



JRC TECHNICAL REPORT

Determination of MOSH and MOAH in muesli and paperboard

Proficiency Test Report
JRC FCM-20/01

Stefanka Bratinova, Pieter Dehouck,
Piotr Robouch, Giorgia Beldi,
Natalia Jakubowska , Eddy Hoekstra



This publication is a Technical report by the Joint Research Centre (JRC), the European Commission's science and knowledge service. It aims to provide evidence-based scientific support to the European policymaking process. The scientific output expressed does not imply a policy position of the European Commission. Neither the European Commission nor any person acting on behalf of the Commission is responsible for the use that might be made of this publication. For information on the methodology and quality underlying the data used in this publication for which the source is neither Eurostat nor other Commission services, users should contact the referenced source. The designations employed and the presentation of material on the maps do not imply the expression of any opinion whatsoever on the part of the European Union concerning the legal status of any country, territory, city or area or of its authorities, or concerning the delimitation of its frontiers or boundaries.

Contact information

Stefanka Bratinova

European Commission, Joint Research Centre

European Union Reference Laboratory for Food Contact Materials

Retieseweg 111, 2440 Geel, Belgium

Email: JRC-EURL-FCM@ec.europa.eu

EU Science Hub

<https://ec.europa.eu/jrc>

JRC126069

EUR 30787 EN

PDF ISBN 978-92-76-40589-4 ISSN 1831-9424 doi:10.2760/05496

Luxembourg: Publications Office of the European Union, 2021

© European Union, 2021



The reuse policy of the European Commission is implemented by the Commission Decision 2011/833/EU of 12 December 2011 on the reuse of Commission documents (OJ L 330, 14.12.2011, p. 39). Except otherwise noted, the reuse of this document is authorised under the Creative Commons Attribution 4.0 International (CC BY 4.0) licence (<https://creativecommons.org/licenses/by/4.0/>). This means that reuse is allowed provided appropriate credit is given and any changes are indicated. For any use or reproduction of photos or other material that is not owned by the EU, permission must be sought directly from the copyright holders.

All content © European Union 2021

How to cite this report: Stefanka Bratinova, Pieter Dehouck, Piotr Robouch, Giorgia Beldi, Natalia Jakubowska, Eddy Hoekstra, *Determination of MOSH and MOAH in muesli and paperboard, Proficiency Test Report JRC FCM-20/01*, EUR 30787 EN, Publications Office of the European Union, Luxembourg, 2021, ISBN 978-92-76-40589-4, doi:10.2760/05496, JRC126069

Table of contents

Executive summary	1
List of abbreviations and symbols.....	2
1 Introduction.....	3
2 Scope.....	3
3 Set up of the exercise	3
3.1 Confidentiality	3
3.2 Time frame	3
3.3 Distribution	3
3.4 Instructions to participants.....	4
4 Test item	5
4.1 Preparation	5
4.2 Homogeneity and stability.....	5
5 Assigned values and corresponding uncertainties.....	6
5.1 Assigned values	6
5.2 Associated uncertainties.....	6
5.1 Standard deviation for proficiency assessment, σ_{pt}	7
6 Evaluation of results	7
6.1 Scores and evaluation criteria.....	7
6.2 General observations.....	9
6.3 Laboratory results and scorings	9
6.3.1 Performances	9
6.3.2 Additional information extracted from the questionnaire	11
7 Conclusion	12
Acknowledgements.....	12
References	13
Annex 1: Invitation letter	14
Annex 2: Test item accompanying letter	15
Annex 3: Confirmation of receipt form	18
Annex 4: Questionnaire	19
Annex 5: Homogeneity and stability results	24
5.1 Paperboard - Homogeneity (all values expressed	24
5.2 Muesli - Homogeneity (all values.....	25
5.3 Stability study for Total MOSH and MOAH in paperboard and muesli	26
Annex 6: Test item characterisation	27
Annex 7: Results for total MOAH mass fraction in paperboard	28
Annex 8: Results for MOAH C10-C16 mass fraction in paperboard	29
Annex 9: Results for MOAH C16-C25 mass fraction in paperboard	30

Annex 10:	Results for MOAH C25-C35 mass fraction in paperboard	31
Annex 11:	Results for MOAH C35-C50 mass fraction in paperboard	32
Annex 12:	Results for Total MOSH mass fraction in paperboard.....	33
Annex 13:	Results for MOSH C10-C16 mass fraction in paperboard	34
Annex 14:	Results for MOSH C16-C20 mass fraction in paperboard	35
Annex 15:	Results for MOSH C20-C25 mass fraction in paperboard	36
Annex 16:	Results for MOSH C25-C35 mass fraction in paperboard	37
Annex 17:	Results for MOSH C35-C40 mass fraction in paperboard	38
Annex 18:	Results for MOSH C40-C50 mass fraction in paperboard	39
Annex 19:	Results for Total MOAH mass fraction in muesli	40
Annex 20:	Results for MOAH C10-C16 mass fraction in muesli	41
Annex 21:	Results for MOAH C16-C25 mass fraction in muesli	42
Annex 22:	Results for MOAH C25-C35 mass fraction in muesli	43
Annex 23:	Results for MOAH C35-C50 mass fraction in muesli	43
Annex 24:	Results for Total MOSH mass fraction in muesli	44
Annex 25:	Results for MOSH C10-C16 mass fraction in muesli	45
Annex 26:	Results for MOSH C16-C20 mass fraction in muesli	46
Annex 27:	Results for MOSH C20-C25 mass fraction in muesli	47
Annex 28:	Results for MOSH C25-C35 mass fraction in muesli	48
Annex 29:	Results for MOSH C35-C40 mass fraction in muesli	49
Annex 30:	Results for MOSH C40-C50 mass fraction in muesli	49
Annex 31:	Results of the questionnaire	50

Executive summary

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM) has organised a proficiency test (FCM-20/01) for the determination of so-called mineral oil saturated hydrocarbons and mineral oil aromatic hydrocarbons (MOSH and MOAH) in muesli and paperboard to support the Commission Recommendation (EU) 84/2017. This proficiency test was open to National Reference Laboratories (NRLs) and Official Control Laboratories (OCLs).

Test item 1 consisted of chopped recycled paperboard, while test item 2 consisted of ground muesli contaminated with MOSH/MOAH. The homogeneity and stability of the test items were evaluated and the assigned values were derived from the results of expert laboratories.

Fourteen NRLs and seven OCLs registered to the exercise, but only 7 NRLs and 5 OCLs, representing 6 EU Member States and Switzerland, reported results.

These results were rated using $D\%$, z , z' and/or zeta (ζ) scores in accordance with ISO 13528:2015. Relative standard deviations for proficiency assessment ($\sigma_{pt,rel}$) ranging from 15 % to 30 % of the respective assigned values were set for the different MOSH/MOAH fractions, based on the perception of experts.

Most of the participating laboratories performed satisfactorily for the determination of the various MOSH/MOAH fractions in muesli and paperboard, when expressed as z scores.

The main outcome is that MOSH/MOAH analysis is still challenging for the NRLs and OCLs in the majority of the MS, even for not complicated samples as paperboard and muesli.

List of abbreviations and symbols

BfR	Bundesinstitut für Risikobewertung (Germany)
DG SANTE	Directorate General for Health and Food Safety
EURL	European Union Reference Laboratory
FCM	Food Contact Materials
LC-GC/FID	Liquid chromatography coupled with gas chromatography and flame ionization detection
ISO	International Organization for Standardization
JRC	Joint Research Centre
LOD	Limit of Detection
LOQ	Limit of Quantification
MOSH/MOAH	Mineral oil saturated/aromatic hydrocarbons
NRL	National Reference Laboratory
OCL	Official Control Laboratory
PT	Proficiency Test
SOP	Standard operating procedure
$D\%$	participant difference from the assigned value expressed as a percentage of x_{pt}
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 so-called mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) in muesli and paperboard, to support the Commission Recommendation (EU) 2017/84 on the monitoring of mineral oil hydrocarbons in food and in materials and articles intended to come into contact with food [1].

This PT was agreed with the Directorate General for Health and Food Safety (DG SANTE) as part of the EURL-FCM annual work programme 2020, thus complying with the mandate set in Regulation (EU) 2017/625 [2]. 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

The present PT aims to assess the performance of NRLs and OCLs in the determination of the mass fractions of MOSH and MOAH in muesli and paperboard.

This PT, organised in line with ISO 17043:2010 [3], is identified as "FCM-20/01".

3 Set up of the exercise

3.1 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 [2] 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.

3.2 Time frame

The organisation of the PT FCM-20/01 exercise was announced by e-mail to NRLs and OCLs on March 2, 2020 (Annex 1). The registration deadline was set to March 13, 2020. Due to the consequences of the COVID-19 pandemic on the working conditions in EU Member States and the Commission, samples were dispatched to participants on October 12, 2020. The deadline for reporting of results was set to December 15, 2020. This deadline was further extended until February 20, 2021.

3.3 Distribution

Each participant received:

- One bottle of Test item 1 (T1) containing 15 g chopped paperboard;
- One bottle of Test item 2 (T2) containing 40 g ground muesli;
- The "Test item accompanying letter" (Annex 2); and
- The "Confirmation of receipt form" (Annex 3).

Samples were sent under normal transport conditions at ambient temperature.

3.4 Instructions to participants

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

The measurands were defined as the mass fractions listed below measured in muesli and paperboard (expressed in mg kg⁻¹):

		Paperboard	Muesli
MOSH	Total (up to C35)	x	
	≥ n-C ₁₀ to ≤ n-C ₁₆	x	x
	> n-C ₁₆ to ≤ n-C ₂₀	x	x
	> n-C ₂₀ to ≤ n-C ₂₅	x	x
	> n-C ₂₅ to ≤ n-C ₃₅	x	x
	> n-C ₃₅ to ≤ n-C ₄₀	<i>optional</i>	x
	> n-C ₄₀ to ≤ n-C ₅₀	<i>optional</i>	x
MOAH	Total (up to C35)	x	
	≥ n-C ₁₀ to ≤ n-C ₁₆	x	x
	> n-C ₁₆ to ≤ n-C ₂₅	x	x
	> n-C ₂₅ to ≤ n-C ₃₅	x	x
	> n-C ₃₅ to ≤ n-C ₅₀	<i>optional</i>	x

Participants were asked (i) to check whether the bottles and vial were undamaged after transport, and (ii) to return the "Confirmation of receipt" form within 3 days after receipt of the samples.

Participants were instructed to store test items 1 and 2 at room temperature, away from any possible contaminations.

Participants were asked to perform two or three independent measurements and to report their calculated mean (x_i) for each of the measurands, the associated expanded measurement uncertainty ($U(x_i)$) together with the coverage factor (k) for total MOSH and total MOAH, 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. Since the homogeneity study was performed with 1 g for test item 1 and 5 g for test item 2, the recommended minimum sample intakes were set to 1 g for test item 1 and 5 g for test item 2.

Participants were informed that the procedure used for the analysis should resemble as closely as possible their routine procedures and should comply with the recommendations of the JRC Guidance document [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 4).

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

4 Test item

4.1 Preparation

The muesli and paperboard raw materials were kindly provided by the Bundesinstitut für Risikoberwertung (BfR, Germany).

The paperboard test items (T1) consisted of recycled paperboard chopped finely at pieces of 3x3 mm and delivered to JRC's Reference Material Processing facility in a stainless still canister of 10 L. Consequently, 15 g aliquots were filled in 100 mL amber glass vials.

The muesli test item (T2) consisted of finely milled muesli that was contaminated at the BfR. This was achieved by gas phase transfer of MOSH and MOAH from a recycled paperboard inserted at layers between the muesli powder. The material was then delivered to JRC's Reference Material Processing facility in a stainless still canister of 10 L. Subsequently, 35 g aliquots were filled in 100 mL amber glass vials.

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

4.2 Homogeneity and stability

Measurements for the homogeneity and short-term stability studies for muesli and paperboard were executed by an external collaborator and by the EURL-FCM, respectively. The statistical treatment of data for both test items were performed by the EURL-FCM.

The assessment of homogeneity was performed after the preparation of the test items and before distribution to the participants. For each test item, ten vials were randomly selected and analysed in duplicate. 1 g paperboard or 20 g of muesli were taken as aliquots for analysis. Results were evaluated according to ISO 13528:2015 [5]. Both items proved to be adequately homogeneous for the investigated analytes (Annex 5.1 and 5.2).

We have monitored only the stability of the TOTAL MOSH/MOAH (as markers for the other measurands).

Short-term stability studies were performed for muesli by the external collaborator and for the paperboard test material by the JRC at three different temperature, namely -25 °C, RT (20 °C) and 40 °C, for a period of 3 months. No significant trends were observed for any of the investigated measurands (Annex 5.3). Hence, the test items could be dispatched at room temperature.

The long-term stability study performed by the EURL-FCM for both materials confirmed the adequate stability of the two test items at room temperature over the whole period of the project (at least 2 years from the date of the homogeneity study and characterisation until the dispatch). Hence, the uncertainty contribution due to stability was set to zero ($u_{st} = 0$) for all the investigated analytes (Annex 5.4).

5 Assigned values and corresponding uncertainties

5.1 Assigned values

Assigned values (x_{pt}) were determined in the summer of 2018 in the frame of a BfR project. The two materials were send to four additional expert laboratories selected by the BfR. The collected results (Annex 6) were sent to the EURL-FCM for statistical treatment. The calculated assigned values (Table 1) were later confirmed by the EURL-FCM, based on the experimental results derived from the stability study.

Table 1: Assigned values (x_{pt}), associated standard uncertainties of the assigned values ($u(x_{pt})$), standard deviation for the PT assessment (σ_{pt}) and other relevant parameters for the assessment of results related to the determination of MOSH and MOAH fractions in muesli and paperboard.

Matrix	Min.Oil.	Fraction	x_{pt} mg/kg	$u(x_{pt})$ mg/kg	σ_{pt} mg/kg	$\sigma_{pt, rel}$	$u(x_{pt})/\sigma_{pt}$	Annex	Score
Paper-board	MOAH	Total	172	10	26	15%	0.39	7	D%
		C10-C16	2.91	0.27	0.87	30%	0.31	8	z'
		C16-C25	101.3	4.3	25.3	25%	0.17	9	z
		C25-C35	67.4	6.1	16.8	25%	0.36	10	z'
		C35-C50	Optional parameter, not for evaluating the performance					11	none
	MOSH	Total	969	26	145	15%	0.18	12	D%
		C10-C16	24.7	2.4	7.4	30%	0.33	13	z'
		C16-C20	254	7.1	38	15%	0.19	14	z
		C20-C25	300	12	45	15%	0.26	15	z
		C25-C35	390	13	59	15%	0.21	16	z
		C35-C40	Optional parameter, not for evaluating the performance					17	none
		C40-C50	Optional parameter, not for evaluating the performance					18	none
Muesli	MOAH	Total	2.74	0.08	0.82	30%	0.10	19	z, ζ
		C10-C16	0.35					20	none
		C16-C25	2.57	0.13	0.77	30%	0.17	21	z
		C25-C35	< 0.5					22	none
		C35-C50	< 0.5					23	none
	MOSH	Total	20.0	1.4	6.0	30%	0.23	24	z, ζ
		C10-C16	2.23	0.19	0.67	30%	0.28	25	z
		C16-C20	12.46	0.77	3.74	30%	0.21	26	z
		C20-C25	4.25	0.43	1.28	30%	0.34	27	z'
		C25-C35	1.23	0.16	0.37	30%	0.44	28	z'
		C35-C40	< 0.5					29	none
		C40-C50	< 0.5					30	none

5.2 Associated uncertainties

The associated standard uncertainties of the assigned values ($u(x_{pt})$, Table 1) 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]:

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

The uncertainty u_{char} is estimated according to the recommendations of ISO 13528:2015:

$$u_{char} = \frac{s}{\sqrt{p}} \quad \text{Eq. 2}$$

where "s" refers to the standard deviation of the mean values obtained by the expert laboratories and "p" refers to the number of expert laboratories.

5.1 Standard deviation for proficiency assessment, σ_{pt}

The relative standard deviations for PT assessment (σ_{pt}) were set, based on expert judgment, to 15-30 % of the respective assigned values for the different fractions and the total content of MOAH and MOSH in paperboard and in muesli (Table 1).

6 Evaluation of results

6.1 Scores and evaluation criteria

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

$$D\% = 100 * \frac{(x_i - x_{pt})}{x_{pt}} \quad \text{Eq. 3}$$

$$z = \frac{x_i - x_{pt}}{\sigma_{pt}} \quad \text{Eq. 4}$$

$$\zeta = \frac{x_i - x_{pt}}{\sqrt{u^2(x_i) + u^2(x_{pt})}} \quad \text{Eq. 5}$$

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 the criteria $u(x_{pt}) < 0.3 \sigma_{pt}$ is not met, the uncertainty of the assigned value ($u(x_{pt})$) should be taken into account by expanding the denominator of the z score and calculating the z' score as follows:

$$z'_i = \frac{x_i - x_{pt}}{\sqrt{\sigma_{pt}^2 + u^2(x_{pt})}} \quad \text{Eq. 6}$$

The interpretation of the z , z' and ζ performance scores is done according to ISO 13528:2015 [5]:

$ score \leq 2$	satisfactory performance	(green in Annexes 6 - 29)
$2 < score < 3$	questionable performance	(yellow in Annexes 6 - 29)
$ score \geq 3$	unsatisfactory performance	(red in Annexes 6 - 29)

The z and 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 $\sqrt{3}$, as recommended by Eurachem [7].

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

Fourteen NRLs and 7 OCLs registered to the exercise, but only 7 NRLs and 5 OCLs - representing 6 EU Member States and Switzerland - reported results. Moreover, only 10 laboratories filled in the questionnaire. The majority of the participants (10 out of 12) applied an on-line MOSH/MOAH separation technique, while two participants reported results by using the off-line MOSH/MOAH separation method.

6.3 Laboratory results and scorings

6.3.1 Performances

Annexes 7 to 30 present the reported results as tables and graphs for each measurand. National Reference Laboratories and Official Control Laboratories are denoted as L-xx and C-xx, respectively.

Out of the 24 measurands considered, the following evaluations were performed, as indicated in Table 1:

- The mass fractions reported for total MOSH and MOAH in paperboard were assessed using the $D\%$, since the measurand was ambiguously defined in the reporting interface. Some of the laboratories reported totals calculated up to C_{50} , while others calculated up to C_{35} as requested in the Accompanying letter (Annex 2) to comply with the recommendations of the EURL Guidance document [4].
- Three measurands were not evaluated, because they were considered as optional (cf. mass fraction of MOAH (C35-C50), MOSH (C35-C40 and C40-C50) in paperboard);
- Five measurands were not evaluated, as they were not detected in the test items (cf. mass fraction of MOAH (C10-C16, C25-C35 and C35-C50) and MOSH (C35-C40 and C40-C50) in muesli);
- Five measurands were evaluated using the z' score, since the criterion $u(x_{pt}) < 0.3 \sigma_{pt}$ was not met (cf. mass fraction of MOAH (C10-C16, C25-C35) and MOSH (C10-C16) in paperboard, and MOSH (C20-C25 and C25-C35) in muesli);
- The remaining 9 measurands were evaluated using the z score;
- In addition, the mass fractions of total MOSH and MOAH in muesli were evaluated using the ζ score, since the associated measurement uncertainties were reported per request.

Figure 1 presents the evaluated performance of the 10 laboratories having reported results for MOAH and MOASH in muesli. Most of the laboratories (above 70 %) reported satisfactory results according to the z score. The low rate of participation may be due to the fact that the presence of MOSH/MOAH in food and FCMs is not regulated and these substances are not routinely monitored by the NRLs and OCLs.

For paperboard, the laboratory performance should be evaluated base on the z -scores for the fractions. Again as with muesli, the response rate was low but slightly over 50 % (12/22 participants) and the performance could be assessed as satisfactory (Figure 2).

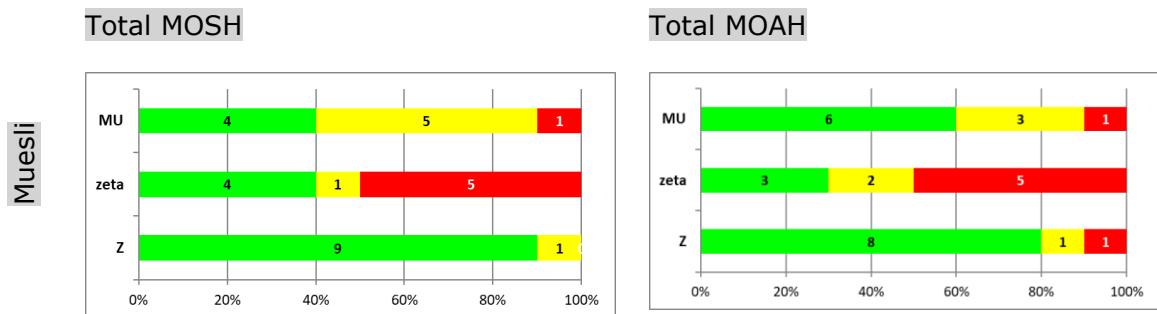


Figure 1: Overview of laboratory performance for total MOSH/MOAH in muesli. Corresponding number of laboratories included in the graph. Satisfactory, questionable and unsatisfactory performances indicated in green, yellow and red, respectively.

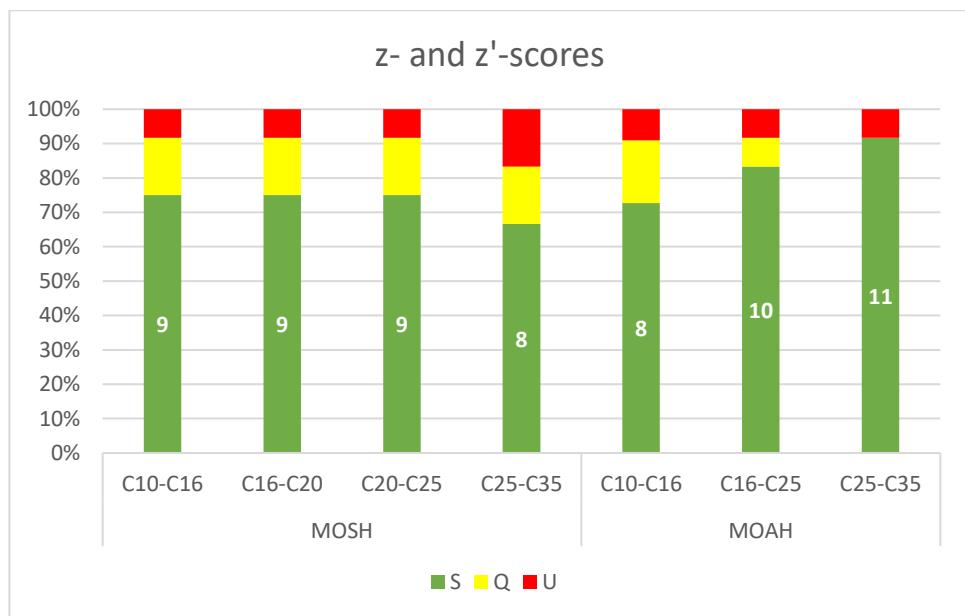


Figure 2 Overview of laboratory performance for the MOSH/MOAH fractions in the paperboard. Corresponding number of laboratories included in the graph. Satisfactory, questionable and unsatisfactory performances indicated in green, yellow and red, respectively

6.3.2 Additional information extracted from the questionnaire

The questionnaire was filled and submitted by 10 of the 12 participants who reported results and gave valuable information about their laboratory and their analytical methods. Annex 31 is summarising the experimental details.

Seven participants stated that they are accredited according to ISO/IEC 17025 for the determination of MOSH/MOAH in paperboard, while only 5 participants are accredited for the determination of MOSH/MOAH in muesli. Two of the participants stated to have little experience in the MOSH/MOAH analysis and have analysed less than 10 samples in 2019.

A similar sample preparation for paperboard was reported. An aliquot of the sample was soaked in an hexane/ethanol mixture 1:1 for 2 h at room temperature. Laboratory C21 soaked the samples overnight. Generally, no additional auxiliary method was applied to the extract. Only laboratory L17 carried out a silica-column clean-up and used an off-line MOSH/MOAH separation method, which is prone to contamination/interferences and could explain the significantly higher results reported for some of the MOAH fractions.

For muesli, different auxiliary techniques were used to remove interferences or for pre-concentration, due to significantly lower MOSH/MOAH levels in a fat-containing matrix (Figure 3). Laboratories L03 and L05 did not apply any auxiliary methods and reported, as expected, higher values for MOSH, as they probably included some non-MOSH peaks in the integration of the chromatographic hump.

Laboratories L16 and C01 reported several underestimated and/or overestimated values for most of the fractions in paperboard or muesli, without providing any experimental details in the questionnaire.

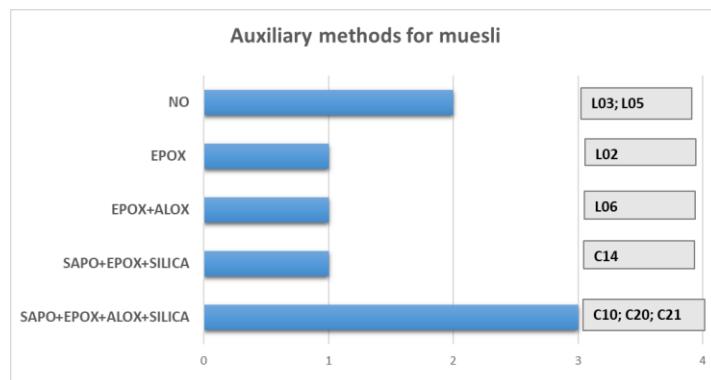


Figure 3 Auxiliary methods applied by the participants for the determination of MOSH/MOAH in the test item "muesli"

7 Conclusion

The proficiency test FCM-20/01 was organised to assess the analytical capabilities of EU NRLs and OCLs to determine the mass fractions of MOSH and MOAH in paperboard and muesli.

Despite the low attendance rate to this PT round, the overall performance of the participants in the determination of the 16 measurands investigated was satisfactory (above 70 %).

The low rate of participation may be attributed to (i) the absence of regulatory limits yet to be enforced, (ii) the sophisticated instrumentation required by this type of analysis, and (iii) the relatively new methodology under development that remains to be ring-trial validated.

Acknowledgements

The 12 laboratories listed hereafter are kindly acknowledged for their participation in the PT. The EURL-FCM would like to acknowledge the Reference Material Unit of the JRC for processing the materials and delivering high quality test items.

Organisation	Country
Health Board, Central Chemistry Laboratory	Estonia
Service Commun des Laboratoires - Laboratoire de Bordeaux	France
Landesbetrieb Hessisches Landeslabor	Germany
CVUA-MEL	Germany
Bundesinstitut für Risikobewertung (BfR)	Germany
CVUA Stuttgart	Germany
LAVES, Institut für Bedarfsgegenständ	Germany
Dublin Public Analyst's Laboratory	Ireland
The Netherlands Food and Consumer Product Safety Authority (NVWA)	Netherlands
Dr. A. Verwey B.V.	Netherlands
CENTRO NACIONAL ALIMENTACION (CNA)-AESAN	Spain
Kantonales Labor Zürich	Switzerland

References

- [1] Commission Recommendation (EU) 2017/84 of 16 January 2017 on the monitoring of mineral oil hydrocarbons in food and in materials and articles intended to come into contact with food. Official Journal of the European Union, OJ L 12/95, 2017.
- [2] Commission Regulation, (EU) No 2017/625 of The European Parliament and of The Council of 15 march 2017 on official controls and other official activities performed to ensure the application of food and feed law, rules on animal health and welfare, plant health and plant protection products. Official Journal of the European Union, OJ L 95/1, 2017.
- [3] ISO/IEC 17043:2010 "*Conformity assessment – General requirements for proficiency testing*". International Organisation for Standardization, Geneva, Switzerland..
- [4] S. Bratinova, E. Hoekstra (Editors) Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials, Luxembourg: Publications Office of the European Union, 2019 ISBN 978-92-76-00172-0, doi:10.2760/208879, JRC115694,
- [5] ISO 13528:2015 "*Statistical methods for use in proficiency testing by interlaboratory comparisons*". International Organisation for Standardization, Geneva, Switzerland.
- [6] ISO 5725-3:1994 "*Accuracy (trueness and precision) of measurement methods and results - Part 3: Intermediate measures of the precision of a standard measurement method*". International Organisation for Standardization, Geneva, Switzerland.
- [7] S L R Ellison and A Williams (Eds). Eurachem/CITAC guide: Quantifying Uncertainty in Analytical Measurement, Third edition, (2012) ISBN 978-0-948926-30-3. Available from www.eurachem.org.

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(2020)1297324 - 02/03/2020

Geel, 2 March 2020

(sent by e-mail)

Subject: Invitation to participate in Proficiency Testing round “FCM-20/01”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-20/01 "Determination of MOSH/MOAH in paperboard and muesli"**. You will receive two test items 1) recycled paperboard and 2) muesli. You will be requested to analyse individual fraction cuts and total MOAH/MOAH, following the requirements of the "Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials on mineral oil hydrocarbons" in the frame of Recommendation (EU) 2017/84.

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

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=2501>

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 13th of March 2020.

Please forward this invitation to the Official Control Laboratories (OCLs) in your network as well as to any OCL in the field of food and feed control that would be interested in participating. They should also register electronically by using the same link above

Samples will be dispatched on the 31st of March 2020.

The deadline for submission of results is the 25th of May 2020.

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

Kind regards,

/signed electronically in Ares/
Dr. P. Dehouck
FCM-20/01 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

 Ref. Ares(2020)5445740 - 13/10/2020



EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate F - Health, Consumers and Reference Materials (Geel)
Food and Feed Compliance



Ispra, 12 October 2020
JRC.F.5/SB/mt/ARES(2020) 20-058

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

Subject: Participation in FCM-20/01 - Determination of MOSH/MOAH in paperboard and muesli

Dear «Title» «Surname»,

Thank you for participating in the FCM-20/01 proficiency test (PT) for the "**Determination of MOSH/MOAH in paperboard and muesli**".

The measurands are defined in the JRC Guidance (2019)¹

- **For paperboard - mass fractions (mg kg^{-1}) of**

MOSH:	MOAH:
total MOSH (up to C ₃₅)	Total MOAH (up to C ₃₅)
MOSH \geq n-C ₁₀ to \leq n-C ₁₆	MOAH \geq n-C ₁₀ to \leq n-C ₁₆
MOSH > n-C ₁₆ to \leq n-C ₂₀	MOAH > n-C ₁₆ to \leq n-C ₂₅
MOSH > n-C ₂₀ to \leq n-C ₂₅	MOAH > n-C ₂₅ to \leq n-C ₃₅
MOSH > n-C ₂₅ to \leq n-C ₃₅	MOAH > n-C ₃₅ to \leq n-C ₅₀

- **For muesli - mass fractions (mg kg^{-1}) of**

MOSH:	MOAH:
total MOSH	Total MOAH
MOSH \geq n-C ₁₀ to \leq n-C ₁₆	MOAH \geq n-C ₁₀ to \leq n-C ₁₆
MOSH > n-C ₁₆ to \leq n-C ₂₀	MOAH > n-C ₁₆ to \leq n-C ₂₅
MOSH > n-C ₂₀ to \leq n-C ₂₅	MOAH > n-C ₂₅ to \leq n-C ₃₅
MOSH > n-C ₂₅ to \leq n-C ₃₅	MOAH > n-C ₃₅ to \leq n-C ₅₀
MOSH > n-C ₃₅ to \leq n-C ₄₀	
MOSH > n-C ₄₀ to \leq n-C ₅₀	

¹ Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials, Luxembourg: Publications Office of the European Union, 2019 ISBN 978-92-76-00172-0, doi:10.2760/208879, JRC115694

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 15 g chopped paperboard on 3x3 mm parts;
- test item 2: one bottle, containing 40 g ground muesli;
- the “Confirmation of receipt” form.

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

The test items 1 and 2 can be stored at room temperature away of any possible contaminations
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 and **should be in compliance with the JRC Guidance**¹.

Please report separately for each item, the following:

- the **mean** of your two or three measurements results (in mg kg⁻¹) for each of the measurands;
- the associated expanded **uncertainty** (in mg kg⁻¹) for total MOSH and total MOAH;
- 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 1 g for test item 1 and 5 g for test item 2 and therefore 1 g and 5 g 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: «**Partkey**».

The system will guide you through the reporting procedure. Do not forget to submit and confirm when required. **Separately next week you will be send a link to a questionnaire via EU Survey platform requiring information about the method applied.**

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 **15/12/2020**.

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.

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. Please be aware of the existence of an appeal procedure in case you disagree with your scores.

Do not hesitate to contact me for further information.

With kind regards,

/signed electronically in Ares/

Dr. Stefanka Bratinova
FCM-20/01 Coordinator

Cc: H. Emons (Head of Unit, Food & Feed Compliance, F.5),
E. Hoekstra (Operating Manager EURL-FCM)
P. Dehouck (FCM-20/01 Deputy PT Coordinator)

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

Geel, 20 October 2020

Ms. Marleen Alttoa
Health Board
Central Chemistry Laboratory
Paldiski mnt 81
10617 Tallinn
ESTONIA

Subject: "Confirmation receipt" form - FCM-20/0 - Determination of MOSH/MOAH in paperboard and muesli

The parcels with the two PT's test items and the instructions were dispatch yesterday.
You have to receive them today or tomorrow.

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:

Stefanka Bratinova
FCM-20/01 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

Save a backup on your local computer (disable if you are using a public/shared computer)
EURL FCM PT 2020/01 - MOSH/MOAH in paperboard and muesli

Fields marked with * are mandatory.

Pages

Start

Experience

Paperboard

Muesli

MOSH/MOAH separation

GC details

Quantification

Other (last)

A Previous experience

* A.1 Name and Surname

* A.2 Institution

* A.3 Your e-mail address

 @

* A.4 Years of experience with MOSH/MOAH analysis

- no experience
- less than 1 year
- 1 - 2 years
- 3 - 5 years
- more than 5 years

* A.5 Number of samples analysed for MOSH/MOAH in 2019

- less than 10
- 10 to 100
- 100 to 500
- more than 500

A.6 Your experience depending on the type of matrices

	Rank (0-3) according to the legend above
* Dry, low fat content sample (< 4% oils/fat)	//
* Higher fat/oil content sample (> 4 % oils/fat)	//
* Oils & Fats	//
* Paperboard	//
* Infant Formula	//

* A.7 Status of your method for MOSH/MOAH in paperboard

- method under development
- method validated
- method accredited

* A.8 Status of your method for MOSH/MOAH in muesli

- method under development
- method validated
- method accredited

Previous

Next

B Sample preparation for paperboard

* B.1 Sample intake

 g

* B.2 Extraction solvent and volume

* B.3 time/Temperature during the extraction

* B.4 Did you apply epoxidation for elimination of interferences?

- Yes for determination of MOAH
- Yes for determination of both MOSH and MOAH
- No

* B.5 What type of epoxidation did you apply?

- in ethanol
- in dichloromethane

B.6 Please describe shortly the epoxidation procedure (mCPBA volume, concentration, t/T, stop reagent)

* B.7 Did you apply ALOX clean-up for determination of MOSH to eliminate the interferences from the n-alkanes?

- Yes
- No

B.8 Please describe shortly the ALOX procedure

* B.9 Did you apply additional column clean-up?

- Yes, before epoxidation
- Yes, after epoxidation
- Yes, before and after epoxidation
- No

B.10 Please describe shortly the column clean-up procedure (type and amount of adsorbent/ eluents, volumes)

[Previous](#)

[Next](#)

C Sample preparation for muesli

* C.1 Sample intake

g

* C.2 Solvent and volume used for the extraction in ml

* C.3 time/Temperature during the extraction

* C.4 Did you apply saponification ?

- Yes, after the extraction
- Yes, simultaneously
- No

C.5 Please describe shortly the saponification step (KOH concentration and volume, t/T)

* C.6 Did you apply epoxidation ?

- Yes, for determination of MOAH
- Yes, for determination of both MOSH and MOAH
- No

C.8 Please describe shortly the epoxidation procedure (mCPBA volume, concentration, t/T, stop reagent)

* C.9 Did you apply ALOX for determination of MOSH?

- Yes
- No

C.10 Please describe shortly the ALOX procedure

* C.11 Did you apply additional column clean-up?

- Yes, before epoxidation
- Yes, after epoxidation
- Yes, before and after epoxidation
- No

C.12 Please describe shortly the column clean-up procedure (type and amount of adsorbent/ eluents, volumes)

C.13 Please specify "other"

Previous

Next

Save a backup on your local computer (disable if you are using a public/shared computer)

EURL FCM PT 2020/01 - MOSH/MOAH in paperboard and muesli

Fields marked with * are mandatory.

Pages

Start

Experience

Paperboard

Muesli

MOSH/MOAH separation

GC details

Quantification

Other (last)

D Analytical setup for MOSH/MOAH separation

- * D.1 Set-up used
- on-line double channel
 - on-line single channel
 - semi-online (collection of fractions in auto-sampler & injection into GC from vial)
 - off-line

[Previous](#) [Next](#)

E Details of GC

- * E.1 Injection system used
- direct coupling with HPLC
 - cold on-column
 - PTV with liner
 - split
 - other

- * E.3 Type of column and dimensions

- * E.4 Oven temperature program

- * E.5 Pressure program

- * E.6 Did you have problem with 
- the baseline
 - the peak tailing/broadening
 - the solvent peak
 - the blank
 - interferences

[Previous](#) [Next](#)

F Quantification

* F.1 How do you quantify MOSH/MOAH? Against which internal standard?

* F.2 Did you remove any riding peaks ?

F.3 To demonstrate the control over the procedure for MOAH, please fill in the respective area ratios. Please report the ratio for each of the 3 replicates in the same cell, separated by comma. If you prefer you could upload an excel file below ⓘ

	TBB/1MN	2MN/1MN	5B/1MN
paperboard	xx xx	xx xx	xx xx
blank PB	xx xx	xx xx	xx xx
muesli	xx xx	xx xx	xx xx
blank MUS	xx xx	xx xx	xx xx
theoretical ratio based on formulation in the IS	xx xx	xx xx	xx xx

F.4 Please upload your excel file with the area ratio ⓘ

* F.5 Please upload MOSH/MOAH chromatograms of the paperboard sample with peak/hump integration and fractionation. Make sure that the humps and the integration lines are clearly visible ⓘ

* F.6 Please upload MOSH/MOAH chromatograms of the muesli sample with peak/hump integration and fractionation. Make sure that the humps and the integration lines are clearly visible ⓘ

* F.7 LOQ for total MOAH is set as:

- max(LOQ) of the various fractions
- sum(LOQ) of the various fractions
- other

* F.8 Please describe "other"

[Previous](#)

[Next](#)

Annex 5: Homogeneity and stability results

5.1 Paperboard - Homogeneity (all values expressed in mg kg⁻¹)

	PaperBoard		MOAH					
	Total		C10-C16		C16-C25		C25-C35	
1	199.1	201.8	4.08	3.95	110.1	111.2	84.9	86.6
2	198.6	195.8	3.96	3.91	109.5	109.0	85.2	82.9
3	214.1	204.4	4.17	4.44	116.4	110.7	93.5	89.3
4	214.5	199.4	4.28	3.77	118.7	106.1	93.5	85.3
5	224.1	190.8	5.59	3.68	108.3	109.9	99.9	81.0
6	195.0	200.5	3.74	3.87	116.8	110.4	83.0	86.8
7	197.8	202.0	3.91	3.99	108.2	108.9	85.6	89.1
8	222.4	215.3	4.03	4.29	104.5	116.0	83.1	95.9
9	202.3	196.5	3.82	3.73	110.1	108.3	88.1	84.5
10	194.1	195.3			107.4	106.7	84.2	83.7
Mean	203.2		4.07		110.4		87.3	
s _w	8.9		0.48		4.3		5.7	
s _x	7.3		0.27		2.2		2.7	
s _s	3.8		0		0		0	
s _{pt}	26.4		0.87		25.3		17.9	
0.3 s _{pt}	7.9		0.26		7.6		5.4	
s _s < 0.3 s _{pt}	passed		passed		passed		passed	

	PaperBoard		MOSH							
	Total		C10-C16		C16-C20		C20-C25		C25-C35	
1	1010	1035	20.46	19.34	255.6	256.1	323.3	331.8	260.7	270.5
2	1009	1007	18.74	18.87	245.7	247.1	318.5	317.7	269.4	269.3
3	1023	1094	19.34	19.33	255.8	251.6	326.7	339.3	264.1	320.8
4	1022	1042	18.90	19.75	253.7	255.7	328.0	341.8	266.1	271.5
5	1034	1013	19.44	19.80	254.7	253.5	328.9	323.6	271.0	264.4
6	1044	1025	19.64	19.75	260.1	254.3	332.7	327.8	272.3	268.6
7	1014	1006	19.04	19.75	250.0	249.1	320.0	320.5	266.2	263.3
8	1030	1011	21.00	21.50	256.1	264.5	334.2	324.5	255.3	248.7
9	1015	999	19.33	18.89	246.4	249.5	318.9	319.5	268.3	260.0
10	1009	1031	18.56	19.82	248.3	255.1	322.0	329.2	263.9	271.8
Mean	1024		19.56		253.1		326.4		268.3	
s _w	20		0.48		3.1		5.6		13.4	
s _x	15		0.67		4.3		5.8		10	
s _s	6		0.58		3.7		4.2		3.1	
s _{pt}	145		7.41		38.1		45.1		58.5	
0.3 s _{pt}	44		2.22		11.4		13.5		17.6	
s _s < 0.3 s _{pt}	passed		passed		passed		passed		passed	

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 Muesli - Homogeneity (all values expressed in mg kg⁻¹)

	Muesli	MOAH
Total = C16-C25		
1	2.62	2.72
2	2.75	2.71
3	2.72	2.38
4	2.81	2.85
5	2.73	2.81
6	2.75	2.63
7	2.82	2.90
8	2.78	2.70
9	2.69	2.78
10	2.71	2.76
Mean	2.73	
s_w	0.09	
s_x	0.09	
s_s	0.06	
σ_{pt}	0.82	
$0.3 \sigma_{pt}$	0.25	
$s_s < 0.3 \sigma_{pt}$	passed	

	Muesli		MOSH					
	Total	C10-C16	C16-C20	C20-C25	C25-C35			
1	21.83	22.78	1.91	1.81	12.45	12.98	4.84	5.05
2	22.00	22.64	1.86	1.82	12.94	13.35	5.03	4.73
3	22.75	21.56	1.85	2.01	13.41	12.42	5.18	4.994
4	21.91	22.17	1.88	1.90	13.35	13.24	5.19	5.38
5	22.21	22.07	1.91	1.93	13.26	13.91	5.19	5.31
6	20.46	21.25	1.98	1.89	13.92	13.12	5.32	5.05
7	22.08	22.26	1.92	1.95	13.73	14.09	5.43	5.61
8	21.81	20.75	1.96	1.79	13.45	13.36	5.22	5.43
9	22.70	23.19	1.90	1.93	13.31	13.87	5.28	5.48
10	21.24	21.59	1.89	1.88	13.39	13.30	5.39	5.41
Mean	22.0	1.90	13.3		5.23		1.50	
s_w	0.5	0.06	0.4		0.14		0.11	
s_x	0.6	0.03	0.4		0.20		0.09	
s_s	0.5	0.00	0.2		0.18		0.05	
σ_{pt}	6.0	0.67	3.7		1.28		0.40	
$0.3 \sigma_{pt}$	1.8	0.20	1.1		0.38		0.12	
$s_s < 0.3 \sigma_{pt}$	passed	passed	passed		passed		passed	

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.

5.3 Stability study for Total MOSH and MOAH in paperboard and muesli

Muesli

by JRC-Ispra	2018	2020	diff		0.3*σ _{pt}	σ _{pt}
MOSH, mg/kg	27.6	28.5	0.9	pass	1.8	6
MOAH, mg/kg	3.3	3.5	0.2	pass	0.24	0.8

by external collaborator	-25°C	RT	40°C	diff (RT - 25°C)	diff (40°C - 25°C)		0.3*σ _{pt}	σ _{pt}
MOSH, mg/kg	18.4	19.9	19.5	1.5	1.1	pass	1.8	6
MOAH, mg/kg	2.5	2.7	2.7	0.2	0.2	pass	0.24	0.8

Paperboard

by JRC-Ispra	2018	2020	diff		0.3*σ _{pt}	σ _{pt}
MOSH, mg/kg	998	981	-17.1	pass	43.5	145
MOAH, mg/kg	186.5	185	-1.5	pass	7.74	25.8

by JRC-Ispra	5°C	RT	40°C	diff (RT - 25°C)	diff (40°C - 25°C)		0.3*σ _{pt}	σ _{pt}
MOSH, mg/kg	1037	1031	1037	-6	0	pass	43.5	145
MOAH, mg/kg	205.5	201.1	198.9	-4.4	-6.6	pass	7.74	25.8

Annex 6: Test item characterisation

Paperboard – MOSH (data in mg/kg)

fractions	Lab 1		Lab 2		Lab 3		Lab 4		x_{pt}	u_{char}
C10-C16	21	24	26	29	20	19	33	26	24.7	2.3
C16-C20	250	262	238	235	262	253	272	258	254	6.1
C20-C25	299	315	270	267	327	313	306	306	300	11
C25-C35	388	393	370	364	424	392	411	380	390	8.6
Total (<C35)	958	994	904	895	1032	977	1022	970	969	24

Paperboard – MOAH (data in mg/kg)

fractions	Lab 1		Lab 2		Lab 3		Lab 4		x_{pt}	u_{char}
C10-C16	2.6	3.1	3.4	3.5	2.6	2.3	3.2	2.6	2.91	0.21
C16-C25	110	115	95	96	103	99	96	97	101.3	3.9
C25-C35	81	82	63	58	71	72	54	58	67.38	5.7
Total (<C35)	194	200	163	158	176	173	153	158	172	9.3

Muesli – MOSH (data in mg/kg)

fractions	Lab 1		Lab 2		Lab 3		Lab 4		Lab 5	x_{pt}	u_{char}
C10-C16	1.7	1.7	2.6	2.7	2.3	2.5	2.5	2.5	1.9	2.23	0.18
C16-C20	11	11	12	11	15	15	11	12	13	12.46	0.75
C20-C25	4.1	4.0	3.6	3.3	5.0	5.4	3.4	3.3	5.2	4.25	0.41
C25-C35	1.3	1.0	1.1	1.0	1.7	1.6	0.8	<0,2	1.5	1.23	0.16
Total (<C35)	18.1	17.7	19.3	18.0	24.0	24.5	17.7	17.0	22	20.01	1.32

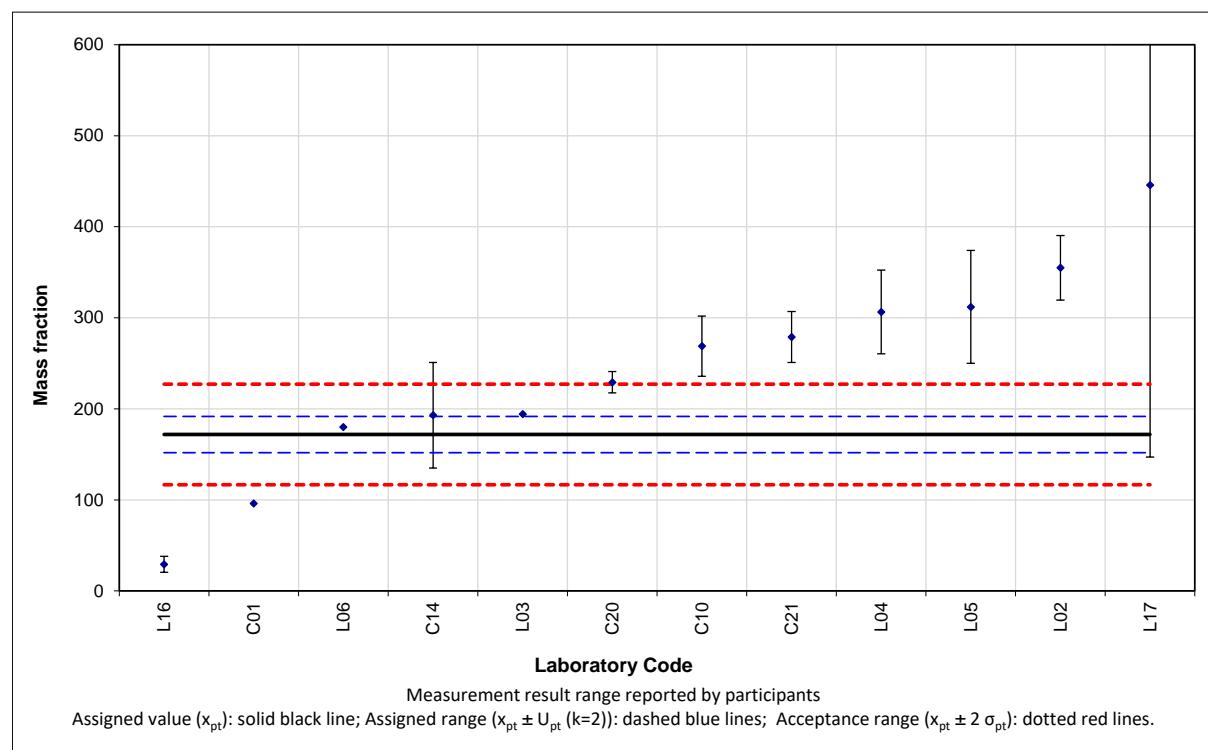
Muesli – MOAH (data in mg/kg)

fractions	Lab 1		Lab 2		Lab 3		Lab 4		Lab 5	x_{pt}	u_{char}
C16-C25	3.0	2.7	2.6	2.7	2.5	2.5	2.1	2.2	2.7	2.57	0.12
Total (<C35)	3.0	2.7	2.6	2.7	2.9	2.9	2.6	2.6	2.7	2.74	0.06

Annex 7: Results for total MOAH mass fraction in paperboard

$x_{pt} = 172$; $u(x_{pt}) = 10$; $\sigma_{pt} = 26$ (all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique	$D\%$	Fraction Used
C01	96			On-line LC-GC-FID	-44%	C10-C50
C10	268.87	33.02	2	On-line LC-GC-FID	56%	C10-C50
C14	193	58	2	On-line LC-GC-FID	12%	C10-C35
C20	229.2	11.77	2.776	On-line LC-GC-FID	33%	C10-C50
C21	279	28	2	On-line LC-GC-FID	62%	C10-C50
L02	355	35.5	2	On-line LC-GC-FID	107%	C10-C50
L03	194.38			On-line LC-GC-FID	13%	C10-C35
L04	306.56	46	1	On-line LC-GC-FID	78%	
L05	312	62	2	Off-line GC-FID	82%	C10-C50
L06	180			On-line LC-GC-FID	5%	C10-C35
L16	29.3	8.8	2	Off-line GC-FID	-83%	
L17	446	299	2	Off-line GC-FID	160%	C10-C50

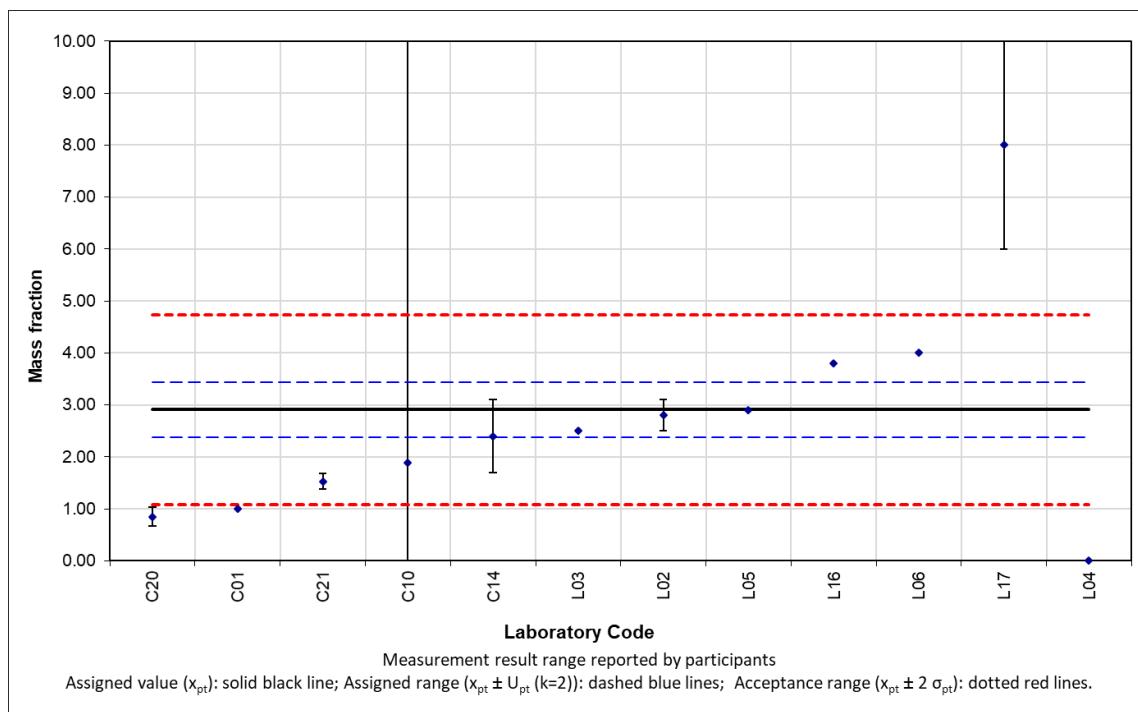


Annex 8: Results for MOAH C10-C16 mass fraction in paperboard

$x_{pt} = 2.91$; $u(x_{pt}) = 0.27$; $\sigma_{pt} = 0.87$ (all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique	z' score
C01	1			On-line LC-GC-FID	-2.09
C10	1.88	36.35	2	On-line LC-GC-FID	-1.13
C14	2.4	0.7	2	On-line LC-GC-FID	-0.56
C20	0.85	0.18	4.303	On-line LC-GC-FID	-2.25
C21	1.53	0.15	2	On-line LC-GC-FID	-1.51
L02	2.8	0.3	2	On-line LC-GC-FID	-0.12
L03	2.5			On-line LC-GC-FID	-0.45
L04					
L05	2.9			Off-line GC-FID	-0.01
L06	4			On-line LC-GC-FID	1.20
L16	3.8			Off-line GC-FID	0.98
L17	8	2	2	Off-line GC-FID	5.58

Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)

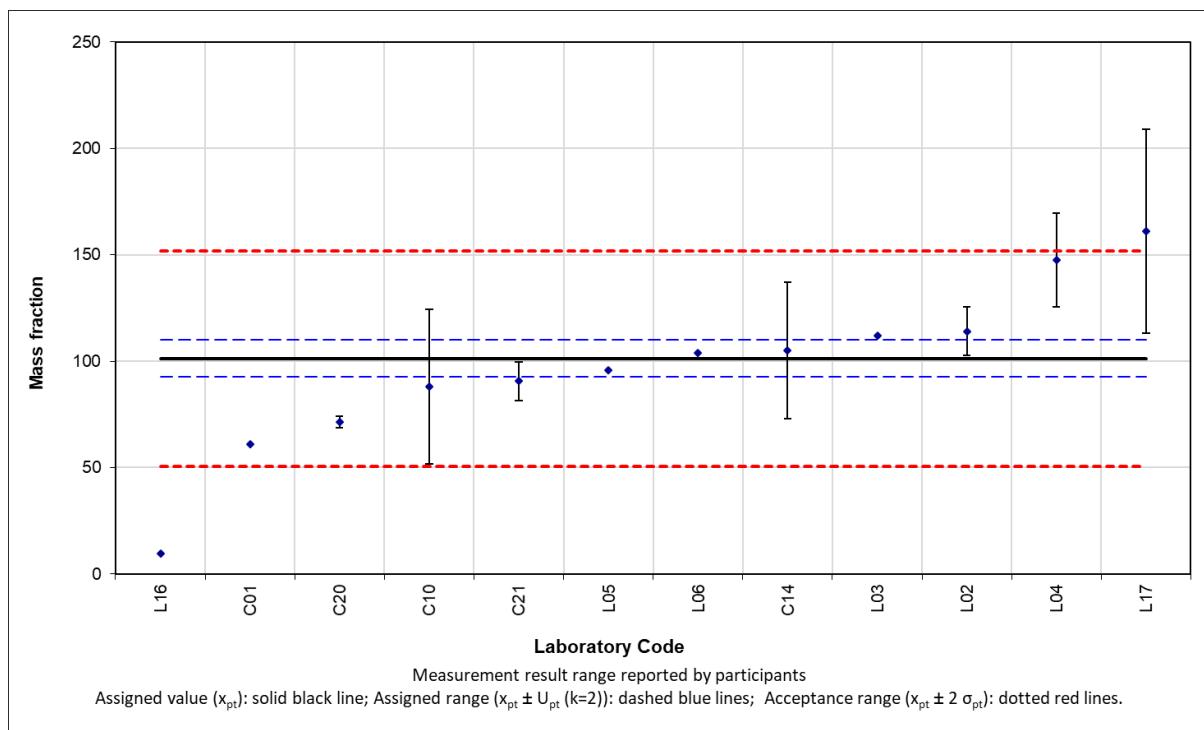


Annex 9: Results for MOAH C16-C25 mass fraction in paperboard

$x_{pt} = 101.3$; $u(x_{pt}) = 4.3$; $\sigma_{pt} = 25.3$ (all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique	z score
C01	61			On-line LC-GC-FID	-1.59
C10	88.19	36.35	2	On-line LC-GC-FID	-0.52
C14	105	32	2	On-line LC-GC-FID	0.15
C20	71.38	2.723	2.776	On-line LC-GC-FID	-1.18
C21	90.6	9.1	2	On-line LC-GC-FID	-0.42
L02	114	11.4	2	On-line LC-GC-FID	0.50
L03	111.9			On-line LC-GC-FID	0.42
L04	147.48	22	1	On-line LC-GC-FID	1.82
L05	95.6			Off-line GC-FID	-0.23
L06	104			On-line LC-GC-FID	0.11
L16	9.5			Off-line GC-FID	-3.62
L17	161	48	2	Off-line GC-FID	2.36

Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)

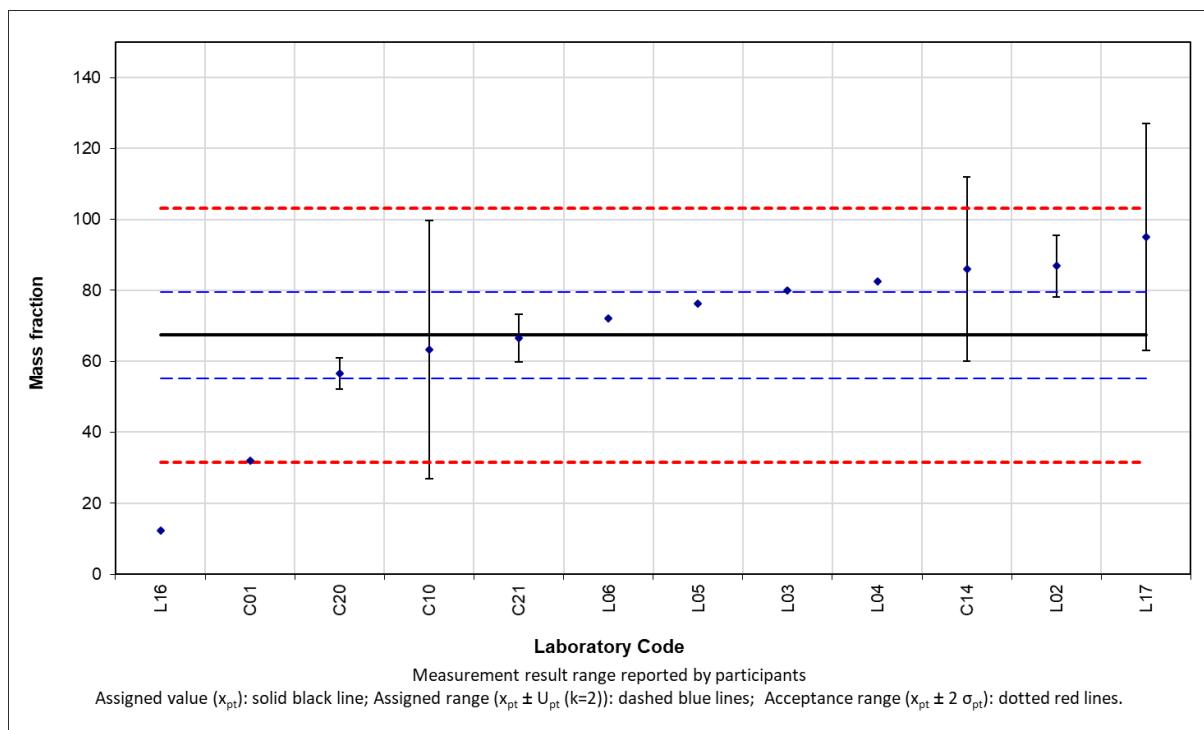


Annex 10: Results for MOAH C25-C35 mass fraction in paperboard

$x_{pt} = 67.4$; $u(x_{pt}) = 6.1$; $\sigma_{pt} = 16.8$ (all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique	z' score
C01	32			On-line LC-GC-FID	-1.97
C10	63.33	36.35	2	On-line LC-GC-FID	-0.23
C14	86	26	2	On-line LC-GC-FID	1.04
C20	56.57	4.431	2.776	On-line LC-GC-FID	-0.60
C21	66.5	6.7	2	On-line LC-GC-FID	-0.05
L02	86.9	8.7	2	On-line LC-GC-FID	1.09
L03	79.97			On-line LC-GC-FID	0.70
L04	82.55		1	On-line LC-GC-FID	0.85
L05	76.3			Off-line GC-FID	0.50
L06	72.1			On-line LC-GC-FID	0.26
L16	12.2			Off-line GC-FID	-3.08
L17	95	32	2	Off-line GC-FID	1.54

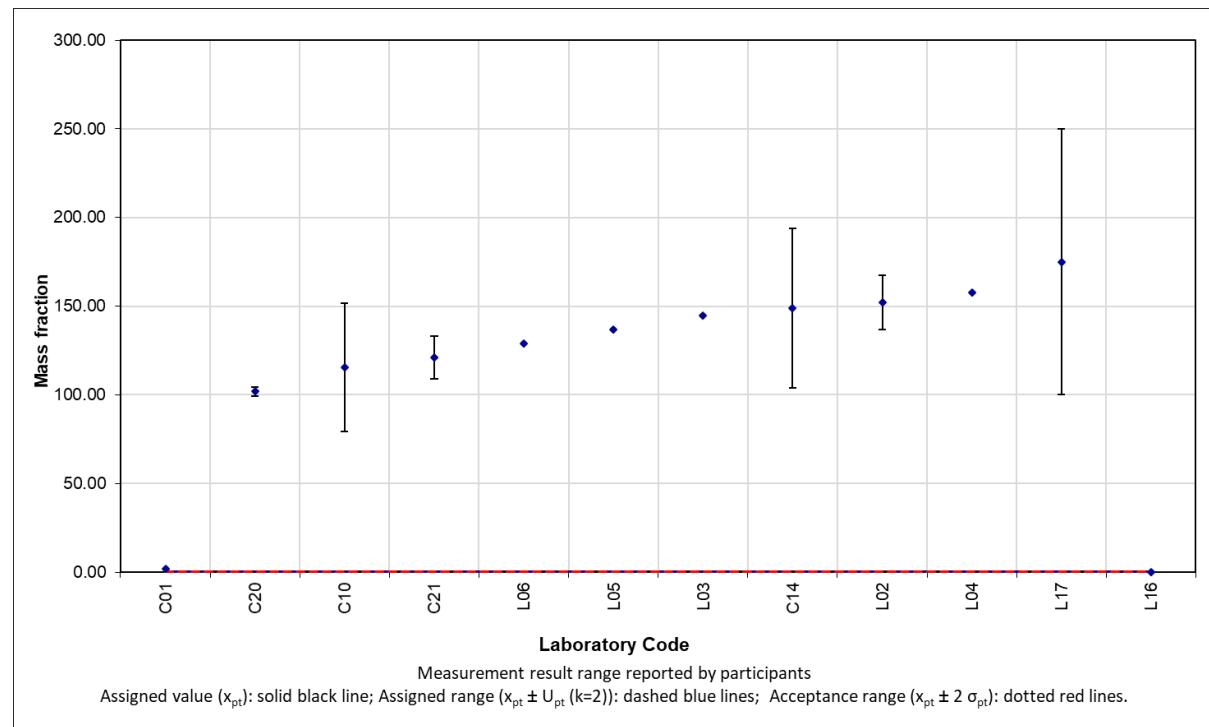
Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)



Annex 11: Results for MOAH C35-C50 mass fraction in paperboard

(all values in mg kg⁻¹)

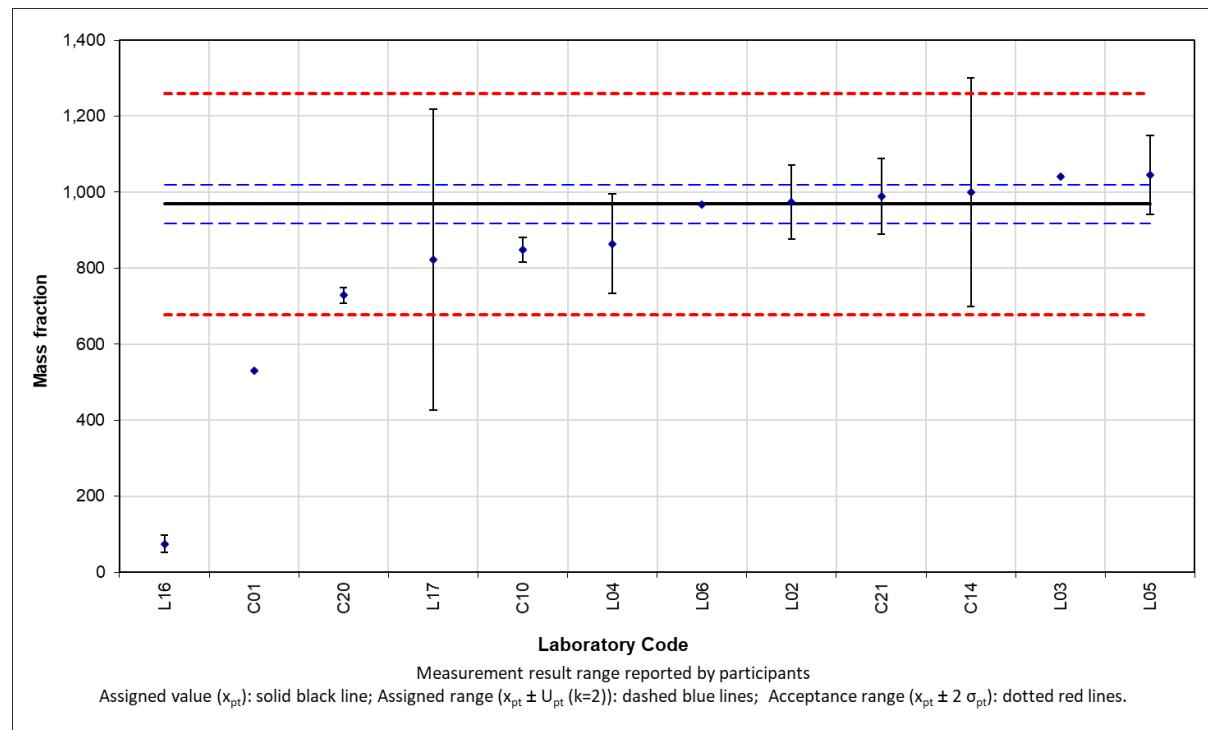
Lab Code	x_i	\pm	k	Technique
C01	1.7			On-line LC-GC-FID
C10	115.48	36.35	2	On-line LC-GC-FID
C14	149	45	2	On-line LC-GC-FID
C20	101.83	2.393	2.776	On-line LC-GC-FID
C21	121	12	2	On-line LC-GC-FID
L02	152	15.2	2	On-line LC-GC-FID
L03	144.66			On-line LC-GC-FID
L04	157.49			On-line LC-GC-FID
L05	137			Off-line GC-FID
L06	129			On-line LC-GC-FID
L16				
L17	175	75	2	Off-line GC-FID



Annex 12: Results for Total MOSH mass fraction in paperboard

$x_{pt} = 969$; $u(x_{pt}) = 26$; $\sigma_{pt} = 145$ (all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique	$D\%$	Fraction Used
C01	530			On-line LC-GC-FID	-45.3%	C10-C50
C10	848.25	33.02	2	On-line LC-GC-FID	-12.5%	C10-C50
C14	1000	300	2	On-line LC-GC-FID	3.2%	C10-C35
C20	728.17	19.76	2.447	On-line LC-GC-FID	-24.9%	C10-C50
C21	989	99	2	On-line LC-GC-FID	2.1%	C10-C35
L02	974	97.4	2	On-line LC-GC-FID	0.5%	C10-C35
L03	1040.3			On-line LC-GC-FID	7.4%	C10-C35
L04	864.4	130	1	On-line LC-GC-FID	-10.8%	C10-C35
L05	1045	104	2	Off-line GC-FID	7.8%	C10-C50
L06	968			On-line LC-GC-FID	-0.1%	C10-C35
L16	74.5	22.4	2	Off-line GC-FID	-92.3%	C10-C50
L17	822	396	2	Off-line GC-FID	-15.2%	C10-C35

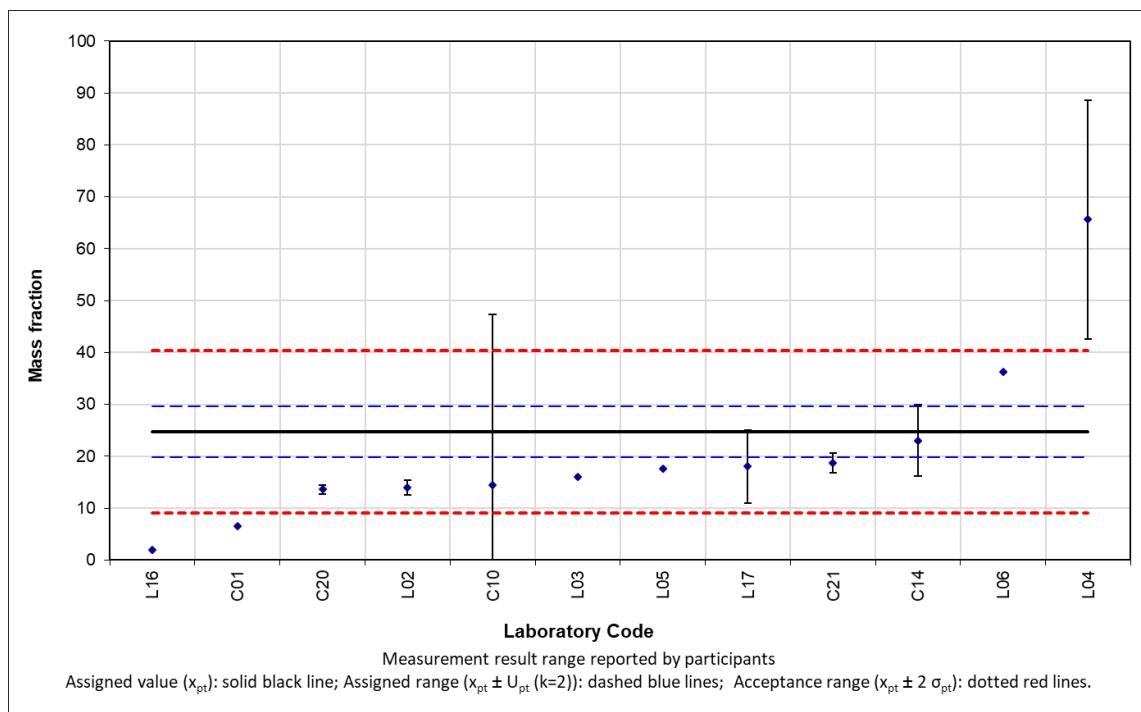


Annex 13: Results for MOSH C10-C16 mass fraction in paperboard

$x_{pt} = 24.7$; $u(x_{pt}) = 2.4$; $\sigma_{pt} = 7.4$ (all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique	z' score
C01	6.5			On-line LC-GC-FID	-2.33
C10	14.37	33.02	2	On-line LC-GC-FID	-1.32
C14	23	6.9	2	On-line LC-GC-FID	-0.22
C20	13.57	0.947	2.447	On-line LC-GC-FID	-1.43
C21	18.7	1.9	2	On-line LC-GC-FID	-0.77
L02	14	1.4	2	On-line LC-GC-FID	-1.37
L03	16.05			On-line LC-GC-FID	-1.11
L04	65.64	23	1	On-line LC-GC-FID	5.25
L05	17.6			Off-line GC-FID	-0.91
L06	36.2			On-line LC-GC-FID	1.48
L16	2			Off-line GC-FID	-2.91
L17	18	7	2	Off-line GC-FID	-0.86

Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)

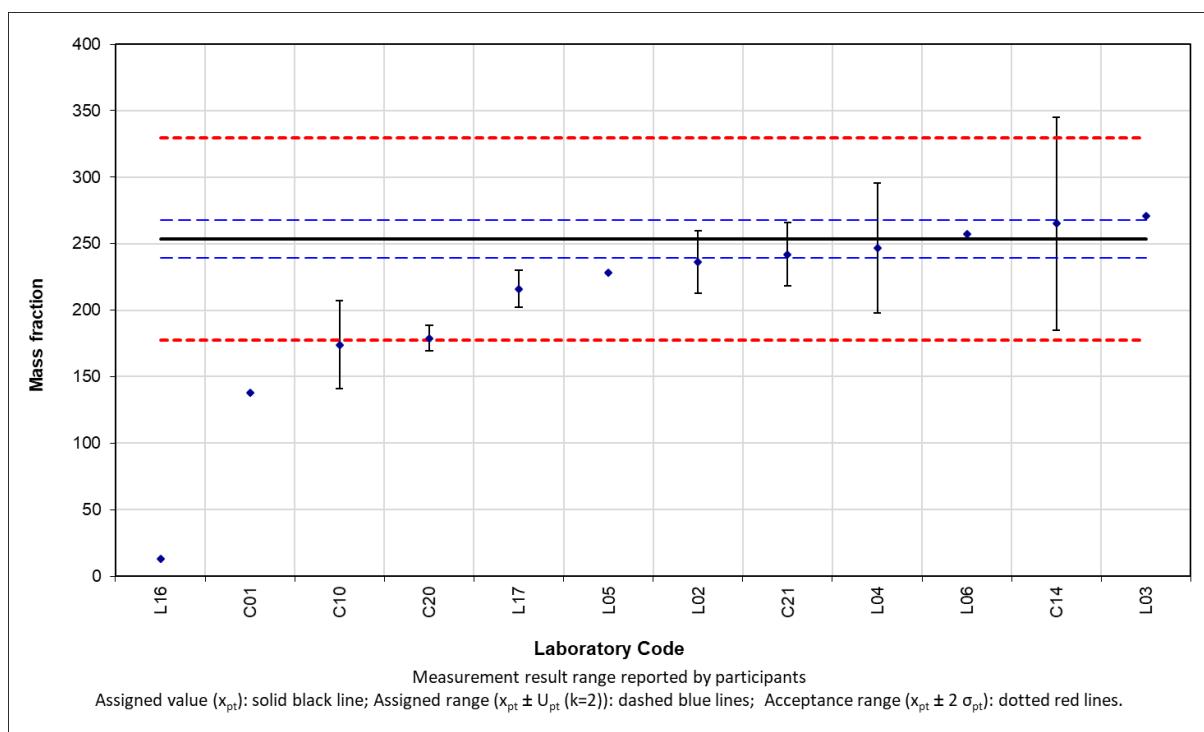


Annex 14: Results for MOSH C16-C20 mass fraction in paperboard

$x_{pt} = 254$; $u(x_{pt}) = 7.1$; $\sigma_{pt} = 38$ (all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique	z score
C01	138			On-line LC-GC-FID	-3.04
C10	173.94	33.02	2	On-line LC-GC-FID	-2.10
C14	265	80	2	On-line LC-GC-FID	0.30
C20	178.92	9.618	2.447	On-line LC-GC-FID	-1.96
C21	242	24	2	On-line LC-GC-FID	-0.31
L02	236	23.6	2	On-line LC-GC-FID	-0.46
L03	270.56			On-line LC-GC-FID	0.44
L04	246.69	49	1	On-line LC-GC-FID	-0.18
L05	228			Off-line GC-FID	-0.68
L06	257			On-line LC-GC-FID	0.09
L16	13.2			Off-line GC-FID	-6.32
L17	216	14	2	Off-line GC-FID	-0.99

Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)

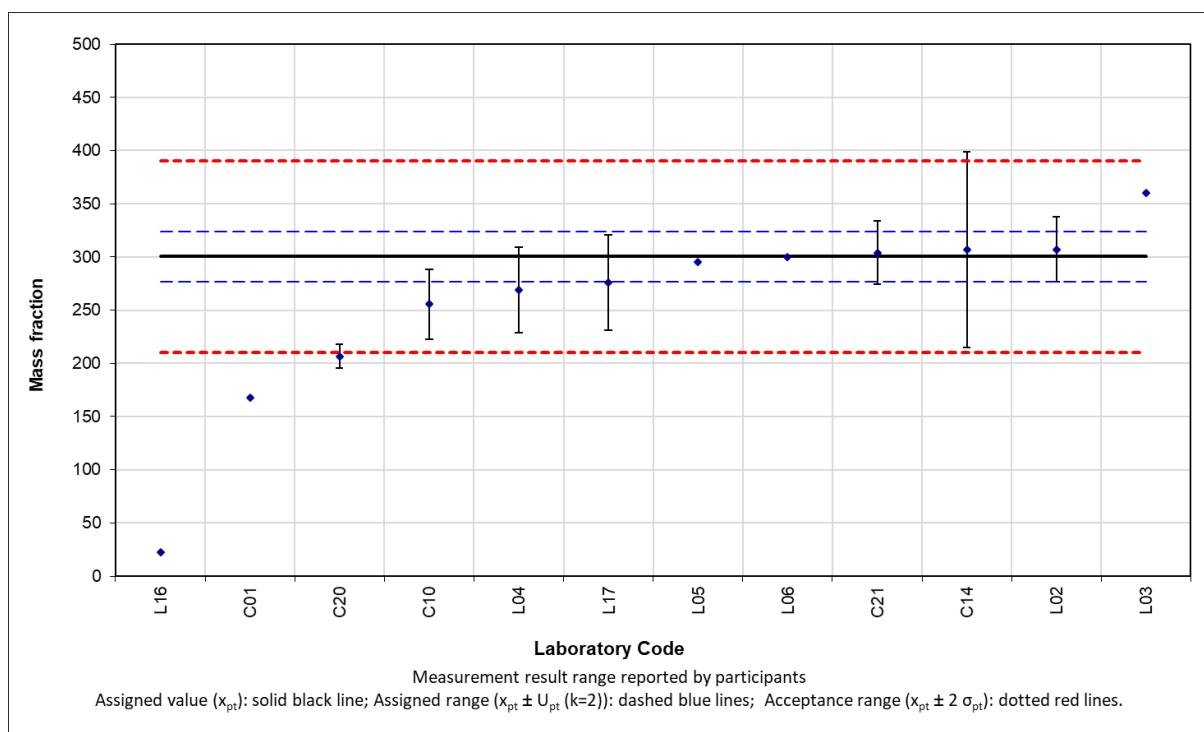


Annex 15: Results for MOSH C20-C25 mass fraction in paperboard

$x_{pt} = 300$; $u(x_{pt}) = 12$; $\sigma_{pt} = 45$ (all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique	z score
C01	168			On-line LC-GC-FID	-2.94
C10	255.6	33.02	2	On-line LC-GC-FID	-0.99
C14	307	92	2	On-line LC-GC-FID	0.15
C20	206.55	11.174	2.447	On-line LC-GC-FID	-2.08
C21	304	30	2	On-line LC-GC-FID	0.08
L02	307	30.7	2	On-line LC-GC-FID	0.15
L03	359.87			On-line LC-GC-FID	1.32
L04	269.07	40	1	On-line LC-GC-FID	-0.69
L05	295			Off-line GC-FID	-0.12
L06	300			On-line LC-GC-FID	-0.01
L16	22.8			Off-line GC-FID	-6.16
L17	276	45	2	Off-line GC-FID	-0.54

Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)

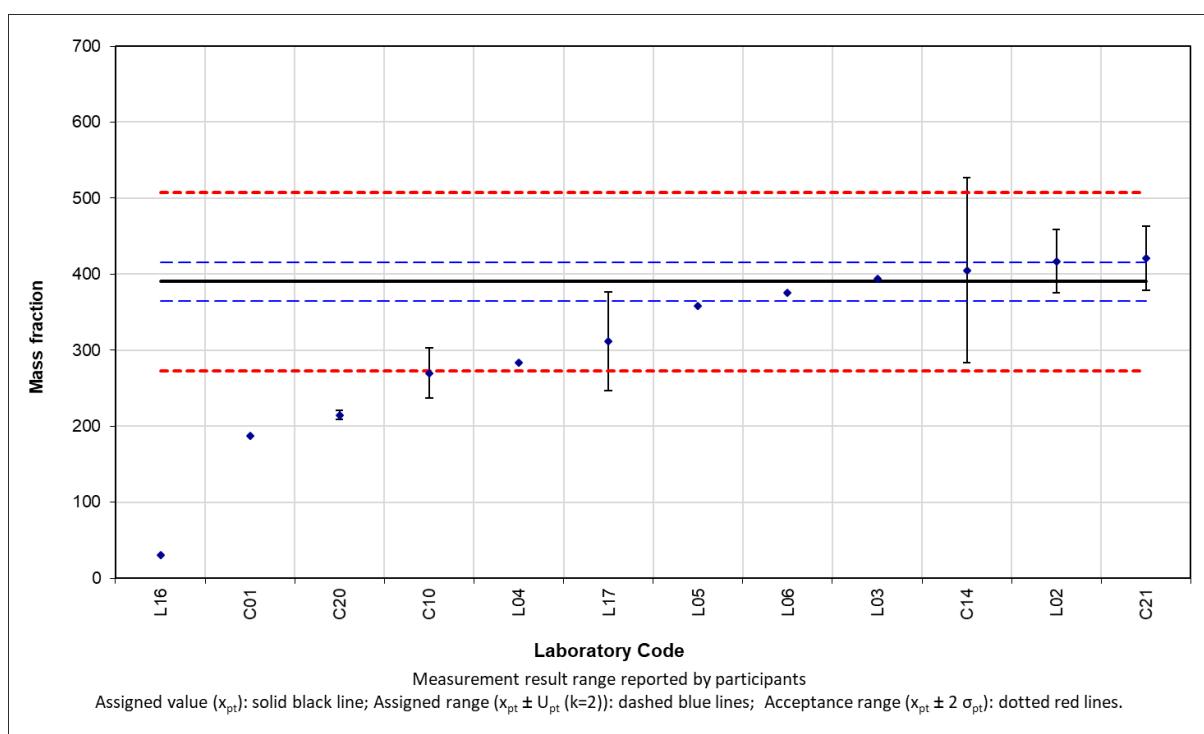


Annex 16: Results for MOSH C25-C35 mass fraction in paperboard

$$x_{pt} = 390 ; u(x_{pt}) = 13 ; \sigma_{pt} = 59 \text{ (all values in mg kg}^{-1}\text{)}$$

Lab Code	x_i	\pm	k	Technique	z score
C01	187			On-line LC-GC-FID	-3.47
C10	269.57	33.02	2	On-line LC-GC-FID	-2.06
C14	405	122	2	On-line LC-GC-FID	0.25
C20	214.71	5.807	2.447	On-line LC-GC-FID	-3.00
C21	421	42	2	On-line LC-GC-FID	0.53
L02	417	41.7	2	On-line LC-GC-FID	0.46
L03	393.81			On-line LC-GC-FID	0.06
L04	283		1	On-line LC-GC-FID	-1.83
L05	358			Off-line GC-FID	-0.55
L06	375			On-line LC-GC-FID	-0.26
L16	30			Off-line GC-FID	-6.15
L17	312	65	2	Off-line GC-FID	-1.34

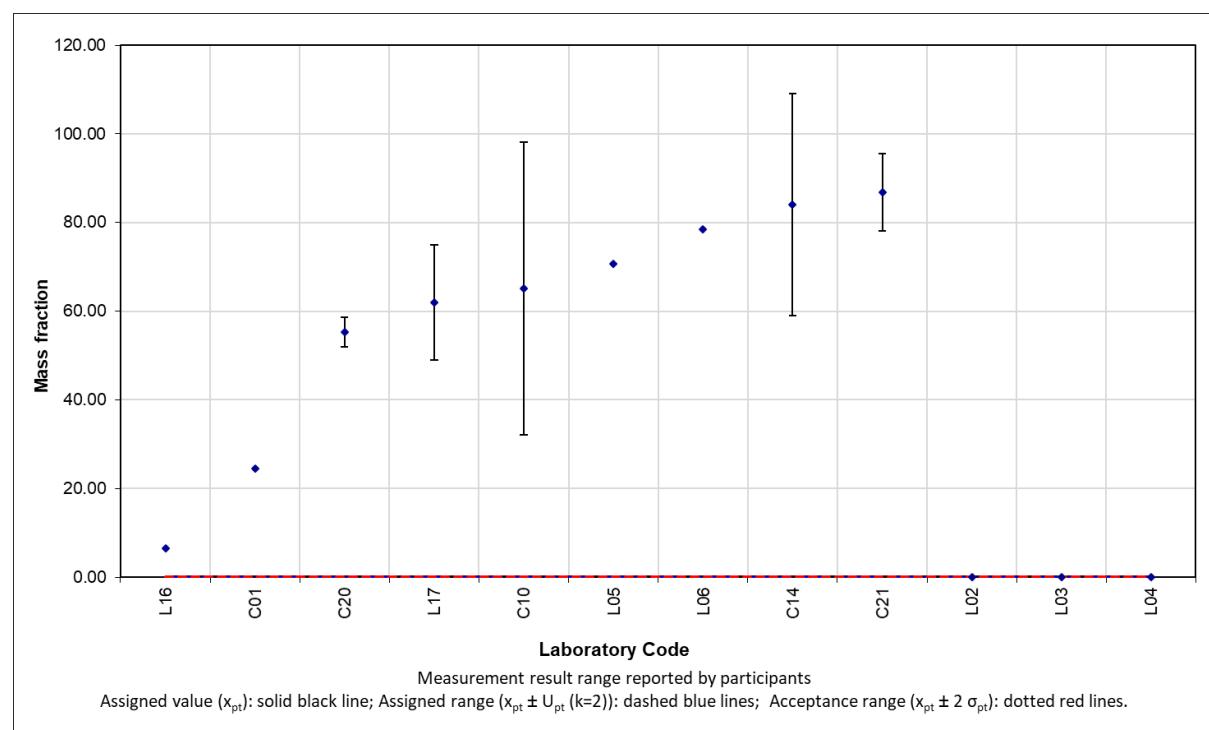
Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)



Annex 17: Results for MOSH C35-C40 mass fraction in paperboard

(all values in mg kg⁻¹)

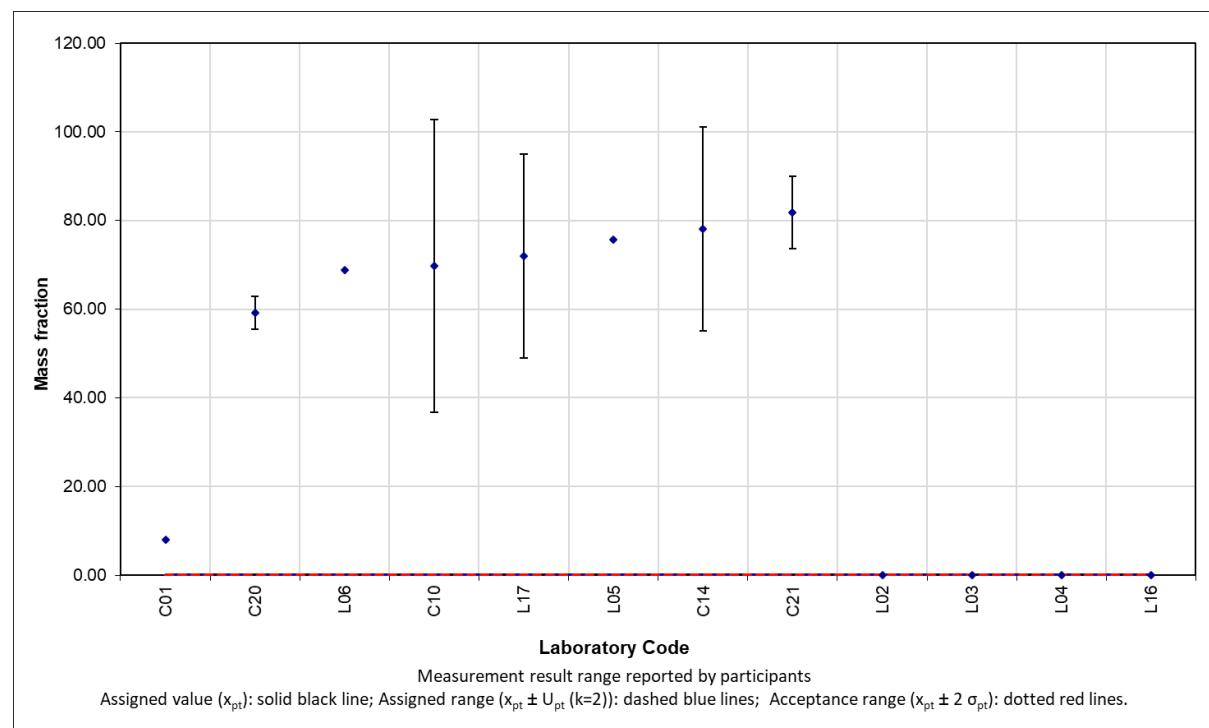
Lab Code	x_i	\pm	k	Technique
C01	24.5			On-line LC-GC-FID
C10	65.04	33.02	2	On-line LC-GC-FID
C14	84	25	2	On-line LC-GC-FID
C20	55.27	3.364	2.447	On-line LC-GC-FID
C21	86.8	8.7	2	On-line LC-GC-FID
L02				
L03				
L04				
L05	70.7			Off-line GC-FID
L06	78.5			On-line LC-GC-FID
L16	6.5			Off-line GC-FID
L17	62	13	2	Off-line GC-FID



Annex 18: Results for MOSH C40-C50 mass fraction in paperboard

(all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique
C01	8			On-line LC-GC-FID
C10	69.73	33.02	2	On-line LC-GC-FID
C14	78	23	2	On-line LC-GC-FID
C20	59.16	3.668	2.447	On-line LC-GC-FID
C21	81.8	8.2	2	On-line LC-GC-FID
L02				
L03				
L04				
L05	75.6			Off-line GC-FID
L06	68.8			On-line LC-GC-FID
L16				
L17	72	23	2	Off-line GC-FID

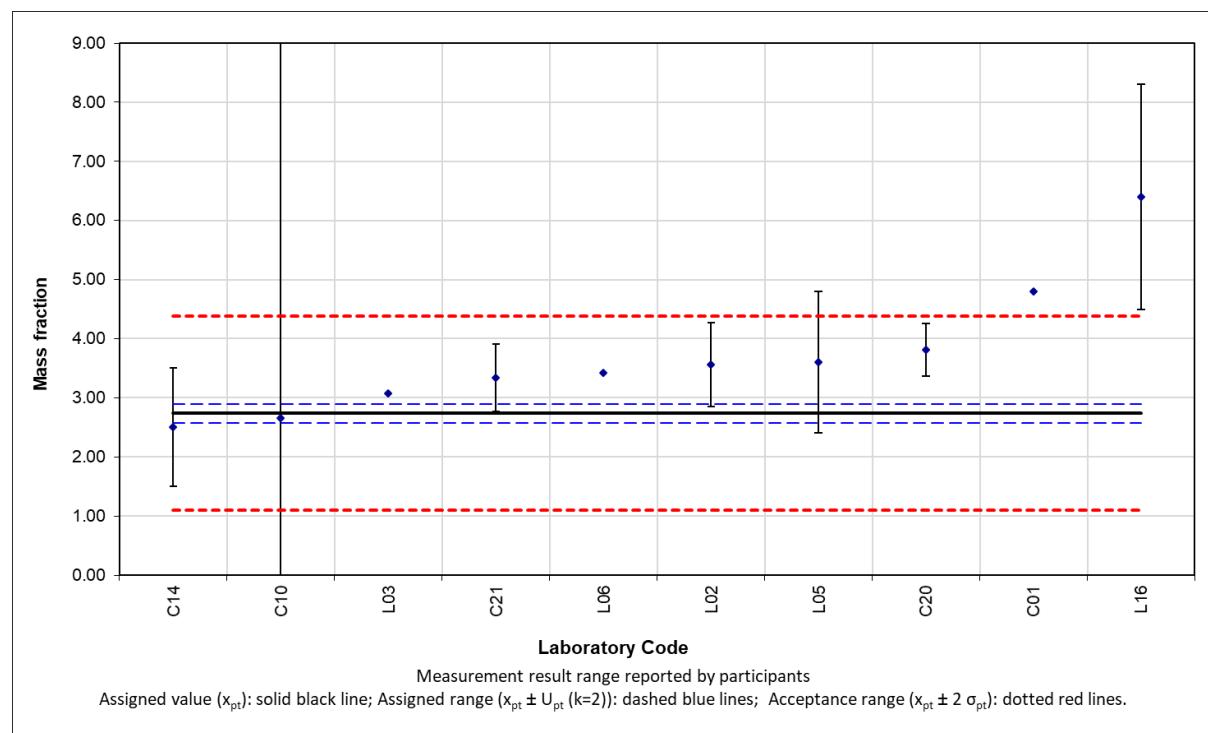


Annex 19: Results for Total MOAH mass fraction in muesli

$x_{pt} = 2.74$; $u(x_{pt}) = 0.08$; $\sigma_{pt} = 0.82$ (all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique	z score	ζ score	MU
C01	4.8			On-line LC-GC-FID	2.52	25.70	b
C10	2.66	18.21	2	On-line LC-GC-FID	-0.09	-0.01	c
C14	2.5	1	2	On-line LC-GC-FID	-0.29	-0.46	a
C20	3.81	0.44	2	On-line LC-GC-FID	1.31	4.59	a
C21	3.34	0.57	2	On-line LC-GC-FID	0.74	2.04	a
L02	3.56	0.71	2	On-line LC-GC-FID	1.01	2.27	a
L03	3.07			On-line LC-GC-FID	0.41	4.17	b
L05	3.6	1.2	2	Off-line GC-FID	1.05	1.43	a
L06	3.42			On-line LC-GC-FID	0.83	8.53	b
L16	6.4	1.9	2	Off-line GC-FID	4.47	3.84	a

Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)



Annex 20: Results for MOAH C10-C16 mass fraction in muesli(all values in mg kg⁻¹)

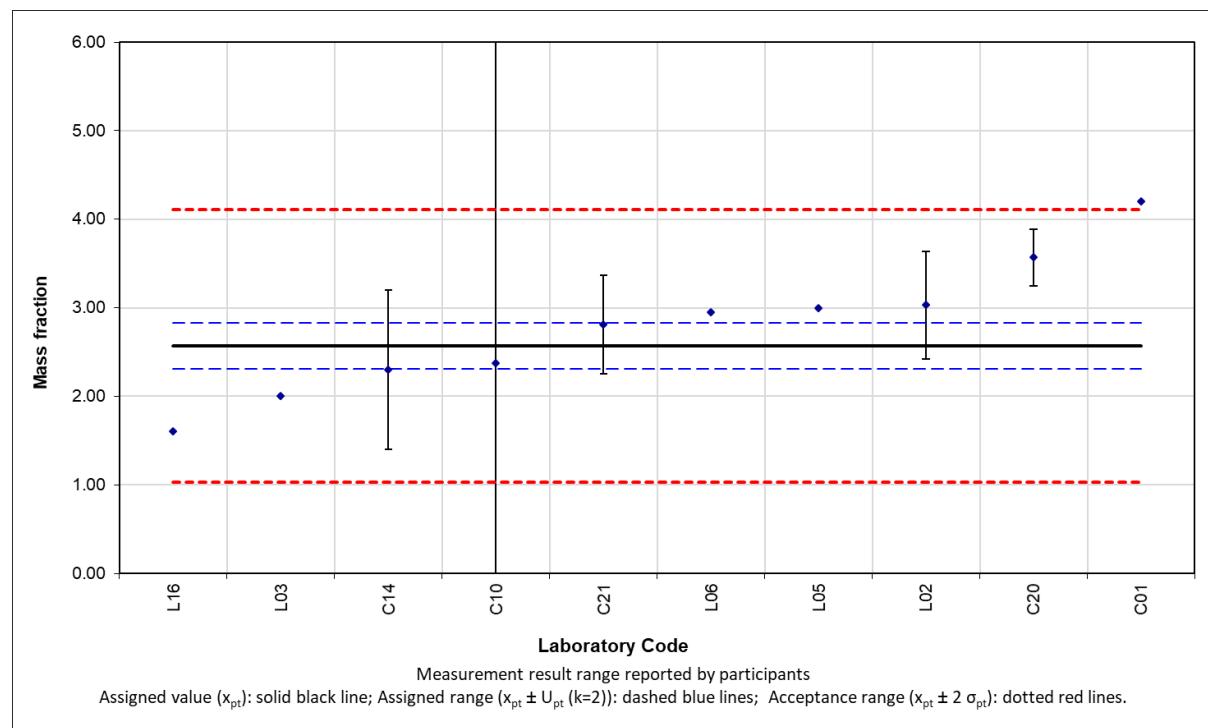
Lab Code	x_i	\pm	k	Technique
C01	0.4			On-line LC-GC-FID
C10	0.26	30.33	2	On-line LC-GC-FID
C14	< 0.5			On-line LC-GC-FID
C20	< 0.5			On-line LC-GC-FID
C21	< 0.5			On-line LC-GC-FID
L02	0.29	0.06	2	On-line LC-GC-FID
L03	0.26			On-line LC-GC-FID
L05	0.44			Off-line GC-FID
L06	< 0.5			On-line LC-GC-FID
L16	0.8			Off-line GC-FID

Annex 21: Results for MOAH C16-C25 mass fraction in muesli

$$x_{pt} = 2.57 ; u(x_{pt}) = 0.13 ; \sigma_{pt} = 0.77 \text{ (all values in mg kg}^{-1}\text{)}$$

Lab Code	x_i	\pm	k	Technique	z score
C01	4.2			On-line LC-GC-FID	2.11
C10	2.37	30.33	2	On-line LC-GC-FID	-0.26
C14	2.3	0.9	2	On-line LC-GC-FID	-0.35
C20	3.57	0.32	2	On-line LC-GC-FID	1.30
C21	2.81	0.56	2	On-line LC-GC-FID	0.31
L02	3.03	0.61	2	On-line LC-GC-FID	0.60
L03	2			On-line LC-GC-FID	-0.74
L05	3			Off-line GC-FID	0.56
L06	2.95			On-line LC-GC-FID	0.49
L16	1.6			Off-line GC-FID	-1.26

Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)



Annex 22: Results for MOAH C25-C35 mass fraction in muesli(all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique
C01	< 0.25			On-line LC-GC-FID
C10	< 0.1			On-line LC-GC-FID
C14	< 0.5			On-line LC-GC-FID
C20	0			On-line LC-GC-FID
C21	< 0.5			On-line LC-GC-FID
L02	0.24	0.048	2	On-line LC-GC-FID
L03	0.77			On-line LC-GC-FID
L05	0.11			Off-line GC-FID
L06	< 0.5			On-line LC-GC-FID
L16	3.1			Off-line GC-FID

Annex 23: Results for MOAH C35-C50 mass fraction in muesli(all values in mg kg⁻¹)

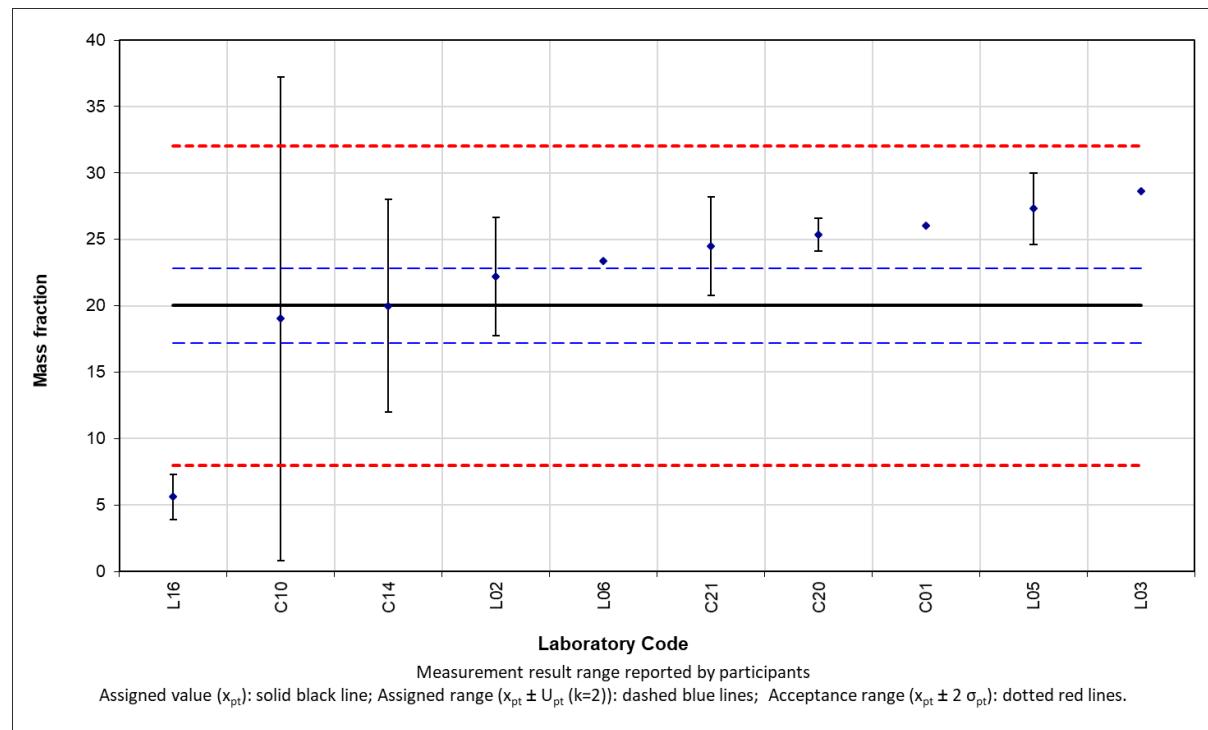
Lab Code	x_i	\pm	k	Technique
C01	< 0.25			On-line LC-GC-FID
C10	0.1	18.21	2	On-line LC-GC-FID
C14	< 0.5			On-line LC-GC-FID
C20	0			On-line LC-GC-FID
C21	< 0.5			On-line LC-GC-FID
L02	< 0.2			On-line LC-GC-FID
L03	0.03			On-line LC-GC-FID
L05	< 0.1			Off-line GC-FID
L06	< 0.5			On-line LC-GC-FID
L16	0.8			Off-line GC-FID

Annex 24: Results for Total MOSH mass fraction in muesli

$x_{pt} = 20.0$; $u(x_{pt}) = 1.4$; $\sigma_{pt} = 6.0$ (all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique	z score	ζ score	MU
C01	26			On-line LC-GC-FID	1.00	4.29	b
C10	19.02	18.21	2	On-line LC-GC-FID	-0.16	-0.11	c
C14	20	8	2	On-line LC-GC-FID	0.00	0.00	a
C20	25.33	1.23	2	On-line LC-GC-FID	0.89	3.49	b
C21	24.5	3.7	2	On-line LC-GC-FID	0.75	1.94	a
L02	22.2	4.45	2	On-line LC-GC-FID	0.36	0.83	a
L03	28.63			On-line LC-GC-FID	1.44	6.17	b
L05	27.3	2.7	2	Off-line GC-FID	1.21	3.75	b
L06	23.35			On-line LC-GC-FID	0.56	2.39	b
L16	5.6	1.7	2	Off-line GC-FID	-2.40	-8.82	a

Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)

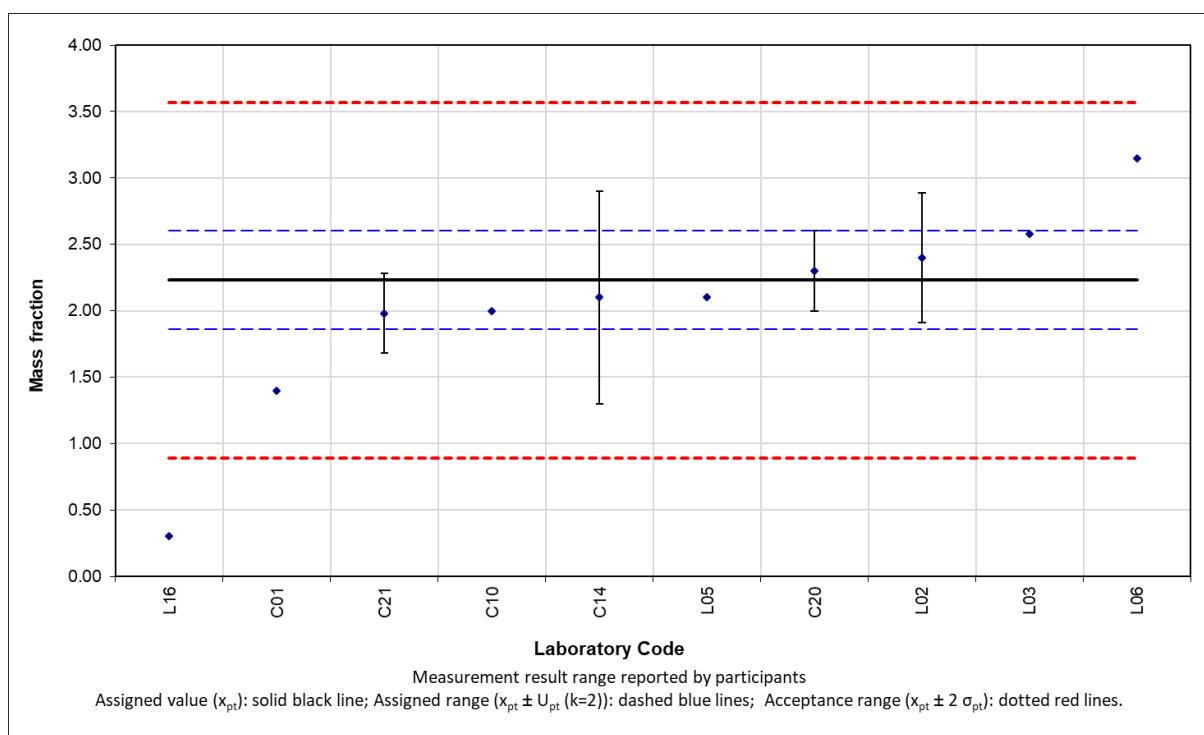


Annex 25: Results for MOSH C10-C16 mass fraction in muesli

$$x_{pt} = 2.23 ; u(x_{pt}) = 0.19 ; \sigma_{pt} = 0.67 \text{ (all values in mg kg}^{-1}\text{)}$$

Lab Code	x_i	\pm	k	Technique	z score
C01	1.4			On-line LC-GC-FID	-1.24
C10	< 2			On-line LC-GC-FID	
C14	2.1	0.8	2	On-line LC-GC-FID	-0.19
C20	2.3	0.3	2	On-line LC-GC-FID	0.10
C21	1.98	0.3	2	On-line LC-GC-FID	-0.37
L02	2.4	0.49	2	On-line LC-GC-FID	0.25
L03	2.58			On-line LC-GC-FID	0.52
L05	2.1			Off-line GC-FID	-0.19
L06	3.15			On-line LC-GC-FID	1.38
L16	0.3			Off-line GC-FID	-2.88

Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)

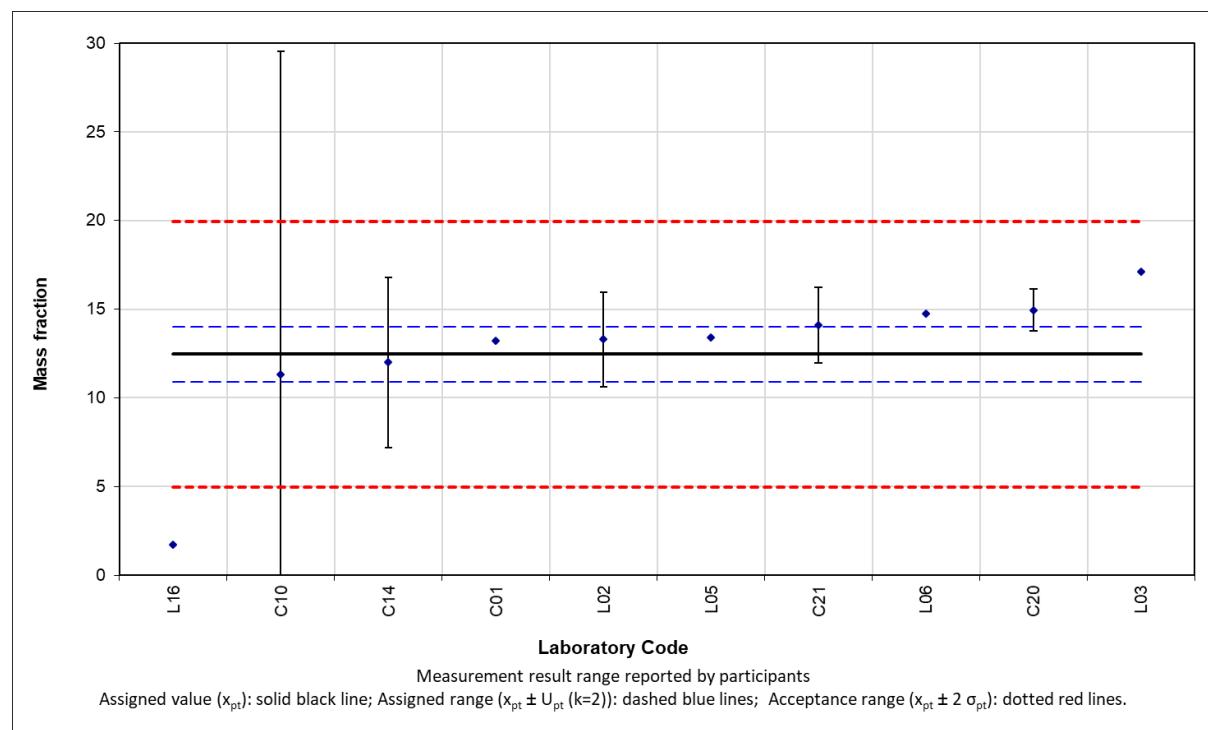


Annex 26: Results for MOSH C16-C20 mass fraction in muesli

$$x_{pt} = 12.46 ; u(x_{pt}) = 0.77 ; \sigma_{pt} = 3.74 \text{ (all values in mg kg}^{-1}\text{)}$$

Lab Code	x_i	\pm	k	Technique	z score
C01	13.2			On-line LC-GC-FID	0.20
C10	11.33	18.21	2	On-line LC-GC-FID	-0.30
C14	12	4.8	2	On-line LC-GC-FID	-0.12
C20	14.95	1.2	2	On-line LC-GC-FID	0.67
C21	14.1	2.12	2	On-line LC-GC-FID	0.44
L02	13.3	2.66	2	On-line LC-GC-FID	0.22
L03	17.11			On-line LC-GC-FID	1.24
L05	13.4			Off-line GC-FID	0.25
L06	14.74			On-line LC-GC-FID	0.61
L16	1.7			Off-line GC-FID	-2.88

Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)

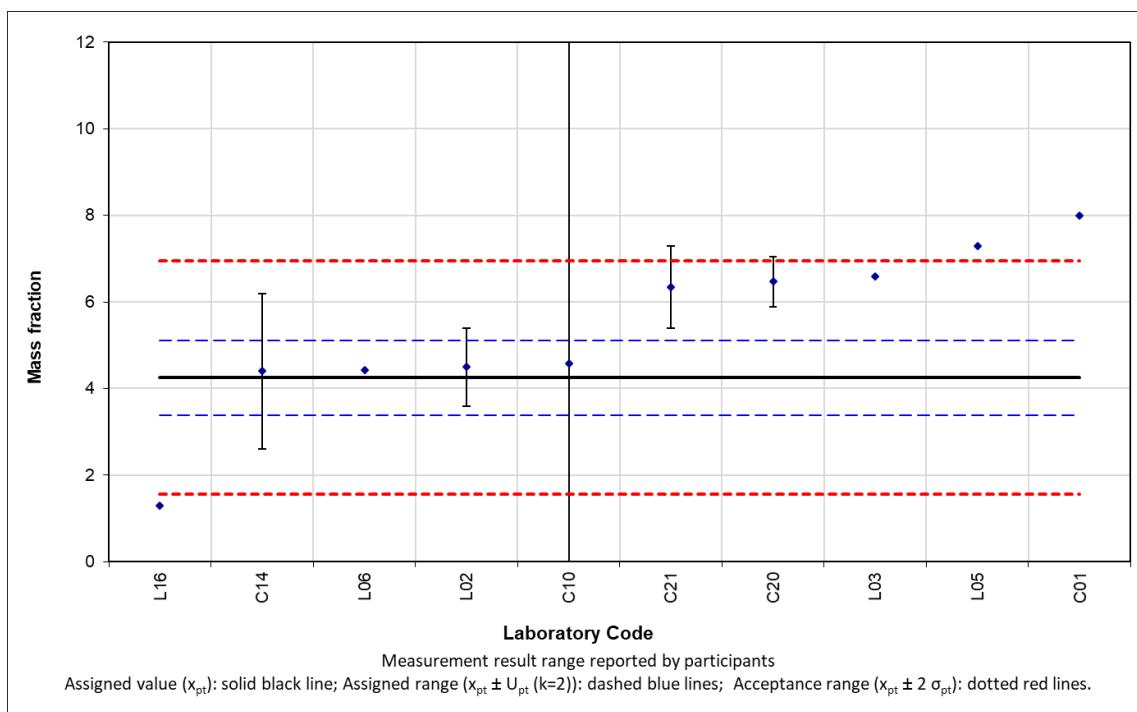


Annex 27: Results for MOSH C20-C25 mass fraction in muesli

$x_{pt} = 4.25$; $u(x_{pt}) = 0.43$; $\sigma_{pt} = 1.28$ (all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique	z' score
C01	8			On-line LC-GC-FID	2.79
C10	4.58	18.21	2	On-line LC-GC-FID	0.25
C14	4.4	1.8	2	On-line LC-GC-FID	0.11
C20	6.47	0.58	2	On-line LC-GC-FID	1.65
C21	6.34	0.95	2	On-line LC-GC-FID	1.55
L02	4.5	0.9	2	On-line LC-GC-FID	0.19
L03	6.59			On-line LC-GC-FID	1.74
L05	7.3			Off-line GC-FID	2.27
L06	4.42			On-line LC-GC-FID	0.13
L16	1.3			Off-line GC-FID	-2.19

Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)

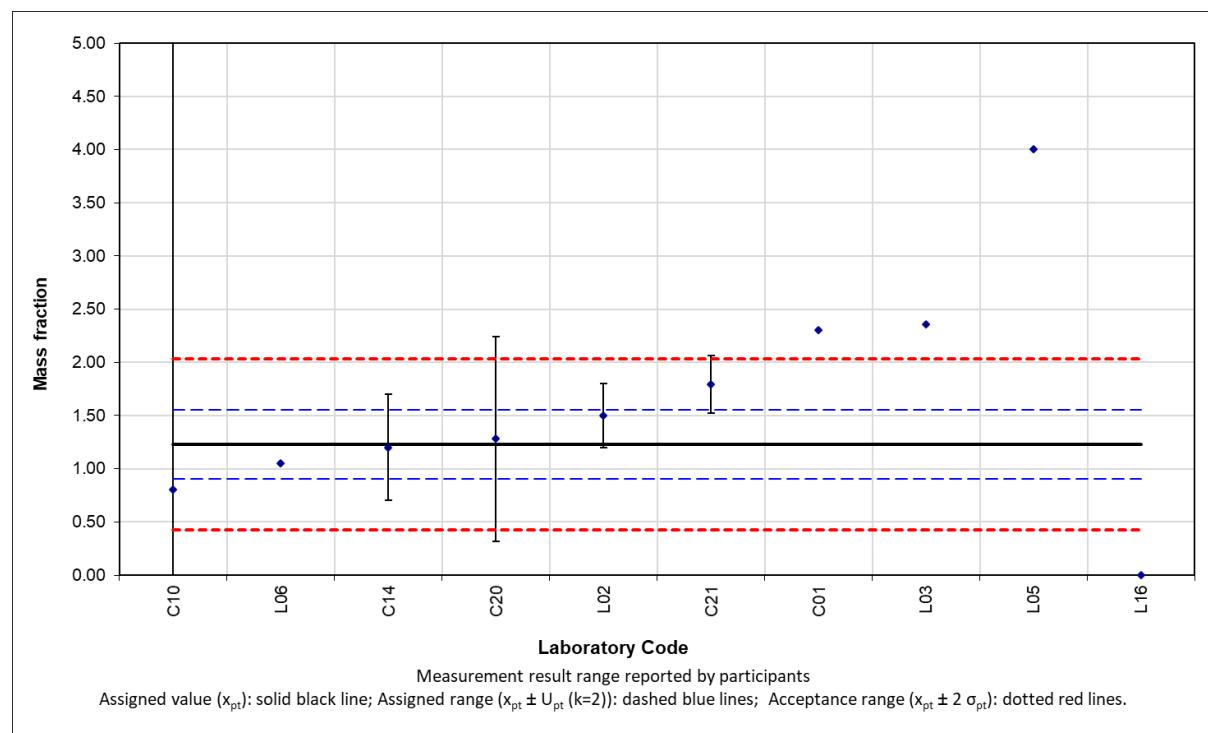


Annex 28: Results for MOSH C25-C35 mass fraction in muesli

$x_{pt} = 1.23$; $u(x_{pt}) = 0.16$; $\sigma_{pt} = 0.37$ (all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique	z' score
C01	2.3			On-line LC-GC-FID	2.67
C10	0.8	18.21	2	On-line LC-GC-FID	-1.06
C14	1.2	0.5	2	On-line LC-GC-FID	-0.06
C20	1.28	0.96	2	On-line LC-GC-FID	0.13
C21	1.79	0.27	2	On-line LC-GC-FID	1.40
L02	1.5	0.3	2	On-line LC-GC-FID	0.68
L03	2.36			On-line LC-GC-FID	2.82
L05	4			Off-line GC-FID	6.90
L06	1.05			On-line LC-GC-FID	-0.44
L16	1.8			Off-line GC-FID	1.43

Performance: Satisfactory (green); Questionable (yellow); Unsatisfactory (red)



Annex 29: Results for MOSH C35-C40 mass fraction in muesli(all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique
C01	< 1			On-line LC-GC-FID
C10	< 0.1			On-line LC-GC-FID
C14	< 0.5			On-line LC-GC-FID
C20	< 0.5			On-line LC-GC-FID
C21	< 0.5			On-line LC-GC-FID
L02	0.27	0.053	2	On-line LC-GC-FID
L03	0			On-line LC-GC-FID
L05	0.34			Off-line GC-FID
L06	< 0.5			On-line LC-GC-FID
L16	0.6			Off-line GC-FID

Annex 30: Results for MOSH C40-C50 mass fraction in muesli(all values in mg kg⁻¹)

Lab Code	x_i	\pm	k	Technique
C01	< 1			On-line LC-GC-FID
C10	< 0.1			On-line LC-GC-FID
C14	< 0.5			On-line LC-GC-FID
C20	0			On-line LC-GC-FID
C21	< 0.5			On-line LC-GC-FID
L02	0.26	0.051	2	On-line LC-GC-FID
L03	0			On-line LC-GC-FID
L05	0.16			Off-line GC-FID
L06	< 0.5			On-line LC-GC-FID
L16				

Annex 31: Results of the questionnaire

Lab No	Years of experience with MOSH/MOAH analysis	Number of samples analysed for MOSH/MOAH in 2019	Your experience depending on the type of matrices					Status of your method for MOSH/MOAH in paperboard	Status of your method for MOSH/MOAH in muesli	Sample intake	Extraction solvent and volume	time/Temperatur during the extraction	epoxidation for elimination of interference used?
			Dry, low fat content sample (< 4% oils/fat) :Rank (0-3) according to the legend above	Higher fat/oil content sample (> 4 % oils/fat): Rank (0-3) according to the legend above	Oils & Fats:Rank (0-3) according to the legend above	Paperboard:Rank (0-3) according to the legend above	Infant Formula: Rank (0-3) according to the legend above						
L02	more than 5 years	100 to 500	3	3	3	3	3	method accredited	method accredited	1	ethanol/water 1+1, 10 ml	2 hours, room temperature	No
L03	1 - 2 years	less than 10	1	1	1	1	1	method under development	method under development	1	1:1 Hexane: Ethanol	2 hours	No
L04	1 - 2 years	10 to 100	0	0	0	10-100	0	method accredited	method under development	1	10 mL hexane:ethanol 1:1	2 hours, ambient	No
L05	3 - 5 years	100 to 500	3	2	2	3	1	method under development	method under development	2	Isohexane (CAS 107-83-5) + Ethanol (50/50), 10mL	2h room temperature	No
L06	more than 5 years	100 to 500	3	2	2	3	2	method accredited	method validated	1	10 mL		
L17	less than 1 year	less than 10	1	0	0	1	1	method under development	n-Hexane/ Ethanol v:v 1:1	2 h / Room temp.	No		
C10	3 - 5 years	100 to 500	2	2	3	3	3	method accredited	method under development	1	Hexane/ethanol (50:50) 10 mL	2 hours / ambient T	No
C14	3 - 5 years	100 to 500	2	2	2	2	1	method accredited	method accredited	1	n-Hexan-Ethanol 1/1v/v 20 ml	2h 22°C	No
C20	more than 5 years	100 to 500	3	2	3	2	2	method accredited	method accredited	3	Ethanol / hexane (1:1 v/v) 30 ml	2 h at room temperature	No
C21	more than 5 years	more than 500	3	2	2	3	3	method accredited	method accredited	1	5 mL Ethanol + 5 mL Hexane	2h room temperature	No

Lab No	What type of epoxidation did you apply?	Please describe shortly the epoxidation procedure (mCPBA volume, concentration, t/T, stop reagent)	Did you apply ALOX clean-up for determination of MOSH to eliminate the interferences from the n-alkanes?	Please describe shortly the ALOX procedure	Did you apply additional column clean-up?	Please describe shortly the column clean-up procedure (type and amount of adsorbent/ eluents, volumes)	Sample intake	Solvent and volume used for the extraction in ml	time/Temperature during the extraction	Did you apply saponification ?	Please describe shortly the saponification step (KOH concentration and volume, t/T)
L02	in ethanol		No		No		10	hexane, 10 ml	>18 hours, room temperature	No	
L03	in ethanol		No		No		5	Hexane	overnight at room temperature	No	
L04	in ethanol		No		No	we used LC-GC-FID	0	-	-	No	
L05	in ethanol		No		No		10	Isohexane (CAS 107-83-5), 10mL	12h (overnight) at room temperature	No	
L06	in ethanol		No		No		5			No	
L17	in ethanol		No		Yes, before epoxidation		0	0	0	No	
C10	in ethanol	no epoxidation used	No		No		5			Yes, after the extraction	10 mL extract + 3 mL KOH 50% aqua.+ 5 mL ethanol shake for 30 min at 60°C
C14	in ethanol		No		No		5	Ethanol / Hexane (1:1 v/v) 30 ml	30 min at 60°C	Yes, after the extraction	To 10 mL extract adding 3 mL of 50% KOH in water
C20	in ethanol	[Please cancel the answer of B5, because we did not apply a epoxidation! see B4: NO]	No		No		10	30 mL EtOH/n-Hexane (1:1 v/v)	30 min / 60°C	Yes, after the extraction	KOH 50% (w in H ₂ O)V = 3 mL Saponification 45 min / 60°C
C21	in ethanol		No		No		4	10 mL hexane/ethanol (1:1) for saponification, further 15 mL hexane for phase separation	60 min at 60°C	Yes, after the extraction	4 mL of KOH solution (50 g KOH/100 mL water) added to sample (in hexane/ethanol), shaking for 60 minutes at 60 °C, cool down to room temperature. extraction: addition of 10 mL water, 10 mL hexane and 2 mL ethanol, extract organic phase, second extraction of aqueous phase with 5 mL hexane washing of organic phase with two times 5 mL of ethanol/water

Lab No	Did you apply epoxidation?	What type of epoxidation did you apply?	Please describe shortly the epoxidation procedure (mCPBA volume, concentration, t/T, stop reagent)	Did you apply ALOX for determination of MOSH?	Please describe shortly the ALOX procedure	Did you apply additional column clean-up?	Please describe shortly the column clean-up procedure (type and amount of adsorbent/eluents, volumes)
L02	Yes, for determination of both MOSH and MOAH	in ethanol	1 mL sample extract, 5x concentrated (hexane)add 0.5 mL m-CPBA solution (20 % in ethanol, freshly prepared, purified)reaction: 15 minutes at 40 °C add 0.5 mL EtOHadd 2 mL sodium thiosulphate (10 % in water) shake well and remove aqueous phaseadd 2 mL sodium carbonate (10 % in water) shake well	No		No	
L03	No			No		No	
L04	No			No		No	
L05	No			No		No	
L06	Yes, for determination of MOAH	in ethanol	1 mL extract + 0,5 mL mCPBA-solution in EtOH(0,2 g/ml) → vortex and shake 15 min at 40°C in waterbad. Add 2 mL Na2S2O3 solution in water (100 mg/ml) → vortex 15 sec. discard lower phase and wash with 2 mL Na2CO3 solution in water (100 mg/ml) by vortex 15 sec	Yes	ALOX-column: glascolumn (Ø 21 mm) field with 10 g Al2O3 (90 active basic 0,063-0,200 mm; activated at 500 °C, 16 h) + 3 g silica (60; 0,063-0,200 mm; activated at 380 °C, 48 h + 1 g Na2SO4) Rinse the column with 20 mL hexane → add 1 mL Extract and eluate with 25 hexane. Add 2 drops Bis(2-ethylhexyl)maleate and reduce until 1 mL with rotorvapor.	No	
L17	No			No		No	
C10	Yes, for determination of MOAH	in ethanol	10 mL saponified extract + 1 mL 20 % CPBA in ethanol shake for 15 min at 40°C, stop reagent: 2 mL (5 g Na2S2O3 + 5 g Na2CO3 /100 ml)	Yes	with 20 mL n-Hexan prewashed Alox-column (10 g AlO2 activated basic +3 g Silicagel+1 g Na2SO4)) 10 mL saponified extract on column, eluated with 20 mL n-hexane concentrate to 1 mL after adding bis-(2ethylhexyl)maleate as keeper	Yes, after epoxidation	3 g silicagel +1 g Na2SO4 column ; epoxidated extract prox. 10 mL on column; elute with 20 mL n-hexane /DCM (7/3)
C14	Yes, for determination of both MOSH and MOAH	in ethanol	to 10 mL extract adding 1 mL mcPBA in ethanol (0,2 g/L) for 15 min at 40°C with shaking. Stopping with adding 8 mL of a mixture of Na2S2O3 / Na2CO3 (150 mg/ml).	No		Yes, after epoxidation	6g silica gel for clean-up column, adding the extract to the column, washing with 1 ml, 1 ml, 2x2mL and 10 mL DCM/Hexane-Solution (30% DCM in hexane)
C20	Yes, for determination of MOAH	in ethanol	V (mCPBA) = 1 ml c = 100 mg/ml stop reagent: Sodiumthiosulfate/Sodiumcarbonate 1:1 in H2O V = 2 ml c = 50 g/l	Yes	column: glaswool + 10 g AlOx + 3 g silica gel conditioning: 20 mL n-Hexane sample: about 10 mL n Hexane elution: 25 mL n-Hexane	Yes, before epoxidation	column: glaswool + 3 g silica gel + Na2SO4 + glaswool conditioning: 10 mL n-Hexane sample: about 10 mL n-Hexane Elution: 14 mL n-Hexane/DCM (70:30 v/v)
C21	Yes, for determination of MOAH	in ethanol	sample concentrated to 1 mL addition of 1 mL mCPBA (0.1 g/mL) in ethanol shaking for 20 minutes at 40 °C addition of 2 mL of stop reagent: sodium thiosulfate/sodium carbonate (0.05 g/mL each) and 0.5 mL ethanol reduce organic phase to 0.3 mL	Yes	column: 10 g aluminum oxide 60, 3 g silica gel 60, 1 g sodium sulfate (all treated at 400 °C before use) rinsing of column with 20 mL hexane eluate sample with 25 mL hexane addition of keeper reduce to 1 mL	Yes, before epoxidation	column: 3 g silica gel 60, 1 g sodium sulfate (all treated at 400 °C before use) rinsing of column with 15 mL hexane/dichloromethane (7:3 v/v) eluate sample with 15 mL hexane/dichloromethane (7:3 v/v)

Lab No	Please specify "other"	Set-up used	Did you follow the JRC SOP provided during the hands-on training ? If not, please briefly describe	HPLC Column used	How do you control the start/end of MOSH fraction?	How you control the start/end of the MOAH fraction?	Injection system used	if "other", specify
L02		on-line single channel		2 x 250mm Lichrospher Si 60A 5um	start: long chain n-alkanes end: cycy	start: DEHB end: per	direct coupling with HPLC	
L03	extract centrifuged at 8000rpm and at 8 deg C to clarify	on-line single channel		Restek Allure Silica 5 um, 250 mm x 2.1 mm Part number 9160572	Cholestane	Perylene	direct coupling with HPLC	
L04		on-line double channel		Phenomenex Luna Silica 100A 250x2mm, 5 um	Inject 100 µL of the diluted reference standard mix solution to check the correct working of the HPLC column and the pre-set time for switching injection on the first and the second GC column.	Inject 100 µL of the diluted reference standard mix solution to check the correct working of the HPLC column and the pre-set time for switching injection on the first and the second GC column.	cold on-column	
L05		off-line	Yes				cold on-	
L06	I have to fill here something to be able to submit the document	on-line double channel		Allure Silica, 250mm x 2.1mm, 5 µm	Start at RT 2 min, Ratio C13 to CyCy≥0.45 (Fraction Length 1.5 min)	Start at RT 4.4 min, Ratio TBB / to 2MN≥0.9 (Fraction Length 1.5 min), LC-Check DEHB, LC check perylene	direct coupling with HPLC	
L17		off-line	yes				cold on-column	
C10	for question C3 : 960 min [16 h] value could not be submitted	on-line double channel		Allure Silica 5 µm 60 A250 mm x 2,1 mm	internal Std, C11 and cholestan	internal Std. TBB / perylen	direct coupling with HPLC	
C14		on-line double channel		Allure Silica 5 µm, 250x 2.1 mm	Via Alkane standard C10 - C50 and Cholestan ISTD	DEHB (in LC-Chromatogram) and TBB/Perylen control in LC/GC	direct coupling with HPLC	
C20		on-line double channel		Restek Allure Silica 5 µm 250 x 2,1 mm (muesli & paperboard)	muesli-method: C11/Cho paperboard-method: Alkane Standard C7-C40	muesli-method: TBB/Perpaperboard-method: Per in the HPLC- chromatogram	direct coupling with HPLC	
C21	We also treated this sample without saponification, alox and epox and obtained very comparable results. The matrix was uncomplicated enough so that it was not necessary. We still provide the samples with saponification, alox and epox because they were carried out three times, the simple procedure was just	on-line double channel		Allure silica 5 um, 250 mm* 2.1 mm i.d. with matching guard cartridge 10mm*2.1mm plus cap frit 2.0 um pore	injection of standard-mix: MOSHslightly visible in UV-spectrum. The standards should be obtained in the GC-chromatogram in the matching ratios. C10-C50 standard mix is used to control the evaporation	injection of standard-mix: standards are visible in the UV-spectrum, the fraction is taken from the beginning of the baseline rise to after the perylene-peak. The standards should be obtained in the GC-chromatogram	direct coupling with HPLC	

Lab No	Type of column and dimentions	Oven temperature program	Pressure program	Did you have problem with
L02	pre-column: 7 m, 0.53 mm i.d., phesil deactivated; separation column: 15 m,0.25 mm i.d., 0.15 um PS- 255	55 °C (4.5 min), 20 °/min 350 °C (8 min)		
L03	Restek MXT Siltek guard column 10 M, 0.53 mm ID.(#573250). Analytical column Restek MXT-1, 15 m,0.25 mm ID, 0.25µm df (#70120)	MOSH 60oC [6 min], @20 oC /min to 350 oC [14.5min] Total run time 35 min.MOAH 60oC [7 min], @20 oC /min to 350 oC [13.5min] Total run time 35min.	hydrogen, 0.6 bar constant pressure	the solvent peak
L04	Pre-column: MXT Siltek Guard Column, uncoated, L= 10 m, I.D. = 0.53 mm Column: Rxi-1HT, L = 15 m, I.D. = 0.25 mm, df = 0.10 µm	: 55 °C (8') – 30 °C·min-1 – 370 °C (16.5')	MOSH- Initial 70 kPa [4min] then to 60kPa @400kPa/min, 60kPa [2], then to 85kPa @1.72kPa/min and remains at that for the run. MOAH-Initial 65 kPa [6min] then to 85kPa @1.32kPa/min and remains at that for the run.	interferences
L05	deactivated precolumn (10m x 0.53mm) + TG-1MT (15m x 0.25mm, 0.25µm 100%dimethyl polysiloxane)	80°C, 9.6min180°C, 20°C/min, 0min 280°C, 25°C/min, 0min 360°C, 30°C/min, 8min	Table 4. MOSH Time (min) TimedCarrierCtrl Timed Pressure (kPa) Initial On 60 0.0 On 90 1.99 On 60 8.0 On 80 12.0 On 100 14.0 On 120 16.0 On 150 Table 5. MOAH Time (min) TimedCarrierCtrl Timed Pressure (kPa) Initial On 60 2.2 On 90 3.93 On 60 8.0 On 80 12.0 On 100 14.0 On 120 16.0 On 15	interferences
L06	MXT-1; 0.25mm ID x 15m;0.25 µm, Retention gap: MXT-Hydroguard 10m, 0.53mm ID	Heat Rate [°C/min]Final Temp [°C]Hold Time [min]Run Time [min]Initial 60 8 820 370 6,5 30	2mL/min, 0.01min 8mL/min, 3.2min 0.4mL/min, 2min 2mL/min, 25min	the baseline; the solvent peak
L17	DB-1HT 100 %polydimethylolysiloxane 15 m x 0.25 mm, 0.10 um (a gap of Deactivated Fused Silica, 10 m x 0.530 mm) was installed previous to the column to allow the injection of a large volume, 40 uL)	75 °C, 9 min/ ramp 20°C min up to 300 °C/ramp 30 °C min up to 380 °C (hold 10 min)	This field is unclear to me? Dop you mean data for the evaporation setting at the interface?	the solvent peak
C10	retention gap: mxt-siltek guard column 10 m x 0,53 mm IDanalytical column : mxt-1 15 m x 0,25 mm ID 0,25 µm df	60°C 8 min 20°/min -> 350°C 6,5 min	Ramp flow: initial: 2.6 mL min (hold 4 min)/ ramp 99 mL min2 up to 5.2 mL min	the solvent peak
C14	MXT-1, 15 m, 0.25 mm ID,0.25 µm df (100% dimethyl polysiloxane) with MXT Siltek Guard Column	Start at 60°C hold for 6 min, with 20°C/min up to 370°C hold for 6,5 min	MOSH/MOAH: 77 kpa (evaporation) 150 kpa analysis pressure	interferences
C20	RESTEK MXT-1; 15 m; 0,25mmID	muesli-method: Temp [°C] / Time [min] / Rate [°C/min]60 / 9 / -120 / - / 15370 / - / 25370 / 7 / -	Chanel A: Start Evaporation Pressure 78 kPA, Chanel B: 68 kPA, Analyse Pressure both 150 kPA, delay time Chanel A: 3.2 min	the solvent peak
C21	Restek MXT-1 15 m*0.25 mm i.d./ 0.25 um with retention gap Restek MXT Siltec guard column 10 m*0.53 mm i.d.	hold at 60 °C for 8 minutes 15 °C/min to 120 °C25 °C/min to 370 °Chold at 370 °C for 8 minutes	muesli-method: MOSH: 75 kPa / on 1,50 min / off 3,80 min - 30 kPa / off 6,90 min - 170 kPa MOAH: 72 kPa / on 4,00 min / off 6,30 min - 165 kPa paperboard-method: MOSH: evaporation pressure 95 kPa / Analytical pressure: 240 kPa MOAH: evaporation pressure 77 kPa / Analytical pressure: 256 kPa	interferences

Lab No	Please describe in more detail the problem	How do you quantify MOSH/MOAH? Against which internal standard?	Did you remove any riding peaks ?	To demonstrate the control over the procedure for MOAH, please fill in the respective area ratios. Please report the ratio for each of the 3 replicates in the same cell, separated by comma. If you prefer you could upload an excel file below theoretical							Please upload your excel file with the area ratio
				muesli: 5B/1MN	MUS:TBB /1MN	MUS:2M N/1MN	MUS:5B/ 1MN	MUS:TBB /1MN	MUS:2M N/1MN	MUS: 5B/ 1MN	
L02	no problems but: "This field is required"	MOSH: cycy MOAH: MNS	paperboard: MOSH: no; MOAH: yes muesli: MOSH: no up to C20, yes after C20; MOAH: yes	0.90, 0.93, 0.88			0.89		1.00	0.97	
L03	Interferences in the MOAH fraction of the muesli from peripheral peaks.	MOSH against cycy and MOAH against 1NM	Yes	0.90, 0.93, 0.88			0.89		1.00	0.97	0.90, 0.93, 0.88
L04	no problems but the field was required...(-:	MOSH: CyCy MOAH: 1-MN	yes, we remove all riding peaks	0.96, 0.96				0.99	1.00	1.00	0.96, 0.96
L05	Baseline drift due to the variability of the percentage of toluene in the solvent peak	MOSH: Cycy MOAH: 1MN	MOSH: no MOAH: yes								
L06	no problems, but I was not able to go on without choosing at least one topic	MOSH against CyCy, MOAH against 2MN	yes	0.95, 0.95, 0.93	1.03, 1.02, 1.04	1.00, 1.01, 1.01	0.97, 0.96, 0.95	1.04	0.99	1.01	0.95, 0.95, 0.93
L17	MOAH fraction: large front eluting peak	MOSH: CyCy MOAH 2 mN	YES	5B/2MN: 0.99, 1.01, 1.00	TBB/2MN: 1.00	1MN/2M N: 1.40	5B/2MN: 0.93	1	1	1	5B/2MN: 0.99, 1.01, 1.00
C10	no problems	MOSH : cycy MOAH : TBB	yes					100	100	100	
C14	I could not submit without clicking any of the fields in E.6 and a no problem answer is missing. So I click the solvent peak, because our GC column had a very poor tailing, but in my opinion not so bad, that I have to do something against it. It has no effect on quantification.	MOSH against Cycy, MOAH in paperboard against 2MN, MOAH in muesli against TBB (because of the saponification / epoxidation step)	We remove riding peaks in every MOAH of every sample and also in MOSH of food samples. We do not remove in MOSH of paper / plastics / mineral oil samples, where the origin of mineral oil is clear.	0.562, 0.548	0.553	0.570	0.577				0.562, 0.548
C20	Interferences in the MOAH fraction of the muesli-method: The chromatogram shows in the area of C35-C40 two little humps. One of these two humps has a big peak on it. Unfortunately it was impossible to identify these substances clearly, therefore we have no idea, if it is a contamination or something else. Usually the peak will be eliminated by the epoxidation step - but this was not successful enough in this case.	muesli-method: MOSH: CyCy / MOAH: 2-MN paperboard-method: MOSH: Cho / MOAH: Per Restek MOSH/MOAH Standard (31070), 150-600 µg/ml, toluene, 1ml/ampulle	muesli-method: yes paperboard-method: no								
C21	we did not have problems, but there was no such option provided	MOSH: Bicyclohexyl MOAH: 1-Methylnaphthaline MOAH after epoxidation: TBB	In order to obtain the hump area, the integration was carried out so that all riding peaks were subtracted from the total area.								

Lab No	LOQ for total MOAH is set as:	Please describe "other"	Any other comment from your side
L02	sum(LOQ) of the various fractions	ddd	
L03	other	Taken from literature	
L04	other	-	I didn't upload chromatograms and area ratio's because at the moment I work from home and I don't have access to this data. If you really need this
L05	sum(LOQ) of the various fractions	/	
L06	other	LOQ for total MOAH was determined by spiking experiments of MOAH containing oil at low level to matrix	Data for GC-pressure an Excel-File for the IS ratios are missing. They can be send later by mail if necessary.
L17	other	Not yet estimated	We had problems for quantitation of .C10-C16 MOAH fraction due to the large solvent peak (manual method) Blank values have been subtracted
C10	max(LOQ) of the various fractions	n.a.?	no
C14	max(LOQ) of the various fractions	I write here, because I could not submit otherwise.	For paperboard we used 2x 3g and 2x 1g as sample weight. To B.7: we must give an answer, for paperboard we did not use epoxidation.
C20	max(LOQ) of the various fractions	-	
C21	max(LOQ) of the various fractions	no further comments	

GETTING IN TOUCH WITH THE EU

In person

All over the European Union there are hundreds of Europe Direct information centres. You can find the address of the centre nearest you at: https://europa.eu/european-union/contact_en

On the phone or by email

Europe Direct is a service that answers your questions about the European Union. You can contact this service:

- by freephone: 00 800 6 7 8 9 10 11 (certain operators may charge for these calls),
- at the following standard number: +32 22999696, or
- by electronic mail via: https://europa.eu/european-union/contact_en

FINDING INFORMATION ABOUT THE EU

Online

Information about the European Union in all the official languages of the EU is available on the Europa website at: https://europa.eu/european-union/index_en

EU publications

You can download or order free and priced EU publications from EU Bookshop at:

<https://publications.europa.eu/en/publications>. Multiple copies of free publications may be obtained by contacting Europe Direct or your local information centre (see https://europa.eu/european-union/contact_en).

**The European Commission's
science and knowledge service**
Joint Research Centre

JRC Mission

As the science and knowledge service of the European Commission, the Joint Research Centre's mission is to support EU policies with independent evidence throughout the whole policy cycle.



EU Science Hub
ec.europa.eu/jrc



@EU_ScienceHub



EU Science Hub - Joint Research Centre



EU Science, Research and Innovation



EU Science Hub



Publications Office
of the European Union

doi:10.2760/05496

ISBN 978-92-76-40589-4