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JRC VALIDATED METHODS, REFERENCE METHODS AND MEASUREMENTS REPORT

Determination of the mass fractions of PBT and PET oligomers in food simulant D1

FCM-18-01 Proficiency Test Report

P. Dehouck, E. Tsochatzis, J. Alberto Lopes,
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European Union Reference Laboratory for Food Contact Materials

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

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Executive summary

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM) organised a proficiency test (FCM-18-01) for the determination of the mass fractions of four oligomers (ethylene terephthalate cyclic dimer (PET cyclic dimer), ethylene terephthalate cyclic trimer (PET cyclic trimer), butylene terephthalate cyclic dimer (PBT cyclic dimer) and butylene terephthalate cyclic trimer (PBT cyclic trimer)) in food simulant D1 (ethanol 50 % v/v) to support Regulation 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 prepared: solution 1 consisted of food simulant D1 spiked with a known amount of the four oligomers; solution 2 was obtained by migration from PET bottles into food simulant D1 and further spiking with the four oligomers. The homogeneity and stability of the test items were evaluated and the assigned values were derived from the results reported by the EURL-FCM.

Twenty seven NRLs from twenty five Member States and Switzerland, and nine OCLs (from Germany and Italy) registered to the exercise. One NRL and one OCL did not report results.

Laboratory results were rated using z (or 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, based on the perception of experts.

More than 79 % and 71 % of the participating laboratories performed satisfactorily (according to the z score) for the analysis of the four oligomers in solution 1 and solution 2, respectively. These results confirm that most NRLs are able to monitor mass fractions of oligomers in the frame of Regulation 10/2011. However further studies including the migration of oligomers from food contact materials are necessary.

Many of the participants underestimated the measurement uncertainty, which may be due to the lack of experience with such analyses. Several laboratories applied analytical methods that were not yet validated and estimated their measurement uncertainty from replicate analyses only.

List of abbreviations and symbols

DAD	Diode Array Detector
DG SANTE	Directorate General for Health and Food Safety
EURL	European Union Reference Laboratory
FCM	Food Contact Materials
GUM	Guide for the Expression of Uncertainty in Measurement
HPLC	High Performance Liquid Chromatography
ISO	International Organization for Standardization
JRC	Joint Research Centre
LC	Liquid Chromatography
LOD	Limit of Detection
NIAS	Non-Intentionally Added Substances
NRL	National Reference Laboratory
OCL	Official Control Laboratory
PBT	Polybutylene terephthalate
PE	Polyethylene
PET	Polyethylene terephthalate
PT	Proficiency Test
K	coverage factor
σ_{pt}	standard deviation for proficiency test assessment
$u(x_i)$	calculated standard measurement uncertainty (of participant "i")
$u(x_{pt})$	standard 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
$U(x_i)$	reported expanded uncertainty by participant "i"
$U(x_{pt})$	expanded uncertainty of the assigned value
x_i	reported mean value by participant "i"
x_{pt}	assigned value
z (or z')	z (or z') score
ζ	zeta 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 the mass fractions of four cyclic oligomers in food simulant D1 (ethanol 50 % v/v). Two cyclic PBT oligomers are regulated as FCM substance No 885 and two cyclic PET oligomers are Non-Intentionally Added Substances (NIAS).

This PT was agreed with the Directorate General for Health and Food Safety (DG SANTE) as part of the EURL-FCM annual work programme 2018. The PT was open to National Reference Laboratories (NRLs) and to Official Control Laboratories (OCLs) willing to participate.

This report summarises the outcome of the PT.

2. Scope

As stated in Regulation (EU) 2017/625 [1], one of the core duties of EURLs is to organise interlaboratory comparisons for the benefit of NRLs.

The present PT aims to assess the performance of NRLs and OCLs in the determination of the mass fractions of ethylene terephthalate cyclic dimer (PET cyclic dimer), butylene terephthalate cyclic dimer (PBT cyclic dimer), ethylene terephthalate cyclic trimer (PET cyclic trimer) and butylene terephthalate cyclic trimer (PBT cyclic trimer) in two solutions of food simulant D1. While the PET oligomers are considered as NIAS, the PBT oligomers are part of a mixture additive FCM No 885, including also the butylene terephthalate cyclic tetramer and pentamer [2].

The reported results were assessed following the administrative and logistic procedures of the JRC Unit in charge of the EURL-FCM, which is accredited for the organisation of PTs according to ISO 17043:2010 [3].

This PT is identified as "FCM-18-01".

3. Set up of the exercise

3.1 Time frame

The organisation of the FCM-18-01 exercise was agreed upon by the EURL-NRL-FCM network at the Plenary Meeting held in Ispra on October 24-26, 2017. An invitation letter was sent (via e-mail) to all NRLs of the network on March 19, 2018 (Annex 1). The registration deadline was set to April 30, 2018. Samples were sent to participants on May 15, 2018. A second dispatch was performed on May 23, 2018 for a few laboratories upon specific request. The dispatch was monitored by the PT coordinator using the messenger's parcel tracking system on the internet. The deadline for reporting of results was set to June 15, 2018. This deadline was extended till June 22, 2018 triggered by the second dispatch.

3.2 Confidentiality

The procedures used here for the organisation of PTs are accredited according to ISO 17043:2010 [3] and guarantee that the identity of the participants and the information provided by them is treated as confidential. However, the laboratory codes of NRLs appointed in line with Regulation (EU) 2017/625 [1] 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 Distribution

Each participant received:

- two test solutions (1 and 2), each in one vial containing approx. 5 mL of test item;
- a calibration solution - mixture containing of target analytes at 20 µg mL^{-1} - in a vial containing approx. 1 mL of hexafluoro-2-propanol/ethanol;
- a temperature sensor/indicator;
- the "Test item accompanying letter" (Annex 2); and
- a "Confirmation of receipt form" to be sent back to the PT coordinator after receipt of the test item (Annex 3).

Samples were sent under cooled conditions with ice packs to ensure a temperature below 4 °C during dispatch.

3.4 Instructions to participants

Detailed instructions were given to participants in the "Test item accompanying letter" mentioned above. The measurands were defined as "the mass fractions of PET cyclic dimer, PBT cyclic dimer, PET cyclic trimer and PBT cyclic trimer in food simulant D1".

Participants were asked (i) to check whether the bottles and vial were undamaged after transport, (ii) to read the temperature sensor included in the container immediately upon arrival of the parcel, and (iii) to report the exposure category in the "Confirmation of receipt form". New samples were dispatched to eight participants having declared that the temperature sensor indicated that test items were exposed to temperatures above 4 °C for a moderate or prolonged time.

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.

A density of 1.0 g mL^{-1} was used as conversion factor for both test solutions. Results were to be reported in the same format (e.g. number of significant figures) as normally reported to customers. Since the homogeneity study was performed with 50 µL sample intakes, the recommended minimum sample intake was set to 50 µL.

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 levels. Since many participants did not have long experience in the analysis of oligomers in food simulants, a technical note was provided describing the method validated by the EURL-FCM (Annex 4).

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 5).

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

4. Test item

4.1 Preparation

Annex III of Regulation (EC) 10/2011 [2] defines "food simulant D1" as a solution of water with ethanol 50 % v/v.

A PET bottle was supplied by ALPLA-Werke Lehner GmbH & Co. KG (Vlotho, Germany), shipped on cardboard base, covered with PE, and stored at room temperature (max 25 °C). This bottle was used for the migration experiment: it was filled with 500 mL of food simulant D1 and the migration experiment was performed at 70 °C for 2 h, as prescribed in Annex V of Regulation (EC) 10/2011 [2].

Half a litre of food simulant D1 solution was gravimetrically spiked with the selected PET and PBT cyclic oligomers (dimers, trimers) and shaken for 5 min to obtain solution 1. Similarly, half a litre of the migrated solution mentioned above was gravimetrically spiked with the selected PET and PBT cyclic oligomers (dimers, trimers) and shaken for 5 min to obtain solution 2. Portions of 5 mL were manually filled into 25 mL screw capped Schott vials and then stored at -18 °C.

In addition, a 20 µg mL^{-1} stock solution containing the selected cyclic oligomers in hexafluoro-2-propanol/EtOH was prepared gravimetrically. Portions of 1 mL were manually transferred to 5 mL amber vials, and stored at -18 °C.

The mass fractions (mg kg^{-1}) of the four oligomers in both solutions were determined as described in Section 5.1.

Each vial was identified with a unique number and the PT identifier.

4.2 Homogeneity and stability

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

High Performance Liquid Chromatography with Diode Array Detection (HPLC-DAD) was used to determine the mass fractions of the selected PET and PBT cyclic oligomers 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. Details of the analytical procedure are given in the Technical Note (Annex 4).

The assessment of homogeneity was performed after the preparation of the test items and before distribution to participants. For each solution, ten vials were randomly selected and analysed in duplicate. Results were evaluated according to ISO 13528:2015 [5]. Both solutions proved to be adequately homogeneous for the investigated analytes (Annex 6.1). The contribution from homogeneity (u_{hom}) to the standard uncertainty of the assigned value ($u(x_{pt})$) was calculated using SoftCRM [6].

Three additional samples of each solution 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 solutions are adequately stable (i) at +4 °C (fridge temperature) over the whole period of time of the PT (8 weeks, from the value assignment till the deadline for reporting results), (ii) for 2 weeks at 20 °C (simulating extreme conditions which may occur during transport) and (iii) at the reference temperature of -18 °C. The uncertainty contribution due to stability was set to zero (u_{st} = 0) for all the investigated analytes (Annex 6.2).

5. Assigned values and corresponding uncertainties

5.1 Assigned values

The assigned values (x_{pt}) of mass fractions for the PET cyclic dimer or trimer, and the PBT cyclic dimer or trimer in the two solutions of food simulant D1 listed in Table 1 were determined by the EURL-FCM applying the validated HPLC-DAD method mentioned above.

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 [5]:

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

where u_{char} was derived from the intermediate precision obtained in the frame of the method validation study, according to ISO 5725 [7].

5.3 Standard deviation for proficiency assessment, σ_{pt}

The relative standard deviations for PT assessment (σ_{pt}) were set for all measurands to 20 % of the respective assigned values, based on expert judgment (Table 1).

Table 1: Assigned value (x_{pt}) and standard deviation for the PT assessment (σ_{pt}) for solutions 1 and 2. All values (excluding the last column) are expressed in mg kg^{-1}.

Sol.	Oligomers	x_{pt}	u_{char}	u_{hom}	$u(x_{pt})$	σ_{pt}	$u(x_{pt})/\sigma_{pt}$
1	PET cyclic dimer	0.0550	0.0026	0.0005	0.0026	0.0110	0.2
	PBT cyclic dimer	0.0538	0.0037	0.0005	0.0037	0.0108	0.3
	PET cyclic trimer	0.0530	0.0026	0.0002	0.0026	0.0106	0.2
	PBT cyclic trimer	0.0502	0.0061	0.0006	0.0061	0.0100	**0.6**
2	PET cyclic dimer	0.0585	0.0028	0.0007	0.0028	0.0117	0.2
	PBT cyclic dimer	0.0706	0.0049	0.0008	0.0049	0.0141	0.3
	PET cyclic trimer	0.1645	0.0080	0.0003	0.0080	0.0329	0.2
	PBT cyclic trimer	0.0509	0.0062	0.0005	0.0062	0.0102	**0.6**

6. Evaluation of results

6.1 Scores and evaluation criteria

The individual laboratory performance was expressed in terms of z and ζ scores according to ISO 13528:2015 [5]:

$$z = \frac{x_i - x_{pt}}{\sigma_{pt}} \qquad \text{Eq. 2}$$

$$\zeta = \frac{x_i - x_{pt}}{\sqrt{u^2(x_i) + u^2(x_{pt})}} \qquad \text{Eq. 3}$$

Where: x_i is the measurement result reported by a participant;

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

x_{pt} is the assigned value;

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

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

According to ISO 13528:2015 [5], when $u(x_{pt}) > 0.3\ \sigma_{pt}$ (cf. PBT cyclic trimer, Table 1) the uncertainty of the assigned value ($u(x_{pt})$) can be taken into account by expanding the denominator of the z score and calculating the z' score, as follows:

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

The interpretation of the *z* (or z') and *ζ* performance scores is done according ISO 13528:2015 [5]:

|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* (or 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.

The 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 [8].

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.

The standard measurement uncertainty from the laboratory $u(x_i)$ is most likely to fall in a range between a minimum and a maximum allowed uncertainty (case "a": $u_{min} \leq u_i \leq u_{max}$). u_{min} is set to the standard uncertainties of the assigned values $u(x_{pt})$. 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} is set to the standard deviation accepted for the PT assessment (σ_{pt}). Consequently, case "a" becomes: $u(x_{pt}) \leq u(x_i) \leq \sigma_{pt}$.

If $u(x_i)$ is smaller than $u(x_{pt})$ (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, measurement uncertainties smaller than $u(x_{pt})$ are possible and plausible.

If $u(x_i)$ 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}" is a normative criterion when set by legislation.

6.2 General observations

Twenty seven NRLs from twenty five Member States and Switzerland, and nine OCLs (from Germany and Italy) registered to the exercise. One NRL and one OCL did not report results. The 34 reporting laboratories, representing most of the EU Member States (except Finland, Latvia, Malta and Romania), reported results for all the 8 measurands; one of them reported "less than" values for the PBT cyclic dimer in both solutions.

6.3 Laboratory results and scorings

6.3.1 Performances

Annexes 7 to 17 present the reported results as tables and graphs for each measurand. 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 [9].

The laboratory performance for the determination of the PET cyclic dimer, PBT cyclic dimer, and PET cyclic trimer in the solutions 1 and 2 was assessed using the *z* and ζ scores. The ISO 13528:2015 [5] recommendation was applied for the PBT cyclic trimer (for which $u(x_{pt}) > 0.3\sigma_{pt}$, cf. Table 1b) and the z' score was used as performance score instead of z.

Figure 1 presents the laboratory performances for the mass fractions investigated in the two solutions. Most of the participants having reported results performed satisfactorily for the four measurands in solution 1: 79 % and above for the *z* or z' score, and 68 % and above for the ζ scores. Slightly lower performances are observed for solution 2: 71 % and above performed satisfactorily according to the *z* or z' score and 51 % and above for the ζ scores. Solution 2 may be more challenging to analyse due to the potential presence of interfering substances resulting from the migration experiment.

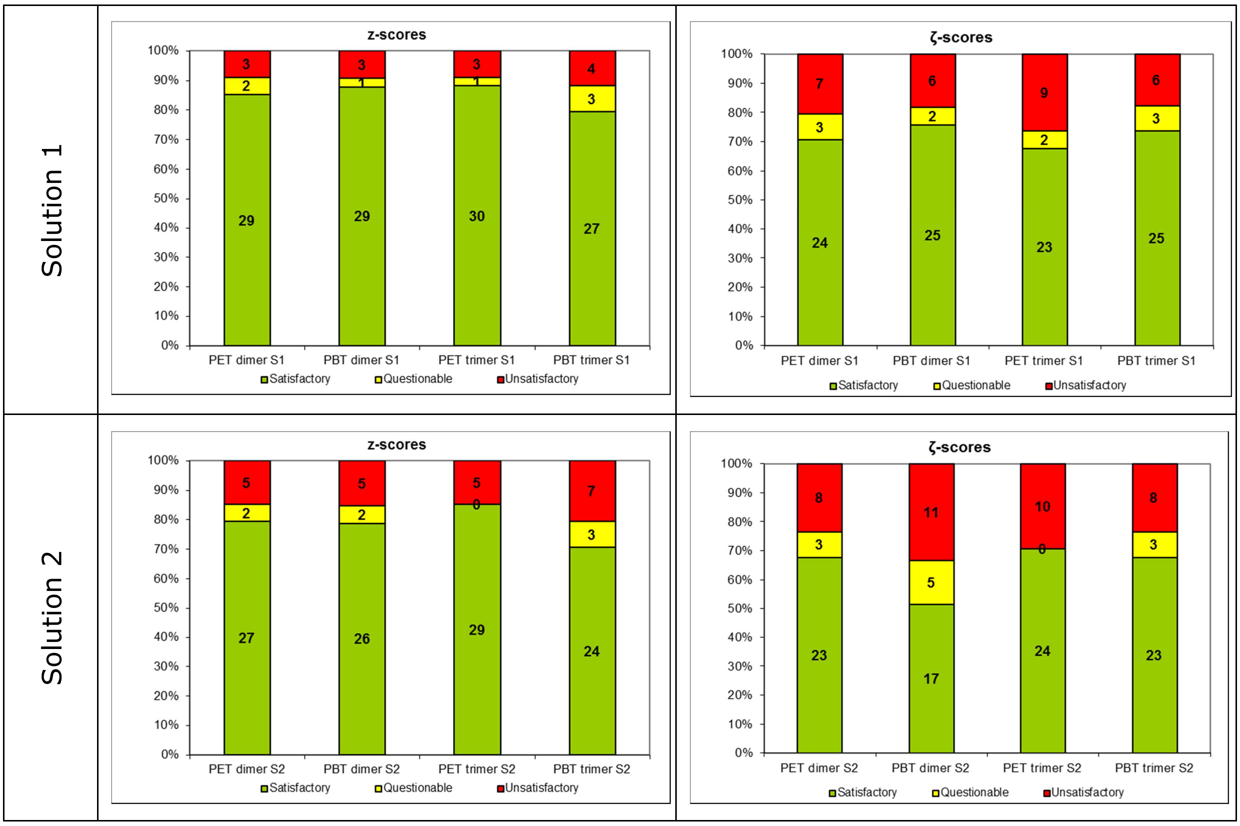

Figure 1: Overview of laboratory performance per measurand according to *z* and *ζ* scores, for the PET cyclic dimer, PBT cyclic dimer, PET cyclic trimer and z' and *ζ* scores for the PBT cyclic trimer in solutions 1 and 2.
Corresponding number of laboratories included in the graph.
Satisfactory, questionable and unsatisfactory performances indicated in green, yellow and red, respectively.

6.3.2 Truncated values

Laboratory N-07 reported truncated values for the PBT cyclic dimer in both solutions ("*less than* 0.04 mg kg^{-1}"), corresponding to the limits of quantification (LOQ) or limits of detection (LOD) of the applied method. These values could not be included in the data evaluation. However, they were compared with the corresponding $x_{pt} - U(x_{pt})$. Since the reported truncated values were lower than these values, the statement was considered incorrect. The laboratory should have detected the PBT cyclic dimer in the two solutions.

6.3.3 Measurement uncertainties

Figure 2 presents the measurement uncertainty evaluation. Most of the participants underestimated their measurement uncertainty (Case "b" (blue): $u(x_i) < u(x_{pt})$). This is due to the fact that they estimated their uncertainty from measurement replicates only (Table 2). This is further confirmed by the larger number of unsatisfactory results assessed according to the ζ score.

More specifically, laboratories N-05, N-30 and N-36 did not provide any measurement uncertainty statement, while laboratory N-14 must have erroneously reported its MU in %, instead of mg kg^{-1}. Furthermore, laboratories N-06 and O-35 did not report any coverage factor and were attributed a k of 1.73 (=√3).

The EURL-FCM will organise a training course on measurement uncertainty dedicated to the network of NRLs following the EURL-NRL-FCM plenary meeting in 2018.

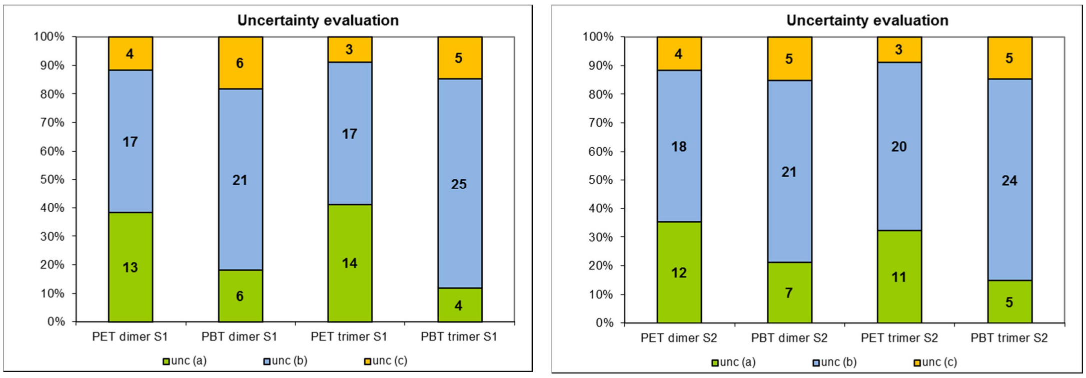

Figure 2: Review of uncertainties reported per measurand result. Corresponding number of laboratories indicated in the graph. Case "a" (green): $u(x_{pt}) \leq u(x_i) \leq \sigma_{pt}$; Case "b" (blue): $u(x_i) < u(x_{pt})$; Case "c" (orange): $u(x_i) > \sigma_{pt}$

6.3.4 Additional information extracted from the questionnaire

The questionnaire was answered by all participants giving valuable information on the laboratories, their way of working and their analytical methods.

All participants, except one, stated that they have an ISO/IEC 17025 accreditation; N-12 referred only to ISO 9001.

The majority of participants (30 out of 34) did not have experience at all with the analysis of the four analytes investigated. However five of them stated they had experience with the analysis of other oligomers, such as polyamide 6 or polyamide 66. Due to the lack of experience in this type of analyses, most of the participants (22 out of 34) applied the Technical Note provided by the EURL-FCM (Annex 4). The experimental details presented in Annexes 15 and 16 do not indicate a direct correlation between the use of the technical note and the performance in the PT study.

The majority of the participants (23 out of 34) 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.

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

Approach	N° of labs
According to ISO-GUM	2
From known uncertainty of a standard method	0
Derived from a single-laboratory validation study	4
Measurement of replicates (precision)	14
Estimation based on judgment	3
Derived from interlaboratory comparison data	0
From Horwitz	4

7. Conclusion

The proficiency test FCM-18-01 was organised to assess the analytical capabilities of EU NRLs and OCLs to determine the mass fractions of the PET cyclic dimer, PBT cyclic dimer, PET cyclic trimer and PBT cyclic trimer in spiked solutions of food simulant D1.

The overall performance of the participants in the determination of these oligomers was satisfactory despite the lack of experience in this type of analysis for most of the participants. Consequently, most of the participants underestimated their measurement uncertainty. After this PT, participants may consider validating these methods of analysis.

Acknowledgements

The thirty four laboratories listed hereafter are kindly acknowledged for their participation in the PT.

Organisation	Country
AGES Austrian Agency for Health & Food Safety	Austria
Scientific Institute of Public Health	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
Danish Veterinary and Food Administration	Denmark
Health Board	Estonia
SCL Service Commun des Laboratoires	France
Landesbetrieb Hessisches Landeslabor	Germany
German Federal Institute for Risk Assessment	Germany
CVUA-MEL	Germany
Thueringer Landesamt fuer Verbraucherschutz	Germany
Landesamt für Verbraucherschutz Sachsen-Anhalt	Germany
Chemisches und Veterinäruntersuchungsamt Stuttgart	Germany
Landesuntersuchungsanstalt für das Gesundheits- und Veterinärwesen Sachsen	Germany
General Chemical State Laboratory	Greece
National Food Chain Safety Office	Hungary
Public Analyst's Laboratory	Ireland
Istituto Superiore di Sanità	Italy
Istituto zooprofilattico sperimentale lombardia emilia romagna	Italy
Settore Laboratorio -APPA	Italy
National Public Health Surveillance Laboratory	Lithuania
Laboratoire National de Santé	Luxembourg
NVWA	Netherlands
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-AECOSAN	Spain
National Food Agency	Sweden
Kantonales Labor Zürich; NRL-CH	Switzerland
Fera Science Ltd	United Kingdom

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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(2018)1503729 - 19/03/2018

Geel, 19 March 2018

(sent by e-mail)

Subject: Invitation to participate in FCM-18-01 PT round

Dear National Reference Laboratory representative,

On behalf of the EURL for Food Contact Materials, we would like to invite you to participate in the Proficiency Test round FCM-18-01 "Determination of oligomers in food simulant D1".

The PT fulfils the EURL-FCM mandate under Regulation (EC) No 882/2004 and 2017/625.

According to Regulation (EC) No 882/2004 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=1981

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 2018.

Please inform us how many OCLs would be interested in participating, together with their addresses, contact number and responsible person. They should register electronically by using the link above.

Samples will be dispatched at 15th of May 2018.

The deadline for submission of results is the 15th of June 2018.

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

Kind regards,

/signed electronically in Ares/	/signed electronically in Ares/
Dr. P. Dehouck	Dr. E. Hoekstra
FCM-18-01 PT Coordinator	Operator Manager EURL-FCM

Cc: Hendrik 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, 15th May 2018
JRC.F.5/PdH/EH/mt/ARES(2018)18-037/

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

Subject: Participation in FCM-18-01 - Determination of the mass fractions of oligomers in food simulant D1

Dear «Title» «Surname»,

Thank you for participating in the FCM-18-01 proficiency test (PT) for the **"Determination of the mass fractions of oligomers in food simulant D1".** This PT is organised in support to *Regulation 10/2011 on plastic materials and articles intended to come into contact with food.*

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

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

- two test solutions (1 and 2), each in one bottle with approx. 5 mL of food simulant D1;
- a calibration solution mixture containing the four target analytes at 20 µg mL^{-1} in a vial of approx. 1 mL of hexafluoro-2-propanol/ethanol;
- the "Confirmation of receipt" form.

Upon arrival of this parcel, please check:

1. immediately the temperature sensor/indicator inside the container, and report the exposure category in the confirmation receipt document.
2. whether the bottles and vial are undamaged after transport.

The test solutions and calibration solution mixture should be stored until analysis in a dark place at a temperature of -18 °C. Send us or email the "Confirmation of receipt" form within 3 days after receipt of the samples.

The measurands are PET cyclic dimer, PET cyclic trimer, PBT cyclic dimer and PBT cyclic trimer in food simulant D1.

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

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

Perform two or three independent measurements and report:

- 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.

A density of 1.0 g mL^{-1} shall be assumed as conversion factor for test solutions 1 and 2. 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 50 µL sample intakes leading to a recommended minimum sample intake level of 50 µL.

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 ILC coordinator as soon as possible before the reporting deadline.

The deadline for submission of results is **15/06/2018**.

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 (EC) No 882/2004 as amended by Regulation (EC) 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.

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. Pieter Dehouck
FCM-18-01 Coordinator

Cc: H. Emons (Head of Unit, Food & Feed Compliance, F.5),
E. Hoekstra (Operating Manager EURL-FCM)

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

Ispra, 15th May 2018
JRC.F.5/PdH/EH/mt/ARES(2018)18-038/

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

Subject: "Confirmation receipt" form - FCM-18-01 Oligomers in food simulant D1

Please return this form at your earliest convenience, to confirm that the package arrived well and specify the exposure category (none, brief, moderate, prolonged) indicated on the temperature sensor upon samples arrival to your laboratory. If samples are damaged or were exposed to inappropriate temperature during transport, please mention it below and contact us as soon as possible.

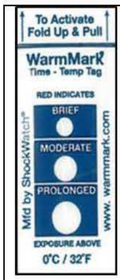
Date of package arrival ______________________________

Were the samples damaged? ☐ YES ☐ NO

Remarks ______________________________

____

T° sensor: Please tick an **X** into the correct box to indicate where red dots are visible.



NO COLOUR	☐	TEST ITEMS OK
BRIEF (RED)	☐	TEST ITEMS OK
MODERATE (RED)	☐	TEST ITEMS NOT OK
PROLONGED (RED)	☐	TEST ITEMS NOT OK

Signature

Thank you for returning this form by email to:

Dr. Pieter Dehouck
FCM-18-01 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: Technical Note

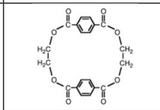
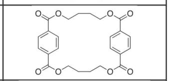
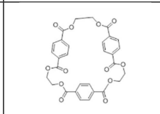
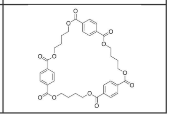
Technical Note

Ref. Ares(2018)1957065 - 12/04/2018

EURL-FCM analytical method for the determination of selected oligomers in food simulant D1 (could be used for the proficiency test FCM-2018-01)

1. Target analytes

in elution order according to the method below

Analyte	Molecular mass (Da)	CAS No.	Chemical name	Chemical formula	Chemical structure
PET 1st series cyclic dimer	384.34	24388-68-9	3,6,13,16-Tetraoxatricyclo[16.2.2.28,11]tetracosa-8,10,18,20,21,23-hexaene-2,7,12,17-tetrone	$C_{20}H_{16}O_8$	
PBT cyclic dimer*	440.44	63440-93-7	3,8,15,20-Tetraoxatricyclo[20.2.2.210,13]octacosa-10,12,22,24,25,27-hexaene-2,9,14,21-tetrone	$C_{24}H_{24}O_8$	
PET 1st series cyclic trimer	576.50	7441-32-9	3,6,13,16,23,26-Hexaoxatetracyclo[26.2.2.28,11.218,21]hexatriaconta-8,10,18,20,28,30,31,33,35-nonaene-2,7,12,17,22,27-hexone	$C_{30}H_{24}O_{12}$	
PBT cyclic trimer*	660.66	63440-94-8	3,8,15,20,27,32-Hexaoxatetracyclo[32.2.2.210,13.222,25]dotetraconta-10,12,22,24,34,36,37,39,41-nonaene-2,9,14,21,26,33-hexone	$C_{36}H_{36}O_{12}$	

* Part of the FCM 885 (Regulation EU No 10/2011).

2. Instrumentation and analytical column

- Agilent HPLC 1200 system
- Agilent UV detector
- Column: Agilent Zorbax Eclipse XDB-C18 (150 x 4.6 mm, 5 µm)

3. Method

A. *Mobile phase:*

1. Solvent A: Acetonitrile HPLC Chromasolv gradient grade
2. Solvent B: Ultrapure Water

B. *Flow*: 2.0 mL/min

C. *Elution*: Apply the linear gradient described hereafter

Time (min) From	Time (min) To	Solvent A (%)	Solvent B (%)	Flow (mL/min)	Notes
0.0	6.0	40	60	2.0	Isocratic
6.0	15.0	75	25	2.0	Linear gradient
15.0	19.0	75	25	2.0	Isocratic
19.0	21.0	95	5	2.0	Linear gradient
21.0	22.0	40	60	2.0	Equilibration
22.0	23.0	40	60	2.0	Equilibration

D. *Injection volume:* 50 µL

E. *Column temperature:* Analytical column temperature set to 40°C (± 1.0 °C).

F. *UV detection wavelength*: 240 nm

Annex 5: Questionnaire

☐ 1. **Are you a National Reference Laboratory (NRL)?** *[Q:110311: CHECKBOX]*

☐ a) Yes *[A:348]*
☐ b) No *[A:349]*

☐ 1.1. If "No" have you been nominated by your NRL? *[Q:110312: CHECKBOX]*

☐ a) Yes *[A:348]*
☐ b) No *[A:349]*

☐ 1.1.1. If "Yes" please identify your NRL *[Q:110313: TEXT]*

☐ 2. **Analytical method(put "X" where applicable)** *[Q:110316: CUSTOM TABLE]*

Analytical method

Questions/Response table	Standard method?	Validated method?	Accredited method?
PBT cyclic dimer			
PBT cyclic trimer			
PET cyclic dimer			
PET cyclic trimer			

☐ 2.1. Did you use the protocol that was sent to you? *[Q:110325: CHECKBOX]*

☐ a) Yes *[A:348]*
☐ b) No *[A:349]*

☐ 2.2. If "No" describe briefly the analytical method used. *[Q:110326: TEXT]*

☐ 3. **Analytical method (LOD, mobile phase composition, detection, column)** *[Q:110317: CUSTOM TABLE]*

Analytical method (details)

Questions/Response table	LOD (mg/kg)	Mobile phase used	Injection volume (µL)	Detection used	Column used
PBT cyclic dimer					
PBT cyclic trimer					
PET cyclic dimer					
PET cyclic trimer					

4. **Does your laboratory carry this type of analysis on a regular basis?** *[Q:110318: CUSTOM TABLE]*

Laboratory experience (samples per year)

Questions/Response table	1) 0-50	2) 50-250	3) 250-1000	4) > 1000	5) Never
PBT cyclic dimer					
PBT cyclic trimer					
PET cyclic dimer					
PET cyclic trimer					
Other oligomers					

4.1. If "other oligomers", specify which ones. *[Q:110334: TEXT]*

5. **What was the basis for your measurement uncertainty evaluation** *[Q:110319: CUSTOM TABLE]*

Measurement uncertainty evaluation (use a to g)

Questions/Response table	PBT cyclic dimer	PBT cyclic trimer	PET cyclic dimer	PET cyclic trimer
a) Uncertainty budget (ISO GUM)				
b) Known uncertainty of standard method (ISO 21748)				
c) From in-house method validation				
d) Measurement of replicates (precision)				
e) Evaluation based on judgment				
f) From interlaboratory comparison				
g) Other (please specify)				

6. **Do you usually provide an uncertainty statement to your customers?** *[Q:110320: CHECKBOX]*

- a) Yes *[A:348]*
- b) No *[A:349]*

7. **Does your laboratory have a quality management system?** *[Q:110321: CHECKBOX]*

- a) Yes *[A:348]*
- b) No *[A:349]*

8. **If "Yes" based on which standard?** *[Q:110322: CHECKBOX]*

- a) ISO 17025 *[A:350]*
- b) ISO 9001 series *[A:2121]*
- c) Other *[A:352]*

9. **Does your laboratory participate in interlaboratory comparisons for this type of analysis?** *[Q:110323: CHECKBOX]*

- a) Yes *[A:348]*
- b) No *[A:349]*

10. **Do you have any comments? Let us know!** *[Q:110324: TEXT]*

Annex 6: Homogeneity and stability results

6.1 Homogeneity study (all values in mg kg^{-1})

Solution 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	0.0570	0.0559	0.0533	0.0543	0.0537	0.0544	0.0502	0.0502
2	0.0548	0.0570	0.0543	0.0554	0.0544	0.0537	0.0502	0.0502
3	0.0559	0.0570	0.0554	0.0554	0.0544	0.0544	0.0493	0.0518
4	0.0559	0.0548	0.0554	0.0554	0.0544	0.0537	0.0493	0.0502
5	0.0559	0.0581	0.0554	0.0554	0.0544	0.0544	0.0493	0.0502
6	0.0570	0.0570	0.0543	0.0543	0.0544	0.0544	0.0518	0.0518
7	0.0570	0.0559	0.0554	0.0564	0.0544	0.0537	0.0518	0.0526
8	0.0548	0.0570	0.0554	0.0554	0.0537	0.0544	0.0518	0.0510
9	0.0559	0.0559	0.0554	0.0543	0.0544	0.0537	0.0493	0.0518
10	0.0548	0.0559	0.0554	0.0554	0.0537	0.0537	0.0518	0.0510
Mean	0.0562		0.0551		0.0541		0.0508	
s_x	0.0006		0.0006		0.0002		0.0009	
s_w	0.0010		0.0005		0.0004		0.0009	
s_s	0		0.0005		0		0.0006	
σ_{pt}	0.0110		0.0108		0.0106		0.0100	
$0.3 * \sigma_{pt}$	0.0033		0.0032		0.0032		0.0030	
$s_s \leq 0.3 * \sigma_{pt}$	passed		passed		passed		passed	

Solution 2								
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	0.0570	0.0581	0.0721	0.0731	0.1668	0.1675	0.0543	0.0551
2	0.0570	0.0592	0.0679	0.0721	0.1661	0.1668	0.0543	0.0526
3	0.0604	0.0592	0.0721	0.0710	0.1682	0.1668	0.0559	0.0551
4	0.0592	0.0581	0.0721	0.0710	0.1682	0.1682	0.0559	0.0526
5	0.0559	0.0570	0.0742	0.0689	0.1675	0.1661	0.0535	0.0551
6	0.0559	0.0570	0.0742	0.0710	0.1675	0.1675	0.0543	0.0535
7	0.0604	0.0592	0.0710	0.0721	0.1675	0.1661	0.0543	0.0551
8	0.0604	0.0559	0.0721	0.0700	0.1675	0.1661	0.0535	0.0518
9	0.0581	0.0581	0.0710	0.0721	0.1668	0.1661	0.0535	0.0543
10	0.0592	0.0581	0.0721	0.0721	0.1682	0.1675	0.0559	0.0551
Mean	0.0582		0.0716		0.1672		0.0543	
s_x	0.0012		0.0008		0.0006		0.0009	
s_w	0.0013		0.0018		0.0007		0.0011	
s_s	0.0007		0		0.0003		0.0004	
σ_{pt}	0.0117		0.0141		0.0329		0.0102	
$0.3 * \sigma_{pt}$	0.0035		0.0042		0.0099		0.0031	
$s_s \leq 0.3 * \sigma_{pt}$	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,

6.2 Stability study (at -18 °C and 4 °C, time in weeks (w), all values in mg kg^{-1})

Solution 1								
	-18 °C			4 °C			Stability criteria [a]	Assessment
		Time			Time			
	Bottle ID	0 w	8 w	Bottle ID	0 w	8 w		
PET Dimer	1	0.0548	0.0537	1	0.0537	0.0548	Passed	Stable
	2	0.0548	0.0537	2	0.0525	0.0537		
PBT Dimer	1	0.0543	0.0533	1	0.0543	0.0533	Passed	Stable
	2	0.0554	0.0554	2	0.0533	0.0543		
PET Trimer	1	0.0537	0.0523	1	0.0537	0.0523	Passed	Stable
	2	0.0530	0.0530	2	0.0530	0.0530		
PBT Trimer	1	0.0510	0.0493	1	0.0493	0.0502	passed	Stable
	2	0.0502	0.0510	2	0.0493	0.0502		

Solution 2								
	-18 °C			4 °C			Stability criteria [a]	Assessment
		Time			Time			
	Bottle ID	0 w	8 w	Bottle ID	0 w	8 w		
PET Dimer	1	0.0570	0.0581	1	0.0548	0.0548	Passed	Stable
	2	0.0570	0.0537	2	0.0559	0.0559		
PBT Dimer	1	0.0689	0.0689	1	0.0710	0.0689	Passed	Stable
	2	0.0710	0.0710	2	0.0679	0.0668		
PET Trimer	1	0.1639	0.1639	1	0.1632	0.1625	Passed	Stable
	2	0.1647	0.1647	2	0.1632	0.1632		
PBT Trimer	1	0.0510	0.0526	1	0.0518	0.0510	passed	Stable
	2	0.0510	0.0510	2	0.0502	0.0526		

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

Annex 7: Results for PET cyclic dimer in solution 1

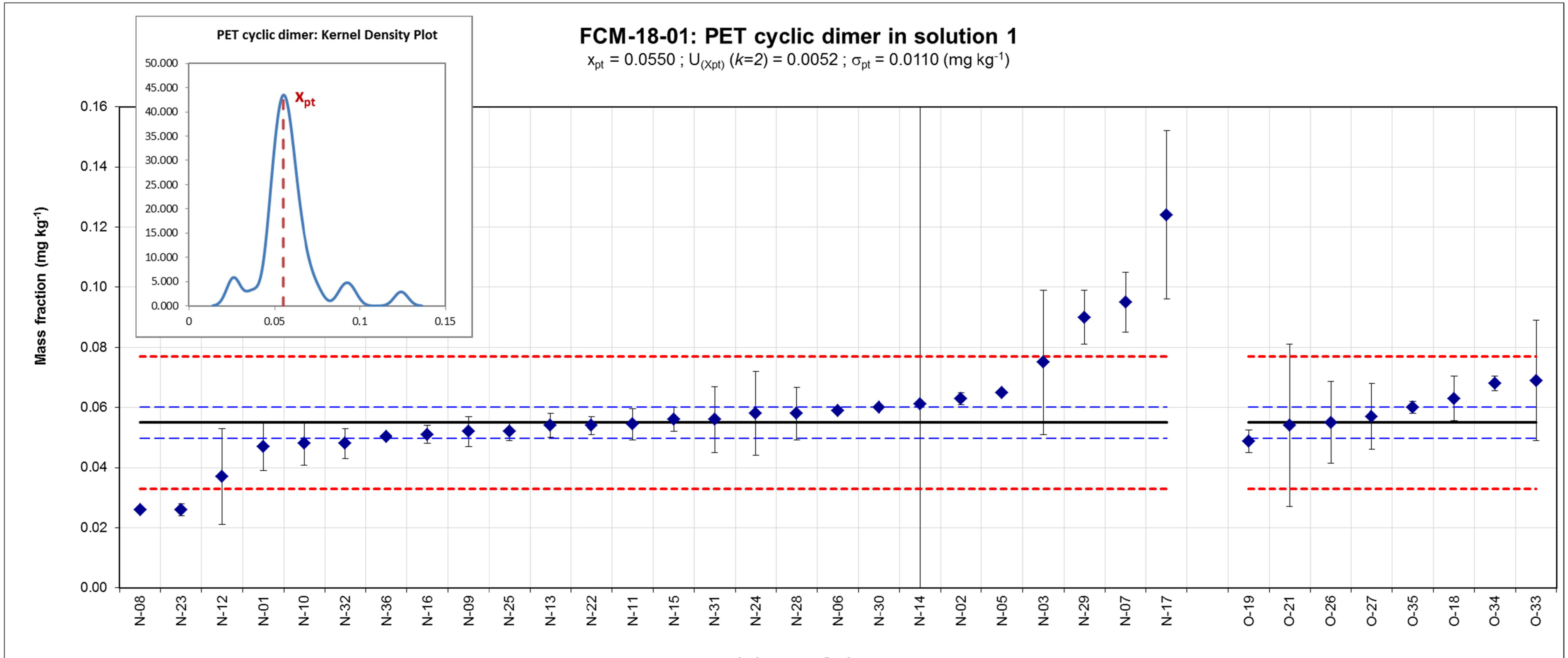
Assigned range: x_{pt} = 0.0550 ± 0.0052 $U(x_{pt}, k = 2.0)$; σ_{pt} = 0.0110 (all values in mg kg^{-1})

Lab Code	X_{lab}	U_{lab}	k	technique	u_{lab}	z-score	zeta-score	uncert.
N-01	0.047	0.008	2	HPLC-UV	0.004	-0.73	-1.68	a
N-02	0.063	0.002	2	HPLC-UV	0.001	0.73	2.87	b
N-03	0.075	0.024	2	HPLC-DAD	0.012	1.82	1.63	c
N-05	0.065			HPLC-DAD	0	0.91	3.85	b
N-06	0.059	0.001	√3	HPLC-UV	0.00057735	0.36	1.50	b
N-07	0.095	0.01	2	HPLC-UV	0.005	3.64	7.10	a
N-08	0.026	0.001	2	HPLC-DAD	0.0005	-2.64	-10.95	b
N-09	0.052	0.005	2	HPLC-DAD	0.0025	-0.27	-0.83	b
N-10	0.048	0.0071	2	HPLC-DAD	0.00355	-0.64	-1.59	a
N-11	0.0544	0.00528	2	HPLC-UV	0.00264	-0.05	-0.16	a
N-12	0.037	0.016	2	LC-MS	0.008	-1.64	-2.14	a
N-13	0.054	0.004	2	HPLC-DAD	0.002	-0.09	-0.30	b
N-14	0.0611	10	2	HPLC-UV	5	0.55	0.00	c
N-15	0.056	0.004	2	HPLC-UV	0.002	0.09	0.30	b
N-16	0.051	0.003	2	HPLC-UV	0.0015	-0.36	-1.33	b
N-17	0.124	0.028	2.2	LC-MS/MS	0.012727273	6.27	5.31	c
N-22	0.054	0.003	2	HPLC-DAD	0.0015	-0.09	-0.33	b
N-23	0.026	0.002	2	HPLC-DAD	0.001	-2.64	-10.41	b
N-24	0.058	0.014	2	HPLC-UV	0.007	0.27	0.40	a
N-25	0.052	0.003	2	HPLC-DAD	0.0015	-0.27	-1.00	b
N-28	0.058	0.0087	2	HPLC-UV	0.00435	0.27	0.59	a
N-29	0.09	0.009	2	HPLC-DAD	0.0045	3.18	6.73	a
N-30	0.06			HPLC-DAD	0	0.45	1.92	b
N-31	0.056	0.011	2	HPLC-DAD	0.0055	0.09	0.16	a
N-32	0.048	0.005	2	HPLC-DAD	0.0025	-0.64	-1.94	b
N-36	0.0503			HPLC-DAD	0	-0.43	-1.81	b
O-18	0.063	0.0074	2	HPLC-DAD	0.0037	0.73	1.77	a
O-19	0.04877	0.00374	3.182	HPLC-DAD	0.001175361	-0.57	-2.18	b
O-21	0.054	0.027	2	HPLC-DAD	0.0135	-0.09	-0.07	c
O-26	0.055	0.0136	2	HPLC-DAD	0.0068	0.00	0.00	a
O-27	0.057	0.011	3	HPLC-DAD	0.003666667	0.18	0.44	a
O-33	0.069	0.02	2	LC-MS/MS	0.01	1.27	1.35	a
O-34	0.068	0.0025	2	HPLC-DAD	0.00125	1.18	4.51	b
O-35	0.06	0.002	√3	HPLC-DAD	0.001154701	0.45	1.76	b

[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 $k = \sqrt{3}$,

[b] Performance scoring: satisfactory (green), questionable (yellow), unsatisfactory (red),

[c] a: $u_{min}(u(X_{pt})) \leq u_{lab} \leq u_{max}(\sigma_{pt})$; b: $u_{lab} < u_{min}$; and c: $u_{lab} > u_{max}$



Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; Reference interval ($x_{pt} \pm U_{(xpt)}$): dashed blue lines; Target interval ($x_{pt} \pm 2\sigma_{pt}$): dotted red lines.

Annex 8: Results for PBT cyclic dimer in solution 1

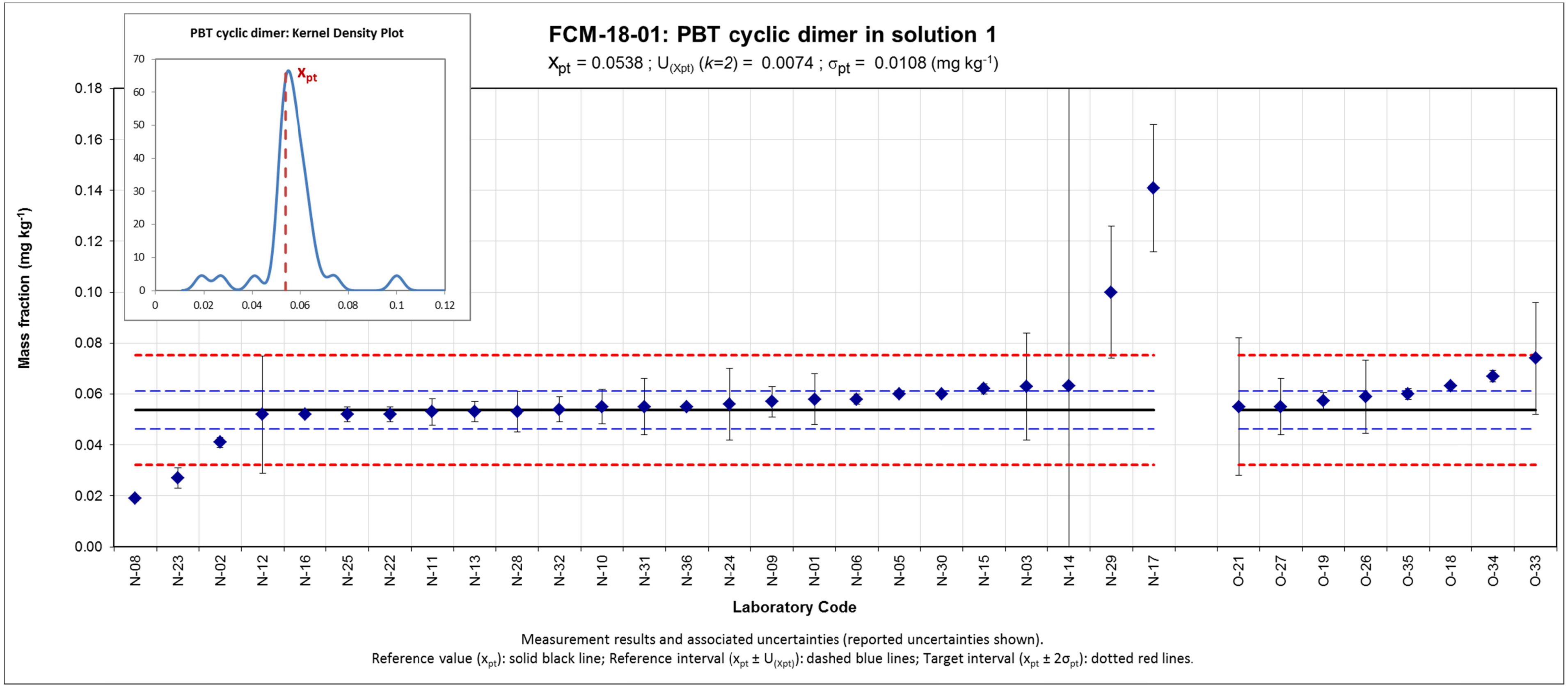
Assigned range: x_{pt} = 0.0538 ± 0.0074 $U(x_{pt}, k = 2.0)$; σ_{pt} = 0.0108 (all values in mg kg^{-1})

Lab Code	X_{lab}	U_{lab}	k	technique	u_{lab}	z-score	zeta-score	uncert.
N-01	0.058	0.01	2	HPLC-UV	0.005	0.39	0.68	a
N-02	0.041	0.002	2	HPLC-UV	0.001	-1.19	-3.34	b
N-03	0.063	0.021	2	HPLC-DAD	0.0105	0.85	0.83	a
N-05	0.0599			HPLC-DAD	0	0.56	1.65	b
N-06	0.058	0.002	√3	HPLC-UV	0.001155	0.39	1.08	b
N-07	<0.04			HPLC-UV				
N-08	0.019	0.001	2	HPLC-DAD	0.0005	-3.22	-9.32	b
N-09	0.057	0.006	2	HPLC-DAD	0.003	0.30	0.67	b
N-10	0.055	0.0068	2	HPLC-DAD	0.0034	0.11	0.24	b
N-11	0.053	0.00514	2	HPLC-UV	0.00257	-0.07	-0.18	b
N-12	0.052	0.023	2	LC-MS	0.0115	-0.17	-0.15	c
N-13	0.053	0.004	2	HPLC-DAD	0.002	-0.07	-0.19	b
N-14	0.0633	10	2	HPLC-UV	5	0.88	0.00	c
N-15	0.062	0.002	2	HPLC-UV	0.001	0.76	2.14	b
N-16	0.052	0.001	2	HPLC-UV	0.0005	-0.17	-0.48	b
N-17	0.141	0.025	2.2	LC-MS/MS	0.011364	8.07	7.30	c
N-22	0.052	0.003	2	HPLC-DAD	0.0015	-0.17	-0.45	b
N-23	0.027	0.004	2	HPLC-DAD	0.002	-2.48	-6.37	b
N-24	0.056	0.014	2	HPLC-UV	0.007	0.20	0.28	a
N-25	0.052	0.003	2	HPLC-DAD	0.0015	-0.17	-0.45	b
N-28	0.053	0.008	2	HPLC-UV	0.004	-0.07	-0.15	a
N-29	0.1	0.026	2	HPLC-DAD	0.013	4.28	3.42	c
N-30	0.06			HPLC-DAD	0	0.57	1.68	b
N-31	0.055	0.011	2	HPLC-DAD	0.0055	0.11	0.18	a
N-32	0.054	0.005	2	HPLC-DAD	0.0025	0.02	0.04	b
N-36	0.055			HPLC-DAD	0	0.11	0.32	b
O-18	0.0633	0.001	2	HPLC-DAD	0.0005	0.88	2.54	b
O-19	0.0573	0.00325	2.776	HPLC-DAD	0.001171	0.32	0.90	b
O-21	0.055	0.027	2	HPLC-DAD	0.0135	0.11	0.09	c
O-26	0.0589	0.0144	2	HPLC-DAD	0.0072	0.47	0.63	a
O-27	0.055	0.011	3	HPLC-DAD	0.003667	0.11	0.23	b
O-33	0.074	0.022	2	LC-MS/MS	0.011	1.87	1.74	c
O-34	0.067	0.0022	2	HPLC-DAD	0.0011	1.22	3.42	b
O-35	0.06	0.002	√3	HPLC-DAD	0.001155	0.57	1.60	b

[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 scoring: satisfactory (green), questionable (yellow), unsatisfactory (red),

[c] a: $u_{min}(u(X_{pt})) \leq u_{lab} \leq u_{max}(\sigma_{pt})$; b: $u_{lab} < u_{min}$; and c: $u_{lab} > u_{max}$


FCM-18-01: PBT cyclic dimer in solution 1
Xpt = 0.0538 ; U(Xpt) (k=2) = 0.0074 ; σpt = 0.0108 (mg kg-1)
PBT cyclic dimer: Kernel Density Plot
Xpt
Mass fraction (mg kg-1)
Laboratory Code
N-08 N-23 N-02 N-12 N-16 N-25 N-22 N-11 N-13 N-28 N-32 N-10 N-31 N-36 N-24 N-09 N-01 N-06 N-05 N-30 N-15 N-03 N-14 N-29 N-17
O-21 O-27 O-19 O-26 O-35 O-18 O-34 O-33
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (xpt): solid black line; Reference interval (xpt ± U(Xpt)): dashed blue lines; Target interval (xpt ± 2σpt): dotted red lines.

Annex 9: Results for PET cyclic trimer in solution 1

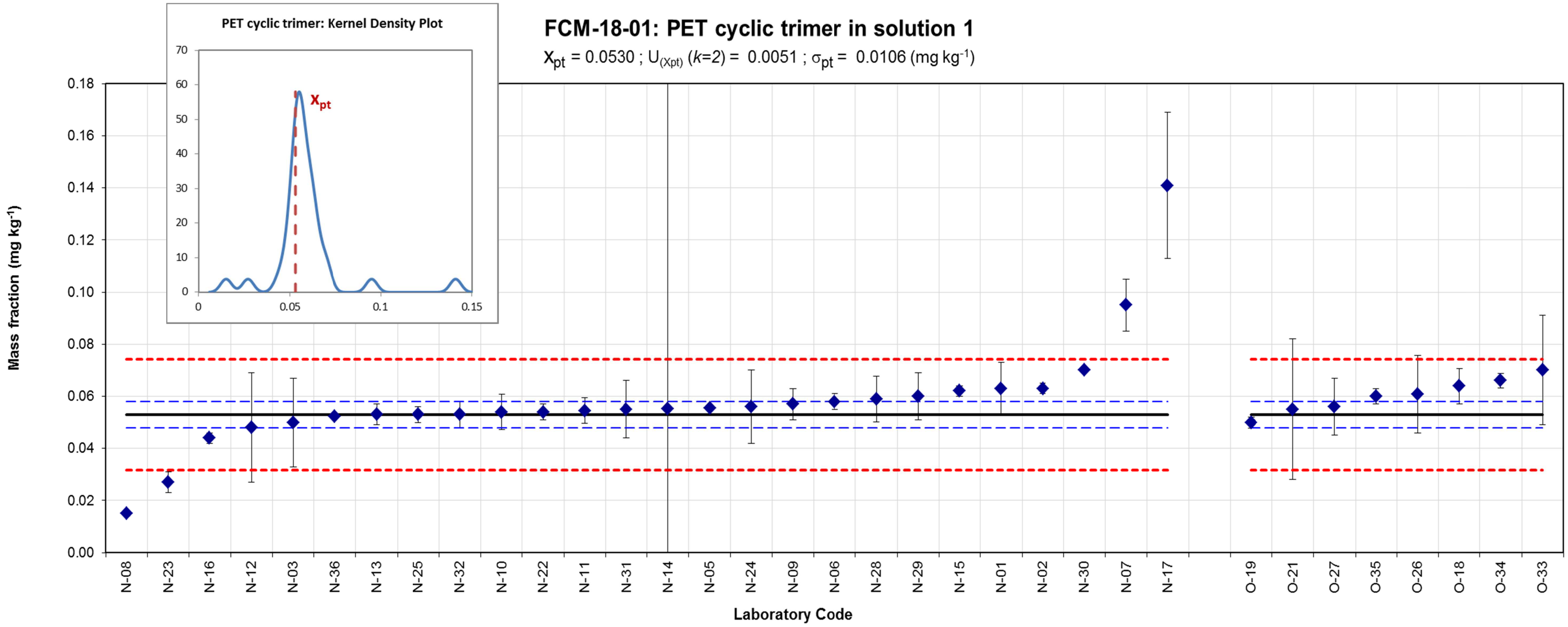
Assigned range: x_{pt} = 0.0530 ± 0.0051 $U(x_{pt}, k = 2.0)$; σ_{pt} = 0.0106 (all values in mg kg^{-1})

Lab Code	X_{lab}	U_{lab}	k	technique	u_{lab}	z-score	zeta-score	uncert.
N-01	0.063	0.01	2	HPLC-UV	0.005	0.94	1.78	a
N-02	0.063	0.002	2	HPLC-UV	0.001	0.94	3.65	b
N-03	0.05	0.017	2	HPLC-DAD	0.0085	-0.28	-0.34	a
N-05	0.0555			HPLC-DAD	0	0.24	0.98	b
N-06	0.058	0.003	√3	HPLC-UV	0.001732	0.47	1.62	b
N-07	0.095	0.01	2	HPLC-UV	0.005	3.96	7.48	a
N-08	0.015	0.001	2	HPLC-DAD	0.0005	-3.58	-14.62	b
N-09	0.057	0.006	2	HPLC-DAD	0.003	0.38	1.02	a
N-10	0.054	0.0067	2	HPLC-DAD	0.00335	0.09	0.24	a
N-11	0.0545	0.00496	2	HPLC-UV	0.00248	0.14	0.42	b
N-12	0.048	0.021	2	LC-MS	0.0105	-0.47	-0.46	a
N-13	0.053	0.004	2	HPLC-DAD	0.002	0.00	0.00	b
N-14	0.0551	15	2	HPLC-UV	7.5	0.20	0.00	c
N-15	0.062	0.002	2	HPLC-UV	0.001	0.85	3.29	b
N-16	0.044	0.002	2	HPLC-UV	0.001	-0.85	-3.29	b
N-17	0.141	0.028	2.2	LC-MS/MS	0.012727	8.30	6.78	c
N-22	0.054	0.003	2	HPLC-DAD	0.0015	0.09	0.34	b
N-23	0.027	0.004	2	HPLC-DAD	0.002	-2.45	-8.02	b
N-24	0.056	0.014	2	HPLC-UV	0.007	0.28	0.40	a
N-25	0.053	0.003	2	HPLC-DAD	0.0015	0.00	0.00	b
N-28	0.059	0.0088	2	HPLC-UV	0.0044	0.57	1.18	a
N-29	0.06	0.009	2	HPLC-DAD	0.0045	0.66	1.35	a
N-30	0.07			HPLC-DAD	0	1.60	6.67	b
N-31	0.055	0.011	2	HPLC-DAD	0.0055	0.19	0.33	a
N-32	0.053	0.005	2	HPLC-DAD	0.0025	0.00	0.00	b
N-36	0.0523			HPLC-DAD	0	-0.07	-0.27	b
O-18	0.0639	0.0067	2	HPLC-DAD	0.00335	1.03	2.59	a
O-19	0.04986	0.00211	2.776	HPLC-DAD	0.00076	-0.30	-1.18	b
O-21	0.055	0.027	2	HPLC-DAD	0.0135	0.19	0.15	c
O-26	0.0608	0.0148	2	HPLC-DAD	0.0074	0.74	1.00	a
O-27	0.056	0.011	3	HPLC-UV	0.003667	0.28	0.67	a
O-33	0.07	0.021	2	LC-MS/MS	0.0105	1.60	1.57	a
O-34	0.066	0.0027	2	HPLC-DAD	0.00135	1.23	4.51	b
O-35	0.06	0.003	√3	HPLC-DAD	0.001732	0.66	2.27	b

[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 scoring: satisfactory (green), questionable (yellow), unsatisfactory (red),

[c] a: $u_{min}(u(X_{pt})) \leq u_{lab} \leq u_{max}(\sigma_{pt})$; b: $u_{lab} < u_{min}$; and c: $u_{lab} > u_{max}$



Measurement results and associated uncertainties (reported uncertainties shown).

Reference value (x_{pt}): solid black line; Reference interval ($x_{pt} \pm U_{(Xpt)}$): dashed blue lines; Target interval ($x_{pt} \pm 2\sigma_{pt}$): dotted red lines.

Annex 10: Results for PBT cyclic trimer in solution 1

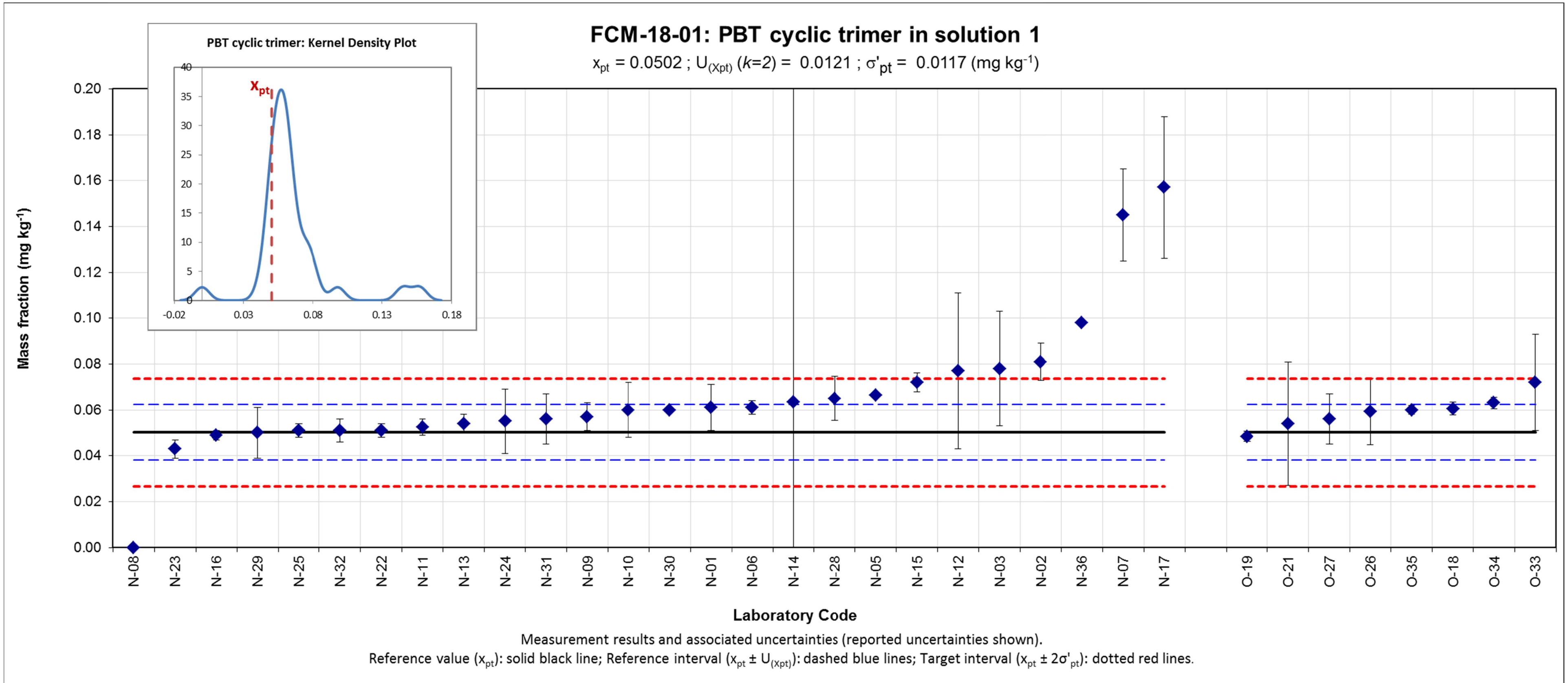
Assigned range: x_{pt} = 0.0502 ± 0.0121 $U(x_{pt}, k = 2.0)$; σ_{pt} = 0.0100 (all values in mg kg^{-1})

Lab Code	X_{lab}	U_{lab}	k	technique	u_{lab}	z'-score	zeta-score	uncert.
N-01	0.061	0.01	2	HPLC-UV	0.005	0.92	1.38	b
N-02	0.081	0.008	2	HPLC-UV	0.004	2.63	4.25	b
N-03	0.078	0.025	2	HPLC-DAD	0.0125	2.37	2.00	c
N-05	0.0663			HPLC-DAD	0	1.37	2.66	b
N-06	0.061	0.003	√3	HPLC-UV	0.001732	0.92	1.72	b
N-07	0.145	0.02	2	HPLC-UV	0.01	8.09	8.11	a
N-08	0	0	2	HPLC-DAD	0	-4.28	-8.30	b
N-09	0.057	0.006	2	HPLC-DAD	0.003	0.58	1.01	b
N-10	0.06	0.012	2	HPLC-DAD	0.006	0.84	1.15	b
N-11	0.0524	0.00351	2	HPLC-UV	0.001755	0.19	0.35	b
N-12	0.077	0.034	2	LC-MS	0.017	2.29	1.49	c
N-13	0.054	0.004	2	HPLC-DAD	0.002	0.32	0.60	b
N-14	0.0635	10	2	HPLC-UV	5	1.13	0.00	c
N-15	0.072	0.004	2	HPLC-UV	0.002	1.86	3.42	b
N-16	0.049	0.002	2	HPLC-UV	0.001	-0.10	-0.20	b
N-17	0.157	0.031	2.2	LC-MS/MS	0.014091	9.11	6.96	c
N-22	0.051	0.003	2	HPLC-DAD	0.0015	0.07	0.13	b
N-23	0.043	0.004	2	HPLC-DAD	0.002	-0.61	-1.13	b
N-24	0.055	0.014	2	HPLC-UV	0.007	0.41	0.52	a
N-25	0.051	0.003	2	HPLC-DAD	0.0015	0.07	0.13	b
N-28	0.065	0.0097	2	HPLC-UV	0.00485	1.26	1.91	b
N-29	0.05	0.011	2	HPLC-DAD	0.0055	-0.02	-0.02	b
N-30	0.06			HPLC-DAD	0	0.84	1.62	b
N-31	0.056	0.011	2	HPLC-DAD	0.0055	0.49	0.71	b
N-32	0.051	0.005	2	HPLC-DAD	0.0025	0.07	0.12	b
N-36	0.098			HPLC-DAD	0	4.08	7.90	b
O-18	0.0606	0.0028	2	HPLC-DAD	0.0014	0.89	1.67	b
O-19	0.04843	0.00225	2.776	HPLC-DAD	0.000811	-0.15	-0.29	b
O-21	0.054	0.027	2	HPLC-DAD	0.0135	0.32	0.26	c
O-26	0.0592	0.0145	2	HPLC-DAD	0.00725	0.77	0.95	a
O-27	0.056	0.011	3	HPLC-DAD	0.003667	0.49	0.82	b
O-33	0.072	0.021	2	LC-MS/MS	0.0105	1.86	1.80	a
O-34	0.063	0.0026	2	HPLC-DAD	0.0013	1.09	2.07	b
O-35	0.06	0.002	√3	HPLC-DAD	0.001155	0.84	1.59	b

[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 scoring: satisfactory (green), questionable (yellow), unsatisfactory (red),

[c] a : $u_{min}(u(X_{pt})) \leq u_{lab} \leq u_{max}(\sigma_{pt})$; b : $u_{lab} < u_{min}$; and c : $u_{lab} > u_{max}$



FCM-18-01: PBT cyclic trimer in solution 1

x_{pt} = 0.0502 ; $U_{(Xpt)}$ (k=2) = 0.0121 ; σ'_{pt} = 0.0117 (mg kg^{-1})

Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; Reference interval ($x_{pt} \pm U_{(Xpt)}$): dashed blue lines; Target interval ($x_{pt} \pm 2\sigma'_{pt}$): dotted red lines.

Annex 11: Results for PET cyclic dimer in solution 2

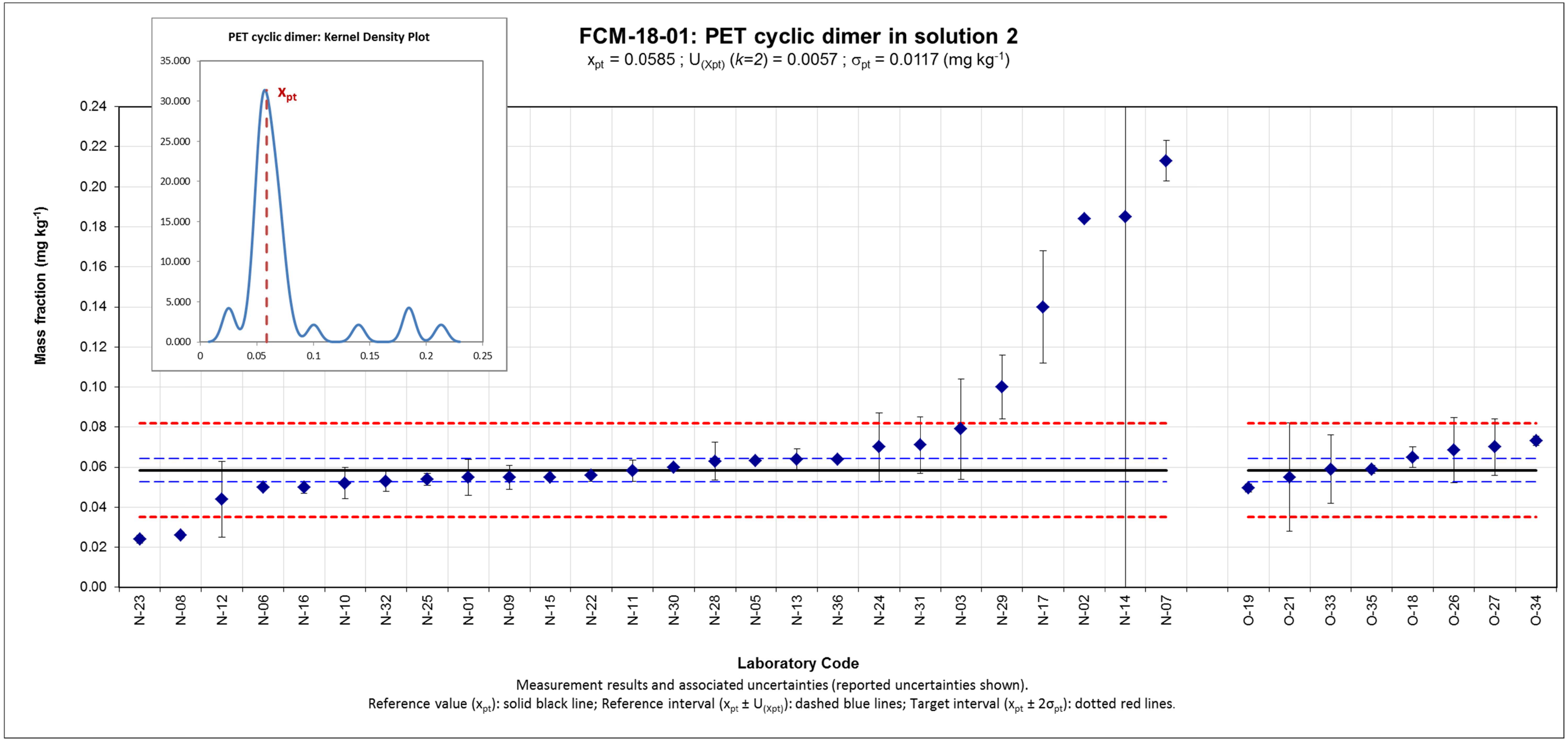
Assigned range: x_{pt} = 0.0585 ± 0.0057 $U(x_{pt}, k = 2.0)$; σ_{pt} = 0.0117 (all values in mg kg^{-1})

Lab Code	X_{lab}	U_{lab}	k	technique	u_{lab}	z-score	zeta-score	uncert.
N-01	0.055	0.009	2	HPLC-UV	0.0045	-0.30	-0.66	a
N-02	0.184	0.001	2	HPLC-UV	0.0005	10.73	43.37	b
N-03	0.079	0.025	2	HPLC-DAD	0.0125	1.75	1.60	c
N-05	0.0632			HPLC-DAD	0	0.40	1.65	b
N-06	0.05	0.001	√3	HPLC-UV	0.00057735	-0.73	-2.92	b
N-07	0.213	0.01	2	HPLC-UV	0.005	13.21	26.85	a
N-08	0.026	0.001	2	HPLC-DAD	0.0005	-2.78	-11.23	b
N-09	0.055	0.006	2	HPLC-DAD	0.003	-0.30	-0.85	a
N-10	0.052	0.0078	2	HPLC-DAD	0.0039	-0.56	-1.35	a
N-11	0.0583	0.00536	2	HPLC-UV	0.00268	-0.02	-0.05	b
N-12	0.044	0.019	2	LC-MS	0.0095	-1.24	-1.46	a
N-13	0.064	0.005	2	HPLC-DAD	0.0025	0.47	1.45	b
N-14	0.185	10	2	HPLC-UV	5	10.81	0.03	c
N-15	0.055	0.001	2	HPLC-UV	0.0005	-0.30	-1.21	b
N-16	0.05	0.003	2	HPLC-UV	0.0015	-0.73	-2.64	b
N-17	0.14	0.028	2.2	LC-MS/MS	0.012727273	6.97	6.25	c
N-22	0.056	0.003	2	HPLC-DAD	0.0015	-0.21	-0.78	b
N-23	0.024	0.002	2	HPLC-DAD	0.001	-2.95	-11.42	b
N-24	0.07	0.017	2	HPLC-UV	0.0085	0.98	1.28	a
N-25	0.054	0.003	2	HPLC-DAD	0.0015	-0.38	-1.40	b
N-28	0.063	0.0094	2	HPLC-UV	0.0047	0.38	0.82	a
N-29	0.1	0.016	2	HPLC-DAD	0.008	3.55	4.89	a
N-30	0.06			HPLC-DAD	0	0.13	0.53	b
N-31	0.071	0.014	2	HPLC-DAD	0.007	1.07	1.65	a
N-32	0.053	0.005	2	HPLC-DAD	0.0025	-0.47	-1.45	b
N-36	0.064			HPLC-DAD	0	0.47	1.93	b
O-18	0.0649	0.0052	2	HPLC-DAD	0.0026	0.55	1.66	b
O-19	0.04955	0.00208	2.571	HPLC-DAD	0.000809024	-0.76	-3.02	b
O-21	0.055	0.027	2	HPLC-DAD	0.0135	-0.30	-0.25	c
O-26	0.0685	0.0164	2	HPLC-DAD	0.0082	0.85	1.15	a
O-27	0.07	0.014	3	HPLC-DAD	0.004666667	0.98	2.10	a
O-33	0.059	0.017	2	LC-MS/MS	0.0085	0.04	0.06	a
O-34	0.073	0.0023	2	HPLC-DAD	0.00115	1.24	4.72	b
O-35	0.059	0.002	√3	HPLC-DAD	0.001154701	0.04	0.16	b

[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 scoring: satisfactory (green), questionable (yellow), unsatisfactory (red),

[c] a: $u_{min}(u(X_{pt})) \leq u_{lab} \leq u_{max}(\sigma_{pt})$; b: $u_{lab} < u_{min}$; and c: $u_{lab} > u_{max}$


FCM-18-01: PET cyclic dimer in solution 2
x_{pt} = 0.0585 ; $U_{(Xpt)}$ (k=2) = 0.0057 ; σ_{pt} = 0.0117 (mg kg^{-1})
PET cyclic dimer: Kernel Density Plot
x_{pt}
Mass fraction (mg kg^{-1})
Laboratory Code
N-23 N-08 N-12 N-06 N-16 N-10 N-32 N-25 N-01 N-09 N-15 N-22 N-11 N-30 N-28 N-05 N-13 N-36 N-24 N-31 N-03 N-29 N-17 N-02 N-14 N-07 O-19 O-21 O-33 O-35 O-18 O-26 O-27 O-34
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; Reference interval ($x_{pt} \pm U_{(Xpt)}$): dashed blue lines; Target interval ($x_{pt} \pm 2\sigma_{pt}$): dotted red lines.

Annex 12: Results for PBT cyclic dimer in solution 2

Assigned range: x_{pt} = 0.0706 ± 0.0097 $U(x_{pt}, k = 2.0)$; σ_{pt} = 0.0141 (all values in mg kg^{-1})

Lab Code	X_{lab}	U_{lab}	k	technique	u_{lab}	z-score	zeta-score	uncert.
N-01	0.065	0.011	2	HPLC-UV	0.0055	-0.40	-0.76	a
N-02	0.042	0.002	2	HPLC-UV	0.001	-2.03	-5.78	b
N-03	0.072	0.023	2	HPLC-DAD	0.0115	0.10	0.11	a
N-05	0.0896			HPLC-DAD	0	1.35	3.92	b
N-06	0.065	0.002	√3	HPLC-UV	0.001155	-0.40	-1.12	b
N-07	<0.04			HPLC-UV				
N-08	0.019	0.002	2	HPLC-DAD	0.001	-3.65	-10.42	b
N-09	0.073	0.007	2	HPLC-DAD	0.0035	0.17	0.40	b
N-10	0.055	0.0072	2	HPLC-DAD	0.0036	-1.10	-2.58	b
N-11	0.0585	0.00667	2	HPLC-UV	0.003335	-0.86	-2.06	b
N-12	0.057	0.025	2	LC-MS	0.0125	-0.96	-1.01	a
N-13	0.056	0.005	2	HPLC-DAD	0.0025	-1.03	-2.68	b
N-14	0.0633	10	2	HPLC-UV	5	-0.52	0.00	c
N-15	0.087	0.001	2	HPLC-UV	0.0005	1.16	3.36	b
N-16	0.072	0.001	2	HPLC-UV	0.0005	0.10	0.29	b
N-17	0.161	0.032	2.2	LC-MS/MS	0.014545	6.40	5.90	c
N-22	0.055	0.003	2	HPLC-DAD	0.0015	-1.10	-3.07	b
N-23	0.038	0.002	2	HPLC-DAD	0.001	-2.31	-6.58	b
N-24	0.064	0.017	2	HPLC-UV	0.0085	-0.47	-0.67	a
N-25	0.056	0.003	2	HPLC-DAD	0.0015	-1.03	-2.88	b
N-28	0.059	0.0089	2	HPLC-UV	0.00445	-0.82	-1.76	b
N-29	0.13	0.038	2	HPLC-DAD	0.019	4.21	3.03	c
N-30	0.07			HPLC-DAD	0	-0.04	-0.12	b
N-31	0.17	0.034	2	HPLC-DAD	0.017	7.04	5.62	c
N-32	0.068	0.005	2	HPLC-DAD	0.0025	-0.18	-0.48	b
N-36	0.064			HPLC-DAD	0	-0.47	-1.36	b
O-18	0.0731	0.0012	2	HPLC-DAD	0.0006	0.18	0.51	b
O-19	0.05247	0.00117	2.447	HPLC-DAD	0.000478	-1.28	-3.72	b
O-21	0.063	0.031	2	HPLC-DAD	0.0155	-0.54	-0.47	c
O-26	0.0692	0.0166	2	HPLC-DAD	0.0083	-0.10	-0.15	a
O-27	0.17	0.034	3	HPLC-DAD	0.011333	7.04	8.06	a
O-33	0.08	0.024	2	LC-MS/MS	0.012	0.67	0.73	a
O-34	0.072	0.0021	2	HPLC-DAD	0.00105	0.10	0.28	b
O-35	0.081	0.002	√3	HPLC-DAD	0.001155	0.74	2.09	b

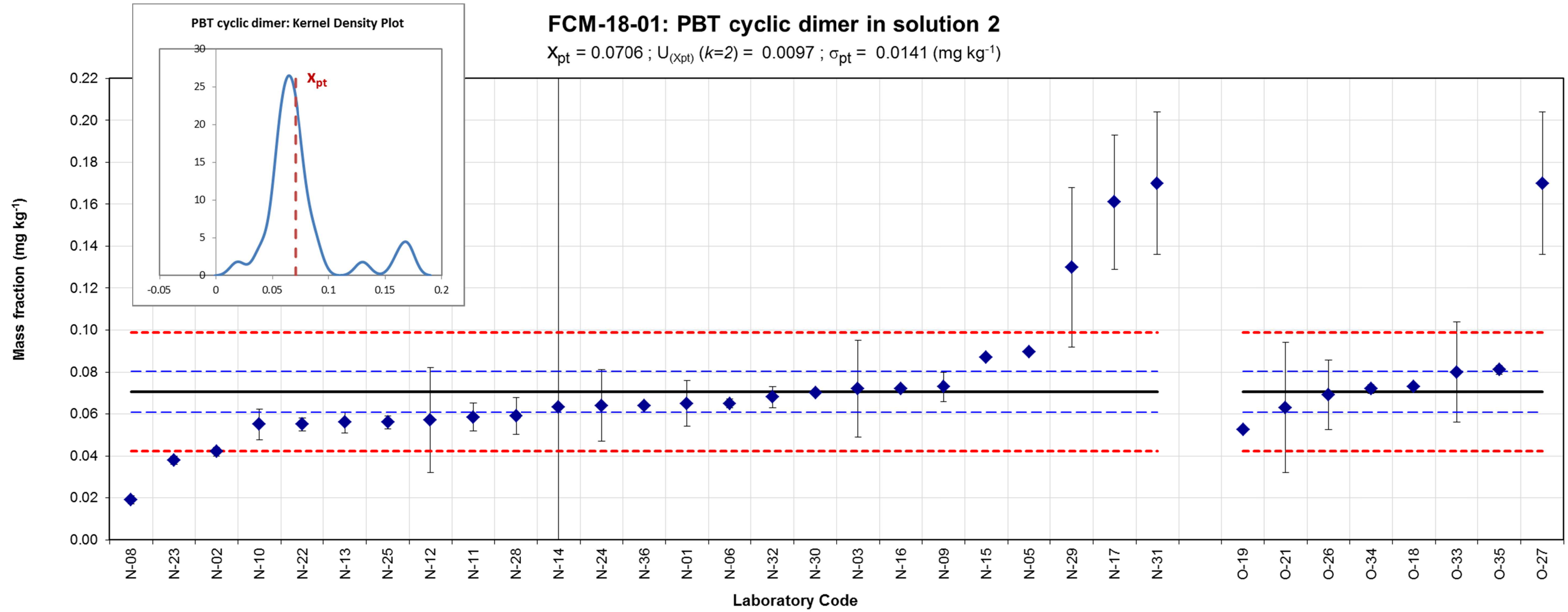
[a] √3 is set by the PT coordinator when no coverage factor *k* is reported. The reported uncertainty was assumed to have a rectangular distribution with *k* = √3,

[b] Performance scoring: satisfactory (green), questionable (yellow), unsatisfactory (red),

[c] a: $u_{min}(u(X_{pt})) \leq u_{lab} \leq u_{max}(\sigma_{pt})$; b: $u_{lab} < u_{min}$; and c: $u_{lab} > u_{max}$

FCM-18-01: PBT cyclic dimer in solution 2

x_{pt} = 0.0706 ; $U_{(Xpt)}$ (k=2) = 0.0097 ; σ_{pt} = 0.0141 (mg kg^{-1})



Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; Reference interval ($x_{pt} \pm U_{(Xpt)}$): dashed blue lines; Target interval ($x_{pt} \pm 2\sigma_{pt}$): dotted red lines.

Annex 13: Results for PET cyclic trimer in solution 2

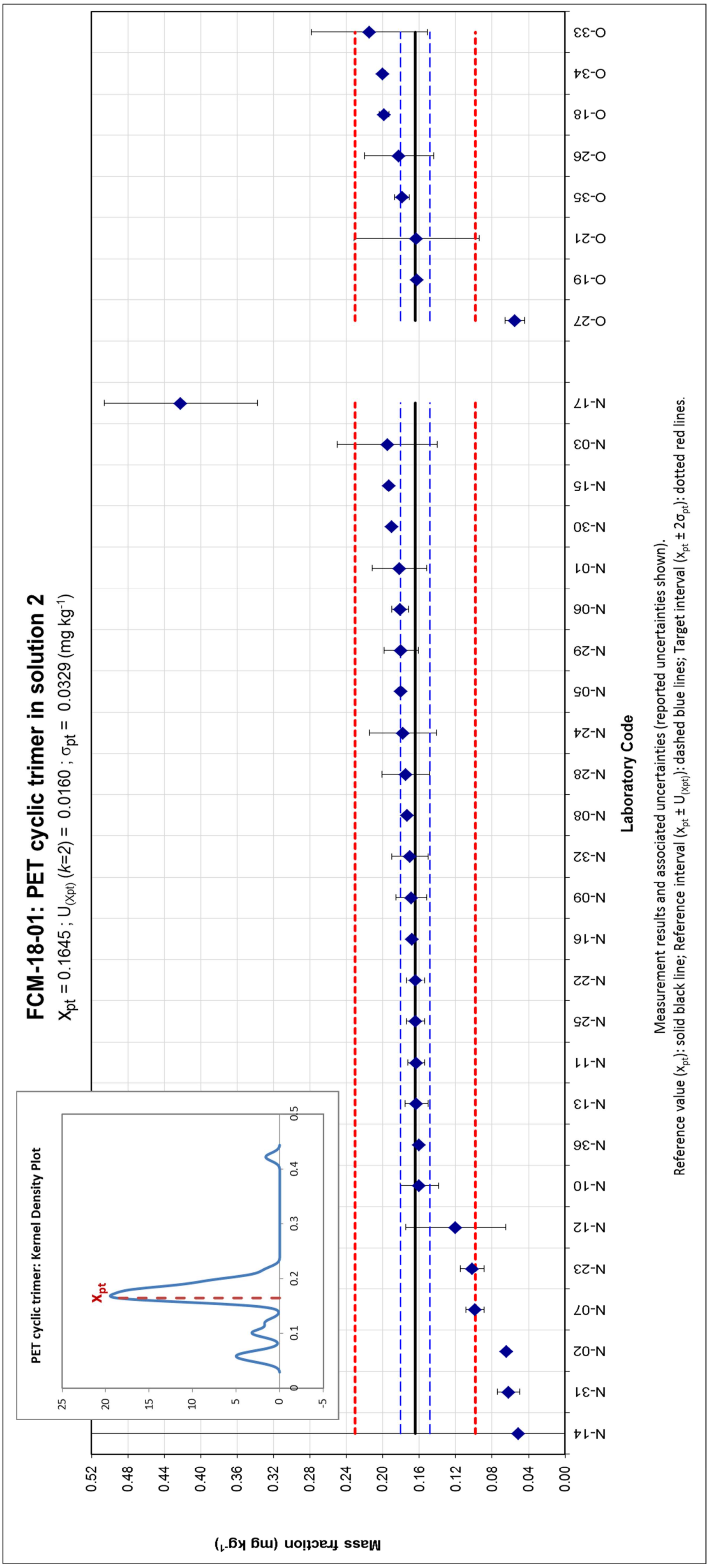
Assigned range: x_{pt} = 0.1645 ± 0.0160 $U(x_{pt}, k = 2.0)$; σ_{pt} = 0.0329 (all values in mg kg^{-1})

Lab Code	X_{lab}	U_{lab}	k	technique	u_{lab}	z-score	zeta-score	uncert.
N-01	0.182	0.03	2	HPLC-UV	0.015	0.53	1.03	a
N-02	0.064	0.003	2	HPLC-UV	0.0015	-3.05	-12.35	b
N-03	0.195	0.055	2	HPLC-DAD	0.0275	0.93	1.06	a
N-05	0.18			HPLC-DAD	0	0.47	1.94	b
N-06	0.181	0.009	√3	HPLC-UV	0.005196	0.50	1.73	b
N-07	0.099	0.01	2	HPLC-UV	0.005	-1.99	-6.94	b
N-08	0.173	0.002	2	HPLC-DAD	0.001	0.26	1.05	b
N-09	0.169	0.017	2	HPLC-DAD	0.0085	0.14	0.39	a
N-10	0.16	0.021	2	HPLC-DAD	0.0105	-0.14	-0.34	a
N-11	0.1632	0.009302	2	HPLC-UV	0.004651	-0.04	-0.14	b
N-12	0.12	0.055	2	LC-MS	0.0275	-1.35	-1.55	a
N-13	0.163	0.013	2	HPLC-DAD	0.0065	-0.05	-0.15	b
N-14	0.0508	15	2	HPLC-UV	7.5	-3.46	-0.02	c
N-15	0.193	0.001	2	HPLC-UV	0.0005	0.87	3.56	b
N-16	0.168	0.002	2	HPLC-UV	0.001	0.11	0.43	b
N-17	0.422	0.084	2.2	LC-MS/MS	0.038182	7.83	6.60	c
N-22	0.164	0.01	2	HPLC-DAD	0.005	-0.02	-0.05	b
N-23	0.102	0.013	2	HPLC-DAD	0.0065	-1.90	-6.06	b
N-24	0.178	0.037	2	HPLC-UV	0.0185	0.41	0.67	a
N-25	0.164	0.01	2	HPLC-DAD	0.005	-0.02	-0.05	b
N-28	0.175	0.0263	2	HPLC-UV	0.01315	0.32	0.68	a
N-29	0.18	0.019	2	HPLC-DAD	0.0095	0.47	1.25	a
N-30	0.19			HPLC-DAD	0	0.78	3.19	b
N-31	0.062	0.012	2	HPLC-DAD	0.006	-3.12	-10.25	b
N-32	0.17	0.02	2	HPLC-DAD	0.01	0.17	0.43	a
N-36	0.1603			HPLC-DAD	0	-0.13	-0.53	b
O-18	0.1985	0.0055	2	HPLC-DAD	0.00275	1.03	4.02	b
O-19	0.1623	0.00232	2.447	HPLC-DAD	0.000948	-0.07	-0.27	b
O-21	0.163	0.069	2	HPLC-DAD	0.0345	-0.05	-0.04	c
O-26	0.1822	0.0377	2	HPLC-DAD	0.01885	0.54	0.86	a
O-27	0.055	0.011	3	HPLC-DAD	0.003667	-3.33	-12.44	b
O-33	0.215	0.064	2	LC-MS/MS	0.032	1.53	1.53	a
O-34	0.2	0.0032	2	HPLC-DAD	0.0016	1.08	4.35	b
O-35	0.179	0.008	√3	HPLC-DAD	0.004619	0.44	1.57	b

[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 scoring: satisfactory (green), questionable (yellow), unsatisfactory (red),

[c] a: $u_{min}(u(X_{pt})) \leq u_{lab} \leq u_{max}(\sigma_{pt})$; b: $u_{lab} < u_{min}$; and c: $u_{lab} > u_{max}$



Measurement results and associated uncertainties (reported uncertainties shown).

Reference value (x_{pt}): solid black line; Reference interval ($x_{pt} \pm U_{(xpt)}$): dashed blue lines; Target interval ($x_{pt} \pm 2\sigma_{pt}$): dotted red lines.

Annex 14: Results for PBT cyclic trimer in solution 2

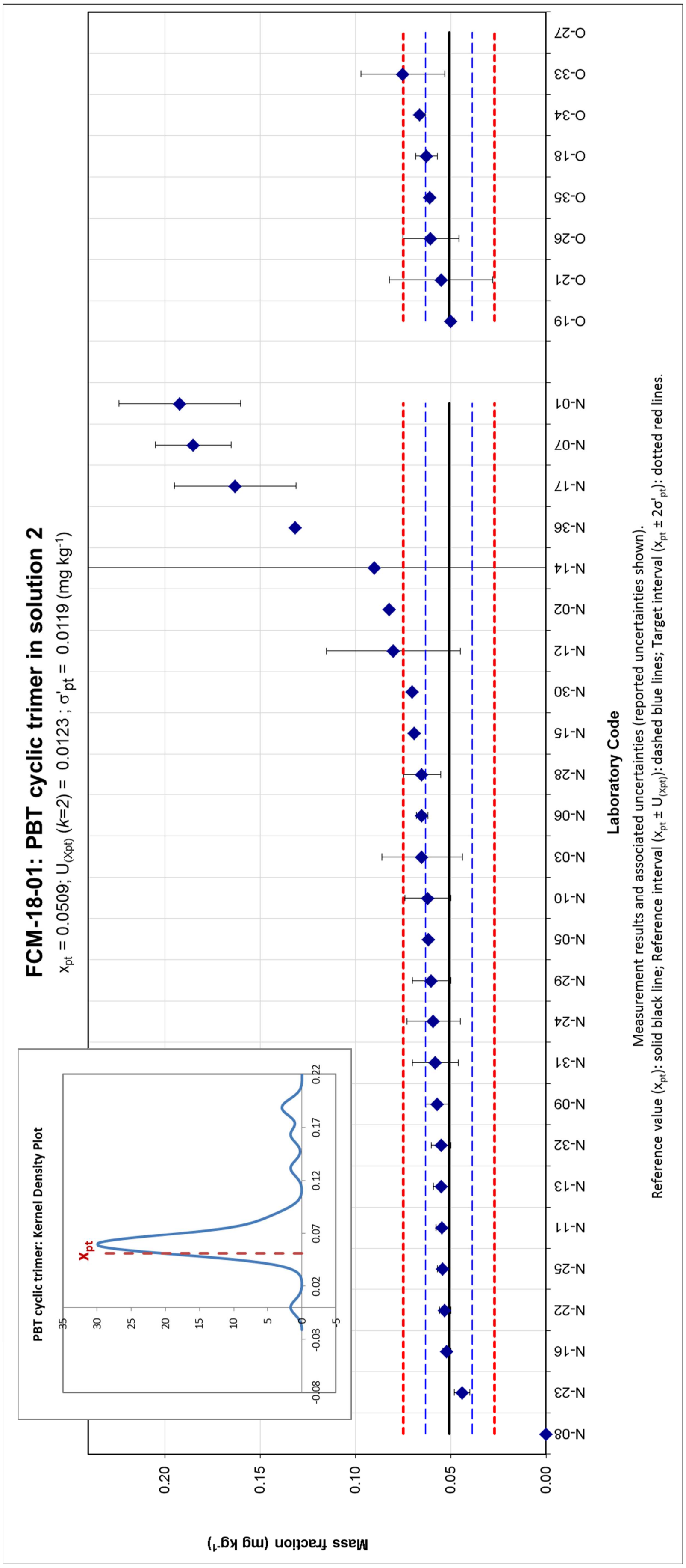
Assigned range: x_{pt} = 0.0509 ± 0.0123 $U(x_{pt}, k = 2.0)$; σ_{pt} = 0.0102 (all values in mg kg^{-1})

Lab Code	X_{lab}	U_{lab}	k	technique	u_{lab}	z'-score	zeta-score	uncert.
N-01	0.192	0.032	2	HPLC-UV	0.016	11.85	8.23	c
N-02	0.082	0.001	2	HPLC-UV	0.0005	2.61	5.04	b
N-03	0.065	0.021	2	HPLC-DAD	0.0105	1.18	1.16	a
N-05	0.0614			HPLC-DAD	0	0.88	1.71	b
N-06	0.065	0.003	√3	HPLC-UV	0.001732	1.18	2.21	b
N-07	0.185	0.02	2	HPLC-UV	0.01	11.26	11.42	a
N-08	0	0	2	HPLC-DAD	0	-4.27	-8.28	b
N-09	0.057	0.006	2	HPLC-DAD	0.003	0.51	0.89	b
N-10	0.062	0.012	2	HPLC-DAD	0.006	0.93	1.29	b
N-11	0.0546	0.00317	2	HPLC-UV	0.001585	0.31	0.58	b
N-12	0.08	0.035	2	LC-MS	0.0175	2.44	1.57	c
N-13	0.055	0.004	2	HPLC-DAD	0.002	0.34	0.63	b
N-14	0.0901	10	2	HPLC-UV	5	3.29	0.01	c
N-15	0.069	0.001	2	HPLC-UV	0.0005	1.52	2.93	b
N-16	0.052	0.002	2	HPLC-UV	0.001	0.09	0.18	b
N-17	0.163	0.032	2.2	LC-MS/MS	0.014545	9.41	7.10	c
N-22	0.053	0.003	2	HPLC-DAD	0.0015	0.18	0.33	b
N-23	0.044	0.004	2	HPLC-DAD	0.002	-0.58	-1.07	b
N-24	0.059	0.014	2	HPLC-UV	0.007	0.68	0.87	a
N-25	0.054	0.003	2	HPLC-DAD	0.0015	0.26	0.49	b
N-28	0.065	0.0098	2	HPLC-UV	0.0049	1.18	1.79	b
N-29	0.06	0.01	2	HPLC-DAD	0.005	0.76	1.15	b
N-30	0.07			HPLC-DAD	0	1.60	3.11	b
N-31	0.058	0.012	2	HPLC-DAD	0.006	0.60	0.83	b
N-32	0.055	0.005	2	HPLC-DAD	0.0025	0.34	0.62	b
N-36	0.1313			HPLC-DAD	0	6.75	13.07	b
O-18	0.0627	0.0056	2	HPLC-DAD	0.0028	0.99	1.75	b
O-19	0.04978	0.00169	2.571	HPLC-DAD	0.000657	-0.09	-0.18	b
O-21	0.055	0.027	2	HPLC-DAD	0.0135	0.34	0.28	c
O-26	0.0604	0.0147	2	HPLC-DAD	0.00735	0.80	0.99	a
O-27	0.57	0.011	3	HPLC-DAD	0.003667	43.58	72.50	b
O-33	0.075	0.022	2	LC-MS/MS	0.011	2.02	1.91	a
O-34	0.066	0.0026	2	HPLC-DAD	0.0013	1.27	2.40	b
O-35	0.061	0.002	√3	HPLC-DAD	0.001155	0.85	1.61	b

[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 scoring: satisfactory (green), questionable (yellow), unsatisfactory (red),

[c] a : $u_{min}(u(X_{pt})) \leq u_{lab} \leq u_{max}(\sigma_{pt})$; b : $u_{lab} < u_{min}$; and c : $u_{lab} > u_{max}$



FCM-18-01: PBT cyclic trimer in solution 2
x_{pt} = 0.0509; $U_{(Xpt)}$ (k=2) = 0.0123 ; σ'_{pt} = 0.0119 (mg kg^{-1})
Mass fraction (mg kg^{-1})
Laboratory Code
N-08 N-23 N-16 N-22 N-25 N-11 N-13 N-32 N-09 N-31 N-24 N-29 N-05 N-10 N-03 N-06 N-28 N-15 N-30 N-12 N-02 N-14 N-36 N-17 N-07 N-01 O-19 O-21 O-26 O-35 O-18 O-34 O-33 O-27
0.00 0.05 0.10 0.15 0.20
PBT cyclic trimer: Kernel Density Plot
x_{pt}
Measurement results and associated uncertainties (reported uncertainties shown).
Reference value (x_{pt}): solid black line; Reference interval ($x_{pt} \pm U_{(xpt)}$): dashed blue lines; Target interval ($x_{pt} \pm 2\sigma'_{pt}$): dotted red lines.

Annex 15: Overview on experimental details

Lab Code	Did you use the protocol that was sent to you?	If "No" describe briefly the analytical method used.	Standard method?	Validated method?	Accredited method?	Do you usually provide an uncertainty statement to your customers?	Does your laboratory have a quality manageme nt system?	If "Yes" based on which standard?	Does your laboratory participate in interlaboratory comparisons for this type of analysis?
N-01	a) Yes	Modified gradient and flow rate	x			b) No	a) Yes	a) ISO 17025	b) No
N-02	b) No	similar method with adapted chromatographic conditions				a) Yes	a) Yes	a) ISO 17025	b) No
N-03	a) Yes		X			a) Yes	a) Yes	a) ISO 17025	b) No
N-05	b) No	Only the wavelength was different, we used 254 nm	X			a) Yes	a) Yes	a) ISO 17025	b) No
N-06	a) Yes		No	No	No	b) No	a) Yes	a) ISO 17025	b) No
N-07	a) Yes			x		a) Yes	a) Yes	a) ISO 17025	b) No
N-08	a) Yes		x			a) Yes	a) Yes	a) ISO 17025, b) ISO 9001 series	b) No
N-09	a) Yes		x			a) Yes	a) Yes	a) ISO 17025	a) Yes
N-10	b) No	it is slightly adjusted to our column, YMC-Pack Pro C18 150x2.1 mm 3.6 µm	x			b) No	a) Yes	a) ISO 17025	b) No
N-11	a) Yes					a) Yes	a) Yes	a) ISO 17025	b) No
N-12	b) No	Used LC method with a differnt column and TOF-MS detection				b) No	a) Yes	b) ISO 9001 series	b) No
N-13	a) Yes		no	no	no	a) Yes	a) Yes	a) ISO 17025	a) Yes
N-14	a) Yes			x		a) Yes	a) Yes	a) ISO 17025	b) No
N-15	a) Yes		x			a) Yes	a) Yes	a) ISO 17025	a) Yes
N-16	a) Yes					b) No	a) Yes	a) ISO 17025	b) No
N-17	b) No	LC-MS/MS after dilution by 2 (in H2O)				b) No	a) Yes	a) ISO 17025	b) No
N-23	a) Yes		X			b) No	a) Yes	a) ISO 17025	b) No
N-24	a) Yes					a) Yes	a) Yes	a) ISO 17025	b) No
N-25	b) No	UHPLC, PDA-detection 240 nm, Waters PFP-column	x			a) Yes	a) Yes	a) ISO 17025	a) Yes
N-28	a) Yes		x			a) Yes	a) Yes	a) ISO 17025	b) No

N-29	a) Yes					a) Yes	a) Yes	a) ISO 17025	b) No
N-30	b) No	We used the protocol that was sent to us with the variation described hereafter: Column (100 x 4.6 mm, 3um), flow (1.5 mL/min), different linear gradient.				a) Yes	a) Yes	a) ISO 17025	b) No
N-31	a) Yes		X			a) Yes	a) Yes	a) ISO 17025	a) Yes
N-32	a) Yes			X		b) No	a) Yes	a) ISO 17025	b) No
N-36	a) Yes		X			b) No	a) Yes	a) ISO 17025	b) No
O-18	b) No	Which protocol? We analysed in dependence on the guideline of our NRL.				a) Yes	a) Yes	a) ISO 17025	a) Yes
O-19	b) No	Method validation is not ready. HPLC-DAD; Column: Agilent Zorbax SB Phenyl; 4,6x250mm; 5µ	No	No	No	b) No	a) Yes	a) ISO 17025	b) No
O-21	b) No	HPLC/DAD; different eluent programme, different column	x			a) Yes	a) Yes	a) ISO 17025	a) Yes
O-22	b) No	UHPLC, PDA-detction 240 nm, Waters PFP-column	x			a) Yes	a) Yes	a) ISO 17025	a) Yes
O-26	a) Yes		no	no	no	a) Yes	a) Yes	a) ISO 17025	b) No
O-27	a) Yes, b) No	differences to the protocol see Nr. 3		x	x	a) Yes, b) No	a) Yes	a) ISO 17025	a) Yes
O-33	a) Yes	We use for mass parameters but we used LCMSMS	x			a) Yes	a) Yes	a) ISO 17025	b) No
O-34	b) No	coulomn zorbax sb C18 150*4.6mm 5 µm temp. 30°C flow 1.2 ml/min mobile phase gradinet 60H20:40 CH3CN to 100 CH3CN	no	no	no	X	a) Yes	a) ISO 17025	a) Yes
O-35	a) Yes					a) Yes	a) Yes	a) ISO 17025	a) Yes

Annex 16: Detailed method parameters and performance

Lab Code	Analyte + Performance Solution 1	Analyte + Performance Solution 2	LOD (mg/kg)	Mobile phase used	Injection volume (µL)	Detection used	Column used
N-01	PBT cyclic dimer	PBT cyclic dimer	0.02	Acetonitrile and Water	50	UV	Phenomenex gemini 150 x 3
	PBT cyclic trimer	PBT cyclic trimer	0.02	Acetonitrile and Water	50	UV	Phenomenex gemini 150 x 3
	PET cyclic dimer	PET cyclic dimer	0.02	Acetonitrile and Water	50	UV	Phenomenex gemini 150 x 3
	PET cyclic trimer	PET cyclic trimer	0.02	Acetonitrile and Water	50	UV	Phenomenex gemini 150 x 3
N-02	PBT cyclic dimer	PBT cyclic dimer	0.02	water/acetonitrile	50	UV	Zorbax Eclipse XDB C18
	PBT cyclic trimer	PBT cyclic trimer	0.02	water/acetonitrile	50	UV	Zorbax Eclipse XDB C18
	PET cyclic dimer	PET cyclic dimer	0.02	water/acetonitrile	50	UV	Zorbax Eclipse XDB C18
	PET cyclic trimer	PET cyclic trimer	0.02	water/acetonitrile	50	UV	Zorbax Eclipse XDB C18
N-03	PBT cyclic dimer	PBT cyclic dimer	0.01	H2O-ACN	50	DAD	C18
	PBT cyclic trimer	PBT cyclic trimer	0.01	H2O-ACN	50	DAD	C18
	PET cyclic dimer	PET cyclic dimer	0.01	H2O-ACN	50	DAD	C18
	PET cyclic trimer	PET cyclic trimer	0.01	H2O-ACN	50	DAD	C18
N-05	PBT cyclic dimer	PBT cyclic dimer					
	PBT cyclic trimer	PBT cyclic trimer					
	PET cyclic dimer	PET cyclic dimer					
	PET cyclic trimer	PET cyclic trimer					
N-06	PBT cyclic dimer	PBT cyclic dimer	0.006	Acetonitrile/water	100	UV-240nm	The same as protocol
	PBT cyclic trimer	PBT cyclic trimer	0.013	Acetonitrile/water	100	UV-240nm	The same as protocol
	PET cyclic dimer	PET cyclic dimer	0.011	Acetonitrile/water	100	UV-240nm	The same as protocol
	PET cyclic trimer	PET cyclic trimer	0.005	Acetonitrile/water	100	UV-240nm	The same as protocol
N-07	PBT cyclic dimer	PBT cyclic dimer	0.04	WATER:ACETONITRILE	50	UV	150X4.6mm,5um C18
	PBT cyclic trimer	PBT cyclic trimer	0.05	WATER:ACETONITRILE	50	UV	150X4.6mm,5um C18
	PET cyclic dimer	PET cyclic dimer	0.03	WATER:ACETONITRILE	50	UV	150X4.6mm,5um C18
	PET cyclic trimer	PET cyclic trimer	0.04	WATER:ACETONITRILE	50	UV	150X4.6mm,5um C18
N-08	PBT cyclic dimer	PBT cyclic dimer		Acetonitrile:water	50	DAD	c18
	PBT cyclic trimer	PBT cyclic trimer		Acetonitrile:water	50	DAD	c18
	PET cyclic dimer	PET cyclic dimer		Acetonitrile:water	50	DAD	c18

	PET cyclic trimer	PET cyclic trimer		Acetonitrile:water	50	DAD	c18
N-09	PBT cyclic dimer	PBT cyclic dimer	0.002		100	DAD	C18 XDB 4.6x150 5 um
	PBT cyclic trimer	PBT cyclic trimer	0.002		100	DAD	C18 XDB 4.6x150 5 um
	PET cyclic dimer	PET cyclic dimer	0.002		100	DAD	C18 XDB 4.6x150 5 um
	PET cyclic trimer	PET cyclic trimer	0.002		100	DAD	C18 XDB 4.6x150 5 um
N-10	PBT cyclic dimer	PBT cyclic dimer	0.01	ACN/Water	8	DAD	YMC-Pack Pro C18 150x2.1
	PBT cyclic trimer	PBT cyclic trimer	0.01	ACN/Water	8	DAD	YMC-Pack Pro C18 150x2.1
	PET cyclic dimer	PET cyclic dimer	0.01	ACN/Water	8	DAD	YMC-Pack Pro C18 150x2.1
	PET cyclic trimer	PET cyclic trimer	0.01	ACN/Water	8	DAD	YMC-Pack Pro C18 150x2.1
N-11	PBT cyclic dimer	PBT cyclic dimer					
	PBT cyclic trimer	PBT cyclic trimer					
	PET cyclic dimer	PET cyclic dimer					
	PET cyclic trimer	PET cyclic trimer					
N-12	PBT cyclic dimer	PBT cyclic dimer		NH4Ac + MeOH	50	TOF-MS	Atlantis dC18
	PBT cyclic trimer	PBT cyclic trimer		NH4Ac + MeOH	50	TOF-MS	Atlantis dC18
	PET cyclic dimer	PET cyclic dimer		NH4Ac + MeOH	50	TOF-MS	Atlantis dC18
	PET cyclic trimer	PET cyclic trimer		NH4Ac + MeOH	50	TOF-MS	Atlantis dC18
N-13	PBT cyclic dimer	PBT cyclic dimer	0.025	acetonitrile/water	100	DAD	Xterra RP18 150x4.6, 5um
	PBT cyclic trimer	PBT cyclic trimer	0.025	acetonitrile/water	100	DAD	Xterra RP18 150x4.6, 5um
	PET cyclic dimer	PET cyclic dimer	0.025	acetonitrile/water	100	DAD	Xterra RP18 150x4.6, 5um
	PET cyclic trimer	PET cyclic trimer	0.025	acetonitrile/water	100	DAD	Xterra RP18 150x4.6, 5um
N-14	PBT cyclic dimer	PBT cyclic dimer	0.02	ACN/Water	10	UV	Poreshell SB C18 2.1*100
	PBT cyclic trimer	PBT cyclic trimer	0.02	ACN/Water	10	UV	Poreshell SB C18 2.1*100
	PET cyclic dimer	PET cyclic dimer	0.02	ACN/Water	10	UV	Poreshell SB C18 2.1*100
	PET cyclic trimer	PET cyclic trimer	0.02	ACN/Water	10	UV	Poreshell SB C18 2.1*100
N-15	PBT cyclic dimer	PBT cyclic dimer	0,007	Acetonotrile/Water	20	UV	X Bridge C18
	PBT cyclic trimer	PBT cyclic trimer	0,009	Acetonitrile/Water	20	UV	X Bridge C18
	PET cyclic dimer	PET cyclic dimer	0,005	Acetonitrile/Water	20	UV	X Bridge C18
	PET cyclic trimer	PET cyclic trimer	0,012	Acetonitrile/Water	20	UV	X Bridge C18
N-16	PBT cyclic dimer	PBT cyclic dimer	0.002	ACN:H2O	50	UV	ZORBAX 5 C18
	PBT cyclic trimer	PBT cyclic trimer	0.001	ACN:H20	50	UV	ZORBAX 5 C18
	PET cyclic dimer	PET cyclic dimer	0.002	ACN:H20	50	UV	ZORBAX 5 C18
	PET cyclic trimer	PET cyclic trimer	0.001	ACN:H20	50	UV	ZORBAX 5 C18

N-17	PBT cyclic dimer	PBT cyclic dimer	0.001	ACN/H2O	20	MS/MS	C18, 100x2.1mm 1.9µm
	PBT cyclic trimer	PBT cyclic trimer	0.0002	ACN/H2O	20	MS/MS	C18, 100x2.1mm 1.9µm
	PET cyclic dimer	PET cyclic dimer	0.004	ACN/H2O	20	MS/MS	C18, 100x2.1mm 1.9µm
	PET cyclic trimer	PET cyclic trimer	0.0003	ACN/H2O	20	MS/MS	C18, 100x2.1mm 1.9µm
N-23	PBT cyclic dimer	PBT cyclic dimer	0.01	ACN-H2O	50	DAD	Phenomenex C18, 150*4.6mm
	PBT cyclic trimer	PBT cyclic trimer	0.01	ACN-H2O	50	DAD	Phenomenex C18, 150*4.6mm
	PET cyclic dimer	PET cyclic dimer	0.01	ACN-H2O	50	DAD	Phenomenex C18, 150*4.6mm
	PET cyclic trimer	PET cyclic trimer	0.01	ACN-H2O	50	DAD	Phenomenex C18, 150*4.6mm
N-24	PBT cyclic dimer	PBT cyclic dimer	0.005	Acetonitrile HPLC/Water	50	UV	LiChrospher-RP18
	PBT cyclic trimer	PBT cyclic trimer	0.005	Acetonitrile HPLC/Water	50	UV	LiChrospher-RP18
	PET cyclic dimer	PET cyclic dimer	0.005	Acetonitrile HPLC/Water	50	UV	LiChrospher-RP18
	PET cyclic trimer	PET cyclic trimer	0.005	Acetonitrile HPLC/Water	50	UV	LiChrospher-RP18
N-25	PBT cyclic dimer	PBT cyclic dimer	0.01	H2O:MeOH	10	PDA	PFP
	PBT cyclic trimer	PBT cyclic trimer	0.01	H2O:MeOH	10	PDA	PFP
	PET cyclic dimer	PET cyclic dimer	0.01	H2O:MeOH	10	PDA	PFP
	PET cyclic trimer	PET cyclic trimer	0.01	H2O:MeOH	10	PDA	PFP
N-28	PBT cyclic dimer	PBT cyclic dimer	0.01	ACN/H2O	20	UV 240 nm	SunShell 150x2.1 2.6um
	PBT cyclic trimer	PBT cyclic trimer	0.01	ACN/H2O	20	UV 240 nm	SunShell 150x2.1 2.6um
	PET cyclic dimer	PET cyclic dimer	0.01	ACN/H2O	20	UV 240 nm	SunShell 150x2.1 2.6um
	PET cyclic trimer	PET cyclic trimer	0.01	ACN/H2O	20	UV 240 nm	SunShell 150x2.1 2.6um
N-29	PBT cyclic dimer	PBT cyclic dimer	0.02	Acetonitrile/H2O	50	UV	Eclipse XDB-C18
	PBT cyclic trimer	PBT cyclic trimer	0.02	Acetonitrile/H2O	50	UV	Eclipse XDB-C18
	PET cyclic dimer	PET cyclic dimer	0.01	Acetonitrile/H2O	50	UV	Eclipse XDB-C18
	PET cyclic trimer	PET cyclic trimer	0.01	Acetonitrile/H2O	50	UV	Eclipse XDB-C18
N-30	PBT cyclic dimer	PBT cyclic dimer	0.01	water/acetonitrile	50	DAD at 240 nm	Roc C18 100x4,6 mm, 3um
	PBT cyclic trimer	PBT cyclic trimer	0.01	water/acetonitrile	50	DAD at 240 nm	Roc C18 100x4,6 mm, 3um
	PET cyclic dimer	PET cyclic dimer	0.01	water/acetonitrile	50	DAD at 240 nm	Roc C18 100x4,6 mm, 3um

	PET cyclic trimer	PET cyclic trimer	0.01	water/acetonitrile	50	DAD at 240 nm	Roc C18 100x4,6 mm, 3um
N-31	PBT cyclic dimer	PBT cyclic dimer	0.01	Water/Acetonitrile	50	DAD	Poroshell 120 EC-C18 2.7
	PBT cyclic trimer	PBT cyclic trimer	0.01	Water/Acetonitrile	50	DAD	Poroshell 120 EC-C18 2.7
	PET cyclic dimer	PET cyclic dimer	0.01	Water/Acetonitrile	50	DAD	Poroshell 120 EC-C18 2.7
	PET cyclic trimer	PET cyclic trimer	0.01	Water/Acetonitrile	50	DAD	Poroshell 120 EC-C18 2.7
N-32	PBT cyclic dimer	PBT cyclic dimer	0.005	ACN/WATER	50	PDA	C18
	PBT cyclic trimer	PBT cyclic trimer	0.005	ACN/WATE	50	PDA	C18
	PET cyclic dimer	PET cyclic dimer	0.005	ACN/WATE	50	PDA	C18
	PET cyclic trimer	PET cyclic trimer	0.005	ACN/WATE	50	PDA	C18
N-36	PBT cyclic dimer	PBT cyclic dimer		ACN/water	50	DAD	XDB-C8
	PBT cyclic trimer	PBT cyclic trimer		ACN/water	50	DAD	XDB-C8
	PET cyclic dimer	PET cyclic dimer		ACN/water	50	DAD	XDB-C8
	PET cyclic trimer	PET cyclic trimer		ACN/water	50	DAD	XDB-C8
O-18	PBT cyclic dimer	PBT cyclic dimer	0.03	ACN/H2O	8	DAD	Zorbax Eclipse XDB-C18
	PBT cyclic trimer	PBT cyclic trimer	0.03	ACN/H2O	8	DAD	Zorbax Eclipse XDB-C18
	PET cyclic dimer	PET cyclic dimer	0.04	ACN/H2O	8	DAD	Zorbax Eclipse XDB-C18
	PET cyclic trimer	PET cyclic trimer	0.03	ACN/H2O	8	DAD	Zorbax Eclipse XDB-C18
O-19	PBT cyclic dimer	PBT cyclic dimer	0.012	Water-MeOH	10 and 20	240	Agilent Zorbax SB Phenyl
	PBT cyclic trimer	PBT cyclic trimer	0.014	Water-MeOH	10 and 20	240	Agilent Zorbax SB Phenyl
	PET cyclic dimer	PET cyclic dimer	0.02	Water-MeOH	10 and 20	240	Agilent Zorbax SB Phenyl
	PET cyclic trimer	PET cyclic trimer	0,02	Water-MeOH	10 and 20	240	Agilent Zorbax SB Phenyl
O-21	PBT cyclic dimer	PBT cyclic dimer	0.005	Acetonitril/Water	20	DAD, 240 nm	Luna 5µ C18(2) 150 x 4,6
	PBT cyclic trimer	PBT cyclic trimer	0.005	Acetonitril/Water	20	DAD, 240 nm	Luna 5µ C18(2) 150 x 4,6
	PET cyclic dimer	PET cyclic dimer	0.005	Acetonitril/Water	20	DAD, 240 nm	Luna 5µ C18(2) 150 x 4,6
	PET cyclic trimer	PET cyclic trimer	0.005	Acetonitril/Water	20	DAD, 240 nm	Luna 5µ C18(2) 150 x 4,6
O-22	PBT cyclic dimer	PBT cyclic dimer	0.01	H2O:MeOH	10	PDA	PFP

	PBT cyclic trimer	PBT cyclic trimer	0.01	H2O:MeOH	10	PDA	PFP
	PET cyclic dimer	PET cyclic dimer	0.01	H2O:MeOH	10	PDA	PFP
	PET cyclic trimer	PET cyclic trimer	0.01	H2O:MeOH	10	PDA	PFP
O-26	PBT cyclic dimer	PBT cyclic dimer	0,0062	Acetonitril/Water	20	DAD	Zorbax Eclipse XDB-C8
	PBT cyclic trimer	PBT cyclic trimer	0,0077	Acetonitril/Water	20	DAD	Zorbax Eclipse XDB-C8
	PET cyclic dimer	PET cyclic dimer	0,0135	Acetonitril/Water	20	DAD	Zorbax Eclipse XDB-C8
	PET cyclic trimer	PET cyclic trimer	0,0052	Acetonitril/Water	20	DAD	Zorbax Eclipse XDB-C8
O-27	PBT cyclic dimer	PBT cyclic dimer	0,01	H20/ACN	10	DAD 240nm	Envirosep PP 150x2,0mm
	PBT cyclic trimer	PBT cyclic trimer	0,01	H20/ACNH20/ACN	10	DAD 240nm	Envirosep PP 150x2,0mm
	PET cyclic dimer	PET cyclic dimer	0,01	H20/ACN	10	DAD 240nm	Envirosep PP 150x2,0mm
	PET cyclic trimer	PET cyclic trimer	0,01	H20/ACN	10	DAD 240nm	Envirosep PP 150x2,0mm
O-33	PBT cyclic dimer	PBT cyclic dimer	0.001	h2o formic acid ACCN	5		
	PBT cyclic trimer	PBT cyclic trimer	0.001	h2o formic acid ACCN	5		
	PET cyclic dimer	PET cyclic dimer	0.001	h2o formic acid ACCN	5		
	PET cyclic trimer	PET cyclic trimer	0.001	h2o formic acid ACCN	5		
O-34	PBT cyclic dimer	PBT cyclic dimer	0.02	gradient	50	DAD	zorbax sb C18 150*4.6mm
	PBT cyclic trimer	PBT cyclic trimer	0.02	gradient	50	DAD	zorbax sb C18 150*4.6mm
	PET cyclic dimer	PET cyclic dimer	0.02	gradient	50	DAD	zorbax sb C18 150*4.6mm
	PET cyclic trimer	PET cyclic trimer	0.01	gradient	50	DAD	zorbax sb C18 150*4.6mm
O-35	PBT cyclic dimer	PBT cyclic dimer	0,005	Acetonitrile/H2O Gradient	20	DAD: 240 nm	RP18 12,5cmx2,1mm; 5µm
	PBT cyclic trimer	PBT cyclic trimer	0,004	Acetonitrile/H2O Gradient	20	DAD: 240 nm	RP18 12,5cmx2,1mm; 5µm
	PET cyclic dimer	PET cyclic dimer	0,006	Acetonitrile/H2O Gradient	20	DAD: 240 nm	RP18 12,5cmx2,1mm; 5µm
	PET cyclic trimer	PET cyclic trimer	0,005	Acetonitrile/H2O Gradient	20	DAD: 240 nm	RP18 12,5cmx2,1mm; 5µm