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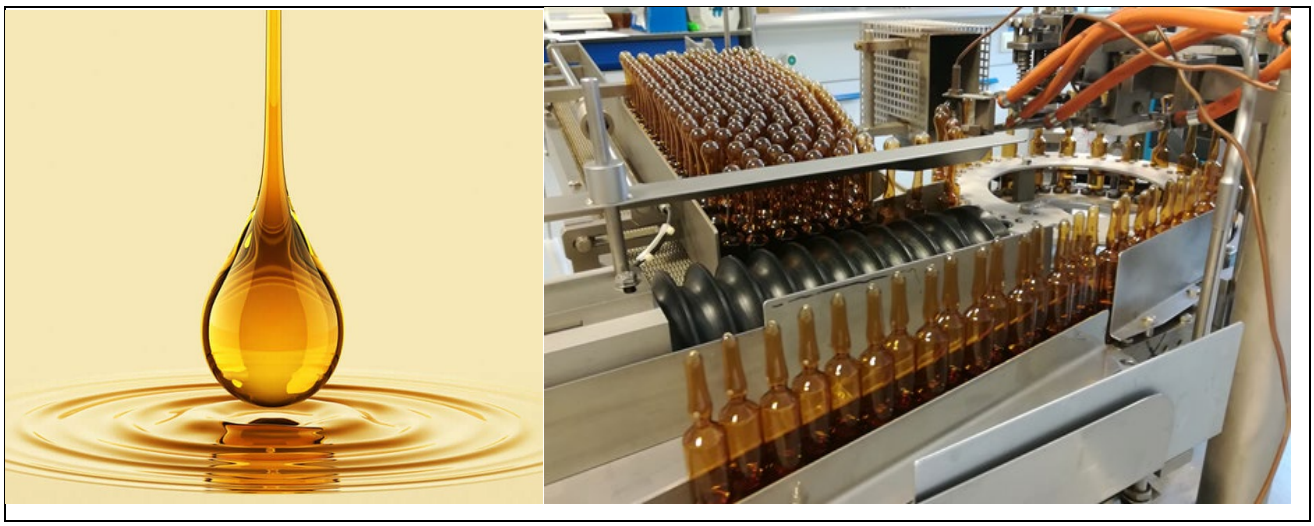
# Determination of MOSH and MOAH in edible oil

*Proficiency Test Report*
*JRC FCM-22/01*

Stefanka Bratinova, Piotr Robouch,
Fernando Cordeiro, Giorgia Beldi, Chiara Senaldi,
Lubomir Karasek and Eddo Hoekstra

2023



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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

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## Executive summary

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM) has organised a proficiency test (FCM-22/01) for the determination of mineral oil saturated hydrocarbons and mineral oil aromatic hydrocarbons (MOSH and MOAH) in edible oil to support the Commission Recommendation (EU) 84/2017. Upon request from DG SANTE, this proficiency test was open to all interested stakeholders from the industry, universities and commercial laboratories, in addition to the National Reference Laboratories (NRLs) and Official Control Laboratories (OCLs).

Test item A and test item B consisted of two edible oils spiked with MOSH/MOAH, while test item C consisted of a spiked hexane solution. The homogeneity and stability of the three test items were evaluated by the EURL and the assigned values were derived from the results from the measurements performed by four expert laboratories.

While 49 laboratories registered to this exercise, only 37 laboratories reported results, and 33 filled in the questionnaire. All results were rated using *z, z' and/or* zeta ($\zeta$) 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 total MOSH or MOAH mass fractions, based on the perception of experts.

Most of the participating laboratories performed satisfactorily (according to the z or z' score) for the determination of the total MOSH/MOAH fractions in edible oil and hexane.

## List of abbreviations and symbols

| | |
|---|---|
| DG SANTE | Directorate General for Health and Food Safety |
| EURL | European Union Reference Laboratory |
| FCM | Food Contact Materials |
| HPLC-FLD | Liquid chromatography coupled with fluorescence detection |
| 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 |
| $k$ | coverage factor |
| $\sigma_{pt}$ | 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 |
| $\zeta$ | zeta score |

# 1 Introduction

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM), hosted by the Joint Research Centre (JRC) of the European Commission, organised a proficiency test (PT) for the determination of the mass fractions of mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) in edible oil, 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) of the European Commission as part of the EURL-FCM annual work programme 2022, thus complying with the mandate set in Regulation (EU) 2017/625 [2]. Upon request from DG SANTE, this PT round was open to all interested stakeholders from the industry, universities and commercial laboratories in addition to the National Reference Laboratories (NRLs) and Official Control Laboratories (OCLs).

This report summarises the outcome of the PT.

# 2 Scope

The present PT aims to assess the performance of the participants in the determination of the mass fractions of MOSH and MOAH in edible oil and in hexane.

This PT, organised in line with ISO 17043:2010 [3], is identified as "FCM-22/01".

# 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-22/01 exercise was announced by e-mail to NRLs and OCLs on May 17, 2022 (Annex 1). The registration deadline was set to June 13, 2022. The samples were dispatched to participants on September 5, 2022. The deadline for reporting of results was set to October 24, 2022. This deadline was further extended until November 4, 2022.

## 3.3 Distribution

Each participant received a parcel containing:

- Two ampoules of test item A containing 5 g of olive oil spiked with mineral oil A;
- Two ampoules of test item B containing 5 g of olive oil spiked with mineral oil B;
- One ampoule of solution C containing 5 ml of mineral oil A in hexane; and
- The "Test item accompanying letter" (Annex 2).

Samples were sent under normal transport conditions at ambient temperature.

In addition, the participants received by e-mail:

- The "instructions to participants" (Annex 3); and
- The "Confirmation of receipt form" (Annex 4).

### 3.4 Instructions to participants

Detailed instructions were provided by email to participants in the "instructions to participants' letter" (Annex 3).

The measurands were defined as:

- mass fraction of total MOSH (nC10-nC50) for test items A and B (expressed in mg/kg);
- mass fraction of total MOAH (nC10-nC50) for test items A and B (expressed in mg/kg);
- concentration of total MOSH (nC10-nC50) in hexane for test items C (expressed in mg/L);
- concentration of total MOAH (nC10-nC50) in hexane for test items C (expressed in mg/L).

Participants were asked (i) to check whether the bottles and vial were undamaged after transport, and (ii) to return the "Confirmation of receipt form" (Annex 4) within 3 days after receipt of the parcel.

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 3 g edible oil, the recommended minimum sample intakes were set to 3 g for test item A and B.

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 5).

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

## 4 Test items

### 4.1 Preparation

The two olive oil samples were spiked with different types of mineral oils. Test item A was spiked with 81.9 mg "Shell Gravex 912" and 83.1 mg "Total engine oil" per kg olive oil. Test item B was spiked with l66.3 mg lubricating oil "Elkalub" (supposed to contain no MOAH) per kg olive oil. However the blank olive oil contained traces of MOSH and MOAH that could not be neglected. Test item C was prepared by simply dissolving the "Shell Gravex 912" and "Total engine oil" in hexane to obtain a final solution of 1000 mg/L mineral oil in hexane.

Five g of each material were ampouled in 10 ml brown glass ampoules at the JRC Reference Material Processing facility. Each vial was identified with a unique number and the PT identifier.

### 4.2 Homogeneity and stability

The measurements and the statistical treatment of data for the homogeneity and short-term stability studies for the test items were executed 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, seven vials were randomly selected and analysed in duplicate. 3 g oil were taken as aliquots for the analysis. Results were evaluated

according to ISO 13528:2015 [5]. Both items proved to be adequately homogeneous for the investigated analytes (Annex 6.1).

Short-term stability studies were performed by the JRC at three different temperatures, namely 4 °C, RT (20 °C) and 40 °C, for a period of 3 weeks in order to mimic the transport conditions. No significant trends were observed for MOSH or MOAH fractions, hence the test items were dispatched at room temperature. Similarly, the long-term stability study performed by the EURL-FCM confirmed the adequate stability of the test items at room temperature over the whole period of the PT (Annex 6.2). Hence, the uncertainty contribution due to stability was set to zero ($u_{st}$ = 0) for all investigated analytes.

# 5 Assigned values and corresponding uncertainties

## 5.1 Assigned values

Assigned values ($x_{pt}$) were determined by four expert laboratories selected by the EURL:

- KLZH - Official Food Control Authority of the Canton of Zurich, Switzerland
- BFR - Bundesinstitut für Risikobewertung, Germany
- CVUA-MEL - Chemisches und Veterinäruntersuchungsamt Münsterland-Emscher-Lippe, Germany
- CVUA Stuttgart - Chemisches und Veterinäruntersuchungsamt Stuttgart, Germany

The statistical treatment of the reported results was performed by the EURL-FCM to derive the assigned values presented in Table 1.

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

| Test item<br>Min.Oil<br>Internal.Std | A<br>MOSH | A<br>MOAH<br>MN | A<br>MOAH<br>TBB | B<br>MOSH | B<br>MOAH<br>MN | B<br>MOAH<br>TBB | C<br>MOSH | C<br>MOAH<br>MN | C<br>MOAH<br>TBB |
|---|---|---|---|---|---|---|---|---|---|
| | mg/kg | mg/kg | mg/kg | mg/kg | mg/kg | mg/kg | **mg/L** | **mg/L** | **mg/L** |
| **$x_{pt}$** | **118.6** | **43.5** | **37.55** | **68.4** | **2.77** | **2.35** | **679.7** | **248.3** | **249** |
| $u_{char}$ | 2.7 | 1.04 | 0.48 | 4.3 | 0.25 | 0.16 | 36 | 7.5 | 11 |
| $u_{hom}$ | 1.3 | 0.45 | 0.39 | 0.75 | 0.04 | 0.034 | 8.4 | 3.0 | 3.0 |
| $u_{stab}$ | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| **$u(x_{pt})$,** k=1 | **3.0** | **1.1** | **0.62** | **4.4** | **0.25** | **0.17** | **37** | **8.0** | **11** |
| $\sigma_{pt}$ | 23.7 | 10.9 | 9.39 | 13.7 | 0.83 | 0.70 | 102 | 37 | 37 |
| $\sigma_{pt}$,% | 20% | 25% | 25% | 20% | 30% | 30% | 15% | 15% | 15% |
| $u(x_{pt})/\sigma_{pt}$ | 0.13 | 0.10 | 0.07 | **0.32** | 0.2998 | 0.23 | **0.36** | 0.22 | 0.2997 |
| scoring | z , ζ | z , ζ | z , ζ | z' , ζ | z , ζ | z , ζ | z' , ζ | z , ζ | z , ζ |

## 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]:

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

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

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

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, $\sigma_{pt}$

The relative standard deviations for PT assessment ($\sigma_{pt}$) were set, based on expert judgment, to 15, 20 , 25 or 30 % of the respective assigned values for the mass fractions of the total content of MOAH and MOSH in edible oil and hexane (Table 1).

## 6 Evaluation of results

### 6.1 Scores and evaluation criteria

The individual laboratory performance was expressed in terms of *z, z'* and $\zeta$ scores, according to ISO 13528:2015 [5].

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

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

Where: $x_i$ 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;
$\sigma_{pt}$ 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:

$$z'_i = \frac{x_i - x_{pt}}{\sqrt{\sigma_{pt}^2 + u^2(x_{pt})}} \qquad \text{Eq. 5}$$

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

| | | |
|---|---|---|
| \|score\| ≤ 2 | satisfactory performance | (green in Annexes 8 - 16) |
| 2 < \|score\| < 3 | questionable performance | (yellow in Annexes 8 - 16) |
| \|score\| ≥ 3 | unsatisfactory performance | (red in Annexes 8 - 16) |

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

The **$\zeta$ score** states 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 $\zeta$ 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 $\zeta$ score

can be caused by an inappropriate estimation of either the concentration or mass fraction, 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 [7].

Uncertainty estimation is not trivial, therefore an additional assessment is provided to the laboratories having reported measurement uncertainty, to indicate how reasonable was their measurement uncertainty estimation.

The relative standard measurement uncertainty was calculated based on the absolute values of the assigned values [$u_{rel}(x_{pt})$ =100*($u(x_{pt})/x_{pt}$)] and of the reported values [$u_{rel}(x_i)=100*(u(x_i)/x_i)$].

The relative standard measurement uncertainty from the laboratory $u_{rel}(x_i)$ is most likely to fall in a range between a minimum and a maximum allowed uncertainty (case "a": $u_{min,rel} \leq u_{rel}(x_i) \leq u_{max,rel}$). $u_{min,rel}$ is set to the standard uncertainties of the assigned values $u_{rel}(x_{pt})$. It is unlikely that a laboratory carrying out the analysis on a routine basis would determine the measurand with a smaller measurement uncertainty than the expert laboratories chosen to establish the assigned value (ISO 13528:2015 §7.6) or, if applicable, by formulation (ISO 13528:2015 §7.3) or than the certified measurement uncertainty associated with a certified reference material property value (ISO 13528:2015 §7.4). $u_{max,rel}$ is set to the standard deviation accepted for the PT assessment, $\sigma_{pt}$ (expressed as a percentage of the assigned value). Consequently, case "a" becomes: $u_{rel}(x_{pt}) \leq u_{rel}(x_i) \leq \sigma_{pt,\%}$.

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

If $u_{rel}(x_i)$ is larger than $\sigma_{pt,\%}$ (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 $\zeta$ score, though the corresponding performance, expressed as a *z* score, may be questionable or unsatisfactory. General observations

## 6.2 General observations

Forty-nine laboratories from 12 countries representing all stakeholders registered to the exercise. 37 laboratories reported results and 33 participants filled in the questionnaire. The majority of the participants were commercial and industrial laboratories as shown on Figure 1.

The low rate of participation from NRLs and OCLs may be due to the fact that the presence of MOSH/MOAH in food and FCMs is not yet regulated, and that these substances are not routinely controlled by the NRLs and OCLs.

Laboratory (L34) reported unrealistic results for MOAH (only) applying the HPLC-FLD method, while L12 reported results only for the two olive oils test items (A and B), but not for the hexane solution (test item C).

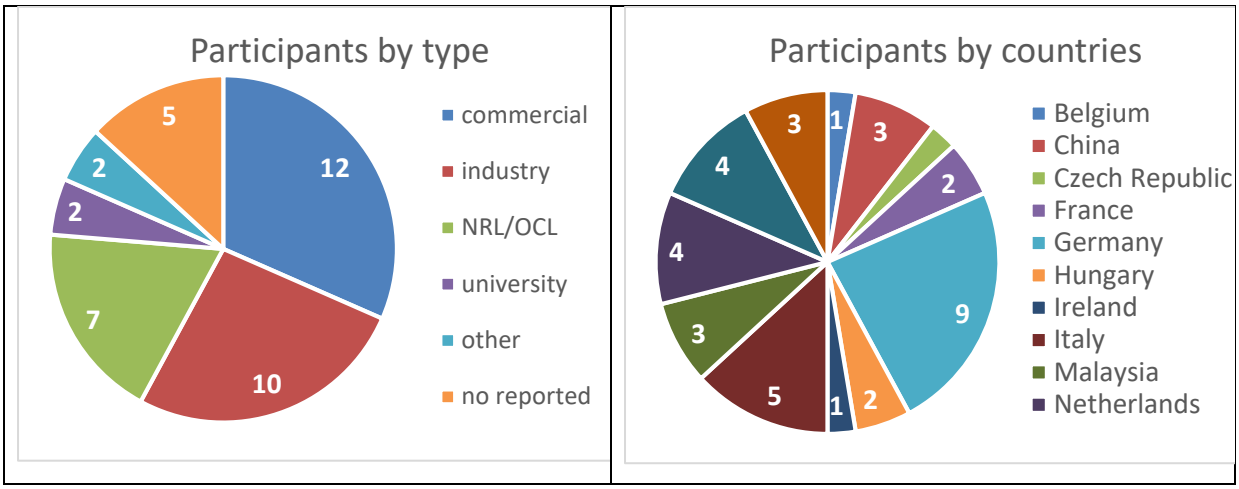


**Figure 1 .** Participating laboratories by country and by type

## 6.3 Laboratory results and scorings

### *6.3.1 Performance*

Annexes 8 to 16 present the reported results as tables and graphs for each measurand.

Table 2 summarises the evaluated performance of the 37 laboratories that submitted results for MOAH and MOSH in edible oil and hexane. Most of the laboratories (above 70 %) reported satisfactory results according to the z or z' score for MOSH in the three test items, and for MOAH in test item A.

Only half of the laboratories reported satisfactory results for MOAH in test item B. This may be attributed to the low MOAH content, close to the maximum tolerable LOQ of 2 mg/kg for MOAH in oils and fats set by the Joint statement of the Member States (dated 21 April 2022) [8]. On the other hand, it was somewhat unexpected that up to eight laboratories would report underestimated mass fractions for MOAH (of ca. 250 mg/L) in the hexane solution (test item C).

**Table 2:** Overview of laboratory performance for the determination of the total mass fraction of MOSH and MOAH in edible oil and hexane. The total number of reported results (N) is compared to the number of Satisfactory (S), Questionable (Q), Unsatisfactory (U) z, z' and ζ scores, together with the truncated "less than" values (LT).

| | | | z score | | | z' score | | | ζ score | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Sample | MO | N | S | Q | U | S | Q | U | S | Q | U | LT |
| A | MOSH | 37 | 35 | 1 | 1 | | | | 20 | 4 | 4 | 0 |
| | MOAH-MN | 38 | 28 | 3 | 5 | | | | 10 | 6 | 11 | 2 |
| | MOAH-TBB | 38 | 31 | 2 | 3 | | | | 15 | 3 | 9 | 2 |
| B | MOSH | 37 | -- | -- | -- | 33 | 1 | 3 | 20 | 2 | 6 | 0 |
| | MOAH-MN | 38 | 23 | 3 | **8** | | | | 7 | 4 | 13 | 4 |
| | MOAH-TBB | 38 | 18 | **8** | **8** | | | | 6 | 7 | 12 | 4 |
| C | MOSH | 36 | -- | -- | -- | 29 | 4 | 3 | 17 | 5 | 7 | 0 |
| | MOAH-MN | 37 | 25 | 3 | **8** | | | | 10 | 2 | 17 | 1 |
| | MOAH-TBB | 37 | 26 | 3 | **7** | | | | 12 | 6 | 10 | 1 |

### *6.3.2 Measurement uncertainty*

Despite the fact that participants were requested to report their results and their associated expanded measurement uncertainty in mg/kg, three laboratories (L14, L35, L48) did not report any uncertainties (flagged as NP), and eight laboratories (L07; L21; L22; L23; L27; L29; L33; L36) may have reported their relative measurement uncertainties expressed in %, which is clearly suggested by the extremely large error bars in the graph of Annex 13. This is further confirmed by the "identical" uncertainty values (±) reported by these laboratories for all the substances (see highlighted values in orange in the tables in Annexes 8 – 16). Therefore, their measurement uncertainties were not taken into account for the calculation of the ζ scores.

### *6.3.3 Additional information extracted from the questionnaire*

The filled in questionnaire was submitted by 33 out of 37 participants having reported results and gave valuable information about their laboratory and their analytical methods. Detailed information is presented in Annex 17.

Half of the participants (19) stated that they are accredited according to ISO/IEC 17025 for the determination of MOSH/MOAH analysis in general, with edible oils and fats included in their analytical scope. Three laboratories are accredited for a whole range of foods while another one is accredited only for paperboard. As for the experience in the field, one of the participants stated to have no experience (0 sample analysed) and five claimed to have analysed less than 10 samples in two years.

***Edible oil test items – A and B***

Fifteen laboratories participated to the ring-trial validation study of the DGF method [9] designed to replace the current standard method EN 16995:2017. They applied a sample preparation protocol including the following steps: (i) saponification; (ii) double extraction in hexane; (iii) silica clean-up before epoxidation; and (iv) epoxidation with m-CPBA for MOAH while aluminium oxide column clean-up for MOSH.

Figures 2 and 3 present the number of laboratories having applied different groups of auxiliary methods during the sample preparation for MOAH and MOSH analysis, respectively

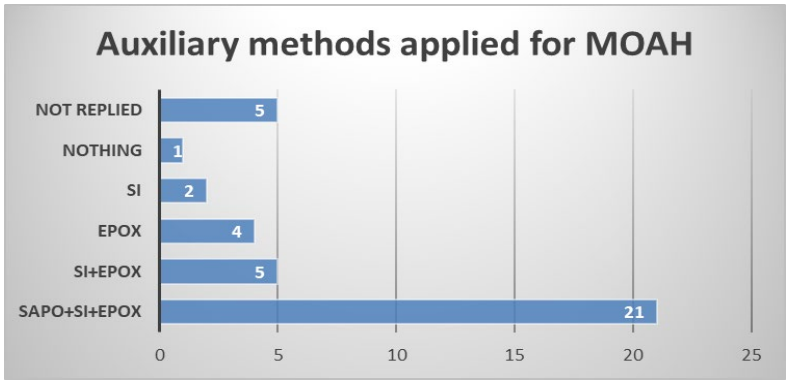


**Figure 2**. Number of laboratories having applied different groups of auxiliary methods during the sample preparation for MOAH analysis
(from the questionnaire)

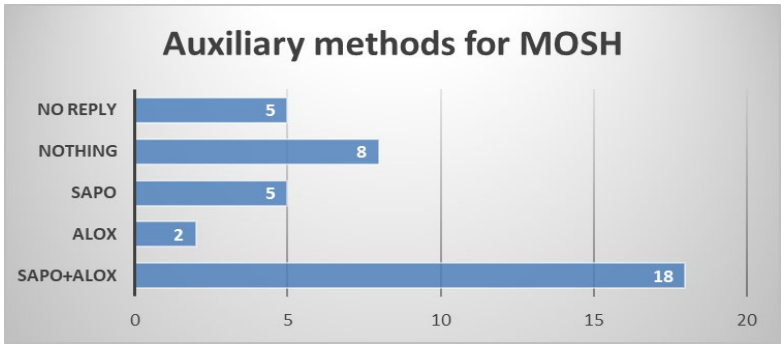


**Figure 3.** Number of laboratories having applied different groups of auxiliary methods during the sample preparation for MOSH analysis
(from the questionnaire)

Despite the different auxiliary methods applied (or not) for sample preparation, no significant difference in results could be identified when analysing high levels of MOSH and MOAH mass fractions (≥ 50 mg/kg) included in the working range of the EN 16995 standard. However, removing the lipids and using higher sample input to the detector are crucial for levels close to the maximum tolerable LOQ of 2 mg MOAH per kg of edible oil.

From the six laboratories (L02, L12, L16, L31, L32, L37) having performed the challenging manual MOSH/MOAH separation, only L12 reported satisfactory and 1 questionable results for all 6 parameters, while L16 and L37 reported unsatisfactory results for MOAH in test item B (low level). The remaining three laboratories (L02, L31 and L32) reported all their results with unsatisfactory scoring.

***Mineral oil solution in hexane – test item C***

Many unsatisfactory z scores were assigned for MOSH/MOAH analysis of the mineral oil solution in hexane. As mentioned before, L34 applied the HPLC-FLD technique for MOAH analysis, while L26 may have reported the results for test item C using wrong units. L02, L16, L31 and most probably L01 applied manual MOSH/MOAH separation, which is challenging even for simple solutions. Similarly, unsatisfactory results were obtained by the participants having applied the on-line method as well. L30, L35, L33 and L40 should check the performance of their on-line system since they obtained very low recovery of the total MO (MOSH+MOAH) from the gravimetrically added content (1000 mg/L). More details on the issue can be found in the JRC report on characterisation of Shell SN500* [10].

# 7 Conclusion

The proficiency test FCM-22/01 was organised to assess the analytical capabilities of different stakeholders to determine the total mass fractions or concentration of MOSH and MOAH in edible oil and a solvent.

The overall performance of the participants was satisfactory (above 70 %).

## Acknowledgements

The 37 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 |
|---|---|
| Primoris | Belgium |
| University of Chemistry and Technology, Prague | Czech Republic |
| ITERG | France |
| Service commun des laboratoires - SCL33 | France |
| Fraunhofer IVV | Germany |
| Bayerisches Landesamt für Gesundheit und Lebensmittelsicherheit | Germany |
| State institut for health and veterinary control saxony | Germany |
| SGS Institut Fresenius GmbH | Germany |
| GBA Gesellschaft für Bioanalytik mbH | Germany |
| Eurofins WEJ Contaminants GmbH | Germany |
| Lebenmittelchemisches Institut des BDSI e.V. | Germany |
| Eurofins Consumer Product Testing GmbH | Germany |
| General Chemical State Laboratory | Greece |
| WESSLING Hungary Kft. | Hungary |
| Dublin Public Analysts Lab. | Ireland |
| CHELAB S.R.L | Italy |
| Carapelli Firenze S.p.A. | Italy |
| Soremartec | Italy |
| Neotron Spa | Italy |
| INNOVHUB-SSI | Italy |
| TLR International Laboratories | Netherlands |
| QTI services B.V. | Netherlands |
| Eurofins Lab Zeeuws-Vlaanderen (CNL027) | Netherlands |
| AGROLAB Dr. Verwey B.V. | Netherlands |
| Centro Nacional De Tecnologia Y Seguridad Alimentaria (CNTA) | Spain |
| Deoleo Global | Spain |
| Instituto de la Grasa -CSIC- | Spain |
| University of Zaragoza | Spain |
| Amt für Verbraucherschutz und Veterinärwesen St.Gallen | Switzerland |
| Nestlé Research | Switzerland |
| Swiss Quality Testing Services | Switzerland |
| Nestle R&D China | China |
| Institute of Analysis and Testing, Beijing Academy of Science and Technology | China |
| Wilmar (Shanghai) Biotechnology R&D Center Co., Ltd | China |
| Unitata Berhad | Malaysia |
| Nisshin Global Research Center Sdn. Bhd. | Malaysia |
| Cargill Palm Product | Malaysia |

# References


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[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,

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[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*". nternational Organisation for Standardization, Geneva, Switzerland.

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# Annex 1: Invitation letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

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

EURL European Union Reference Laboratory for Food Contact Materials

Ispra, 17 May 2022
JRC.F.5 UV/sb/bk/ARES(2022) 22-035

*(sent by e-mail)*

**Subject: Invitation to participate in Proficiency Testing round "FCM-22/01"**

Dear all,

On behalf of the European Union Reference Laboratory for Food Contact Materials (EURL-FCM) managed by the Joint Research Center (JRC) of the European Commission (EC), we invite you to participate in the Proficiency Testing round **FCM-22/01** for the "**Determination of MOSH and MOAH in edible oil**".

Three test items will be dispatched: two olive oils spiked with mineral oils together with a solution of mineral oil in hexane. The PT will be organised under ISO 17043 accreditation from BELAC (Belgian accreditation body). The assigned values for proficiency assessment will be derived by the PT provider from the results provided by the subcontracted expert laboratories, independently from the results reported by the participants.

As announced earlier, your **participation is free of charge**, but we will accept a **maximum of 50 participants.**

If you intend to participate, please register electronically as soon as possible by using the link below and follow the instructions provided.

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=2741

Once you have submitted your registration electronically, you will have to sign it, date it and send it by e-mail to JRC-EURL-FCM@ec.europa.eu.

**The deadline for registration is set to 13th of June 2022.** However, it may be closed earlier, as soon as the maximum number of participants will be reached.

Samples will be dispatched in September (date will be announced later).

The deadline for submission of results will be 6 weeks after the dispatch.

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

Kind regards,

/signed electronically in Ares/
Dr. S. Bratinova
FCM-22/01 PT Coordinator

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

Cc: Dr. U.Vincent (Head of Unit, Food & Feed Compliance, F.5)

Commission européenne/Europese Commissie, Retiesweg 111, 2440 Geel, BELGIQUE/BELGIE
Office: 010 01/057 - Tel. direct line +32 (0)14 571 207

Commissione europea, Via Enrico Fermi 2749, 21027 Ispra (VA), ITALIA - Tel. +39 332 78-9111

Ursula.VINCENT@ec.europa.eu

# Annex 2: Test item accompanying letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate F - Health, Consumers and Reference Materials
**Food and Feed Compliance**

EURL
European Union Reference Laboratory
for Food Contact Materials

Geel, 05 September 2022

**Subject:** **Participation in FCM-22/01 - Determination of MOSH/MOAH in edible oil**

Dear participant,

Thank you for participating in the FCM-22/01 proficiency test (PT) for the **" Determination of MOSH/MOAH in edible oil ".**

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

- test item A: two ampoules containing 5 g each of olive oil spiked with mineral oil A;
- test item B: two ampoules containing 5 g each of olive oil spiked with mineral oil B;
- solution C: one ampoule containing 5 ml of mineral oil A – (mineral oil A content > 0.8 mg/l) in hexane

Upon arrival of this parcel, please check whether the ampoules are undamaged after the transport and promptly inform us if this is not the case. There is no need to send proof of delivery to the EURL-FCM.

The test items should be stored until analysis in a dark place at room temperature (20 °C ± 2 °C) away of any possible contaminations.

Further instructions on this PT round, your individual lab code and passcode for entering the results have been provided by e-mail to the person that register for this round.

Do not hesitate to contact me for all issues related to this PT.

Thank you for your collaboration.

Your sincerely,

e-signed

**Stefanka Bratinova**
PT Coordinator
European Union Reference Laboratory for Food Contact Materials

Cc: Fernando Cordeiro – Deputy PT coordinator
Eddo Hoekstra – Manager EURL-FCM;

# Annex 3: Instructions to participants letter

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate F - Health, Consumers and Reference Materials
**Food and Feed Compliance**

Geel, 19 December 2022
JRC.F.5/SB/xx/ARES(2022) 22-065

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

| | |
|---|---|
| **Reporting website** | https://europa.eu/!jyvjGB |
| EU login | For help, see the Participant's guidelines |
| Password for reporting | **«Part_key»** |
| LabCode | **«LCode»** |
| **Questionnaire** | EUSurvey<br>https://ec.europa.eu/eusurvey |

**Subject: Participation in FCM-22/01 - Determination of MOSH/MOAH in edible oil**

Dear «Title» «Surname»,

Thank you for participating in the FCM-22/01 proficiency test (PT) for the **" Determination of MOSH/MOAH in edible oil ".**

The parcels are dispatch today. Each parcel contains:

- test item A: two ampoules containing 5 g each of olive oil spiked with mineral oil A;
- test item B: two ampoules containing 5 g each of olive oil spiked with mineral oil B;
- solution C: one ampoule containing 5 ml of mineral oil A – (mineral oil A content > 0.8 mg/l) in hexane
- the "Confirmation of receipt" form.

Upon arrival of this parcel, please check whether the ampoules are undamaged after the transport.

The test items A, B and C should be stored until analysis in a dark place at room temperature (20 °C ± 2 °C) away of any possible contaminations.

The measurands are

- **mass fractions (mg $kg^{-1}$) of total MOSH (nC10-nC50) for test items A and B;**
- **mass fractions (mg $kg^{-1}$) of total MOAH (nC10-nC50) for test items A and B;**
- **concentrations of total MOSH (nC10-nC50) in hexane for test items C**
- **concentrations of total MOAH (nC10-nC50) in hexane for test items C.**

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

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

Please report separately for each item, the following:

- for test items A and B - the **mean** of your two measurements results (one per ampule) in mg $kg^{-1}$ for each of the measurands and the associated expanded **uncertainty** (in mg $kg^{-1}$);
- for test items C - the **mean** of your two measurements results (in mg $l^{-1}$) for each of the measurands and the associated expanded **uncertainty** (in mg $l^{-1}$);
- the **coverage factor** for the uncertainties of the test items A, B and C; 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 2.5 g for test item A and B and therefore the recommended minimum sample intakes for test items A and B is 2.5 g.

You can find the MILC reporting website at https://europa.eu/!jyvjGB . You need first to login with your EU login account (see detailed guideline) and then enter the personal password. Your unique password is indicated above in the box under your address data. The system will guide you through the reporting procedure. Do not forget to submit and confirm when required.

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

**The deadline for submission of results is 24/10/2022**. It will not be possible to submit your results after the deadline.

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 PT 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/*

PT Coordinator

# Annex 4: 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, «Date»

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

**Subject: "Confirmation receipt" form -**
**FCM-22/01 - Determination of MOSH/MOAH in edible oil**

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-22/01 Coordinator
e-mail : jrc-eurl-fcm@ec.europa.eu

European Commission, Retieseweg 111 – 2440 Geel, Belgium
Tel: +32 14 57 12 11 • e-mail: jrc-eurl-fcm@ec.europa.eu
https://ec.europa.eu/jrc/en/eurl/food-contact-materials

## Annex 5: Questionnaire

Draft ID: 307cd79b-290b-44d2-8016-5b13e2dfe0c9
Date: 31/01/2023 12:25:02

# EURL-FCM PT 2022/01

Fields marked with * are mandatory.

This questionnaire aims to collect information about your routine method, applied for the analyses of the test samples from the PT FCM22/01

Thank you for your contribution
Stefanka-Petkova.BRATINOVA@ec.europa.eu
ILC-coordinator of the "MOAH in IF"

## Identification

* I. Laboratory code

* II. Laboratory name/Organisation

* III. Contact person

* IV. Please select what is relevant for your laboratory:

- [ ] National Reference Laboratory for process contaminants or FCM (NRL)
- [ ] Official control laboratory (OCL)
- [ ] industry lab
- [ ] commercial lab
- [ ] university lab
- [ ] other

## General Questions

* A. How many edible oils@fats samples did you analyse for MOSH/MOAH during the 2021 and 2022?

1

* B. How many MOSH/MOAH analyses in general did you perform in 2021 and in 2022?

* C. What type of matrices were the majority of the samples?

* D. Are you accredited for MOSH/MOAH analyses?

- ( ) Yes
- ( ) No

* F. Did you participate in the 2022 collaborative trial for MV of the MOSH/MOAH in edible oils and fats

- ( ) Yes
- ( ) No

## Outcome

* 21. Did you dilute test item C before the injection?

- ( ) Yes
- ( ) No

* 22. What was the ratio C50:C20

* 23. Were all verification standards within the limits prescribed in the Guidance document on MOSH/MOAH? Please mention those that deviate?

* 24. Did you perform background compensation ?

- ( ) Yes
- ( ) No

* 25. How did you perform background compensation ? Please describe

2

26. Please report the values (if you hqve quantified it) for the total MOAH (TBB) content in your reagent blank (mg MOAH/kg edible oil) - what you have subtracted from the sample A and sample B

27. Please report the values (if you hqve quantified it) for the total MOAH (2MN) content in your reagent blank (mg MOAH/kg edible oil) - what you have subtracted from the sample A and sample B

28. Please report the values (if you hqve quantified it) for the total MOSH content in your reagent blank (mg MOSH/kg edible oil) - what you have subtracted from the sample A and sample B

* 29. Did you encounter any problems during the sample preparation, please describe?

* 30. Did you encounter problems during the integration and the interpretation of the results

31. Any other comments?

Please upload two picture (one per sample) of the MOAH chromatograms for the samples A and B , overlaying the respective reagent blank? Please scale the picture so that the MOAH hump could be visible at at least 2/3 of the scale.



Please upload two picture (one per sample) of the MOSH chromatograms for the samples A and B , overlaying the respective reagent blank? Please scale the picture so that the MOSH hump could be visible at at least 2/3 of the scale.

Please upload *.cvs or *.txt file of your MOAH reagent blank **chromatograms** (via export file function of your software)

Please upload your *.cvs or *.txt file of your Test item B MOAH **chromatogram** (via export file function of your software)

**Contact**

Contact Form

# Annex 6: Homogeneity and stability results

## 6.1 Homogeneity (normalised)

| | MOAH-TBB | | | | | |
|---|---|---|---|---|---|---|
| | Test item A | | Test item B | | Test item C | |
| 1 | 100% | 99% | 102% | 106% | 100% | 98% |
| 2 | 98% | 97% | 105% | 101% | 103% | 97% |
| 3 | 103% | 101% | 99% | 96% | 102% | 99% |
| 4 | 97% | 100% | 101% | 95% | 101% | 99% |
| 5 | 99% | 104% | 96% | 100% | 99% | 99% |
| 6 | 99% | 102% | 101% | 98% | 99% | 97% |
| 7 | 103% | 99% | 102% | 99% | 98% | 98% |
| 8 | | | | | 103% | 104% |
| 9 | | | | | 105% | 101% |
| Mean | 100% | | 100% | | 100% | |
| $u_{hom}$ | 1.0% | | 1.4% | | 1.2% | |
| $\sigma_{pt}$ | 25% | | 30% | | 15% | |
| 0.3 $\sigma_{pt}$ | 7.5% | | 9.0% | | 4.5% | |
| $u_{hom}$ < 0.3 $\sigma_{pt}$ | passed | | passed | | passed | |

| | MOSH | | | | | |
|---|---|---|---|---|---|---|
| | Test item A | | Test item B | | Test item C | |
| 1 | 101% | 101% | 102% | 104% | 103% | 97% |
| 2 | 102% | 102% | 100% | 99% | 103% | 98% |
| 3 | 101% | 100% | 102% | 98% | 102% | 97% |
| 4 | 98% | 97% | 99% | 101% | 103% | 98% |
| 5 | 98% | 102% | 99% | 97% | 98% | 99% |
| 6 | 101% | 100% | 100% | 99% | 97% | 98% |
| 7 | 100% | 99% | 99% | 102% | 98% | 98% |
| 8 | | | | | 103% | 103% |
| 9 | | | | | 102% | 103% |
| Mean | 100% | | 100% | | 100% | |
| $u_{hom}$ | 1.1% | | 1.1% | | 1.2% | |
| $\sigma_{pt}$ | 20% | | 20% | | 15% | |
| 0.3 $\sigma_{pt}$ | 6.0% | | 6.0% | | 4.5% | |
| $u_{hom}$ < 0.3 $\sigma_{pt}$ | passed | | passed | | passed | |

Where: $\sigma_{pt}$ is the standard deviation for the PT assessment,
$u_{hom}$ is the standard uncertainty contribution due to homogeneity

## 6.2 Stability

| | MOAH-TBB | | MOSH | |
|---|---|---|---|---|
| | Sample A | Sample B | Sample A | Sample B |
| $t_0$ | 32.5 | 1.7 | 121.3 | 77.1 |
| $t_{10}$ | 31.7 | 1.8 | 121.2 | 77.0 |
| $\lvert t_0-t_{10}\rvert$ | 0.8 | 0.1 | 0.1 | 0.1 |
| $0.3\ \sigma_{pt}$ | 2.8 | 0.2 | 7.1 | 4.1 |
| $\lvert t_0-t_{10}\rvert < 0.3\ \sigma_{pt}$ | passed | passed | passed | passed |

$t_0$ –initial time

$t_{10}$ – 10 weeks later (after closing the PT)

## Annex 7: Test item characterisation

| | | Certifier | *replicates* | | | | $x_{pt}$ | $u(x_{pt})$ |
|---|---|---|---|---|---|---|---|---|
| MOSH | Test item A | C1 | 110 | 113 | | | 118.6 | 2.7 |
| | | C2 | 118.6 | 118.7 | | | | |
| | | C3 | 120.79 | 118.29 | | | | |
| | | C4 | 121 | 120 | 125 | 133 | | |
| | Test item B | C1 | 61 | 61.6 | | | 68.40 | 4.30 |
| | | C2 | 66.03 | 66.3 | | | | |
| | | C3 | 63.23 | 67.21 | | | | |
| | | C4 | 80.1 | 80.5 | 80.9 | 82.1 | | |
| | Test item C | C1 | 653 | 612 | | | 679.7 | 35.9 |
| | | C2 | 691.5 | 692.8 | | | | |
| | | C3 | 616 | 620 | | | | |
| | | C4 | 776 | | | | | |
| MOAH-MN | Test item A | C1 | 44.4 | 42.5 | | | 43.54 | 1.04 |
| | | C2 | 42.13 | 42.07 | | | | |
| | | C3 | 47.9 | 45.1 | | | | |
| | | C4 | 44.7 | 33.4 | 44.7 | 45.6 | | |
| | Test item B | C1 | 3.19 | 3.15 | | | 2.765 | 0.245 |
| | | C2 | 2.52 | 2.44 | | | | |
| | | C3 | 3.18 | 3.2 | | | | |
| | | C4 | 2.09 | 2.25 | 2.17 | 2.37 | | |
| | Test item C | C1 | 263 | 242 | | | 248.3 | 7.5 |
| | | C2 | 240.4 | 241.6 | | | | |
| | | C3 | 232 | 233 | | | | |
| | | C4 | 267 | | | | | |
| MOAH-TBB | Test item A | C1 | 37.2 | 39.2 | | | 37.55 | 0.48 |
| | | C2 | 38.54 | 38.54 | | | | |
| | | C3 | 37.28 | 36.44 | | | | |
| | | C4 | 37.7 | 32.5 | 37.9 | 38.3 | | |
| | Test item B | C1 | 2.65 | 2.59 | | | 2.347 | 0.161 |
| | | C2 | 2.24 | 2.1775 | | | | |
| | | C3 | 2.55 | 2.65 | | | | |
| | | C4 | 2 | 1.96 | 1.86 | 2.01 | | |
| | Test item C | C1 | 254 | 238 | | | 249.3 | 10.8 |
| | | C2 | 256 | 257.2 | | | | |
| | | C3 | 221 | 222 | | | | |
| | | C4 | 273 | | | | | |
| | | | *all values in mg/kg* | | | | | |

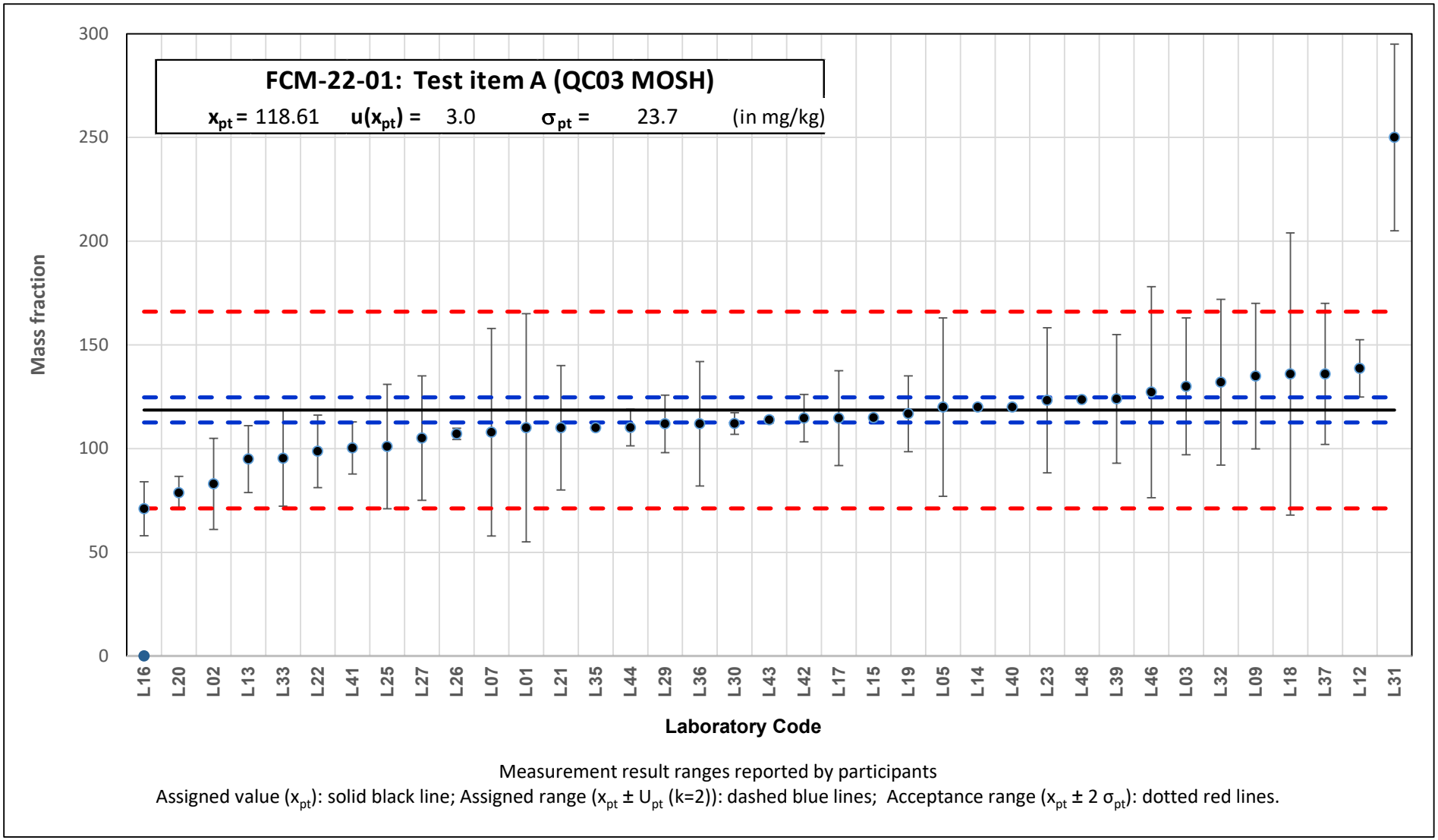
## Annex 8: Results for total MOSH mass fraction in Test item A

$x_{pt}$ = 118.6 ; $u(x_{pt})$ = 3.0 ; $\sigma_{pt}$ = 23.7 (all values in mg/kg)

| LabCode | $x_i$ | ± | k | Comment | z score | ζ score | MU |
|---|---|---|---|---|---|---|---|
| L01 | 110 | 55 | 2 | | -0.36 | -0.31 | c |
| L02 | 83 | 22 | 2 | | -1.50 | -3.12 | a |
| L03 | 130 | 33 | 2 | | 0.48 | 0.68 | a |
| L05 | 120 | 43 | 2 | | 0.06 | 0.06 | a |
| L07 | 107.9 | 50 | 2 | ± in % | -0.45 | | |
| L09 | 134.96 | 35.09 | 2 | | 0.69 | 0.92 | a |
| L12 | 138.63 | 13.8 | 2 | | 0.84 | 2.66 | a |
| L13 | 94.94 | 16.14 | 2 | | -1.00 | -2.75 | a |
| L14 | 120 | | | MU not provided | 0.06 | 0.46 | NP |
| L15 | 114.9 | 1.06 | 2 | | -0.16 | -1.21 | b |
| L16 | 71 | 13 | 2 | | -2.01 | -6.64 | a |
| L17 | 114.7 | 22.9 | 2 | | -0.16 | -0.33 | a |
| L18 | 136 | 68 | 2 | | 0.73 | 0.51 | c |
| L19 | 116.8 | 18.3 | 2 | | -0.08 | -0.19 | a |
| L20 | 78.69 | 7.96 | 1 | | -1.68 | -4.69 | a |
| L21 | 110 | 30 | 1.73 | ± in % | -0.36 | | |
| L22 | 98.7 | 17.5 | 2 | ± in % | -0.84 | | |
| L23 | 123.29 | 35 | 2 | ± in % | 0.20 | | |
| L25 | 101 | 30 | 1.73 | | -0.74 | -1.00 | a |
| L26 | 107.1 | 2.7 | 2 | | -0.49 | -3.48 | b |
| L27 | 105.02 | 30 | 2 | ± in % | -0.57 | | |
| L29 | 111.92 | 13.9 | 2 | | -0.28 | -0.88 | a |
| L30 | 112.04 | 5.2 | 2 | | -0.28 | -1.65 | b |
| L31 | 250 | 45 | 1 | | 5.54 | 2.91 | a |
| L32 | 132 | 40 | 2 | | 0.56 | 0.66 | a |
| L33 | 95.3 | 23 | 3 | ± in % | -0.98 | | |
| L34 | -- | -- | | | | | |
| L35 | 110 | | | MU not provided | -0.36 | -2.85 | NP |
| L36 | 112 | 30 | 1.73 | ± in % | -0.28 | | |
| L37 | 136 | 34 | 2 | | 0.73 | 1.01 | a |
| L39 | 124 | 31 | 25 | ± % instead of k | 0.23 | | |
| L40 | 120 | 0.5 | 2 | | 0.06 | 0.46 | b |
| L41 | 100.3 | 12.6 | 2 | | -0.77 | -2.62 | a |
| L42 | 114.67 | 11.47 | 2 | | -0.17 | -0.61 | a |
| L43 | 113.85 | 1.56 | 2 | | -0.20 | -1.53 | b |
| L44 | 110.17 | 8.81 | 2 | | -0.36 | -1.58 | a |
| L46 | 127.2 | 50.9 | 2.8 | | 0.36 | 0.47 | a |
| L48 | 123.6306 | | | MU not provided | 0.21 | 1.66 | NP |

Performance (z, z', ζ): Satisfactory (green); Questionable (yellow); Unsatisfactory (Red)

MU - (a): $u(x_{pt,rel}) \leq u(x_{i,rel}) \leq \sigma_{pt,rel}$ ; (b): $u(x_{i,rel}) < u(x_{pt,rel})$; (c): $u(x_{i,rel}) > \sigma_{pt,rel}$; NP: *not provided*




Measurement result ranges reported by participants

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

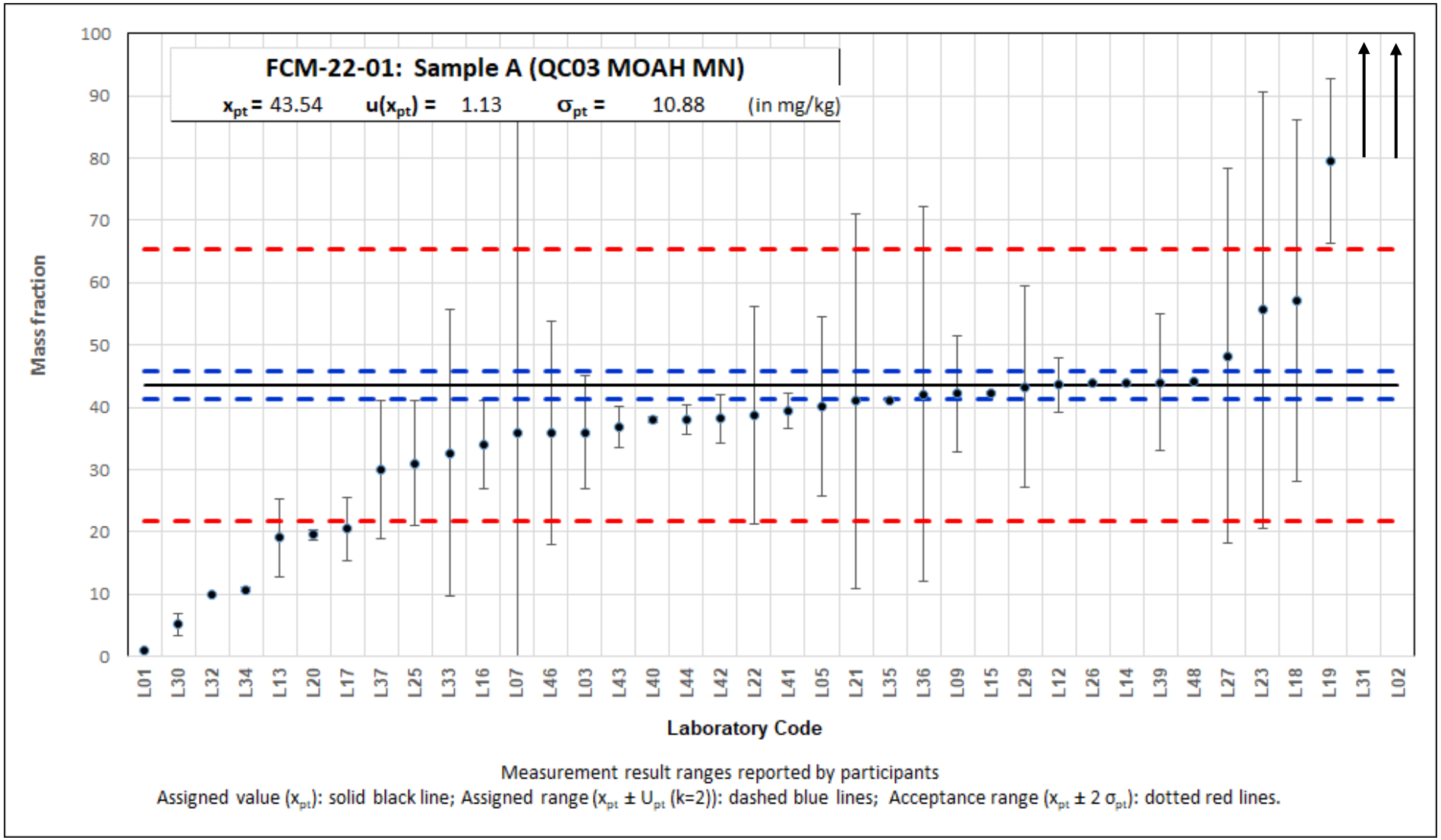
## Annex 9: Results for total MOAH-MN mass fraction in Test item A

$x_{pt}$ = 43.5 ; $u(x_{pt})$ = 1.1 ; $\sigma_{pt}$ = 10.9 (all values in mg/kg)

| LabCode | $x_i$ | ± | k | Comment | z score | ζ score | MU |
|---|---|---|---|---|---|---|---|
| L01 | < 1 | | | Less than | | | |
| L02 | 572 | 101 | 2 | | 48.55 | 10.46 | a |
| L03 | 36 | 9 | 2 | | -0.69 | -1.62 | a |
| L05 | 40.1 | 14.4 | 2 | | -0.32 | -0.47 | a |
| L07 | 35.8 | 50 | 2 | ± in % | -0.71 | | |
| L09 | 42.2 | 9.28 | 2 | | -0.12 | -0.28 | a |
| L12 | 43.56 | 4.35 | 2 | | 0.00 | 0.01 | a |
| L13 | 19.07 | 6.29 | 2 | | -2.25 | -7.32 | a |
| L14 | 44 | | | MU not provided | 0.04 | 0.41 | NP |
| L15 | 42.3 | 0.28 | 2 | | -0.11 | -1.09 | b |
| L16 | 34 | 7 | 2 | | -0.88 | -2.59 | a |
| L17 | 20.5 | 5.1 | 2 | | -2.12 | -8.26 | a |
| L18 | 57 | 29 | 2 | | 1.24 | 0.93 | c |
| L19 | 79.5 | 13.2 | 2 | | 3.30 | 5.37 | a |
| L20 | 19.52 | 0.74 | 1 | | -2.21 | -17.77 | a |
| L21 | 41 | 30 | 1.73 | ± in % | -0.23 | | |
| L22 | 38.7 | 17.5 | 2 | ± in % | -0.44 | | |
| L23 | 55.6 | 35 | 2 | ± in % | 1.11 | | |
| L25 | 31 | 10 | 1.73 | | -1.15 | -2.13 | a |
| L26 | 43.88 | 0.12 | 2 | | 0.03 | 0.30 | b |
| L27 | 48.25 | 30 | 2 | ± in % | 0.43 | | |
| L29 | 43.3 | 16.1 | 2 | | -0.02 | -0.03 | a |
| L30 | 5.18 | 1.8 | 2 | | -3.52 | -26.54 | a |
| L31 | 295 | 46 | 1 | | 23.10 | 5.46 | a |
| L32 | < 10 | | | Less than | | | |
| L33 | 32.7 | 23 | 3 | ± in % | -1.00 | | |
| L34 | 10.78 | 0.28 | 2 | | -3.01 | -28.74 | b |
| L35 | 41 | | | MU not provided | -0.23 | -2.24 | NP |
| L36 | 42.1 | 30 | 1.73 | ± in % | -0.13 | | |
| L37 | 30 | 11 | 2 | | -1.24 | -2.41 | a |
| L39 | 44 | 11 | 25 | ± % instead of k | 0.04 | | |
| L40 | 38 | 0.5 | 2 | | -0.51 | -4.78 | b |
| L41 | 39.4 | 2.9 | 2 | | -0.38 | -2.25 | a |
| L42 | 38.16 | 3.82 | 2 | | -0.49 | -2.42 | a |
| L43 | 36.85 | 3.25 | 2 | | -0.61 | -3.38 | a |
| L44 | 38.03 | 2.28 | 2 | | -0.51 | -3.43 | a |
| L46 | 35.9 | 18 | 2.8 | | -0.70 | -1.17 | a |
| L48 | 44.0702 | | | MU not provided | 0.05 | 0.47 | NP |

Performance (z, z', ζ): Satisfactory (green); Questionable (yellow); Unsatisfactory (Red)

MU - (a): $u(x_{pt,rel}) \leq u(x_{i,\,rel}) \leq \sigma_{pt,rel}$ ; (b): $u(x_{i,rel}) < u(x_{pt,rel})$; (c): $u(x_{i,rel}) > \sigma_{pt,rel}$; NP: *not provided*




Measurement result ranges reported by participants

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

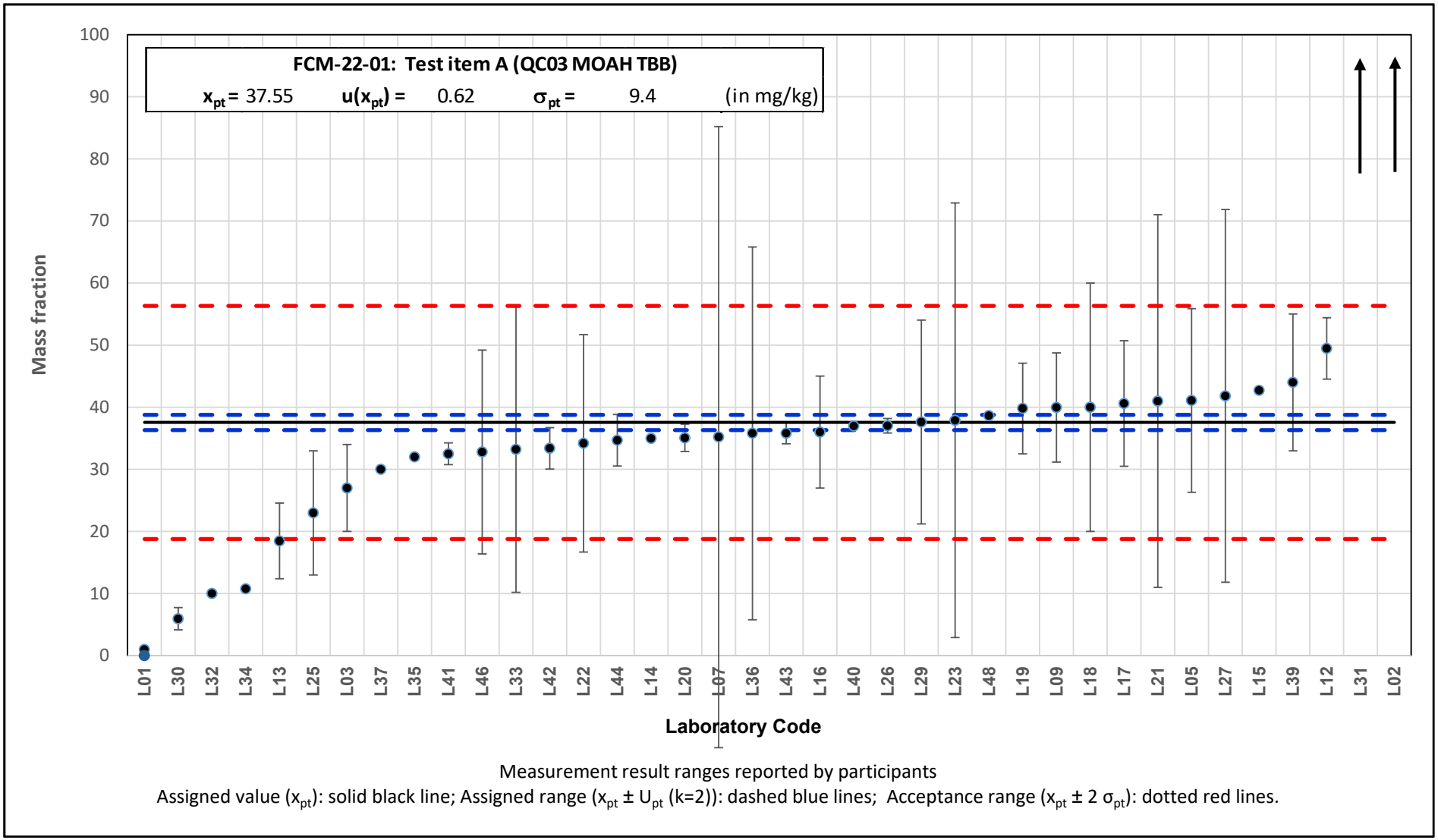
## Annex 10: Results for total MOAH-TBB mass fraction in Test item A

$x_{pt}$ = 37.55 ; $u(x_{pt})$ = 0.62 ; $\sigma_{pt}$ = 9.39 (all values in mg/kg)

| LabCode | $x_i$ | ± | k | Comment | z score | ζ score | MU |
|---|---|---|---|---|---|---|---|
| L01 | < 1 | | | Less than | | | |
| L02 | 484 | 78 | 2 | | 47.56 | 11.45 | a |
| L03 | 27 | 7 | 2 | | -1.12 | -2.97 | a |
| L05 | 41.1 | 14.8 | 2 | | 0.38 | 0.48 | a |
| L07 | 35.2 | 50 | 2 | ± in % | -0.25 | | |
| L09 | 39.96 | 8.79 | 2 | | 0.26 | 0.54 | a |
| L12 | 49.46 | 4.95 | 2 | | 1.27 | 4.67 | a |
| L13 | 18.47 | 6.1 | 2 | | -2.03 | -6.13 | a |
| L14 | 35 | | | MU not provided | -0.27 | -4.12 | NP |
| L15 | 42.7 | 0.3 | 2 | | 0.55 | 8.09 | b |
| L16 | 36 | 9 | 2 | | -0.17 | -0.34 | a |
| L17 | 40.6 | 10.1 | 2 | | 0.32 | 0.60 | a |
| L18 | 40 | 20 | 2 | | 0.26 | 0.24 | a |
| L19 | 39.8 | 7.3 | 2 | | 0.24 | 0.61 | a |
| L20 | 35.07 | 2.21 | 1 | | -0.26 | -1.08 | a |
| L21 | 41 | 30 | 1.73 | ± in % | 0.37 | | |
| L22 | 34.2 | 17.5 | 2 | ± in % | -0.36 | | |
| L23 | 37.9 | 35 | 2 | ± in % | 0.04 | | |
| L25 | 23 | 10 | 1.73 | | -1.55 | -2.51 | c |
| L26 | 37.02 | 1.16 | 2 | | -0.06 | -0.63 | b |
| L27 | 41.83 | 30 | 2 | ± in % | 0.46 | | |
| L29 | 37.63 | 16.4 | 2 | | 0.01 | 0.01 | a |
| L30 | 5.95 | 1.8 | 2 | | -3.37 | -28.94 | a |
| L31 | 460 | 334 | 1 | | 45.00 | 1.26 | c |
| L32 | < 10 | | | Less than | | | |
| L33 | 33.2 | 23 | 3 | ± in % | -0.46 | | |
| L34 | 10.78 | 0.28 | 2 | | -2.85 | -42.21 | b |
| L35 | 32 | | | MU not provided | -0.59 | -8.97 | NP |
| L36 | 35.8 | 30 | 1.73 | ± in % | -0.19 | | |
| L37 | 30 | | | MU not provided | -0.80 | -12.20 | NP |
| L39 | 44 | 11 | 25 | ± % instead of k | 0.69 | | |
| L40 | 37 | 0.5 | 2 | | -0.06 | -0.82 | b |
| L41 | 32.5 | 1.75 | 2 | | -0.54 | -4.71 | a |
| L42 | 33.39 | 3.34 | 2 | | -0.44 | -2.34 | a |
| L43 | 35.8 | 1.7 | 2 | | -0.19 | -1.66 | a |
| L44 | 34.69 | 4.16 | 2 | | -0.30 | -1.32 | a |
| L46 | 32.8 | 16.4 | 2.8 | | -0.51 | -0.81 | a |
| L48 | 38.636 | | | MU not provided | 0.12 | 1.76 | NP |

Performance (z, z', ζ): Satisfactory (green); Questionable (yellow); Unsatisfactory (Red)

MU - (a): $u(x_{pt,rel}) \leq u(x_{i,\,rel}) \leq \sigma_{pt,rel}$ ; (b): $u(x_{i,rel}) < u(x_{pt,rel})$; (c): $u(x_{i,rel}) > \sigma_{pt,rel}$; NP: *not provided*




Measurement result ranges reported by participants

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

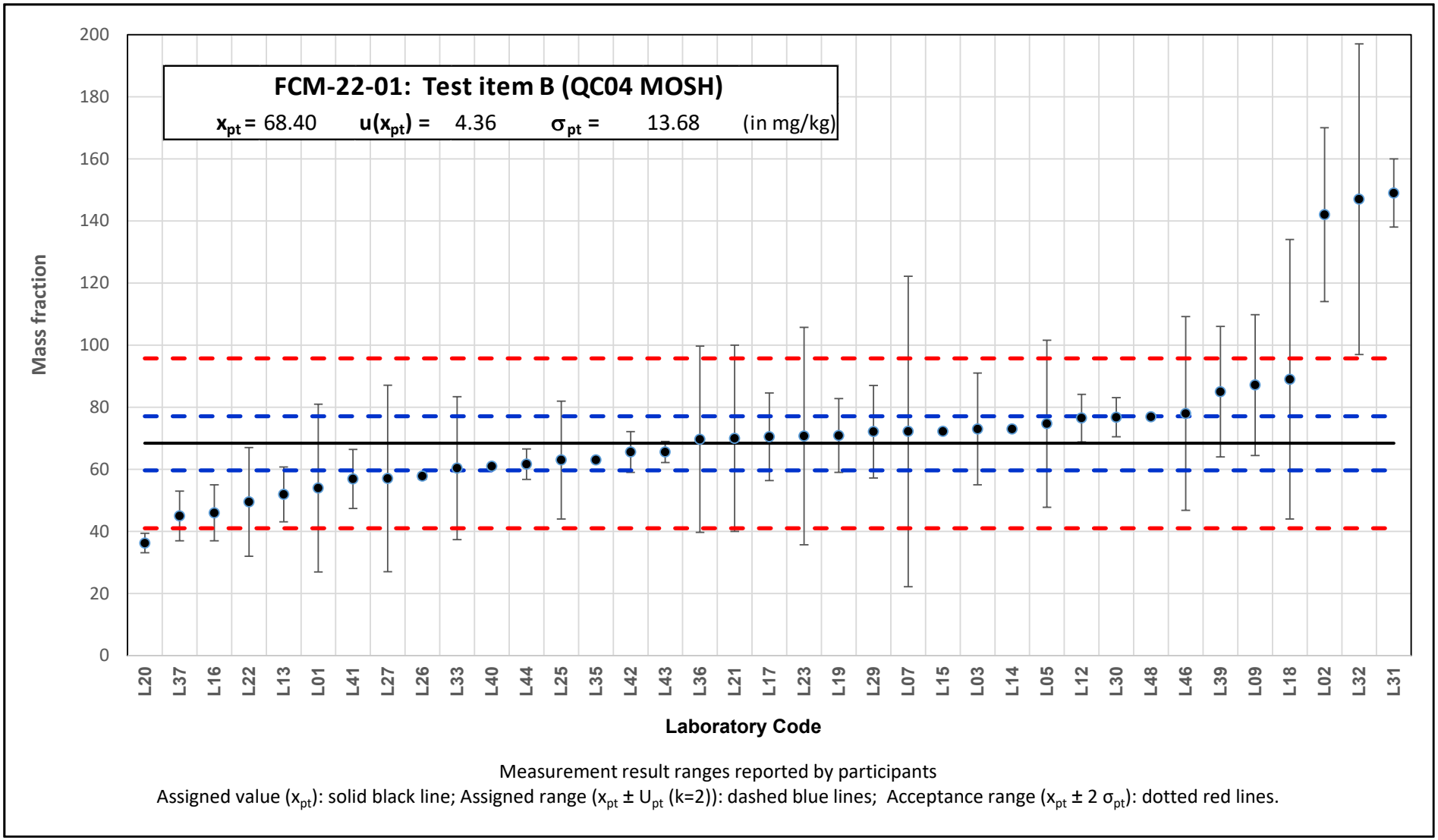
## Annex 11: Results for total MOSH mass fraction in Test item B

$x_{pt}$ = 68.40 ; $u(x_{pt})$ = 4.36 ; $\sigma_{pt}$ = 13.7; $\sigma'_{pt}$ = 13.7 (all values in mg/kg)

| LabCode | $x_i$ | ± | k | Comment | z prime | ζ score | MU |
|---|---|---|---|---|---|---|---|
| L01 | 54 | 27 | 2 | | -1.00 | -1.01 | c |
| L02 | 142 | 28 | 2 | | 5.13 | 5.02 | a |
| L03 | 73 | 18 | 2 | | 0.32 | 0.46 | a |
| L05 | 74.7 | 26.9 | 2 | | 0.44 | 0.45 | a |
| L07 | 72.2 | 50 | 2 | ± in % | 0.26 | | |
| L09 | 87.14 | 22.66 | 2 | | 1.31 | 1.54 | a |
| L12 | 76.51 | 7.65 | 2 | | 0.57 | 1.40 | b |
| L13 | 51.96 | 8.83 | 2 | | -1.14 | -2.65 | a |
| L14 | 73 | | | MU not provided | 0.32 | 1.05 | NP |
| L15 | 72.2 | 0.61 | 2 | | 0.26 | 0.87 | b |
| L16 | 46 | 9 | 2 | | -1.56 | -3.57 | a |
| L17 | 70.5 | 14.1 | 2 | | 0.15 | 0.25 | a |
| L18 | 89 | 45 | 2 | | 1.43 | 0.90 | c |
| L19 | 70.9 | 11.9 | 2 | | 0.17 | 0.34 | a |
| L20 | 36.27 | 3.12 | 1 | | -2.24 | -5.99 | a |
| L21 | 70 | 30 | 1.73 | ± in % | 0.11 | | |
| L22 | 49.5 | 17.5 | 2 | ± in % | -1.32 | | |
| L23 | 70.7 | 35 | 2 | ± in % | 0.16 | | |
| L25 | 63 | 19 | 1.73 | | -0.38 | -0.46 | a |
| L26 | 57.83 | 0.96 | 2 | | -0.74 | -2.41 | B |
| L27 | 57.07 | 30 | 2 | ± in % | -0.79 | | |
| L29 | 72.14 | 14.9 | 2 | | 0.26 | 0.43 | a |
| L30 | 76.76 | 6.3 | 2 | | 0.58 | 1.55 | b |
| L31 | 149 | 11 | 1 | | 5.61 | 6.81 | a |
| L32 | 147 | 50 | 2 | | 5.47 | 3.10 | a |
| L33 | 60.4 | 23 | 3 | ± in % | -0.56 | | |
| L34 | -- | -- | | | | | |
| L35 | 63 | | | MU not provided | -0.38 | -1.24 | NP |
| L36 | 69.7 | 30 | 1.73 | ± in % | 0.09 | | |
| L37 | 45 | 8 | 2 | | -1.63 | -3.95 | a |
| L39 | 85 | 21 | 25 | ± % instead of k | 1.16 | | |
| L40 | 61 | 0.5 | 2 | | -0.52 | -1.69 | b |
| L41 | 56.9 | 9.5 | 2 | | -0.80 | -1.78 | a |
| L42 | 65.58 | 6.56 | 2 | | -0.20 | -0.52 | b |
| L43 | 65.6 | 3.39 | 2 | | -0.19 | -0.60 | b |
| L44 | 61.67 | 4.93 | 2 | | -0.47 | -1.34 | b |
| L46 | 78 | 31.2 | 2.8 | | 0.67 | 0.80 | a |
| L48 | 76.9214 | | | MU not provided | 0.59 | 1.95 | NP |

Performance (z, z', ζ): Satisfactory (green); Questionable (yellow); Unsatisfactory (Red)

MU - (a): $u(x_{pt,rel}) \leq u(x_{i,rel}) \leq \sigma_{pt,rel}$ ; (b): $u(x_{i,rel}) < u(x_{pt,rel})$; (c): $u(x_{i,rel}) > \sigma_{pt,rel}$; NP: *not provided*




Measurement result ranges reported by participants

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

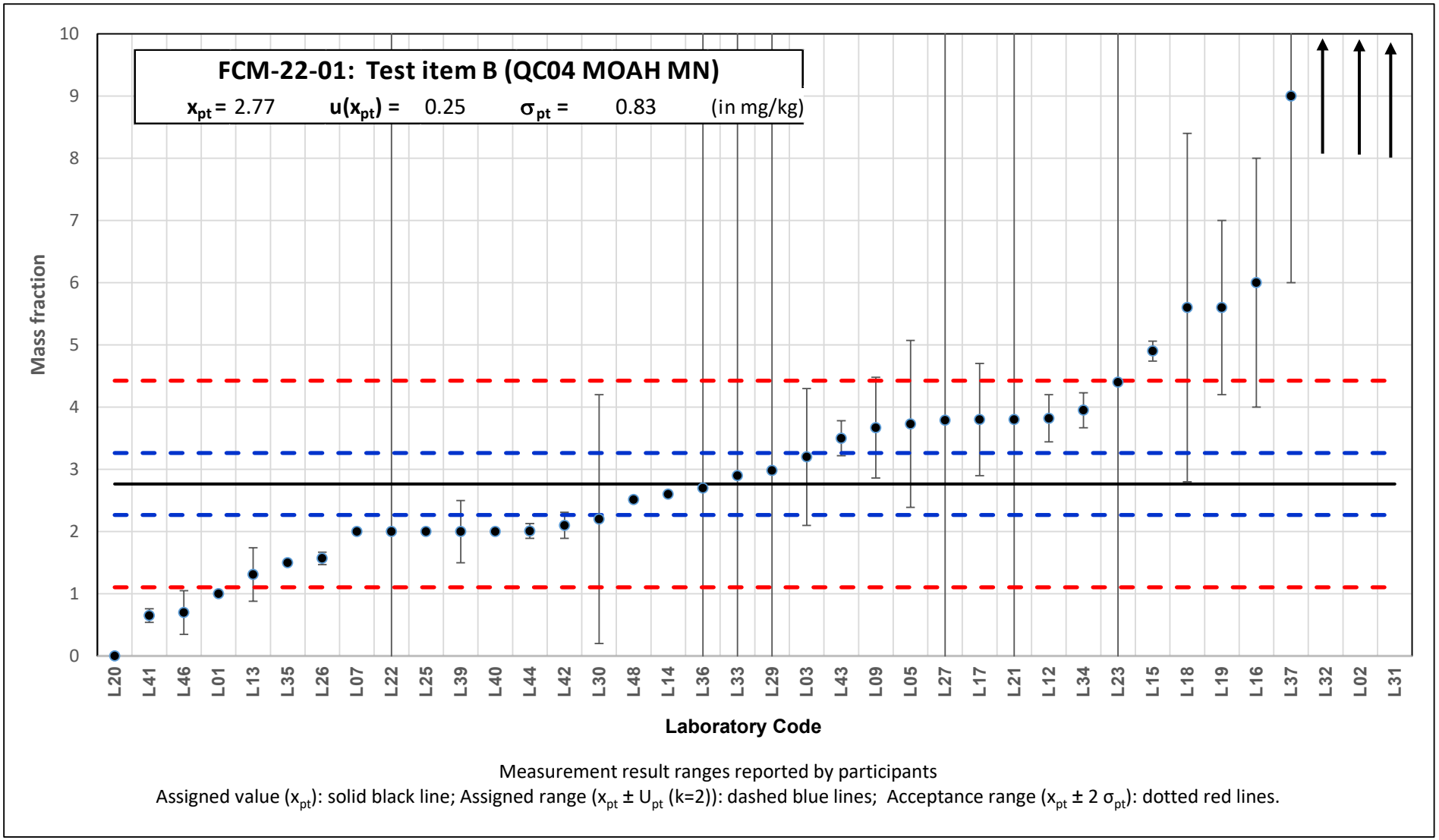
## Annex 12: Results for total MOAH-MN mass fraction in Test item B

$x_{pt}$ = 2.77 ; $u(x_{pt})$ = 0.25 ; $\sigma_{pt}$ = 0.83 (all values in mg/kg)

| LabCode | $x_i$ | ± | k | Comment | z score | ζ score | MU |
|---|---|---|---|---|---|---|---|
| L01 | < 1 | | | Less than | | | |
| L02 | 194 | 47 | 2 | | 230.54 | 8.14 | a |
| L03 | 3.2 | 1.1 | 2 | | 0.52 | 0.72 | a |
| L05 | 3.73 | 1.34 | 2 | | 1.16 | 1.35 | a |
| L07 | < 2 | | | Less than | | | |
| L09 | 3.67 | 0.81 | 2 | | 1.09 | 1.90 | a |
| L12 | 3.82 | 0.38 | 2 | | 1.27 | 3.37 | b |
| L13 | 1.31 | 0.43 | 2 | | -1.75 | -4.43 | a |
| L14 | 2.6 | | | MU not provided | -0.20 | -0.66 | NP |
| L15 | 4.9 | 0.16 | 2 | | 2.57 | 8.17 | b |
| L16 | 6 | 2 | 2 | | 3.90 | 3.14 | a |
| L17 | 3.8 | 0.9 | 2 | | 1.25 | 2.01 | a |
| L18 | 5.6 | 2.8 | 2 | | 3.42 | 1.99 | a |
| L19 | 5.6 | 1.4 | 2 | | 3.42 | 3.82 | a |
| L20 | 0 ? | | 1 | Reported result? | | | |
| L21 | 3.8 | 30 | 1.73 | ± in % | 1.25 | | |
| L22 | 2 | 17.5 | 2 | ± in % | -0.92 | | |
| L23 | 4.4 | 35 | 2 | ± in % | 1.97 | | |
| L25 | < 2 | | | Less than | | | |
| L26 | 1.57 | 0.1 | 2 | | -1.44 | -4.71 | b |
| L27 | 3.79 | 30 | 2 | ± in % | 1.24 | | |
| L29 | 2.98 | 24 | 2 | ± in % | 0.26 | | |
| L30 | 2.2 | 2 | 2 | | -0.68 | -0.55 | c |
| L31 | 223 | 26 | 1 | | 265.50 | 8.47 | a |
| L32 | 29 | 12 | 2 | | 31.63 | 4.37 | a |
| L33 | 2.9 | 23 | 3 | ± in % | 0.16 | | |
| L34 | 3.95 | 0.28 | 2 | | 1.43 | 4.15 | b |
| L35 | 1.5 | | | MU not provided | -1.53 | -5.09 | NP |
| L36 | 2.7 | 30 | 1.73 | ± in % | -0.08 | | |
| L37 | 9 | 3 | 2 | | 7.52 | 4.10 | a |
| L39 | 2 | 0.5 | 25 | ± % instead of k | -0.92 | | |
| L40 | < 2 | | | Less than | | | |
| L41 | 0.65 | 0.11 | 2 | | -2.55 | -8.30 | b |
| L42 | 2.1 | 0.21 | 2 | | -0.80 | -2.46 | b |
| L43 | 3.5 | 0.28 | 2 | | 0.89 | 2.58 | b |
| L44 | 2.01 | 0.12 | 2 | | -0.91 | -2.95 | b |
| L46 | 0.7 | 0.35 | 2.8 | | -2.49 | -7.42 | a |
| L48 | 2.5143 | | | MU not provided | -0.30 | -1.01 | NP |

Performance (z, z', ζ): Satisfactory (green); Questionable (yellow); Unsatisfactory (Red)

MU - (a): $u(x_{pt,rel}) \leq u(x_{i,rel}) \leq \sigma_{pt,rel}$ ; (b): $u(x_{i,rel}) < u(x_{pt,rel})$; (c): $u(x_{i,rel}) > \sigma_{pt,rel}$; NP: *not provided*




Measurement result ranges reported by participants

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

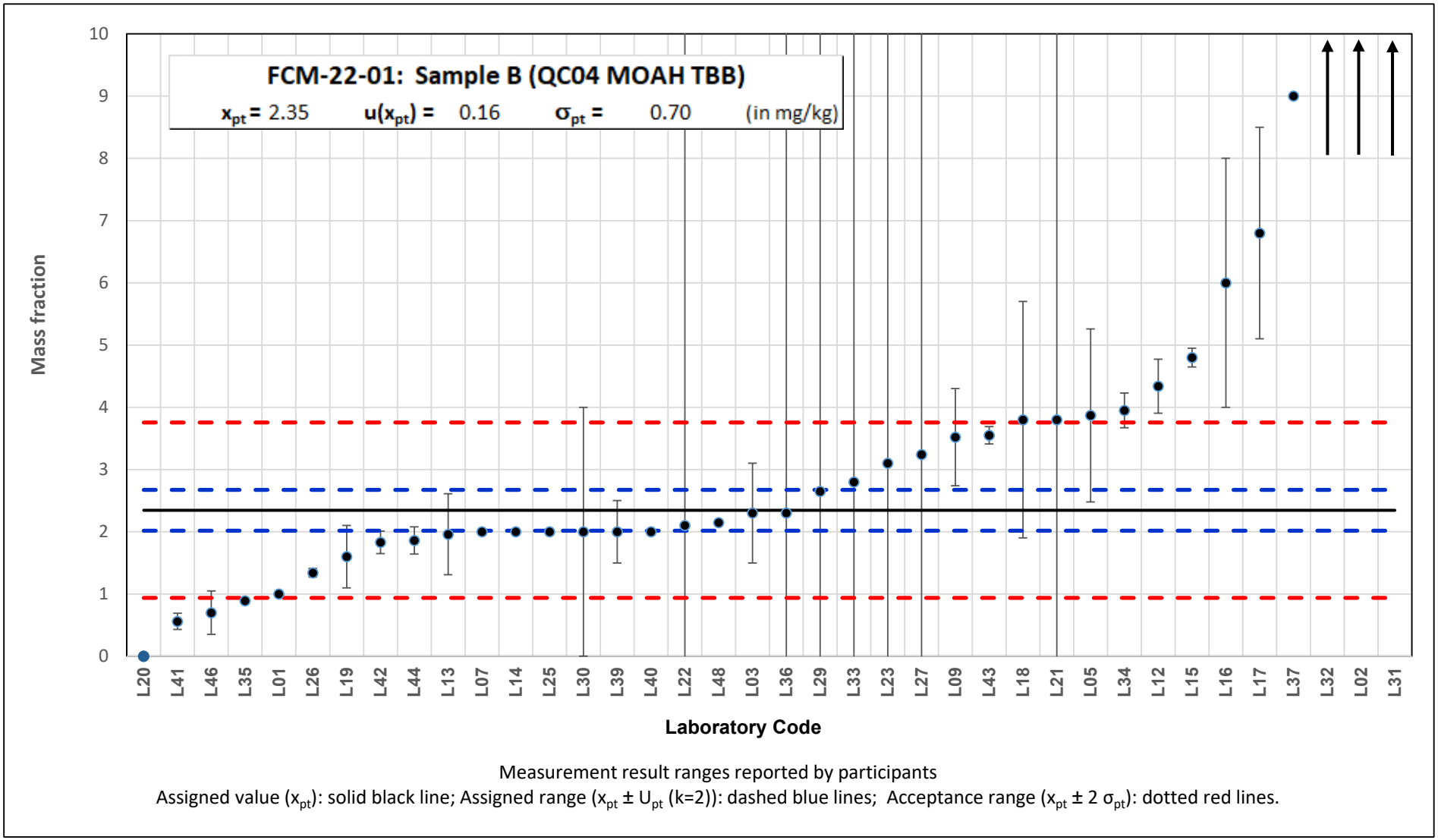
## Annex 13: Results for total MOAH-TBB mass fraction in Test item B

$x_{pt}$ = 2.35; $u(x_{pt})$ = 0.16 ; $\sigma_{pt}$ = 0.70 (all values in mg/kg)

| LabCode | $x_i$ | ± | k | Comment | z score | ζ score | MU |
|---|---|---|---|---|---|---|---|
| L01 | < 1 | | | Less than | | | |
| L02 | 193 | 52 | 2 | | 270.83 | 7.33 | a |
| L03 | 2.3 | 0.8 | 2 | | -0.07 | -0.11 | a |
| L05 | 3.87 | 1.39 | 2 | | 2.16 | 2.13 | a |
| L07 | < 2 | | | Less than | | | |
| L09 | 3.52 | 0.78 | 2 | | 1.67 | 2.77 | a |
| L12 | 4.34 | 0.434 | 2 | | 2.83 | 7.33 | b |
| L13 | 1.96 | 0.65 | 2 | | -0.55 | -1.06 | a |
| L14 | 2 | | | MU not provided | -0.49 | -2.11 | NP |
| L15 | 4.8 | 0.15 | 2 | | 3.49 | 13.60 | b |
| L16 | 6 | 2 | 2 | | 5.19 | 3.61 | a |
| L17 | 6.8 | 1.7 | 2 | | 6.33 | 5.14 | a |
| L18 | 3.8 | 1.9 | 2 | | 2.06 | 1.51 | a |
| L19 | 1.6 | 0.5 | 2 | | -1.06 | -2.50 | a |
| L20 | 0 ? | | 1 | Reported result? | | | |
| L21 | 3.8 | 30 | 1.73 | ± in % | 2.06 | | |
| L22 | 2.1 | 17.5 | 2 | ± in % | -0.35 | | |
| L23 | 3.1 | 35 | 2 | ± in % | 1.07 | | |
| L25 | < 2 | | | Less than | | | |
| L26 | 1.34 | 0.07 | 2 | | -1.43 | -6.00 | b |
| L27 | 3.24 | 30 | 2 | ± in % | 1.27 | | |
| L29 | 2.65 | 24.4 | 2 | | 0.43 | 0.02 | c |
| L30 | 2 | 2 | 2 | | -0.49 | -0.34 | c |
| L31 | 248 | 88 | 1 | | 348.96 | 2.79 | c |
| L32 | 52 | 20 | 2 | | 70.53 | 4.96 | a |
| L33 | 2.8 | 23 | 3 | ± in % | 0.64 | | |
| L34 | 3.95 | 0.28 | 2 | | 2.28 | 7.43 | b |
| L35 | 0.89 | | | MU not provided | -2.07 | -8.87 | NP |
| L36 | 2.3 | 30 | 1.73 | ± in % | -0.07 | | |
| L37 | 9 | | | MU not provided | 9.45 | 40.54 | NP |
| L39 | 2 | 0.5 | 25 | ± % instead of k | -0.49 | | |
| L40 | < 2 | | | Less than | | | |
| L41 | 0.56 | 0.13 | 2 | | -2.54 | -10.12 | a |
| L42 | 1.83 | 0.18 | 2 | | -0.73 | -2.76 | b |
| L43 | 3.55 | 0.14 | 2 | | 1.71 | 6.74 | b |
| L44 | 1.86 | 0.22 | 2 | | -0.69 | -2.46 | b |
| L46 | 0.7 | 0.35 | 2.8 | | -2.34 | -7.98 | a |
| L48 | 2.1456 | | | MU not provided | -0.29 | -1.22 | NP |

Performance (z, z', ζ): Satisfactory (green); Questionable (yellow); Unsatisfactory (Red)

MU - (a): $u(x_{pt,rel}) \leq u(x_{i,rel}) \leq \sigma_{pt,rel}$ ; (b): $u(x_{i,rel}) < u(x_{pt,rel})$; (c): $u(x_{i,rel}) > \sigma_{pt,rel}$; NP: *not provided*




Measurement result ranges reported by participants

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

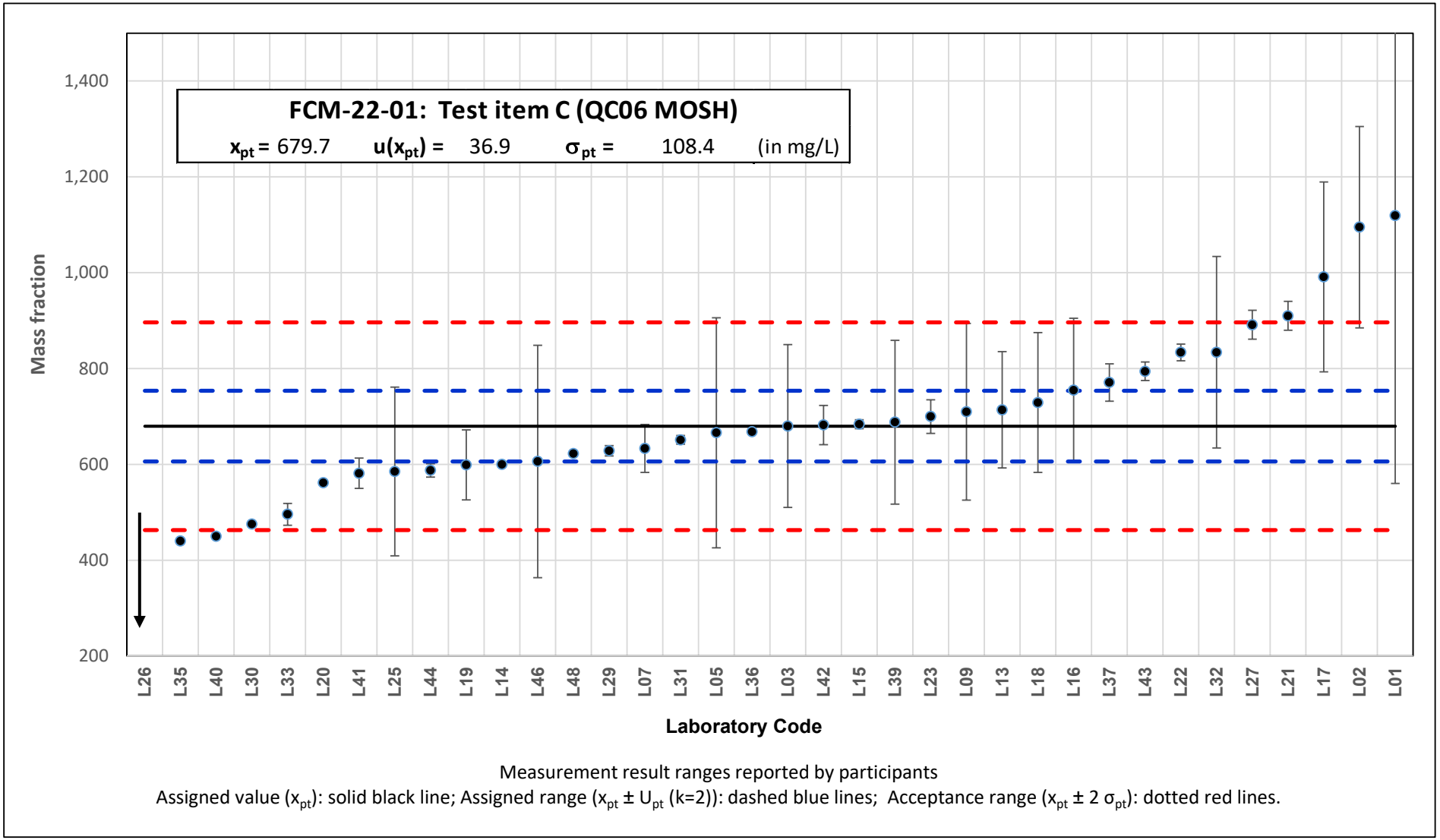
## Annex 14: Results for total MOSH concentration in Test item C

$x_{pt}$ = 680 ; $u(x_{pt})$ = 37 ; $\sigma_{pt}$ = 102; $\sigma'_{pt}$ = 108 (all values in mg/L)

| LabCode | $x_i$ | ± | k | Comment | z prime | ζ score | MU |
|---|---|---|---|---|---|---|---|
| L01 | 1119 | 559 | 2 | | 4.05 | 1.56 | c |
| L02 | 1095 | 210 | 2 | | 3.83 | 3.73 | a |
| L03 | 680 | 170 | 2 | | 0.00 | 0.00 | a |
| L05 | 666 | 240 | 2 | | -0.13 | -0.11 | c |
| L07 | 633.2 | 50 | 2 | ± in % | -0.43 | | |
| L09 | 709.78 | 184.54 | 2 | | 0.28 | 0.30 | a |
| L13 | 713.78 | 121.34 | 2 | | 0.31 | 0.48 | a |
| L14 | 600 | | | MU not provided | -0.73 | -2.16 | NP |
| L15 | 683.9 | 9.38 | 2 | | 0.04 | 0.11 | b |
| L16 | 755 | 150 | 2 | | 0.69 | 0.90 | a |
| L17 | 991.2 | 198 | 2 | | 2.87 | 2.95 | a |
| L18 | 729 | 146 | 2 | | 0.46 | 0.60 | a |
| L19 | 599 | 73.2 | 2 | | -0.74 | -1.55 | a |
| L20 | 561.8 | 2.09 | 1 | | -1.09 | -3.19 | b |
| L21 | 910 | 30 | 1.73 | ± in % | 2.12 | | |
| L22 | 833.6 | 17.5 | 2 | ± in % | 1.42 | | |
| L23 | 699.7 | 35 | 2 | ± in % | 0.18 | | |
| L25 | 585 | 176 | 1.73 | | -0.87 | -0.88 | c |
| L26 | 0.83 | 0 | 2 | | -6.26 | -18.41 | b |
| L27 | 891.41 | 30 | 2 | ± in % | 1.95 | | |
| L29 | 628.38 | 10.7 | 2 | | -0.47 | -1.38 | b |
| L30 | 475.45 | 2.4 | 2 | | -1.88 | -5.54 | b |
| L31 | 651 | 9 | 1 | | -0.26 | -0.76 | b |
| L32 | 834 | 200 | 2 | | 1.42 | 1.45 | a |
| L33 | 495.7 | 23 | 3 | ± in % | -1.70 | | |
| L34 | -- | -- | | | | | |
| L35 | 440 | | | MU not provided | -2.21 | -6.50 | NP |
| L36 | 668 | | | MU not provided | -0.11 | -0.32 | NP |
| L37 | 771 | 39 | 2 | | 0.84 | 2.19 | b |
| L39 | 688 | 171 | 25 | ± % instead of k | 0.08 | | |
| L40 | 450 | 0.5 | 2 | | -2.12 | -6.23 | b |
| L41 | 581.4 | 31.8 | 2 | | -0.91 | -2.45 | b |
| L42 | 682.21 | 40.93 | 2 | | 0.02 | 0.06 | b |
| L43 | 794.3 | 19.23 | 2 | | 1.06 | 3.01 | b |
| L44 | 587.56 | 14 | 2 | | -0.85 | -2.45 | b |
| L46 | 606.1 | 242.4 | 2.8 | | -0.68 | -0.78 | a |
| L48 | 622.2383 | | | MU not provided | -0.53 | -1.56 | NP |

Performance (z, z', ζ): Satisfactory (green); Questionable (yellow); Unsatisfactory (Red)

MU - (a): $u(x_{pt,rel}) \leq u(x_{i,rel}) \leq \sigma_{pt,rel}$ ; (b): $u(x_{i,rel}) < u(x_{pt,rel})$; (c): $u(x_{i,rel}) > \sigma_{pt,rel}$; NP: *not provided*




Measurement result ranges reported by participants

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

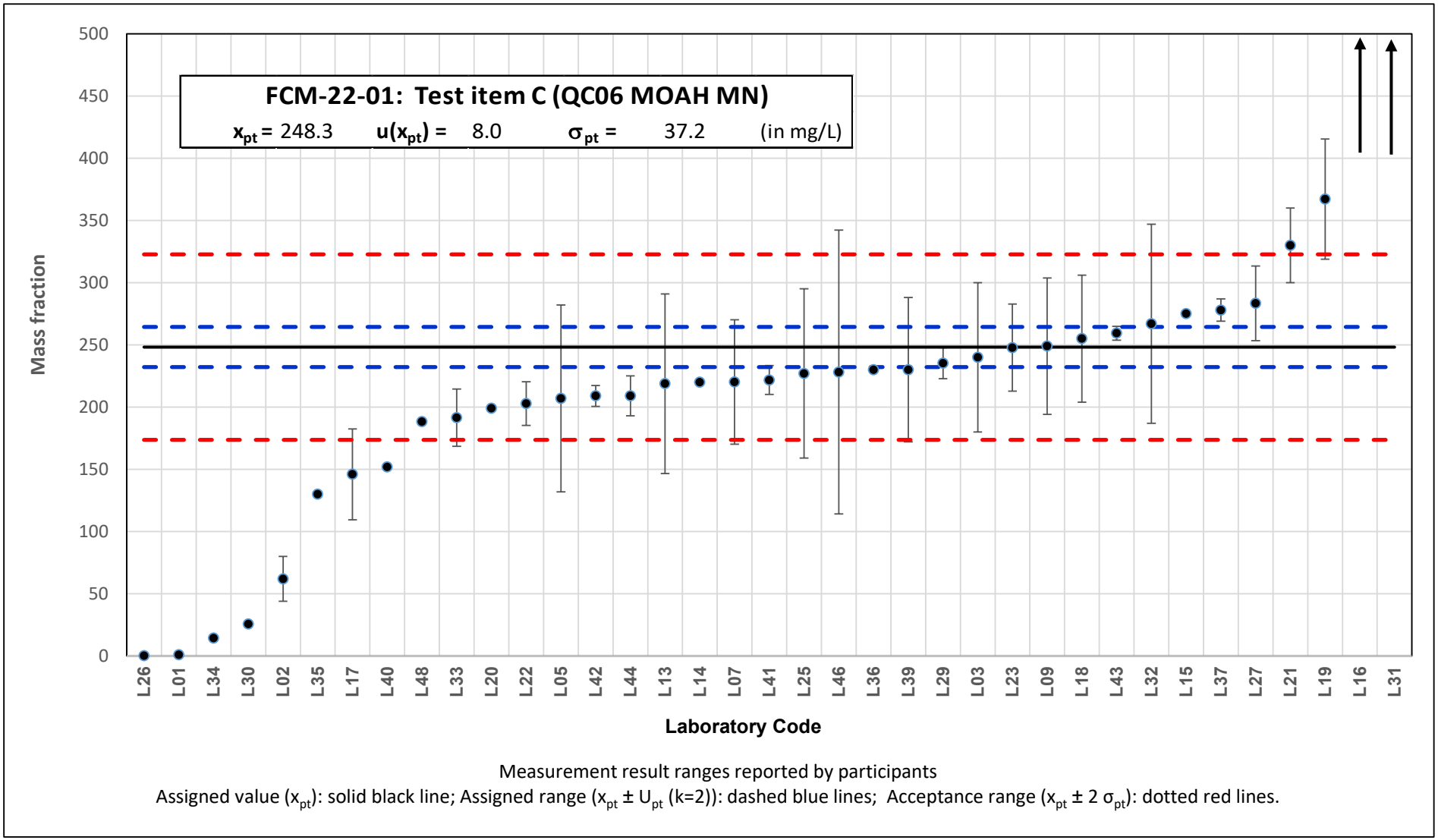
## Annex 15: Results for total MOAH-MN concentration in Test item C

$x_{pt}$ = 248.3; $u(x_{pt})$ = 8.0 ; $\sigma_{pt}$ = 37.2 (all values in mg/L)

| LabCode | $x_i$ | ± | k | Comment | z score | ζ score | MU |
|---|---|---|---|---|---|---|---|
| L01 | < 1 | | | Less tan | | | |
| L02 | 62 | 18 | 2 | | -5.00 | -15.43 | a |
| L03 | 240 | 60 | 2 | | -0.22 | -0.27 | a |
| L05 | 207 | 75 | 2 | | -1.11 | -1.08 | c |
| L07 | 220.2 | 50 | 2 | ± in % | -0.75 | | |
| L09 | 249 | 54.78 | 2 | | 0.02 | 0.03 | a |
| L13 | 218.79 | 72.2 | 2 | | -0.79 | -0.80 | c |
| L14 | 220 | | | MU not provided | -0.76 | -3.51 | NP |
| L15 | 275 | 1.12 | 2 | | 0.72 | 3.32 | b |
| L16 | 783 | 160 | 2 | | 14.36 | 6.65 | a |
| L17 | 146 | 36.5 | 2 | | -2.75 | -5.13 | a |
| L18 | 255 | 51 | 2 | | 0.18 | 0.25 | a |
| L19 | 367.1 | 48.3 | 2 | | 3.19 | 4.67 | a |
| L20 | 199.13 | 1.52 | 1 | | -1.32 | -6.00 | b |
| L21 | 330 | 30 | 1.73 | ± in % | 2.20 | | |
| L22 | 202.8 | 17.5 | 2 | ± in % | -1.22 | | |
| L23 | 247.8 | 35 | 2 | ± in % | -0.01 | | |
| L25 | 227 | 68 | 1.73 | | -0.57 | -0.53 | c |
| L26 | 0.28 | 0 | 2 | | -6.66 | -30.82 | b |
| L27 | 283.37 | 30 | 2 | ± in % | 0.94 | | |
| L29 | 235.29 | 12.4 | 2 | | -0.35 | -1.28 | b |
| L30 | 25.64 | 1.1 | 2 | | -5.98 | -27.60 | b |
| L31 | 933 | 51 | 1 | | 18.39 | 13.26 | a |
| L32 | 267 | 80 | 2 | | 0.50 | 0.46 | a |
| L33 | 191.5 | 23 | 3 | ± in % | -1.52 | | |
| L34 | 14.49 | 0.28 | 2 | | -6.28 | -29.05 | b |
| L35 | 130 | | | MU not provided | -3.18 | -14.70 | NP |
| L36 | 230 | | | MU not provided | -0.49 | -2.27 | NP |
| L37 | 278 | 9 | 2 | | 0.80 | 3.23 | b |
| L39 | 230 | 58 | 25 | ± % instead of k | -0.49 | | |
| L40 | 152 | 0.5 | 2 | | -2.58 | -11.96 | b |
| L41 | 221.7 | 11.6 | 2 | | -0.71 | -2.68 | b |
| L42 | 209.01 | 8.36 | 2 | | -1.05 | -4.33 | b |
| L43 | 259.35 | 5.52 | 2 | | 0.30 | 1.31 | b |
| L44 | 209.07 | 16 | 2 | | -1.05 | -3.45 | a |
| L46 | 228.2 | 114.1 | 2.8 | | -0.54 | -0.48 | c |
| L48 | 188.3382 | | | MU not provided | -1.61 | -7.45 | NP |

Performance (z, z', ζ): Satisfactory (green); Questionable (yellow); Unsatisfactory (Red)

MU - (a): $u(x_{pt,rel}) \leq u(x_{i,rel}) \leq \sigma_{pt,rel}$ ; (b): $u(x_{i,rel}) < u(x_{pt,rel})$; (c): $u(x_{i,rel}) > \sigma_{pt,rel}$; NP: *not provided*




Measurement result ranges reported by participants

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

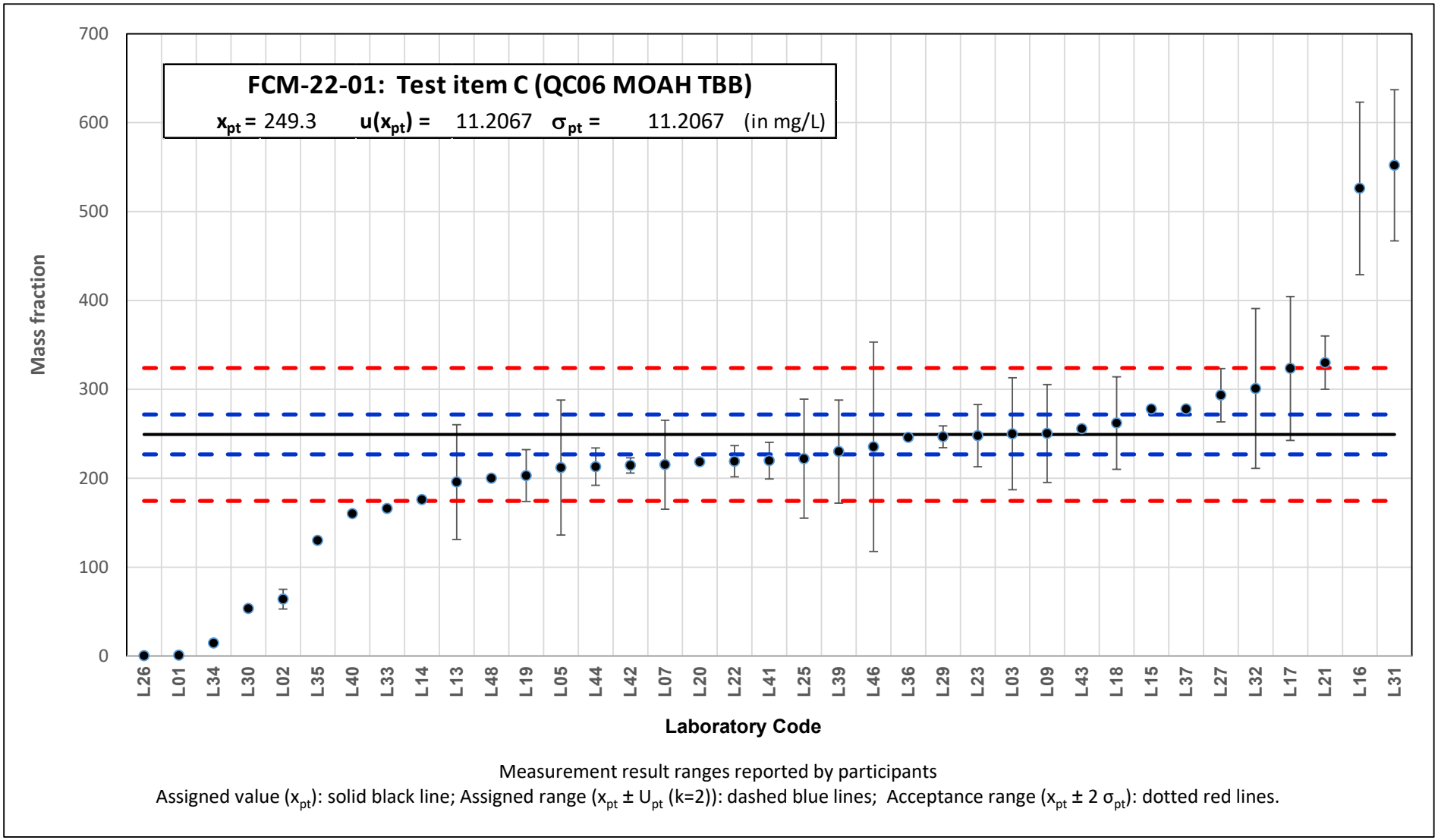
## Annex 16: Results for total MOAH-TBB concentration in Test item C

$x_{pt}$ = 249.3 ; $u(x_{pt})$ = 11.2 ; $\sigma_{pt}$ = 37.4 (all values in mg/L)

| LabCode | $x_i$ | ± | k | Comment | z score | ζ score | MU |
|---|---|---|---|---|---|---|---|
| L01 | < 1 | | | Less tan | | | |
| L02 | 64 | 11 | 2 | | -4.96 | -14.84 | a |
| L03 | 250 | 63 | 2 | | 0.02 | 0.02 | a |
| L05 | 212 | 76 | 2 | | -1.00 | -0.94 | c |
| L07 | 215.2 | 50 | 2 | ± in % | -0.91 | | |
| L09 | 250.36 | 55.08 | 2 | | 0.03 | 0.04 | a |
| L13 | 195.64 | 64.56 | 2 | | -1.43 | -1.57 | c |
| L14 | 176 | | | MU not provided | -1.96 | -6.54 | NP |
| L15 | 277.8 | 0.35 | 2 | | 0.76 | 2.54 | b |
| L16 | 526 | 97 | 2 | | 7.40 | 5.56 | a |
| L17 | 323.5 | 80.9 | 2 | | 1.98 | 1.77 | a |
| L18 | 262 | 52 | 2 | | 0.34 | 0.45 | a |
| L19 | 203 | 29.2 | 2 | | -1.24 | -2.52 | a |
| L20 | 218.48 | 0.93 | 1 | | -0.82 | -2.74 | b |
| L21 | 330 | 30 | 1.73 | ± in % | 2.16 | | |
| L22 | 219.1 | 17.5 | 2 | ± in % | -0.81 | | |
| L23 | 247.9 | 35 | 2 | ± in % | -0.04 | | |
| L25 | 222 | 67 | 1.73 | | -0.73 | -0.68 | c |
| L26 | 0.3 | 0 | 2 | | -6.66 | -22.22 | b |
| L27 | 293.41 | 30 | 2 | ± in % | 1.18 | | |
| L29 | 246.61 | 12.4 | 2 | | -0.07 | -0.21 | b |
| L30 | 53.5 | 1.1 | 2 | | -5.24 | -17.45 | b |
| L31 | 552 | 85 | 1 | | 8.09 | 3.53 | c |
| L32 | 301 | 90 | 2 | | 1.38 | 1.11 | a |
| L33 | 166 | | | ± in % | -2.23 | | |
| L34 | 14.49 | 0.28 | 2 | | -6.28 | -20.95 | b |
| L35 | 130 | | | MU not provided | -3.19 | -10.65 | NP |
| L36 | 246 | | | MU not provided | -0.09 | | NP |
| L37 | 278 | | | MU not provided | 0.77 | 2.56 | NP |
| L39 | 230 | 58 | 25 | ± % instead of k | -0.52 | | |
| L40 | 160 | 0.5 | 2 | | -2.39 | -7.97 | b |
| L41 | 219.8 | 20.54 | 2 | | -0.79 | -1.94 | a |
| L42 | 214.44 | 8.58 | 2 | | -0.93 | -2.91 | b |
| L43 | 255.75 | 1.84 | 2 | | 0.17 | 0.57 | b |
| L44 | 212.98 | 21 | 2 | | -0.97 | -2.37 | a |
| L46 | 235.4 | 117.7 | 2.8 | | -0.37 | -0.32 | c |
| L48 | 199.9507 | | | MU not provided | -1.32 | -4.40 | NP |

Performance (z, z', ζ): Satisfactory (green); Questionable (yellow); Unsatisfactory (Red)

MU - (a): $u(x_{pt,rel}) \leq u(x_{i,rel}) \leq \sigma_{pt,rel}$ ; (b): $u(x_{i,rel}) < u(x_{pt,rel})$; (c): $u(x_{i,rel}) > \sigma_{pt,rel}$; NP: *not provided*




Measurement result ranges reported by participants

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

## Annex 17: Results of the questionnaire

| I. Lab code | IV. Please select what is relevant for your laboratory: | A. How many edible oils@fats samples did you analyse for MOSH/ MOAH during the 2021 and 2022? | B. How many MOSH/MOAH analyses in general did you perform in 2021 and in 2022? | C. What type of matrices were the majority of the samples? | D. Are you accredited for MOSH/MOAH analyses? | E. In which samples/matrices? | F. Did you participate in the 2022 trial for MV of the MOSH/ MOAH in edible oils and fats | G. Did you follow exactly the SOP? |
|---|---|---|---|---|---|---|---|---|
| L02 | National Reference Laboratory for process contaminants or FCM (NRL) | 0 | 0 | 0 | No | | Yes | No |
| L03 | commercial lab | 750 | 5060 | Fats and oils, cocoa products, pastry, cereal products, nuts, fat sauces. | Yes | Fats and oils, cocoa products, pastry, cereal products, nuts, fat sauces. dairy products, vegetables, paper and cardboard | No | |
| L07 | commercial lab | 200 | 350 | edible oils | No | | No | |
| L09 | National Reference Laboratory for process contaminants or FCM (NRL); Official control laboratory (OCL) | 0 | 30 | cry cereal based foods, breakfast cereals, pasta and breads. | No | | No | |
| L12 | National Reference Laboratory for process contaminants or FCM (NRL) | 0 | 7 | Canned fishes and fruits of the sea. | Yes | Vegetable oils and extracted fats. | No | |
| L13 | commercial lab | 2021: 179<br><br>2022: 165 | 2021: 902<br><br>2022: 673 | Chocolate and rice | Yes | Yes, for coffee, rice and cereals, pasta, vegetable oil, cacao and dried fruits. | No | |

| I. Lab code | IV. Please select what is relevant for your laboratory: | A. How many edible oils@fats samples did you analyse for MOSH/ MOAH during the 2021 and 2022? | B. How many MOSH/MOAH analyses in general did you perform in 2021 and in 2022? | C. What type of matrices were the majority of the samples? | D. Are you accredited for MOSH/MOAH analyses? | E. In which samples/matrices? | F. Did you participate in the 2022 trial for MV of the MOSH/ MOAH in edible oils and fats | G. Did you follow exactly the SOP? |
|---|---|---|---|---|---|---|---|---|
| L14 | commercial lab | We are a starting lab therefore our numbers are not representative<br>In 2020 and begin 2021 only a few after accreditation in september 2021 some more. | We are a starting lab therefore our numbers are not representative<br>In 2020 and begin 2021 only a few after accreditation in september 2021 some more. | Oils & fats | Yes | Vegetable and animal oils, fats and fatty acids | No | |
| L15 | commercial lab; other | < 10 | ~ 200 | packaging material (polymer, P&B) | Yes | edible oil, chocolate, dry & wet food, fatty food like milk powder, polymers, Tenax, | No | |
| L16 | industry lab | about 200 samples | about 500 analyses | extra virgin olive oil | No | | No | |
| L17 | industry lab | Aprox 1000 | Aprox 1000 | Olive Oil | No | | No | |
| L18 | Official control laboratory (OCL) | 24 | 90 | dry, low-fat contant (e.g. bread) as well as products with higher fat contant (chocolate products) or edible oils | Yes | different matrices (dry, low fat; high fat; edible oils), paper-based packaging material | No | |
| L19 | industry lab | 800 | 2000 | edible oils and fats | Yes | edible oils and fats | Yes | Yes |
| L20 | other | 2020:262<br>2021: 265 | 2021:265<br>2022 (Jan-Oct): 437 | Olive oil, olive pomace oil, sunflower oil, eventually other edible oils. | No | | No | |
| L21 | commercial lab | more than 1000 | more than 2500 | Food | Yes | Food | Yes | Yes |
| L22 | industry lab | 60 | 200 | Food samples | No | | No | |
| L23 | Official control laboratory (OCL) | 70 | 330 | Oil/Fat, Products with a fat content higher than 20% | Yes | Oil | Yes | Yes |
| L25 | other | 1100 samples | 1100 samples | oils and fats | Yes | vegetable and animal oils and fats | No | |

| I. Lab code | IV. Please select what is relevant for your laboratory: | A. How many edible oils@fats samples did you analyse for MOSH/ MOAH during the 2021 and 2022? | B. How many MOSH/MOAH analyses in general did you perform in 2021 and in 2022? | C. What type of matrices were the majority of the samples? | D. Are you accredited for MOSH/MOAH analyses? | E. In which samples/matrices? | F. Did you participate in the 2022 trial for MV of the MOSH/ MOAH in edible oils and fats | G. Did you follow exactly the SOP? |
|---|---|---|---|---|---|---|---|---|
| L26 | industry lab | 100 | 150 | Oils, infant formula | No | | Yes | Yes |
| L27 | commercial lab | 2021: 5000 approx samples edible oile and fatt, 2022: 5000 until now. | 2021: 10´750 Analyses, 2022: 10´200 until now | edible fatt and oils, chocolate, dry food, | Yes | fatt/oil, chocolate, reis, packaging, cosmetic | Yes | Yes |
| L29 | industry lab | 487 | 2514 | food matrices | Yes | Cereals, olive oil and paper | Yes | Yes |
| L30 | industry lab | 2021 - 2055 samples 2021 - 900 samples | 2021 - 4110 2022 - 1800 | Palm Oil | No | | Yes | minor deviations |
| L31 | university lab | 10 | 10 | Pellets of polymers, oils | No | | No | |
| L32 | National Reference Laboratory for process contaminants or FCM (NRL); Official control laboratory (OCL) | 40 | 250 | food (dry, oils/fats, IF, chocolates)paperboard/cardboard | No | | No | |
| L33 | commercial lab | ~ 1000 | ~7500 | tea, herbs, spices, oils | Yes | food in general | No | |
| L35 | commercial lab | more than 10000 | more than 40000 | oils, spices, bakery products, meat, tea, pasta, rice, milk powder | Yes | all food and feed matrices | Yes | Yes |
| L37 | university lab | 2 | 7 | paper and cardboard | No | | No | |
| L39 | commercial lab | 500 | 3500 | cocoa | Yes | cocoa, fat, FCM, migration solution | Yes | Yes |
| L40 | commercial lab | 0 | >1500 | Migration | Yes | carton, paperboard, Tenax- and Ethanol-migration solutions, care products and raw material | No | |

| I. Lab code | IV. Please select what is relevant for your laboratory: | A. How many edible oils@fats samples did you analyse for MOSH/MOAH during the 2021 and 2022? | B. How many MOSH/MOAH analyses in general did you perform in 2021 and in 2022? | C. What type of matrices were the majority of the samples? | D. Are you accredited for MOSH/MOAH analyses? | E. In which samples/matrices? | F. Did you participate in the 2022 collaborative trial for MV of the MOSH/MOAH in edible oils and fats | G. Did you follow exactly the SOP undergoing validation in that study? |
|---|---|---|---|---|---|---|---|---|
| L42 | Official control laboratory (OCL) | about 200 | about 500 | edible oils and milk powder | Yes | edible oil, food and food cantact material | Yes | Yes |
| L43 | industry lab | 2000 | 2000 | Edible oil | No | | Yes | Yes |
| L44 | industry lab | 1000+ | 500+ | vegetable oil | No | | Yes | Yes |
| L46 | commercial lab | ~3500 | ~6500 | Vegetable oils | Yes | Vegetable oils and foodstuff on basis of vegetable oils<br><br>Packaging materials, food and feed and feedingstuffs (low fat content) | Yes | Yes |
| L48 | industry lab | about 300 | about 300 | edible oil (soft seed oil and tropical oil) | No | | Yes | Yes |

| I. Lab code | H. Please describe these minor deviations | 1.What is the aliquot taken from samples A and B for the analyses? | 2. What is the volume and the composition of the hexane/ethanol mixture used to prepare the initial solutions for test items A and B? | 3. Did you use the same solution for further MOSH and for MOAH analyses? | 4. What was the volume taken for MOSH and for MOAH analyses? | 5. Did you perform saponification (SAPO)? | Please describe "other" |
|---|---|---|---|---|---|---|---|
| L02 | | 0,2 g | The sample was diluted in 1,7 ml hexane | Yes | After the epoxidation app. 1 ml (upper layer) has been collected and inserted into the AgNO3 enriched column. For the further MOSH analysis the received eluent has been concentrated to 2 ml in rota vapor. | No | No saponification applied |
| L03 | | 5 | We use 100 ml of ethanolic KOH (130 g/L) for saponification and 35 ml of Hexane for extraction | Yes | 35 ml of Hexane and concentrated to 0,75 ml | Yes, for MOSH and for MOAH together in one aliquot | - |
| L07 | | 0.3g | n.a. | Yes | n.a. | No | n.a. |
| L09 | | 1g | 1g mixed with 2mL hexane, | Yes | all of it. | Other | (1g oil + 2mL hexane), was put through a column with 12g of silica, eluted with 45mL of 20:DCM:Hexane and eluate evaporated to ca 1mL. |
| L12 | | 2.5 g. | 1 ml hexane, 0 ml ethanol. | Yes | 1 ml. | Yes, for MOSH and for MOAH together in one aliquot | - |
| L13 | | / | / | No | 0.5 g - MOSH, 1 g - MOAH | No | / |
| L14 | | 2 grams | 20 ml (10 ml hexane / 10 ml ethanol) | Yes | Question not clear. We used; injection: 50 µl reagent: 16 ml hexane, 11 ml water and 15 ml ethanol and 6 ml KOH in water | Yes, for MOSH and for MOAH together in one aliquot | NA |

| I. Lab code | H. Please describe these minor deviations | 1.What is the aliquot taken from samples A and B for the analyses? | 2. What is the volume and the composition of the hexane/ethanol mixture used to prepare the initial solutions for test items A and B? | 3. Did you use the same solution for further MOSH and for MOAH analyses? | 4. What was the volume taken for MOSH and for MOAH analyses? | 5. Did you perform saponification (SAPO)? | Please describe "other" |
|---|---|---|---|---|---|---|---|
| L15 | | 300 mg | 0.7 mL hexane<br>Further solutions during online epoxidation:<br>0.5 mL ethanol (including mCPBA) + 1 mL ethanol for the reaction with Na2S2O3/Na2CO3 solution | Yes | As their was an online epoxidation process the whole volume was taken. | No | - |
| L16 | | 0.3 grams | . | No | 15 mL of n-hexane for MOSH fraction and 22 mL of n-hexane / dichloromethane 75/25 | No | . |
| L17 | | 0.11-0.14 g | 0.280 ml only Hexane, after that we add 0.400 ml EtOH and 0.900 ml H2O in the epoxidation step | Yes | 0.050 ml (injected with syringe to the HPLC System) | No | n/a |
| L18 | | 2.5 g | 20 ml (hexan/ethanol 1:1, v/v) | Yes | MOAH: 10 ml, MOSH: hexane volume left after removal of 10 ml for MOAH analysis (roughly 8 ml) | Yes, for MOSH and for MOAH together in one aliquot | - |
| L19 | | | | | | | |
| L20 | | 0.3 g | There were not initial solutions. Samples are epoxidized directly after adding 1.5 mL n-hexane. | Yes | All of it. | No | Not relevant. |
| L21 | | | | | | | |
| L22 | | 1 g | 10 mL hexane/ethanol 1:1, v/v | Yes | na | Yes, for MOSH and for MOAH together in one aliquot | na |
| L23 | | | | | | | |

| I. Lab code | H. Please describe these minor deviations | 1.What is the aliquot taken from samples A and B for the analyses? | 2. What is the volume and the composition of the hexane/ethanol mixture used to prepare the initial solutions for test items A and B? | 3. Did you use the same solution for further MOSH and for MOAH analyses? | 4. What was the volume taken for MOSH and for MOAH analyses? | 5. Did you perform saponification (SAPO)? | Please describe "other" |
|---|---|---|---|---|---|---|---|
| L25 | | for MOSH analysis : 300 mg - for MOAH analysis : 1 g | 10 ml hexane/ethanol 50:50 for MOAH analysis | No | for MOAH analysis : 1 g of oil + 10 ml hexane/ethanol 50:50 for MOSH analysis : 300 mg oil +600 µl hexane | Other | no saponification for MOSH content (content higher than 10 mg/kg) saponification for MOAH content |
| L26 | | | | | | | |
| L27 | | | | | | | |
| L29 | | | | | | | |
| L30 | 1. Saponification by water bath sonicator for 30 minutes at 60-degree celcius.<br><br>2. Modification of alox clean up procedure. Doubled up the aluminium oxide (20g), silica gel (6g) and sodium sulphate (2g).<br><br>3. Increased elution with n-hexane to 50 ml to facilitate removal of filtrate through the double amount of aluminium oxide, silica gel and sodium sulphate. This was followed with a complete evaporation step before reconstituting the residue with 1 ml n-hexane. | | | | | | |

| I. Lab code | H. Please describe these minor deviations | 1.What is the aliquot taken from samples A and B for the analyses? | 2. What is the volume and the composition of the hexane/ethanol mixture used to prepare the initial solutions for test items A and B? | 3. Did you use the same solution for further MOSH and for MOAH analyses? | 4. What was the volume taken for MOSH and for MOAH analyses? | 5. Did you perform saponification (SAPO)? | Please describe "other" |
|---|---|---|---|---|---|---|---|
| L31 | | 0,2 g | 1 g of pure hexane | No | MOSH was eluted with 6 mL and reconcentrated to 0,5 mL<br><br>MOAH was eluted with 12 mL and concentrated to 0,5 mL | No | not applied |
| L32 | | 100mg (MOSH), 200mg (MOAH) | 1mL (isohexane) | No | 1mL | No | no sapo |
| L33 | | 2 g | 10 mL hexane (100%) | Yes | 10 mL | No | - |
| L35 | | | | | | | |
| L37 | | 1 g (from each ampoule) | 5 ml of hexane | Yes | 1 ml | No | - |
| L39 | | | | | | | |
| L40 | | 3g | 30 ml Hexane/Ethanol (50/50) | Yes | 10 ml | Yes, for MOSH and for MOAH together in one aliquot | for MOSH and for MOAH together in one aliquot (see above) |
| L42 | | | | | | | |
| L43 | | | | | | | |
| L44 | | | | | | | |
| L46 | | | | | | | |
| L48 | | | | | | | |

| I. Lab code | 6. What was the composition of the saponifying mixture - KOH concentration and volume added? | 7. What were the conditions for the saponification - time and temperature? | 8. Did you perform second extraction? | 9. Did you perform aluminum column clean up (ALOX) for MOSH fraction? | 10. Did you perform silica gel column clean-up for MOAH fraction? | 11. Did you perform epoxidation (EPOX)? | 12. What epoxidation agent did you apply? | 13. What device did you use for reducing the volumes (evaporation of the solvent)? | 14. Did you use a keeper? Which one? When you introduce it? | 15. Final volume of the extract before injection in ul, approx? |
|---|---|---|---|---|---|---|---|---|---|---|
| L02 | No saponification applied | No saponifi-cation applied | No | Yes, | No | Yes | mCPBA in ethanol | Rotary perforator with vacuum | Toluene | 300 ul |
| L03 | We use 100 ml of ethanolic KOH (130 g/L) for saponification and 35 ml of Hexane for extraction | 30 minutes at 80 °C | Yes, for both MOSH and MOAH fractions | Yes, | Yes | Yes | other | N2 (nitrogen) | Yes. Bis (2-ethylesil-maleate). After the clean-up. before concentration. | 750 |
| L07 | n.a. | n.a. | No | No | Yes | Yes | mCPBA in ethanol | n.a. | n.a. | 1 ml |
| L09 | NA | NA | No | No | Yes | Yes | mCPBA in ethanol | Syncore evaporator. | No keeper required as system maintains residual volume of 1mL | 1.5mL |
| L12 | 50 ml 3 M KOH in ethanol/water 1:1. | 16 h at room temperature. | No | No | Yes | Yes | other | Rotary vap after SAPO, nitrogen flow after column clean-up. | 300 ul isooctane for MOSH manual separation. | 1000 ul. |
| L13 | / | / | No | Yes, | Yes | Yes | mCPBA in ethanol | Polyvap | yes, Bis(2-ethylhexyl) maleate before concentrating the sample with the polyvap. | 1000 |
| L14 | Concentration: 500 g/l, Volume added: 6 ml | 30 minutes @ 70°C | Yes, only for MOAH fraction | No | Yes | Yes | mCPBA in ethanol | Chronect LCGC-box | No | 3000 µl (3 ml) |
| L15 | - | - | No | No | No | Yes | mCPBA in ethanol | not used | - | ~ 800 µL |
| L16 | . | . | No | No | No | No | other | vacuum at 35°C | no | 500 microL |
| L17 | n/a | n/a | No | Yes, | Yes | Yes | mCPBA in ethanol | We don't reduce the solvent | No | 0.280 ml |

| I. Lab code | 6. What was the composition of the saponifying mixture - KOH concentration and volume added? | 7. What were the conditions for the saponification - time and temperature? | 8. Did you perform second extraction? | 9. Did you perform aluminum column clean up (ALOX) for MOSH fraction? | 10. Did you perform silica gel column clean-up for MOAH fraction? | 11. Did you perform epoxidation (EPOX)? | 12. What epoxidation agent did you apply? | 13. What device did you use for reducing the volumes (evaporation of the solvent)? | 14. Did you use a keeper? Which one? When you introduce it? | 15. What was the final volume of the extract before injection in ul, approx? |
|---|---|---|---|---|---|---|---|---|---|---|
| L18 | 50 %, 6 ml | 45 min, 70 °C | No | Yes, | Yes | Yes | mCPBA in ethanol | parallel evaporator (Multivapor P-12, Büchi) | yes, Bis(2-ethylhexyl)maleat, 2-3 drops, directly before evaporation | 1.5 ml |
| L19 | | | | | | | | | | |
| L20 | I did not do saponification. | I did not do saponification. | No | Yes, | Yes | Yes | mCPBA in ethanol | I did not reduce volumes. | No, I did not. | 2000 |
| L21 | | | | | | | | | | |
| L22 | 3 mL KOH 0.33 g/mL | 60 ºC, 30 min | No | Yes, | Yes | Yes | mCPBA in ethanol | Buchi, vacum system | yes, | 100 per fraction (MOSH / MOAH) |
| L23 | | | | | | | | | | |
| L25 | KOH 33 g/100 g in water | 30 min at 60 °C | Yes, only for MOAH fraction | No | Yes | Yes | mCPBA in ethanol | Rotavapor for MOAH determination<br><br>No evaporation for MOSH determination | no keeper | 1000 µl |
| L26 | | | | | | | | | | |
| L27 | | | | | | | | | | |
| L29 | | | | | | | | | | |
| L30 | | | | | | | | | | |
| L31 | not appplied | not applied | No | No | Yes | No | other | N2 streamer | no | 500 uL |
| L32 | no sapo | no sapo | No | Yes, | No | Yes | mCPBA in ethanol | rotavapor + N2 | toluene (added before concentration by evaporation) | 200µL (MOSH), 400µL (MOAH) |

| I. Lab code | 6. What was the composition of the saponifying mixture - KOH concentration and volume added? | 7. What were the conditions for the saponification - time and temperature? | 8. Did you perform second extraction? | 9. Did you perform aluminum column clean up (ALOX) for MOSH fraction? | 10. Did you perform silica gel column clean-up for MOAH fraction? | 11. Did you perform epoxidation (EPOX)? | 12. What epoxidation agent did you apply? | 13. What device did you use for reducing the volumes (evaporation of the solvent)? | 14. Did you use a keeper? Which one? When you introduce it? | |
|---|---|---|---|---|---|---|---|---|---|---|
| L33 | - | - | No | No | No | Yes | mCPBA in ethanol | no evaporation | - | 1000 µl |
| L35 | | | | | | | | | | |
| L37 | - | - | No | No | Yes | No | other | vacuum rotary evap. and gentle stream of N | toluene for MOAH fraction (during SPE clean-up) | 250 |
| L39 | | | | | | | | | | |
| L40 | 3 ml KOH (50g KOH in 100ml water) | 30 min/60°C | No | Yes, | Yes | Yes | mCPBA in ethanol | Rotary Evaporator | Yes, Bis(2-ethylhexyl)maleat was added before evaporation. | |
| L42 | | | | | | | | | | |
| L43 | | | | | | | | | | |
| L44 | | | | | | | | | | |
| L46 | | | | | | | | | | |
| L48 | | | | | | | | | | |

| I. Lab code | 16. How many ul did you inject? | 17. Please specify the instrumentation used – on-line system brands. Does it incorporate automation for some of the sample preparation steps? Which ones? | 18. GC column? | 19. FID temperature? | 20. Please specify the software used for integration; Is an option to subtract the reagent blank chromatogram from the sample chromatogram and visualise the resulting chromatogram available? | 21. Did you dilute test item C before the injection? | 22. What was the ratio C50:C20 | 23. Were all verification standards within the limits prescribed in the Guidance document on MOSH/MOAH? Please mention those that deviate? |
|---|---|---|---|---|---|---|---|---|
| L02 | 90 | No automatisation. We applied the manual method | Optima 1, | 240 | ChromQuest | Yes | Not tested | Not checked |
| L03 | 50 | LC-GC-FID Brechbuehler. No automation. | ZB-1 capillary column (15 m x 0.25 mm i.d., 0.25 µm film thickness) | 360 °C | Chromeleon. Yes, it is an option. | No | 0.58 | Yes. |
| L07 | 50 | Automated online LC-GC-FID | MXT-1 Crossbond | 350°C | Clarity; substraction would be performed outside the software | Yes | 0.95 | yes |
| L09 | 100 | LC-GC consisting of Thermo U3000 HPLC, Brechbuhler switching valves and 1310 GC-FID with heated SVE, | MXT Siltek 10m x 0.53 mmID Guard Column and MXT separation column 15m x 0.25mmID x 0.25uM | 380 deg C | Chromeleon. No subtraction. Subtraction possible using file transfer to MS Excel. | Yes | 1:3.1 (Restek 31076 Retention time standard) | 2MN/1MN 2MN/TBB 5B/TBB CyCy/TBB CyCy/C13<br>1.002 0.972 0.920 0.814 1.722<br>All agreed within +/-5% |
| L12 | 5 ul. | GC-FID with offline manual separation. | MEGA-PS255, 15mx0.25mmx0.15um. | 350°C. | Agilent OpenLAB CDS ChemStation Edition Rev. C.01.05. | No | 1.07. | C7-C10 lost in solvent signal. Cholestane coeluted with C28. Part of volatiles were lost as indicated by C11 and 5Bz values and target ratios. |
| L14 | 50 µl | HPLC-GC-FID-PAL | Analytical column: MXT-1 15 m x 0,25 mm x 0,25 µm Guard column: Guard MXT, 10 m x 0,53 mm | 350°C | Chrolibri version 1.2.2.2 | Yes | 80% | Yes |
| L15 | 50 µL | LC-GC-FID by Axel Semrau | Rxi-5Sil MS, 15m×0.25mmID×0.25 µm, RESTEK GmbH | 350°C | Clarity ba Axel Semrau | Yes | 1.15 | yes |
| L13 | 50 µl for MOSH and 100 µl for MOAH | Axel Semrau - Agilent with automatic epoxidation | MXT-1 (Crossbond 100% dimethyl polysiloxane) 15m, 0.25 mmID, 0.1 µm df; precolumn: MXT siltek guard column 10 m, 0.53 mmID. | 380°C | Chrolibri, Yes. | Yes | / | / |

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|---|---|---|---|---|---|---|---|---|
| L16 | 1.5 microL | GC-FID off line method | High temperature DB-5HT 15M, 0.32MM, 0.10U from Agilent | 365°C | OpenLab | No | . | . |
| L17 | 50 uL | HPLC-GC-FID Thermo-Fisher with autosampler CTC model TriPlus. All the steps are automatizaded by the CombiPal | MXT-1 15x0.25x0.1 Restek | 380 | Chromaleon 7.3, ThermoFisher Instruments | No | 0.53 The ratio to C20 are between 0.8-1,1 but C50:C20 always is lower | Verification MOSH, C11, C13, Cholestane are all between 80-120% relative to CyCy Verification MOAH Perylene, 1MN, TBB. 5B are all between 80-120% relative to 2MN |
| L18 | 80 µl | Scientific Instruments Manufacturer GmbH (SIM): Agilent HPLC 1260 Infinity System, Agilent GC 7890B, CHRONECT LC-GC interface, CHRONECT Robotic PAL RTC autosampler | Restek MXT-1: 15 m x 0,25 mm x 0,1 µm together with Restek MXT Siltek Guard Column: 10 m x 0,53 mm | 380 °C | ChemStation Software (OpenLAB), no reagent blank subtraction in the software possible | Yes | 83 % (MOSH); 89 % (MOAH) | Areas for pentylbenzene were low in olive oil samples (54-70% of TBB-area) |
| L19 | | | | | | Yes | 0.90 | Yes |
| L20 | 100 | On-line Agilent HPLC-GC-FID with Gerstel robot | MXT-1, 0.25 microm x 15 m x 0.25 mmI D | 380 °C | Gerstel Enterprise MOSH-MOAH version 2 | No | 0.6 | Perylene and cholestane deviated. |
| L21 | | | | | | Yes | 0.604 | no deviation |
| L22 | 15 | GCxGC-FID (Agilent GC and Leco cryo-modulator) | mid polar - non polar | 370 | ChromaTOF | Yes | 0.987 | Yes |
| L23 | | | | | | Yes | 0.7 : 1 | The difference between TBB and 2 MN was bigger than normal. |
| L25 | 50 µl | Axel-Semrau equipment | Resteck MXT1 (15m - 0.25mm - 0.1µm) | 350°C | clarity | Yes | 0.90 | yes |
| L26 | | | | | | Yes | 80% | Yes |

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|---|---|---|---|---|---|---|---|---|
| L27 | | | | | | No | 0,85 | yes |
| L29 | | | | | | Yes | 0,8 | ratio between the Internal standard:<br><br>C11/CyCy - C13/CyCy - Cho/CyCy (Cho interfered and overestimated > 200%)<br><br>5B/2MN - 1MN/2MN TTBB72MN - Per/2-MN (note < 200%) |
| L30 | | | | | | No | 1.0 | Yes |
| L31 | 50 uL | GC FID from agilent, no automation for sample preparation steps | DB5 | 280 C | GC 8860 Data Analysyus | No | 0,2 | yes |
| L32 | 40µL (GC-FID) | manual method + GC-FID (Trace 1310 Thermofisher) | TG-1MT (15m x 0.25mm, 100% PDMS, 0.25µm) | 350°C | Chromeleon (7.2) (blank subtraction available) | Yes | not measured | ratio TBB/1-MN and 5B/1-MN under specifications when strong epoxidation is performed |
| L33 | 90 µL | Agilent online system with automated epoxidation | Restek MXT-1 | 380 °C | Chrolibri | Yes | 0.80 | yes |
| L35 | | | | | | Yes | 0,8:1 | yes |
| L37 | 15 | SPE fractionation was done offline, GC-FID was Agilent 7890B | DB-1HT (15 m x 0.25 mm x 0.1 µm) | 360 °C | ChromaTOF for BT | Yes | not analysed | - |
| L39 | | | | | | No | 1 | yes |
| L40 | 50 µl | online LC-GC-FID, it does not include automation of preparation steps. | MXT-1 | 370°C | Clarity, no | Yes | 0.8 | yes |

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|---|---|---|---|---|---|---|---|---|
| L42 | | | | | | Yes | In MOSH channel the ratio C50:C20 is 0.88, In MOAH channel the ratio C50:C20 is 0.90 | Yes, all operations were followed the Guidance document, no deviate. |
| L43 | | | | | | Yes | 1.03 | Yes |
| L44 | | | | | | Yes | 82% | No |
| L46 | | | | | | Yes | Ratio C20/C40 was 92.4 % | Yes, but in our internal standard mixture we have replaced perylen with pyrene |
| L48 | | | | | | No | 0.81 | except Perylene |

| I. Lab code | 24. Did you perform background compensation? | 25. How did you perform background compensation ? Please describe | 26. total MOAH (TBB) content in your reagent blank (mg MOAH/kg edible oil) - what you have subtracted from the sample A and sample B | 27. total MOAH (2MN) content in your reagent blank - what you have subtracted from the sample A and sample B | 28. total MOSH content in your reagent blank (mg MOSH/kg edible oil) - what you have subtracted from the sample A and sample B |
|---|---|---|---|---|---|
| L02 | Yes | By subtracting all sharp peaks´area | 293 ppm (sample A), 5 ppm (sample B) based on 1-MN | 249 ppm (sample A), 5 ppm (sample B) based on Sum 1MN+2MN | 64 ppm (sample A), 42 ppm (sample B) |
| L03 | Yes | Baseline subtraction by a blank run without solvent. | 3.6 | 4.7 | 1,7 |
| L07 | No | No substraction | No substraction | No substraction | No substracted |
| L09 | Yes | I overlayed the chromatograms of the sample with the reagent blank. I manually drew in the baseline from the intersections (beginning and end) of the reagent blank with the sample. When required I used the split peak function and baseline adjustment function to shaped the baseline, so it matched, as close to possible to the reagent blank. | No applicable. Subtraction was done graphically. | No applicable. Subtraction was done graphically. | No applicable. Subtraction was done graphically. |
| L12 | No | - | 1.33, but not substracted | [1.44, but not substracted] | 7.25 (CyCy), but not substracted] |
| L13 | No | / | / | / | / |
| L14 | Yes | With a blank procedure | 0,75 | 0,88 | 1,9 |
| L15 | Yes | A separate analysis blank was measured and the signals above the baseline were quantified and subtracted from the values of the samples. Both in the reagent blank and the sample shoulder peaks were excluded for quantification. | Usually, we subtract the amount of substances in ng (before calculation with weight of sample taken). For your purpose we calculated in approximation with the median sample weight: 4 mg/kg | Usually, we subtract the amount of substances in ng For your purpose we calculated in approx: 3,7 mg/kg | Usually, we subtract the amount of substances in ng (before calculation with weight of sample taken). For your purpose we calculated in approximation with the median sample weight: 5,2 mg/kg |
| L16 | Yes | Instrumental compensation | . | . | . |
| L17 | Yes | The MOSH MOAH standard solution injected everyday in Hexane for verfication is used as blank solvent for background compesation of the injected samples. | n/a | 0.60 | 0.76 |
| L18 | Yes | Baseline of an hexane blank was used as baseline for integration of all samples.<br>Amount in reagent blank was determined. Amount in samples was determined followed by substraction of amount in reagent blank. | 0.17 mg/kg | 0.21 mg/kg | 0.93 mg/kg |
| L19 | Yes | Reagent blank substracted | 0.4 | 0.4 | 0.2 |
| L20 | Yes | Using the software iteration button. | 2.34 | 2.81 | 14.26 |

| I. Lab code | 24. Did you perform background compensation? | 25. How did you perform background compensation ? Please describe | 26. total MOAH (TBB) content in your reagent blank (mg MOAH/kg edible oil) - what you have subtracted from the sample A and sample B | 27. total MOAH (2MN) content in your reagent blank (mg MOAH/kg edible oil) - what you have subtracted from the sample A and sample B | 28. total MOSH content in your reagent blank (mg MOSH/kg edible oil) - what you have subtracted from the sample A and sample B |
|---|---|---|---|---|---|
| L21 | Yes | with procedure Blanc correction. | | | |
| L22 | Yes | Automatically by software | 2.13 | 2.34 | 3.33 |
| L23 | Yes | We substract the blind value. | 0.3 mg/kg | 0.4 mg/kg | 0.2 mg/kg |
| L25 | No | no background compensation | no subtraction - blank level : 0.33 mg/kg | no subtraction - blank level : 0.33 mg/kg | no subtraction - Blank level : 1 mg/kg |
| L26 | Yes | Integrate the area of blank and calculate the MOSH/MOAH content. The value of samples decreases the value of blank. | 0 | 0 | 0 |
| L27 | No | no | | | |
| L29 | Yes | subtraction mathematically of the blank | 0.00 | 0,00 | 0.23 |
| L30 | No | - | 0.33 mg/kg | 0.24 mg/kg | 0.27 mg/kg |
| L31 | Yes | Subtraction of blank signal | not quantified | not quantified | not quantified |
| L32 | Yes | subtraction of solvent blank with integration software | not subtracted | not subtracted | not subtracted |
| L33 | No | - | | | |
| L35 | No | No compensation performed | | | |
| L37 | Yes | by blank substraction | | | |
| L39 | Yes | blank sample was used | | | |
| L40 | No | We did no background compensation. | | | |
| L42 | No | the reagent blank was low, so we didn't perform background compensation. | 0.00 | 0.00 | 0.12 |
| L43 | No | No background compensation. | | | |
| L44 | No | We will control that the background values of reagents etc. do not interfere with the analysis of MOSH and MOAH. | | | |
| L46 | Yes | Blank signals substracted using Chrolibri software when necessary. With epoxydation (contamination from mCPBA) | 2.0 mg/kg | 2.1 mg/kg | No substraction for MOSH |
| L48 | No | N/A | | | |

| I. Lab code | 29. Did you encounter any problems during the sample preparation, please describe? | 30. Did you encounter problems during the integration and the interpretation of the results | 31. Any other comments? |
|---|---|---|---|
| L02 | The absolute recovery rate of the standards was very low. Where the estimation of 2 ppm of mineral oils in sample should have been achievable, when using the AgNO3/Silica column the absolute recovery was extremely low making it impossible to approach the wished LOQ. | We were not sure whether a peak should be considered as a sharp one or not. There were some broad peaks with other peaks on them and we didn;t know how to subtract the areas. | Our method is not fit for purpose as tested to achieve the proposed maximum permitted level for MOAH for olive oil. We are sorry but we could not present the chromatograms as requested. |
| L03 | No | No | - |
| L07 | No problems | No problems | You require no information on LOQ's? Ours is 2 mg/kg. |
| L09 | No problems. | No. | 12g of silica is quite a large volume and presumably it can't be regenerated and re-used. |
| L12 | - | - | The analytical column previously used for the analysis of MOSH content has been changed for a new one. Sample C not analyzed. |
| L13 | No. | In test Item C the internal standards were not baseline separated because the hump of the mineral oil was under them. It was difficult to make the right integration of the internal standards, especially for cholestane and perylene. | |
| L14 | No | No | No |
| L15 | no | no | |
| L16 | No | No | . |
| L17 | No | The concentrations found in the samples of this ring test were high enough so that we did not have any problem.<br>When concentrations found, specially for MOAH, are below 2 mg/kg, our results are higher than that offered by external labs. | |
| L18 | no problems discovered | no problems discovered | - |
| L19 | Sample C was too concentrated, for this reason we perfomed the analysis different times | Due to the high concentration of the samples A and C, the internal standards were hardly integrated properly | In our opinion TBB is the most reliable and repeatable internal standard to quantify the MOAH content.<br><br>Epoxidation was performed also for the sample C in order to have the same conditions of the other samples. |

| I. Lab code | 29. Did you encounter any problems during the sample preparation, please describe? | 30. Did you encounter problems during the integration and the interpretation of the results | 31. Any other comments? |
|---|---|---|---|
| L20 | Sample preparation is made by the system itself. | Yes. By looking at the chromatograms of both, sample and blank, I cannot understand how it is possible to have such high values for the C10-C16 cut in some of the samples. | The software developer explained to me that the apparent MOSH-MOAH concentration in my blanks were not actually due to the MOH presence, but to the normal behaviour of the electric system. I do not know what to think when I see my results anyway. Maybe I should do manual baseline integration, but then I have the feeling I'm manipulating the results. |
| L21 | NO | NO | NO |
| L22 | No | No | na |
| L23 | No | We always observe a difference between 2 MN and TBB during our analysis, but in try trial it was bigger than normal. We normally use TBB as ISTD. | |
| L25 | no | no | no |
| L26 | no | no | |
| L27 | Sample C, mineral oil: with dilution 1:1000 was no Hump to see, so we measured with no diluition. | no | |
| L29 | none | none | none |
| L30 | NO | Having heavy solvent peak tailing at the MOAH chromatogram. | Test items A and B<br><br>- Using automated DIN EN16995:2017 Mod. (Modification: Saponification) and manual alox clean up for the MOSH fraction<br><br>- Using automated DIN16995:2017 Mod. (Modification: Saponification) with epoxidation purification for the MOAH fraction.<br><br>Test Item C<br><br>- Using automated DIN EN16995:2017 without saponification and without alox clean up for the MOSH fraction<br><br>-Using automated DIN16995:2017 with epoxidation purification for the MOAH fraction. |

| I. Lab code | 29. Did you encounter any problems during the sample preparation, please describe? | 30. Did you encounter problems during the integration and the interpretation of the results | 31. Any other comments? |
|---|---|---|---|
| L31 | no comments | Problem with TBB standard that was different in different replicates, and not very reproducible. Probably it has been evaporated during sample preparation. | no comments |
| L32 | no | important interferences still remaining even after strong epoxidation so that LoQ is set to 10 mg/kg (MOAH) | |
| L33 | no | no | |
| L35 | no | no | |
| L37 | no | no | |
| L39 | No problems known | No problems known | |
| L40 | none | none | |
| L42 | No | Yes, we were a little confused about the integration of solution C. The guidance document does not mention the integration method of solution C. For mineral oil products, we didn't know if we need to subtract all the sharp peaks, or just subtract the internal standard peaks. | |
| L43 | Test sample C in hexane can easily evaporate and thus might affect the concentration during the process. | Test sample C has a high concentration of MOSH and MOAH that it has carryover. So needed to repeat with lower amount of sample. | |
| L44 | No | About solution C, is it necessary to eliminate the sharp peaks above the hump when calculating the MOSH and MOAH? Because the solution C is the standard of MOH, the calculation of MOH is uncertain. | |
| L46 | N/A | N/A | Getting errors uploading .CSV files. Saying the files are too large 1 MB despite them being barely 3 kB. |
| L48 | No | TBB is on the hump. not sure will it affect the result. | |

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