European Commission

JRC TECHNICAL REPORT

Determination of MOAH in Infant Formula

JRC IF 2020-01 - an exploratory interlaboratory comparison

Bratinova S., Robouch P.

Karasek L., Goncalves C., Beldi G., Senaldi C., Jakubowska N., Valzacchi S. Conneely P.

Hoekstra E., Emons H.

Joint Research Centre

JRC 121915 EN

Contact information
Name: Stefanka Bratinova
Address: European Commission
Joint Research Centre (JRC)
Retieseweg 111
Geel 2440, Belgium
Email: Stefanka-Petkova.BRATINOVA@ec.europa.eu
Tel.: 0032 14 571800
EU Science Hub
https://ec.europa.eu/jrc

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Executive summary

An exploratory interlaboratory comparison (ILC) was organised by the Joint Research Centre (JRC) in the frame of developing on request of DG SANTE a harmonised method for the determination of mineral oil aromatic hydrocarbons (MOAH) in infant formula (IF). This report presents the outcome of the ILC called JRC IF 2020-01 and attended by 27 participants from 8 European countries.

A thorough questionnaire related to the employed experimental details was elaborated by the organisers and filled in by the participants. The results reported were used to identify the best experimental practices in view of selecting the best analytical approach. This study should further advance the harmonisation of a standard operating procedure which will be in a next step validated using a ring-trial.

1 Introduction

Following the RASFF notification message 2019.3734 (dated 25/10/2019) [1] and the Foodwatch findings [2] related to 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 organise a Roundtable Workshop on the determination of MOAH in IF [3]. The meeting held in Brussels on December 5, 2019, was attended by various stakeholders (e.g. official control laboratories, industry and NGOs), DG SANTE and EFSA. The comparability and reliability of the analytical procedures applied by laboratories to monitor the MOAH content in IF was thoroughly discussed. A broad variety of experimental procedures were reviewed. Participants agreed to simplify the experimental protocols and identified the need for a harmonised method to be validated and further standardised.

JRC committed (i) to coordinate the work; (ii) to collect the available standard operating procedures used by the experienced laboratories; (iii) to draft a harmonised SOP to be reviewed; and (iv) to organise a ring-trial exercise for the validation of the harmonised protocol. Hence, the JRC decided to organise an exploratory interlaboratory comparison (ILC) to evaluate the analytical procedures applied to determine the MOAH mass fraction in an infant formula sample. This report represents the outcome of the ILC **JRC IF 2020-01**.

2 Scope

JRC IF 2020-01 aimed to collect and evaluate the different SOPs applied by a variety of participants (official control laboratories, industry, NGOs and universities) when analysing the MOAH content in a well characterised infant formula test item. Successful/satisfactory results will be used to identify the reliability and robustness of experimental steps to be used, thus resulting in a detailed SOP to be further investigated.

3 Set-up of the exercise

In February 2020, the JRC identified and purchased at a local supermarket three 800 g cans (displaying the same batch number) of a commercial IF powder with a suitable MOAH content for the interlaboratory comparison. The material was mixed, homogenised and bottled (25 g aliquots in 100 ml brown glass bottles) by the JRC. All necessary measures were taken to prevent cross-contaminations:

- the bottles were baked before filling at 400 °C for at least 6 h;
- the crimp cap used for closure contained Teflon lining; and
- an aluminium (Al) foil was inserted between the caps and the bottle neck. In addition, the bottles were wrapped in Al foil to prevent any potential gas-phase cross contaminations during the shipment and storage.

Due to the imposed COVID-19 lockdown in Belgium, the homogeneity study could not be finalised. However, the thorough mixing of the content of three cans of the commercial powder IF, originating from the same batch, is assumed as sufficient to produce homogeneous test items.

(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) Report from the Roundtable meeting: https://ec.europa.eu/jrc/en/eurl/food-contact-materials/technical-guidelines

Confidentiality

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

Time frame

JRC IF 2020-01 was announced by email on February 13, 2020 (Annex 1, Invitation letter). Due to the upcoming COVID-19 pandemic, with a lockdown expected to be implemented shortly in Belgium, a strict deadline for registration was set to March 17, 2020 - 09:00 h. On that day, all samples were dispatched to participants. At first a tentative deadline for reporting of results was set to May 10, 2020. It was first extended to end of May, due to the ongoing COVID-19 pandemics, with the last reported results accepted on June 18, 2020.

Distribution

Each participant received:

- One bottle containing 25 g of powder IF in a 100 ml brown glass bottle;
- The "Instruction to participants" (Annex 2); and
- The "Confirmation of receipt form" to be sent back to the PT coordinator after receipt of the test item (Annex 3).

Instructions to participants

Detailed instructions were provided to the participants by e-mail (Annex 2). They were requested to apply experimental protocols complying with (i) the decisions taken during the Roundtable meeting and (ii) the requirements set in the "EURL-FCM Guidance on sampling, analysis and data reporting" [4].

The following measurands were defined:

- "the mass fraction of total MOAH in IF", expressed in mg kg^{-1}
- "the mass fraction of the MOAH in IF corresponding to the retention time of n-alkanes from n-C35 to n-C50 (MOAH C35-C50)", expressed in mg kg^{-1}
- "the mass fraction of the MOAH in IF corresponding to the retention time of n-alkanes from n-C25 to n-C35 (MOAH C25-C35)", expressed in mg kg^{-1}
- ""the mass fraction of the MOAH in IF corresponding to the retention time of n-alkanes from n-C16 to n-C25 (MOAH C16-C25)", expressed in mg kg^{-1}
- "the mass fraction of the MOAH in IF corresponding to the retention time of n-alkanes from n-C10 to n-C16 (MOAH C10-C16)", expressed in mg kg^{-1}

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

In addition, participants were requested to:

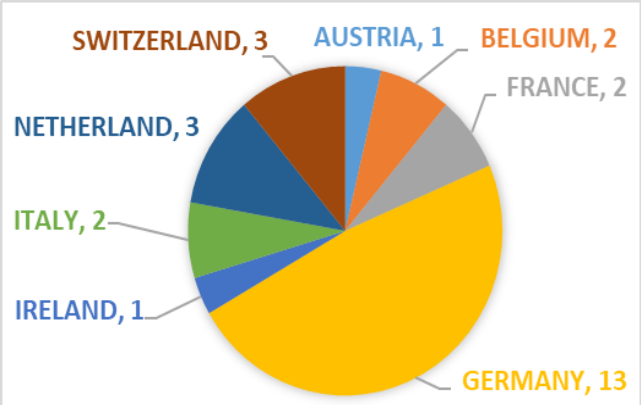
- Perform three independent measurements;

(4) JRC Report – Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials (EUR-29666, 2019) https://europa.eu/!nx87Th

- Report their calculated mean (in mg kg^{-1}), derived from their replicates - as they would report to their customer;
- Provide the associated expanded uncertainty (in mg kg^{-1}), specifying the coverage factor;
- Fill the experimental details into the online questionnaire [5] (Annex 4) about the applied method; and
- Provide the recorded chromatograms.

4 Results and Discussions

A total of 30 laboratories registered to the JRC IF 2020-01 round. Twenty-seven participants from 8 EU countries reported results; of which 26 filled in the detailed online questionnaire related to their standard operation procedure (SOP) and 25 provided the requested chromatograms.



4.1 Results

This exploratory interlaboratory comparison aimed to evaluate the analytical procedures used when monitoring MOAH in IF. This will allow the identification of the relevant experimental steps to be included in the harmonised/standard analytical method for further validation.

JRC IF 2020-01 was not intended as a proficiency testing exercise, hence no reported results were scored. In addition, the "assigned values" that could have been obtained applying robust statistics are not considered as reliable estimates of the "true values".

Participants reported quantitative results (numerical values) for total MOAH (Annex 5) and the C25-C35, C35-C50 fractions (Annexes 6-7). "Less values" were mainly reported for the C10-C16 and C16-C25 fractions (Annexes 7-8). The reported results together with their associated expanded uncertainties are presented graphically in Figure 1.

Based on the kernel distribution presented in Figure 2, the reported total MOAH mass fractions ranging from 2.2 to 3.7 mg kg^{-1} (bracketing the main mode at 3.0 mg kg^{-1}) are considered suitable for the identification of proper analytical steps and experimental procedures. Results below 1.4 mg kg^{-1} or above 4.2 mg kg^{-1} seem to be unsatisfactory.

While the JRC Guidance document [4] recommends to integrate the entire chromatogram when determining the total MOAH content, many participants reported the total MOAH as sum of the content of the different (quantified) fractions, applying a lower bound approach. Such an approach would provide underestimated results, when MOAH is detected but not quantified in some of the fractions.

(5) https://ec.europa.eu/eusurvey/runner/JRC_IF_2020_01A

Participants (with experience or novel in the field) displayed a broad range for the relative intermediate precision parameter (up to 60 %) at LOQ levels. This should be significantly improved in order to comply with the 25 % limit required by the JRC Guidelines for official controls.

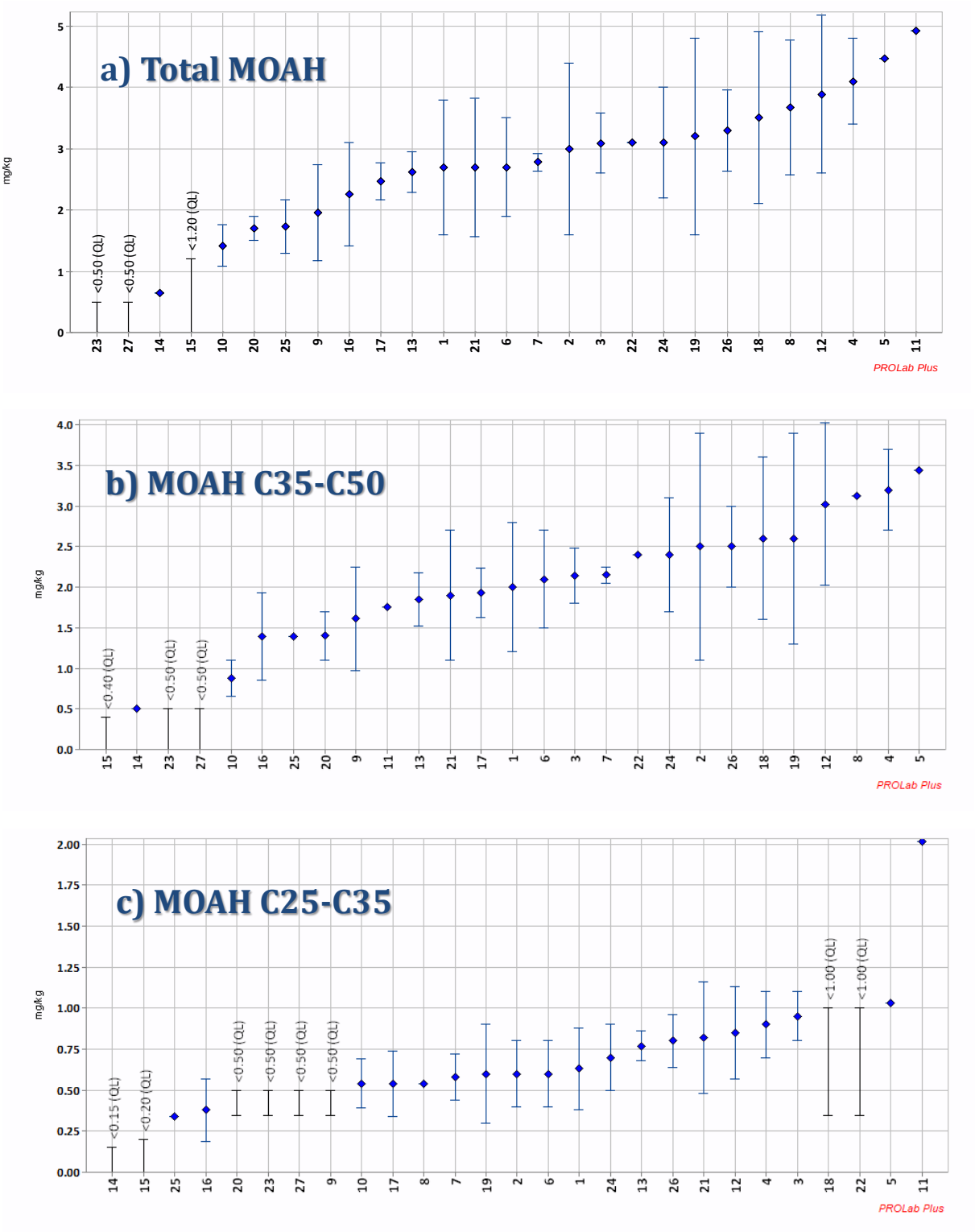


Figure 1 *Distribution of the results from the participants in JRC IF 2020-01; a) total MOAH; b) MOAH C35-C50; c) MOAH C25-C35*

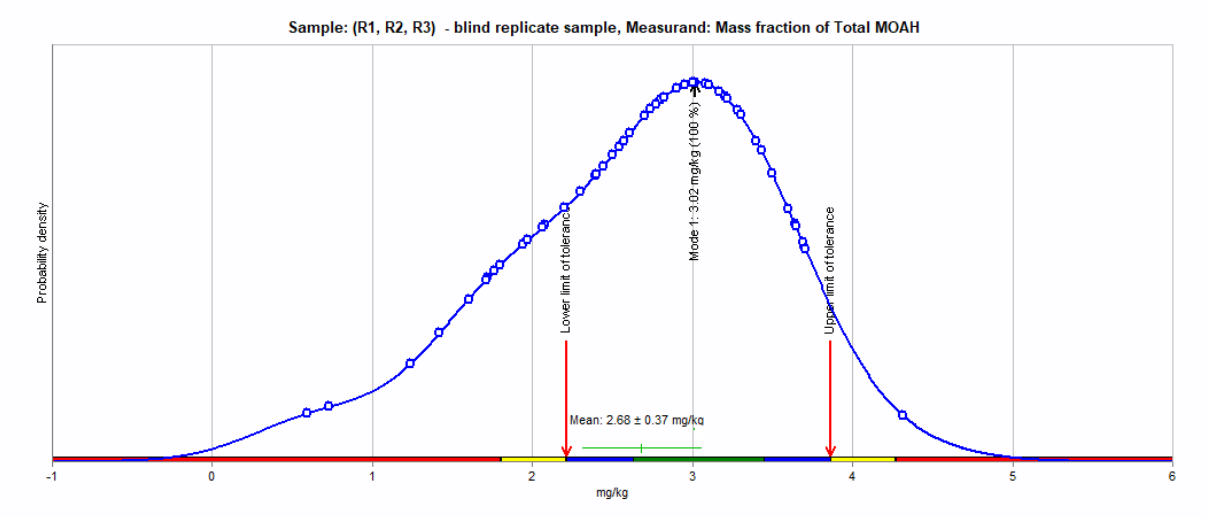


Figure 2 *Kernel density plot and Kernel density mode for total MOAH results*

4.2 Questionnaire

The structure of the online questionnaire (Annex 4, [5]) was based on the main experimental steps (listed hereafter) that were identified and discussed during the Roundtable meeting:

- ☐ Saponification;
- ☐ Extraction;
- ☐ Epoxidation;
- ☐ Column clean-up;
- ☐ On-line or off-line LC-GC MOSH/MOAH separation; and
- ☐ FID chromatogram quantification.

4.3 Details about the experimental procedures used

Important conclusions about the good practices and critical steps requiring further investigations were derived from the experimental details provided by the laboratories in the questionnaire:

- ✓ Satisfactory total MOAH mass fractions were reported by laboratories having applied the experimental pathways A, B, C, D and E (Figure 3);
- ✓ Largely scattered (and often unsatisfactory) results were reported when proceeding without reconstitution of the powdered IF (G and H). This supports the Roundtable decision recommending the reconstitution of the powder IF before further steps.

At that time, questions were raised by the organisers whether the different experimental pathways chosen by laboratories (e.g. saponification and extraction) would significantly influence the measurement results. After experimental comparison of the A-D approaches and experts' consultations, the use of hot water reconstitution of the IF, followed by saponification and extraction was chosen to be the most effective procedure.

The following important observations concerning the experimental steps were made:

- ✓ Approximately 2 g KOH (from 1.5 to 2.5 g) were used for the saponification of a 5 g sample intake, performed at 60 to 80 °C for 30 to 120 min, using different mixing/shaking approaches (see Figure 4).
- ✓ Although the Roundtable recommended the use of an ethanolic KOH saturated solution for saponification, satisfactory results (12 out of 18) were also obtained when using a 50 % water solution of KOH, which is more favourable for handling.
- ✓ Applying saponification in the organic phase only after extraction (E) does not comply with the Roundtable decisions. Despite the good results reported by two participants, this approach may not be applicable to all kinds of IF and should not be recommended at this point.
- ✓ Epoxidation and additional clean-up are the most critical steps. They have to be applied with due care and must be assessed for their effectiveness and robustness. Despite the Roundtable requirement of implementing the so-called *Nestola procedure* (including epoxidation in ethanolic media) [6], two participants successfully applied a different protocol and performed epoxidation at sub-ambient temperature in DCM.
- ✓ Most of the participants used 100 to 200 mg chlorobenzoic acid (mCPBA) for epoxidation. Only 5 laboratories reported preliminary purification of the acid by washing with hexane. Consequently, purification should be performed (if needed), depending on the purity of each new batch of the reagent. The mCPBA amount depends on whether there is a column clean-up step before the epoxidation.

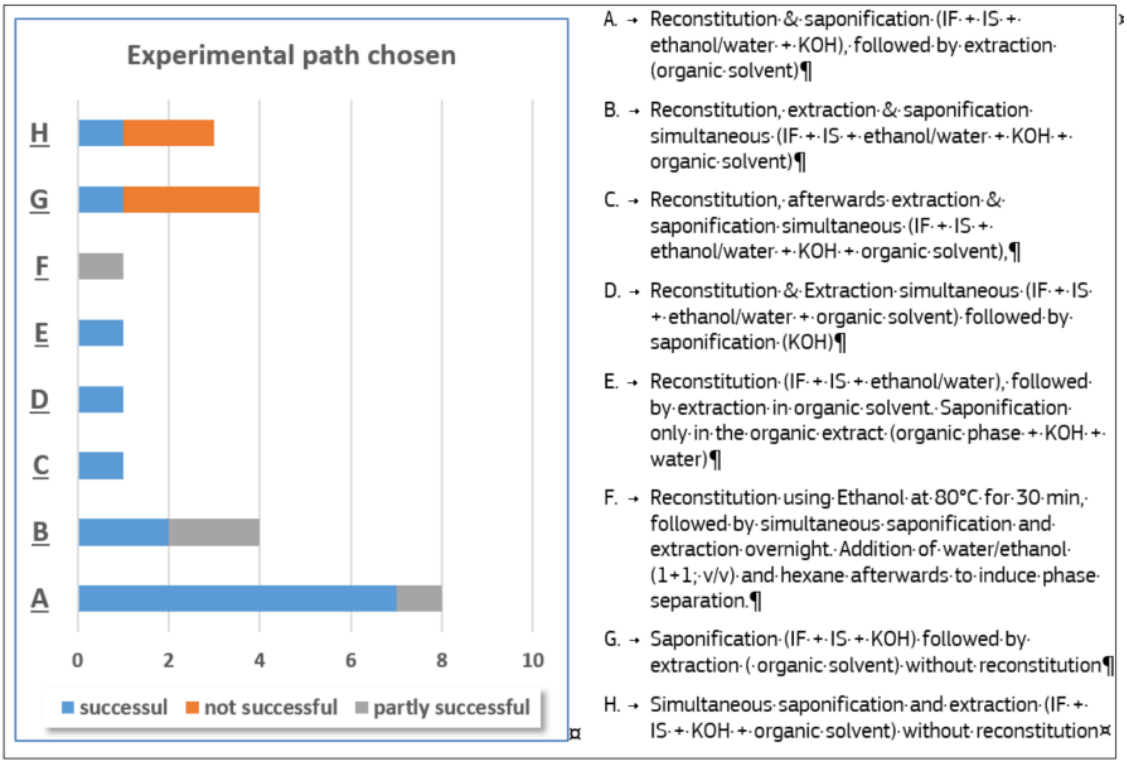


Figure 3. *Influence of the experimental path chosen for the results of the ring trial*

[6] Nestola M., M., Schmidt T. Journal of Chromatography A, 1505 (2017) 69–76

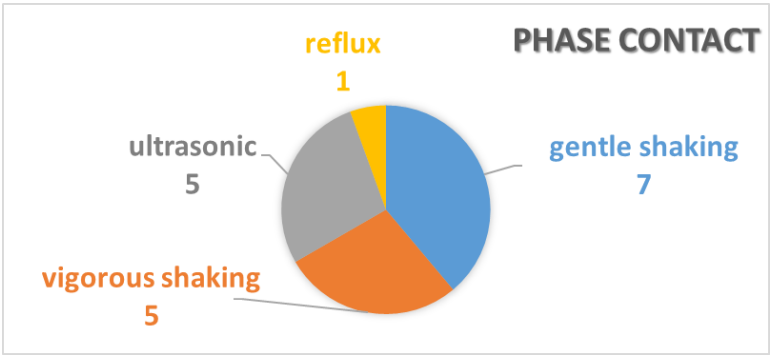


Figure 4. *Different way of insuring phase contact during the saponification step*

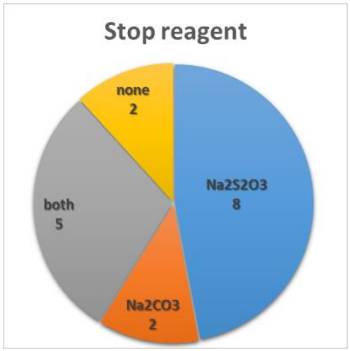


Figure 5 *Type of the stop reagent for the reaction of epoxidation*

- ✓ Half (8/17) of the laboratories used only sodium thiosulfate ($Na_2S_2O_3$) as a stop reagent. However, due to the excess of acid, some participants observed crystal formation in the organic solvent when concentrating the extract to smaller volumes (to reduce the LOQ). Such a phenomenon was not observed with all types of IF.
- ✓ The formation of crystals should not affect the analysis after their removal, but it could cause clogging in the chromatographic system. In order to remove the excess of acid from the organic phase and to protect the chromatographic device, washing with sodium carbonate (Na_2CO_3) is recommended.

The details of the clean-up procedure using chromatographic column filled with activated silica gel and the point in the analytcal procedure performing it (before or after epoxidation) are relevant topics to be further investigated.

From the 13 satisfactory results reported (Figure 5), 7 were obtained when the column clean-up was applied "after epoxidation", as required by the Roundtable decisions, 3 were obtained when the column clean-up was applied "before epoxidation", and the clean-up step was omitted by 3 participants.

Skipping the clean-up step does not comply with the decisions of the Roundtable and doesn't seem to be a reliable approach for IFs containing possible interferences.

More investigations on column clean-up are necessary to propose the most robust procedure for different varieties of IF.

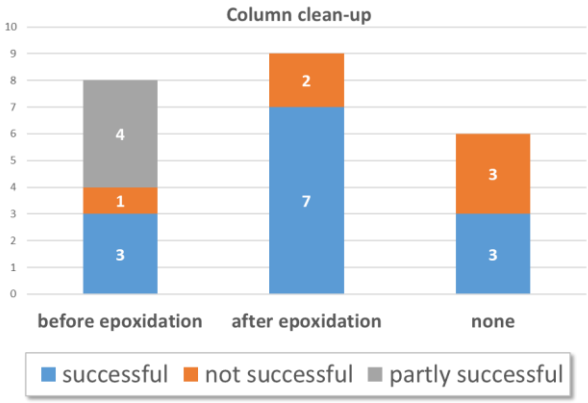


Figure 6. *Resuts obtained with/without column clean-up performed before/after epoxidation*

4.4 Limit of quantification

The limit of quantification (LOQ) for MOAH in IF depends obviously also on the amount of sample (representative for the initial IF sample intake) injected into the chromatographic system and reaching the FID detector. Table 1 presents the injected amounts (calculated by the organisers, based on the answers from the questionnaire) ranging from 0.025 to 1.3 g and resulting in LOQ values ranging from 0.06 to 2 mg/kg per fraction and for total MOAH.

The majority of the laboratories (16) reported LOQs of 0.5 mg/kg per fraction and for total MOAH; 4 laboratories reported no LOQ for total MOAH; 6 laboratories expressed their LOQ for total MOAH as the sum of the LOQs per fractions.

Most of the participants reported the same LOQ for each individual fraction. In general, most of them evaluate different LOQs for different type of IF, depending on the matrix interferences. Some laboratories evaluate the LOQ based on a visual approach (Figure 7, option 2), which indicates the hump when compared to the signal of the blank and takes into account any detected interferences that could not be removed by the procedure. These laboratories report usually different LOQs (i) for different IF samples, depending on the interferences in the sample and/or (ii) for different replicates of the same sample, depending on the blank.

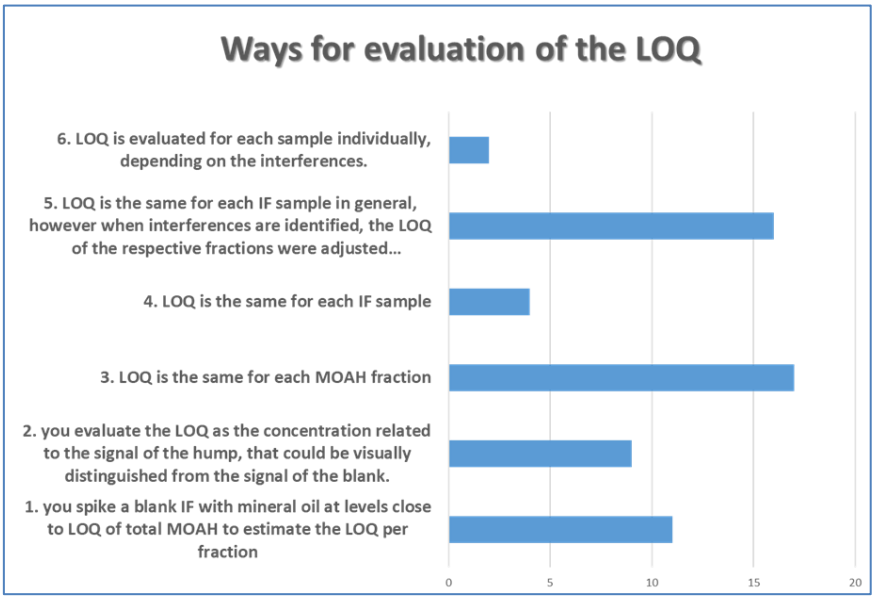


Figure 7 Number of laboratories using a specific approach to evaluate LOQs per MOAH fraction

Table 1. Sample equivalent injected (SEI, in g) in the chromatographic system as calculated by the ILC organizer compared to the reported LOQs per fraction and for total MOAH (expressed in in mg kg^{-1}).

SEI	LOQ C10-C16	LOQ C16-C25	LOQ C25-C35	LOQ C35-C50	LOQ total MOAH
0.24	1	1	1	1	1
0.33	0.5	0.5	0.5	0.5	0.5
0.59	No LOQ	1	1	1	1
0.25	0.5	0.5	0.5	0.5	No LOQ
0.5	0.5	0.5	0.5	0.5	0.5
0.38	0.5	0.5	0.5	0.5	0.5
Not enough data to calculate	0.5	0.5	0.5	0.5	0.5
0.7	0.07	0.07	0.07	0.07	0.07
Not enough data to calculate	0.5	0.5	0.5	0.5	0.5
0.2	0.5	0.5	0.5	0.5	0.5
0.375	0.2	0.2	0.2	0.2	0.8
0.25	0.15	0.15	0.15	0.15	0.15
1.32	0.5	0.5	0.5	0.5	1
0.16	0.5	0.5	0.5	0.5	1
0.27	1	0.5	0.5	0.5	0.5
0.36	0.5	0.5	0.5	0.5	0.5
0.1	No LOQ	No LOQ	No LOQ	No LOQ	No LOQ
0.5	No LOQ	No LOQ	No LOQ	No LOQ	No LOQ
0.025	0.5	0.5	0.5	0.5	No LOQ
0.025	0.04	0.08	0.06	0.07	0.26
0.054	2	2	2	2	2
0.83	0.5	0.5	0.5	0.5	0.5
0.267	0.1	0.1	0.1	0.1	0.5
Not enough data to calculate	0.05	0.05	0.05	0.05	0.2
0.25	0.15	0.15	0.15	0.15	1
0.2	0.2	0.4	0.2	0.4	1.2

4.5 Laboratory experience

Figure 8 indicates that most of the participants have at least five years of experience in the determination of MOSH/MOAH in different type of matrices, such as rice, cereals, oil and fats or paperboards. However, some of them were not used to determine MOAH in infant formula.

It is worth noting that satisfactory results were reported by accredited laboratories as well as participants having only recently implemented their method of analysis, and/or having analysed only a few IF samples in 2019. Only one laboratory reported significantly underestimated values for the five measurands investigated, while claiming to have performed 800 analyses of IF samples over the past 12 months.

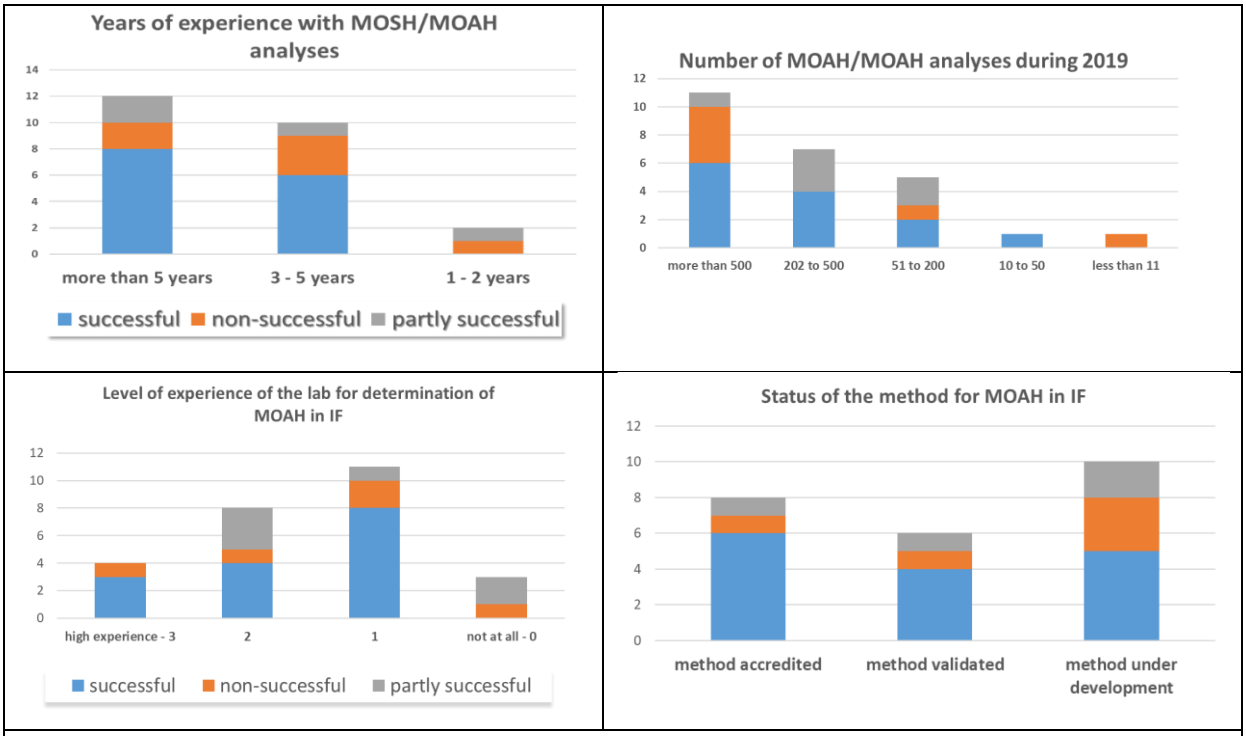


Figure 8: a) years of experience of the laboratories in MOAH analyses;
b) number of MOSH/MOAH analyses in 2019;
c) (self-assessed) level of experience v.s. quality of the reported results;
d) status of the MOAH method of analysis.

5 Conclusions

This successful exploratory interlaboratory comparison was organised for the determination of MOAH in commercial IF. The satisfactory results reported and the experimental details provided in the on-line questionnaire were thoroughly scrutinised to identify the relevant experimental steps to be applied as listed below. An analytical protocol will be drafted and then reviewed also by expert laboratories. Afterwards, this harmonised protocol will be validated in a ring-trial. The resulting standard operating procedure will enable the comparability of MOAH in IF results obtained by different laboratories.

A number of experimental conditions (listed hereafter) remain to be discussed and further agreed on:

	Experimental conditions of the steps in the procedure to be followed	**Parameter to be agreed**
1	hot water reconstitution of the IF – 5 g powder IF in 10 mL hot water at XX °C	temperature ? (40 - 60 °C)
2	Alkaline digestion and saponification of the reconstituted IF with KOH (XX g) in saturated ethanolic solution or 50 % aqueous solution in the presence of ethanol at 60 °C for XX min; preparation of saturated EtOH solution (X g KOH / mL EtOH)	KOH quantity? (1.5 - 3 g)
		saponification time? (30 - 60 min)
3	extraction of the saponified solution with XX mL hexane	V (hexane) ?
4	epoxidation of the organic phase in ethanol with m-CPBA (previously purified/washed in hexane, if needed) at XX °C for XX min ;	40 °C ? 15 min ?
	quantity of m-CPBA (XX g) to be determined depends on whether the column clean-up is before or after the epoxidation	g m-CPBA
5.	Addition of stop reagents - sodium thiosulfate and sodium carbonate - after epoxidation	amount
	washing of the organic phase with ethanol/water after epoxidation required?	Y/N?
6.	Column cleanup and sample enrichment	Before/after epoxidation or both
	column clean-up on XX g of activated silica and elution with DCM/hexane	m(Silica) = 3 or 12 g?
7	0.5 g sample equivalent reaching FID	
8.	Quantification of the MOAH against methyl naphtalenes or TBB	Equivalence to be demonstrated
	Quantification of the total MOAH based on the integration of the entire hump	
9.	Mineral oil solution in hexane with known MOAH content to be proposed as a reference for LOQ determination	Composition /concentration

Acknowledgements

The EURL-FCM acknowledges the contribution of JRC's Reference Material Unit for processing the powder infant formula and delivering promptly high quality proficiency test items, just before the March 2020 lockdown. Furthermore, the 27 laboratories listed hereafter are kindly acknowledged for their participation to this exercise.

Organisation	Country
Graz University of Technology	Austria
Primoris	Belgium
Sciensano	Belgium
ITERG	France
NQAC NESTLE France Laboratory	France
Bavarian Health and Food Safety Authority	Germany
bilacon GmbH	Germany
Bundesinstitut für Risikobewertung (BfR)	Germany
Chemisches und Veterinäruntersuchungsamt Münsterland-Emscher-Lippe (CVUA-MEL)	Germany
CVUA Stuttgart	Germany
Eurofins WEJ Contaminants GmbH	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
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
Nestlé Research	Switzerland
Official Food Control Authority of the Canton of Zurich	Switzerland
Swiss Quality Testing Services	Switzerland

Annex 1: Invitation letter

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

Ref. Ares(2020)1559091 - 13/03/2020

Geel, 13 March 2020

(sent by e-mail)

Subject: Invitation to participate in a ring trial round "JRC IF 2020/01"

Dear all,

Following the conclusions of the Roundtable of 05 December 2019 for further harmonisation, the JRC is launching a study for the evaluation of analytical procedures for the analysis of MOAH in infant formula (IF).

The JRC would appreciate if you would be willing to participate in this important exercise.

A test sample of 25 g IF will be dispatched by the end of the next week. You would be requested to perform the analysis in triplicate, starting always from a new sample aliquot. **The analytical procedures applied should follow the decisions from the Roundtable and the requirements of the "Guidance** on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials on mineral oil hydrocarbons" in the frame of Recommendation (EU) 2017/84. You will receive a detailed questionnaire on the procedure applied by your laboratory. Only test results accompanied by the filled-in questionnaire would be subject to further evaluation.

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=2541

Please, register at the latest by Tuesday, 17th of March 2020 until 9 am.

Please, forward this invitation to all laboratories and organisations that would be interested and competent to participate. They should also register electronically by using the same link above.

Samples will be dispatched on the 17th of March 2020.

The planned deadline for submission of results is the 10th of May 2020.

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

Kind regards,

/signed electronically in Ares/
S. BRATINOVA

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

Annex 2: Instructions to 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, 17th March 2020
Ref. Ares (2020)1614616 - 17/03/2020

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

Subject: Participation in JRC IF 2020/01 – "Determination of MOAH in IF"

Dear «Title» «Surname»,

Thank you for participating in the pre-trial **JRC-IF-2020/01 – "Determination of MOAH in IF".** This round is organised to harmonise the analytical procedure for the analysis of MOAH in infant formula (IF) as discussed in Brussels during the Roundtable of December 5, 2019.

The measurands are mass fractions (mg kg^{-1}) of total MOAH 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 on mineral oil hydrocarbons" in the frame of Recommendation (EU) 2017/84.

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

The parcels are dispatched today. They contain one 100 ml brown glass bottle filled with approximately 26 g powder IF, crimp capped and wrapped in Al foil each.

Upon arrival of this parcel, please check whether the bottle 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.

The procedure used for the analyses should **follow the decisions from the Roundtable of 05 December 2019 and the requirements of the "Guidance on sampling, analysis and data reporting"** (**https://europa.eu/!YF46DN**).

Please report the following:

- the results from the **three replicate** measurements (in mg kg^{-1})
- the **final value** you would report to customers (may be different from the mean of the 3 replicates);
- the associated expanded **uncertainty** of the mean value (in mg kg^{-1});
- the **coverage factor;** and
- the analytical technique used (on-line LC-GC or manual MOSH/MOAH separation).

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

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

The reporting website **https://europa.eu/!QG86xB** will be open on **March 30, 2020.**

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

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

Only results accompanied by the filled-in questionnaire will be taken into account for further evaluation!

At present, the deadline for submission of results is set to **May 10, 2020**.

A report to participants will be circulated shortly after the end of the round to present

(i) the reported values from all participants with their lab codes and
(ii) a proposed SOP to be further validated.

However, the laboratory code will be disclose only to the respective participant, 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 2020/01 Coordinator

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

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

Annex 3: Confirmation of receipt form

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

EURL European Union Reference Laboratory for Food Contact Materials

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

Subject: Participation in JRC IF 2020/01 – "Determination of MOAH in IF"

Please return this form within 3 days of reception, 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: _____/_____/2020

Was the sample damaged? ☐ YES ☐ NO

Remarks

……..

……..

……..

……..

Signature

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

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

Annex 4. Eusurvey – online questionnaire
to collect experimental details used by the participants to analyse MOAH in IF
(https://ec.europa.eu/eusurvey/runner/JRC_IF_2020_01A)

MOAH in Infant Formula

This questionnaire aims to collect the analytical procedure that you used for the determination of MOAH in Infant Formula (IF), in the frame of the present interlaboratory comparison.

Consequently, the JRC intends to identify best practices, that would result in a reliable, robust and harmonised method to be further ring-trial validated.

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

A Previous experience

* A.1 Specify your confidential "Participation Key"

* A.2 Institution

* A.3 Your e-mail address

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

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

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

- ○ less than 10
- ○ 10 to 50
- ○ 51 to 200
- ○ 201 to 500
- ○ more than 500

A.6 Your experience depending on the type of matrices

Classification according to the EURL-FCM Guidance document
0 - no experience
1 - very little experience
2 - moderate experience
3 - high expertise

	Rank (0-3) the matrices you are most experienced in
*Dry, low fat content sample (< 4% oils/fat)	
*Higher fat/oil content sample (> 4 % oils/fat)	
*Oils & Fats	
*Paperboard	
*Infant Formula	

* A.7 Number of samples analysed for MOAH in Infant formula during the past 12 months

* A.8 Status of your method for MOAH in IF

- method under development
- method validated
- method accredited
- other (e.g. direct implementation of an SOP, developed by other lab)

* A.9 Please specify

B Sample preparation

* B.1 Sample intake

g

* B.2 Source of the Internal & Verification Standards

- commercial
- home made from individual compounds

* B.3 Please give reference to the commercial standard. Is it diluted before use?

* B.4 Please describe the Internal & Verification Standards (compounds and concentration levels of the solution used for spiking)

* B.5 solvent used for the IS

* B.6 Volume of the IS solution added to the sample

uL

C Saponification & Extraction

* C.1 Experimental path chosen

- ○ **Reconstitution, Extraction & Saponification simultaneous** (IF + IS + ethanol/water + KOH + organic solvent)
- ○ **Reconstitution and saponification** (IF + IS + ethanol/water + KOH), **followed by extraction** (organic solvent)
- ○ **Reconstitution** (IF + IS + ethanol/water), followed by **extraction** in organic solvent. **Saponification only on the organic extract** (organic phase + KOH + water)
- ○ **Saponification** (IF + IS + KOH) followed by **extraction** (organic solvent) **without reconstitution**
- ○ **Simultaneous saponification and extraction** (IF + IS + KOH + organic solvent) **without reconstitution**
- ○ Other (except for additional saponification and washing as the questions will follow)

* C.2 Intermediate steps

- ○ none
- ○ shaking/heating before adding KOH **without** organic solvent (for reconstitution of the Infant Formula)
- ○ shaking/heating before adding KOH **with** organic phase (for partial fat extraction)
- ○ other

* C.3 Temperature

[ ] oC

* C.4 Time

[ ] min

* C.5 If "other" specify other intermediate steps

[ ]

* C.6 Please describe in detail any other sample treatment path applied by you

[ ]

C.7 Table of the reagent for the saponification&extraction step

Please fill some of the cells with N/A

	Solvent	Concentraction, %	Volume, mL
*water			
*ethanol			
*KOH in			
*organic solvent			

* C.8 Amount of KOH

- ○ Fixed amount for all matrices
- ○ Adjusted to the fat content in the sample

* C.9 Temperature during saponification

oC

* C.10 Time for saponification

min

* C.11 Aqueous/Organic phase mixing during saponification/extraction, ensured by:

- ○ gentle shaking
- ○ vigorous shaking
- ○ ultrasonic
- ○ reflux
- ○ other

* C.12 Please describe if "other"

* C.13 Phase separation, using

- ○ separatory funnels
- ○ only vials & pipettes

* C.14 Please specify the type of pipettes you are using during the procedure

* C.15 Further steps

(multiple choice)

- ☐ none
- ☐ second saponification step on organic phase
- ☐ washing of the organic extract with solvent
- ☐ re-concentration of the organic extract before next step

* C.16 Please describe the second saponification step

C.17 Please specify the solvent for washing

* C.18 How many times

* C.19 Till what volume

mL

* C.20 How is the pre-concentration done?

- flow of nitrogen
- rotavapor
- other

* C.21 Please specify "other"

C.22 Please describe any deviations from the above mentioned steps

D Epoxidation

* D.1 Which part of the extract undergoes epoxidation?

- all the organic phase
- an aliquot of the organic phase

D.2 Please specify what part from the total organic phase is taken for epoxidation

either in fraction number or how many mL from the total extract in mL

* D.3 Epoxidation agent (mCPBA) in which solvent?

- in dichloromethane
- in ethanol

* D.4 Do you purify mCPBA before use?

- No
- Yes

* D.5 How is mCPBA purified?

* D.6 Do you use the same amount of mCPBA for all types of samples with different fat contents?

- Yes
- No

D.7 Table of the reagents for the epoxidation step

for the organic extract the cell concentration should be empty

	solvent	concentration (g/L)	volume added (mL)
*organic extract			
*mCPBA			
*$Na_2S_2O_3$			
*Na_2CO_3			
*other			

* D.8 please specify "other"

* D.9 at what temperature the epoxidation is carried out?

oC

* D.10 for how long?

min

* D.11 Do you wash the organic phase after the epoxidation?

- Yes
- No

* D.12 Please describe the washing step

* D.13 Do you pre-concentrate the organic phase after epoxidation?

- No
- Yes

D.14 till what volume (uL)

* D.15 How is the pre-concentration done?

- blow of nitrogen
- rotavapor
- other

* D.16 Please specify "other"

E Column clean-up

* E.1 Column clean-up

- before epoxidation
- after epoxidation
- none

* E.2 Which part of the organic phase undergoes clean-up?

- all the organic phase
- an aliquot of the organic phase

E.3 Please specify what part from the total organic phase is taken for clean-up

either in fraction number or how many mL from the total extract in mL

* E.4 Type and amount of the sorbents loaded onto the column

* E.5 activated sorbent?

- No
- Yes

* E.6 activated for how long?

min

* E.7 activated at what temperature?

oC

E.8 Sequence of the solvent for washing and elution

In the column "volume collected", please specify only the volume collected for further processing; some cells SHOULD be empty

	Eluent	Volume used (mL)	Volume collected (mL)
1			
2			
3			
4			

* E.9 Do you pre-concentrate the eluate before further processing?

- ○ No
- ○ Yes

* E.10 till what volume ?

mL

* E.11 How is the pre-concentration done?

- ○ flow of nitrogen
- ○ rotavapor
- ○ other

* E.12 Please specify "other"

F Analytical setup for MOSH/MOAH separation

* F.1 Separation performed

- ○ before epoxidation
- ○ after epoxidation

* F.2 Set-up used

- ○ on-line
- ○ semi-online (collection of fractions in auto-sampler & injection into GC from vial)
- ○ off-line

* F.3 Please describe the instrument you used for the analyses

G Manual MOSH/MOAH separation

* G.1 Column used

* G.2 Type of and quantities of the sorbents filled in the column

G.3 Sequence of eluents

In the column "volume collected", please specify only the volume collected for MOSH and MOAH analyses; some cells SHOULD be empty

	Eluent	Volume used (mL)	Volume collected (mL)
1			
2			
3			
4			

* G.4 When you start collection of MOAH fraction, please describe

* G.5 How you control the start and the end of the MOAH fraction

* G.6 Do you pre-concentrate the MOAH fraction?

- No
- Yes

* G.7 till what volume ?

mL

* G.8 How is the pre-concentration done?

- blow of nitrogen
- rotavapor
- other

* G.9 Please specify "other"

H Details of HPLC for the on-line MOSH/MOAH separation

* H.1 Type of the HPLC column used and its dimentions

* H.2 Volume of extract injected

ul

* H.3 Initial eluent used

* H.4 Gradient used

* H.5 When the MOAH fraction collection begins?

- Just after MOSH
- Using the Retention Time of TBB
- Using the Retention Time of DEHB
- Other

* H.6 Please describe "other"

I Details of GC

* I.1 Injection system used

- direct coupling with HPLC
- cold on-column
- PTV with liner
- split
- other

* I.2 if "other", specify

* I.3 Volume injected in GC (in uL)

uL

I.4 Is the total MOAH fraction transfered to the GC via on-line interface

- YES
- NO

* I.5 What part of the total MOAH fraction is injected in the GC port?

Please indicate 1/2; 1/3; 1/4; 1/5 etc...

fraction

* I.6 Type of column and dimentions

* I.7 Type of pre-column

* I.8 Oven temperature program

* I.9 Additional information

(multiple choice)

- ☐ have a retention gap installed
- ☐ vent the injected solvent before entering the separation column

* I.10 Do you have problem with

(multiple choice)

- ☐ the baseline
- ☐ the the peak tailing/broadening
- ☐ the solvent peak
- ☐ the blank
- ☐ interferences

* I.11 Please describe in more detail the problem

J Quantification

* J.1 How do you quantify MOAH? Against which standard?

* J.2 Did you remove any riding peaks ?

* J.3 How do you calculate the total MOAH

- ○ as the sum of different fractions applying the lower bond approach (if less than LOQ then set to zero)
- ○ based on the integration of the entirre chromatogram (from C10 to C50)

* J.4 Peak/Hump integration

- ○ Manual
- ○ Automatic

* J.5 Software used for peak evaluation

* J.6 Baseline/blank compensation

- ○ Manual - blank correction
- ○ Automatic - software compensation

* J.7 If "manual", please explain "how"

* J.8 If "automatic", do you visually check the correctness of the peak/hump integration for EACH sample?

- ○ Yes
- ○ No

K Limit of Quantification (LOQ) and measurement uncertainty (U)

K.1 Please report LOQ for different fractions and the total

	LOQ
1. MOAH ≥ n-C10 to ≤ n-C16	
2. MOAH > n-C16 to ≤ n-C25	
3. MOAH > n-C25 to ≤ n-C35	
4. MOAH > n-C35 to ≤ n-C50	
5. Total MOAH	

* K.2 How do you evaluate the LOQ for the individual fraction of MOAH in IF? Which of the following statement is correct?

Multiple answers are accepted

- ☐ 1. you spike a blank IF with mineral oil at levels close to LOQ of total MOAH to estimate the LOQ per fraction
- ☐ 2. you evaluate the LOQ as the concentration related to the signal of the hump, that could be visually distinguished from the signal of the blank.
- ☐ 3. LOQ is the same for each MOAH fraction
- ☐ 4. LOQ is the same for each IF sample
- ☐ 5. LOQ is the same for each IF sample in general, however when interferences are identified, the LOQ of the respective fractions were adjusted depending on the interferences.
- ☐ 6. LOQ is evaluated for each sample individually, depending on the interferences.

* K.3 What is the spiking level of the mineral oil and the type of the mineral oil? What was the MOAH content in it?

* K.4 Which fractions does it cover?

* K.5 Do you extrapolate the estimated LOQ to the other fractions?

- Yes
- No

* K.6 LOQ for total MOAH is set as:

- max(LOQ) of the various fractions
- sum(LOQ) of the various fractions
- based on the integration of the entire hump
- other

* K.7 Please describe "other"

* K.8 Estimated relative intermediate precision around the LOQ

%

* K.9 How did you estimate the measurement uncertainty of the reported result

- replicate analyses (repeatability)
- validation study (intermediate precision)
- from previous proficiency tests
- expert opinion
- according to Guide for the expression of the Measurement Uncertainty (GUM)
- other

* K.10 Which Quality Control measures do you apply in general during analyses of IF for MOAH?

- criteria for the intensity of peaks - IS and verification standards
- recovery
- reagent blank
- replicate analyses
- QC samples
- PTs for the determination of MOSH/MOAH in other matrices
- other

* K.11 how do you evaluate recovery for the MOAH in IF?

20

* K.12 Do you always analyse replicate sample when the result for MOAH in IF is positive?

* K.13 Specify which QC sample was used with the current analyses

* K.14 Please mention the successful participation in a PT for MOSH/MOAH in other food matreces

* K.15 If "other", specify

L Other

L.1 Any other comment from your side

Thank you for your contribution.
Rest assured that this information will be treated with due confidentiality

MOAH in
Infant Formula

Annex 5. Results as reported by the participants for **total MOAH in IF** (in mg kg^{-1})

LabCode	Result	MU (k=2)	Rep1	Rep2	Rep3	Method
1	2.7	1.1	2.7	2.6	2.7	ON-line LC-GC-FID
2	3	1.4	2.2	3.7	3.1	ON-line LC-GC-FID
3	3.09	0.49	3.17	3.08	3.02	ON-line LC-GC-FID
4	4.1	0.7	4.1	4	4	ON-line LC-GC-FID
5	4.47		4.54	4.476	4.395	ON-line LC-GC-FID
6	2.7	0.8	2.7	2.8	2.4	ON-line LC-GC-FID
7	2.78	0.14	2.82	2.74	2.77	ON-line LC-GC-FID
8	3.67	1.1	3.69	3.64		
9	1.96	0.78	1.94	1.97	1.97	ON-line LC-GC-FID
10	1.42	25	1.6	1.24	1.42	ON-line LC-GC-FID
11	4.919	0				OFF-line GC-FID
12	3.888	1.283	4.313	3.645	3.706	ON-line LC-GC-FID
13	2.62	0.33	2.3	2.95	2.61	ON-line LC-GC-FID
14	0.64		0.59	0.73	0.59	ON-line LC-GC-FID
15	< 1.2		< 1.2	< 1.2	< 1.2	OFF-line GC-FID
16	2.26	0.84	2.08	2.54	2.06	ON-line LC-GC-FID
17	2.47	0.3	2.44	2.39	2.57	ON-line LC-GC-FID
18	3.5	1.4	3.4	3.5	3.6	ON-line LC-GC-FID
19	3.2	1.6	3.1	3.2	3.4	ON-line LC-GC-FID
20	1.7	0.2	1.6	1.8		
21	2.7	1.13	2.9	2.8	2.5	ON-line LC-GC-FID
22	3.1		3.1	3.1	3.1	ON-line LC-GC-FID
23	< 0.5		< 0.5	< 0.5	< 0.5	Semi-ON line LC-GC-FID
24	3.1	0.9	3.1	3.3	3	ON-line LC-GC-FID
25	1.73	0.43	1.72	1.76	1.71	ON-line LC-GC-FID
26	3.3	0.66	3.28	3.43	3.22	ON-line LC-GC-FID
27	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID

Annex 6. Results as reported by the participants for the **MOAH C35-C50 fraction in IF** (in mg kg^{-1})

LabCode	Result	MU (k=2)	Rep1	Rep2	Rep3	Method
1	2	0.8	2.1	2	2.1	ON-line LC-GC-FID
2	2.5	1.4	1.8	3.1	2.5	ON-line LC-GC-FID
3	2.14	0.34	2.19	2.14	2.11	ON-line LC-GC-FID
4	3.2	0.5	3.2	3.1	3.2	ON-line LC-GC-FID
5	3.438		3.5	3.39	3.424	ON-line LC-GC-FID
6	2.1	0.6	2.1	2.2	1.9	ON-line LC-GC-FID
7	2.15	0.1	2.17	2.12	2.17	ON-line LC-GC-FID
8	3.12		3.17	3.07		
9	1.61	0.64	1.61	1.61	1.61	ON-line LC-GC-FID
10	0.88	25	0.98	0.78	0.88	ON-line LC-GC-FID
11	1.759	0				OFF-line GC-FID
12	3.023	0.997	3.369	2.784	2.915	ON-line LC-GC-FID
13	1.85	0.33	1.49	2.13	1.94	ON-line LC-GC-FID
14	0.5		0.37	0.59	0.43	ON-line LC-GC-FID
15	< 0.4		< 0.4	< 0.4	< 0.4	OFF-line GC-FID
16	1.39	0.54	1.23	1.52	1.2	ON-line LC-GC-FID
17	1.93	0.3	1.92	1.82	2.05	ON-line LC-GC-FID
18	2.6	1	2.6	2.6	2.7	ON-line LC-GC-FID
19	2.6	1.3	2.5	2.7	2.7	ON-line LC-GC-FID
20	1.4	0.3	1.6	1.3		
21	1.9	0.8	2	2	1.8	ON-line LC-GC-FID
22	2.4		2.4	2.4	2.4	ON-line LC-GC-FID
23	< 0.5		< 0.5	< 0.5	< 0.5	Semi-ON line LC-GC-FID
24	2.4	0.7	2.4	2.6	2.3	ON-line LC-GC-FID
25	1.39		1.37	1.42	1.38	ON-line LC-GC-FID
26	2.5	0.5	2.46	2.57	2.42	ON-line LC-GC-FID
27	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID

Annex 7. Results as reported by the participants for the **MOAH C25-C35 fraction in IF** (in mg kg^{-1})

LabCode	Result	MU (k=2)	Rep1	Rep2	Rep3	Method
1	0.63	0.25	0.65	0.61	0.63	ON-line LC-GC-FID
2	0.6	0.2	0.4	0.6	0.7	ON-line LC-GC-FID
3	0.95	0.15	0.98	0.94	0.92	ON-line LC-GC-FID
4	0.9	0.2	0.8	0.9	0.8	ON-line LC-GC-FID
5	1.032		1.04	1.086	0.971	ON-line LC-GC-FID
6	0.6	0.2	0.6	0.6	0.5	ON-line LC-GC-FID
7	0.58	0.14	0.63	0.57	0.55	ON-line LC-GC-FID
8	0.54		0.52	0.56		
9	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
10	0.54	25	0.62	0.46	0.54	ON-line LC-GC-FID
11	2.013	0				OFF-line GC-FID
12	0.852	0.281	0.934	0.835	0.788	ON-line LC-GC-FID
13	0.77	0.09	0.82	0.82	0.67	ON-line LC-GC-FID
14	< 0.15		0.19	0.13	0.12	ON-line LC-GC-FID
15	< 0.2		< 0.2	< 0.2	< 0.2	OFF-line GC-FID
16	0.38	0.19	0.32	0.41	0.37	ON-line LC-GC-FID
17	0.54	0.2	0.51	0.57	0.53	ON-line LC-GC-FID
18	< 1		< 1	< 1	< 1	ON-line LC-GC-FID
19	0.6	0.3	0.6	0.5	0.6	ON-line LC-GC-FID
20	< 0.5		0.41	0.51		
21	0.82	0.34	0.84	0.86	0.77	ON-line LC-GC-FID
22	< 1		< 1	< 1	< 1	ON-line LC-GC-FID
23	< 0.5		< 0.5	< 0.5	< 0.5	Semi-ON line LC-GC-FID
24	0.7	0.2	0.6	0.7	0.8	ON-line LC-GC-FID
25	0.34		0.35	0.34	0.33	ON-line LC-GC-FID
26	0.8	0.16	0.82	0.85	0.8	ON-line LC-GC-FID
27	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID

Annex 8. Results as reported by the participants for the **MOAH C16-C25 fraction in IF** (in mg kg^{-1})

LabCode	Result	MU (k=2)	Rep1	Rep2	Rep3	Method
1	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
2	< 0.08		< 0.05	< 0.13	< 0.06	ON-line LC-GC-FID
3	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
4	< 0.1		< 0.1	< 0.1	< 0.1	ON-line LC-GC-FID
5						ON-line LC-GC-FID
6	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
7	< 0.07		< 0.07	< 0.07	< 0.07	ON-line LC-GC-FID
8	0.01					OFF-line GC-FID
9						ON-line LC-GC-FID
10	< 0.05		< 0.05	< 0.05	< 0.05	ON-line LC-GC-FID
11	1.146					
12						ON-line LC-GC-FID
13	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
14	< 0.3		< 0.3	< 0.3	< 0.3	ON-line LC-GC-FID
15	< 0.4		< 0.4	< 0.4	< 0.4	OFF-line GC-FID
16	< 0.15		< 0.15	< 0.15	< 0.15	ON-line LC-GC-FID
17	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
18	< 1		< 1	< 1	< 1	ON-line LC-GC-FID
19	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
20	< 0.5		< 0.5	< 0.5		
21	< 0.3		< 0.3	< 0.3	< 0.3	ON-line LC-GC-FID
22	< 1		< 1	< 1	< 1	ON-line LC-GC-FID
23	< 0.5		< 0.5	< 0.5	< 0.5	Semi-ON line LC-GC-FID
24	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
25	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
26	< 0.2		< 0.2	< 0.2	< 0.2	ON-line LC-GC-FID
27	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID

Annex 9. Results as reported by the participants for the **MOAH C10-C16 fraction in IF** (in mg kg^{-1})

LabCode	Result	MU (k=2)	Rep1	Rep2	Rep3	Method
1	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
2	< 0.04		< 0.03	< 0.07	< 0.03	ON-line LC-GC-FID
3	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
4	< 0.1		< 0.1	< 0.1	< 0.1	ON-line LC-GC-FID
5						ON-line LC-GC-FID
6	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
7	< 0.07		< 0.07	< 0.07	< 0.07	ON-line LC-GC-FID
8						
9						ON-line LC-GC-FID
10	< 0.05		< 0.05	< 0.05	< 0.05	ON-line LC-GC-FID
11						OFF-line GC-FID
12						ON-line LC-GC-FID
13	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
14	< 0.15		< 0.15	< 0.15	< 0.15	ON-line LC-GC-FID
15	< 0.2		< 0.2	< 0.2	< 0.2	OFF-line GC-FID
16	< 0.15		< 0.15	< 0.15	< 0.15	ON-line LC-GC-FID
17	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
18	< 1		< 1	< 1	< 1	ON-line LC-GC-FID
19	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
20	< 1		< 1	< 1		
21	< 0.3		< 0.3	< 0.3	< 0.3	ON-line LC-GC-FID
22	< 1		< 1	< 1	< 1	ON-line LC-GC-FID
23	< 0.5		< 0.5	< 0.5	< 0.5	Semi-ON line LC-GC-FID
24	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
25	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID
26	< 0.2		< 0.2	< 0.2	< 0.2	ON-line LC-GC-FID
27	< 0.5		< 0.5	< 0.5	< 0.5	ON-line LC-GC-FID

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