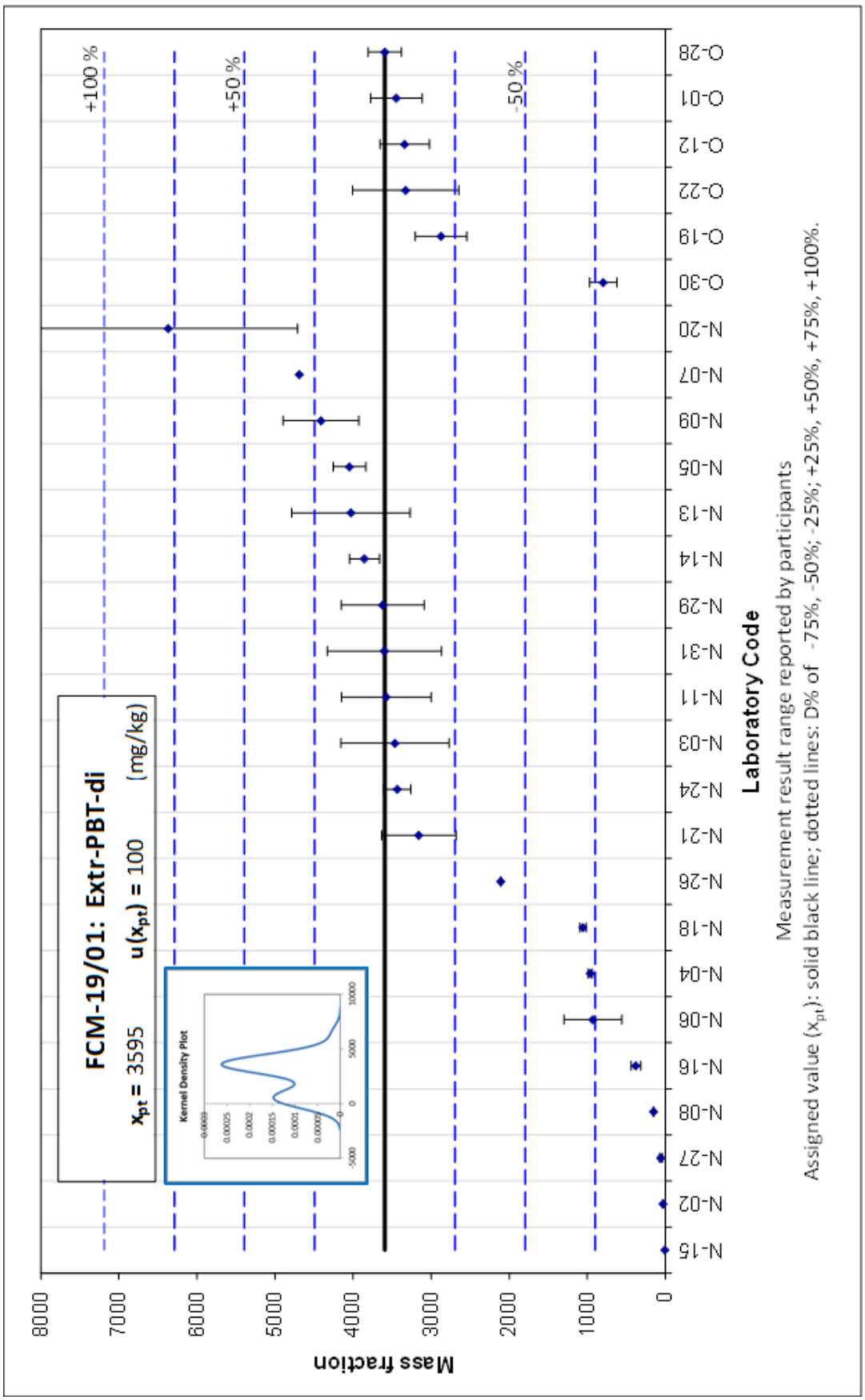
Test item 1 (all values in mg kg^{-1})					
Analyte	**Time (9 w), 20 °C**			**Stability criteria [a]**	**Assessment**
	Bottle ID	**Replicate 1**	**Replicate 2**		
PET Dimer	**1**	3553	3532	Passed	Stable
	2	3486	3486		
PBT Dimer	**1**	1403	1403	Passed	Stable
	2	1376	1381		
PET Trimer	**1**	671	671	Passed	Stable
	2	655	651		
PBT Trimer	**1**	396	399	passed	Stable
	2	404	405		
Test item 2 (all values in mg L^{-1})					
Analyte	**Time (9 w), 20 °C**			**Stability criteria [a]**	**Assessment**
	Bottle ID	**Replicate 1**	**Replicate 2**		
PBT Dimer	**1**	2.22	2.23	Passed	Stable
	2	2.14	2.12		
PBT Trimer	**1**	0.352	0.351	Passed	Stable
	2	0.337	0.340		
PET Tetramer	**1**	0.0373	0.0378	Passed	Stable
	2	0.0361	0.0365		
PBT Pentamer	**1**	0.0245	0.0245	passed	Stable
	2	0.0235	0.0235		

[a] Stability criteria according to ISO 13528:2015 § B.5.

Annex 6: Results for PBT cyclic dimer in Test item 1

Assigned range: x_{pt} = 3595 ± 201 *(k = 2)*; all values in mg kg^{-1}

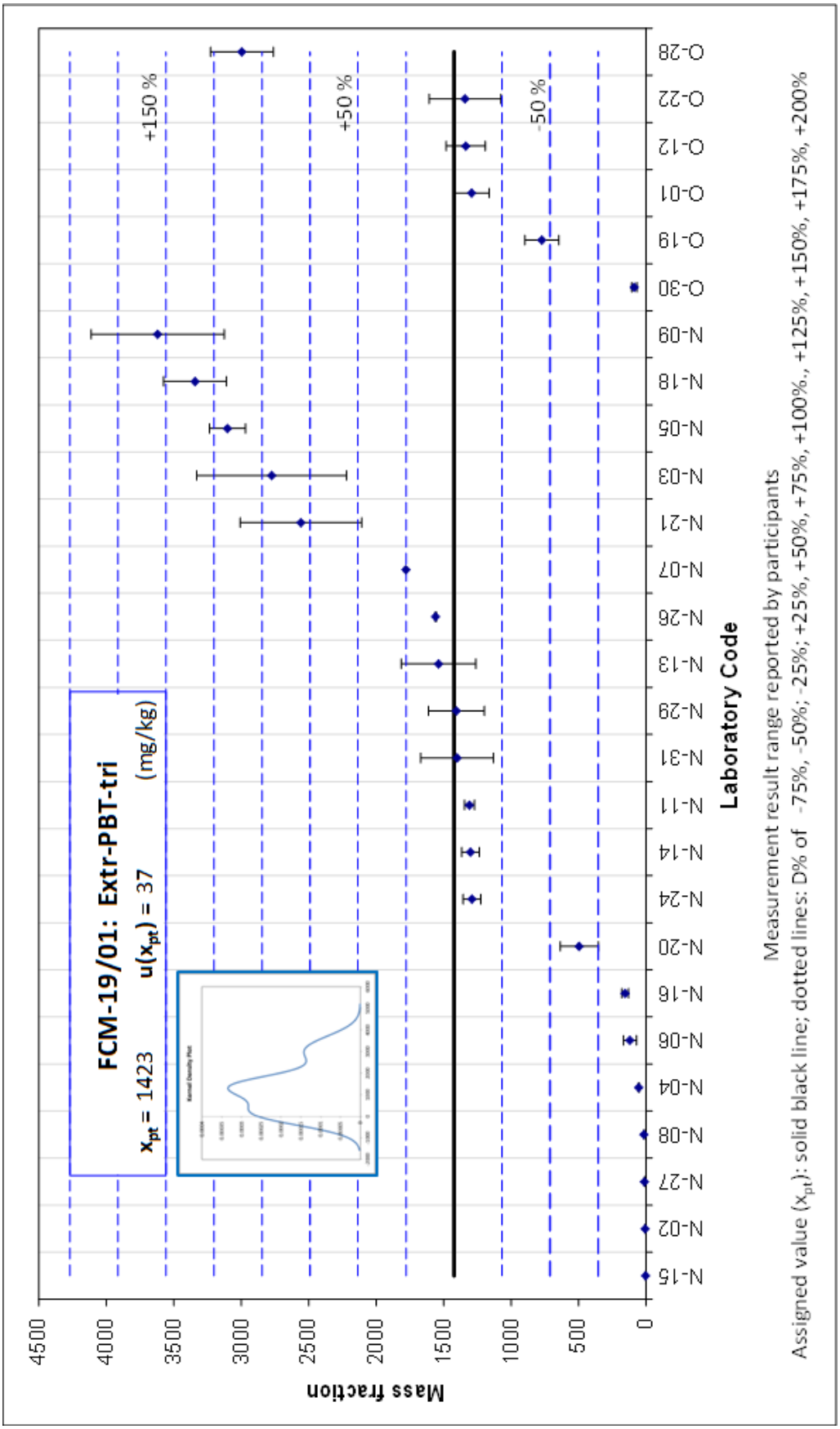
Code	x_i	$U(x_i)$	k	Technique	$D\%$
N-02	29.45	1.47	2	HPLC-UV	-99%
N-03	3463	693	2	HPLC-UV	-4%
N-04	962	28		HPLC-UV	-73%
N-05	4046	208	2	HPLC-UV	13%
N-06	930	370	2	HPLC-UV	-74%
N-07	4690			LC-MS/MS	30%
N-08	152.4	5.8	2	HPLC-UV	-96%
N-09	4411	485	2	HPLC-UV	23%
N-11	3574.7	575	2	HPLC-UV	-1%
N-13	4027.5	756.2	2	HPLC-DAD	12%
N-14	3855	193	2	HPLC-DAD	7%
N-15	5.67	0.82	2	HPLC-UV	-100%
N-16	379.1	62.9	2	HPLC-UV	-89%
N-17	--				
N-18	1056	38	2	HPLC-UV	-71%
N-20	6366	1655	2	HPLC-UV	77%
N-21	3160	477	2	HPLC-UV	-12%
N-24	3435	172	2	HPLC-UV	-4%
N-26	2109	4	2	HPLC-DAD	-41%
N-27	60.2	17	3	LC-MS/MS	-98%
N-29	3620	532	2	HPLC-UV	1%
N-31	3600	730	2	HPLC-UV	0%
O-01	3446	330	2	HPLC-UV	-4%
O-12	3341	315	2	HPLC-UV	-7%
O-19	2874	331	2	HPLC-UV	-20%
O-22	3327	680		HPLC-UV	-7%
O-28	3594	212	2	HPLC-UV	0%
O-30	795.96	175.11	2	LC-MS/MS	-78%


FCM-19/01: Extr-PBT-di
x_{pt} = 3595 $u(x_{pt})$ = 100 (mg/kg)
Kernel Density Plot
+100 %
+50 %
-50 %
Mass fraction
Laboratory Code
Measurement result range reported by participants
Assigned value (x_{pt}): solid black line; dotted lines: D% of -75%, -50%; -25%; +25%, +50%, +75%, +100%.

Annex 7: Results for PBT cyclic trimer in Test item 1

Assigned range: x_{pt} = 1423 ± 74 *(k = 2)*; all values in mg kg^{-1}.

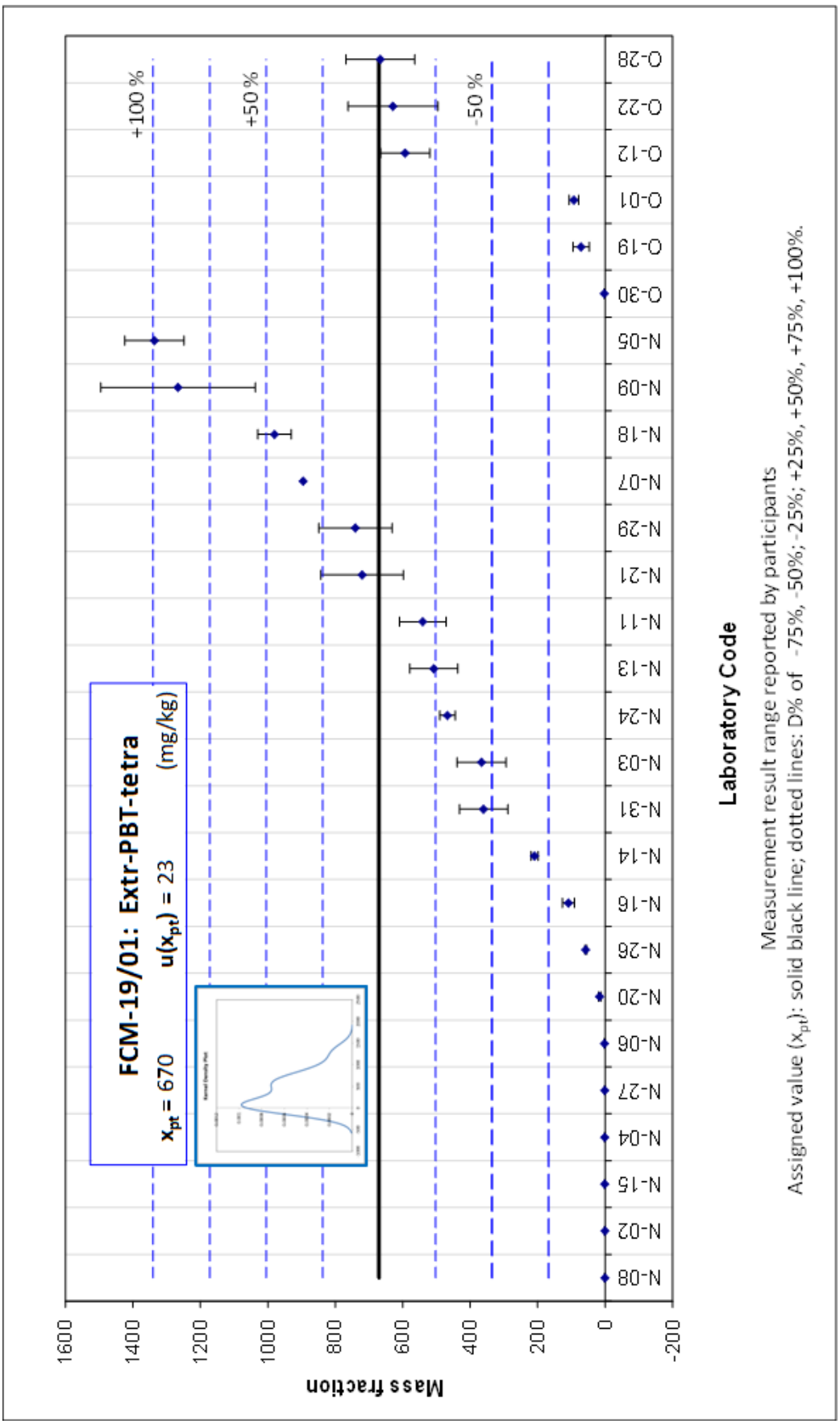
Code	x_i	$U(x_i)$	k	Technique	$D\%$
N-02	5.56	0.55	2	HPLC-UV	-100%
N-03	2775	555	2	HPLC-UV	95%
N-04	53.3	3.4		HPLC-UV	-96%
N-05	3103	134	2	HPLC-UV	118%
N-06	120	49	2	HPLC-UV	-92%
N-07	1780			LC-MS/MS	25%
N-08	16.1	1	2	HPLC-UV	-99%
N-09	3620	493	2	HPLC-UV	154%
N-11	1308.2	37.8	2	HPLC-UV	-8%
N-13	1538.4	277.2	2	HPLC-DAD	8%
N-14	1302	65	2	HPLC-DAD	-9%
N-15	2.53	0.35	2	HPLC-UV	-100%
N-16	154.9	25.7	2	HPLC-UV	-89%
N-17	--				
N-18	3343	232	2	HPLC-UV	135%
N-20	495	141	2	HPLC-UV	-65%
N-21	2558	450	2	HPLC-UV	80%
N-24	1290	65	2	HPLC-UV	-9%
N-26	1560	4	2	HPLC-DAD	10%
N-27	11.2	2	3	LC-MS/MS	-99%
N-29	1405	207	2	HPLC-UV	-1%
N-31	1400	270	2	HPLC-UV	-2%
O-01	1292	130	2	HPLC-UV	-9%
O-12	1337	145	2	HPLC-UV	-6%
O-19	773	126	2	HPLC-UV	-46%
O-22	1342	267		HPLC-UV	-6%
O-28	2995	233	2	HPLC-UV	110%
O-30	87.58	19.26	2	LC-MS/MS	-94%


FCM-19/01: Extr-PBT-tri
x_{pt} = 1423 $u(x_{pt})$ = 37 (mg/kg)
+150 %
+50 %
-50 %
Mass fraction
Laboratory Code
Measurement result range reported by participants
Assigned value (x_{pt}): solid black line; dotted lines: D% of -75%, -50%; -25%; +25%, +50%, +75%, +100%., +125%, +150%, +175%, +200%

Annex 8: Results for PBT cyclic tetramer in Test item 1

Assigned range: x_{pt} = 670 ± 47 *(k = 2)*; all values in mg kg^{-1}

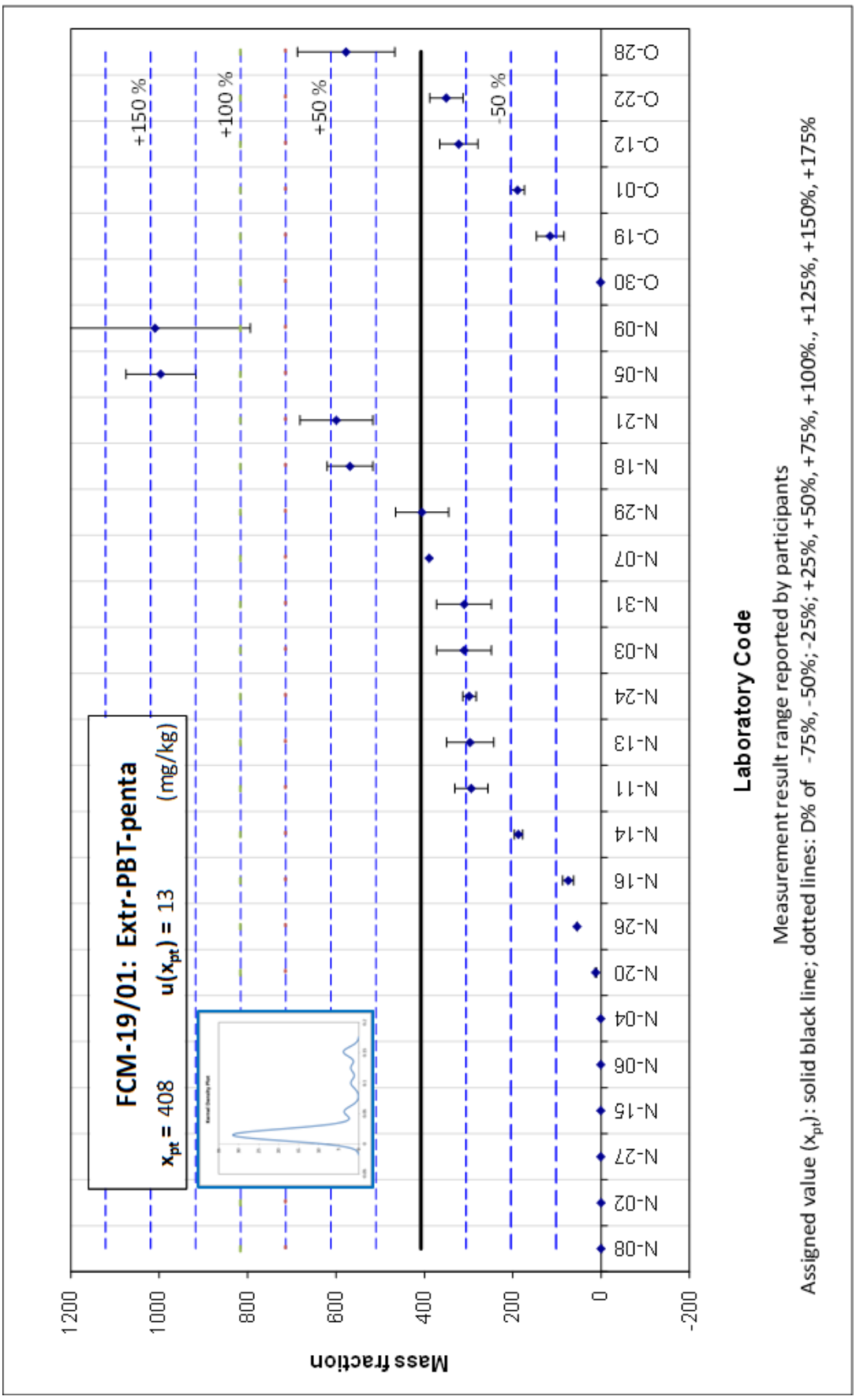
Code	x_i	$U(x_i)$	k	Technique	$D\%$
N-02	0.39	0.04	2	HPLC-UV	-100%
N-03	366	73	2	HPLC-UV	-45%
N-04	0.59	0.04		HPLC-UV	-100%
N-05	1336	88	2	HPLC-UV	99%
N-06	1.63	0.65	2	HPLC-UV	-100%
N-07	895			LC-MS/MS	34%
N-08	0	0.46	2	HPLC-UV	-100%
N-09	1266	229	2	HPLC-UV	89%
N-11	540.5	69.5	2	HPLC-UV	-19%
N-13	507.7	70.9	2	HPLC-DAD	-24%
N-14	209.1	10.5	2	HPLC-DAD	-69%
N-15	0.46	0.19	2	HPLC-UV	-100%
N-16	108.5	18	2	HPLC-UV	-84%
N-17	--				
N-18	980	50	2	HPLC-UV	46%
N-20	16	3	2	HPLC-UV	-98%
N-21	720	122	2	HPLC-UV	7%
N-24	467	23	2	HPLC-UV	-30%
N-26	57.2	2	2	HPLC-DAD	-91%
N-27	0.72	0.04	3	LC-MS/MS	-100%
N-29	740	109	2	HPLC-UV	10%
N-31	360	72	2	HPLC-UV	-46%
O-01	92.3	14.5	2	HPLC-UV	-86%
O-12	593	73	2	HPLC-UV	-11%
O-19	71	24	2	HPLC-UV	-89%
O-22	629	133		HPLC-UV	-6%
O-28	666	102	2	HPLC-UV	-1%
O-30	2.13	0.47	2	LC-MS/MS	-100%


FCM-19/01: Extr-PBT-tetra
x_{pt} = 670
$u(x_{pt})$ = 23
(mg/kg)
+100 %
+50 %
-50 %
Mass fraction
1600
1400
1200
1000
800
600
400
200
0
-200
N-08
N-02
N-15
N-04
N-27
N-06
N-20
N-26
N-16
N-14
N-31
N-03
N-24
N-13
N-11
N-21
N-29
N-07
N-18
N-09
N-05
O-30
O-19
O-01
O-12
O-22
O-28
Laboratory Code
Measurement result range reported by participants
Assigned value (x_{pt}): solid black line; dotted lines: D% of -75%, -50%; -25%; +25%, +50%, +75%, +100%.

Annex 9: Results for PBT cyclic pentamer in Test item 1

Assigned range: x_{pt} = 408 ± 26 *(k = 2)*; all values in mg kg^{-1}.

Code	x_i	$U(x_i)$	k	Technique	$D\%$
N-02	0.23	0.03	2	HPLC-UV	-100%
N-03	310	62	2	HPLC-UV	-24%
N-04	0.61	0.02		HPLC-UV	-100%
N-05	996	79	2	HPLC-UV	144%
N-06	0.44	0.18	2	HPLC-UV	-100%
N-07	389			LC-MS/MS	-5%
N-08	0	0.45	2	HPLC-UV	-100%
N-09	1009	215	2	HPLC-UV	147%
N-11	293.5	37.2	2	HPLC-UV	-28%
N-13	296.3	53.4	2	HPLC-DAD	-27%
N-14	186.8	9.3	2	HPLC-DAD	-54%
N-15	0.42	0.09	2	HPLC-UV	-100%
N-16	74.2	12.3	2	HPLC-UV	-82%
N-18	568	52	2	HPLC-UV	39%
N-20	12	2	2	HPLC-UV	-97%
N-21	599	83	2	HPLC-UV	47%
N-24	298	15	2	HPLC-UV	-27%
N-26	54.3	0.5	2	HPLC-DAD	-87%
N-27	0.41	0.03	3	LC-MS/MS	-100%
N-29	405	60	2	HPLC-UV	-1%
N-31	310	62	2	HPLC-UV	-24%
O-01	189	16	2	HPLC-UV	-54%
O-12	322	43	2	HPLC-UV	-21%
O-19	115	31	2	HPLC-UV	-72%
O-22	350	38		HPLC-UV	-14%
O-28	577	110	2	HPLC-UV	42%
O-30	1.34	0.29	2	LC-MS/MS	-100%


FCM-19/01: Extr-PBT-penta
x_{pt} = 408 $u(x_{pt})$ = 13 (mg/kg)
+150 %
+100 %
+50 %
-50 %
Mass fraction
1200
1000
800
600
400
200
0
-200
O-28
O-22
O-12
O-01
O-19
O-30
N-09
N-05
N-21
N-18
N-29
N-07
N-31
N-03
N-24
N-13
N-11
N-14
N-16
N-26
N-20
N-04
N-06
N-15
N-27
N-02
N-08
Laboratory Code
Measurement result range reported by participants
Assigned value (x_{pt}): solid black line; dotted lines: D% of -75%, -50%; -25%; +25%, +50%, +75%, +100%., +125%, +150%, +175%

Annex 10: Results for PBT cyclic dimer in Test item 2

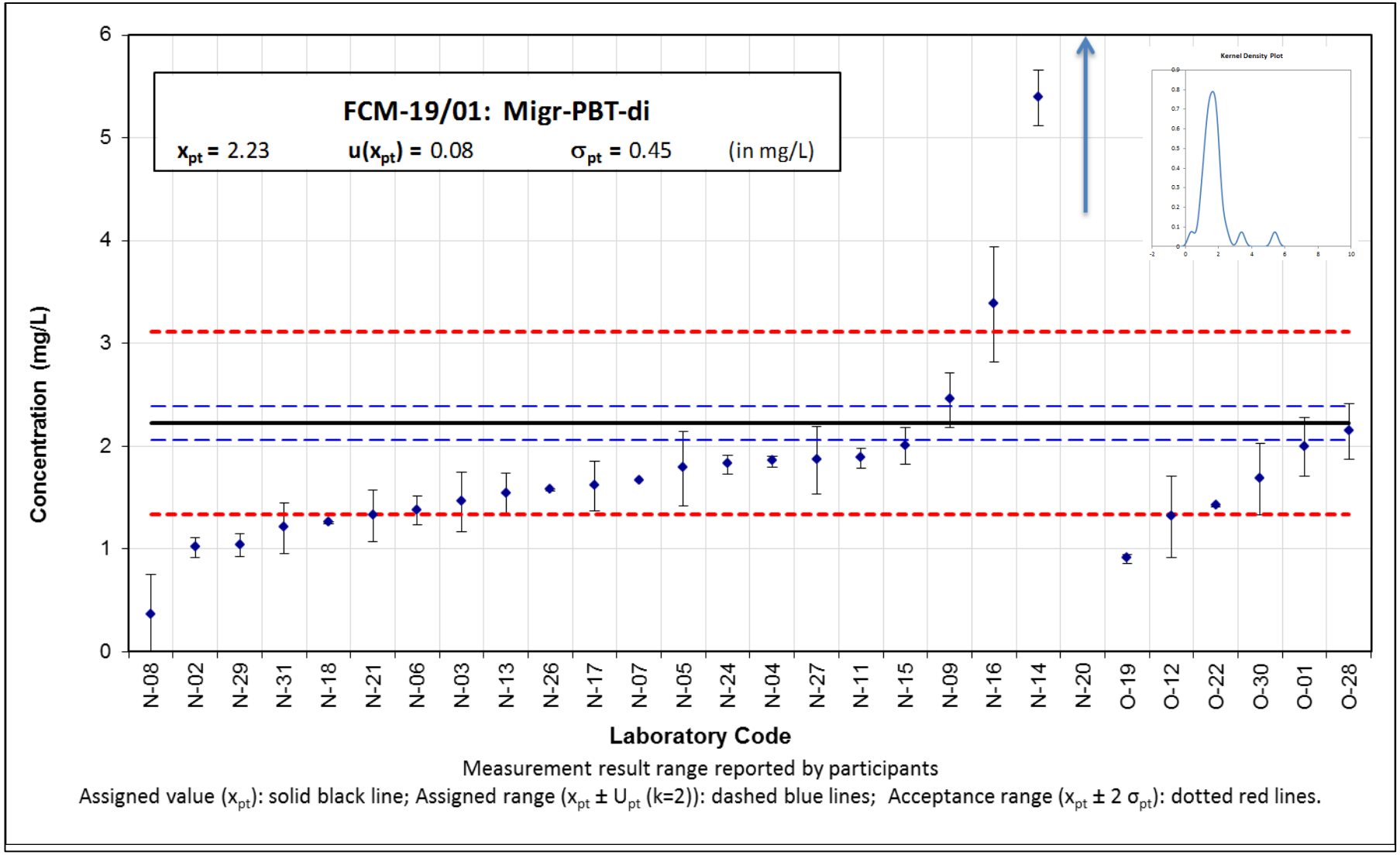
Assigned range: x_{pt} = 2.23 ± 0.16 $U(x_{pt}, k = 2.0)$; σ_{pt} = 0.45 (all values in mg L^{-1})

Code	x_i	$U(x_i)$	k	Technique	$u(x_i)$	z score	ζ score	unc.
N-02	1.01	0.1	2	HPLC-UV	0.050	-2.7	-12.7	a
N-03	1.46	0.29	2	HPLC-UV	0.145	-1.7	-4.6	a
N-04	1.848	0.055	√3	HPLC-UV	0.032	-0.8	-4.3	b
N-05	1.782	0.362	2	HPLC-UV	0.181	-1.0	-2.24	a
N-06	1.37	0.14	2	HPLC-UV	0.070	-1.9	-8.0	a
N-07	1.66			LC-MS/MS	0.000	-1.3	-7.0	b
N-08	0.35	0.4	2	HPLC-UV	0.200	-4.2	-8.7	c
N-09	2.4462	0.2688	2	HPLC-UV	0.134	0.5	1.4	a
N-11	1.88	0.1	2	HPLC-UV	0.050	-0.8	-3.6	b
N-13	1.53	0.21	2	HPLC-DAD	0.105	-1.6	-5.2	a
N-14	5.39	0.27	2	HPLC-DAD	0.135	7.1	20.1	b
N-15	2	0.18	2	HPLC-UV	0.090	-0.5	-1.9	a
N-16	3.38	0.56	2	HPLC-UV	0.280	2.6	4.0	a
N-17	1.61	0.24	2	HPLC-UV	0.120	-1.4	-4.3	a
N-18	1.25	0.0097	2	HPLC-PDA	0.005	-2.2	-12.0	b
N-20	1727	371	2	HPLC-UV	185.5	3873.4	9.3	a
N-21	1.319	0.2516	2	HPLC-UV	0.126	-2.04	-6.1	a
N-24	1.82	0.091	2	HPLC-UV	0.046	-0.9	-4.4	b
N-26	1.571	0.007	2	HPLC-DAD	0.004	-1.5	-8.0	b
N-27	1.86	0.33	3	LC-MS/MS	0.110	-0.8	-2.7	a
N-29	1.034	0.113	2	HPLC-UV	0.057	-2.7	-12.0	a
N-31	1.2	0.25	2	HPLC-UV	0.125	-2.3	-6.9	a
O-01	1.99	0.287	2	HPLC-UV	0.144	-0.5	-1.4	a
O-12	1.31	0.4	2	HPLC-UV	0.200	-2.1	-4.2	a
O-19	0.903	0.0435	2	HPLC-UV	0.022	-2.97	-15.7	b
O-22	1.421	0.01	√3	HPLC-UV	0.006	-1.8	-9.9	b
O-28	2.139	0.269	2	HPLC-UV	0.135	-0.2	-0.6	a
O-30	1.68	0.35	2	LC-MS/MS	0.175	-1.2	-2.8	a

[a] √3 is set by the PT coordinator when no coverage factor *k* is reported. The reported uncertainty was assumed to have a rectangular distribution with *k* = √3,

[b] Performance: satisfactory, questionable, unsatisfactory,

[c] a: $u_{min,rel}(u_{rel}(x_{pt,})) \leq u_{rel}(x_i) \leq u_{max,rel}(\sigma_{pt})$; b: $u_{rel}(x_i) < u_{min,rel}(u_{rel}(x_{pt,}))$; and c: $u_{rel}(x_i) > u_{max,rel}(\sigma_{pt})$



Measurement result range reported by participants

Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 11: Results for PBT cyclic trimer in Test item 2

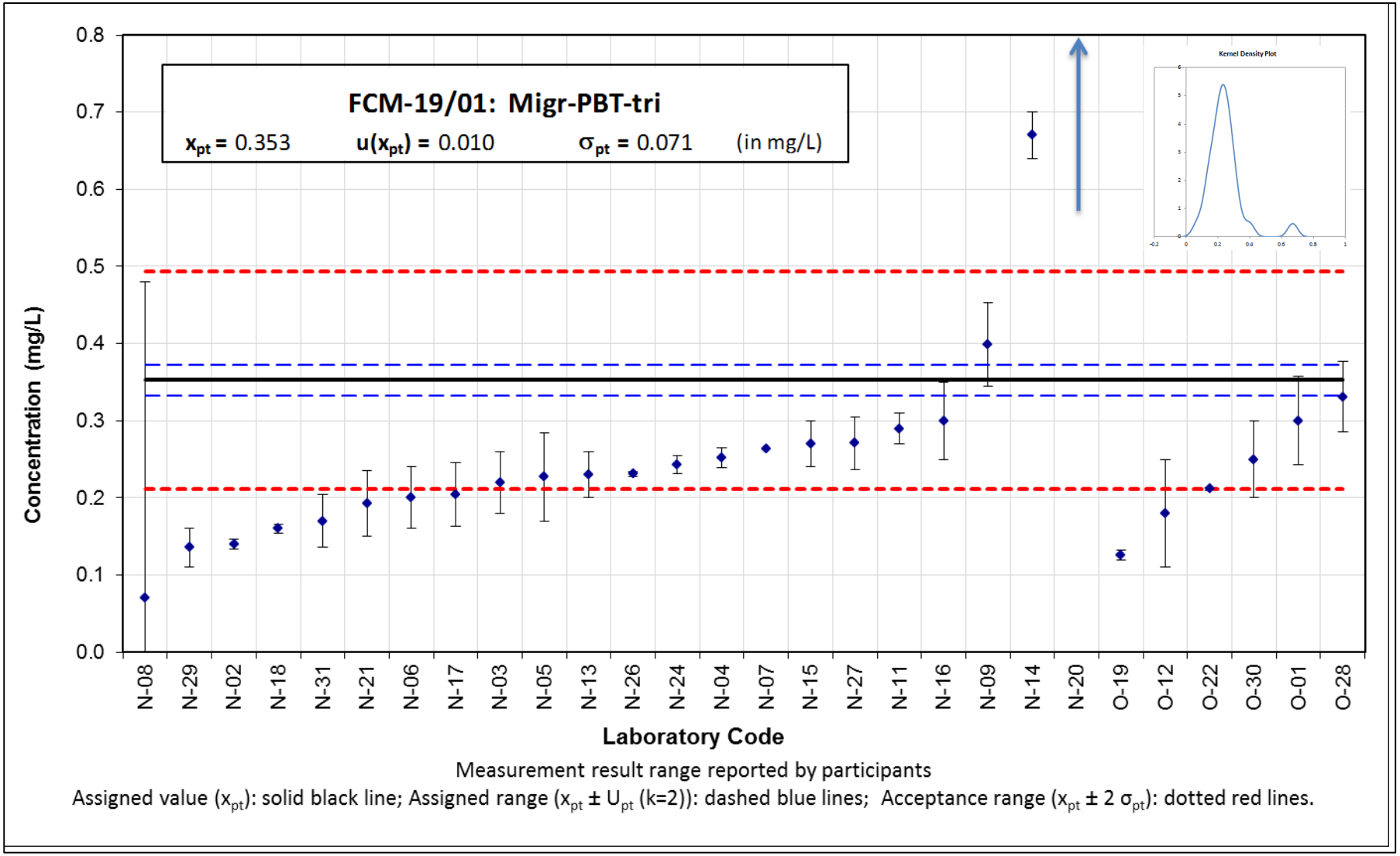
Assigned range: x_{pt} = 0.353 ± 0.020 $U(x_{pt}, k = 2.0)$; σ_{pt} = 0.071 (all values in mg L^{-1})

Code	x_i	$U(x_i)$	k	Technique	$u(x_i)$	z score	ζ score	unc.
N-02	0.14	0.007	2	HPLC-UV	0.004	-3.0	-20.3	b
N-03	0.22	0.04	2	HPLC-UV	0.020	-1.9	-6.0	a
N-04	0.252	0.013	√3	HPLC-UV	0.008	-1.4	-8.1	a
N-05	0.227	0.057	2	HPLC-UV	0.029	-1.8	-4.2	a
N-06	0.2	0.04	2	HPLC-UV	0.020	-2.2	-6.8	a
N-07	0.263			LC-MS/MS	0.000	-1.3	-9.1	b
N-08	0.07	0.41	2	HPLC-UV	0.205	-4.0	-1.4	c
N-09	0.3987	0.0542	2	HPLC-UV	0.027	0.7	1.6	a
N-11	0.29	0.02	2	HPLC-UV	0.010	-0.9	-4.5	a
N-13	0.23	0.03	2	HPLC-DAD	0.015	-1.7	-6.8	a
N-14	0.67	0.03	2	HPLC-DAD	0.015	4.5	17.7	b
N-15	0.27	0.03	2	HPLC-UV	0.015	-1.2	-4.6	a
N-16	0.3	0.05	2	HPLC-UV	0.025	-0.7	-1.96	a
N-17	0.204	0.041	2	HPLC-UV	0.021	-2.1	-6.5	a
N-18	0.16	0.0058	2	HPLC-PDA	0.003	-2.7	-18.8	b
N-20	512	79	2	HPLC-UV	39.50	7253.2	13.0	a
N-21	0.1926	0.0428	2	HPLC-UV	0.021	-2.3	-6.8	a
N-24	0.243	0.0121	2	HPLC-UV	0.006	-1.6	-9.5	b
N-26	0.231	0.003	2	HPLC-DAD	0.002	-1.7	-12.2	b
N-27	0.271	0.034	3	LC-MS/MS	0.011	-1.2	-5.4	a
N-29	0.136	0.025	2	HPLC-UV	0.013	-3.1	-13.6	a
N-31	0.17	0.034	2	HPLC-UV	0.017	-2.6	-9.3	a
O-01	0.3	0.057	2	HPLC-UV	0.029	-0.7	-1.7	a
O-12	0.18	0.07	2	HPLC-UV	0.035	-2.4	-4.7	a
O-19	0.126	0.006	2	HPLC-UV	0.003	-3.2	-22.0	b
O-22	0.212	0.002	√3	HPLC-UV	0.001	-2.0	-14.2	b
O-28	0.331	0.046	2	HPLC-UV	0.023	-0.3	-0.9	a
O-30	0.25	0.05	2	LC-MS/MS	0.025	-1.5	-3.8	a

[a] √3 is set by the PT coordinator when no coverage factor *k* is reported. The reported uncertainty was assumed to have a rectangular distribution with *k* = √3,

[b] Performance: satisfactory, questionable, unsatisfactory,

[c] a: $u_{min,rel}(u_{rel}(x_{pt,})) \leq u_{rel}(x_i) \leq u_{max,rel}(\sigma_{pt})$; b: $u_{rel}(x_i) < u_{min,rel}(u_{rel}(x_{pt,}))$; and c: $u_{rel}(x_i) > u_{max,rel}(\sigma_{pt})$



Measurement result range reported by participants

Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 12: Results for PBT cyclic tetramer in Test item 2

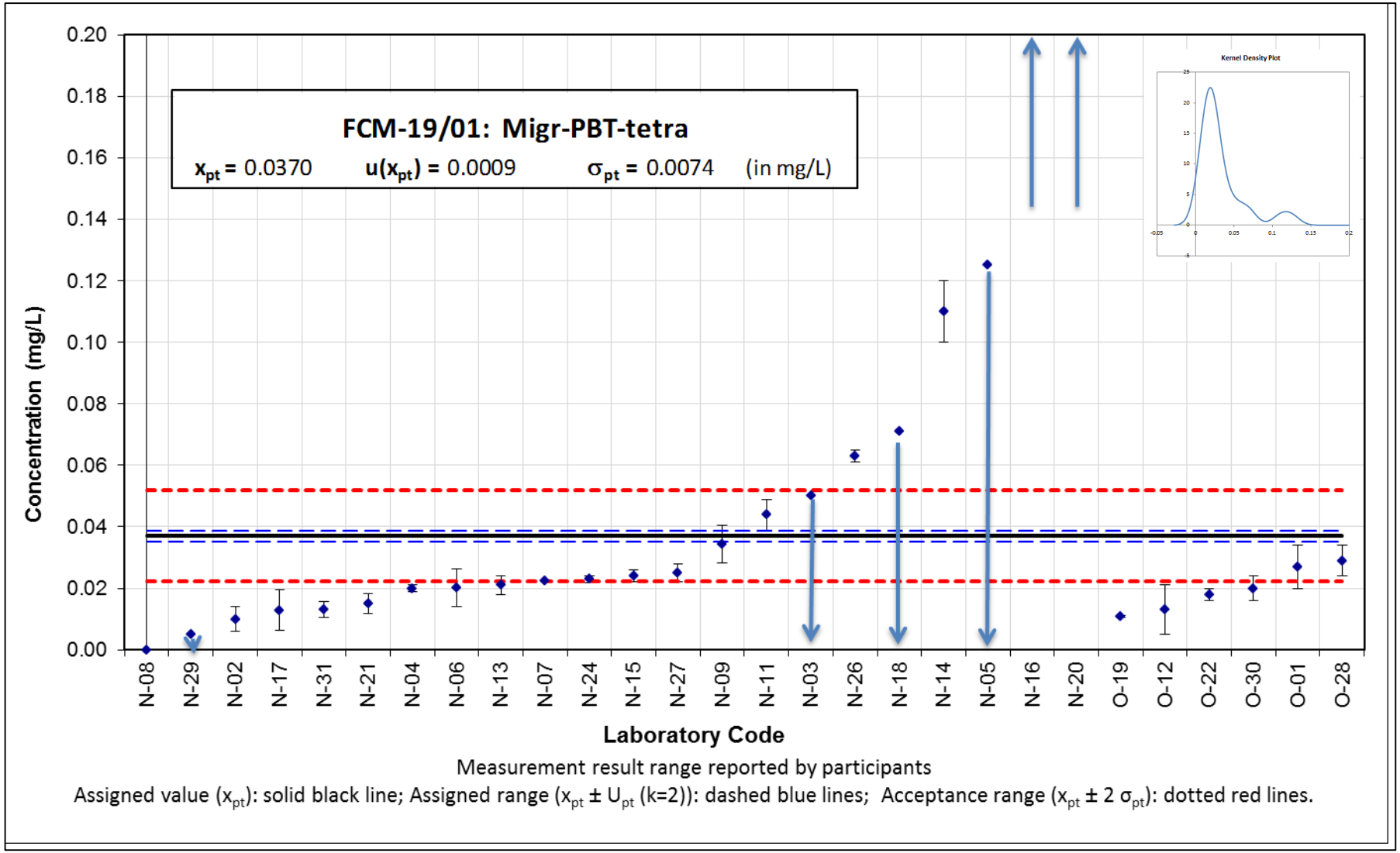
Assigned range: x_{pt} = 0.0370 ± 0.0018 $U(x_{pt}, k = 2.0)$; σ_{pt} = 0.0074 (all values in mg L^{-1})

Code	x_i	$U(x_i)$	k	Technique	$u(x_i)$	z score	ζ score	unc.
N-02	0.01	0.004	2	HPLC-UV	0.002	-3.6	-12.3	a
N-03	< 0.05			HPLC-UV				
N-04	0.02	0.001	√3	HPLC-UV	0.001	-2.3	-16.0	a
N-05	< 0.125			HPLC-UV				
N-06	0.0203	0.0061	2	HPLC-UV	0.003	-2.3	-5.3	a
N-07	0.0224			LC-MS/MS	0.000	-2.0	-16.4	b
N-08	0	0.46	2	HPLC-UV	0.230	-5.0	-0.2	
N-09	0.0344	0.0062	2	HPLC-UV	0.003	-0.4	-0.8	a
N-11	0.044	0.005	2	HPLC-UV	0.003	0.9	2.6	a
N-13	0.021	0.003	2	HPLC-DAD	0.002	-2.2	-9.2	a
N-14	0.11	0.01	2	HPLC-DAD	0.005	9.9	14.4	a
N-15	0.024	0.002	2	HPLC-UV	0.001	-1.8	-9.7	a
N-16	0.5	0.08	2	HPLC-UV	0.040	62.6	11.6	a
N-17	0.0129	0.0066	2	HPLC-UV	0.003	-3.3	-7.0	c
N-18	< 0.071			HPLC-PDA				
N-20	16	4	2	HPLC-UV	2.000	2157.6	8.0	a
N-21	0.0151	0.0032	2	HPLC-UV	0.002	-3.0	-12.0	a
N-24	0.023	0.0011	2	HPLC-UV	0.001	-1.9	-13.4	b
N-26	0.063	0.002	2	HPLC-DAD	0.001	3.5	19.4	b
N-27	0.025	0.003	3	LC-MS/MS	0.001	-1.6	-9.0	a
N-29	< 0.005			HPLC-UV				
N-31	0.013	0.0026	2	HPLC-UV	0.001	-3.2	-15.2	a
O-01	0.027	0.007	2	HPLC-UV	0.004	-1.4	-2.8	a
O-12	0.013	0.008	2	HPLC-UV	0.004	-3.2	-5.9	c
O-19	0.0108	0.0004	2	HPLC-UV	0.000	-3.5	-28.7	b
O-22	0.018	0.002	√3	HPLC-UV	0.001	-2.6	-13.0	a
O-28	0.029	0.005	2	HPLC-UV	0.003	-1.1	-3.0	a
O-30	0.02	0.004	2	LC-MS/MS	0.002	-2.3	-7.8	a

[a] √3 is set by the PT coordinator when no coverage factor *k* is reported. The reported uncertainty was assumed to have a rectangular distribution with *k* = √3,

[b] Performance: satisfactory, questionable, unsatisfactory,

[c] a: $u_{min,rel}(u_{rel}(x_{pt,})) \leq u_{rel}(x_i) \leq u_{max,rel}(\sigma_{pt})$; b: $u_{rel}(x_i) < u_{min,rel}(u_{rel}(x_{pt,}))$; and c: $u_{rel}(x_i) > u_{max,rel}(\sigma_{pt})$



Measurement result range reported by participants

Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 13: Results for PBT cyclic pentamer in Test item 2

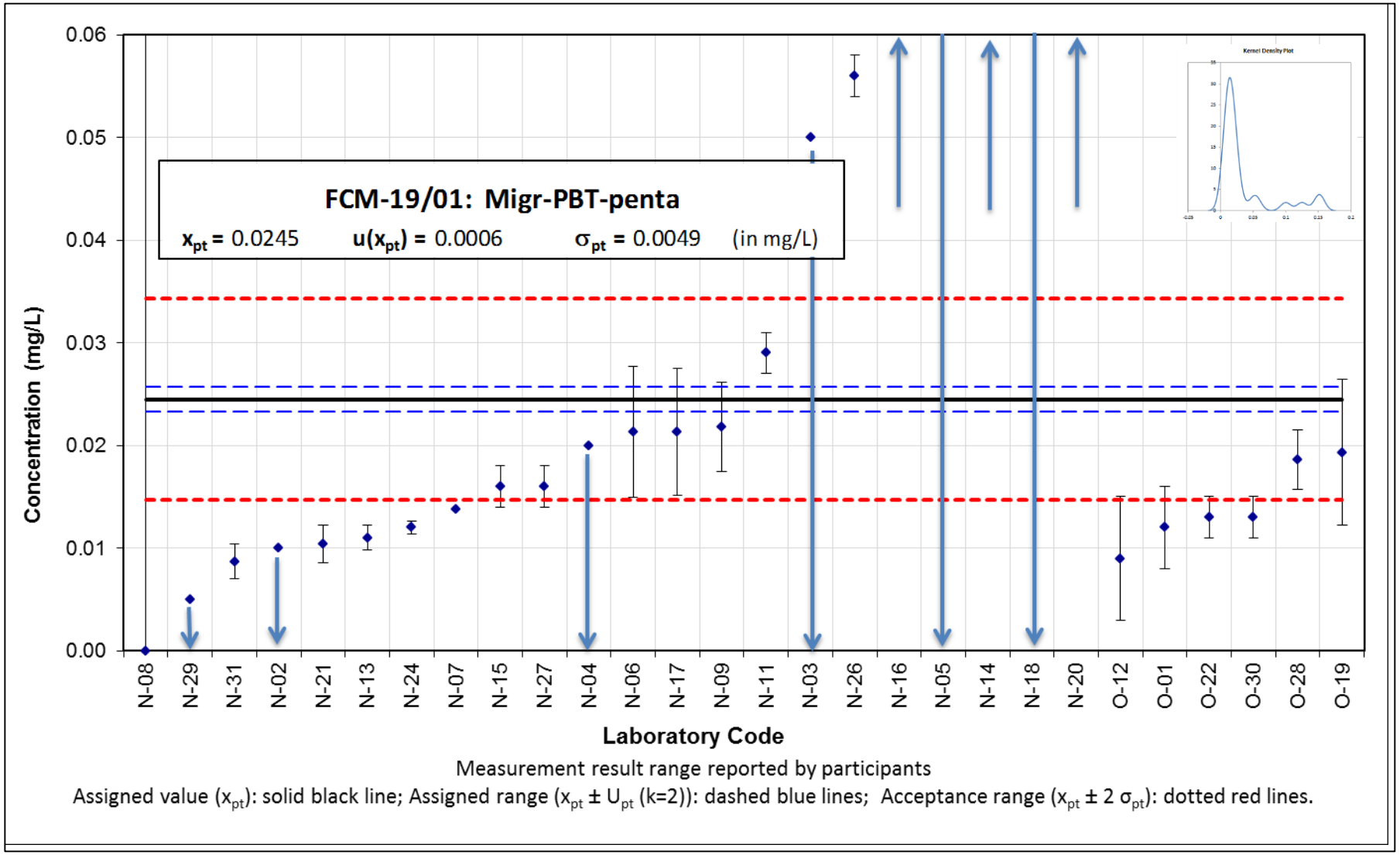
Assigned range: x_{pt} = 0.0245 ± 0.0012 $U(x_{pt}, k = 2.0)$; σ_{pt} = 0.0049 (all values in mg L^{-1})

Code	x_i	$U(x_i)$	k	Technique	$u(x_i)$	z score	ζ score	unc.
N-02	< 0.01			HPLC-UV				
N-03	< 0.05			HPLC-UV				
N-04	< 0.02			HPLC-UV				
N-05	< 0.125			HPLC-UV				
N-06	0.0213	0.0064	2	HPLC-UV	0.003	-0.7	-1.0	a
N-07	0.0138			LC-MS/MS	0.000	-2.2	-17.2	b
N-08	0	0.45	2	HPLC-UV	0.225	-5.0	-0.1	
N-09	0.0218	0.0043	2	HPLC-UV	0.002	-0.6	-1.2	a
N-11	0.029	0.002	2	HPLC-UV	0.001	0.9	3.8	a
N-13	0.011	0.0012	2	HPLC-DAD	0.001	-2.8	-15.6	a
N-14	0.15	0.01	2	HPLC-DAD	0.005	25.6	24.9	a
N-15	0.016	0.002	2	HPLC-UV	0.001	-1.7	-7.2	a
N-16	0.1	0.02	2	HPLC-UV	0.010	15.4	7.5	a
N-17	0.0213	0.0062	2	HPLC-UV	0.003	-0.7	-1.0	a
N-18	< 0.153			HPLC-PDA				
N-20	22	7	2	HPLC-UV	3.500	4485.2	6.3	a
N-21	0.0104	0.0018	2	HPLC-UV	0.001	-2.9	-12.9	a
N-24	0.012	0.0006	2	HPLC-UV	0.000	-2.6	-18.1	b
N-26	0.056	0.002	2	HPLC-DAD	0.001	6.4	26.8	b
N-27	0.016	0.002	3	LC-MS/MS	0.001	-1.7	-9.3	a
N-29	< 0.005			HPLC-UV				
N-31	0.0087	0.0017	2	HPLC-UV	0.001	-3.2	-15.0	a
O-01	0.012	0.004	2	HPLC-UV	0.002	-2.6	-6.0	a
O-12	0.009	0.006	2	HPLC-UV	0.003	-3.2	-5.1	c
O-19	0.0193	0.0071	2	HPLC-UV	0.004	-1.1	-1.4	a
O-22	0.013	0.002	1.73	HPLC-UV	0.001	-2.3	-8.8	a
O-28	0.0186	0.0029	2	HPLC-UV	0.001	-1.2	-3.7	a
O-30	0.013	0.002	2	LC-MS/MS	0.001	-2.3	-9.8	a

[a] √3 is set by the PT coordinator when no coverage factor *k* is reported. The reported uncertainty was assumed to have a rectangular distribution with *k* = √3,

[b] Performance: satisfactory, questionable, unsatisfactory,

[c] a: $u_{min,rel}\,(u_{rel}(x_{pt,})) \leq u_{rel}(x_i) \leq u_{max,rel}\,(\sigma_{pt})$; b: $u_{rel}(x_i) < u_{min,rel}\,(u_{rel}(x_{pt,}))$; and c: $u_{rel}(x_i) > u_{max,rel}\,(\sigma_{pt})$



Measurement result range reported by participants

Assigned value (x_{pt}): solid black line; Assigned range ($x_{pt} \pm U_{pt}$ (k=2)): dashed blue lines; Acceptance range ($x_{pt} \pm 2\ \sigma_{pt}$): dotted red lines.

Annex 14: Detailed analytical method parameters and performance for Test item 2

LabCode	Analyte+	Same analytical method used for both test items?	stationary phase	mobile phase	injection volume (µL)	Limit of Detection (LOD)	detection
N-02	**PBT dimer**	Yes	C18	Water-acetonitrile	50	0.01	UV
	PBT trimer	Yes	C18	Water-acetonitrile	50	0.01	UV
	PBT tetramer	Yes	C18	Water-acetonitrile	50	0.01	UV
	PBT pentamer	Yes	C18	Water-acetonitrile	50	0.01	UV
N-03	**PBT dimer**	Yes	C18 Xterra Waters	acetonitrile/water	70	0.025	DAD 240nm
	PBT trimer	Yes	C18 Xterra Waters	acetonitrile/water	70	0.025	DAD 240nm
	PBT tetramer	Yes	C18 Xterra Waters	acetonitrile/water	70	0.025	DAD 240nm
	PBT pentamer	Yes	C18 Xterra Waters	acetonitrile/water	70	0.025	DAD 240nm
N-04	**PBT dimer**	Yes	Zorbax Eclipse XDB-C18	acetonitrile/water	100	0.020 mg/L	UV-240nm
	PBT trimer	Yes	Zorbax Eclipse XDB-C18	acetonitrile/water	100	0.020 mg/L	UV-240nm
	PBT tetramer	Yes	Zorbax Eclipse XDB-C18	acetonitrile/water	100	0.020 mg/L	UV-240nm
	PBT pentamer	Yes	Zorbax Eclipse XDB-C18	acetonitrile/water	100	0.020 mg/L	UV-240nm
N-05	**PBT dimer**	Yes	C18	Water/ACN	10	-	UV
	PBT trimer	Yes	C18	Water/ACN	10	-	UV
	PBT tetramer	Yes	C18	Water/ACN	10	-	UV
	PBT pentamer	Yes	C18	Water/ACN	10	-	UV
N-06	**PBT dimer**	Yes	Poroshell 120 EC-C18	H2O/ACN	50	0.005 mg/L	UV
	PBT trimer	Yes	Poroshell 120 EC-C18	H2O/ACN	50	0.005 mg/L	UV
	PBT tetramer	Yes	Poroshell 120 EC-C18	H2O/ACN	50	0.005 mg/L	UV
	PBT pentamer	Yes	Poroshell 120 EC-C18	H2O/ACN	50	0.005 mg/L	UV
N-07	**PBT dimer**	Yes	C18; 1,7x2,1x50 130Å	0,1% formic acid	10	-	SIM
	PBT trimer	Yes	C18; 1,7x2,1x50 130Å	0,1% formic acid	10	-	SIM
	PBT tetramer	Yes	C18; 1,7x2,1x50 130Å	0,1% formic acid	10	-	SIM
	PBT pentamer	Yes	C18; 1,7x2,1x50 130Å	0,1% formic acid	10	-	SIM
N-08	**PBT dimer**	Yes	XDB-C18	ACN and water	20	0.12 mg/L	UV
	PBT trimer	Yes	XDB-C18	ACN and water	20	0.12 mg/L	UV
	PBT tetramer	Yes	XDB-C18	ACN and water	20	0.12 mg/L	UV
	PBT pentamer	Yes	XDB-C18	ACN and water	20	0.12 mg/L	UV
N-09	**PBT dimer**	Yes	C18	ACN/H2O	10	9 ng/mL	DAD
	PBT trimer	Yes	C18	ACN/H2O	10	9 ng/mL	DAD
	PBT tetramer	Yes	C18	ACN/H2O	10	9 ng/mL	DAD
	PBT pentamer	Yes	C18	ACN/H2O	10	9 ng/mL	DAD
N-11	**PBT dimer**	Yes	C18	ACN/H2O	50	0.01 mg/L	PDA
	PBT trimer	Yes	C18	ACN/H2O	50	0.01 mg/L	PDA
	PBT tetramer	Yes	C18	ACN/H2O	50	0.01 mg/L	PDA
	PBT pentamer	Yes	C18	ACN/H2O	50	0.01 mg/L	PDA
N-13	**PBT dimer**	Yes	C18	ACN-H2O	50	0.01 µg/mL	DAD
	PBT trimer	Yes	C18	ACN-H2O	50	0.01 µg/mL	DAD
	PBT tetramer	Yes	C18	ACN-H2O	50	0.01 µg/mL	DAD
	PBT pentamer	Yes	C18	ACN-H2O	50	0.01 µg/mL	DAD
N-14	**PBT dimer**	Yes	C18	Acetonitrile/water	100	0.5 µg/mL	HPLC_DAD
	PBT trimer	Yes	C18	Acetonitrile/water	100	0.5 µg/mL	HPLC_DAD
	PBT tetramer	Yes	C18	Acetonitrile/water	100	0.5 µg/mL	HPLC_DAD
	PBT pentamer	Yes	C18	Acetonitrile/water	100	0.5 µg/mL	HPLC_DAD
N-15	**PBT dimer**	Yes	C18	Acetonitrile/water	100	0.01 mg/kg	UV
	PBT trimer	Yes	C18	Acetonitrile/water	100	0.01 mg/kg	UV
	PBT tetramer	Yes	C18	Acetonitrile/water	100	0.01 mg/kg	UV

LabCode	Analyte+	Same analytical method used for both test items?	stationary phase	mobile phase	injection volume (µL)	Limit of Detection (LOD)	detection
	PBT pentamer	Yes	C18	Acetonitrile/water	100	0.01 mg/kg	UV
N-16	**PBT dimer**	Yes	C18	A: 0.015% Formic Acid B:	10	0.1 mg/kg	UV
	PBT trimer	Yes	C18	A: 0.015% Formic Acid B:	10	0.1 mg/kg	UV
	PBT tetramer	Yes	C18	A: 0.015% Formic Acid B:	10	0.1 mg/kg	UV
	PBT pentamer	Yes	C18	A: 0.015% Formic Acid B:	10	0.1 mg/kg	UV
N-17	**PBT dimer**	b) No	RP18	ACN:H2O	50	-	UV
	PBT trimer	b) No	RP18	ACN:H2O	50	-	UV
	PBT tetramer	b) No	RP18	ACN:H2O	50	-	UV
	PBT pentamer	b) No	RP18	ACN:H2O	50	-	UV
N-18	**PBT dimer**	b) No	C18	Water/Acetonitrile	50	-	PDA
	PBT trimer	b) No	C18	Water/Acetonitrile	50	-	PDA
	PBT tetramer	b) No	C18	Water/Acetonitrile	50	-	PDA
	PBT pentamer	b) No	C18	Water/Acetonitrile	50	-	PDA
N-20	**PBT dimer**	Yes	C18	ACN/H2O	20	70 ng/ml	UV 240 nm
	PBT trimer	Yes	C18	ACN/H2O	20	70 ng/ml	UV 240 nm
	PBT tetramer	Yes	C18	ACN/H2O	20	70 ng/ml	UV 240 nm
	PBT pentamer	Yes	C18	ACN/H2O	20	70 ng/ml	UV 240 nm
N-21	**PBT dimer**	Yes	C18	Water/Acn	50	-	240nm
	PBT trimer	Yes	C18	Water/Acn	50	-	240nm
	PBT tetramer	Yes	C18	Water/Acn	50	-	240nm
	PBT pentamer	Yes	C18	Water/Acn	50	-	240nm
N-24	**PBT dimer**	Yes	RP18	ACN/H2O	5-20	0.005 mg/l	UV 240 nm
	PBT trimer	Yes	RP18	ACN/H2O	5-20	0.005 mg/l	UV 240 nm
	PBT tetramer	Yes	RP18	ACN/H2O	5-20	-	UV 240 nm
	PBT pentamer	Yes	RP18	ACN/H2O	5-20	0.005 mg/l	UV 240 nm
N-26	**PBT dimer**	Yes	C18	Acetonitrile/H2O	50	0.05	DAD
	PBT trimer	Yes	C18	Acetonitrile/H2O	50	0.05	DAD
	PBT tetramer	Yes	C18	Acetonitrile/H2O	50	0.05	DAD
	PBT pentamer	Yes	C18	Acetonitrile/H2O	50	0.05	DAD
N-27	**PBT dimer**	Yes	100x2.1mm, 1.9µm/C18	H2O/ACN	5	2 µg/L	MS/MS (APCI)
	PBT trimer	Yes	100x2.1mm, 1.9µm/C18	H2O/ACN	5	2 µg/L	MS/MS (APCI)
	PBT tetramer	Yes	100x2.1mm, 1.9µm/C18	H2O/ACN	5	2 µg/L	MS/MS (APCI)
	PBT pentamer	Yes	100x2.1mm, 1.9µm/C18	H2O/ACN	5	2 µg/L	MS/MS (APCI)
N-29	**PBT dimer**	Yes	C18	acetonitrile/water	100	0.002 mg/L	240 nm
	PBT trimer	Yes	C18	acetonitrile/water	100	0.002 mg/L	240 nm
	PBT tetramer	Yes	C18	acetonitrile/water	100	0.002 mg/L	240 nm
	PBT pentamer	Yes	C18	acetonitrile/water	100	0.002 mg/L	240 nm
N-31	**PBT dimer**	Yes	XDB-C18	ACN/H2O	100	0.002 mg/L	UV
	PBT trimer	Yes	XDB-C18	ACN/H2O	100	0.002 mg/L	UV
	PBT tetramer	Yes	XDB-C18	ACN/H2O	100	0.002 mg/L	UV
	PBT pentamer	Yes	XDB-C18	ACN/H2O	100	0.002 mg/L	UV
O-01	**PBT dimer**	Yes	Zorbax Elclipse XDB-C8	Acetonitril/Water	20	0.015 mg/l	240 nm
	PBT trimer	Yes	Zorbax Elclipse XDB-C8	Acetonitril/Water	20	0.015 mg/l	240 nm
	PBT tetramer	Yes	Zorbax Elclipse XDB-C8	Acetonitril/Water	20	0.015 mg/l	240 nm
	PBT pentamer	Yes	Zorbax Elclipse XDB-C8	Acetonitril/Water	20	0.015 mg/l	240 nm
O-12	**PBT dimer**	Yes	Luna 5µ C18; 150x4,6 mm	ACN/H2O (Gradient)	20	0.005	DAD
	PBT trimer	Yes	Luna 5µ C18; 150x4,6 mm	ACN/H2O (Gradient)	20	0.005	DAD
	PBT tetramer	Yes	Luna 5µ C18; 150x4,6 mm	ACN/H2O (Gradient)	20	0.005	DAD
	PBT pentamer	Yes	Luna 5µ C18; 150x4,6 mm	ACN/H2O (Gradient)	20	0.005	DAD
O-19	**PBT dimer**	Yes	RP C18	MeOH-Water 95:5 isocratic	5	0.01 mg/kg	UV (240 nm)
	PBT trimer	Yes	RP C18	MeOH-Water 95:5 isocratic	5	0.01 mg/kg	UV (240 nm)
	PBT tetramer	Yes	RP C18	MeOH-Water 95:5 isocratic	5	0.01 mg/kg	UV (240 nm)
	PBT pentamer	Yes	RP C18	MeOH-Water 95:5 isocratic	5	0.01 mg/kg	UV (240 nm)

LabCode	Analyte+	Same analytical method used for both test items?	stationary phase	mobile phase	injection volume (µL)	Limit of Detection (LOD)	detection
O-22	**PBT dimer**	Yes	RP-18	water/ACN (gradient)	10	0.0025 mg/L	DAD (240 nm)
	PBT trimer	Yes	RP-18	water/ACN (gradient)	10	0.0025 mg/L	DAD (240 nm)
	PBT tetramer	Yes	RP-18	water/ACN (gradient)	10	0.0025 mg/L	DAD (240 nm)
	PBT pentamer	Yes	RP-18	water/ACN (gradient)	10	0.0025 mg/L	DAD (240 nm)
O-28	**PBT dimer**	b) No	Zorbax XDB C18	Acetonitrile Water	10	0.014	UV
	PBT trimer	b) No	Zorbax XDB C18	Acetonitrile Water	10	0.011	UV
	PBT tetramer	b) No	Zorbax XDB C18	Acetonitrile Water	10	0.014	UV
	PBT pentamer	b) No	Zorbax XDB C18	Acetonitrile Water	10	0.018	UV
O-30	**PBT dimer**	Yes	C18	formic acid	5	3	msms
	PBT trimer	Yes	C18	formic acid	5	3	msms
	PBT tetramer	Yes	C18	formic acid	5	3	msms
	PBT pentamer	Yes	C18	formic acid	5	3	msms

Annex 15: Overview on the experimental details related to extraction with Test item 1

LabCode	Time	Temperature	Solvents
N-02	2hrs	ambient	ethanol
N-03	30min, ultrasound	25 °C	hexafluoro-2-propanol
N-04	24 hours	40°C	diclroromethane
N-05	-	-	-
N-06	1h	60 °C	50% EtOH/H2O
N-07	60 min	40 °C	dichloromethane
N-08	2h	70 degrees	50% EtOH
N-09	3x 30 min	40°C	HFIP + DCM
N-11	50h	boiling temperature	CH_2Cl_2
N-13	20 min	30°C	Chloroform
N-14	24 h	40°C	AcCN, $CHCl_3$
N-15	20	100°C	Acetonitrile
N-16	60 minutes	60 °C	Dichloromethane
N-18	1 hr	120 °C	Dichloromethane
N-20	2 h	70 °C	D1: 50% EtOH
N-21	2*30min	RT	(HFIP/DCM)-IP
N-24	2x 18 h	60 °C	THF
N-26	20 min	30 ^{o}C	HFIP/Acetonitil
N-27	24h	23°C	Ethanol
N-29	36	ambient	DCM
N-31	67h	ambient	DCM
O-01	18 h	60 °C	Acetonitrol
O-12	60 min	60°C	dichlormethan
O-19	2 h	40 °C	Acetone
O-22	1 h	60 °C in ultrasonic bath	dichloromethane
O-28	30	Roomtemp	Hexafluorisopropanol, DCM
O-30	2h	70 °C	D1

Annex 16:Detailed migration testing parameters for Test item 2

LabCode	Analyte	Temperature of the oven used.	cups preheat before migration?	controlled temperature of Sim.D1?	Volume Simulant D1 used for migration	If Yes, how was temperature controlled?.
N-02	**PBT dimer**	70°C	Yes	No	280ml	
	PBT trimer	70°C	Yes	No	280ml	
	PBT tetramer	70°C	Yes	No	280ml	
	PBT pentamer	70°C	Yes	No	280ml	
N-03	**PBT dimer**	70°C	No	Yes	280 mL	Calibrated datalogger
	PBT trimer	70°C	No	Yes	280 mL	Calibrated datalogger
	PBT tetramer	70°C	No	Yes	280 mL	Calibrated datalogger
	PBT pentamer	70°C	No	Yes	280 mL	Calibrated datalogger
N-04	**PBT dimer**	70 C	No	Yes	300 mL	Non-calibrated thermometer
	PBT trimer	71 C	No	Yes	300 mL	Non-calibrated thermometer
	PBT tetramer	72 C	No	Yes	300 mL	Non-calibrated thermometer
	PBT pentamer	73 C	No	Yes	300 mL	Non-calibrated thermometer
N-05	**PBT dimer**	72	No	Yes	300 ml	Calibrated thermometer
	PBT trimer	72	No	Yes	300 ml	Calibrated thermometer
	PBT tetramer	72	No	Yes	300 ml	Calibrated thermometer
	PBT pentamer	72	No	Yes	300 ml	Calibrated thermometer
N-06	**PBT dimer**	70	Yes	Yes	300	Calibrated thermometer
	PBT trimer	70	Yes	Yes	300	Calibrated thermometer
	PBT tetramer	70	Yes	Yes	300	Calibrated thermometer
	PBT pentamer	70	Yes	Yes	300	Calibrated thermometer
N-07	**PBT dimer**	70 °C	No	Yes	300 ml	Calibrated thermometer
	PBT trimer	70 °C	No	Yes	300 ml	Calibrated thermometer
	PBT tetramer	70 °C	No	Yes	300 ml	Calibrated thermometer
	PBT pentamer	70 °C	No	Yes	300 ml	Calibrated thermometer
N-08	**PBT dimer**	75	No	No	298	
	PBT trimer	75	No	No	298	
	PBT tetramer	75	No	No	298	
	PBT pentamer	75	No	No	298	
N-09	**PBT dimer**	set 73°C (71,7°C)	Yes	Yes	275	Calibrated datalogger
	PBT trimer	set 73°C (71,7°C)	Yes	Yes	275	Calibrated datalogger
	PBT tetramer	set 73°C (71,7°C)	Yes	Yes	275	Calibrated datalogger
	PBT pentamer	set 73°C (71,7°C)	Yes	Yes	275	Calibrated datalogger
N-11	**PBT dimer**	70 °C	Yes	Yes	300 ml	Calibrated thermometer
	PBT trimer	70 °C	Yes	Yes	300 ml	Calibrated thermometer
	PBT tetramer	70 °C	Yes	Yes	300 ml	Calibrated thermometer
	PBT pentamer	70 °C	Yes	Yes	300 ml	Calibrated thermometer
N-13	**PBT dimer**	72°C	No	Yes	300 ml	Calibrated datalogger
	PBT trimer	72°C	No	Yes	300 ml	Calibrated datalogger
	PBT tetramer	72°C	No	Yes	300 ml	Calibrated datalogger
	PBT pentamer	72°C	No	Yes	300 ml	Calibrated datalogger
N-14	**PBT dimer**	70	Yes	No	300 ml	
	PBT trimer	70	Yes	No	300 ml	
	PBT tetramer	70	Yes	No	300 ml	
	PBT pentamer	70	Yes	No	300 ml	
N-15	**PBT dimer**	70	No	No	300	
	PBT trimer	70	No	No	300	
	PBT tetramer	70	No	No	300	
	PBT pentamer	70	No	No	300	
N-16	**PBT dimer**	70	No	No	250	

LabCode	Analyte	Temperature of the oven used.	cups preheat before migration?	controlled temperature of Sim.D1?	Volume Simulant D1 used for migration	If Yes, how was temperature controlled?.
	PBT trimer	70	No	No	250	
	PBT tetramer	70	No	No	250	
	PBT pentamer	70	No	No	250	
N-17	**PBT dimer**	70	Yes	Yes	300	Calibrated thermometer
	PBT trimer	70	Yes	Yes	300	Calibrated thermometer
	PBT tetramer	70	Yes	Yes	300	Calibrated thermometer
	PBT pentamer	70	Yes	Yes	300	Calibrated thermometer
N-18	**PBT dimer**	70 °C	No	No	300 ml	
	PBT trimer	70 °C	No	No	300 ml	
	PBT tetramer	70 °C	No	No	300 ml	
	PBT pentamer	70 °C	No	No	300 ml	
N-20	**PBT dimer**	2 h, 70°C	No	Yes	280 ml	Calibrated thermometer
	PBT trimer	2 h, 70°C	No	Yes	280 ml	Calibrated thermometer
	PBT tetramer	2 h, 70°C	No	Yes	280 ml	Calibrated thermometer
	PBT pentamer	2 h, 70°C	No	Yes	280 ml	Calibrated thermometer
N-21	**PBT dimer**	70	No	Yes	285	Calibrated datalogger
	PBT trimer	70	No	Yes	285	Calibrated datalogger
	PBT tetramer	70	No	Yes	285	Calibrated datalogger
	PBT pentamer	70	No	Yes	285	Calibrated datalogger
N-24	**PBT dimer**	70 °C	Yes	No	240 ml	
	PBT trimer	70 °C	Yes	No	240 ml	
	PBT tetramer	70 °C	Yes	No	240 ml	
	PBT pentamer	70 °C	Yes	No	240 ml	
N-26	**PBT dimer**	70°C	No	Yes	250 ml	Calibrated thermometer
	PBT trimer	70°C	No	Yes	250 ml	Calibrated thermometer
	PBT tetramer	70°C	No	Yes	250 ml	Calibrated thermometer
	PBT pentamer	70°C	No	Yes	250 ml	Calibrated thermometer
N-27	**PBT dimer**	72	No	No	280	
	PBT trimer	72	No	No	280	
	PBT tetramer	72	No	No	280	
	PBT pentamer	72	No	No	280	
N-29	**PBT dimer**	70	No	Yes		Calibrated thermometer
	PBT trimer	70	No	Yes		Calibrated thermometer
	PBT tetramer	70	No	Yes		Calibrated thermometer
	PBT pentamer	70	No	Yes		Calibrated thermometer
N-31	**PBT dimer**	70	No	Yes	300	Calibrated datalogger
	PBT trimer	70	No	Yes	300	Calibrated datalogger
	PBT tetramer	70	No	Yes	300	Calibrated datalogger
	PBT pentamer	70	No	Yes	300	Calibrated datalogger
O-01	**PBT dimer**	70	No	Yes	300	Calibrated thermometer
	PBT trimer	70	No	Yes	300	Calibrated thermometer
	PBT tetramer	70	No	Yes	300	Calibrated thermometer
	PBT pentamer	70	No	Yes	300	Calibrated thermometer
O-12	**PBT dimer**	70	No	No	250	Other
	PBT trimer	70	No	No	250	Other
	PBT tetramer	70	No	No	250	Other
	PBT pentamer	70	No	No	250	Other
O-19	**PBT dimer**	70	Yes	Yes	280	Calibrated thermometer
	PBT trimer	70	Yes	Yes	280	Calibrated thermometer
	PBT tetramer	70	Yes	Yes	280	Calibrated thermometer
	PBT pentamer	70	Yes	Yes	280	Calibrated thermometer
O-22	**PBT dimer**	70 °C	No	No	250 mL	
	PBT trimer	71 °C	No	No	250 mL	
	PBT tetramer	72 °C	No	No	250 mL	
	PBT pentamer	73 °C	No	No	250 mL	
O-28	**PBT dimer**	73-75°C (oven);	Yes	Yes	284 mL; /290 mL / 290	Calibrated thermometer

LabCode	Analyte	Temperature of the oven used.	cups preheat before migration?	controlled temperature of Sim.D1?	Volume Simulant D1 used for migration	If Yes, how was temperature controlled?.
		68-70°C (Simulant)			mL (at 20°C)	
	PBT trimer	73-75°C (oven); 68-70°C (Simulant)	Yes	Yes	284 mL; /290 mL / 290 mL (at 20°C)	Calibrated thermometer
	PBT tetramer	73-75°C (oven); 68-70°C (Simulant)	Yes	Yes	284 mL; /290 mL / 290 mL (at 20°C)	Calibrated thermometer
	PBT pentamer	73-75°C (oven); 68-70°C (Simulant)	Yes	Yes	284 mL; /290 mL / 290 mL (at 20°C)	Calibrated thermometer
O-30	**PBT dimer**	70	No	Yes	320	Calibrated thermometer
	PBT trimer	70	No	Yes	320	Calibrated thermometer
	PBT tetramer	70	No	Yes	320	Calibrated thermometer
	PBT pentamer	70	No	Yes	320	Calibrated thermometer

Annex 17: Overview of the reported densities for conversion of concentrations to mass fractions

LabCode	kg/L					RSD
	PBT dimer	PBT trimer	PBT tetramer	PBT pentamer	Average/lab	
N-02	0.89	0.90	0.91	-	0.90	0.9 %
N-03	1.00	1.00	-	-	1.00	0.0 %
N-04	1.20	1.20	1.25	-	1.22	2.3 %
N-05	0.92	0.92	-	-	0.92	0.0 %
N-06	0.94	0.92	0.94	0.93	0.93	0.8 %
N-07	1.14	1.14	1.14	1.14	1.14	0.1 %
N-08	1.17	1.17	-	-	1.17	0.0 %
N-09	0.93	0.93	0.93	1.07	0.97	7.4 %
N-11	0.93	0.91	0.92	0.94	0.92	1.4 %
N-13	0.91	0.88	0.91	0.85	0.89	3.5 %
N-14	1.00	1.00	1.00	1.00	1.00	0.0 %
N-15	1.00	1.00	1.00	1.00	1.00	0.0 %
N-16	1.03	1.03	1.04	1.00	1.03	1.8 %
N-17	0.93	0.93	0.93	0.93	0.93	0.2 %
N-18	0.91	0.89			0.90	1.8 %
N-20	0.89	0.89	0.89	0.92	0.90	1.5 %
N-21	0.92	0.92	0.92	0.92	0.92	0.3 %
N-24	1.36	1.36	1.35	1.36	1.36	0.3 %
N-26	1.00	1.00	1.00	1.00	1.00	0.0 %
N-27	0.91	0.91	0.89	0.89	0.90	1.3 %
N-29	0.93	0.93			0.93	0.0 %
N-31	0.92	0.94	0.93	0.94	0.93	1.0 %
O-01	1.00	1.00	1.00	1.00	1.00	0.0 %
O-12	0.92	0.95	0.93	0.90	0.92	2.1 %
O-19	0.98	0.98	0.98	0.98	0.98	0.2 %
O-22	0.92	0.92	0.90	0.93	0.92	1.3 %
O-28	0.93	0.93	0.93	0.93	0.93	0.1 %
O-30	1.06	1.04	1.05	1.08	1.06	1.7 %
Average	**0.99**	**0.99**	**0.99**	**0.99**		
Min	0.89	0.88	0.89	0.85		
Max	1.36	1.36	1.35	1.36		

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