European Commission

JRC TECHNICAL REPORT

Determination of MOSH/MOAH in Shell SN500* mineral oil

JRC IF 2021-03 - The third interlaboratory comparison

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Joint Research Centre

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Executive summary

In order to harmonise the determination of the mineral oil aromatic hydrocarbons (MOAH) in infant formulas (IF), a third interlaboratory comparison (ILC) JRC IF 2021-03 was organised by the Joint Research Centre (JRC) of the European Commission. This ILC was designed to (i) evaluate the consistency of the reported results for solutions mineral oil in hexane at different concentration levels; and (ii) determine the mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) composition of a specific Shell SN500* mineral oil batch. This batch was used to spike various infant formula materials for the next method validation study, planned for the beginning of 2022.

The test sample named QC10, provided to the participants, consisted of a hexane solution containing the Shell SN500* mineral oil. Participants were requested to perform four different dilutions and to quantify and report MOSH and MOAH concentrations. Thirty participants from eight European countries reported results. Six of them reported consistent results for the various dilutions, and provided proper recoveries. These results were selected for the characterisation of the MOSH and MOAH percent composition in QC10.

The following “reference” values and their expanded measurement uncertainty (k=2) were derived: MOSH % in SN500* = 60.1 ± 2.1 % and MOAH % in SN500* = 34.5 ± 1.2 %. In addition, the major MOSH and MOAH fractions in SN500* were identified.

1 Introduction

Following the Rapid Alert System for Food and Feed (RASFF) notification Nr.2019.3734 (dated 25/10/2019) [1] and the Foodwatch report (dated 24.10.2019) [2] related to the presence of mineral oil aromatic hydrocarbons (MOAH) in infant formula and follow-on formula (IF), the Directorate General for Health and Food Safety (DG SANTE) of the European Commission requested the Joint Research Centre (JRC) to provide a harmonised and validated method for the determination of MOAH mass fractions in infant formula in order to ensure comparable and reliable results.

In 2021, the JRC organised the second interlaboratory comparison JRC IF 2020-02 [3] to evaluate the draft standard operating procedure (SOP). This ILC identified the need for further optimisation of (i) the sample preparation and (ii) the integration of the chromatographic hump.

This third ILC (JRC IF 2021-03) was organised to characterise the MOSH and MOAH composition of the Shell SN500* mineral oil, used to spike the infant formula. This report presents the outcome of this exercise.

2 Scope

JRC IF 2021-03 was designed (i) to evaluate the consistency of the reported results for diluted solutions of mineral oil in hexane at different concentration levels, and (ii) to determinine the MOSH/MOAH composition of the Shell SN500* mineral oil that was used to spike various infant formula materials for the next method validation study planned in 2022.

3 Set-up of the exercise

Preparation of the QC10 solution

The mineral oil SN500* used for the preparation of the QC10 and for the spiking of the infant formula is not a routine product and does not exist commercially – it was sampled directly as a distillation fraction in an oil refinery. This was the only way to obtain a mineral oil product with the requested profile (i.e. with a large percentage of high molecular weight MOAH) since such products are not for sale anymore.

An aliquot (2.024 g) of the Shell SN500* mineral oil (high viscosity base oil, Lot number ID 878338) was accurately weighted in a beaker and dissolved in hexane. Then it was quantitatively transferred into a 1 L volumetric flask, and diluted to the mark with hexane at room temperature (20°C), to produce the QC10 stock solution. 5 mL QC10 aliquots were filled in 10 mL brown glass ampules under inert argon atmosphere. Samples were stored at room temperature.

Time frame

The JRC IF 2021-03 round was announced by e-mail and open for registration on June 7, 2021 (Annex 1). All samples were dispatched to the registered participants on July 6, 2021. The deadline for reporting of results was set to August 27, 2021. It was further extended until September 15, 2021, upon request from several laboratories.

[1] https://www.foodwatch.org/fileadmin/-DE/Themen/Mineraloel/Dokumente/Mineraloel_RASFF_BVL_30-03-2020.pdf
[2] https://www.foodwatch.org/en/news/2019/foodwatch-laboratory-tests-suspected-carcinogenic-mineral-oil-residues-in-baby-milk/
[3] Bratinova S., Robouch P., Beldi G., Senaldi C., Goncalves C., Karasek L., Valzacchi S., Conneely P., Hoekstra E., Emons H. Determination of MOAH in Infant Formula, JRC IF 2020-02 - The 2nd interlaboratory comparison, European Commission, Geel, 2021, JRC 125669 EN. https://europa.eu/!BHj8r9.

Confidentiality

The procedures used for the organisation of this ILC guarantee that the identity of the participants and the information they provided are treated as confidential. The participants in this ILC received a unique laboratory code used throughout this report.

Distribution

Each participant received:

- The SOP to be applied without any modifications;
- One ampoule with 5 ml of the QC10 solution;
- The "Instruction to participants" (Annex 2); and
- The "Confirmation of receipt" form to be sent back to the organiser after receipt of the test item (Annex 3).

Instructions to participants

Participants were asked to check whether the test item was received undamaged after transport and to report any damage, if necessary, using the "Confirmation of receipt form" (Annex 3).

Participants were requested to open the ampoule carefully and prepare four diluted test samples following the instructions presented in Table 1.

Table 1. Dilution parameters - as per instruction

Sample name	(50 µL injected) µg on column	C (mg L^{-1})	QC10 (µL)	IS (µL)	Hexane (µL)	Total volume (µL)
QC10-1	0.5	10.1	10	20	1970	2000
QC10-2	1	20.2	10	10	980	1000
QC10-3	3	60.7	30	10	960	1000
QC10-4	10	202.4	100	10	890	1000

The measurands were defined as the concentration of the "fraction cut" of the "mineral oil" in the "QC solutions" expressed in mg L^{-1}, where:

"Mineral oil"	"Fraction cuts" [4], corresponding to the retention time of n-alkanes from-to:	QC solution
MOAH	C10-C16; C16-C25; C25-C35; C35-C50; C50+(additional) C10-C50 (total)	QC-10-4 All QC
MOSH	C10-C16; C16-C20; C20-C25; C25-C35; C35-C40; C40-C50; C50+ (additional) C10-C50 (total)	QC10-4 All QC

Participants were requested to report

- the concentrations of "total MOSH" and "total MOAH" (C10-C50) expressed in mg L^{-1}, in the four diluted solutions prepared (QC10-1 to 4); and
- the concentrations of the "fraction cuts" defined in the JRC Guidelines [4] and the fraction above C50 (C50+) for MOSH and MOAH in the QC10-4 solution, expressed in mg L^{-1}.

In addition, participants were requested to provide

(i) the integrated MOSH and MOAH chromatograms for the lowest concentration level (QC10-1);

(ii) chromatograms of MOSH and MOAH for QC10-4 with the visualisation of the fraction cuts;

(iii) the chromatograms of the procedural blank and the RT mix of n-alkanes.

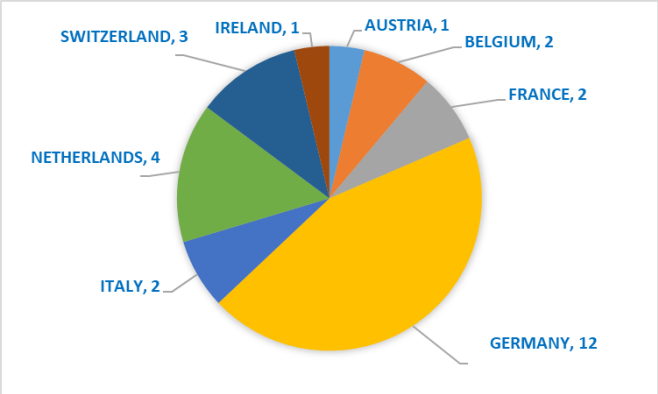
4 Results and Discussions

4.1 Results

The main objective of this ILC was to characterise the MOSH and MOAH composition in the Shell SN500* mineral oil, and to evaluate the proper operation of the on-line LC-GC-FID systems used by the participants.

Twenty-nine laboratories from 7 EU Member States and Switzerland reported the results presented in Annexes 4-7.

The laboratory performance was not assessed, since this ILC is not a proficiency test.



Despite the apparent absence of analytical challenges on the determination of MOSH and MOAH in hexane solutions (e.g. no sample preparation needed, no interferences), highly scattered results were observed for MOSH and MOAH contents up to 1 µg loaded on the column (QC10-1 and 2, see Figure 1). This range corresponds to the levels encountered when analysing commercial infant formula samples after multi-step sample preparation and would therefore result in higher standard deviations.

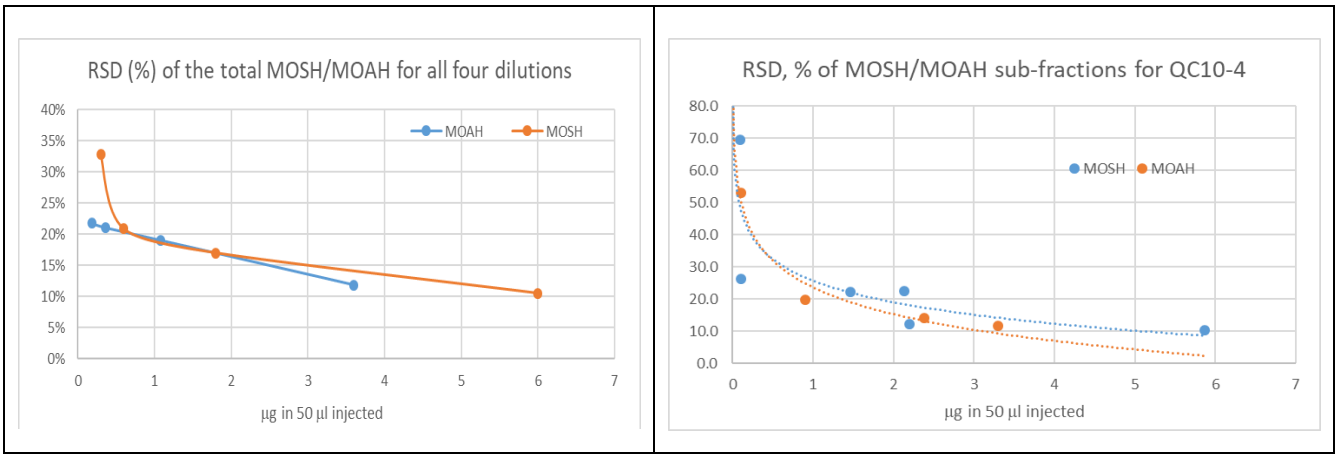


Figure 1. Relative robust standard deviation of the reported results for (a) total MOSH/MOAH (C10-C50) in the four dilutions and (b) MOSH/MOAH fractions and sub-fractions in QC10-4, as a function of the amount in µg reaching the column/detector when 50 µL of the test solution is injected in the HPLC column.

4.2 Evaluation of the instrumental performance

The results reported for MOSH and MOAH (C10-C50) in the four dilutions (QC10-1 to QC10-4) were used to calculate the percent composition of MOSH and MOAH in Shell SN500*, used for the preparation of the QC10 and for spiking of the IF (see Annexes 8-9, Figure 2).

Two main issues were observed. Some laboratories reported:

- highly scattered results for MOSH and MOAH for the four QC10 dilutions (Annex 8-9), which have the same composition and/or
- significantly low recoveries for %MOSH+%MOAH (Annex 10)

Additionally, upward or downward trends in the MOSH and MOAH content could be detected in some of the participants' datasets with the increase of the Shell SN500* concentration from QC10-1 to QC10-4 (Figure 2)

Higher recoveries at lower Shell SN500* content (lowest concentration for QC10-1) as observed by L02, L10, L11 and L28 may be due to an inappropriate hump integration, where the background and/or interfering peaks are not properly subtracted. The reverse trend showed by L03, L05, L22 (low recoveries) indicates a technical problem of the on-line LC-GC-FID system that may require maintenance. These trends were observed independently from the gas chromatography capillary columns used (metal or fused silica) or the instrument brand (Braechbuhler – Thermo or Axel Semrau) interfaced with a variety of LC and GC apparatus.

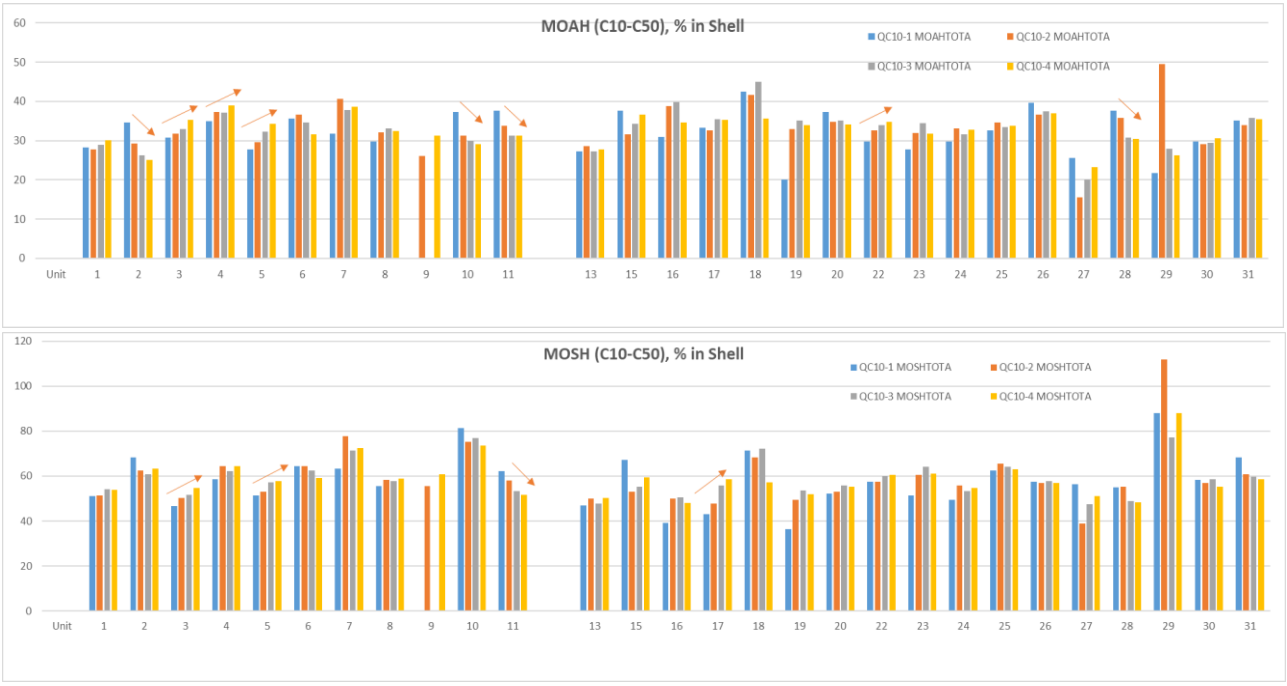


Figure 2. Calculated total MOAH (2a) and MOSH (2b) percent composition in Shell SN500* in the four diluted solutions (QC10-1 to 4), see Annex 8

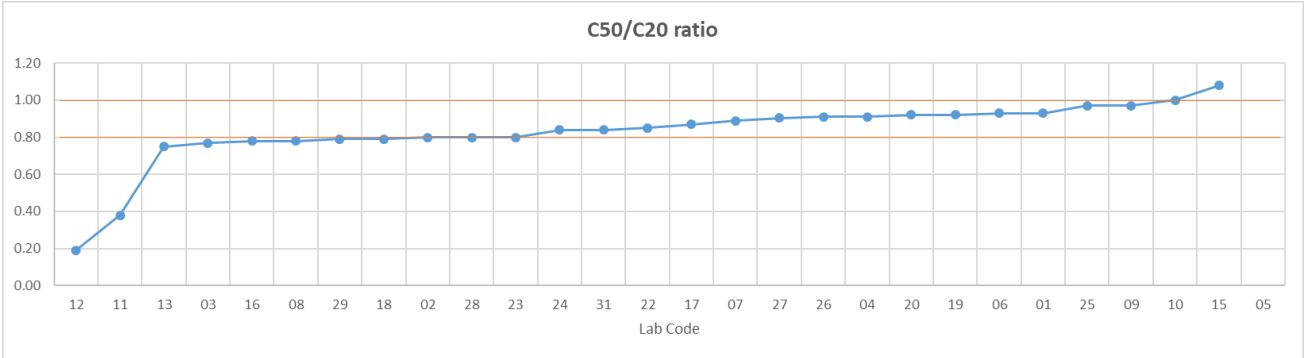


Figure 3. n-C50/n-C20 ratios reported in the questionnaire

While the Shell SN500* mineral oil and the majority of infant formula formulations contain C-fraction beyond the RT of C50 n-alkanes, the instrumental analysis beyond C50 is known to be challenging, as the absence of discrimination could not be guaranteed. An acceptance criterion assuring for proper analyses up to C50 was set for the ratio of the signals for n-C50 relative to n-C20, which should be in the range of 0.8-1.2. Though for the majority of the participants this value ranges from 0.8-1.0 (Figure 3), the variation in the ratio C50/C20 would contribute to the increased RSD in the results for the C-fractions around and above the RT of n-C50.

According to the JRC Guideline [4] the MOSH and the MOAH were requested to be reported up to the RT corresponding to the C50 n-alkane. In addition, to allow for determination of the total % MOSH+% MOAH recovery in Shell SN500*, data for the fractions with RT > C50+ were asked for QC10-4 (Annexes 6-7). Highly scattered results were reported for the C50+ fraction, which may be attributed to the variations of the C50/C20 ratio mentioned above, or to inappropriate RT calibration on the MOSH and MOAH channels. L7 and L17 reported a significant contribution of C50+ to the total MOSH and MOAH (above 15%, Annexes 6-7). All the other laboratories reported a negligible contribution of C50+ to the total (below 3 %). Therefore the reference values were established based on the MOSH/MOAH C10-C50, in compliance with the requirements set by the JRC Guidance document [4].

4.3 Characterisation of Shell SN500* for the batch used for spiking of the IF and for QC10 preparation

Two dedicated experiments were conducted in the JRC laboratory to set a target recovery range for "total MOSH" and "total MOAH". At first, an aliquot of QC10 solution was injected directly on-column in a GC-FID to quantify the total hump without MOSH and MOAH separation against the area of CyCy, and a recovery close to 100% was obtained.

The second experiment was conducted to account for polar fractions that could be eventually retained onto the HPLC column, an additional experiment was conducted using manual MOSH and MOAH fraction collection. The capillaries coming from the selection valve were disconnected from the respective columns and placed inside vials. Both the MOSH and the MOAH fractions were analysed off-line in a GC-FID confirming the recovery for the sum close to 100%. This fact confirmed the Shell SN500* oil does not contain any major polar substances. Connecting back the columns to the selection valve, maximum recovery obtained was around 96%. This recovery was supported by the results from two external expert laboratories and the value was considered as acceptable to be fixed as target recovery for % MOAH + % MOAH in Shell SN500*.

Hence, the following two assessment criteria were set for the selection of the results for characterisation:

1. The relative standard deviation (**RSD**) of the percent compositions of the %MOSH + %MOAH (C10-C50) observed for the four QC10 solutions (Figure 2 a, b) should be **smaller than 5%**;
2. The percent **recovery** of total MOSH+MOAH in QC10-4 (computed using the extended range C10-C50+) should be **between 92-100%.**

Figure 4 highlights the six laboratories (L6, L8, L22, L25, L26 and L31) that met the two criteria mentioned above. The reported concentrations for "total MOSH" and "total MOAH" (C10-C50) in QC10-1 to QC10-4 were selected to characterise the MOSH and MOAH percent compositions in the Shell SN500* batch used by the JRC. However, the integration of the chromatogram of the QC10-1 solution (lowest concentration level) was proven to be challenging by the high scatter of reported results, due to different ways of background correction and riding peak subtraction. Therefore, the QC10-1 results were not used.

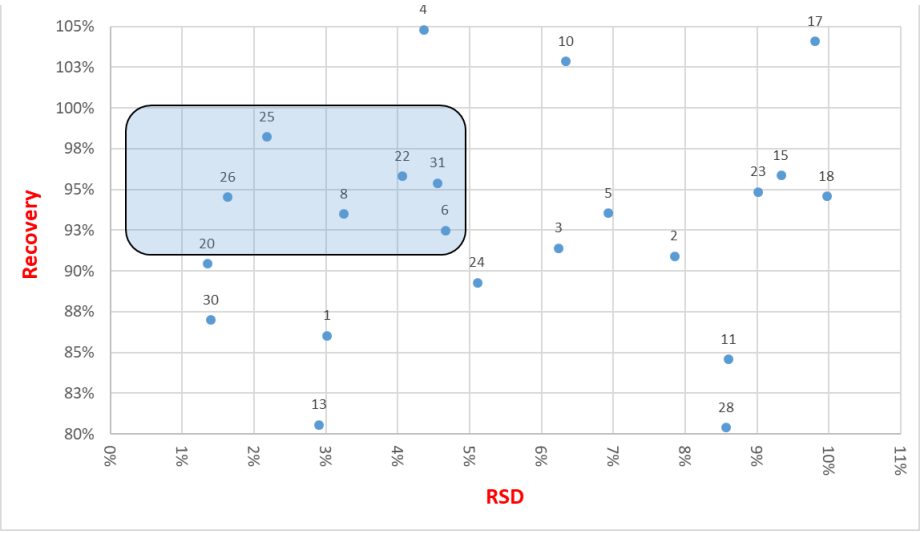
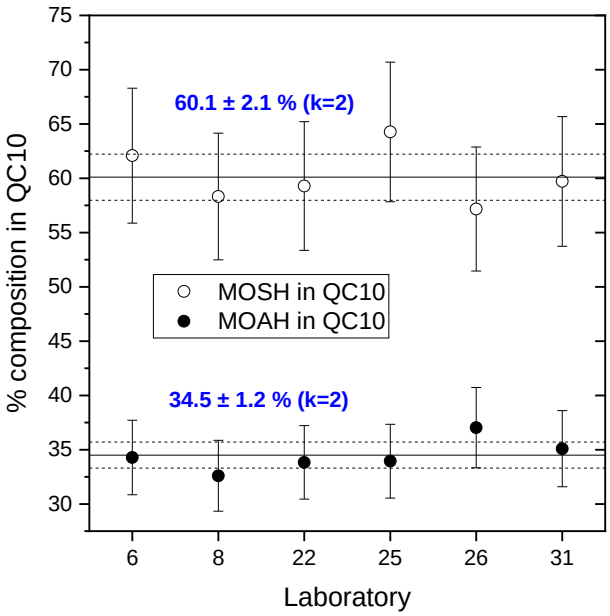


Figure 4. Recovery of MOSH/MOAH in QC10-4 v.s. the relative standard deviation (RSD) of total MOSH and MOAH (C10-C50) for QC10-1 to 4. The blue area delimits the recovery range 92-100% and RSD < 5 %.

Table 2 and Figure 5. Characterisation of the MOSH and MOAH percent composition in the Shell SN500* mineral oil used for spiking of the IF and for QC10 preparation.

MOAH %				
Lab	QC10-2	QC10-3	QC10-4	mean
6	36.63	34.60	31.62	34.28
8	32.18	33.11	32.51	32.60
22	32.67	33.94	34.88	33.83
25	34.60	33.43	33.80	33.94
26	36.63	37.40	37.06	37.03
31	33.91	35.83	35.54	35.09
		% MOAH in QC10:		**34.5 %**
			Stdev:	1.5 %

MOSH, %				
Lab	QC10-2	QC10-3	QC10-4	mean
6	64.36	62.60	59.29	62.08
8	58.42	57.66	58.89	58.32
22	57.43	59.97	60.47	59.29
25	65.54	64.28	62.98	64.27
26	56.93	57.66	56.92	57.17
31	60.69	59.77	58.68	59.71
		% MOSH in QC10:		**60.1 %**
			Stdev:	2.6 %



While the integration of MOSH and MOAH chromatograms is considered to be challenging at low levels, as in QC10-1, due to the need for background correction and riding peaks subtraction, reasonable consensus

was reached by the six laboratories selected for the total MOSH and MOAH percent composition in QC10-2 to QC10-4. Table 2 and Figure 5 present the averages and standard deviations used to derive the reference values, and the associated expanded uncertainties (k = 2) calculated as $U = 2\,s\,/\sqrt{6}$ (assuming a negligible inhomogeneity of MOSH and MOAH in the QC10 hexane solution), where "s" is the standard deviation of the means (of 6 selected labs).

The following reference percent compositions are calculated:

MOAH$_{\%}$ in SN500* = 34.5 ± 1.2 % (k=2)

MOSH$_{\%}$ in SN500* = 60.1 ± 2.1 % (k=2)

Note: These values apply only to the specific Shell SN500* batch used by the JRC for the preparation of the QC-10 solution. They do not apply to other Shell SN500* batches.

Figure 6 shows that even the six selected laboratories reported highly scattered results for the individual MOSH/MOAH fraction cuts, probably influenced by various RT calibrations. Relative standard deviations ranging from 9 % (for the MOAH C35-C50) to 56 % (MOAH C16-25) were obtained from Annexes 6-7. The "cutting position" affects directly the two neighbouring fractions, where one fraction may be overestimated to the detriment of the next one that will be underestimated. Nevertheless, the following **major fractions (contributing to more than 95 %) in the Shell SN500*** can be identified**:**

"C25-C35" and "C35-C50" for MOAH;

"C25-C35", C35-C40" and "C40-C50" for MOSH.

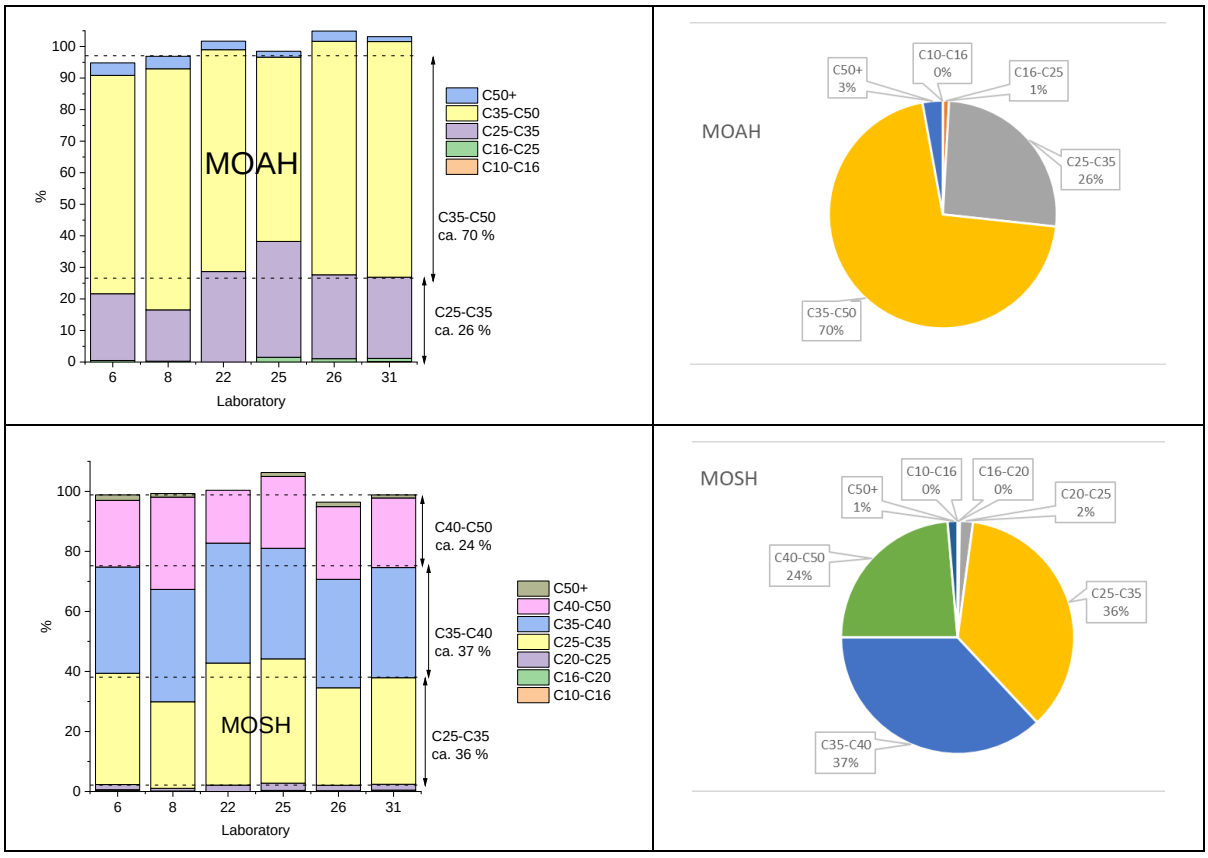


Figure 6. MOSH and MOAH fraction-cut distribution in Shell SN500*

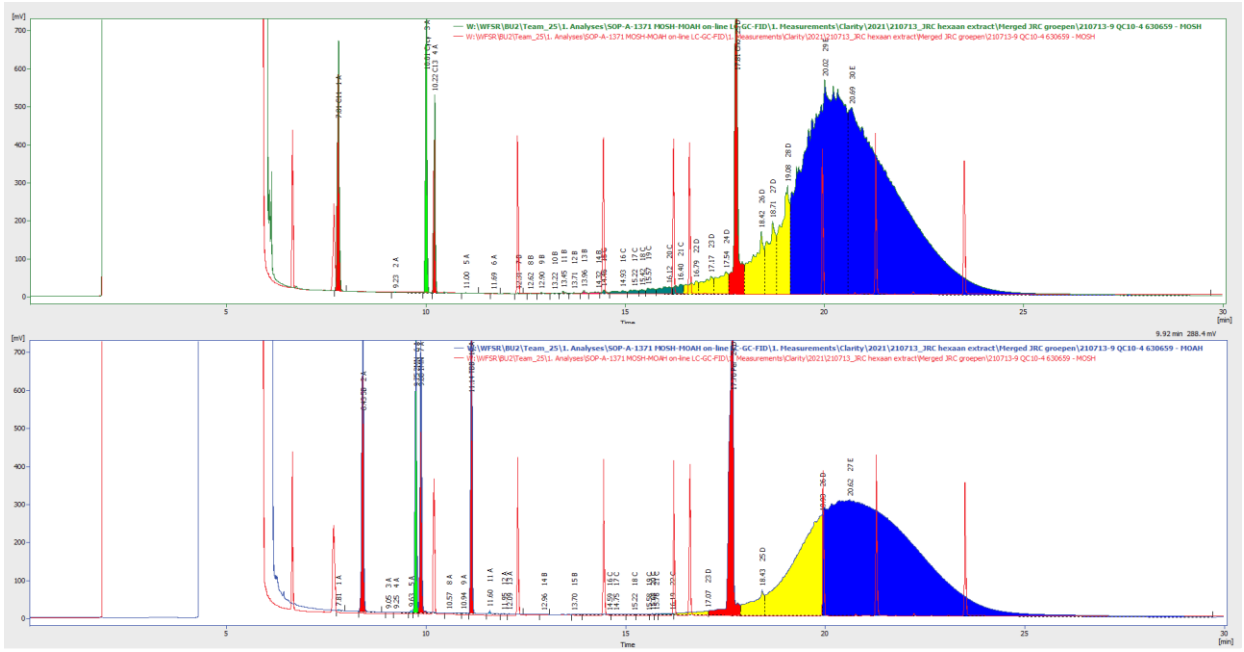
4.4 Questionnaire

Most of the laboratories (20 out 28) used the on-line LC-GC-FID instrumentation with the Axel Semrau interface and five laboratories used the Braechbuhler–Thermo equipment. Only L29 applied manual MOSH/MOAH separation, and provided chromatograms showing significant interferences due to impurities.

Some of the results reported for the C50+ fraction are not coherent with the MOSH and MOAH chromatograms provided. For example, L7 and L17 reported a contribution of ca. 10 % (Annexes 6-7), which cannot be justified by the relatively small tailing of the hump above C50 (Figure 7a). On the contrary, L15 reported a value below the detection limit for C50+, when the chromatograms display a clear tailing of the hump beyond C50 (Figure 7b). Three laboratories (L11, L27 and L30) reported a C50+ contribution below LOD without providing the corresponding chromatograms.

The MOSH and MOAH chromatograms were recorded with a two-channel LC-GC system using two different GC columns, and applying the same GC oven temperature program and the same inlet pressures. A shift in the apex of the MOSH and MOAH humps was observed in most of the chromatograms (Figures 7-8). The resulting total shift of ca. 0.5 min may be due to differences in film thickness and/or column flow rates. Hence, **dedicated calibrations of the RT windows on both channels should always be performed** with the n-alkane mixture to ensure the accurate delimitations of the fraction cuts.

7a



7b

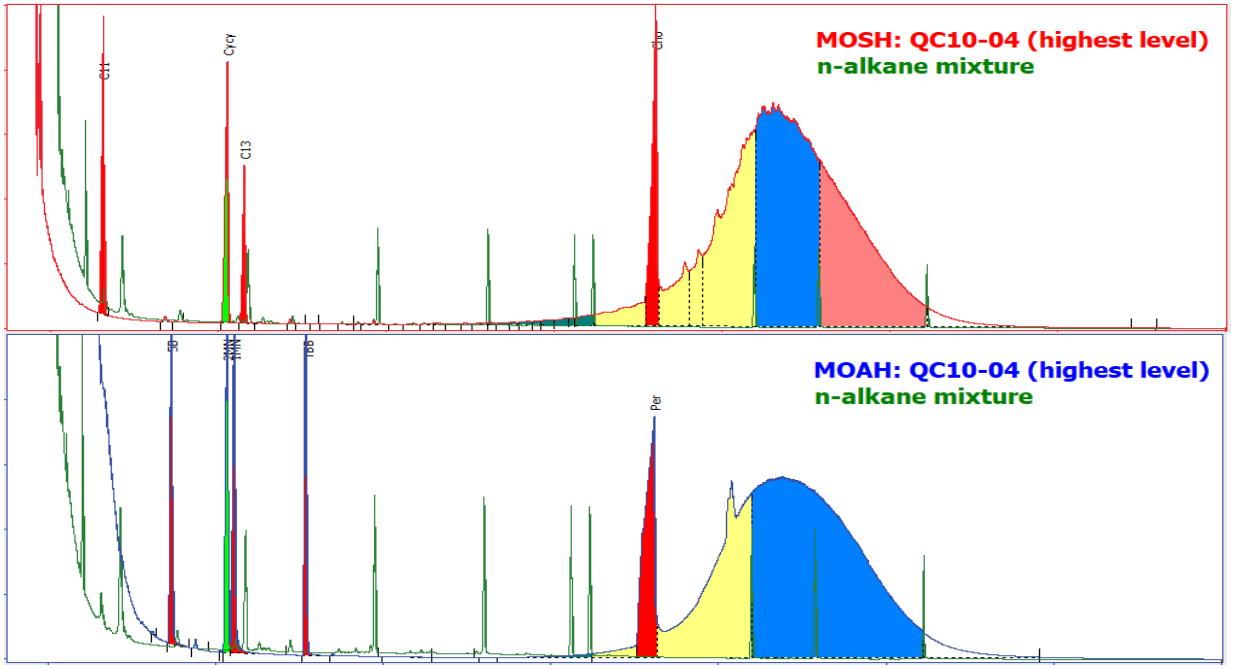


Figure 7. MOSH/MOAH chromatograms of QC10-4 overlaid with n-alkane C10-C50 mixture where (a) L17 reported MOAH-C50+ = 11.8 mg/L; (b) L15 reported MOAH-C50+ <0.1 mg/L.

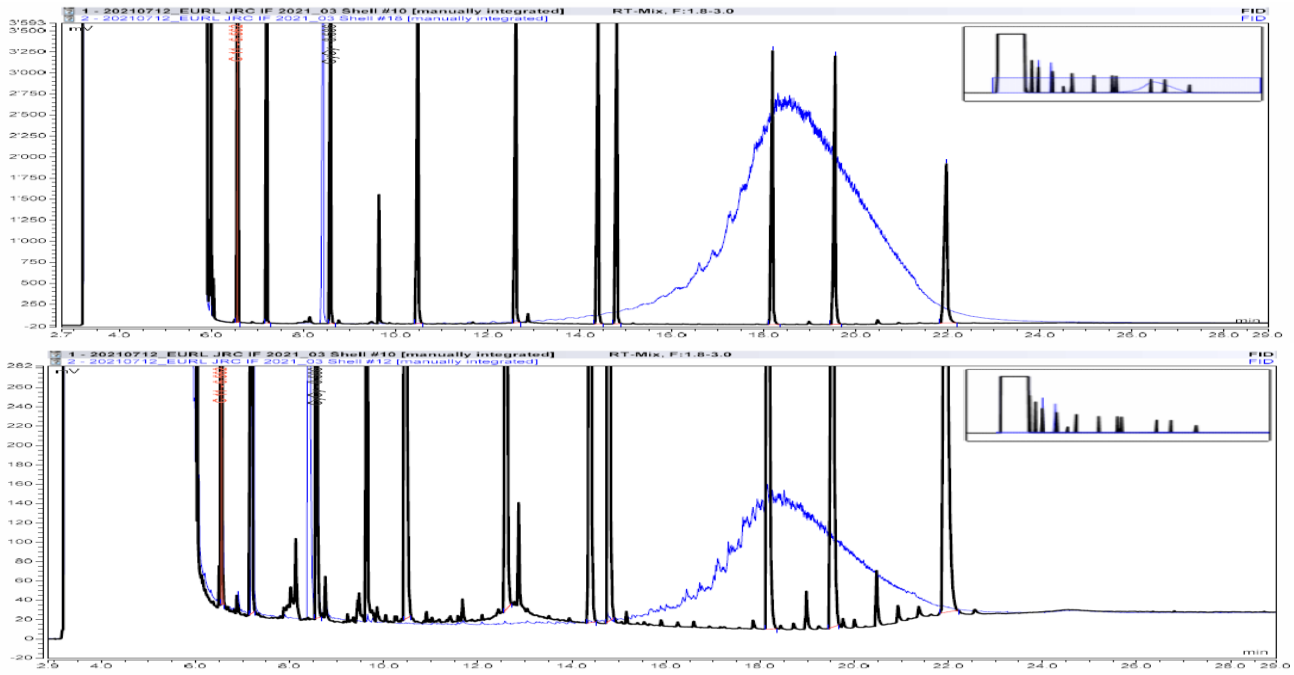


Figure 8. MOSH (above) and MOAH (below) chromatograms (L26) for the QC10-4 overlaid with the RT calibration mix from a dual channel on-line LC-GC system

5 Conclusions

The JRC organised the third interlaboratory comparison (JRC IF 2021-03) to characterise the specific Shell SN500* mineral oil batch used to prepare the QC10 test sample and to spike different infant formula formulation for the next method validation study. Six laboratories (out of 30) reported consistent results for the different QC10 diluted samples and satisfactory recoveries. Their results were used to derive the "reference" percent compositions of MOSH and MOAH in Shell SN500* mineral oil. Some other laboratories reported lower recoveries and highly scattered results, requiring further instrumental optimisation.

The integration of the internal standard peaks (eg. 1-MN and 2-MN) and/or the chromatographic humps in “simple” QC10 diluted solutions seemed to be challenging. How to select the baseline for the background correction? How to subtract correctly a riding peak? Such questions require clear guidance to ensure a reliable integration. In this context, the JRC intends to organise another “virtual” interlaboratory comparison, where a set of chromatograms (files) will be sent to laboratories in order to assess the quality of their integrations.

Acknowledgements

The European Union Reference Laboratory for Food Contact Materials (EURL-FCM) thanks Special Nutrition Europe (NESTLE pilot plant) for producing the different powder infant formulas and the Reference Materials Unit of the JRC for processing the material and delivering high quality test items. Furthermore, the 28 laboratories listed hereafter are kindly acknowledged for their participation in this ILC round.

Organisation	Country
Graz University of Technology	Austria
Primoris	Belgium
Liege University	Belgium
Sciencesano	Belgium
ITERG	France
NQAC NESTLE France Laboratory	France
Bavarian Health and Food Safety Authority	Germany
Bundesinstitut für Risikobewertung (BfR)	Germany
Chemisches und Veterinäruntersuchungsamt Münsterland-Emscher-Lippe (CVUA-MEL)	Germany
CVUA Stuttgart	Germany
Fraunhofer IVV	Germany
GALAB Laboratories GmbH	Germany
GBA Gesellschaft für Bioanalytik mbH	Germany
Institut Kirchhoff Berlin GmbH	Germany
Landesbetrieb Hessisches Landeslabor	Germany
mas GmbH	Germany
SGS Institut Fresenius GmbH	Germany
IFP Institut für Produktqualität GmbH	Germany
Dublin Public Analyst's Laboratrory	Ireland
NEOTRON SPA	Italy
Unversity of Udine	Italy
NOFALAB	Netherland
Eurofins Lab Zeeuws-Vlaanderen (CNL027)	Netherlands
Wageningen Food Safety Research	Netherlands
Dr A Verwey	Netherlands
Centro Nacional Alimentación-AESAN	Spain
Nestlé Research	Switzerland
Official Food Control Authority of the Canton of Zurich	Switzerland
Swiss Quality Testing Services	Switzerland

List of abbreviations

DG SANTE	Directorate General for Health and Food Safety
JRC	Joint Research Centre
ILC	interlaboratory comparison
LC-GC-FID	liquid chromatography hyphenated with gas-chromatography with flame ionisation detector
MOAH	mineral oil aromatic hydrocarbons
MOSH	mineral oil saturated hydrocarbons

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Annex 1. Invitation letter

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

Geel, 29 October 2021

(sent by e-mail)

Subject: Invitation to participate in a ring trial round "JRC IF 2021/03"

Dear all,

As we have informed you already with our letter from May 28, the JRC is launching a study for the characterisation of MOSH and MOAH in mineral oils (MO) with the aim to evaluate the consistency of results obtained at different concentration levels in a hexane solution.

Two test samples of approx. 5 ml with a known mass fraction of MO in hexane will be dispatched during the **first half of July**. You would be requested to perform several dilutions in hexane and analyse the MOSH/MOAH content. Results should be expressed and reported in mg MOSH/MOAH per kg MO. The instrumental setup should be the same as for the analysis of MOAH in IF.

The participation is free of charge and open for all interested laboratories.

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

https://web.jrc.ec.europa.eu/ilcRegistrationWeb/registration/registration.do?selComparison=2641

Please, register at the latest by Tuesday, 15th of June 2020.

The planned deadline for submission of results is the 3rd of September 2021.

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

Kind regards,

/signed electronically in Ares/
S. BRATINOVA

Cc: H. Emons (JRC.F.5)

Annex 2. Instructions to the participants

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Directorate F - Health, Consumers & Reference Materials (Geel/Ispra)
Food & Feed Compliance

EURL
European Union Reference Laboratory
for Food Contact Materials

Geel, 07 July 2021

Attn.: «Title» «Firstname» «Surname»
«Organisation»
«Department»

«Zip» «Town»
«Country»

Subject: Participation in JRC IF 2021/03 – "Characterisation of MO in hexane solution"

Dear «Title» «Surname»,

Thank you for participating in the trial **JRC-IF-2021/03 – "Characterisation of MO in hexane solution".** This round is organised to characterise MOSH and MOAH in Shell SN500 mineral oil and to evaluate the consistency of results obtained at different concentration levels in a hexane solution.

The measurands are concentration (mg/l) of total MOSH and MOAH (C10-C50) in hexane and the corresponding **fraction cuts**, as defined in the "Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials" in the frame of Recommendation (EU) 2017/84.

The parcel contains an amber glass ampoule with approx. 5 ml of Shell SN500 oil in hexane (QC10) with known content as follows:

QC10	g/kg	g/L
SN 500	3.064	2.024

Upon arrival of this parcel, please check whether the ampoule is undamaged after transport.

You are requested to send the "Confirmation of receipt" form within 3 days after receipt of the samples to Stefanka-Petkova.BRATINOVA@ec.europa.eu.

Please open the ampoule carefully and prepare the four test samples by dilutions following the instructions in Table 1. **The leftover should be transferred into a vial and kept in the refrigerator as it could be used later on** for LOQ estimation in IF (procedure will follow) and as QC sample.

European Commission, JRC-Geel, Belgium. Telephone: (32) 14571800.
e-mail: jrc-eurl-fcm@ec.europa.eu URL: https://ec.europa.eu/jrc/en/eurl/food-contact-materials

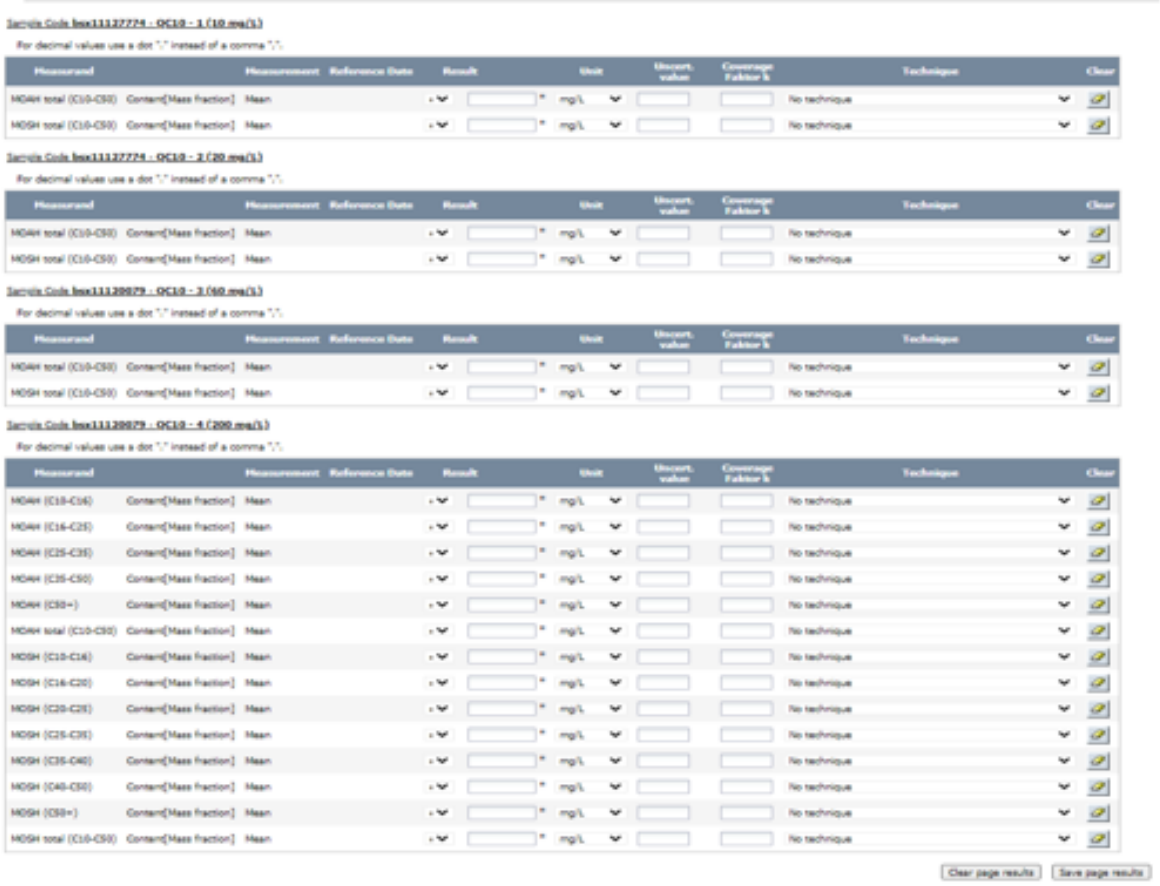
Table 1. Dilutions to be made with hexane for preparation of the four test items

	50 ul inj		QC10	IS	C6H6	total volume
Sample name	µg in column	C mg/l	ul	ul	ul	ul
QC10-1	0.5	10.1	10	20	1970	2000
QC10-2	1	20.2	10	10	980	1000
QC10-3	3	60.7	30	10	960	1000
QC10-4	10	202.4	100	10	890	1000

Please report the following:

- The results from the measurements of total MOSH (**C10-C50**) and total MOAH (**C1 C50**) in mg/l hexane for the four different test samples QC10-1; QC10-2; QC10-3 a QC10-4.
- The fraction cuts for MOSH and MOAH according to the JRC Guidance and additional the amount beyond C50 - >C50 only for the test sample with the highest concentrati (QC10-4);
- The integrated MOSH/MOAH chromatograms for the lowest level (QC10-1) togeth with the chromatograms of the n-alkanes mixture (C10-C50).

The reporting website **https://web.jrc.ec.europa.eu/ilcReportingWeb/** will be opened 05/07/2021. The reporting page will look as the picture below:



Your personal password key that you need for the access to the reporting website is **«Part_key»**

Please do not forget to answer the questions in the questionnaire via EUSurvey platform https://ec.europa.eu/eusurvey/runner/JRC_IF_2021_03

The deadline for submission of the results is set to **August 30, 2021**.

A draft report to participants will be circulated shortly after the end of the round to present the reported values from all participants with their lab codes. The laboratory code will be disclosed only to the respective participant together with the password key for reporting, to preserve the confidentiality of the data reported.

Your participation in this project is greatly appreciated.

Do not hesitate to contact me for further information.

With kind regards,

/signed electronically in Ares/

Dr. Stefanka Bratinova
JRC IF 2021/03 Coordinator

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

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Annex 3. Confirmation of receipt

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Directorate F – Health, Consumers and Reference Materials
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European Union Reference Laboratory
for Food Contact Materials

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Subject: Participation in JRC IF 2021/03 – "Characterisation of Shell SN500 mineral oil in hexane solution"

Please return this form within 3 days of reception, to confirm that the package arrived well to your laboratory. If the ampule is damaged, please mention it below and contact us as soon as possible.

Date of package arrival: _____/_____/2021

Was the sample damaged? ☐ YES ☐ NO

Remarks

……..

……..

……..

……..

Signature

Thank you for returning this form by email to:
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CC: jrc-eurl-fcm@ec.europa.eu

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Annex 4. Reported concentration of total MOAH (C10-C50) in QC10-1, 2, 3, 4 expressed in mg L^{-1}

	QC10-1	QC10-2	QC10-3	QC10-4
	mg/L	mg/L	mg/L	mg/L
1	2.86	5.62	17.60	61.10
2	3.50	5.90	15.90	50.90
3	3.11	6.43	20.05	71.39
4	3.53	7.54	22.50	78.80
5	2.80	5.99	19.59	69.46
6	3.60	7.40	21.00	64.00
7	3.21	8.20	22.95	78.22
8	3.00	6.50	20.10	65.80
9		5.28		63.40
10	3.77	6.32	18.20	58.85
11	3.80	6.84	18.94	63.38
12	5.64	10.47	32.91	123.50
13	2.75	5.79	16.51	56.25
15	3.80	6.40	20.80	74.10
16	3.12	7.85	24.19	70.23
17	3.36	6.59	21.53	71.61
18	4.30	8.40	27.30	72.10
19	2.02	6.67	21.32	68.78
20	3.76	7.04	21.30	69.00
22	3.00	6.60	20.60	70.60
23	2.81	6.46	20.91	64.41
24	3.00	6.70	19.20	66.30
25	3.29	6.99	20.29	68.41
26	4.00	7.40	22.70	75.00
27	2.59	3.14	12.14	46.99
28	3.80	7.24	18.72	61.47
29	2.20	10.00	17.00	53.20
30	3.01	5.89	17.85	62.05
31	3.55	6.85	21.75	71.93

Annex 5. Reported concentrations of total MOSH (C10-50) in QC10-1, 2, 3, 4, expressed in mg L^{-1}

	QC10-1	QC10-2	QC10-3	QC10-4
	mg/L	mg/L	mg/L	mg/L
1	5.17	10.40	32.90	109.00
2	6.90	12.60	36.90	127.90
3	4.70	10.17	31.31	110.46
4	5.91	13.00	37.80	130.50
5	5.20	10.70	34.67	116.96
6	6.50	13.00	38.00	120.00
7	6.39	15.72	43.37	146.44
8	5.60	11.80	35.00	119.20
9		11.23		123.00
10	8.21	15.21	46.63	149.00
11	6.28	11.72	32.42	104.38
12	10.33	19.10	61.23	283.71
13	4.75	10.09	28.94	101.91
15	6.80	10.70	33.60	120.10
16	3.95	10.10	30.70	97.17
17	4.36	9.65	33.86	118.34
18	7.20	13.80	43.80	115.80
19	3.67	10.00	32.49	105.18
20	5.27	10.70	33.80	112.00
22	5.80	11.60	36.40	122.40
23	5.20	12.25	38.96	123.73
24	5.00	11.30	32.30	110.90
25	6.31	13.24	39.02	127.47
26	5.80	11.50	35.00	115.20
27	5.70	7.88	28.75	103.20
28	5.56	11.15	29.62	98.05
29	8.90	22.60	46.90	178.10
30	5.89	11.50	35.55	111.64
31	6.91	12.26	36.28	118.77

Annex 6. Reported concentrations of MOAH fractions in QC10-4, expressed in mg L^{-1}

	C10-C16	C16-C25	C25-C35	C35-C50	C50+	TOTAL
	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
1	< 0.20	0.36	16.40	44.30	2.02	61.10
2	< 0.10	0.60	13.80	36.60	2.70	50.90
3	< 0.50	< 0.50	23.85	47.04	2.13	71.39
4	0	0.29	18.40	60.10	1.86	78.80
5	0	0.29	18.34	50.83	1.51	69.46
6	0	0.34	15.00	49.00	2.80	64.00
7	< 0.15	0.39	20.89	56.94	11.81	78.22
8	0	0.20	11.50	54.10	2.80	65.80
9	0	< 0.50	15.50	47.80	1.90	63.40
10	0.10	0.22	16.63	40.90	1.00	58.85
11	0.435	0.936	15.18	46.83	3.42	63.39
12	0	0	26.41	96.42	3.82	123.50
13	< 0.20	0.20	11.97	44.08	3.73	56.25
15	0.20	< 0.50	17.70	56.00	< 0.10	74.10
16	< 0.15	0.78	17.41	51.94	0.37	70.23
17	< 0.10	0.26	19.23	52.12	11.03	71.61
18	< 1.00	< 1.00	20.70	50.60	3.90	72.10
19	< 0.50	< 0.50	20.04	48.46	2.52	68.78
20	< 0.30	< 0.30	19.10	49.40	2.17	69.00
22	< 1.00	< 1.00	20.30	49.80	1.90	70.60
23	< 0.50	< 0.50	14.63	49.54	3.02	64.41
24	0	0.50	19.90	45.90	1.90	66.30
25	0	1.07	26.00	41.34	1.32	68.41
26	< 0.10	0.77	18.80	52.40	2.30	75.00
27	0	0.17	17.66	29.16	0	46.99
28	< 0.50	0.51	17.04	43.86	2.54	61.47
29	0	0.80	14.10	37.10	2.80	53.20
30	0	0.26	17.47	44.32	0.41	62.05
31	0.08	0.75	18.21	52.88	1.10	71.93

Annex 7. Reported concentrations of MOSH fractions in QC10-4, expressed in mg L^{-1}

	C10-C16	C16-C20	C20-C25	C25-C35	C35-C40	C40-C50	C50+	TOTAL
	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L	mg/L
1	< 0.20	0.31	2.12	38.50	41.80	26.70	1.62	109.00
2	< 0.10	0.60	2.70	48.70	46.60	29.30	2.40	127.90
3	< 0.50	< 0.50	1.95	44.14	37.25	26.91	1.70	110.46
4	0	0.20	2.09	44.40	50.80	33.00	0.96	130.50
5	0	0.10	1.78	40.31	44.95	29.82	1.40	116.96
6	0.3	0.44	2.10	45.00	43.00	27.00	2.20	120.00
7	< 0.15	0.27	2.21	50.12	54.78	39.34	5.88	146.44
8	0	0.20	1.10	35.00	45.50	37.30	1.50	119.20
9	0	< 0.50	1.49	41.30	47.50	32.70	1.60	123.00
10	0.11	0.20	1.87	57.51	54.98	32.12	2.50	149.00
11	0.4	0.68	2.10	34.29	45.65	21.27		104.38
12	0	0	3.73	97.19	113.03	69.76	4.34	283.71
13	< 0.20	0.27	1.98	43.67	34.15	21.85	1.16	101.91
15	< 0.20	0.20	1.60	16.40	101.90	< 0.10	< 0.10	120.10
16	< 1.00	< 1.00	1.32	19.98	35.65	40.00	2.65	97.17
17	< 0.10	0.21	1.77	37.04	45.79	33.52	9.67	118.34
18	< 1.00	< 1.00	2.30	39.50	47.20	26.10	1.10	115.80
19	< 0.50	< 0.50	1.60	38.41	40.22	24.82	1.57	105.18
20	< 0.30	< 0.30	2.01	43.10	40.00	26.00	1.28	112.00
22	< 1.00	< 1.00	2.60	49.4	48.50	21.40	< 1.00	122.40
23	< 0.50	< 0.50	2.19	49.59	45.36	26.22	1.40	123.73
24	0	0.20	1.80	41.10	42.40	25.40	1.60	110.90
25	0	0.47	2.90	50.28	44.77	29.05	1.61	127.47
26	< 0.10	0.36	2.20	39.40	43.90	29.40	1.80	115.20
27	0	0.04	1.39	40.70	42.93	18.13		103.20
28	< 0.50	< 0.50	1.70	36.56	34.54	24.57	1.37	98.05
29	0	0.40	2.80	57.30	69.70	49.00	5.10	178.10
30	0	0.23	2.34	41.10	41.30	26.67	1.95	111.64
31	0.11	0.46	2.38	43.12	44.54	28.16	1.28	118.77

Annex 8. Calculated total MOAH (C10-C50) % composition in Shell SN500* in QC10-1 to 4

	QC10-1 % SN500	QC10-2 % SN500	QC10-3 % SN500	QC10-4 % SN500	**RSD, %**
1	28.3	27.8	29.0	30.2	**3.6**
2	34.7	29.2	26.2	25.1	14.8
3	30.8	31.8	33.0	35.3	**5.9**
4	35.0	37.3	37.1	38.9	**4.4**
5	27.7	29.7	32.3	34.3	9.3
6	35.6	36.6	34.6	31.6	**6.3**
7	31.8	40.6	37.8	38.6	10.2
8	29.7	32.2	33.1	32.5	**4.7**
9		26.1		31.3	12.8
10	37.3	31.3	30.0	29.1	11.6
11	37.6	33.9	31.2	31.3	8.1
12	55.8	51.8	54.2	61.0	7.0
13	47.0	50.0	47.7	27.8	**2.5**
15	37.6	31.7	34.3	36.6	7.6
16	30.9	38.9	39.9	34.7	11.4
17	33.3	32.6	35.5	35.4	**4.3**
18	42.6	41.6	45.0	35.6	9.7
19	20.0	33.0	35.1	34.0	23.2
20	37.2	34.9	35.1	34.1	**3.8**
22	29.7	32.7	33.9	34.9	6.9
23	27.8	32.0	34.4	31.8	8.7
24	29.7	33.2	31.6	32.8	**4.9**
25	32.6	34.6	33.4	33.8	**2.5**
26	39.6	36.6	37.4	37.1	**3.5**
27	25.6	15.5	20.0	23.2	20.7
28	37.6	35.8	30.8	30.4	10.7
29	21.8	49.5	28.0	26.3	39.4
30	29.8	29.2	29.4	30.7	**2.2**
31	35.1	33.9	35.8	35.5	**2.4**

Annex 9. Calculated total MOSH (C10-C50) % composition in Shell SN500* in QC10-1 to 4

	QC10-1 % SN500	QC10-2 % SN500	QC10-3 % SN500	QC10-4 % SN500	**RSD, %**
1	51.2	51.5	54.2	53.9	**3.0**
2	68.3	62.4	60.8	63.2	**5.1**
3	46.5	50.3	51.6	54.6	6.6
4	58.5	64.4	62.3	64.5	**4.5**
5	51.5	53.0	57.1	57.8	**5.6**
6	64.4	64.4	62.6	59.3	**3.8**
7	63.3	77.8	71.4	72.4	8.4
8	55.4	58.4	57.7	58.9	**2.6**
9		55.6		60.8	6.3
10	81.3	75.3	76.8	73.6	**4.3**
11	62.2	58.0	53.4	51.6	**5.4**
12	102.3	94.6	100.9	140.2	18.9
13	47.0	50.0	47.7	50.4	**3.4**
15	67.3	53.0	55.4	59.3	10.7
16	39.1	50.0	50.6	48.0	11.3
17	43.2	47.8	55.8	58.5	13.8
18	71.3	68.3	72.2	57.2	10.2
19	36.3	49.5	53.5	52.0	16.4
20	52.2	53.0	55.7	55.3	**3.2**
22	57.4	57.4	60.0	60.5	**2.8**
23	51.5	60.6	64.2	61.1	9.2
24	49.5	55.9	53.2	54.8	**5.3**
25	62.5	65.5	64.3	63.0	**2.2**
26	57.4	56.9	57.7	56.9	**0.6**
27	56.4	39.0	47.4	51.0	15.1
28	55.0	55.2	48.8	48.4	7.2
29	88.1	111.9	77.3	88.0	16.0
30	58.3	56.9	58.6	55.2	**2.7**
31	68.4	60.7	59.8	58.7	7.2

Annex 10. Calculated % recoveries of total (MOSH+MOAH) in QC10-1 to QC10-4

	MOSH+MOAH (C10-C50)				C10-C50+
	QC10-1	QC10-2	QC10-3	QC10-4	QC10-4
1	79.5	79.3	83.2	84.0	86.0
2	103.0	91.6	87.0	88.3	90.9
3	77.3	82.2	84.6	89.8	91.4
4	93.5	101.7	99.3	103.4	104.8
5	79.2	82.6	89.4	92.1	93.5
6	100.0	101.0	97.2	90.9	92.3
7	95.0	118.4	109.3	111.0	119.9
8	85.1	90.6	90.8	91.4	93.5
9		81.7		92.1	93.8
10	118.6	106.6	106.8	102.7	102.7
11	99.8	91.9	84.6	82.9	83.8
12					
13	74.3	78.6	74.9	78.1	80.6
15	105.0	84.7	89.6	95.9	95.8
16	70.0	88.9	90.4	82.7	84.0
17	76.4	80.4	91.3	93.8	104.1
18	113.9	109.9	117.1	92.8	94.6
19	56.3	82.5	88.6	85.9	87.8
20	89.4	87.8	90.8	89.4	90.4
22	87.1	90.1	93.9	95.4	95.8
23	79.3	92.6	98.6	93.0	94.8
24	79.2	89.1	84.8	87.5	89.3
25	95.0	100.1	97.7	96.8	98.2
26	97.0	93.6	95.1	94.0	94.5
27	82.1	54.6	67.4	74.2	74.2
28	92.7	91.0	79.6	78.8	80.4
29	109.9	161.4	105.3	114.3	118.1
30	88.1	86.1	88.0	85.8	87.0
31	103.6	94.6	95.6	94.2	95.3

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