



JRC TECHNICAL REPORT

Determination of MOAH in infant formula

JRC IF 2022-05 – the ring trial validation study

Bratinova S., Robouch P.,
Beldi G., Senaldi C., Karasek L., Gonçalves C.,
Valzacchi S., Garcia-Ruiz S., Hoekstra E.

2023



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

Contact information
Stefanka Bratinova
European Commission
Joint Research Centre (JRC)
Retieseweg 111
2440 Geel, Belgium
Email: JRC-EURL-FCM@ec.europa.eu
Tel.: +32 14 571800

EU Science Hub
<https://ec.europa.eu/jrc>

JRC 133243

EUR 31476 EN

PDF ISBN 978-92-68-01815-6 ISSN 1831-9424 [doi:10.2760/606967](https://doi.org/10.2760/606967) KJ-NA-31-476-EN-N

Luxembourg: Publications Office of the European Union, 2023

© European Union, 2023



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

For any use or reproduction of photos or other material that is not owned by the European Union/European Atomic Energy Community, permission must be sought directly from the copyright holders.

How to cite this report: Bratinova S., Robouch P., Beldi G., Senaldi C., Karasek L., Gonçalves C., Valzacchi S., Garcia-Ruiz S., Hoekstra E., *Determination of MOAH in Infant Formula*, JRC IF 2022-05 – the ring trial validation study, Publications Office of the European Union, Luxembourg, 2023, doi:10.2760/606967, JRC133243

Table of Contents

Executive summary	1
1 Introduction.....	2
2 Scope.....	2
3 Set-up of the exercise	2
4 Results and Discussions.....	6
4.1 Ring trial.....	6
4.2 Statistical analysis.....	6
4.3 Performance characteristics	7
4.4 Additional information	13
5 Conclusions.....	17
Acknowledgements.....	18
References.....	19
Annex 1: Homogeneity study (all values in mg kg ⁻¹).....	20
Annex 2. Invitation letter.....	21
Annex 3. SOP	22
Annex 4. Instructions to the participants.....	23
Annex 5: Results as reported by the participants.....	26
Annex 6: Chromatograms' compilation.....	72

Executive summary

A ring trial validation study for the determination of mineral oil aromatic hydrocarbons (MOAH) in infant formula (IF) "JRC-IF-2022/05" was organised by the European Union Reference Laboratory for food contact materials (EURL-FCM) upon request by DG SANTE. The goal was to provide a harmonised method and, therefore, to increase the comparability of the results between the laboratories performing the analyses.

Fifteen test items were prepared at the Joint Research Centre (JRC) using materials provided by Special Nutrition Europe (SNE) and produced at the Nestle pilot plant. Homogeneity and stability were proven by the JRC and the test items were distributed for analysis to 26 participants from 8 European countries and China. Participants were requested to apply strictly the standard operating procedure (SOP) provided, i.e. no deviations allowed, and to report mass fractions of total MOAH and mineral oil saturated hydrocarbons (MOSH) for each sample. This report presents the satisfactory method performance characteristics obtained for MOAH level above 1 mg/kg, and the indicative values determined for MOSH.

1 Introduction

Following the Rapid Alert System for Food and Feed (RASFF) notification message 2019.3734 (dated 25/10/2019) [1] and the Foodwatch report [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 harmonise the method for determination of MOAH in IF.

Several interlaboratory comparisons were organised by the JRC in the past three years:

- i. To assess the initial performance of laboratories in determining the mass fraction of total MOAH in an infant formula sample (JRC IF 2020-01 [3]);
- ii. To check the suitability of a draft Standard Operating Procedure (SOP) proposed to quantify MOAH in IF down to 2-3 mg/kg (JRC IF 2020-02 [4]);
- iii. To characterise a reference mineral oil material Shell SN500* used to spike IF samples, and to evaluate the performance of on-line LC-GC-FID instrumental systems applied for the analyses JRC IF 2021-03 [5]; and
- iv. To evaluate the ability of laboratories to integrate predefined mineral oil saturated hydrocarbons (MOSH) and MOAH chromatograms (virtual intercomparison **JRC IF 2021-04** [6]).

Based on the experience acquired and the advice of several national experts in the field, an improved SOP was drafted by the JRC. This report presents the outcome of the ring-trial validation study (JRC IF 2022-05) organised in 2022 where the improved SOP was used for the determination of the total mass fraction of MOAH (and MOSH) in various IF formulations.

2 Scope

The scope of this ring-trial validation study (JRC IF 2022-05) was to establish the performance characteristics (e.g. repeatability, reproducibility, recovery) of the improved SOP for determination of MOAH in IF.

3 Set-up of the exercise

To achieve the challenging goal, the JRC organised four collaborative pre-trials over the period of 3 years (2020-2022) [3-6]. ILC JRC IF 2022-05 is the final collaborative trial of the MOAH in IF project.

Test materials preparation

At the beginning of the project, the JRC collaborated with Special Nutrition Europe (SNE) to produce tailored test materials (i) based on different formulations (prone or not to chromatographic interferences), (ii) containing different MOAH contents (iii) in amounts suitable for running a method validation ring trial.

It was then decided to spike a blank IF matrix with different recipes representing easy or challenging analytical scenarios. Three IF recipes were identified by the SNE members as representative of the market. They were spiked with one type of mineral oil (MO) only. It was chosen based on the MOAH volatility profile in the contaminated IF from the occurrence data. No MO with the required volatility range of the hydrocarbons was commercially available. Hence, a tailor-made

distillation fraction was provided to SNE by an oil refinery (Shell SN500*), having a large percentage of high molecular weight MOAH.

The following strategy was designed in collaboration with the SNE representatives and adopted:

- I. August 2020 - Five bulk IF materials of different compositions were produced at the Nestle pilot plant:
 - **Rapeseed oil based IF** blank material (should not contain MOAH), with little expected chromatographic interferences for the MOAH determination (**BL1**)
 - **Palm oil based IF** blank material (should not contain MOAH) - a “worst case” matrix, resulting in complex chromatograms with interferences (**BL2**)
 - **Rapeseed oil based IF** blank material (should not contain MOAH) with **hydrolysed protein** milk used in hypoallergenic infant formulas (**HABL**)
 - **BL1 spiked** with 50 mg kg⁻¹ SN500 mineral oil (**BL1SP**). The spiking was performed in the oil ingredient before mixing and spray drying of the IF, at a pilot plant, to ensure a homogeneous distribution of the mineral oil in the spiked material
 - **HABL spiked** with 50 mg kg⁻¹ SN500 mineral oil (**HASP**). The spiking was performed in the oil ingredient before mixing and spray drying of the IF, at the pilot plant, to ensure a homogeneous distribution of the mineral oil in the spiked material
- II. In August 2021, the JRC Reference Material Unit blended BL1, BL2 and HABL with the BL1SP and HASP to produce a total of 15 test items (Table 1) with different MOAH level to be used in the collaborative trial for method validation. Since BL1SP contained a high amount of MO, blending small aliquots of this did not change significantly the BL2 matrix composition. The bulk materials mentioned above were spiked with the well-characterised reference material Shell SN500* consisting of 60.1 % MOSH and 36.0 % MOAH¹ [5]. The added MOAH contents are presented in Table 1 for each of the test items.
- III. The JRC homogenised the newly produced materials and filled 100 ml amber glass bottles each with 40 g of powder. All necessary measures were taken to prevent cross-contamination:
 - the bottles were baked before filling at 400 °C for at least 6 h;
 - the crimp caps 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 contaminations during the shipment and storage.
- IV. In January 2022, the JRC assessed the homogeneity of the test items before dispatch to the participants. Seven bottles per test item were randomly selected and two replicates from each bottle were analysed using on-line LC-GC/FID. Results were evaluated according to ISO 13528:2015 [7] and the test items were proven to be adequately homogeneous (Annex 1)

¹ The remaining 3.9 % are polar compounds that are retained on the HPLC column before the GC.

Test item name		MOAH added	used in the previous ILC as
		mg/kg	
IF03A	BL1 - rapeseed oil based	-	
IF03D	BL1+BL1SP	0.75	
IF03E	BL1+BL1SP	1.21	
IF03F	BL1+BL1SP	2.03	
IF03G	BL1+BL1SP	3.30	IF02A - JRC 2020/02
IF03B	BL2 - palm oil based	-	
IF03H	BL2+BL1SP	1.17	
IF03I	BL2+BL1SP	2.43	
IF03J	BL2+BL1SP	3.60	IF02B - JRC 2020/02
IF03K	BL2+BL1SP	7.80	
IF03C	HA BL - rapeseed hydrolyzed protein	-	
IF03L	HABL+HASP	1.20	
IF03M	HABL+HASP	2.40	
IF03N	HABL+HASP	8.10	
IF03Q	commercial IF, sunflower oil based	-	IF01 - JRC 2020/01

Table 1. Test items for the method validation study (MVS). The added MOAH content was calculated based on the reference value of 36.0 % for the MOAH content in the Shell SN500* [5].

Confidentiality

The procedures used for the organisation of this ring-trial guarantee that the identity of the participants and the information provided by them are treated as confidential. The participants to this round received a unique laboratory code used throughout this report.

Time frame

JRC IF 2022-05 was formally announced by e-mail on February 16, 2022² (Annex 2). The proposed SOP was sent to the interested participants in December 20 (Annex 3). All samples were dispatched on March 28, 2022 to the registered participants. Initially, the deadline for the reporting of results was set to May 22, 2022. However, the deadline was extended to June 20, 2022 due to several requests received from the laboratories.

Distribution

Each participant received:

- Fifteen test items containing approx. 40 g of powder;
- The standard operating procedure (Annex 3)
- The "Instruction to participants" (Annex 4).

² The first announcement was done in 2020 when presenting the collaborative trials foreseen in the frame of the IF study.

Instructions to participants

Detailed instructions were provided to the participants by e-mail (Annex 4). They were requested to apply the experimental protocol described in the SOP (Annex 3) strictly.

The measurands, expressed in mg/kg, were defined as:

- (i) the **mass fraction of total MOAH (C10-C50) in IF**; and
- (ii) the **mass fraction of total MOSH (C10-C50) in IF**.

Participants were requested to analyse each sample (bottle) in **duplicate** and report:

- any deviations from the prescribed SOP;**
- the mass fraction of **total MOAH (C10-C50) calculated vs 2MN;**
- the mass fraction of **total MOAH (C10-C50) calculated vs TBB;**
- the mass fraction of **total MOSH (C10-C50) calculated vs CyCy;**
- the associate **relative expanded uncertainties** (expressed in %, coverage factor k=2)
- the mass fraction of the **C50+ for MOSH and MOAH (vs TBB)**, expressed in mg/kg;
- the “2MN/1MN”, “2MN/TBB”, “5B/TBB”, “CyCy/TBB”, “C20/C50” **ratios;**
- the concentration of **total MOSH and total MOAH in JRC QC10-1** -Shell SN500* in hexane, sample sent as a quality control sample to the participants in July 2021, JRC IF 2021-03 [5]; , expressed in mg/L;
- the **content of the sub-fraction cuts** (as defined in the EURL guide [8]) for samples IF03G and IF03J (**vs TBB**), expressed in mg/kg.

Participants were requested to report all quantifiable results for total MOSH and total MOAH, based on the integration of the **entire hump** for the range C10-C50, but to highlight in red those results that they would normally not report to the customer.

In addition, the following chromatograms (screenshots) were requested to be sent, together with the Reporting form, to show the integration of the chromatograms, riding peaks and background compensation:

- overlaid original chromatograms of IF03 (A, B, D, E, H, I) each with hexane and the reagent blank, properly scaled to show clearly the hump;
- chromatograms of IF03 (E, I) properly scaled, to show (i) the trimmed riding peaks and (ii) the baseline subtracted from the hump.

4 Results and Discussions

4.1 Ring trial

Twenty-eight laboratories registered to the exercise, representing a broad variety of stakeholders (e.g. National Reference Laboratories (NRLs), Official Control Laboratories (OCLs), commercial, or industrial or university laboratories) (Figure 1).. Twenty-six of them - from 8 European countries and China – reported results for MOAH and MOSH. –

The reported results and the corresponding graphs are presented in Annex 5.

All participants were familiar with the different steps included in the SOP to be applied, since they participated in the pre-trial study [4]. No major deviations from the prescribed SOP were identified.

Most of the participants were experienced in the field, since they are analysing 200 to 40000 samples per year, covering a broad variety of matrices, such as oil and fats, dry food and paperboard, while few laboratories analysed 20 to 30 samples per year only. In the last two years, laboratory L11 performed no MOSH/MOAH analyses, while L12 did not analyse any IF samples. Seventeen laboratories were accredited for MOSH/MOAH analyses in different commodities, according to ISO 17025.

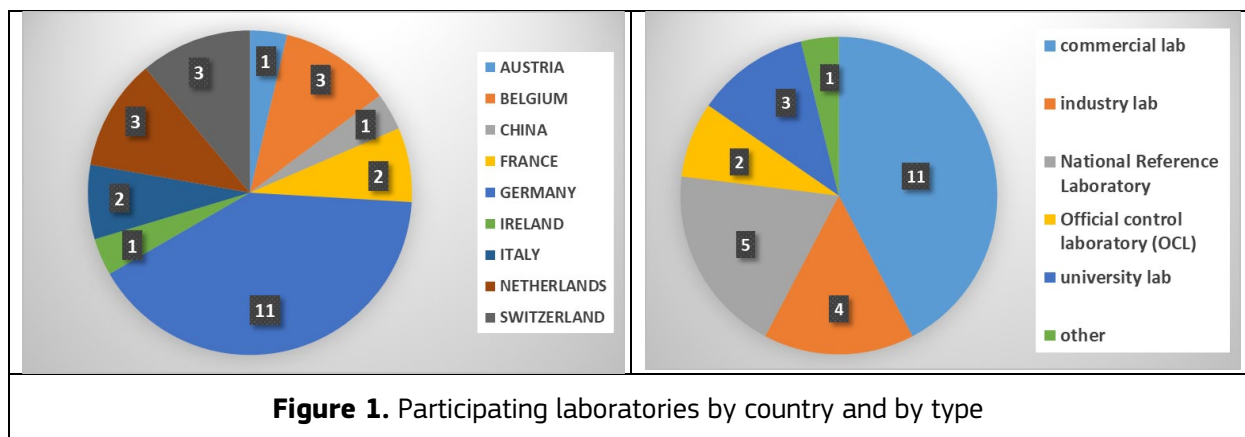


Figure 1. Participating laboratories by country and by type

4.2 Statistical analysis

Statistical evaluation of the data was performed following international standard recommendations (ISO 5725-2:2019 [9]). The commercial software PROLab developed by QUODATA [10] was used to perform the statistical treatment and the following tests were performed:

- ✓ Check for outliers in the laboratory precision (variance) applying the Cochran test. This test compares the highest laboratory internal repeatability variance with the sum of reported variances from all the participants;
- ✓ Check for laboratory outliers within the series of independent replicates applying the Grubbs-internal test (repeatability). This test is of particular relevance for laboratories being flagged as stragglers by the Cochran test;
- ✓ Check for outliers in the laboratory mean applying the Grubbs test. This test checks for laboratory means deviating significantly from the total mean calculated from data reported from all participants.

Method performance characteristics (repeatability and reproducibility) were estimated after the identification and elimination (if applicable) of outlier results. Outlying data were investigated and discarded, in line with ISO 5725-2. The remaining number of valid data sets was 22 to 25 for each of the measurand.

4.3 Performance characteristics

In order to monitor the behaviour and the extraction efficiency of the two internal standards (2MN and TBB) used for quantification of the MOAH fraction, participants were asked to quantify and report two MOAH values (MOAH-2MN and MOAH-TBB). The performance characteristics were evaluated for both sets of data. In addition, even though the harmonised SOP did not target explicitly the MOSH fraction, the results for MOSH obtained (after epoxidation that removes part of the interfering unsaturated hydrocarbons but without any ALOX clean-up) were also evaluated.

The 22 to 25 valid data sets reported by laboratories were used to derive the method performance characteristics for the determination of (i) MOAH-2MN, (ii) MOAH-TBB (see Table 2 and 3), and (iii) MOSH-CyCy (indicative values, Table 4). All the values were confirmed by an alternative approach based on robust statistics, which do not require the exclusion of outliers.

The results submitted by laboratory Lab 11 were excluded from the final calculation, due to the lack of experience in the field and several experimental problems mentioned by the laboratory. Similarly, the results of L02 and L15 were flagged as outliers. Lab15 may have swapped some of the samples.

For L12 the low values were due to the very unfavourable C50:C20 ratio of 0.37, which had an adverse effect on the quantification of MOAH in the samples with high boiling MO fractions. Nevertheless, the data from this lab were not removed, since they were not identified as outliers during the tests. Unusually high variations in the ratio TBB/2MN (L25) and TBB/CyCy (L02) across the sample set were observed for two labs, suggesting unreproducible extraction of the IS in hexane after saponification (TBB/2MN) or unreproducible epoxidation (TBB/CyCy). Only the values identified as outliers for these two labs were removed from the dataset for performance evaluation.

The following observations derived from Tables 2-4 are worth noting:

- Three blank samples (IF03A-C) were provided, containing reasonably low mass fractions of total MOAH (below 0.6 mg/kg), but significantly higher levels of total MOSH (from 5 to 7 mg/kg).
- Four formulations based on rapeseed- (IF03D-G and IF03L-N), palm- (IF03H-K) or sunflower (IF03Q) oils, resulted in the 12 additional samples that were distributed.
- The mass fractions of total MOAH above 1 mg/kg and total MOSH above 5 mg/kg (due to the high MOSH content in the blank samples used) were investigated.
- The calculated mass fractions of total [MOAH-2MN] and total [MOAH-TBB] are strongly correlated, with a correlation coefficient $R^2 > 0.999$, and a slope of 1.13 (Figure 2), which implies that the total [MOAH-TBB] is 13 % lower than total [MOAH-2MN]. A similar correlation was observed in the interlaboratory comparison for the determination of MOSH/MOAH content in edible oils and fats [report to be published]. However, this should be considered with caution. Figure 2 shows that the TBB to 2MN ratio varies considerably (from 0.9 to 1.4) between laboratories or even within a single laboratory (L25).
- Similar precisions are derived for the determination of total MOAH-2MN and total MOAH-TBB (Figure 4).

- The observed relative standard deviations for **repeatability** (RSD_r) – when excluding the blank samples - ranged from 5 % to 13 % for total MOAH, and from 4 % to 14 % for total MOSH.
- The observed relative standard deviations for **reproducibility** (RSD_R) - when excluding the blank samples - ranged from 13 % to 32 % for total MOAH and from 13 to 23 % for total MOSH. Despite the different formulations investigated (including the challenging palm oil based matrix), the uniform decrease of RSD_R with increasing mass fraction of total MOAH confirms the expected Horwitz trumpet as shown in Figure 5.
- Based on the known spiking values (Table 1), the **recoveries** were calculated after subtracting the content of the sample blanks (Table 4). As expected, recoveries ranging from 90 % to 110 % were obtained for total MOSH in infant formula, since no ALOX clean-up was applied. On the contrary, significantly lower recoveries were obtained for MOAH-2MN (ranging from 60 % to 70 %) and MOAH-TBB (52-63%), which may be attributed to the loss of MOAH during epoxidation. The superior MOAH recovery when quantified versus 2MN could be explained with the slight compensation for the MOAH losses during epoxidation by the not full 2MN extraction into the hexane from the saponified solution under the condition of full saponification. On the other hand, it is known that the MOAH recoveries values depend not only on the composition of the matrix but also on the composition of the MO contaminating the matrix (spiked). Therefore, no uniform recovery factor can be assigned and applied.
- The acceptance criteria recommended by CEN ($HorRat \leq 2$) was met for all the spiked samples (IF03D-Q), which demonstrates that the **investigated method is suitable for the determination of the mass fraction of total MOAH in infant formula above 1 mg/kg, regardless of the infant formula composition explored.**
- Higher HorRat values were observed for the blank samples (IF03A-C) containing low levels of MOAH (close to or below 0.5 mg/kg). In this context, the laboratories were requested to report their “calculated” mass fractions, even if in routine they would have reported < LOQ. In fact, 11 laboratories (out of 26) clearly stated that they would have reported values < 0.5 mg/kg for the blank samples IF02A and IF03C, and 9 labs would have reported < 0.5 mg/kg for IF03B.

Acknowledged method validation limitations

- ❖ Only Shell SN500* (a high boiling point mineral oil) was used to spike all bulk materials, as indicated in the test material preparation section, which may have facilitated the proper identification of the hump profile in samples containing low levels of total MOAH. In routine, higher RSD could be expected in the lower working range levels, especially < 1.5 mg/kg ;
- ❖ Only four types of matrices were investigated (rapeseed-, sunflower-, palm-, or hypo-allergenic palm oil based), as they were considered to represent most of the infant food formulations.
- ❖ **Only “indicative” performance characteristics are presented for MOSH** (Table 3) since the SOP investigated was initially designed for the quantification of total MOAH in infant formula. All the mass fractions for total MOSH were reported after epoxidation but without ALOX clean-up of interfering naturally occurring n-alkanes. All IF samples investigated showed MOSH chromatograms with clearly identifiable humps where the trimming of the riding peak was fairly straightforward.

Table 2. Method performance characteristics for the determination of the mass fraction of total **MOAH-2MN** in infant formula

matrix sample	BL1	BL2	HABL	Rapeseed oil based				Palm oil based				HA, Rapeseed oil based			Sunflower oil based
	IF03A	IF03B	IF03C	IF03D	IF03E	IF03F	IF03G	IF03H	IF03I	IF03J	IF03K	IF03L	IF03M	IF03N	IF03Q
N_{tot}	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25
N_G											1			1	
N_C	2	1							1	2			1		1
N	23	24	25	25	25	25	25	25	24	23	24	25	24	24	24
x (mg/kg)	0.44	0.55	0.37	0.92	1.26	1.77	2.49	1.38	2.15	2.88	5.33	1.11	1.82	5.15	3.92
s_r (mg/kg)	0.07	0.12	0.03	0.08	0.11	0.24	0.18	0.14	0.12	0.16	0.35	0.10	0.14	0.36	0.22
RSD_r	17%	22%	9.0%	8.1%	9.1%	13%	7.3%	9.8%	5.7%	5.5%	6.6%	9.4%	7.9%	7.0%	5.7%
r (mg/kg)	0.21	0.34	0.09	0.21	0.32	0.66	0.51	0.38	0.35	0.44	0.99	0.29	0.40	1.01	0.63
s_R (mg/kg)	0.29	0.29	0.19	0.29	0.34	0.40	0.42	0.37	0.46	0.50	0.73	0.27	0.33	0.69	0.70
RSD_R	66%	52%	51%	32%	27%	23%	17%	27%	21%	17%	14%	24%	18%	13%	18%
R (mg/kg)	0.81	0.80	0.53	0.82	0.95	1.12	1.18	1.05	1.28	1.41	2.05	0.75	0.91	1.94	1.95
RSD _{HR}	18%	17%	19%	16%	15%	15%	14%	15%	14%	14%	12%	16%	15%	13%	13%
HorRat	3.6	3.0	2.7	2.0	1.8	1.5	1.2	1.8	1.5	1.3	1.1	1.5	1.2	1.1	1.4

Table 3. Method performance characteristics for the determination of the mass fraction of total **MOAH-TBB** in infant formula

sample matrix	BL1	BL2	HABL	Rapeseed oil based				Palm oil based				HA, Rapeseed oil based			Sunflower oil based
	IF03A	IF03B	IF03C	IF03D	IF03E	IF03F	IF03G	IF03H	IF03I	IF03J	IF03K	IF03L	IF03M	IF03N	IF03Q
N_{tot}	25	25	25	25	25	25	25	25	25	25	25	25	25	25	25
N_G											1		1		1
N_C	1	2	1		2				1	1			2	2	
N	24	23	24	25	23	25	25	25	24	24	24	25	22	23	24
x (mg/kg)	0.40	0.47	0.31	0.81	1.06	1.54	2.18	1.21	1.87	2.56	4.72	1.00	1.55	4.57	3.40
s_r (mg/kg)	0.07	0.10	0.02	0.07	0.05	0.16	0.14	0.12	0.11	0.16	0.30	0.10	0.09	0.19	0.14
RSD_r	18%	22%	7.7%	8.7%	4.3%	11%	6.2%	9.9%	5.9%	6.4%	6.3%	9.9%	5.7%	4.2%	4.1%
r (mg/kg)	0.20	0.29	0.07	0.20	0.13	0.45	0.38	0.34	0.31	0.46	0.83	0.28	0.25	0.53	0.39
s_R (mg/kg)	0.26	0.26	0.16	0.27	0.28	0.35	0.36	0.34	0.42	0.53	0.68	0.30	0.26	0.74	0.58
RSD_R	64%	55%	53%	34%	26%	23%	16%	28%	22%	21%	14%	30%	17%	16%	17%
R (mg/kg)	0.73	0.73	0.45	0.77	0.78	0.97	0.99	0.94	1.17	1.50	1.91	0.83	0.72	2.06	1.61
RSD _{HR}	18%	18%	19%	17%	16%	15%	14%	16%	15%	14%	13%	16%	15%	13%	13%
HorRat	3.5	3.1	2.8	2.1	1.7	1.5	1.1	1.8	1.5	1.5	1.1	1.9	1.1	1.3	1.3

Table 4. Indicative method performance characteristics for the determination of mass fraction of total **MOSH in infant formula**

matrix sample	BL1	BL2	HABL	Rapeseed oil based				Palm oil based				HA, Rapeseed oil based			Sunflower oil based
	IF03A	IF03B	IF03C	IF03D	IF03E	IF03F	IF03G	IF03H	IF03I	IF03J	IF03K	IF03L	IF03M	IF03N	IF03Q
N	22	24	25	25	25	25	23	25	25	24	24	24	24	24	21
x (mg/kg)	5.9	6.4	5.0	7.2	8.1	9.2	11	8.2	10	12	19	6.8	9.2	18	10
RSD _r	8%	6%	7%	6%	7%	10%	5%	7%	6%	7%	4%	8%	8%	5%	5%
RSD _R	27%	23%	22%	23%	19%	20%	17%	15%	16%	17%	13%	22%	23%	18%	14%
HorRat	2.2	1.9	1.7	1.9	1.7	1.8	1.6	1.3	1.4	1.5	1.3	1.8	2.0	1.8	1.2

List of notations used in Tables 2-4:

N _{tot}	No. of laboratories that submitted results
N _G	No. of Type B outliers (Grubbs)
N _C	No. of Type C outliers (Cochran)
N	No. of laboratories after elimination of outliers
x (mg/kg)	Mean value
s _r (mg/kg)	Standard deviation for repeatability
RSD _r	Relative standard deviation for repeatability
r (mg/kg)	Limit of repeatability, $r = 2.8 s_r$
s _R (mg/kg)	Standard deviation for reproducibility
RSD _R	Relative standard deviation for reproducibility
R (mg/kg)	Limit of reproducibility, $R = 2.8 s_R$
RSD _{HR}	Horwitz relative standard deviation for reproducibility
HorRat	Horrat value = RSD_R / RSD_{HR} (unit less)

Table 5. Recovery for total MOSH and total MOAH from infant formula samples

	MOSH-CyCy				MOAH-TBB				MOAH-2MN			
	total mg/kg	total-blk mg/kg	added mg/kg	Recovery %	total mg/kg	total-blk mg/kg	added mg/kg	Recovery %	total mg/kg	total-blk mg/kg	added mg/kg	Recovery %
IF03A	5.90				0.40				0.44			
IF03D	7.24	1.34	1.25	107%	0.81	0.40	0.75	54%	0.92	0.47	0.75	63%
IF03E	8.13	2.23	2.01	111%	1.06	0.66	1.21	54%	1.26	0.81	1.21	67%
IF03F	9.17	3.27	3.39	97%	1.54	1.14	2.03	56%	1.77	1.32	2.03	65%
IF03G	11.18	5.28	5.50	96%	2.18	1.77	3.30	54%	2.49	2.05	3.30	62%
IF03B	6.41				0.47				0.55		0.00	
IF03H	8.19	1.78	1.95	91%	1.21	0.74	1.17	63%	1.38	0.83	1.17	71%
IF03I	10.34	3.93	4.05	97%	1.87	1.40	2.43	58%	2.15	1.60	2.43	66%
IF03J	12.40	6.00	6.00	100%	2.56	2.09	3.60	58%	2.88	2.33	3.60	65%
IF03K	18.74	12.33	13.00	95%	4.72	4.25	7.80	54%	5.33	4.78	7.80	61%
IF03C	4.97				0.31				0.37		0.00	
IF03L	6.81	1.84	2.00	92%	1.00	0.69	1.20	58%	1.11	0.74	1.20	62%
IF03M	9.16	4.19	4.00	105%	1.55	1.25	2.40	52%	1.82	1.45	2.40	61%
IF03N	17.90	12.93	13.50	96%	4.57	4.26	8.10	53%	5.15	4.78	8.10	59%
			average	99%				56%				64%

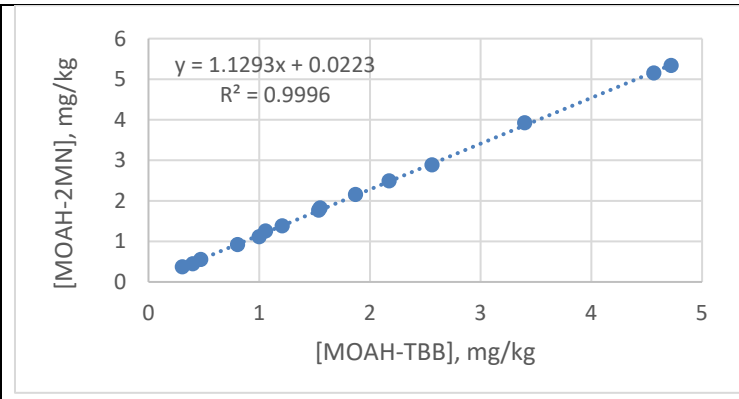


Figure 2.
Correlation between mass fractions MOAH-2MN vs MOAH-TBB, with mean values derived from Tables 1-2.

Figure 3.
Variations in the TBB/2MN ratio for each sample per participant

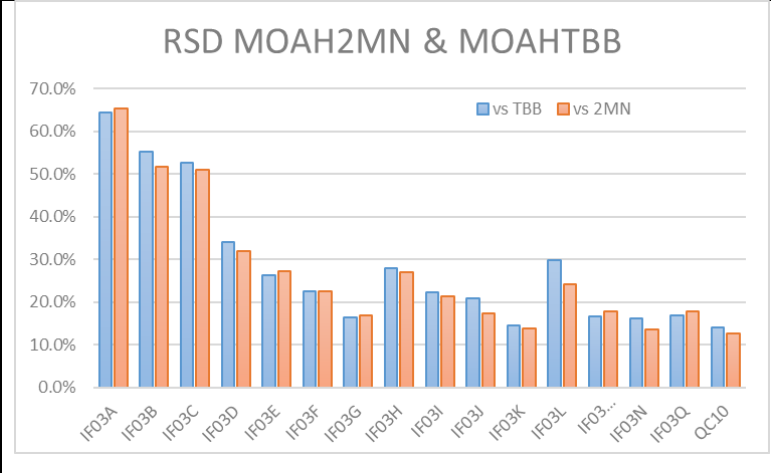
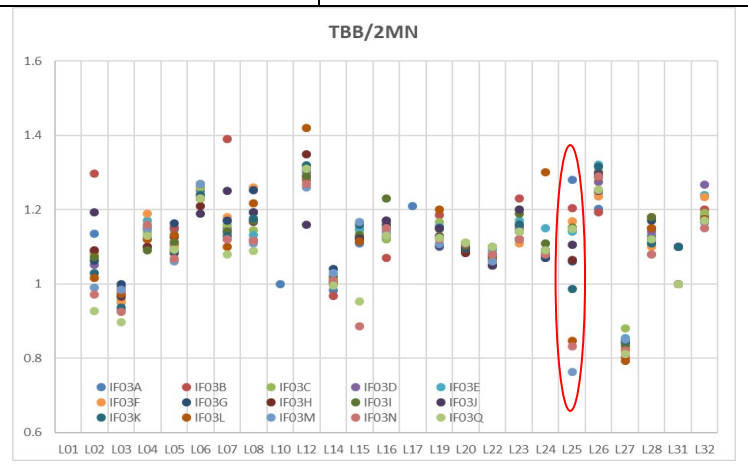
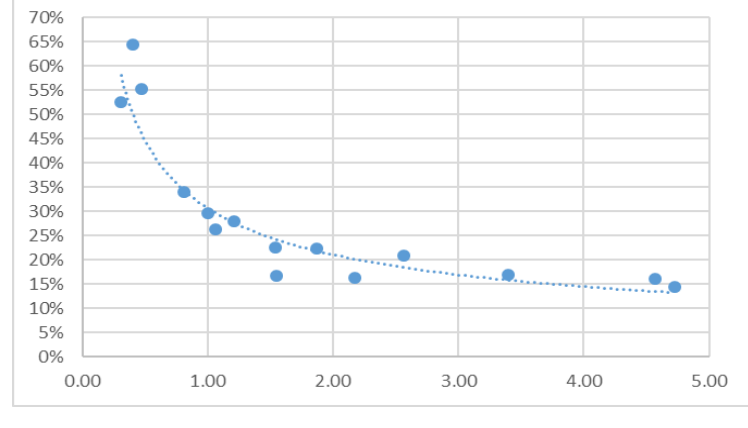


Figure 4.
Comparison of $RSD_R(\text{MOAH-2MN})$ and $RSD_R(\text{MOAH-TBB})$

Figure 5.
Decreasing relative standard deviation for reproducibility $RSD_R(\text{MOAH-TBB})$,% with increasing mass fraction of MOAH-TBB (in mg/kg) in infant formula formulations



MOSH/MOAH sub-fractions

In addition to the mass fractions of total MOSH and MOAH (C10-C50), participants were requested to quantify and report the MOSH and MOAH sub-fractions in samples IF03G and IF03J. These samples were previously analysed in the frame JRC IF 2020-02 [4]. Figure 6 presents a graphical overview of the results, while Table 6 lists the corresponding relative standard deviations for reproducibility.

As expected, slightly higher RSD_R values are observed when quantifying sub-fractions comparing to those of total MOAH (C10-C50). This could be due to the fact that sub-fractions are influenced by the calibration of the MOAH retention time windows. In addition, the interfering riding peaks of the non-fully saponified olefins may have increased the standard deviation of reproducibility for the MOAH C25-C35 sub-fraction. The RSD_R of the C10-C16 and C16-C20 sub-fractions is high due to the very low MOAH content at these C-ranges



Figure 6. Distribution of the MOSH/MOAH content per sub-fraction, mg/kg

MOSH-CyCy	fraction	C10-C16	C16-C20	C20-C25	C25-C35	C35-C40	C40-C50
	mean (mg/kg)	0.135	0.356	0.758	5.30	3.55	2.62
	RSD_R	43%	18%	18%	18%	25%	21%
MOAH-TBB	fraction	C10-C16	C16-C25	C25-C35	C35-C50		
	mean (mg/kg)	0.0186	0.139	0.921	1.58		
	RSD_R	87%	53%	29%	20%		

Table 6 Precision of the determination of the MOSH/MOAH sub-fractions in sample IF03J

MOAH C50+ fraction

Participants were requested to report MOAH C50+ sub-fraction separately. Figure 7 clearly shows that the current analytical instrumentation used does not allow a sufficiently reproducible determination of the high boiling compounds even above 1 mg/kg (IF03Q). This may be due to the discrimination of n-alkanes above n-C50 in the chromatographic system.

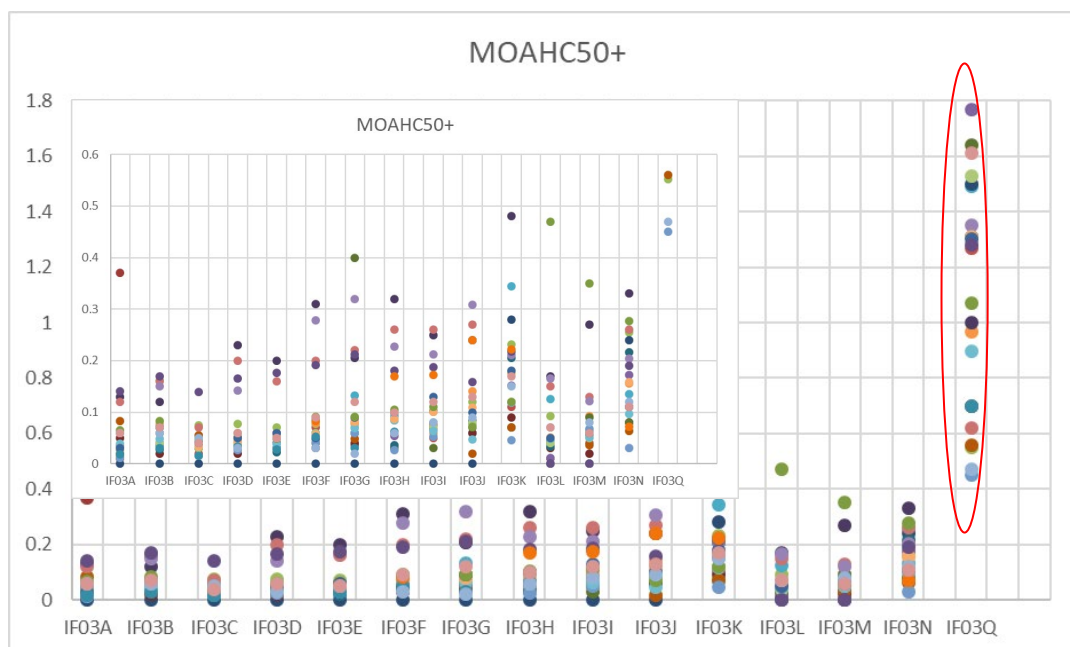


Figure 7. MOAH C50+ fraction in IF samples

4.4 Additional information

Even though no major deviations from the prescribed SOP have been reported by the participants in the questionnaire, some minor variations, not influencing the results, were identified, as discussed hereafter.

SOP implementation

The addition of the IS is one of the critical steps in the procedure for the MOSH/MOAH quantification, which could be considered as an one-point only calibration procedure. The majority of the labs used a syringe for the addition of 10-25 μL volume of the IS solution. Few labs reported using pipettes for 10 and 20- μL volume, which however did not influence the results outcome. **Special care should be taken and only *calibrated* devices should be used in order to limit the contribution of this factor over the total reproducibility of the method.**

Two labs (L14 and L24) observed clumps after reconstitution of the powder IF in water but this did not have an adverse effect on the results of these two labs.

Sixteen out of 24 laboratories confirmed that for some of the samples they still observed an oil layer above the aqueous phase after the first 30 min of saponification. All of them reached full saponification – the majority by proceeding with another 30 min at 60 °C. L01 and L06 needed only 15 min, while L20 applied additional 60 minutes until full saponification.

The slow saponification procedure is one of the drawbacks of the proposed SOP. It could be overcome by proceeding with partial, but still sufficient saponification for MOSH/MOAH release. However this approach requires thorough clean-up over a larger silica gel column (12 g instead of the 3 g preferred by the participants) that has the capacity to retain the remaining lipids. Such procedure would have also a positive effect over the TBB/2MN ratio, known to deviate from the target value of 1 under the conditions of full saponification due to the different distribution of both IS between the phases during the extraction with hexane.

All participants washed the combined hexane extract with either 15 ml (according to the text) or 10 ml (according to the flow chart in Annex 1) water/ethanol. No influence of the volume used for washing was observed on the results.

Using glass chromatographic columns filled with a layer of pre-heated at 400 °C silica gel (3 g) with different height (3-10 cm) was the preferred choice for retaining the polar substances. Six participants however reported the use of SPE cartridges, pre-filled with silica gel. In both cases, participants pre-washed the columns/cartridges with 10 ml hexane/dichloromethane with the exception of L10, which used the SPE cartridge directly. This fact could explain the high contamination background observed in the chromatograms of that lab (Annex 6).

Re-concentration of the extracts was performed either with a stream of nitrogen (10 labs) or via the use of vacuum evaporators such as Turbovap, Polyvap, Rotavap etc. (11 labs). The majority of participants (13 labs) introduced bis(2-ethylhexyl) maleate as keeper, but three labs applying vacuum evaporators did not use a keeper, without adverse effect. As only high boiling point mineral oils were the target analytes in the test items, 5BP behaviour was not monitored for further insight on the loss of volatile compounds, however losses of MNs were not observed.

Comments from participants

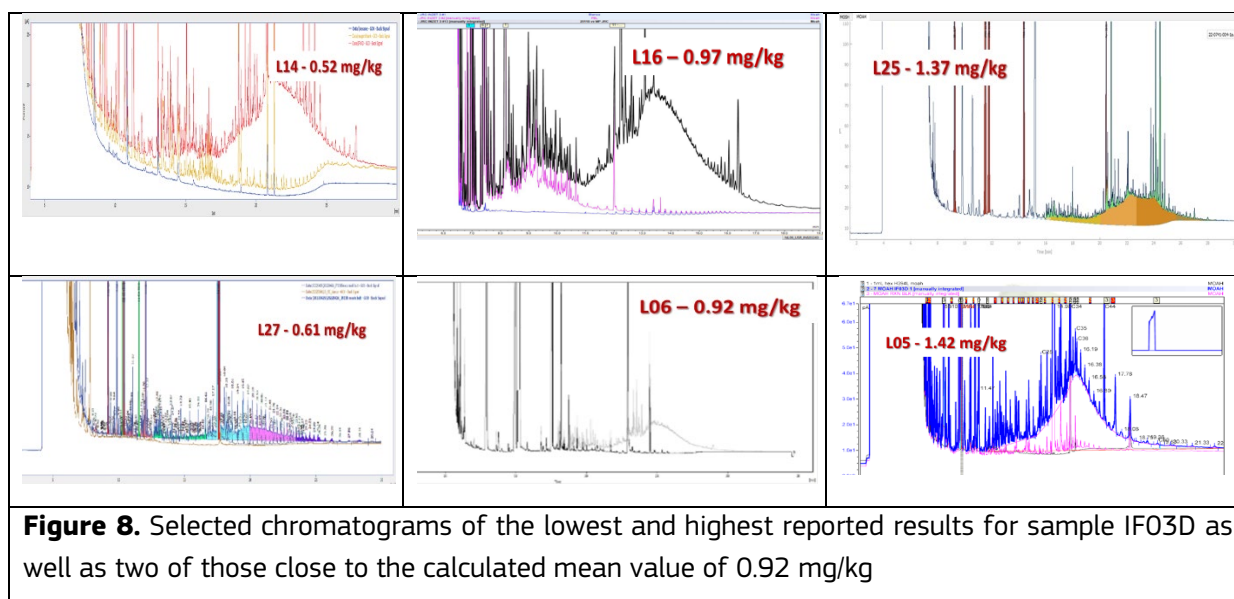
No major problems were encountered by the participants during the sample preparation and analyses of the test items as reported by them.

The main comments received were related to the length and laboriousness of the procedure, requiring many manual operations, which render it not very practical for a routine laboratory with a high throughput of samples. Other remarks concerned some encountered challenges with the phase separation and the necessity to add a keeper from the beginning to prevent IS losses in the blank if quantification is needed.

Examples of chromatograms

Annex 6 includes the compilation of the MOAH and MOSH chromatograms of a number of pre-defined test items from all the participants. The pictures show that efficient removal of the interfering peaks from MOAH humps has been achieved by the majority of the participants for samples based on rapeseed oil IF03A, IF03D and IF03E. As expected, palm oil interferences were more difficult to eliminate compared to interferences from other oils, especially for sample ID03B that was supposed to be blank. In fact, for this sample, the hump under interferences could not be unambiguously defined and the results should be reported as lower than LOQ. Actually only 9 out of 26 participants noted that they would have reported < 0.5 mg/kg.

The lowest spiked sample was IF03D with 0.75 mg/kg MOAH added. Taking into account the presence of the native MOAH of 0.4 mg, the target MOAH value was set to 1.2 mg/kg, but the participants' mean value was calculated to be 0.92 mg/kg (<1 mg/kg). Two selected chromatograms of the lowest and highest reported values as well as two chromatograms with reported values close to the mean are illustrated in Fig. 6. No sound explanation could be found for a 3-fold difference in the MOAH content based only on the visual comparison of the chromatograms. If the maximum LOQ of 1 mg/kg MOAH agreed by the EU member states in a Joint statement from April 2022 [11], is considered, the fact that 17 labs reported values < 1 mg/kg, while 9 labs reported values above 1 mg/kg is a good achievement. Much better is the situation with the samples IF03E (rapeseed oil based) and IF03H (palm oil based), where 1.2 mg/kg MOAH was added to the blank IF matrix. The majority (22 labs) reported values > 1 mg/kg while only 4 labs in both cases reported values < 1 mg/kg.



A further look at the chromatograms' compilation from all participants demonstrated that some labs (e.g. L28 from Annex 6) had constantly or occasionally repeated sharp peaks on the humps. However this did not had significant adverse effect on the quantification. Presumably they are due to the siloxanes coming from the silicon containing septa (especially after being pierced once during the automated epoxidation for example) or silicon caps for the disposable pipettes (when hexane unnoticeably enter the silicon cap when drawn up)

5 Conclusions

The EURL-FCM organised a ring trial to validate the harmonised Standard Operating Procedure investigated. The performance characteristics for the determination of the mass fraction of total MOAH in infant formula were derived from the results reported by 25 participants. Indicative values are also provided for MOSH.

The mass fractions of total MOAH above 1 mg/kg and total MOSH above 5 mg/kg (due to the high MOSH content in the blank samples used) were investigated. The observed relative standard deviations for **repeatability** (RSD_r) – when excluding the blank samples – ranged from 5 % to 13 % for total MOAH, and from 4 % to 14 % for total MOSH. The observed relative standard deviations for **reproducibility** (RSD_R) – when excluding the blank samples – ranged from 13 % to 32 % for total MOAH and from 13 to 23 % for total MOSH. No influence was observed on the type of IF composition.

Despite the low recovery of MOAH (55-65%) attributed to losses during the epoxidation, the method is considered suitable for determination of the mass fraction of total MOAH (C10-C50) above 1 mg/kg in different infant food formulations based on rapeseed, sunflower or palm oils. However it is known that the MOAH recovery values depend not only on the composition of the matrix but as well on the composition of the MO contaminating the matrix (spiked). Therefore no uniform recovery factor can be assigned and applied.

Even if statistically negligible, the mass fraction of total MOAH referred to TBB was constantly 13 % lower than the one obtained using 2MN. It is therefore recommended, in order to ensure proper comparability of results, to report the MOAH in IF using TBB as the internal standard.

Still, users should be aware that this experimental protocol does not fully remove all the interfering compounds present in some challenging matrices, which may require a cautious interpretation and integration of the recorded chromatograms. Further characterisation with two-dimensional GC-GC techniques may be required.

Acknowledgements

The EURL-FCM would like to acknowledge Prof. Dr. H. Emons for his contribution to the initial stage of the MOAH in IF project. Special Nutrition Europe (NESTLE pilot plant) is acknowledged for producing the different powder infant formulas and the Reference Material Unit of the JRC is acknowledged for processing the material and producing high quality test items. Furthermore, the 28 laboratories listed hereafter are kindly acknowledged for their participation in this round.

Organisation	Country
Graz University of Technology	Austria
Primoris	Belgium
Liege University	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
Institute Kirchhoff Berlin GmbH	Germany
Landesbetrieb Hessisches Landeslabor	Germany
mas GmbH	Germany
SGS Institut Fresenius GmbH	Germany
Dublin Public Analyst's Laboratory	Ireland
NEOTRON SPA	Italy
University of Udine	Italy
NOFALAB	Netherlands
Eurofins Lab Zeeuws-Vlaanderen (CNL027)	Netherlands
Wageningen Food Safety Research	Netherlands
Dr A Verwey	Netherlands
Nestlé Research	Switzerland
Official Food Control Authority of the Canton of Zurich	Switzerland
Swiss Quality Testing Services	Switzerland

References

- 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., Karasek L., Gonçalves C., Beldi G., Senaldi C., Jakubowska N., Valzacchi S., Conneely P., Hoekstra E., Emons H. (2020) Determination of MOAH in Infant Formula, JRC IF 2020-01 - an exploratory interlaboratory comparison, European Commission, Geel, JRC 121915
- 4 Bratinova S., Robouch P., Beldi G., Senaldi C., Gonçalves 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 E
- 5 Bratinova S., Robouch P., Gonçalves C., Karasek L., Beldi G., Senaldi C., Valzacchi S., Hoekstra E., H. *Determination of MOSH/MOAH in Shell SN500* mineral oil; JRC IF 2021-03 - The 3rd interlaboratory comparison*, Publications Office (OP) of the European Union, Luxembourg, 2022, ISBN 978-92-76-47525-5, doi:10.2760/23771, JRC 127743
- 6 Robouch P., Bratinova S., Gonçalves C., Karasek L., Beldi G., Senaldi C., Valzacchi S. and Hoekstra E.. *Mineral oils in infant formulas – guidelines for integrating chromatogram; JRC IF 2022-04 – a virtual interlaboratory comparison*. Publications Office (OP) of the European Union, Luxembourg, 2022,
- 7 ISO 13528:2015 Statistical methods for use in proficiency testing by interlaboratory comparison <https://www.iso.org/obp/ui/#iso:std:iso:13528:ed-2:v2:en>
- 8 Bratinova S., 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.
- 9 ISO 5725-2:1994 Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard.
- 10 Software for PT programs and collaborative studies, ProLab; <http://quodata.de/en/software/for-interlaboratory-tests.html>
- 11 Joint statement of 21 April 2022 of the Member States regarding the presence of Mineral Oil Aromatic Hydrocarbons in food, including food for infants and young children [joint statement](#)

Annex 1: Examples from the homogeneity study (all values in mg kg⁻¹)

Sample	IF03G		IF03J	
1	2.384	2.335	2.824	2.805
2	2.115	2.236	2.746	2.630
3	2.456	2.291	2.874	2.728
4	2.211	2.325	2.904	2.959
5	2.212	2.216	2.795	2.891
6	2.140	2.258	2.958	2.878
7	2.117	2.132	2.747	2.668
8	2.250	2.270	2.938	2.853
9	2.192	2.214	2.947	2.753
10	2.162	2.177	2.833	2.758
mean	2.235		2.825	
S_{bb}	0.081		0.082	
S_r	0.060		0.074	
U_{hom}	0.069		0.063	
σ_{pt} (20 %)	0.447		0.565	
$0.3 \sigma_{pt}$	0.134		0.169	
$U_{hom} < 0.3 \sigma_{pt}$	passed		passed	

Annex 2. Invitation letter



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

Geel, 16 February 2022

(sent by registered e-mail in ARES)

Subject: Invitation to participate in a ring trial “JRC IF 2022/05”

Dear Colleagues,

The JRC intends to launch a ring trial validation study of the measurement procedure for the **determination of MOAH in infant formula (IF)**. You are invited to participate to this round.

You will be provided with a detailed **Standard Operating Procedure to be strictly applied**. All experimental deviations will have to be properly documented.

Fifteen samples will be delivered to you, that you will have to analyse **in duplicate**, starting from two independent sample intakes that will undergo the whole measurement procedure.

You will have to (i) report the total mass fraction in mg/kg of MOAH (C10-C50) in IF; and provide (ii) selected chromatograms (screenshots and/or cdf/csv files), as well as (iii) the relevant information proving the performance of your system. In addition, (iv) the total mass fraction of MOSH (C10-C50) in IF, and (v) sub-fraction cuts (as defined in the EURL guide [1]¹) for 2 test samples, will be requested.

We would like to draw your attention to the fact that the performance characteristics will be calculated after careful review of the results and chromatogram submitted.

If you intend to contribute to this round, please register via the link below:

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

The following deadline are foreseen:

- Registration: February 23, 2022;
- Sample dispatch: mid-March, 2022; and
- Submission of results: mid-May 2022.

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

Kind regards,

/signed electronically /

S. BRATINOVA

Ring-trial coordinator

Cc: U.Vincent (Head of Unit, Food & Feed Compliance, JRC.F.5),
E. Hoekstra (EURL-FCM operating manager)

¹ 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.

Annex 3. SOP

link to the JRC EURL-FCM website - [link to the SOP](#)

Annex 4. Instructions to the participants



EUROPEAN COMMISSION
JOINT RESEARCH CENTRE
Directorate F - Health, Consumers & Reference Materials
Food & Feed Compliance Unit
European Union Reference Laboratory for Food Contact Materials



Geel, 04 July 2022
Ref. Ares (2022)xxxx - 04/07/2022

Subject: Ring-trial validation study “JRC IF 2022/05” for the determination of MOAH in infant formulas
- Instructions for participants

Dear Colleague,

Thank you for participating in the ring-trial validation study **JRC-IF-2022/05**. This study is organised, upon request from DG SANTE, to harmonise the official control **method for the determination of MOAH in infant formulas (IF)**.

Triggered by the interest expressed by many of you in the previous interlaboratory comparison rounds, and knowing that the analytical method under investigation provides results for both MOSH and MOAH in IF samples, we will collect data for MOSH content as well and we will provide indicative performance parameters for MOSH.

The measurands, expressed in mg/kg, are defined as:

- (i) the mass fraction of total MOAH (C10-C50) in IF; and
- (ii) the mass fraction of total MOSH (C10-C50) in IF.

You received a parcel consisting of fifteen samples (100 ml glass bottles crimp capped and wrapped in Al foil; containing ca. 40 g powder). Please check upon arrival whether all bottles are undamaged after transport; and send the “**Confirmation of receipt**” form to jrc-eurl-fcm@ec.europa.eu, within 3 days after receipt of the samples.

You were provided with a detailed **Standard Operating Procedure (SOP) to be strictly applied**. All experimental deviations have to be properly documented.

You are requested to analyse every sample (bottle) in **duplicate** (starting from two independent sample intakes that will undergo the whole measurement procedure) and report:

- any deviations from the prescribed SOP;**
- the mass fraction of **total MOAH (C10-C50) calculated vs 2MN (#)**;
- the mass fraction of **total MOAH (C10-C50) calculated vs TBB (#)**;
- the mass fraction of **total MOSH (C10-C50) calculated vs CyCy (#)**;
(#) for every replicate; expressed in mg/kg;
- the associate **relative expanded uncertainties** (expressed in %, coverage factor k=2)
- the mass fraction of the **C50+ for MOSH and MOAH (vs TBB)**, expressed in mg/kg;
- the “**2MN/1MN**”, “**2MN/TBB**”, “**5B/TBB**”, “**CyCy/TBB**”, “**C20/C50**” ratios;
- the concentration of total MOSH and total MOAH in JRC QC10-1 (Shell SN500* in hexane, sample sent to you in July 2021), expressed in mg/L; Information about how to prepare JRC QC10-1 in an attached file (from JRC IF 2021/03).
- the content of the sub-fraction cuts (as defined in the EURL guide [1]) for samples IF03G and IF03J (vs TBB), expressed in mg/kg.

¹ 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.

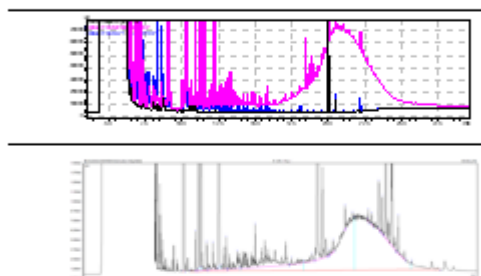
Please report all the quantifiable results for MOSH and MOAH total, based on the integration of the **entire hump** for the range C10-C50 (even if below your usual LOQ), but please highlight in red those that you wouldn't normally report to the customer

All results should be reported in the attached "Reporting Form" (Excel template) with **three significant figures** (e.g. 12.0 or 1.20 mg/kg).

Your LabCode will be provided to you in a separate mail. Introduce this code in the XLS template and name the file accordingly (for example: JRC-IF-2022/05-L01.xlsx).

In addition, the following chromatograms (screenshots) should be provided together with the Reporting form, mentioned above, to show the integration of the chromatograms, riding peaks and background compensation:

- overlaid original chromatograms of IF03 (A, B, D, E, H, I) each with hexane and the reagent blank, properly scaled to show clearly the hump;
- chromatograms of IF03 (E, I) properly scaled, to show (i) the trimmed riding peaks and (ii) the baseline subtracted from the hump.



When deemed necessary, you will be contacted for additional information or chromatograms.

You will receive shortly the link to the [EUSurvey](#) questionnaire. The system will guide you through the reporting procedure. Do not forget to submit and confirm when required. Please note that submitted results without filled questionnaires will not be used in the final calculation.

The deadline for reporting is set to 22 May, 2022

A preliminary report to participants will be circulated shortly after the end of the round to present the reported values from all participants.

Your participation in this project is greatly appreciated.
Do not hesitate to contact us for further information.

Kind regards,

/signed electronically in Ares/

Dr. Stefanka [Bratinova](#)
JRC IF 2022/05 Coordinator

Cc: [U. Vincent](#) (Head of Unit, Food & Feed Compliance, F.5),
[E. Hoekstra](#) (Operating Manager EURL-FCM)

Reporting form

LabCode																			
Organisation																			
Reporting person																			

Note: Only the yellow & green cells can be edited
 Please integrate the entire hump from C10 to C50, and report all quantifiable results for total MOSH & MOAH even if the results would fall below the LOQ you usually report.
 Please highlight such results in red.

Analyte	IS	Sample	Unit	Samples														QC10-01		
				IF03A	IF03B	IF03C	IF03D	IF03E	IF03F	IF03G	IF03H	IF03I	IF03J	IF03K	IF03L	IF03M	IF03N		IF03Q	
Total mass fraction (C10-C50)	tot MOAH	2MN	Replicate 1	mg/kg																
		Replicate 2	mg/kg																	
		U _{rel} (k=2)	%																	
	tot MOAH	TBB	Replicate 1	mg/kg																
	Replicate 2	mg/kg																		
		U _{rel} (k=2)	%																	
Total mass fraction (C10-C50)	tot MOSH	CyCy	Replicate 1	mg/kg																
			Replicate 2	mg/kg																
		U _{rel} (k=2)	%																	
	Individual fractions cuts	MOSH	CyCy	C10-C16	mg/kg															
C16-C20				mg/kg																
C20-C25				mg/kg																
C25-C35				mg/kg																
C35-C40				mg/kg																
C40-C50				mg/kg																
		C50+	mg/kg																	
MOAH		TBB	C10-C16	mg/kg																
			C16-C25	mg/kg																
			C25-C35	mg/kg																
			C35-C50	mg/kg																
				C50+	mg/kg															
	1MN/2MN *	ratio																		
	TBB/2MN *	ratio																		
	TBB/5B *	ratio																		
	TBB/CyCy *	ratio																		
	C50/C20 *	ratio																		

(*) the ratios could be filled only once if constant for all the samples, otherwise fill in the deviations
 U_{rel} (k=2) expanded relative uncertainty, expressed in %

Annex 5: Results as reported by the participants

Table 1: Reported results for MOAH vs 2MN in IF03A

Lab	M 1	M 2	MU [%]
L01	0.320	0.370	0
L02	1.230	1.177	4.4
L03	0.307	0.233	13
L04	0.624	0.676	0.07
L05	0.636	0.703	0
L06	0.440	0.500	30
L07	0.390	0.350	20
L08	0.090	0.100	
L10	1.176	0.908	25.7
L11	1.638	1.814	0.05
L12	0.005	0.201	0.33
L14	0.188	0.170	13.9
L15	3.370	0.412	221
L16	0.620	0.580	0.3
L17	0.150	0.100	56.5
L19	0.651	0.421	36
L20	0.362	0.442	19.9
L22	0.264	0.262	40
L23	0.410	0.340	36.6
L24	0.370	0.440	30
L25	0.571	0.744	25
L26	0.672	0.698	20
L27	0.190	0.074	43
L28	0.561	1.394	30
L31	0.380	0.400	7.1
L32	0.250	0.240	1.4

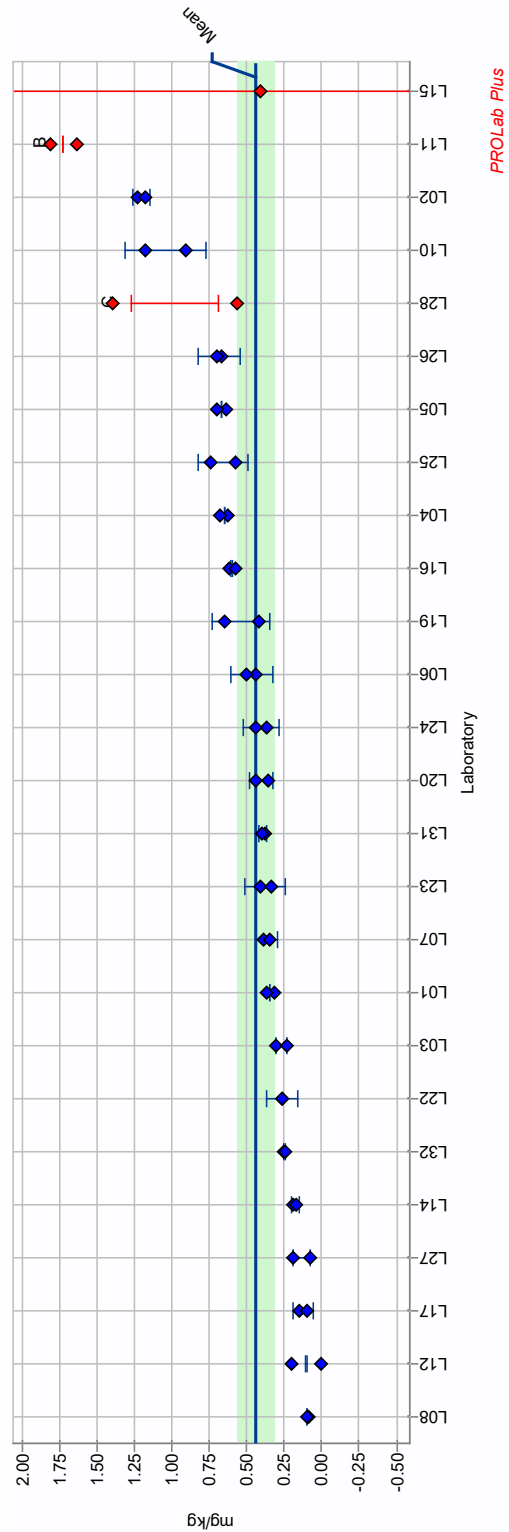


Figure 1. MOAH2MN in IF03A.

Results in red are outliers:

B - Grubbs (laboratory mean),

C - Cochran (excessive standard deviation)

Table 2: Reported results for MOAH vs TBB in IF03A

Lab	M 1	M 2	MU [%]
L01	0.240	0.300	0
L02	1.177	0.968	19.5
L03	0.333	0.240	13
L04	0.538	0.601	0.9
L05	0.554	0.629	0
L06	0.340	0.360	0
L07	0.340	0.300	20
L08	0.080	0.080	
L10	1.074	0.878	20.1
L11	1.206	1.405	0.07
L12	0.004	0.159	0.33
L14	0.189	0.176	10.6
L15	3.000	0.356	223
L16	0.530	0.520	0.3
L17	0.120	0.090	40.4
L19	0.563	0.362	36
L20	0.316	0.384	19.4
L22	0.249	0.248	40
L23	0.370	0.310	37.2
L24	0.340	0.390	30
L25	0.576	0.581	25
L26	0.559	0.580	20
L27	0.147	0.140	43
L28	0.466	0.737	30
L31	0.360	0.380	6
L32	0.210	0.200	1.4

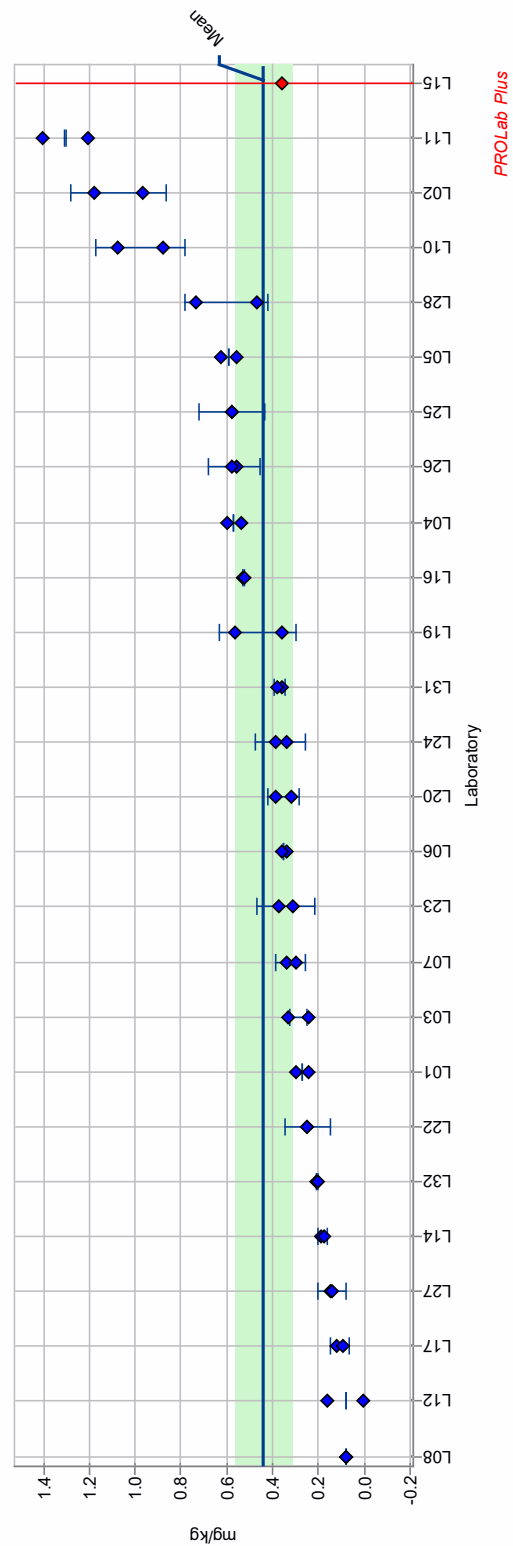


Figure 2. MOAHTBB in IF03A.

Results in red are outliers:

C – Cochran (excessive standard deviation)

Table 3: Reported results for MOSH vs CyCy in IF03A

Lab	M 1	M 2	MU [%]
L01	4.93	4.82	0
L02	17.11	13.93	20.5
L03	6.72	6.33	23
L04	6.29	6.59	0.42
L05	6.56	6.97	0
L06	5.48	5.08	30
L07	7.12	7.04	10
L08	9.22	7.26	
L10	8.29	8.72	5.0
L11	15.40	18.00	0.07
L12		5.08	0.17
L14	4.53	4.63	3.1
L15	20.00	5.40	162
L16	6.09		0.3
L17	8.69	8.47	3.6
L19	7.24	5.71	36
L20	2.02	2.20	8.4
L22	4.84	4.76	40
L23	6.36	5.91	24.2
L24	4.90	5.52	30
L25	6.78	6.92	25
L26	5.44	5.96	20
L27	5.55	5.79	40
L28	4.66	11.10	30
L31	4.79	4.65	4.2
L32	3.99	3.47	73.5

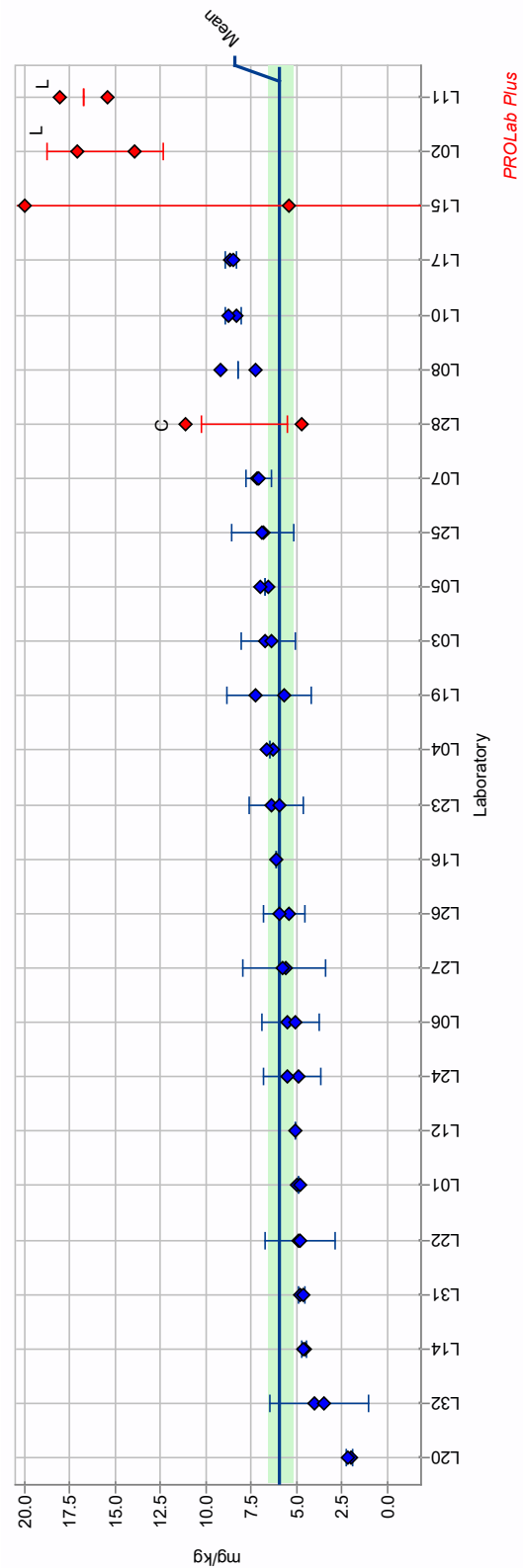


Figure 3. MOSH in IF03A.
 Results in red are outliers:
 C – Cochran (excessive standard deviation)
 L - Grubbs (laboratory mean for 2),

Table 4: Reported results for MOAH vs 2MN in IF03B

Lab	M 1	M 2	MU [%]
L01	0.740	0.590	0
L02	4.632	3.760	20.7
L03	0.435	0.366	13
L04	0.715	0.769	0.07
L05	1.170	1.103	0
L06	0.460	0.640	30
L07	0.430	0.430	20
L08	0.233	0.215	
L10	0.893	1.152	25.3
L11	3.029	3.259	0.03
L12	0.038	0.218	0.33
L14	0.145	0.141	3.5
L15	0.986	0.489	95
L16	1.080	0.560	0.3
L17	0.170	0.140	27.3
L19	0.536	0.542	36
L20	0.467	0.476	1.9
L22	0.500	0.486	40
L23	0.590	0.580	34.6
L24	0.720	0.730	30
L25	0.919	0.883	25
L26	0.721	0.674	20
L27	0.264	0.356	43
L28	0.673	0.509	30
L31	0.270	0.230	23.2
L32	0.550	0.550	0

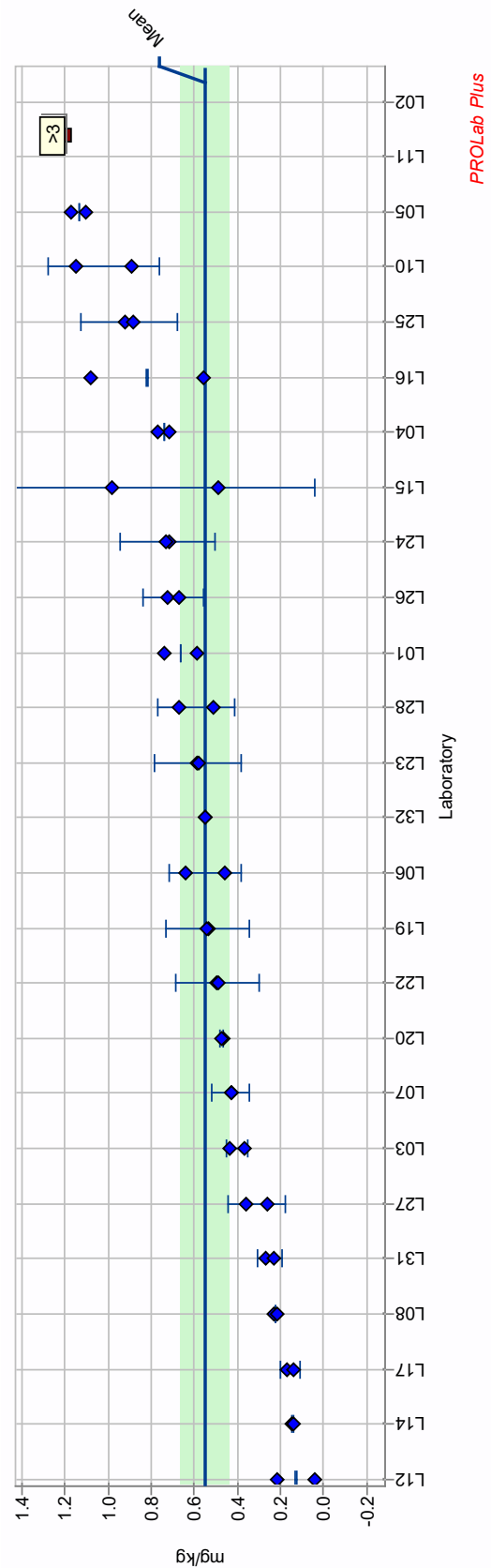


Figure 4. MOAH2MN in IF03B.
Results in yellow box are Grubbs outliers.

Table 5: Reported results for MOAH vs TBB in IF03B

Lab	M 1	M 2	MU [%]
L01	0.560	0.420	0
L02	3.646	2.850	24.5
L03	0.437	0.356	13
L04	0.637	0.626	0.02
L05	1.014	0.982	0
L06	0.360	0.500	0
L07	0.320	0.310	20
L08	0.208	0.184	
L10	0.886	1.100	21.5
L11	0.430	1.711	0.6
L12	0.030	0.176	0.33
L14	0.149	0.147	1.4
L15	0.834	0.412	96
L16	0.940	0.520	0.3
L17	0.140	0.120	21.7
L19	0.452	0.461	36
L20	0.420	0.409	2.6
L22	0.478	0.458	40
L23	0.460	0.440	36
L24	0.670	0.670	30
L25	0.752	0.733	25
L26	0.585	0.585	20
L27	0.173	0.237	43
L28	0.536	2.857	30
L31	0.250	0.220	20.9
L32	0.460	0.470	1.4

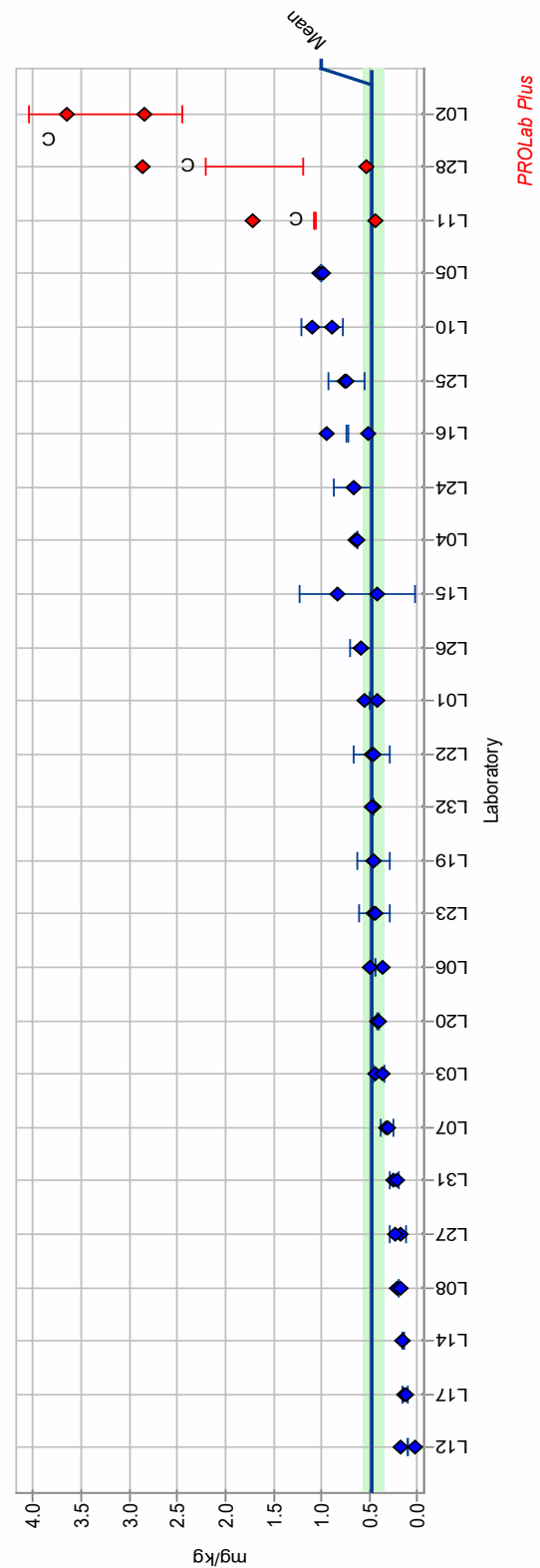


Figure 5. MOAHTBB in IF03B.
 Results in red are outliers:
 C – Cochran (excessive standard deviation)

Table 6: Reported results for MOSH vs CyCy in IF03B

Lab	M 1	M 2	MU [%]
L01	5.69	5.91	0
L02	8.66	7.37	16
L03	6.54	6.48	23
L04	5.82	6.31	0.7
L05	8.79	9.89	0
L06	5.74	5.82	30
L07	6.27	6.33	10
L08	7.60	7.09	
L10	8.99	8.18	9.4
L11	4.21	15.10	0.5
L12		4.63	0.17
L14	4.99	5.18	5.1
L15	10.10	6.64	58
L16	5.90		0.3
L17	6.58	6.51	1.5
L19	6.58	6.08	36
L20	3.92	3.74	4.6
L22	5.66	5.82	40
L23	7.63	7.65	23.6
L24	5.92	5.53	30
L25	8.42	8.02	25
L26	7.18	6.99	20
L27	7.22	8.63	40
L28	5.49	4.91	30
L31	5.28	4.90	10.5
L32	4.04	3.86	25.4

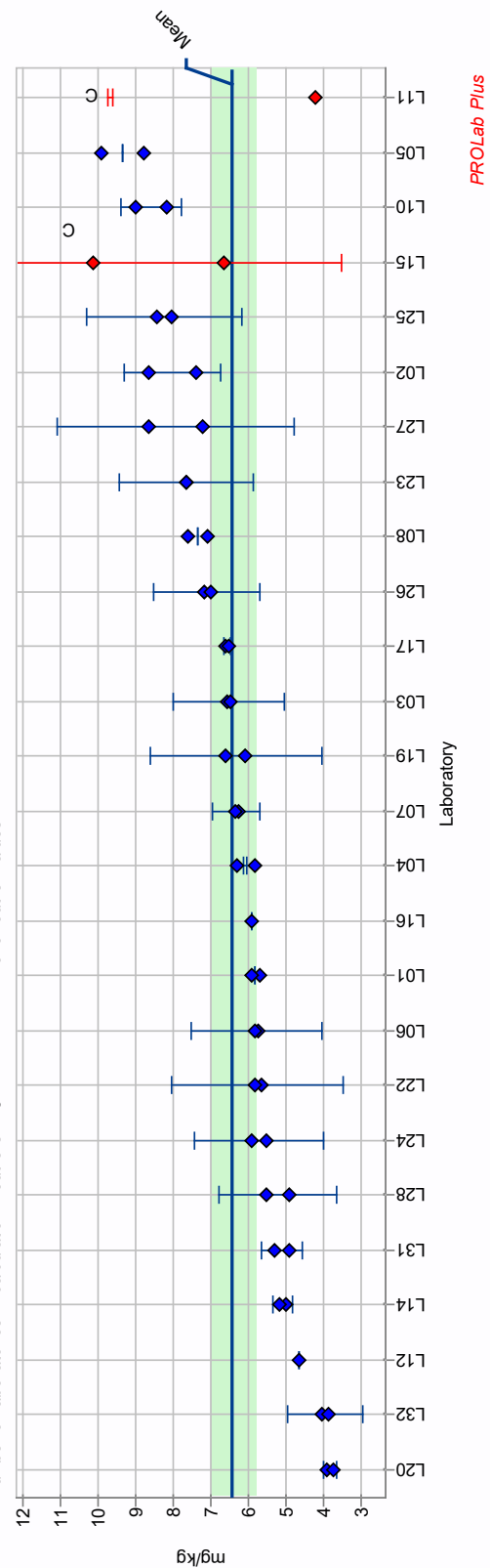


Figure 6. MOSH in IF03B.
 Results in red are outliers:
 C – Cochran (excessive standard deviation)

Table 7: Reported results for MOAH vs TBB in IF03C

Lab	M 1	M 2	MU [%]
L01	0.280	0.230	0
L02	0.680	0.543	22.5
L03	0.258	0.206	13
L04	0.431	0.466	0.05
L05	0.572	0.641	0
L06	0.320	0.300	0
L07	0.330	0.350	20
L08	0.080	0.100	
L10	0.456	0.493	8
L11	1.429	1.060	0.14
L12	0.089	0.086	0.33
L14	0.138	0.131	6.7
L15	0.279	0.286	3.7
L16	0.500	0.520	0.3
L17	0.080	0.080	0
L19	0.300	0.300	36
L20	0.309	0.297	3.8
L22	0.225	0.239	40
L23	0.290	0.250	38.6
L24	0.290	0.240	30
L25	0.530	0.543	25
L26	0.583	0.521	20
L27	0.082	0.069	43
L28	0.517	0.485	30
L31	0.300	0.250	25.5
L32	0.200	0.190	1.4

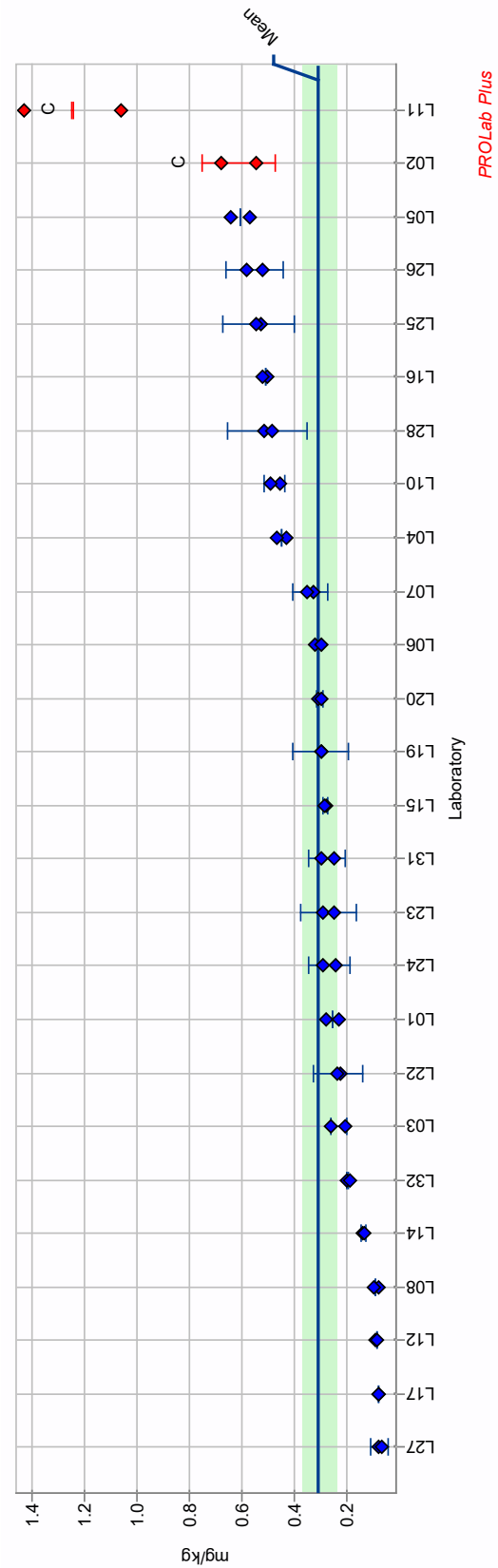


Figure 7. MOAHTBB in IF03C.
 Results in red are outliers:
 C – Cochran (excessive standard deviation)

Table 8: Reported results for MOAH vs 2MN in IF03C

Lab	M 1	M 2	MU [%]
L01	0.370	0.310	0
L02	0.731	0.599	19.7
L03	0.250	0.212	13
L04	0.491	0.554	0.09
L05	0.633	0.706	0
L06	0.420	0.410	30
L07	0.380	0.410	20
L08	0.090	0.110	
L10	0.476	0.517	8
L11	1.753	1.453	0.1
L12	0.130	0.110	0.33
L14	0.141	0.131	10.2
L15	0.332	0.342	4.2
L16	0.560	0.580	0.3
L17	0.110	0.100	13.4
L19	0.350	0.350	36
L20	0.359	0.341	5.4
L22	0.243	0.259	40
L23	0.340	0.300	37.6
L24	0.320	0.260	30
L25	0.586	0.642	25
L26	0.661	0.721	20
L27	0.161	0.132	43
L28	0.609	0.553	30
L31	0.350	0.290	22.7
L32	0.230	0.230	0

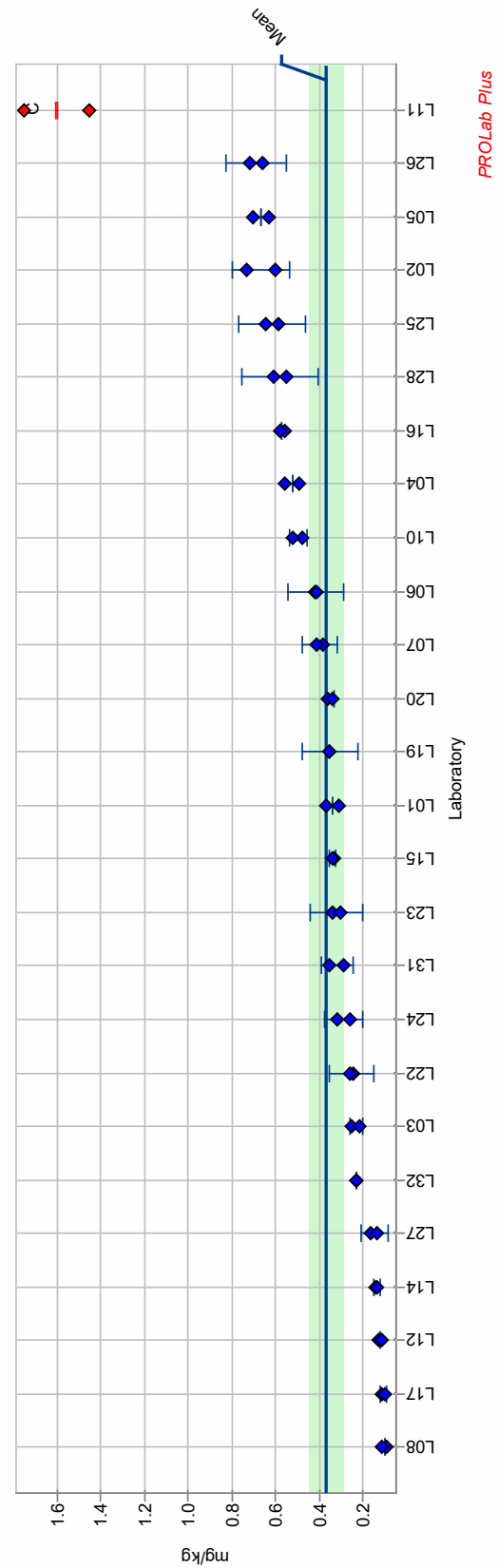


Figure 8. MOAH2MN in IF03C.
Results in red are outliers:
C – Cochran (excessive standard deviation)

Table 9: Reported results for MOSH vs CyCy in IF03C

Lab	M 1	M 2	MU [%]
L01	4.23	4.43	0
L02	7.42	6.70	10.2
L03	4.79	4.85	23
L04	4.46	5.17	1
L05	6.12	6.91	0
L06	4.83	4.33	30
L07	5.26	5.32	10
L08	5.10	5.03	
L10	5.57	6.05	8.3
L11	11.70	11.06	0.03
L12	6.66	5.18	0.17
L14	4.03	3.99	1.16
L15	5.13	4.88	7.2
L16	4.79		0.3
L17	6.61	6.18	9.5
L19	4.69	4.51	36
L20	3.30	3.35	7.7
L22	4.03	4.10	40
L23	5.20	5.29	25
L24	4.05	4.06	30
L25	5.99	5.92	25
L26	4.96	5.52	20
L27	4.70	5.57	40
L28	3.68	3.46	30
L31	6.04	5.69	8.3
L32	2.75	2.67	11

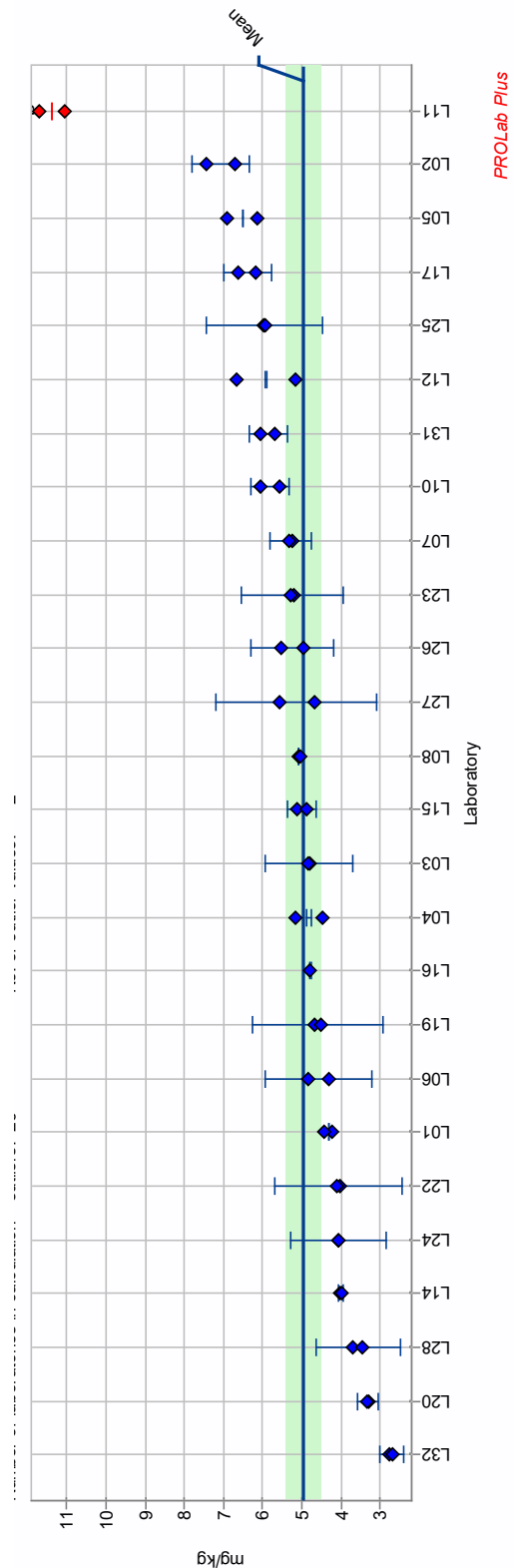


Figure 9. MOSH in IF03C.
Results in red are outliers:
B - Grubbs (laboratory mean),

Table 10: Reported results for MOAH vs 2MN in IF03D

Lab	M 1	M 2	MU [%]
L01	0.780	0.910	0
L02	1.178	0.894	27.4
L03	0.787	0.711	13
L04	1.160	1.110	0.07
L05	1.393	1.453	0
L06	0.930	0.900	30
L07	1.030	1.020	20
L08	0.520	0.570	
L10	1.466	1.678	13.4
L11	10.335	9.936	0.02
L12	0.265	0.517	0.33
L14	0.571	0.480	24
L15	0.882	0.892	1.6
L16	1.010	0.920	0.3
L17	0.650	0.640	2.1
L19	0.863	0.811	36
L20	0.769	0.759	1.24
L22	0.764	0.720	40
L23	0.820	0.820	33
L24	0.850	0.830	30
L25	1.351	1.391	25
L26	1.380	1.290	20
L27	0.652	0.572	43
L28	0.943	1.041	30
L31	1.040	1.000	4.6
L32	0.900	0.990	12.7

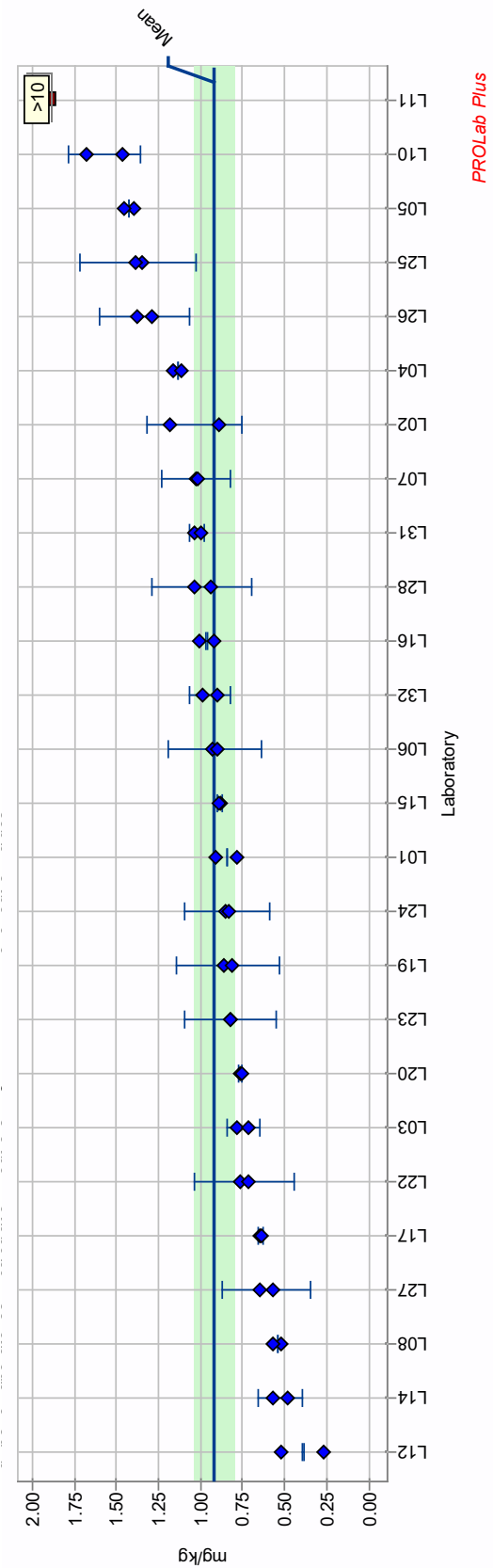


Figure 10. MOAH2MN in IF03D. Results in yellow box are Grubbs outliers.

Table 11: Reported results for MOAH vs TBB in IF03D

Lab	M 1	M 2	MU [%]
L01	0.600	0.730	0
L02	1.113	0.858	25.8
L03	0.832	0.704	13
L04	0.996	0.963	0.05
L05	1.258	1.328	0
L06	0.720	0.780	0
L07	0.890	0.870	20
L08	0.450	0.490	
L10	1.439	1.647	13.4
L11	9.025	9.139	0.006
L12	0.195	0.398	0.33
L14	0.570	0.483	23.3
L15	0.785	0.753	5.7
L16	0.920	0.820	0.3
L17	0.530	0.530	0
L19	0.785	0.715	36
L20	0.689	0.630	8.8
L22	0.734	0.707	40
L23	0.730	0.710	33.6
L24	0.790	0.750	30
L25	1.181	1.134	25
L26	1.040	1.050	20
L27	0.536	0.453	43
L28	0.864	0.794	30
L31	0.950	0.920	4.3
L32	0.710	0.780	9.8

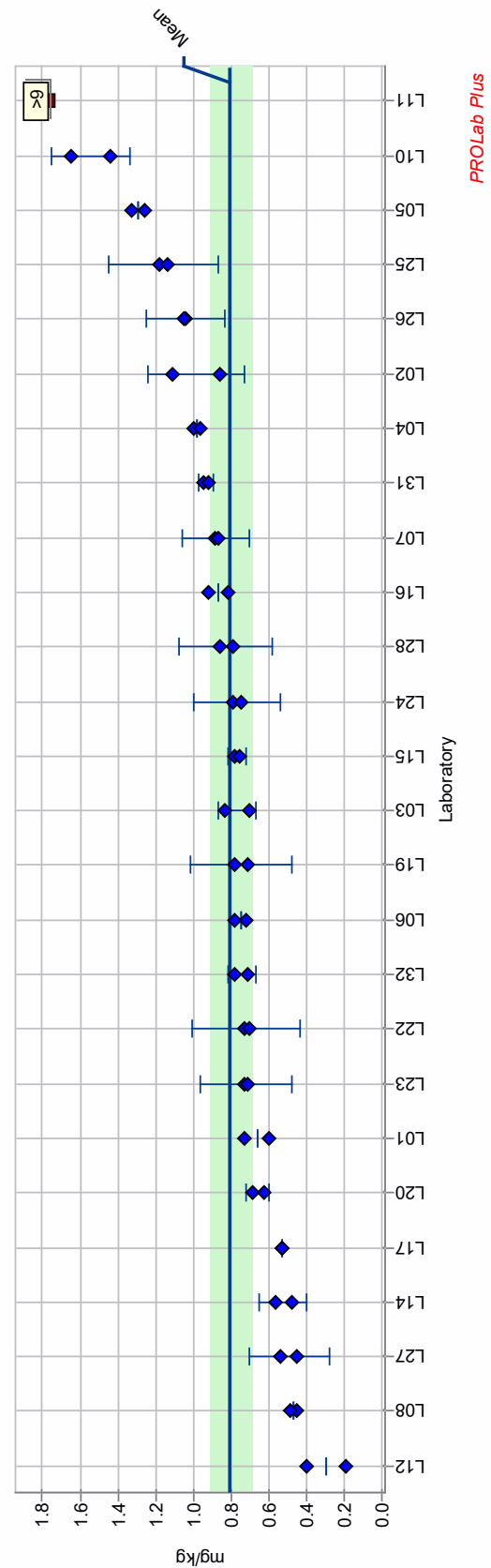


Figure 11. MOAHTBB in IF03D.
Results in yellow box are Grubs outliers.

Table 12: Reported results for MOSH vs CyCy in IF03D

Lab	M 1	M 2	MU [%]
L01	6.25	6.11	0
L02	7.09	8.83	21.8
L03	7.64	7.58	23
L04	6.49	7.24	1.06
L05	9.54	10.79	0
L06	6.72	5.89	30
L07	7.71	7.83	10
L08	8.32	7.78	
L10	7.35	6.93	5.8
L11	17.23	16.76	0.014
L12		6.29	0.17
L14	5.77	5.59	4.3
L15	7.92	8.59	11.5
L16	9.11		0.3
L17	10.96	11.56	7.5
L19	6.63	6.84	36
L20	4.17	4.08	2.18
L22	6.08	6.44	40
L23	7.37	7.18	23.7
L24	6.19	6.36	30
L25	8.64	8.74	25
L26	7.71	7.29	20
L27	7.47	8.85	40
L28	5.87	6.17	30
L31	7.80	7.15	12.3
L32	4.19	4.48	41

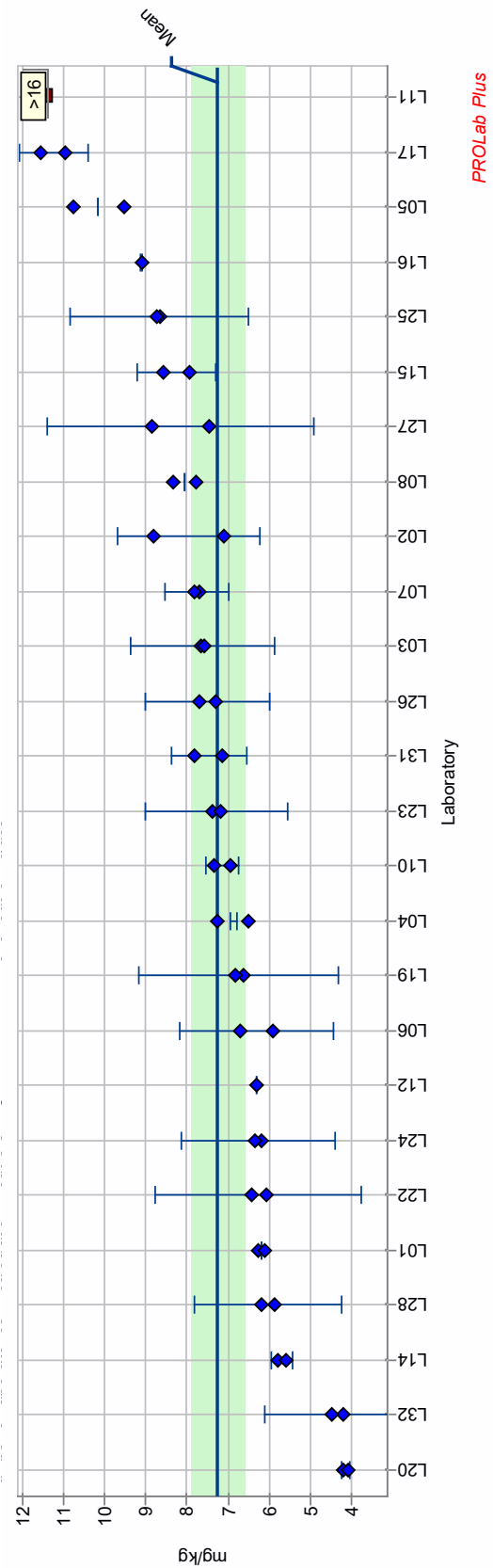


Figure 12. MOSH in IF03D. Results in yellow box are Grubbs outliers.

Table 13: Reported results for MOAH vs 2MN in IF03E

Lab	M 1	M 2	MU [%]
L01	1.51	1.27	0
L02	1.49	1.34	11.1
L03	1.19	0.98	13
L04	1.54	1.40	0.189
L05	1.91	1.88	0
L06	1.23	1.15	30
L07	1.23	1.23	20
L08	0.93	0.91	
L10	1.89	1.47	24.9
L11	9.71	10.01	0.015
L12	0.54	0.72	0.33
L14	0.70	0.68	3.3
L15	1.15	1.23	9.2
L16	1.43	1.33	0.3
L17	0.97	0.91	9
L19	1.12	1.11	36
L20	1.09	1.08	1
L22	1.09	1.04	40
L23	1.13	1.14	31
L24	1.16	1.24	30
L25	1.61	2.10	25
L26	1.78	1.81	20
L27	0.93	0.93	43
L28	1.26	1.17	30
L31	1.57	1.58	1
L32	1.38	1.27	15

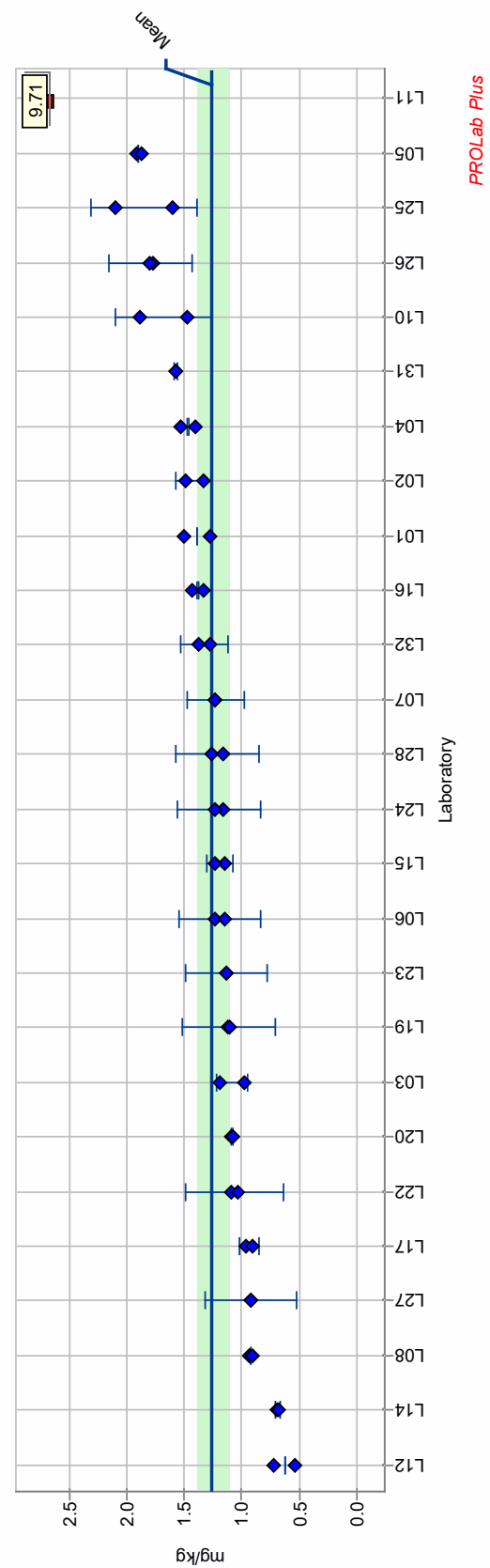


Figure 13. MOAH2MN in IF03E. Results in yellow box are Grubbs outliers.

Table 14: Reported results for MOAH vs TBB in IF03E

Lab	M 1	M 2	MU [%]
L01	1.10	1.01	0
L02	1.37	1.25	9.52
L03	1.30	1.02	13
L04	1.30	1.20	0.15
L05	1.75	1.68	0
L06	0.99	0.91	0
L07	1.11	1.10	20
L08	0.82	0.77	
L10	1.81	1.42	23.7
L11	8.16	9.02	0.05
L12	0.42	0.51	0.33
L14	0.68	0.69	0.13
L15	1.00	1.03	4.2
L16	1.11	1.16	0.3
L17	0.80	0.74	11
L19	0.97	0.97	36
L20	0.99	0.96	2.5
L22	1.00	0.96	40
L23	1.02	0.97	31.9
L24	1.08	1.07	30
L25	1.41	1.48	25
L26	1.34	1.38	20
L27	0.78	0.72	43
L28	1.11	1.00	30
L31	1.44	1.43	1.3
L32	1.10	1.02	11.3

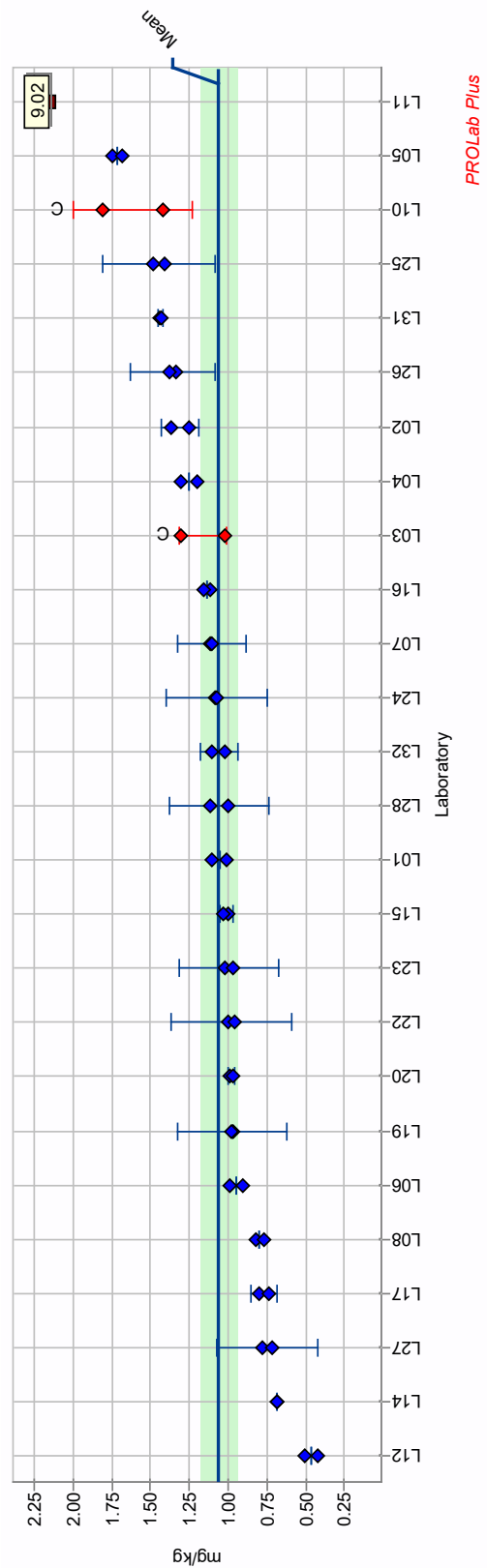


Figure 14. MOAHTBB in IF03E.
 Results in yellow box are Grubs outliers.
 C – Cochran (excessive standard deviation)

Table 15: Reported results for MOSH vs CyCy in IF03E

Lab	M 1	M 2	MU [%]
L01	7.89	6.80	0
L02	9.89	9.91	0.2
L03	9.70	8.79	23
L04	9.77	7.77	2.8
L05	11.90	10.19	0
L06	7.41	6.74	30
L07	8.32	8.35	10
L08	8.76	9.36	
L10	7.44	7.80	4.8
L11	9.91	19.59	0.3
L12		7.06	0.17
L14	6.45	6.48	0.8
L15	9.29	8.71	9.1
L16	9.42		0.3
L17	10.12	11.51	18.1
L19	7.49	7.45	36
L20	5.44	5.75	5.4
L22	7.03	6.85	40
L23	8.49	8.62	23.2
L24	6.95	7.19	30
L25	9.58	9.09	25
L26	8.97	8.94	20
L27	7.60	8.43	40
L28	6.69	6.16	30
L31	8.99	8.94	0.9
L32	5.04	4.79	35.3

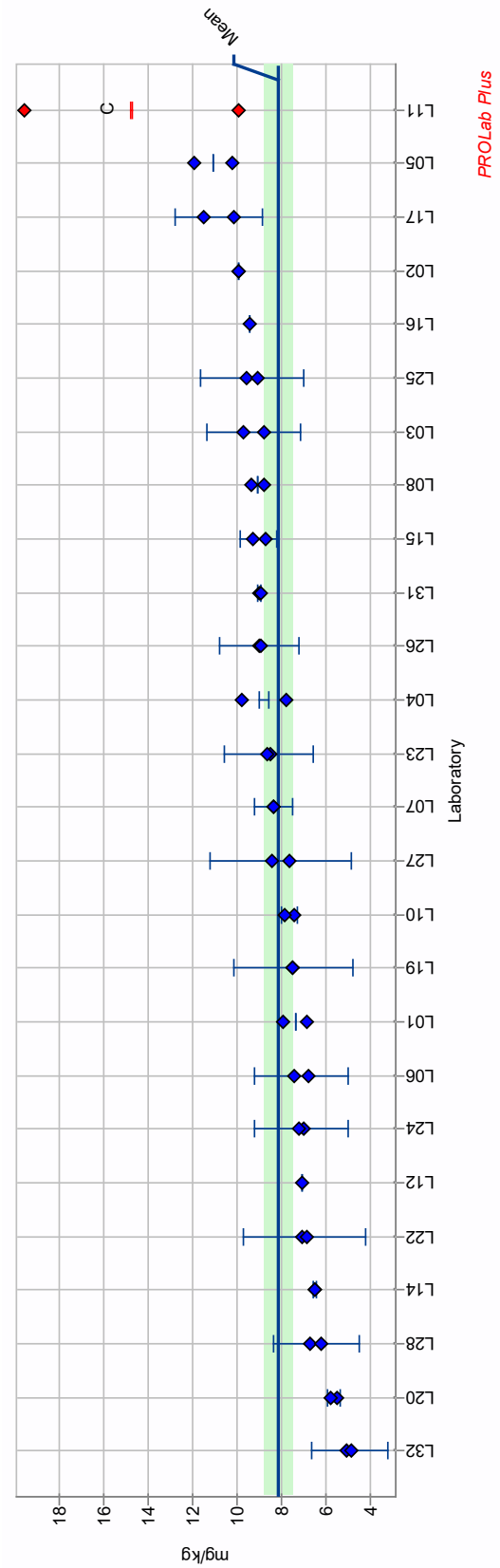


Figure 15. MOSH in IF03E.
 Results in red are outliers:
 C – Cochran (excessive standard deviation)

Table 16: Reported results for MOAH vs 2MN in IF03F

Lab	M 1	M 2	MU [%]
L01	1.92	1.92	0
L02	2.00	2.29	13.8
L03	1.38	1.41	13
L04	1.69	1.72	0.04
L05	2.36	2.72	0
L06	1.73	1.56	30
L07	1.61	1.61	20
L08	2.37	1.37	
L10	2.14	1.79	17.9
L11	2.85	2.72	0.02
L12	1.01	1.14	0.33
L14	1.21	1.04	21.5
L15	1.82	1.94	8.6
L16	1.81	1.89	0.3
L17	1.97	1.42	45.8
L19	1.75	1.75	36
L20	1.62	1.49	8.7
L22	1.51	1.49	40
L23	1.58	1.60	29.9
L24	1.66	1.69	30
L25	2.62	2.53	25
L26	2.43	2.07	20
L27	1.43	1.49	43
L28	2.15	1.21	30
L31	1.65	1.67	1.7
L32	2.07	2.04	4.2

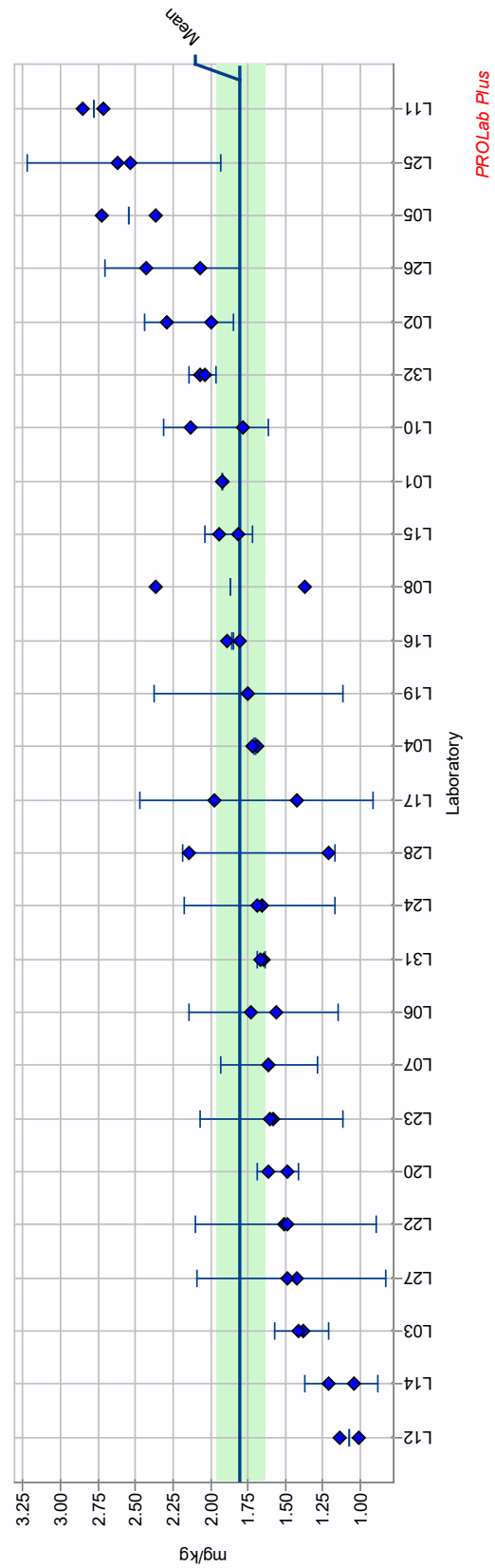


Figure 16. MOAH2MN in IF03F.

Table 17: Reported results for MOAH vs TBB in IF03F

Lab	M 1	M 2	MU [%]
L01	1.41	1.45	0
L02	2.11	1.88	11.4
L03	1.47	1.47	13
L04	1.58	1.44	0.2
L05	2.15	2.49	0
L06	1.39	1.25	0
L07	1.39	1.34	20
L08	1.88	1.17	
L10	2.07	1.71	18.9
L11	2.41	2.19	0.04
L12	0.75	0.93	0.33
L14	1.17	1.06	13.8
L15	1.63	1.63	4.7
L16	1.65	1.63	0.3
L17	1.43	1.18	27.1
L19	1.55	1.55	36
L20	1.47	1.34	8.8
L22	1.41	1.38	40
L23	1.42	1.43	30.4
L24	1.53	1.54	30
L25	2.24	2.17	25
L26	1.86	1.77	20
L27	1.17	1.18	43
L28	1.59	1.01	30
L31	1.61	1.70	8.4
L32	1.68	1.66	2.8

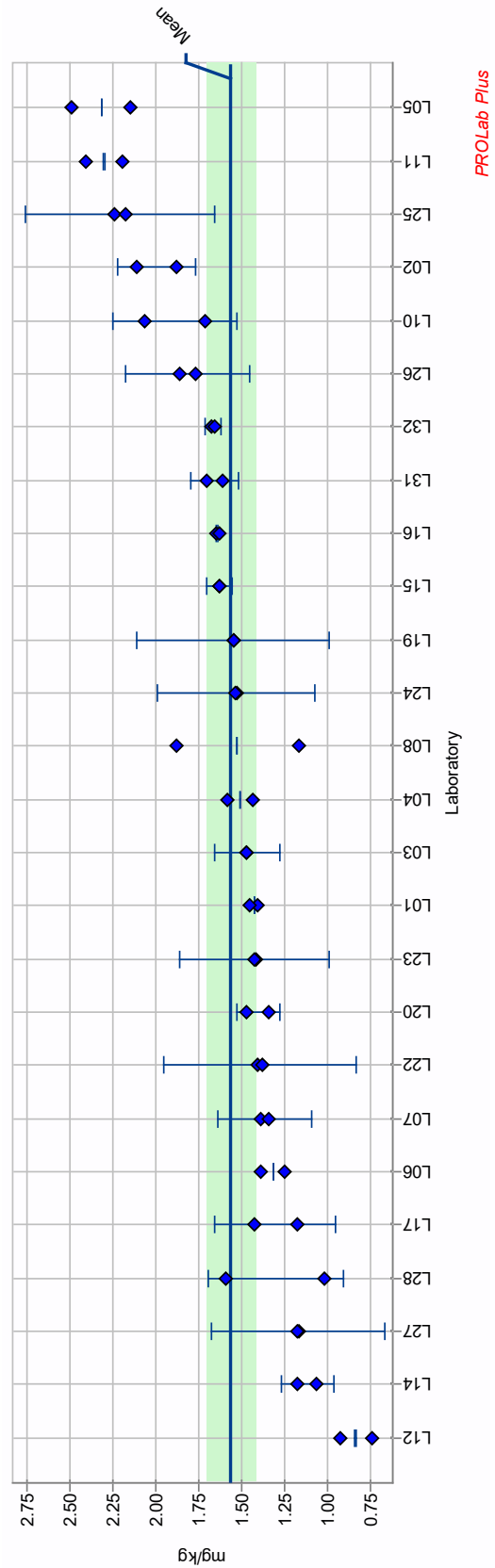


Figure 17. MOAHTBB in IF03F.

Table 18: Reported results for MOSH vs CyCY in IF03F

Lab	M 1	M 2	MU [%]
L01	8.20	8.25	0
L02	13.13	9.23	34.8
L03	9.62	10.00	23
L04	8.15	8.33	0.26
L05	12.22	13.67	0
L06	8.82	8.34	30
L07	9.34	9.39	10
L08	10.07	10.08	
L10	8.26	8.87	7.14
L11	14.10	14.23	0.005
L12		8.27	0.17
L14	7.83	7.19	12
L15	10.10	11.80	22
L16	9.73		0.3
L17	11.35	11.85	6.1
L19	8.86	8.89	36
L20	5.24	5.92	12.2
L22	8.03	7.82	40
L23	9.07	9.08	23
L24	8.23	8.78	30
L25	12.34	12.01	25
L26	9.94	9.34	20
L27	9.10	9.73	40
L28	9.56	5.73	30
L31	9.21	9.02	3.1
L32	5.92	6.28	50.9

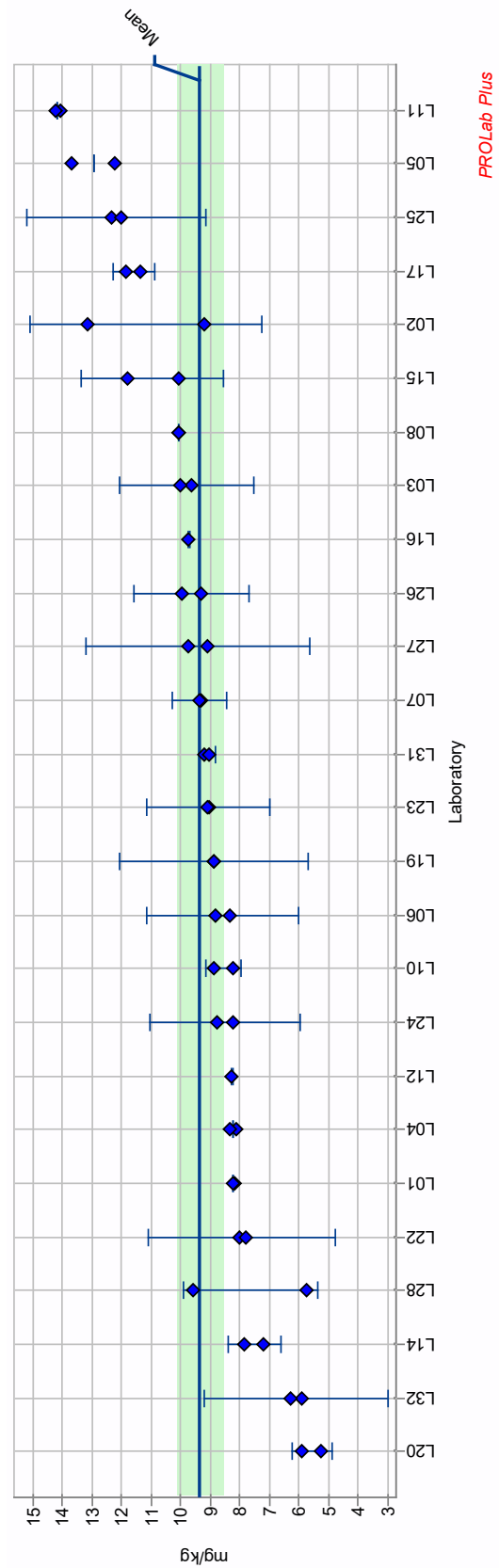


Figure 18. MOSH in IF03F.

Table 19: Reported results for MOAH vs 2MN in IF03G

Lab	M 1	M 2	MU [%]
L01	2.73	2.60	0
L02	2.71	2.66	1.67
L03	2.23	2.25	13
L04	3.32	2.68	0.9
L05	3.23	3.26	0
L06	2.52	2.36	30
L07	2.31	2.35	20
L08	2.32	2.46	
L10	2.17	2.33	7
L11	4.35	4.18	0.02
L12	1.61	1.93	0.33
L14	1.92	1.66	20.9
L15	2.38	2.80	23
L16	2.67	2.70	0.3
L17	2.16	2.22	3.8
L19	2.46	2.48	36
L20	2.25	2.25	0.3
L22	2.33	2.32	40
L23	2.41	2.17	28
L24	2.24	2.20	30
L25	3.18	3.43	25
L26	2.85	3.14	20
L27	2.16	1.77	43
L28	2.62	3.27	30
L31	2.45	2.53	4.9
L32	2.76	2.72	5.6

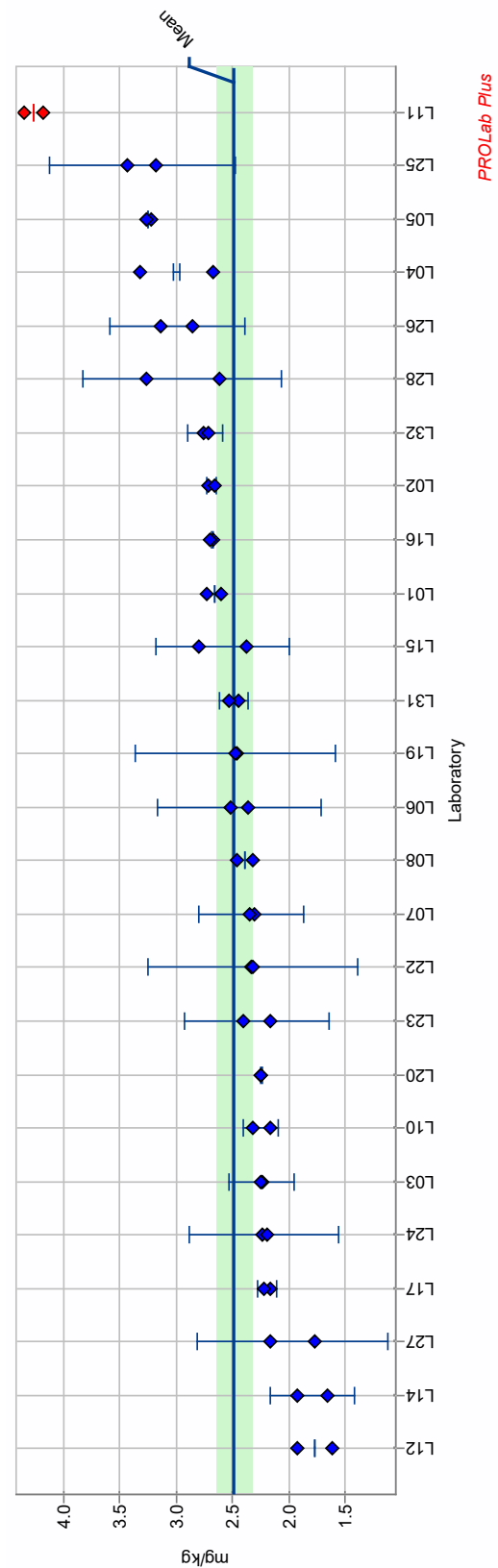


Figure 19. MOAH2MN in IF03G.
Results in red are outliers
B - Grubbs (laboratory mean),

Table 20: Reported results for MOAH vs TBB in IF03G

Lab	M 1	M 2	MU [%]
L01	2.11	2.09	0
L02	2.51	2.55	1.6
L03	2.33	2.23	13
L04	2.84	2.43	0.5
L05	2.77	2.74	0
L06	2.04	1.86	0
L07	2.02	2.01	20
L08	1.85	2.18	
L10	2.08	2.23	6.7
L11	3.75	3.55	0.026
L12	1.29	1.47	0.33
L14	1.73	1.70	1.9
L15	2.10	2.52	26
L16	2.46	2.34	0.3
L17	1.86	1.88	1.5
L19	2.23	2.17	36
L20	2.06	2.04	1.12
L22	2.21	2.20	40
L23	2.16	1.94	28.5
L24	2.09	1.96	30
L25	2.99	2.89	25
L26	2.27	2.35	20
L27	1.80	1.46	43
L28	2.07	2.43	30
L31	2.26	2.27	0.5
L32	2.36	2.32	5.6

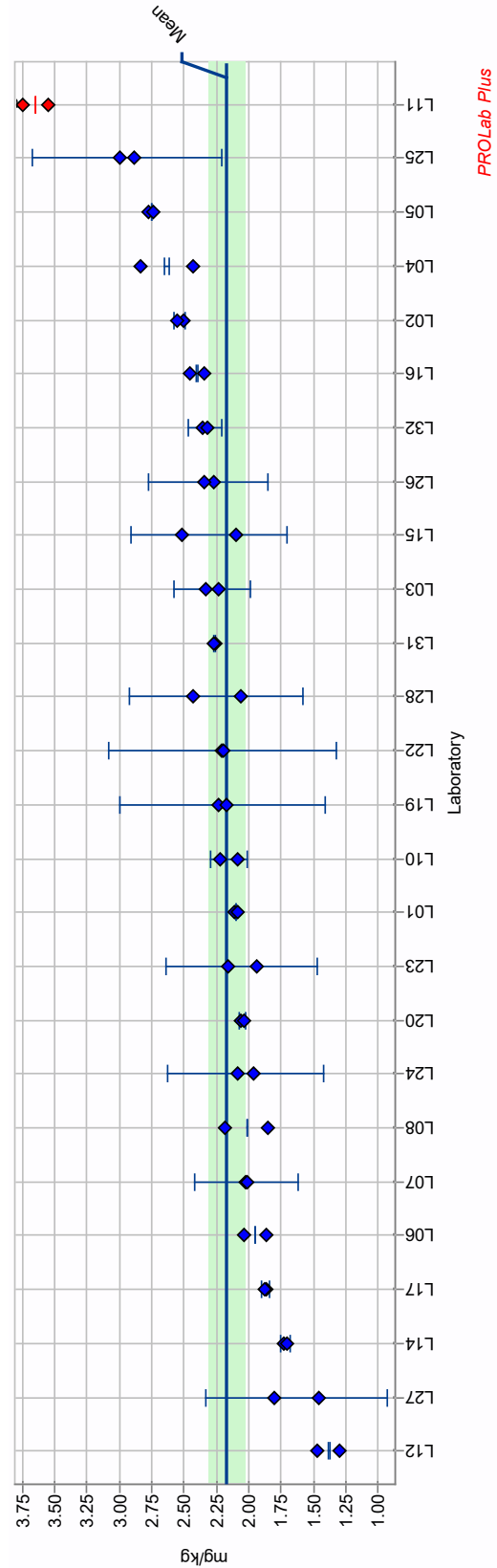


Figure 20. MOAH2MN in IF03G.

Results in red are outliers:

B - Grubbs (laboratory mean),

Table 21: Reported results for MOSH vs CyCy in IF03G

Lab	M 1	M 2	MU [%]
L01	10.2	9.8	0
L02	11.8	12.2	3.1
L03	11.4	12.8	23
L04	14.7	10.2	5.57
L05	13.3	14.2	0
L06	10.4	9.8	30
L07	11.2	11.2	10
L08	12.4	11.9	
L10	9.3	9.9	7.15
L11	19.0	17.5	0.04
L12		10.5	0.17
L14	9.5	9.3	3.7
L15	12.5	14.6	21
L16	12.8		0.3
L17	14.3	15.2	8.9
L19	10.4	10.6	36
L20	7.5	7.6	1.3
L22	9.9	10.0	40
L23	11.3	11.5	22.2
L24	9.5	10.2	30
L25	14.0	14.0	25
L26	10.7	11.2	20
L27	11.3	12.6	40
L28	9.9	13.4	30
L31	12.3	11.3	12.6
L32	7.6	7.5	12.7

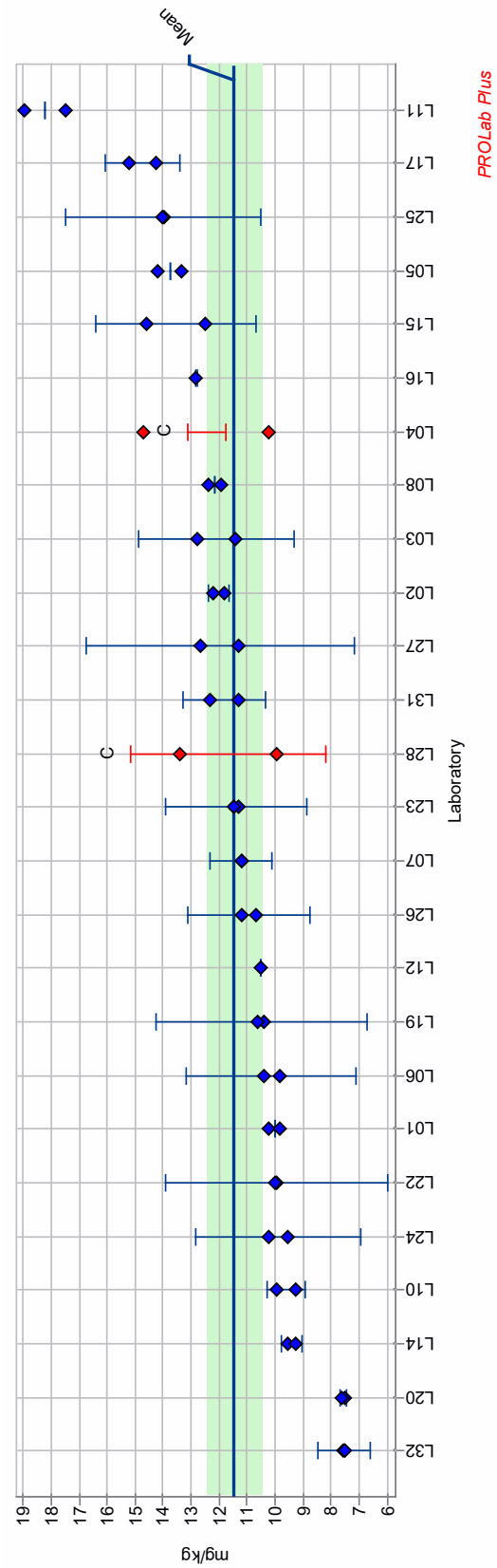


Figure 21. MOSH in IF03G.
Results in red are outliers:
C – Cochran (excessive standard deviation)

Table 22: Reported results for MOAH vs 2MN in IF03H

Lab	M 1	M 2	MU [%]
L01	1.30	1.49	0
L02	1.93	2.04	5.7
L03	0.92	1.12	13
L04	1.72	2.00	0.4
L05	2.10	2.07	0
L06	1.35	1.33	30
L07	1.21	1.24	20
L08	1.34	0.95	
L10	1.28	1.22	4.9
L11	3.47	3.27	0.03
L12	0.69	0.73	0.33
L14	0.87	0.87	0.47
L15	1.14	1.43	32
L16	1.89	1.45	0.3
L17	0.93	0.96	4.49
L19	1.41	1.40	36
L20	1.19	1.38	15.1
L22	1.29	1.29	40
L23	1.46	1.31	30.2
L24	1.55	1.46	30
L25	2.05	2.17	25
L26	1.65	1.53	20
L27	1.07	0.84	43
L28	1.23	1.55	30
L31	1.37	1.18	20.3
L32	1.48	1.59	15.5

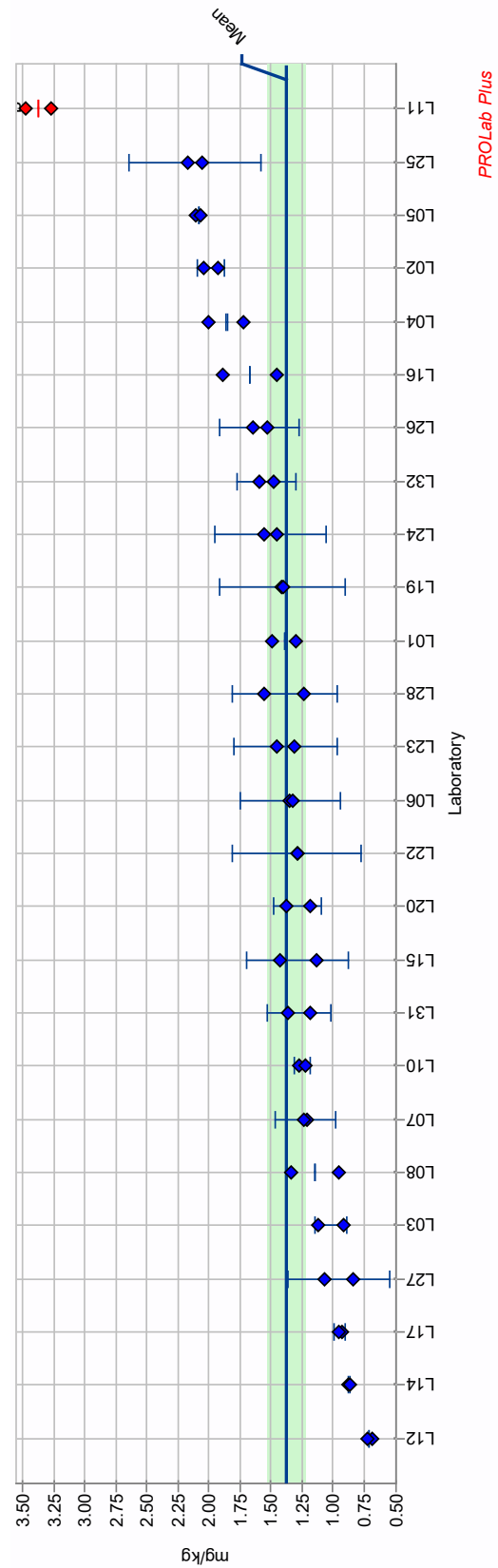


Figure 22. MOAH2MN in IF03H.
Results in red are outliers:
B - Grubbs (laboratory mean),

Table 23: Reported results for MOAH vs TBB in IF03H

Lab	M 1	M 2	MU [%]
L01	1.02	1.16	0
L02	1.73	1.92	10.4
L03	1.01	1.15	13
L04	1.49	1.81	0.44
L05	1.87	1.80	0
L06	1.10	1.06	0
L07	1.07	1.09	20
L08	1.14	0.84	
L10	1.17	1.25	6.8
L11	2.80	2.32	0.09
L12	0.55	0.57	0.33
L14	0.85	0.89	6.65
L15	0.98	1.27	36
L16	1.67	1.23	0.3
L17	0.79	0.78	1.8
L19	1.25	1.24	36
L20	1.09	1.23	11.5
L22	1.21	1.20	40
L23	1.28	1.13	30.8
L24	1.43	1.24	30
L25	1.93	1.82	25
L26	1.30	1.25	20
L27	0.87	0.65	43
L28	1.09	1.10	30
L31	1.21	1.11	12.7
L32	1.26	1.35	12.7

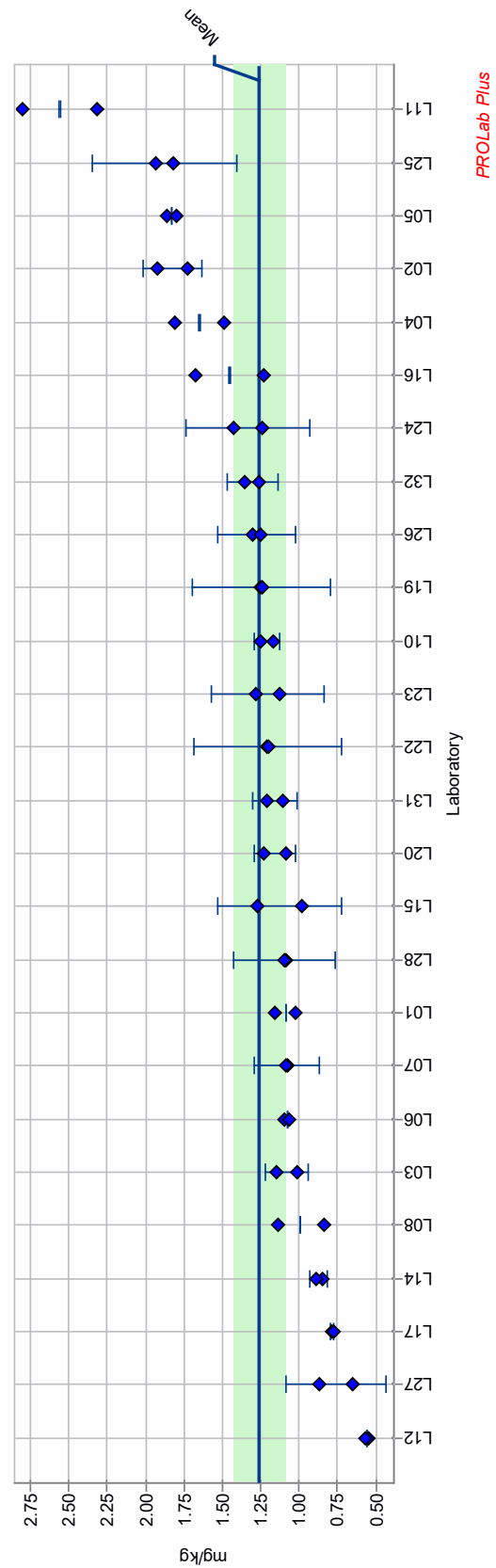


Figure 23. MOAHTBB in IF03H.

Table 24: Reported results for MOSH vs CyCy in IF03H

Lab	M 1	M 2	MU [%]
L01	7.33	8.22	0
L02	9.99	10.37	3.7
L03	8.67	8.50	23
L04	9.32	8.83	0.7
L05	9.36	10.35	0
L06	7.94	7.17	30
L07	8.08	8.10	10
L08	8.77	8.26	
L10	7.59	7.06	7.2
L11	11.50	11.53	0.001
L12		6.58	0.17
L14	7.56	7.09	9.1
L15	8.80	10.30	19
L16	8.04		0.3
L17	8.51	9.25	11.7
L19	8.17	8.06	36
L20	6.52	6.36	2.4
L22	7.54	7.38	40
L23	9.20	9.12	22.9
L24	7.75	7.66	30
L25	10.19	10.54	25
L26	8.61	7.78	20
L27	6.65	8.09	40
L28	6.79	8.40	30
L31	9.31	8.04	20.7
L32	5.37	5.61	33.9

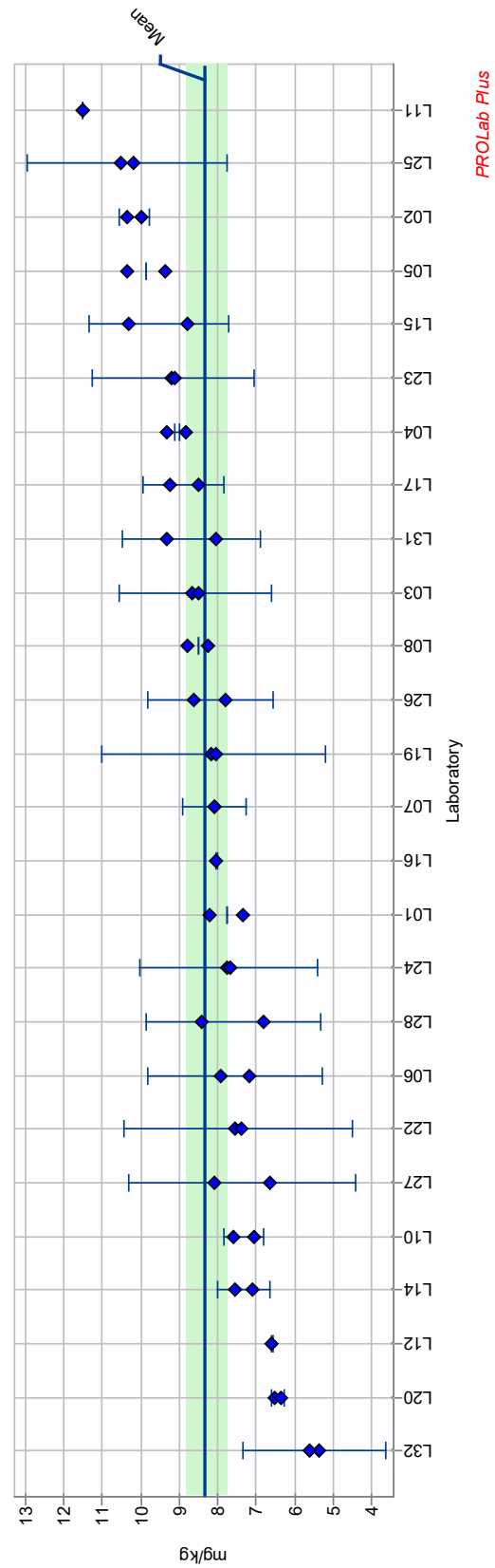


Figure 24. MOSH in IF03H.

Table 25: Reported results for MOAH vs 2MN in IF03I

Lab	M 1	M 2	MU [%]
L01	2.19	2.58	0
L02	2.78	2.91	4.47
L03	2.04	1.88	13
L04	2.45	2.14	0.45
L05	2.71	2.69	0
L06	2.25	2.16	30
L07	1.83	1.82	20
L08	1.73	1.84	
L10	2.12	2.13	0.8
L11	4.59	4.85	0.028
L12	1.58	1.47	0.33
L14	1.61	1.33	27
L15	2.36	1.96	26
L16	3.27	2.47	0.3
L17	1.66	1.70	3.3
L19	2.23	2.19	36
L20	1.73	1.81	4.5
L22	2.01	2.01	40
L23	2.22	2.34	28.4
L24	2.53	2.38	30
L25	3.27	3.38	25
L26	2.65	2.46	20
L27	1.49	1.31	43
L28	2.24	2.37	30
L31	2.00	2.16	11.2
L32	2.34	2.33	1.4

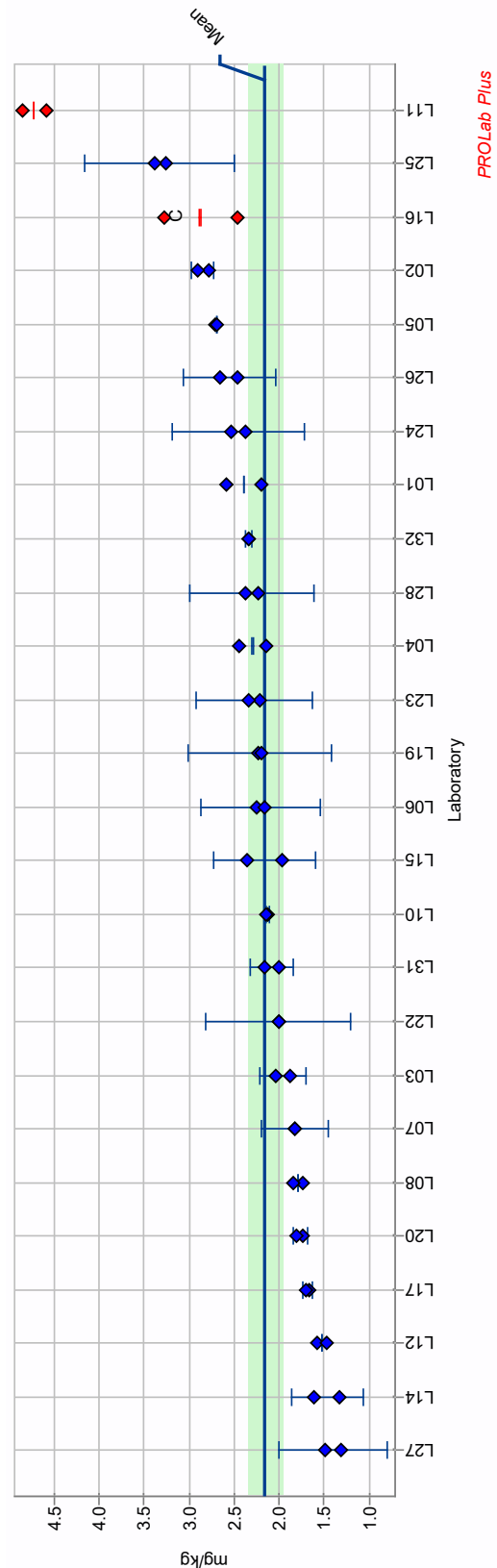


Figure 25. MOAH2MN in IF03I.
 Results in red are outliers:
 C – Cochran (excessive standard deviation)
 B - Grubbs (laboratory mean),

Table 26: Reported results for MOAH vs TBB in IF03I

Lab	M 1	M 2	MU [%]
L01	1.76	1.93	0
L02	2.61	2.71	3.5
L03	2.30	1.93	13
L04	2.08	1.95	0.18
L05	2.43	2.43	0
L06	1.81	1.69	0
L07	1.62	1.61	20
L08	1.48	1.60	
L10	2.01	2.13	6.1
L11	3.38	3.78	0.05
L12	1.08	1.15	0.33
L14	1.53	1.34	18.5
L15	2.06	1.72	25
L16	2.89	2.01	0.3
L17	1.42	1.44	1.9
L19	1.94	1.92	36
L20	1.52	1.62	6.3
L22	1.85	1.90	40
L23	1.91	1.97	29
L24	2.29	1.99	30
L25	2.83	2.76	25
L26	1.98	1.95	20
L27	1.15	1.07	43
L28	1.74	1.77	30
L31	1.82	2.08	18.9
L32	2.00	1.99	1.4

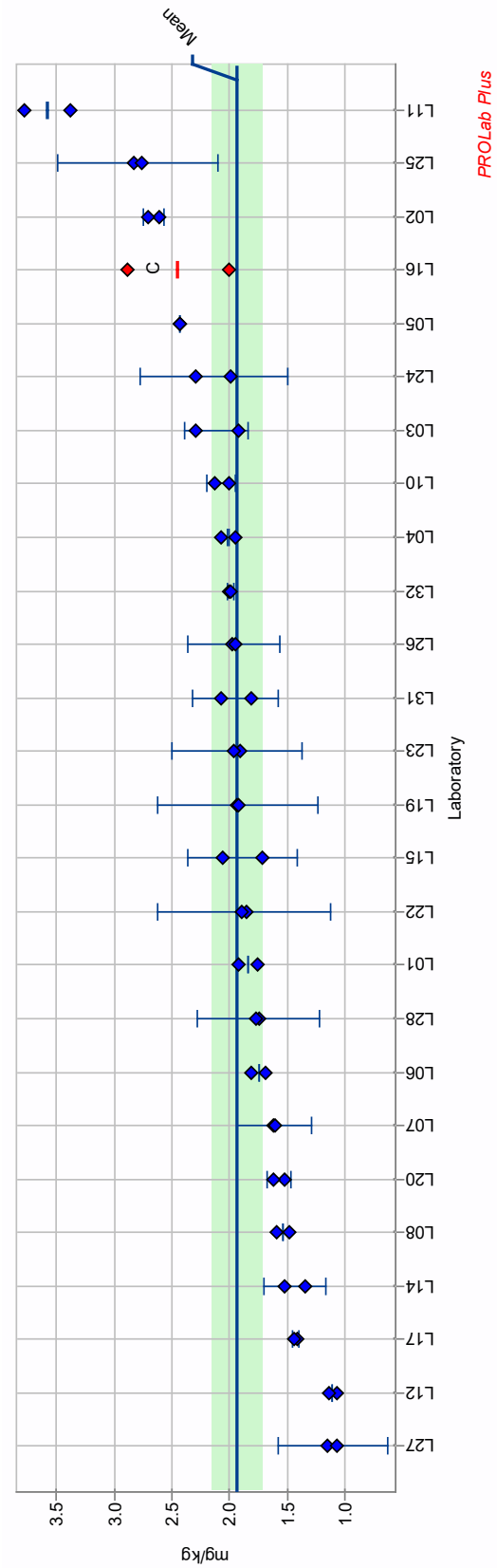


Figure 26. MOAHTBB in IF03I.
 Results in red are outliers:
 C – Cochran (excessive standard deviation)

Table 27: Reported results for MOSH vs CyCy in IF03I

Lab	M 1	M 2	MU [%]
L01	9.4	10.5	0
L02	12.4	10.9	12.5
L03	11.9	11.3	23
L04	11.3	8.8	3.5
L05	13.1	12.0	0
L06	10.1	9.2	30
L07	9.8	9.8	10
L08	11.7	11.2	
L10	8.2	8.5	4.6
L11	15.3	15.3	0.002
L12	9.0	8.5	0.17
L14	9.4	8.6	12.9
L15	12.6	13.1	4.9
L16	10.2		0.3
L17	10.3	10.9	7.3
L19	10.3	10.1	36
L20	7.6	7.9	3.3
L22	9.2	9.2	40
L23	11.1	11.9	22.3
L24	11.1	10.3	30
L25	14.0	13.6	25
L26	10.2	9.9	20
L27	10.1	10.4	40
L28	10.4	10.1	30
L31	10.4	11.4	13
L32	7.0	7.2	41.0

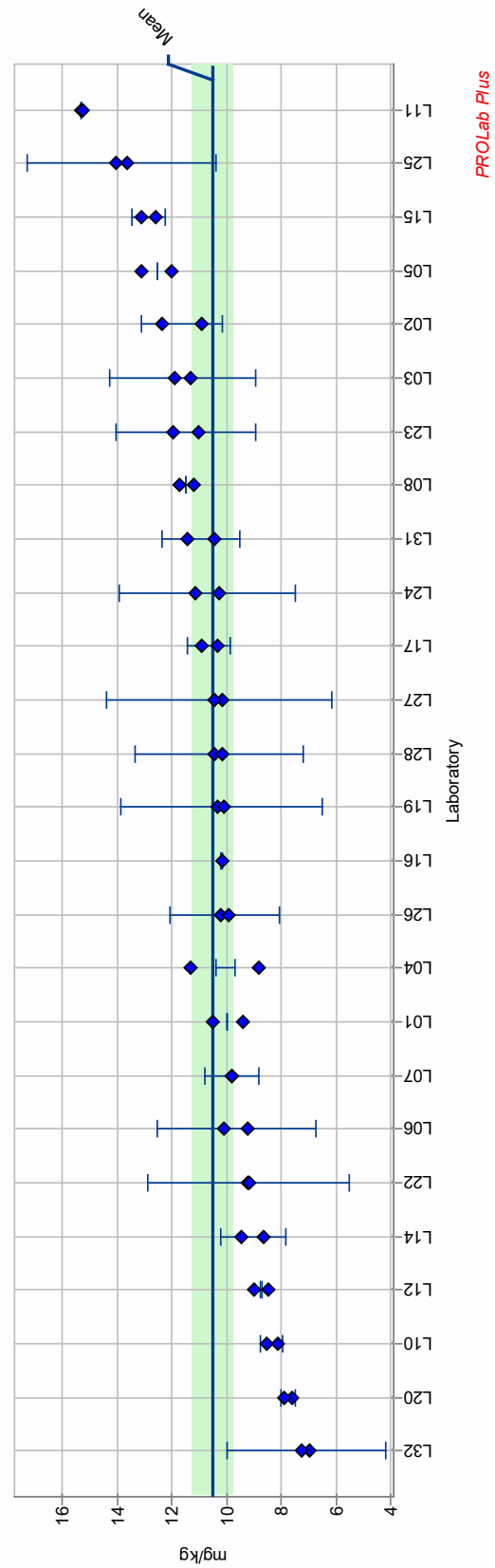


Figure 27. MOSH in IF03I.

Table 28: Reported results for MOAH vs 2MN in IF03J

Lab	M 1	M 2	MU [%]
L01	3.01	3.17	0
L02	3.64	5.13	33.9
L03	2.96	2.70	13
L04	3.22	3.13	0.13
L05	3.83	3.47	0
L06	2.94	2.95	30
L07	2.75	2.82	20
L08	2.86	2.59	
L10	2.82	2.61	8
L11	5.09	4.90	0.019
L12	2.12	2.18	0.33
L14	2.20	2.02	12.3
L15	3.15	4.26	43
L16	3.23	2.94	0.3
L17	2.55	2.41	7.9
L19	3.14	3.09	36
L20	2.34	2.56	8.6
L22	2.60	2.61	40
L23	2.95	2.43	27.2
L24	2.92	3.04	30
L25	3.78	4.25	25
L26	3.24	3.39	20
L27	2.09	1.89	43
L28	2.76	2.64	30
L31	3.23	3.22	0.5
L32	3.44	3.53	12.7

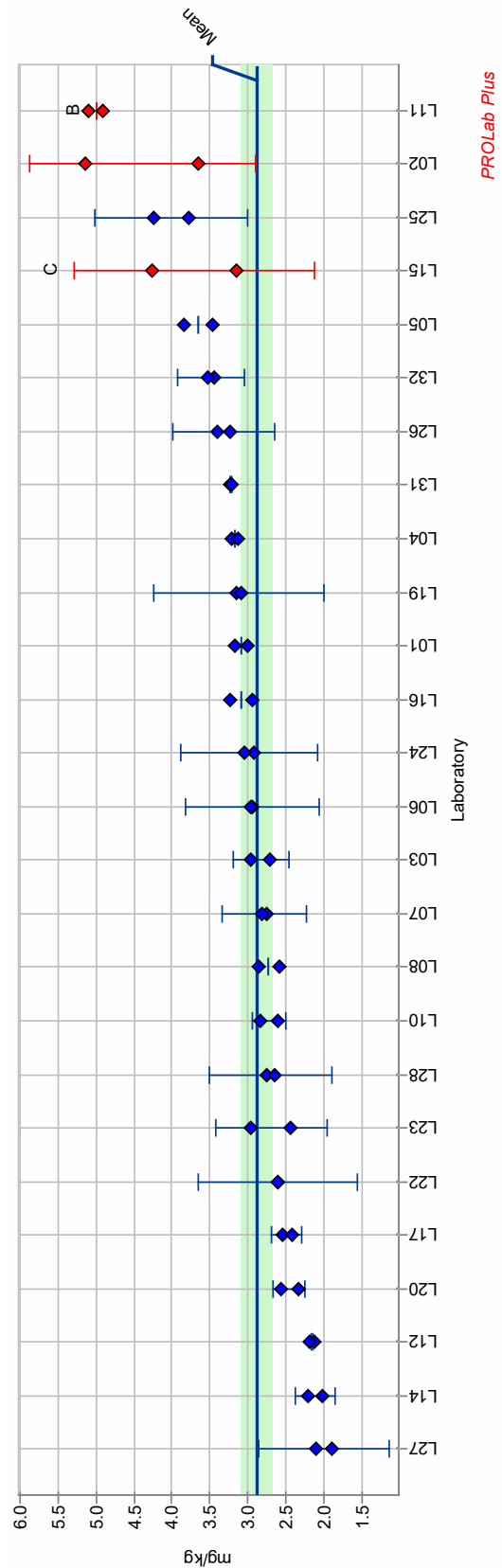


Figure 28. MOAH2MN in IF03J.
 Results in red are outliers:
 B - Grubbs (laboratory mean),
 C - Cochran (excessive standard deviation)

Table 29: Reported results for MOAH vs TBB in IF03J

Lab	M 1	M 2	MU [%]
L01	2.38	2.45	0
L02	3.42	3.90	13.1
L03	3.07	2.77	13
L04	2.77	2.71	0.09
L05	3.53	3.18	0
L06	2.45	2.15	0
L07	2.21	2.25	20
L08	2.40	2.32	
L10	2.69	2.62	3
L11	4.15	3.90	0.032
L12	1.49	1.69	0.33
L14	2.17	2.03	9.3
L15	2.79	3.78	43
L16	3.02	2.51	0.3
L17	2.16	2.05	7.3
L19	2.73	2.66	36
L20	2.16	2.30	6.1
L22	2.47	2.48	40
L23	2.47	2.02	27.9
L24	2.69	2.75	30
L25	3.46	3.61	25
L26	2.58	2.52	20
L27	1.69	1.57	43
L28	2.38	2.03	30
L31	3.05	3.01	2
L32	2.94	3.00	8.4

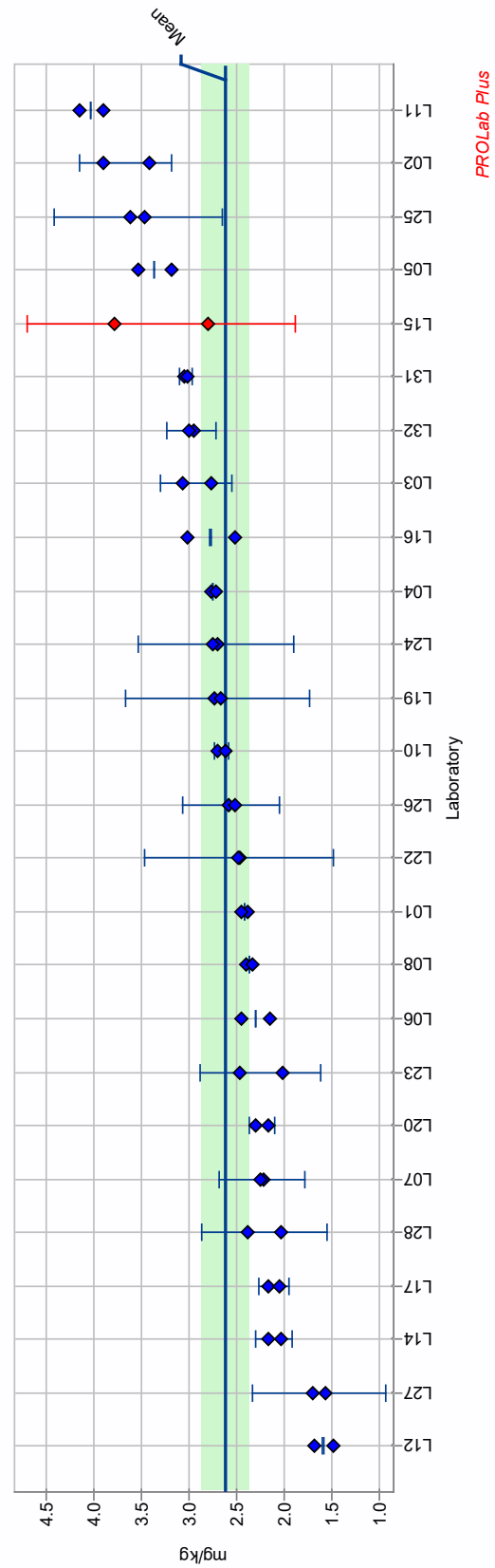


Figure 29. MOAHTBB in IF03J.
 Results in red are outliers:
 C – Cochran (excessive standard deviation)

Table 30: Reported results for MOSH vs CyCy in IF03J

Lab	M 1	M 2	MU [%]
L01	11.0	11.6	0
L02	12.3	12.5	1.6
L03	14.6	13.6	23
L04	13.7	11.5	3.1
L05	17.9	15.0	0
L06	11.4	11.2	30
L07	12.0	12.0	10
L08	13.1	13.3	
L10	10.2	10.8	5.2
L11	20.0	19.0	0.024
L12	14.1		0.17
L14	11.0	10.9	1.3
L15	15.8	23.0	52
L16	12.9		0.3
L17	14.5	13.7	8.5
L19	12.6	13.0	36
L20	9.6	9.9	3.8
L22	10.5	10.5	40
L23	13.4	13.9	21.7
L24	11.4	12.3	30
L25	15.2	18.4	25
L26	12.2	12.0	20
L27	11.8	10.3	40
L28	10.8	10.9	30
L31	15.4	14.5	8.3
L32	8.5	8.9	50.9

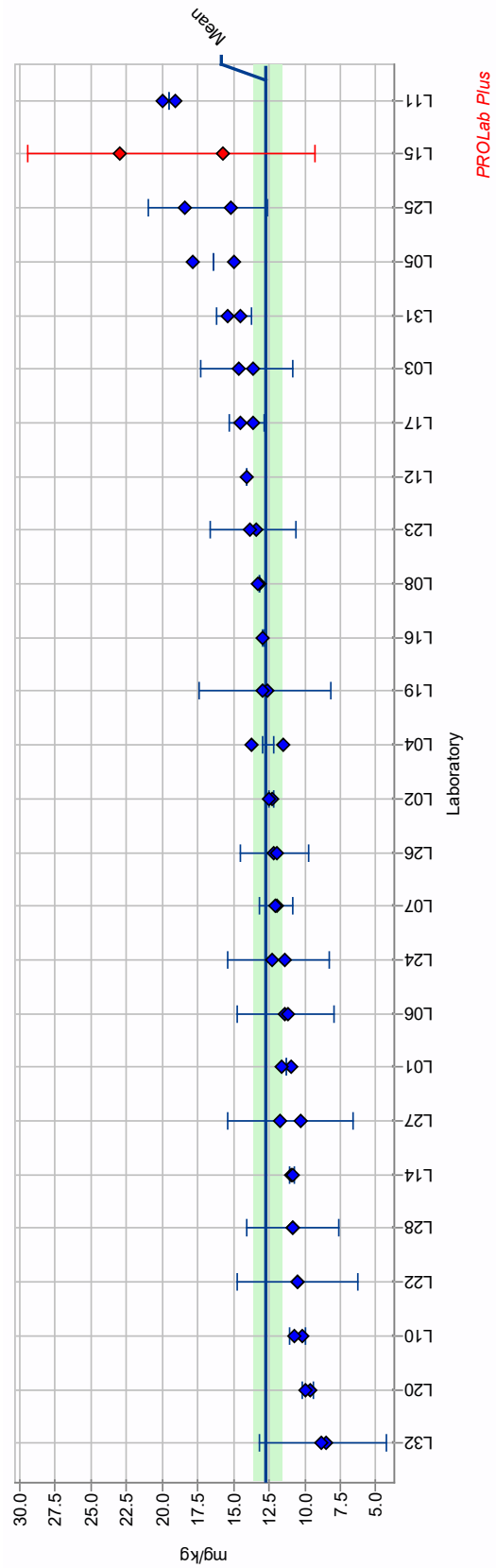


Figure 30. MOSH in IF03J.
 Results in red are outliers:
 C – Cochran (excessive standard deviation)

Table 31: Reported results for MOAH vs 2MN in IF03J

Lab	M 1	M 2	MU [%]
L01	5.81	6.21	0
L02	8.46	8.37	1.06
L03	5.10	4.71	13
L04	5.93	5.25	0.9
L05	6.30	6.24	0
L06	5.52	5.04	30
L07	4.94	5.01	20
L08	5.11	4.67	
L10	5.13	4.81	6.6
L11	10.09	10.41	0.016
L12	5.25	4.43	0.33
L14	4.10	3.92	6.3
L15	7.26	5.86	30
L16	5.20	5.34	0.3
L17	5.37	5.23	3.7
L19	5.47	5.50	36
L20	4.14	4.36	5.1
L22	5.58	5.25	40
L23	5.77	4.76	24.6
L24	5.62	5.50	30
L25	5.72	6.29	25
L26	6.36	6.11	20
L27	4.24	4.14	43
L28	4.46	5.06	30
L31	5.51	5.69	4.7
L32	6.33	6.42	12.7

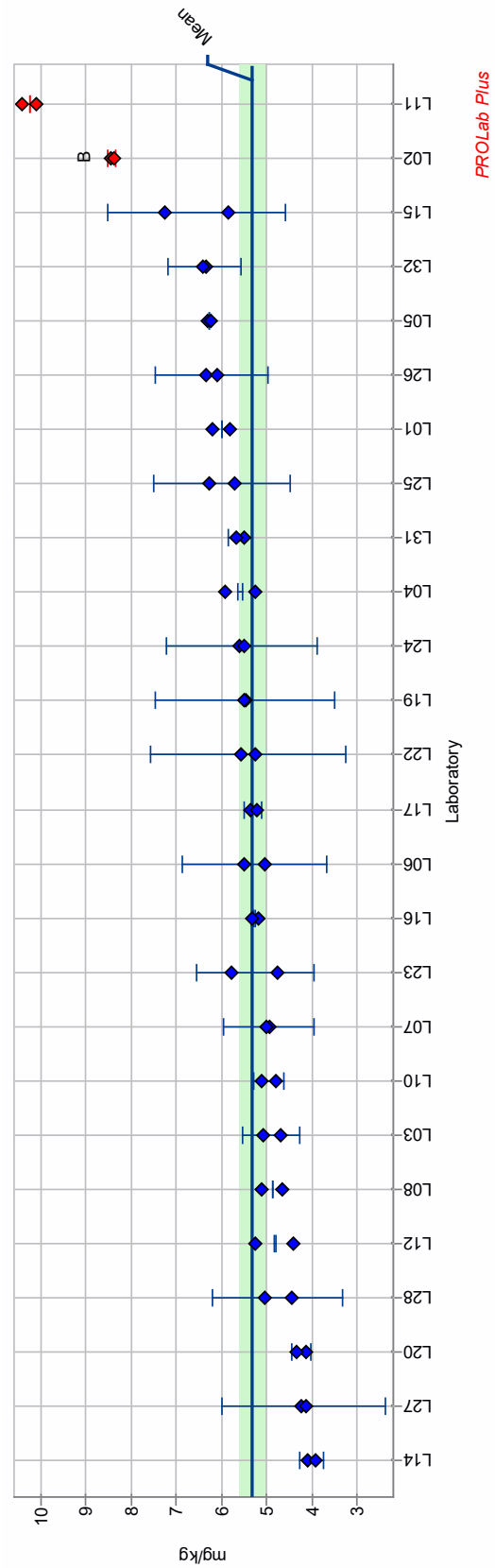


Figure 31. MOAH2MN in IF03J.
Results in red are outliers:
B - Grubbs (laboratory mean),

Table 32: Reported results for MOAH vs TBB in IF03K

Lab	M 1	M 2	MU [%]
L01	4.62	4.66	0
L02	8.30	8.11	2.2
L03	5.41	5.01	13
L04	5.25	4.44	1.14
L05	5.78	5.58	0
L06	4.46	4.02	0
L07	4.42	4.44	20
L08	4.34	4.22	
L10	4.88	4.73	3.17
L11	8.39	7.79	0.037
L12	3.52	3.52	0.33
L14	4.17	3.80	13.2
L15	6.23	4.97	33
L16	5.15	4.73	0.3
L17	4.50	4.41	2.8
L19	4.86	5.00	36
L20	3.78	3.92	3.7
L22	5.09	4.97	40
L23	5.02	4.12	25.1
L24	5.16	5.02	30
L25	5.80	6.03	25
L26	4.75	4.72	20
L27	3.49	3.48	43
L28	4.09	4.54	30
L31	5.27	5.43	4.3
L32	5.39	5.49	14.1

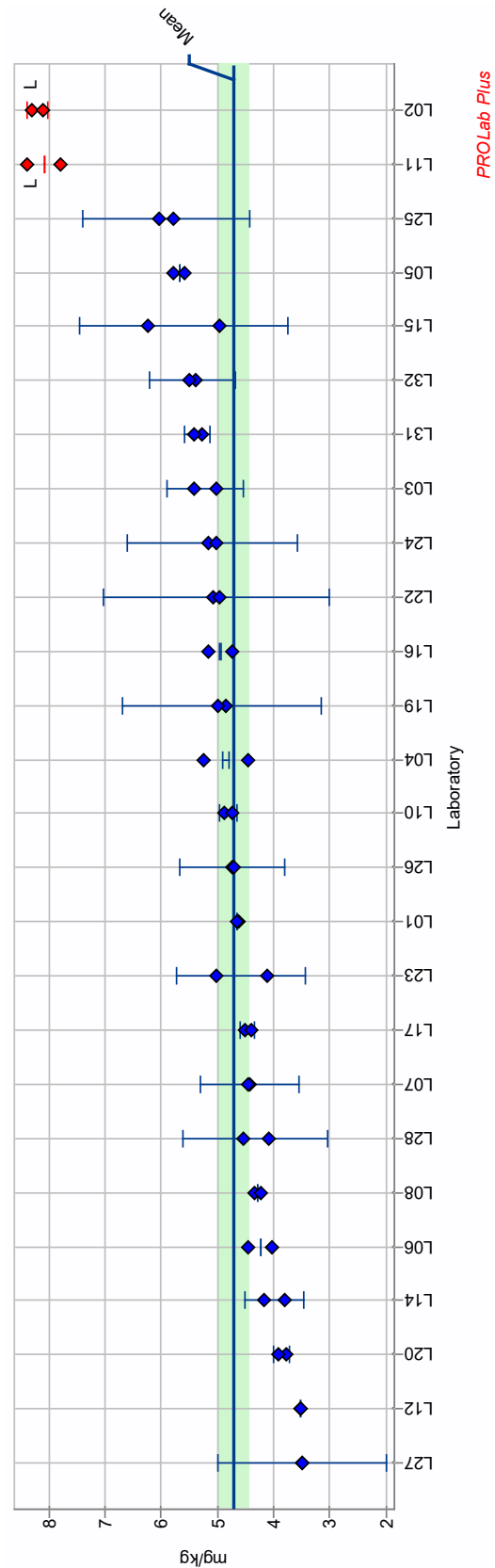


Figure 32. MOAHTBB in IF03K.
 Results in red are outliers:
 L - Grubbs (laboratory mean for 2),

Table 33: Reported results for MOSH vs CyCy in IF03K

Lab	M 1	M 2	MU [%]
L01	17.5	18.4	0
L02	21.9	21.9	0.14
L03	21.0	19.7	23
L04	18.4	19.0	0.85
L05	22.7	23.4	0
L06	18.7	16.9	30
L07	17.6	17.5	10
L08	20.8	19.6	
L10	17.0	15.8	7.3
L11	34.6	35.7	0.015
L12	17.3	19.1	0.17
L14	17.1	16.6	3.7
L15	30.2	23.7	34
L16	20.5		0.3
L17	23.1	22.4	3.9
L19	18.4	18.8	36
L20	14.7	14.6	0.29
L22	18.8	17.1	40
L23	21.3	21.2	20.2
L24	17.9	18.5	30
L25	21.9	21.5	25
L26	20.6	19.3	20
L27	18.3	17.9	40
L28	17.5	18.4	30
L31	16.6	16.5	1.1
L32	13.5	13.4	5.65

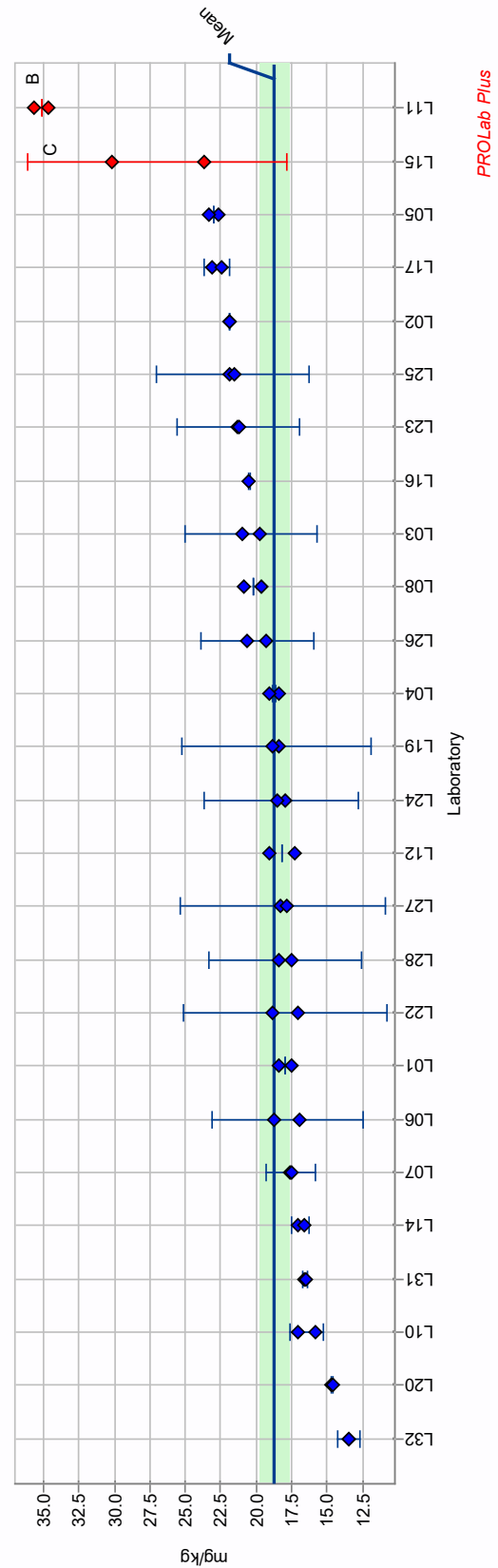


Figure 33. MOSH in IF03K.
 Results in red are outliers:
 B - Grubbs (laboratory mean),
 C - Cochran (excessive standard deviation)

Table 34: Reported results for MOAH vs 2MN in IF03L

Lab	M 1	M 2	MU [%]
L01	1.10	1.26	0
L02	1.52	1.78	16.1
L03	0.86	0.91	13
L04	1.44	1.20	0.33
L05	1.54	1.42	0
L06	1.18	1.10	30
L07	1.08	1.11	20
L08	0.90	1.13	
L10	1.24	1.55	22.6
L11	0.38		0
L12	0.55	0.68	0.33
L14	0.72	0.64	17
L15	1.33	1.28	5.9
L16	1.09	0.98	0.3
L17	0.87	0.92	7.9
L19	1.20	1.20	36
L20	0.85	0.88	5.8
L22	0.93	0.90	40
L23	1.14	1.07	31.4
L24	1.05	1.08	30
L25	1.41	1.32	25
L26	1.49	1.52	20
L27	1.00	0.64	43
L28	0.88	0.97	30
L31	1.24	1.10	18.6
L32	1.16	1.13	4.2

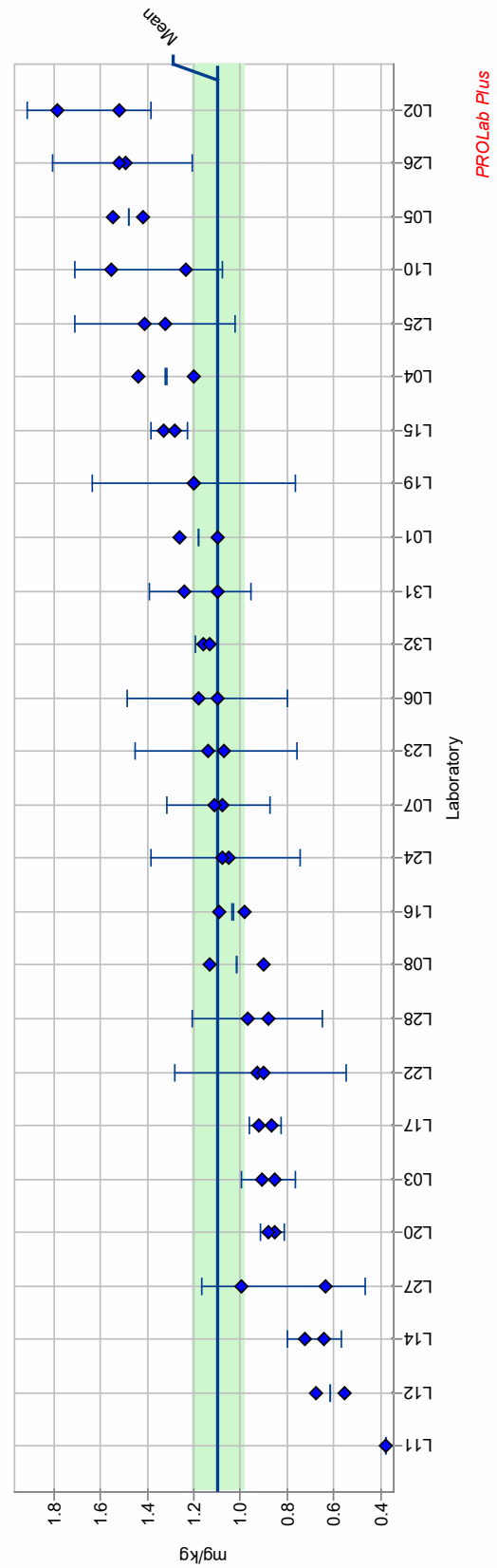


Figure 34. MOAH2MN in IF03L.

Table 35: Reported results for MOAH vs TBB in IF03L

Lab	M 1	M 2	MU [%]
L01	0.840	0.920	0
L02	1.473	1.803	20.1
L03	0.972	0.916	13
L04	1.320	1.070	0.34
L05	1.362	1.355	0
L06	0.950	0.910	0
L07	0.980	1.010	20
L08	0.743	1.006	
L10	1.123	1.506	29.1
L11	0.116		0
L12	0.429	0.478	0.33
L14	0.703	0.660	8.9
L15	1.190	1.120	7.7
L16	1.000	0.860	0.3
L17	0.700	0.710	2
L19	1.000	1.100	36
L20	0.777	0.797	2.6
L22	0.866	0.863	40
L23	1.020	0.940	31.9
L24	0.950	0.930	30
L25	1.666	1.637	25
L26	1.160	1.180	20
L27	0.596	0.460	43
L28	0.870	0.908	30
L31	1.220	1.070	18
L32	0.990	0.960	4.2

Figure 35. Summary graph for MOAHTBB in IF03L. Results in red are outliers: B - Grubbs (laboratory mean), C - Cochran (excessive standard deviation)

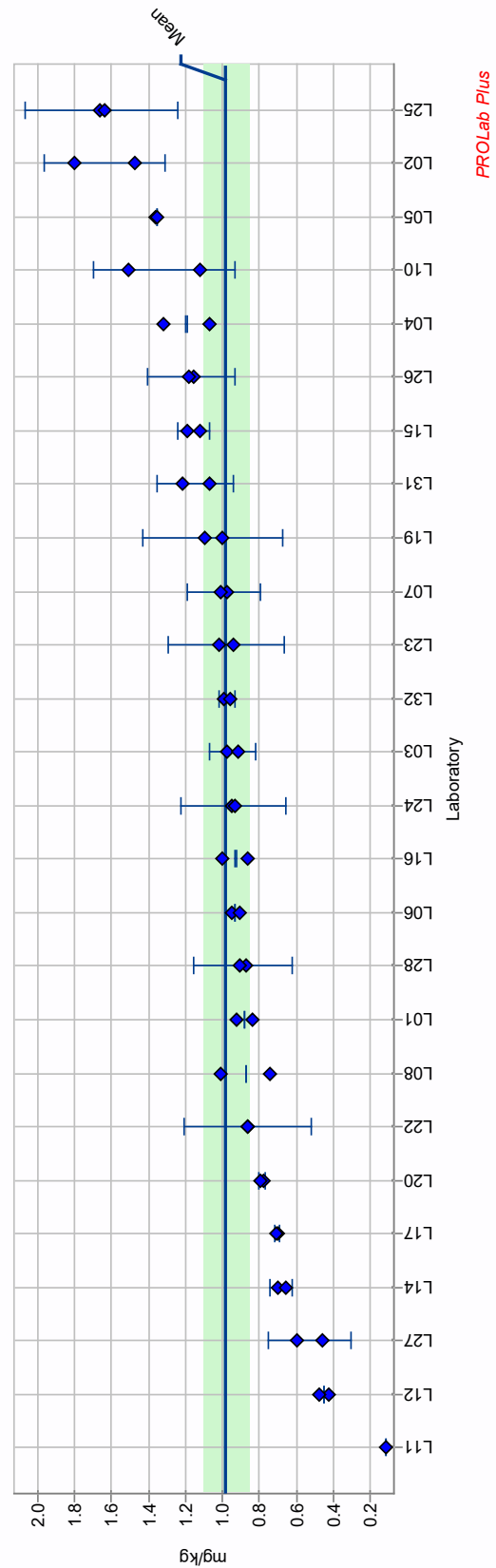


Table 36: Reported results for MOSH vs CyCy in IF03L

Lab	M 1	M 2	MU [%]
L01	5.74	6.42	0
L02	9.38	8.38	11.3
L03	6.64	7.29	23
L04	7.96	6.02	2.75
L05	8.06	7.77	0
L06	6.86	6.21	30
L07	7.71	7.69	10
L08	8.51	8.88	
L10	7.26	7.63	5
L11	12.27	14.24	0.074
L12	9.00	5.89	0.17
L14	6.09	5.85	5.4
L15	9.42	7.62	30
L16	7.21		0.3
L17	9.81	10.12	4.3
L19	6.80	6.28	36
L20	3.57	3.28	8.4
L22	5.53	5.44	40
L23	7.50	7.39	23.6
L24	6.63	5.91	30
L25	6.32	6.48	25
L26	6.40	6.94	20
L27	6.72	5.76	40
L28	6.08	6.26	30
L31	5.51	5.77	6.6
L32	4.34	4.43	12.7

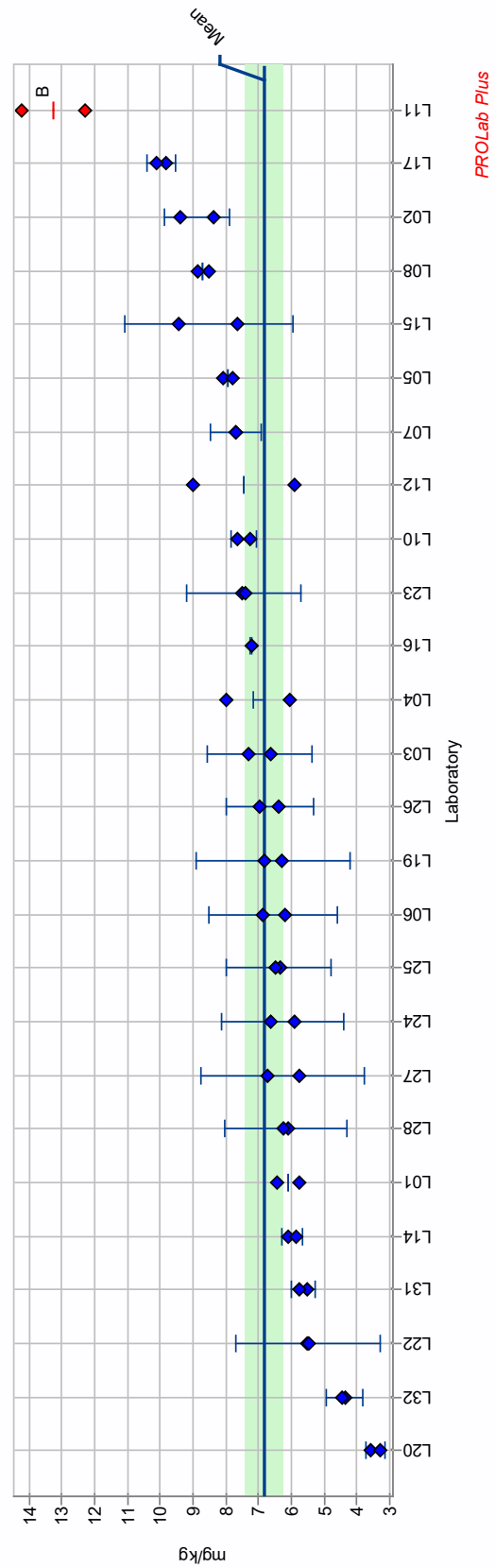


Figure 36. MOSH in IF03L.
Results in red are outliers:
B - Grubbs (laboratory mean),

Table 37: Reported results for MOAH vs 2MN in IF03M

Lab	M 1	M 2	MU [%]
L01	1.95	2.07	0
L02	2.22	2.71	19.8
L03	1.64	1.58	13
L04	2.41	2.02	0.5
L05	2.18	2.18	0
L06	1.91	1.82	30
L07	1.78	1.77	20
L08	1.62	1.94	
L10	1.55	1.78	13.8
L11	1.90	1.76	0.04
L12	1.37	1.29	0.33
L14	1.35	1.15	23.2
L15	4.28	5.42	33
L16	1.81	1.83	0.3
L17	1.99	2.30	20.4
L19	1.97	1.95	36
L20	1.51	1.68	11
L22	1.64	1.66	40
L23	1.88	1.80	29.1
L24	1.82	1.79	30
L25	2.00	2.25	25
L26	2.12	2.22	20
L27	1.24	1.17	43
L28	1.51	1.89	30
L31	1.71	1.81	8
L32	1.82	1.82	0

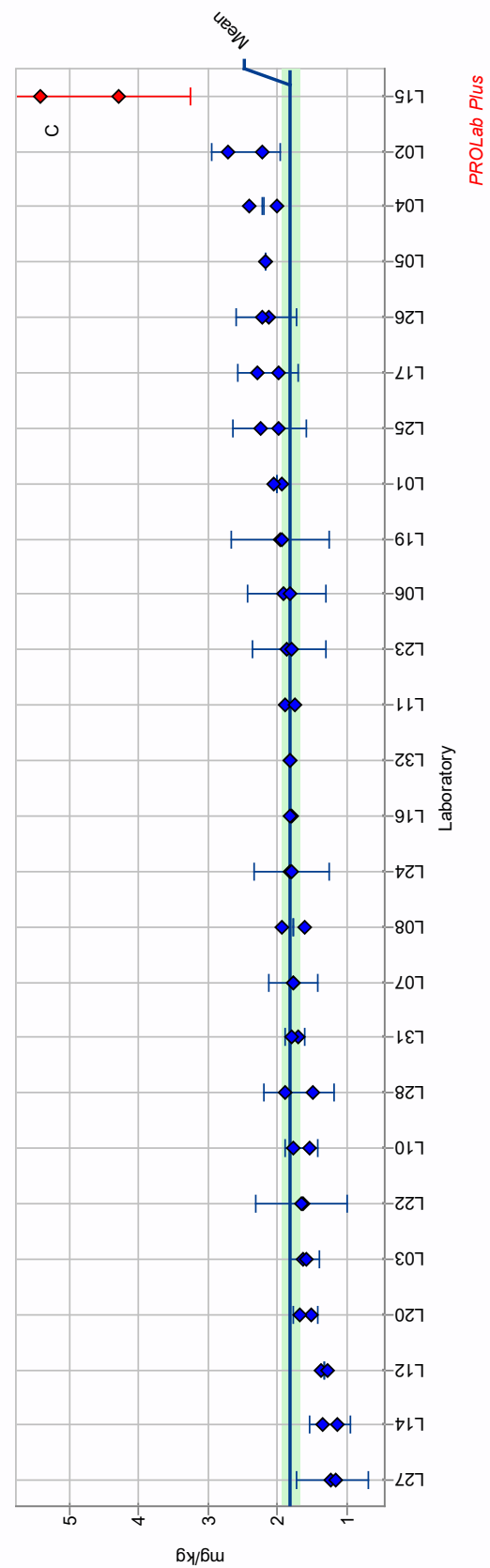


Figure 37. MOAH2MN in IF03M.
Results in red are outliers:
C – Cochran (excessive standard deviation)

Table 38: Reported results for MOAH vs TBB in IF03M

Lab	M 1	M 2	MU [%]
L01	1.50	1.53	0
L02	2.20	3.16	35.6
L03	1.90	1.59	13
L04	2.09	1.75	0.47
L05	2.05	2.05	0
L06	1.50	1.46	0
L07	1.59	1.56	20
L08	1.46	1.69	
L10	1.46	1.38	5.3
L11	1.50	1.45	0.02
L12	0.96	1.05	0.33
L14	1.25	1.17	9.5
L15	3.54	4.73	41
L16	1.71	1.62	0.3
L17	1.37	1.40	3
L19	1.78	1.68	36
L20	1.52	1.50	1.6
L22	1.54	1.57	40
L23	1.67	1.58	29.6
L24	1.68	1.65	30
L25	2.62	2.76	25
L26	1.73	1.74	20
L27	1.02	0.97	43
L28	1.46	1.57	30
L31	1.65	1.73	6.7
L32	1.56	1.57	1.4

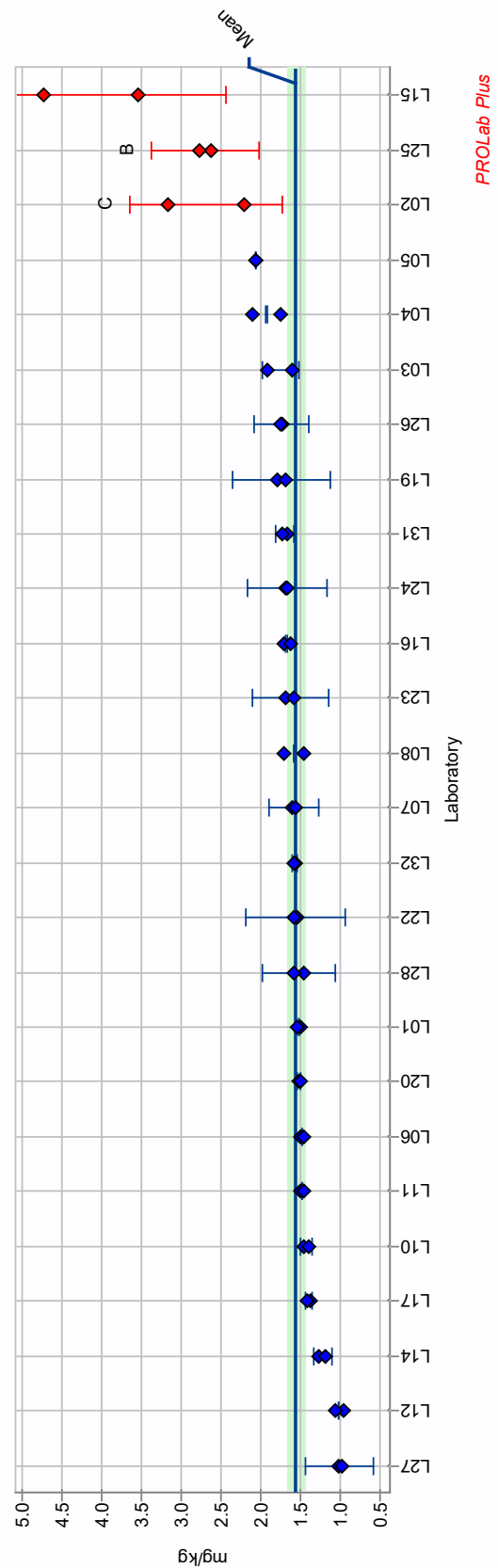


Figure 38. MOAHTBB in IF03M.
 Results in red are outliers:
 B - Grubbs (laboratory mean),
 C - Cochran (excessive standard deviation)

Table 39: Reported results for MOSH vs CyCY in IF03M

Lab	M 1	M 2	MU [%]
L01	7.91	8.27	0
L02	9.86	10.64	7.6
L03	10.70	9.56	23
L04	11.86	8.60	4.6
L05	9.80	10.52	0
L06	8.52	8.33	30
L07	8.83	8.76	10
L08	10.26	11.52	
L10	10.03	9.86	1.75
L11	9.97	10.31	0.017
L12	11.95	8.96	0.17
L14	7.78	7.10	13
L15	19.00	28.90	58
L16	9.09		0.3
L17	13.67	14.98	12.9
L19	8.12	8.64	36
L20	6.51	6.28	3.6
L22	7.20	7.22	40
L23	9.49	9.63	22.8
L24	8.17	8.47	30
L25	8.16	8.46	25
L26	8.07	8.36	20
L27	13.77	13.54	40
L28	7.90	7.40	30
L31	8.26	8.57	5.1
L32	5.39	5.45	8.4

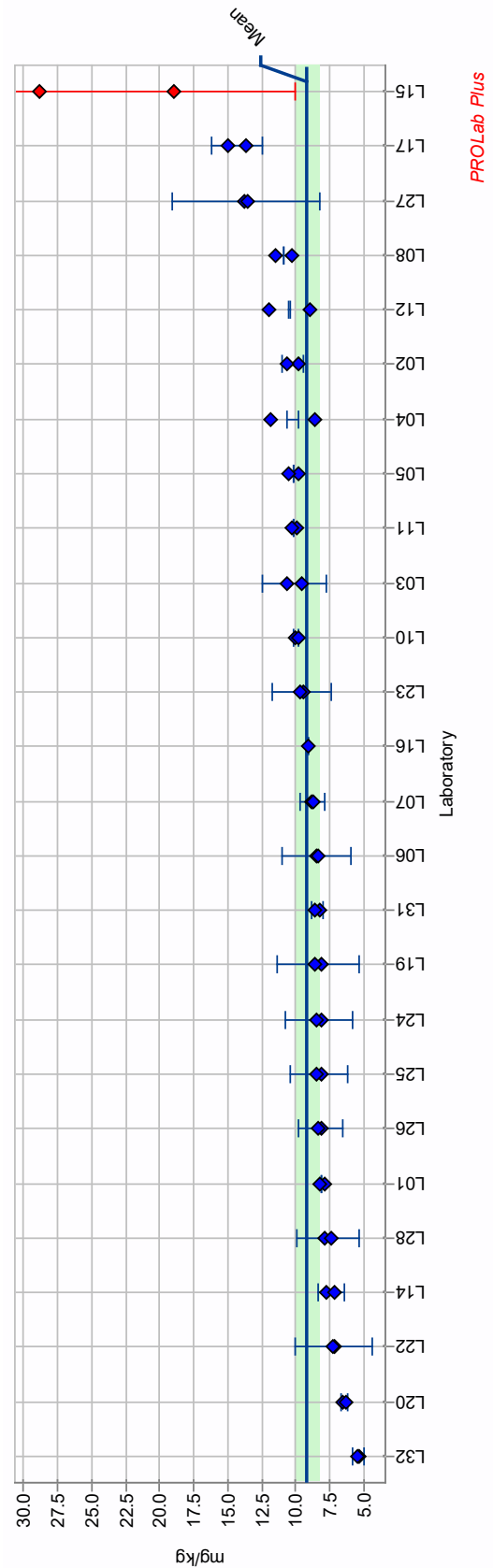


Figure 39. MOSH in IF03M.
Results in red are outliers:
C – Cochran (excessive standard deviation)

Table 40: Reported results for MOAH vs 2MN in IF03N

Lab	M 1	M 2	MU [%]
L01	5.34	6.10	0
L02	9.33	11.35	19.5
L03	4.89	4.69	13
L04	5.57	5.88	0.44
L05	5.95	6.40	0
L06	5.27	5.02	30
L07	4.82	4.79	15
L08	5.41	5.01	
L10	4.33	4.94	13.1
L11	9.11	8.91	0.01
L12	5.12	4.80	0.33
L14	3.96	3.56	15
L15	6.76	4.97	43
L16	5.97	5.69	0.3
L17	5.46	5.75	7.3
L19	5.60	5.37	36
L20	3.57	3.82	6.6
L22	5.14	5.15	40
L23	5.04	5.23	25.1
L24	5.21	5.09	30
L25	5.54	4.93	25
L26	5.85	6.10	20
L27	4.39	4.02	43
L28	4.41	4.88	30
L31	5.32	5.13	5.3
L32	5.29	5.70	57.9

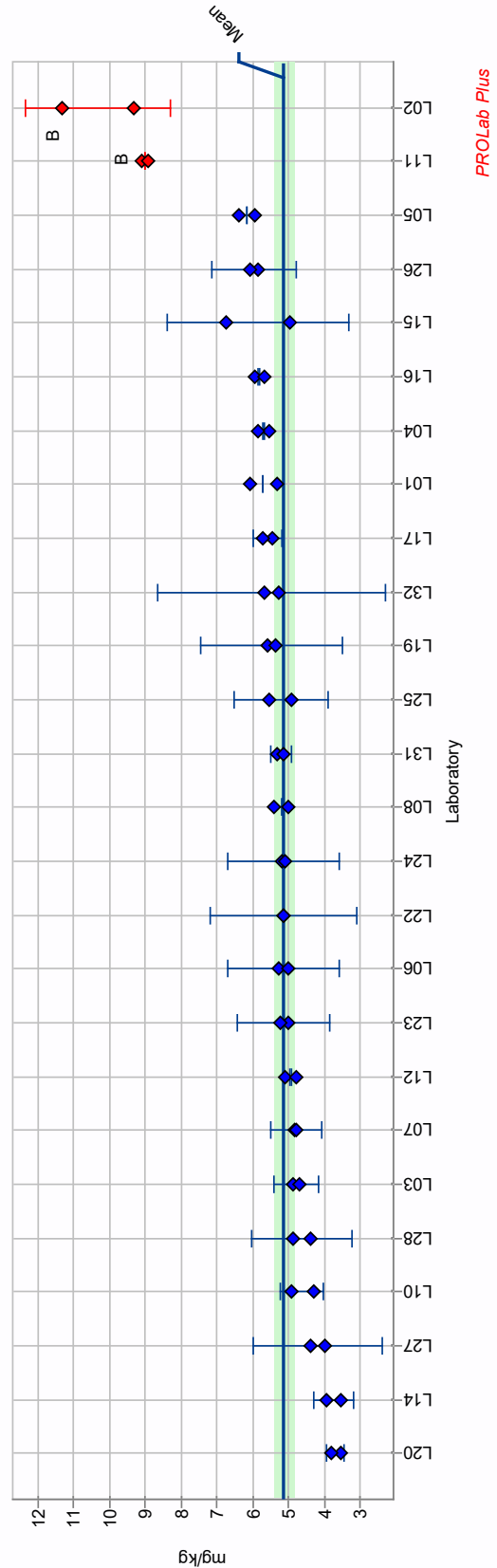


Figure 40. MOAH2MN in IF03N.
Results in red are outliers:
B - Grubbs (laboratory mean),

Table 41: Reported results for MOAH vs TBB in IF03N

Lab	M 1	M 2	MU [%]
L01	4.32	4.37	0
L02	9.72	11.90	20.1
L03	5.19	5.07	13
L04	4.80	5.07	0.38
L05	5.56	5.90	0
L06	4.28	3.86	0
L07	4.32	4.33	15
L08	4.85	4.55	
L10	4.16	4.46	7.09
L11	8.06	7.63	0.03
L12	3.50	3.77	0.33
L14	3.84	3.63	7.8
L15	8.28	5.13	66
L16	5.41	4.97	0.3
L17	4.35	4.32	1
L19	5.00	4.75	36
L20	3.16	3.43	8.
L22	4.78	4.69	40
L23	4.60	4.67	25.4
L24	4.84	4.65	30
L25	6.67	6.47	25
L26	4.53	4.74	20
L27	3.52	3.31	43
L28	4.16	4.37	30
L31	4.97	4.37	12.2
L32	4.60	4.89	41

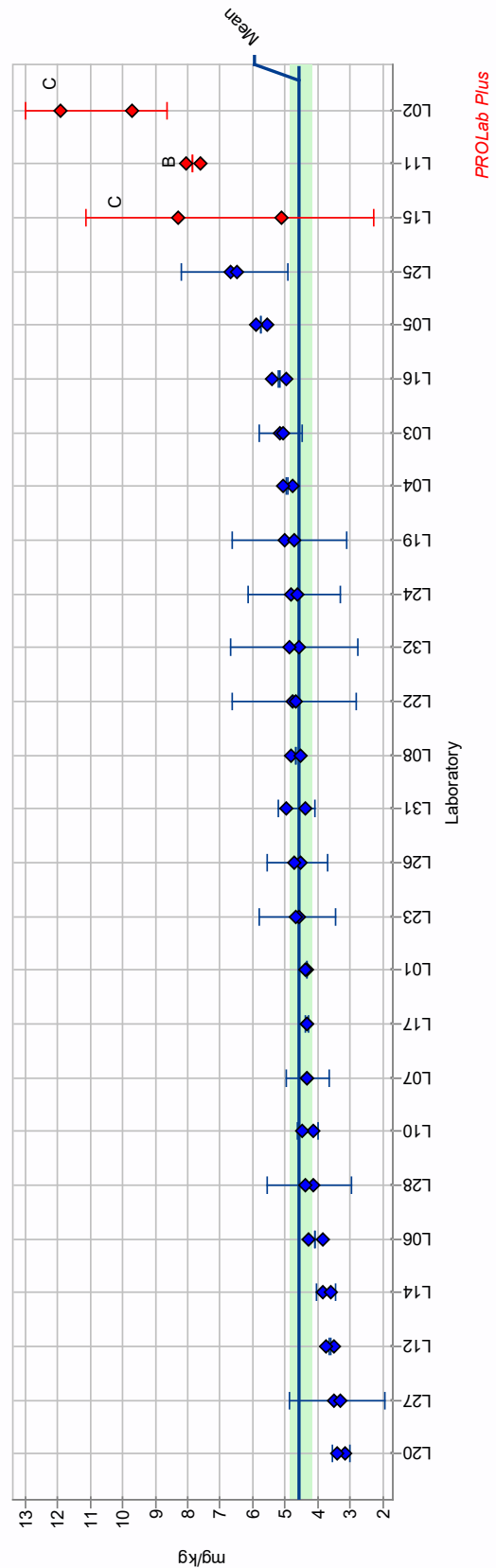


Figure 41. MOAHTBB in IF03N.
 Results in red are outliers:
 B - Grubbs (laboratory mean),
 C - Cochran (excessive standard deviation)

Table 42: Reported results for MOSH vs CyCY in IF03N

Lab	M 1	M 2	MU [%]
L01	16.1	17.2	0
L02	17.3	18.7	8
L03	20.3	19.3	23
L04	20.6	21.1	0.67
L05	23.2	26.5	0
L06	17.3	16.9	30
L07	17.3	17.4	10
L08	20.8	20.9	
L10	15.5	15.0	2.9
L11	31.6	30.1	0.02
L12	20.2	22.4	0.17
L14	16.4	14.6	16.1
L15	37.4	23.8	63
L16	19.0		0.3
L17	26.0	23.7	13.2
L19	17.0	17.5	36
L20	11.8	13.8	15.8
L22	16.3	16.6	40
L23	19.1	19.3	20.5
L24	17.4	17.2	30
L25	18.7	17.0	25
L26	18.2	19.1	20
L27	17.4	16.2	40
L28	16.6	16.6	30
L31	14.3	14.2	1.3
L32	11.3	12.3	152

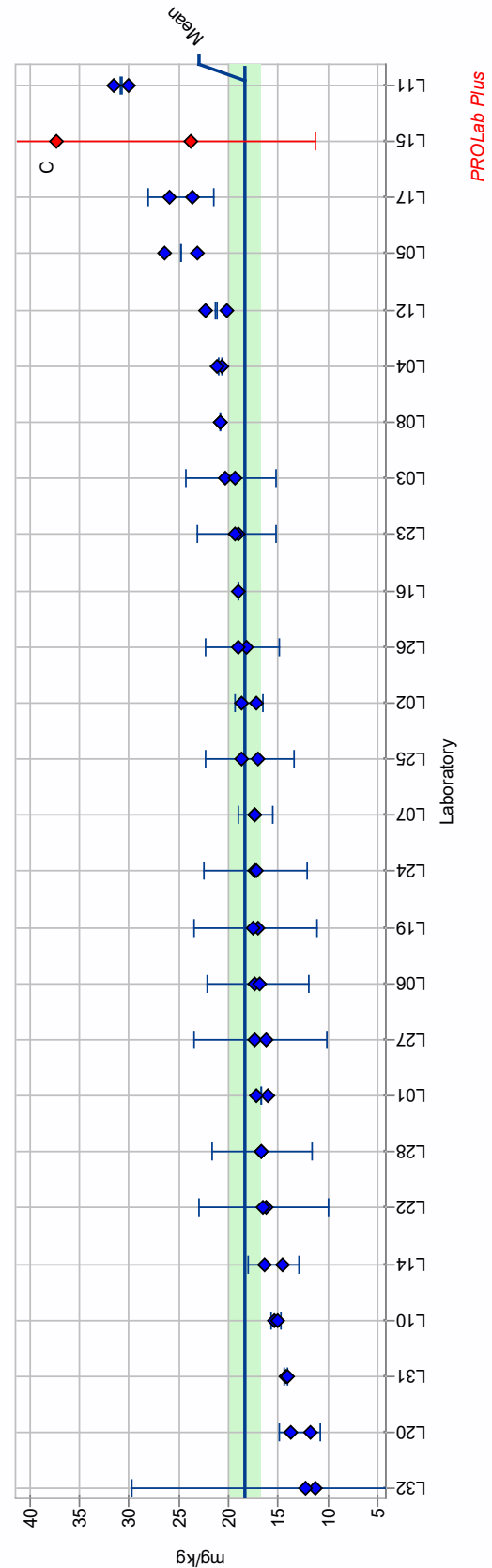


Figure 42. MOSH in IF03N.
Results in red are outliers:
C – Cochran (excessive standard deviation)

Table 43: Reported results for MOAH vs 2MN in IF03Q

Lab	M 1	M 2	MU [%]
L01	3.71	4.21	0
L02	5.39	5.55	2.9
L03	3.66	3.60	13
L04	4.69	4.67	0.02
L05	4.62	4.52	0
L06	3.87	3.27	30
L07	3.65	3.64	15
L08	3.44	3.68	
L10	3.99	4.15	4.1
L11	5.40	5.78	0.03
L12	2.73	2.65	0.33
L14	2.89	2.38	27.3
L15	2.21	3.99	81
L16	3.75	4.25	0.3
L17	4.58	5.11	15.4
L19	4.27	4.40	36
L20	2.86	3.14	9.5
L22	3.54	3.47	40
L23	3.78	3.42	26.2
L24	3.22	3.53	30
L25	4.25	4.15	25
L26	4.21	3.95	20
L27	3.87	3.96	43
L28	3.77	3.46	30
L31	4.31	4.85	16.6
L32	4.49	4.75	36.7

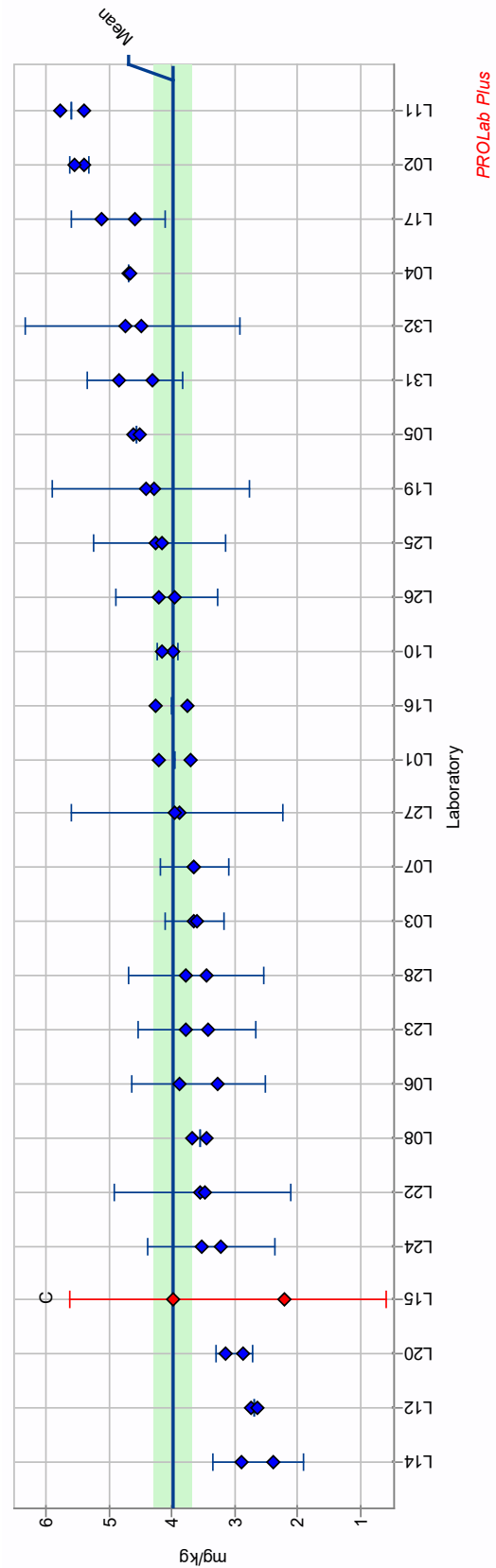


Figure 43. MOAH2MN in IF03Q.
 Results in red are outliers:
 C – Cochran (excessive standard deviation)

Table 44: Reported results for MOAH vs TBB in IF03Q

Lab	M 1	M 2	MU [%]
L01	3.03	3.16	0
L02	5.96	5.92	0.7
L03	4.11	4.02	13
L04	4.13	4.12	0.016
L05	4.22	4.13	0
L06	3.13	2.91	0
L07	3.37	3.43	15
L08	3.17	3.35	
L10	3.69	4.01	8.2
L11	4.73	4.74	0.001
L12	1.96	2.02	0.33
L14	2.84	2.45	20.7
L15	2.83	3.25	20
L16	3.53	3.76	0.3
L17	3.61	3.62	0.39
L19	3.80	3.89	36
L20	2.53	2.81	10.2
L22	3.21	3.20	40
L23	3.22	3.01	26.8
L24	2.96	3.15	30
L25	3.69	3.73	25
L26	3.21	3.28	20
L27	3.00	3.24	43
L28	3.34	3.25	30
L31	4.37	4.57	6.3
L32	3.82	4.02	28.2

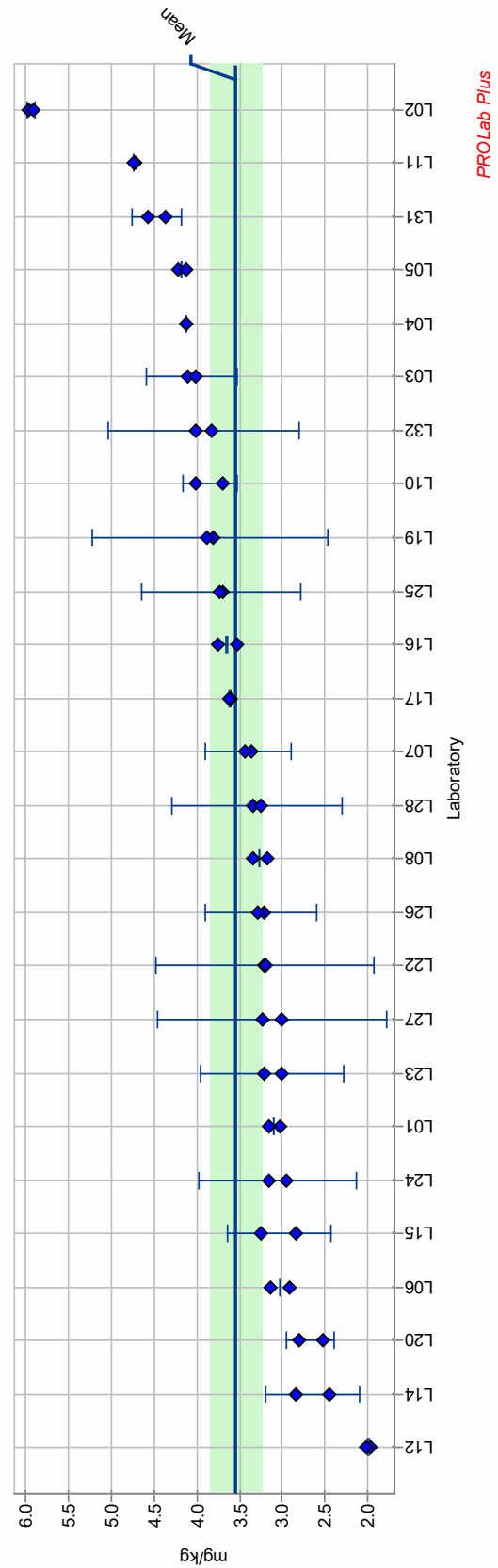


Figure 44. MOAHTBB in IF03Q.

Table 45: Reported results for MOSH vs CyCY in IF03

Lab	M 1	M 2	MU [%]
1	8.5	9.2	0
02	9.8	11.7	17.9
L03	12.1	11.3	23
L04	12.1	11.5	2.3
L05	14.9	14.1	0
L06	9.8	8.5	30
L07	10.4	10.4	10
L08	11.9	11.5	
L10	11.7	12.2	4.8
L11	12.6	12.3	0.01
L12	14.3	9.9	0.17
L14	9.2	8.7	7.4
L15	9.3	10.5	17
L16	10.0		0.3
L17	16.4	15.6	7.1
L19	10.4	10.5	36
L20	9.2	9.6	4.3
L22	8.8	9.0	40
L23	11.0	10.2	22.3
L24	9.0	9.7	30
L25	16.2	12.8	25
L26	10.2	9.6	20
L27	12.0	11.6	40
L28	9.3	9.7	30
L31	9.3	10.4	15.3
L32	6.5	6.8	53.7

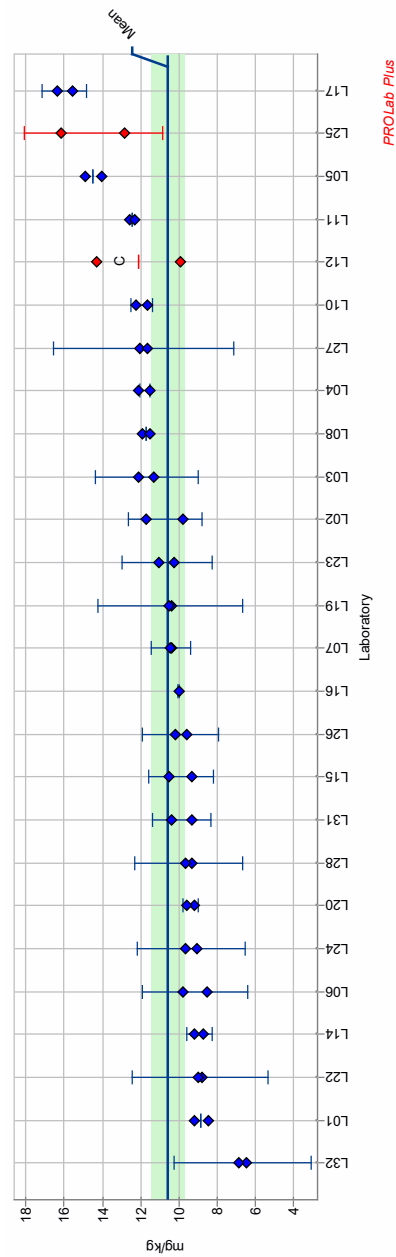
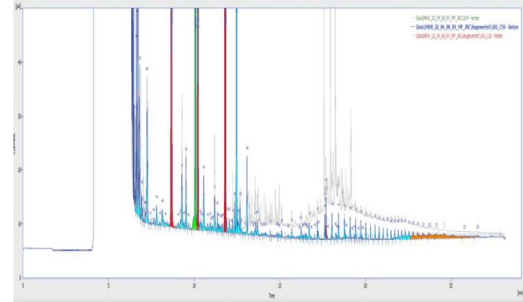


Figure 45. MOSH in IF03Q.
 Results in red are outliers:
 C – Cochran (excessive standard deviation)

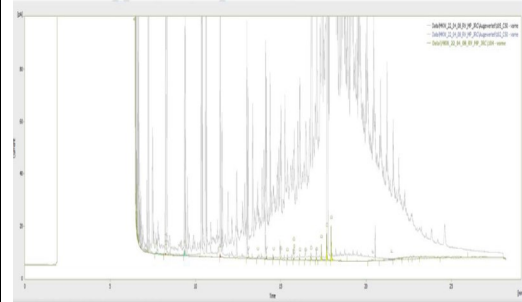
Annex 6. Chromatograms compilation

Lab 01

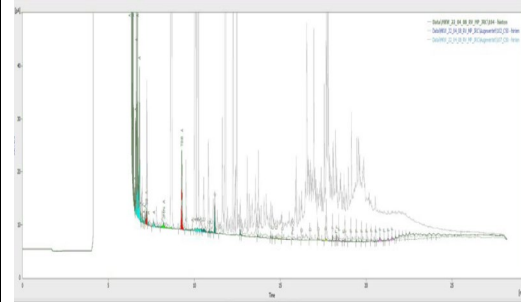
IF03A MOAH



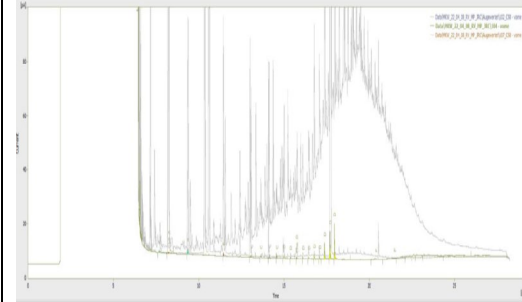
IF03A MOSH



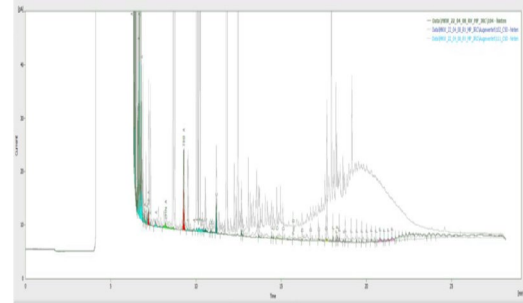
IF03B MOAH



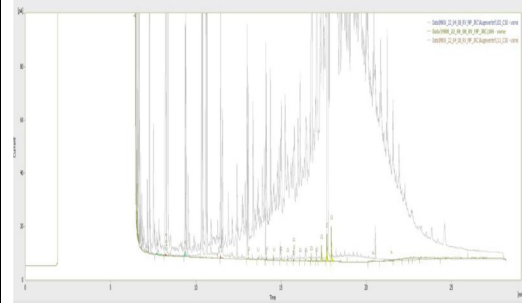
IF03B MOSH



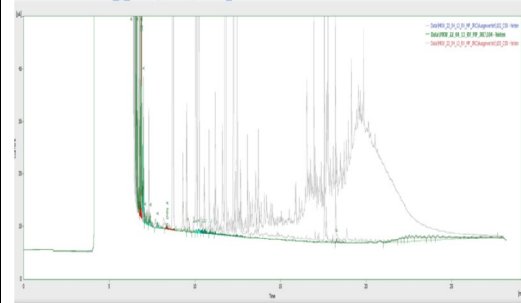
IF03D MOAH



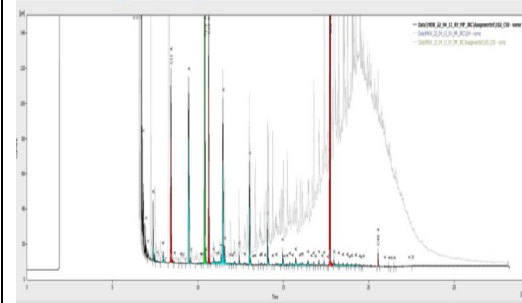
IF03D MOSH



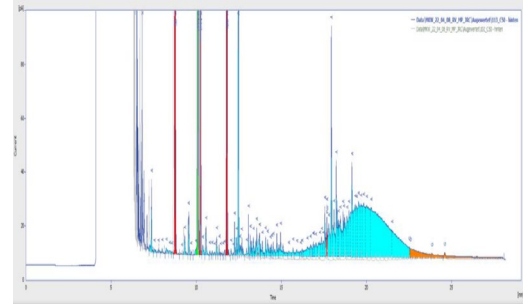
IF03H MOAH



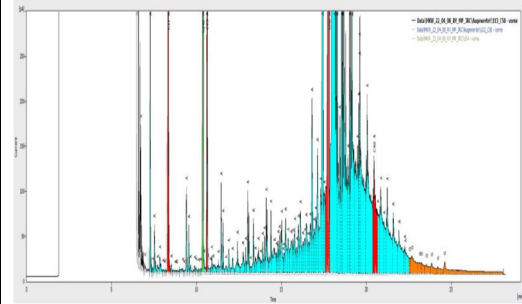
IF03H MOSH



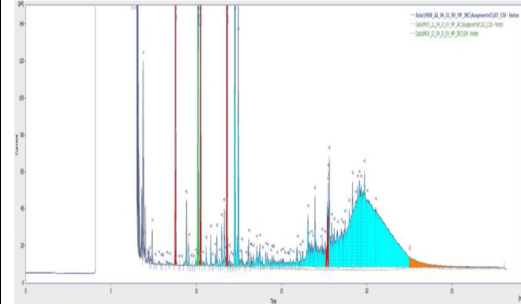
IF03E MOAH



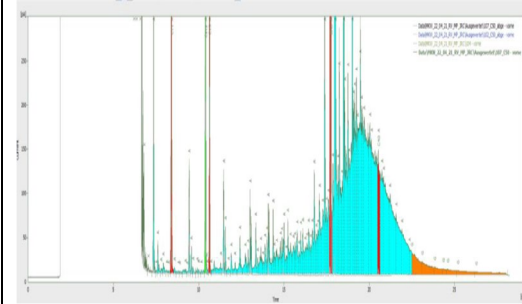
IF03E MOSH



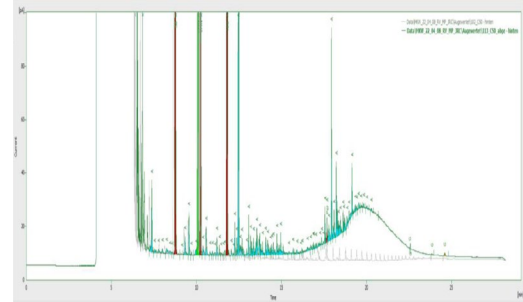
IF03I MOAH



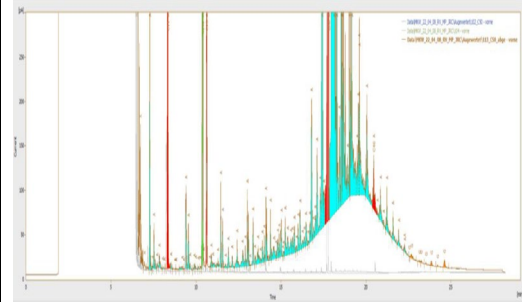
IF03I MOSH



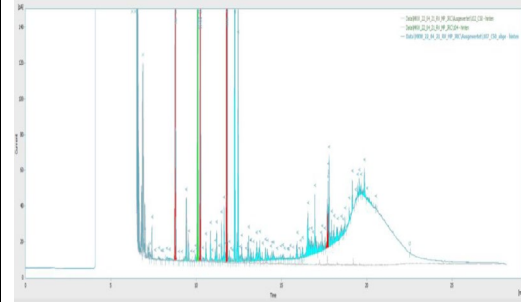
IF03E MOAH trimmed



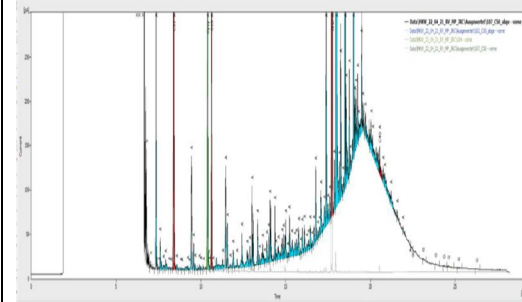
IF03E MOSH trimmed



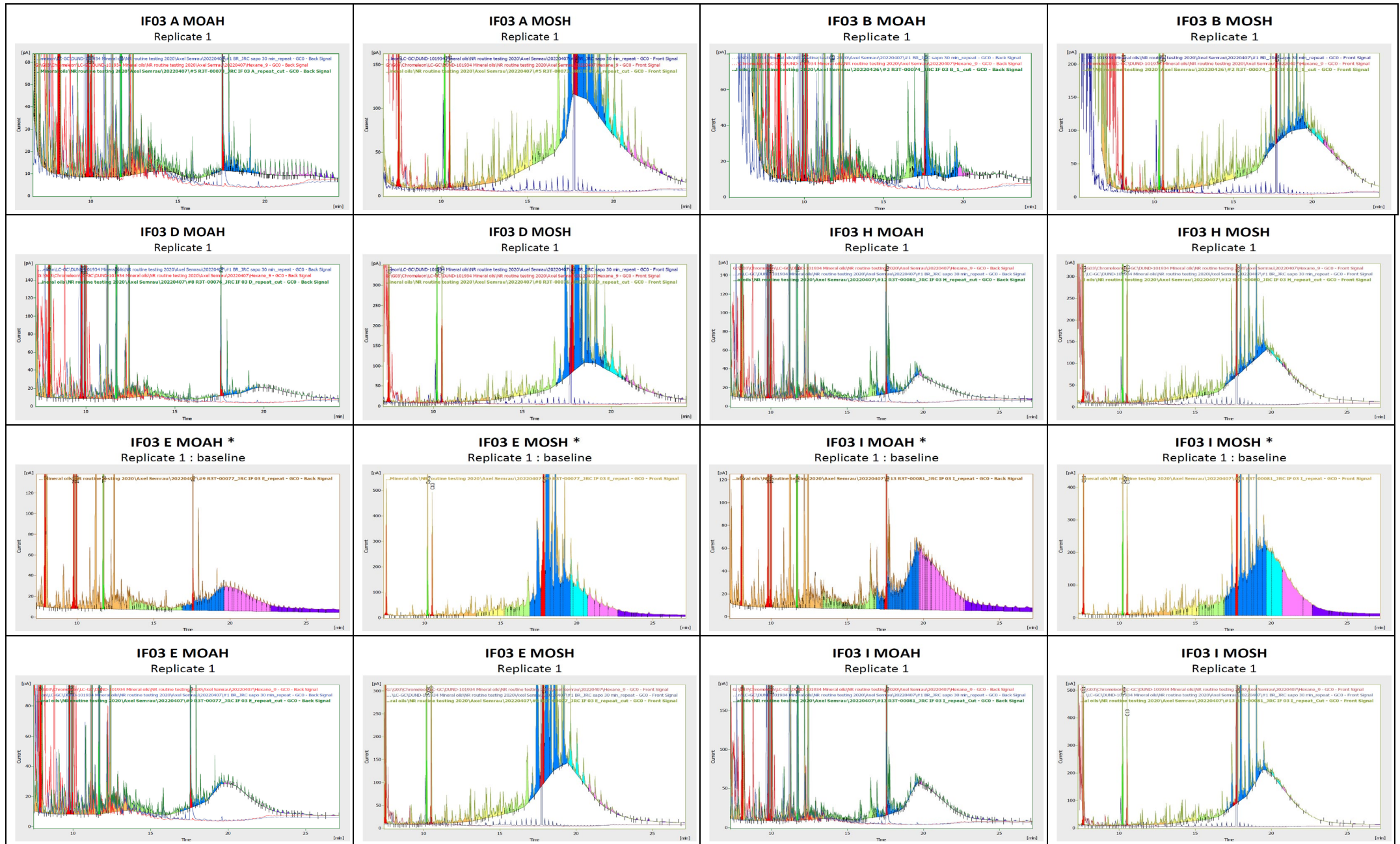
IF03I MOAH trimmed



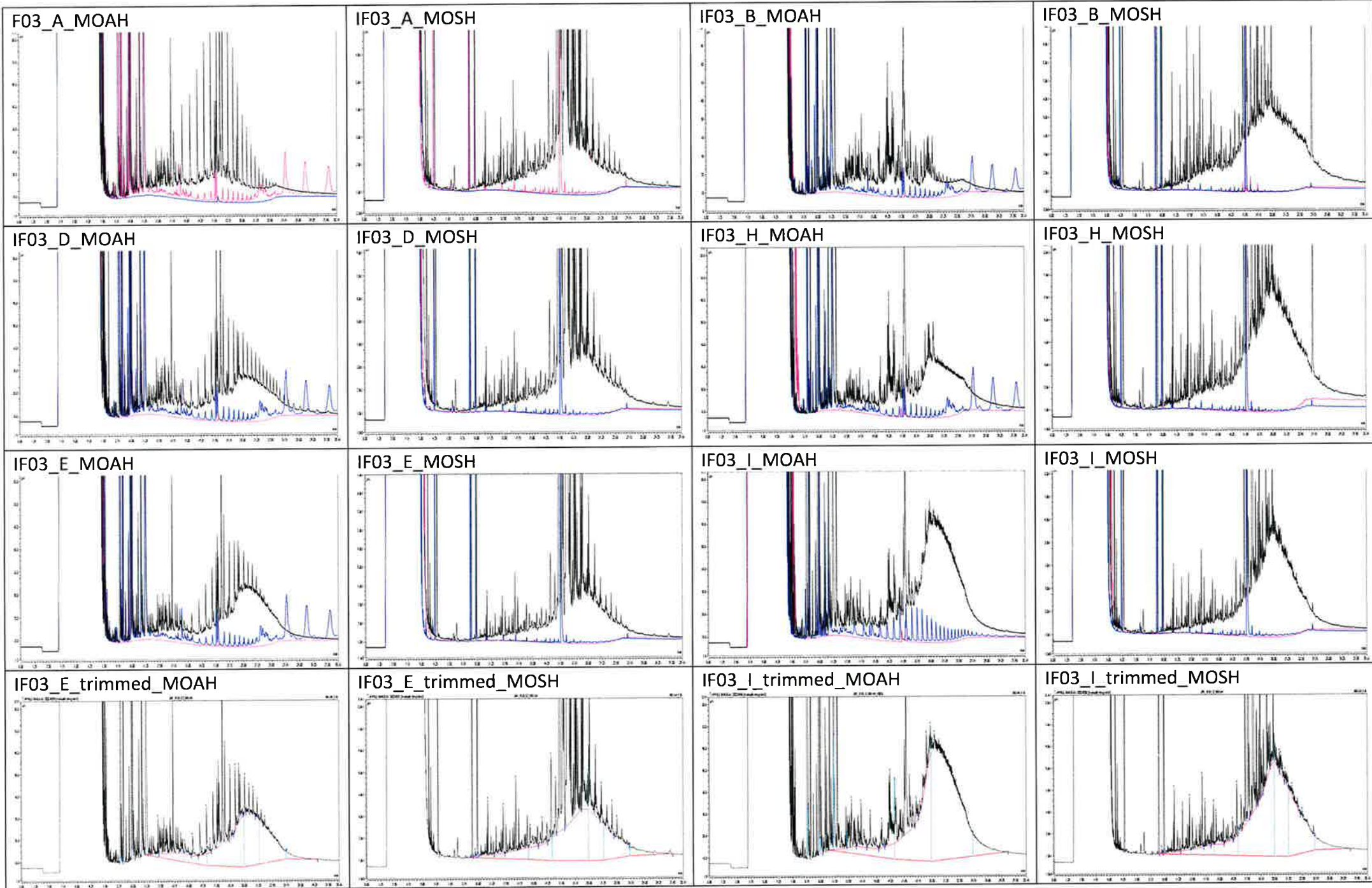
IF03I MOSH trimmed



Lab 03



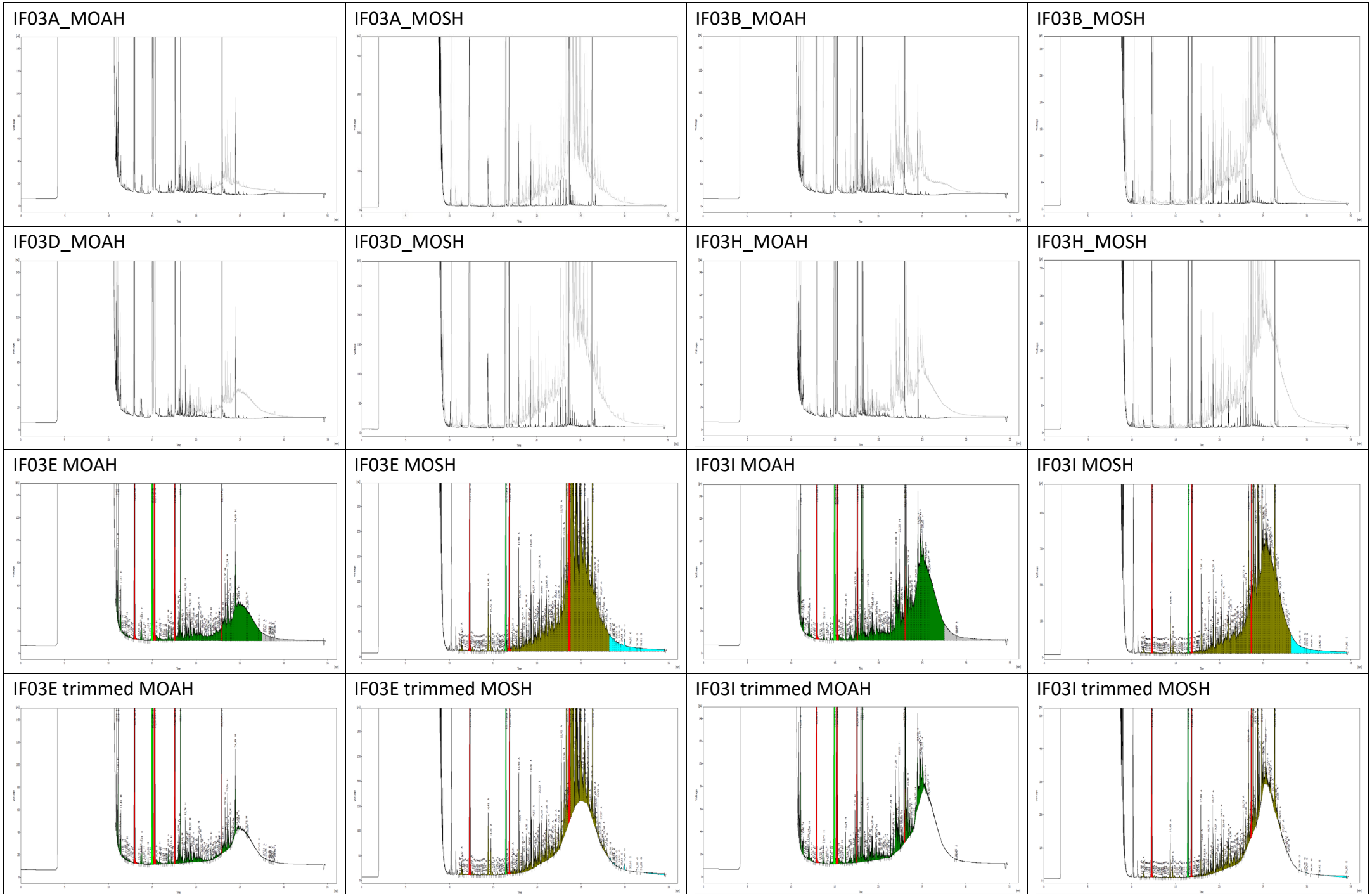
Lab 04



Lab 05

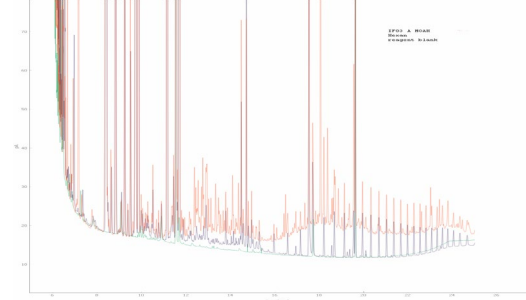
F03_A_MOAH	IF03_A_MOSH	IF03_B_MOAH	IF03_B_MOSH
IF03_D_MOAH	IF03_D_MOSH	IF03_H_MOAH	IF03_H_MOSH
<p>IF03_E_MOAH</p>	IF03_E_MOSH	<p>IF03_I_MOAH</p>	IF03_I_MOSH
<p>IF03_E_trimmed_MOAH</p>	IF03_E_trimmed_MOSH	<p>IF03_I_trimmed_MOAH</p>	IF03_I_trimmed_MOSH

Lab 06



Lab 07

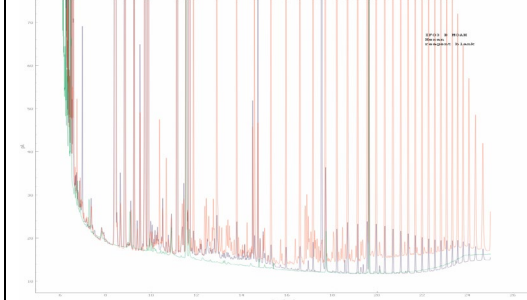
IF03_A_MOAH



IF03_A_MOSH



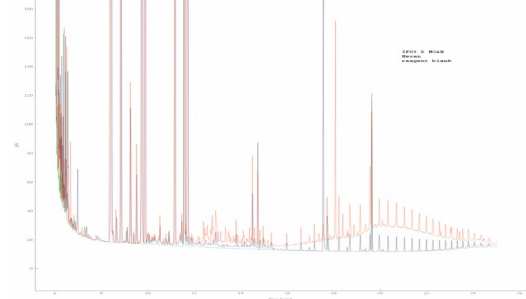
IF03_B_MOAH



IF03_B_MOSH



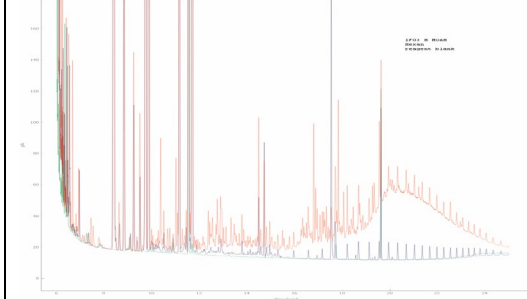
IF03_D_MOAH



IF03_D_MOSH



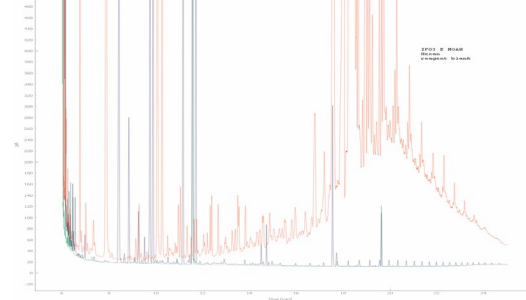
IF03_H_MOAH



IF03_H_MOSH



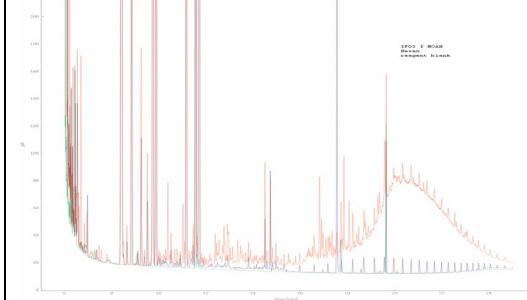
IF03_E_MOAH



IF03_E_MOSH



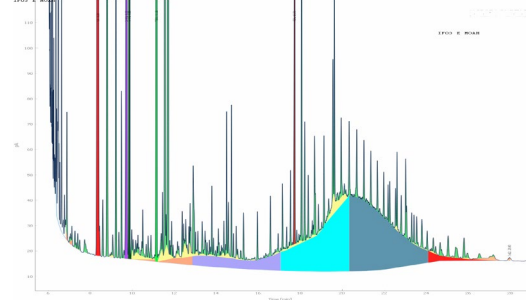
IF03_I_MOAH



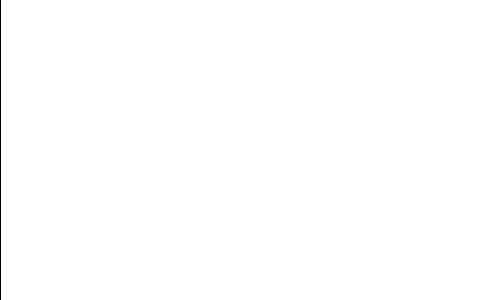
IF03_I_MOSH



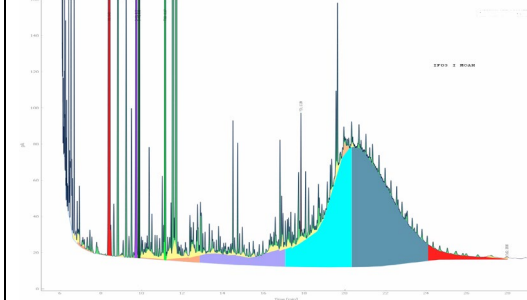
IF03_E_trimmed_MOAH



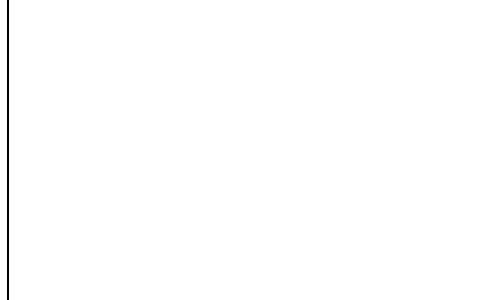
IF03_E_trimmed_MOSH



IF03_I_trimmed_MOAH

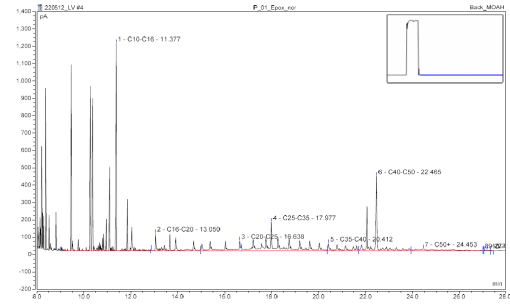


IF03_I_trimmed_MOSH

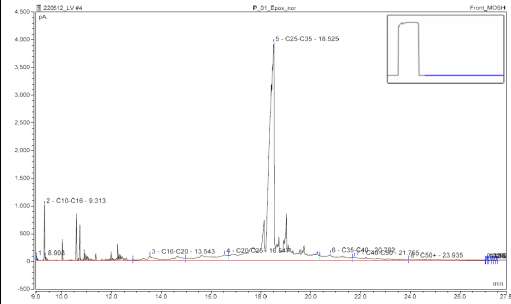


Lab 10

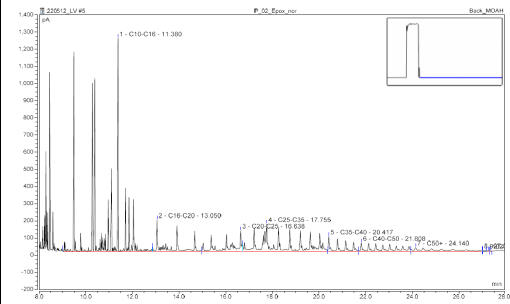
IF03_A_MOAH



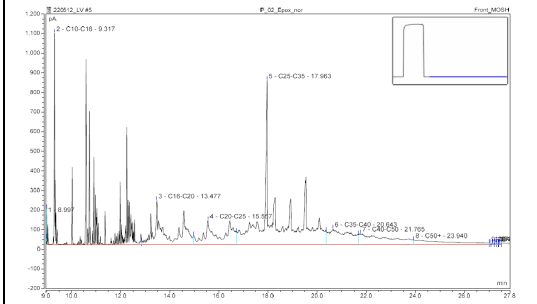
IF03_A_MOSH



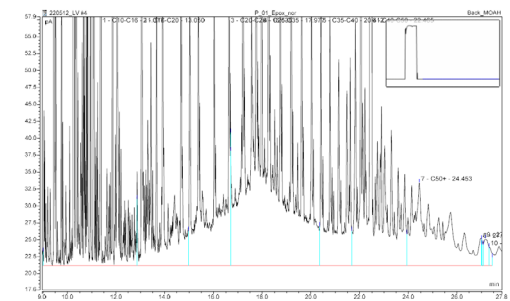
IF03_B_MOAH



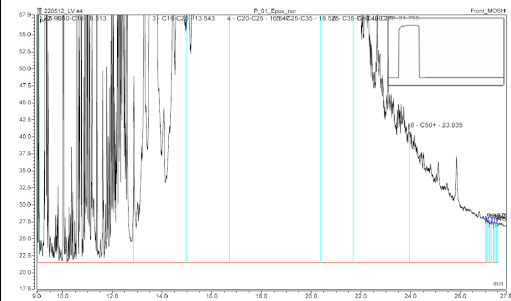
IF03_B_MOSH



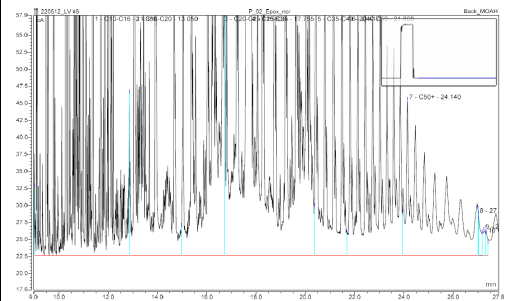
IF03_A_MOAH zoom



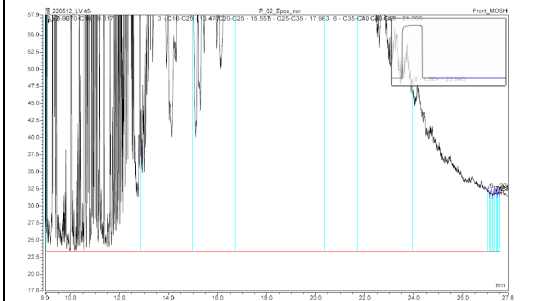
IF03_A_MOSH zoom



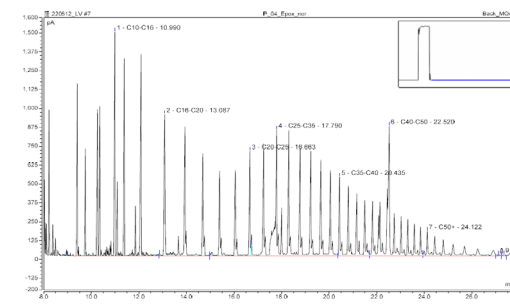
IF03_B_MOAH zoom



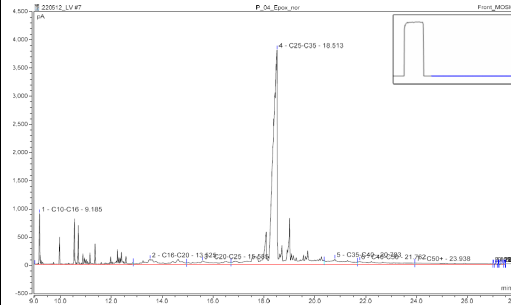
IF03_B_MOSH zoom



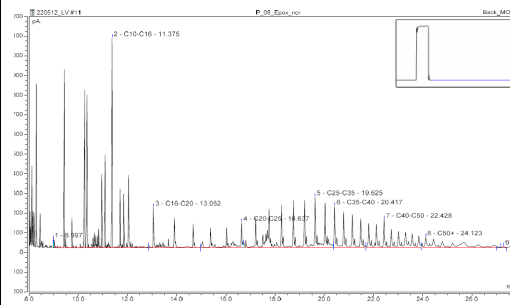
IF03_D_MOAH



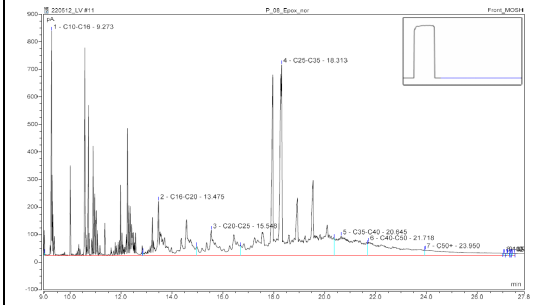
IF03_D_MOSH



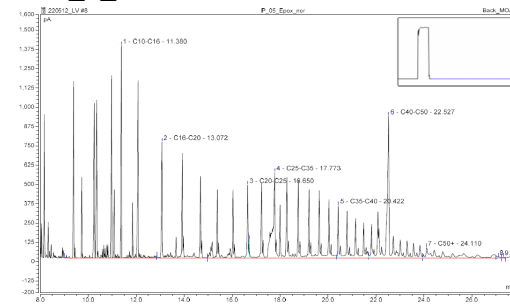
IF03_H_MOAH



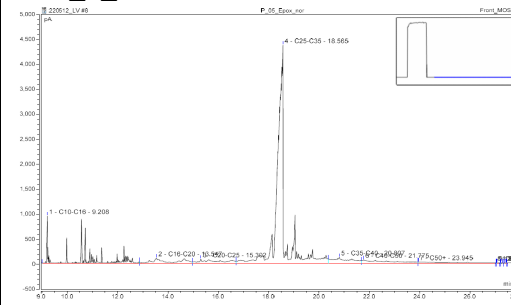
IF03_H_MOSH



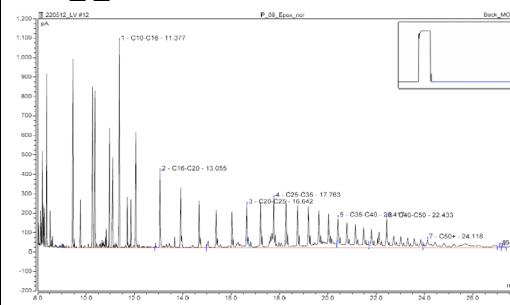
IF03_E_MOAH



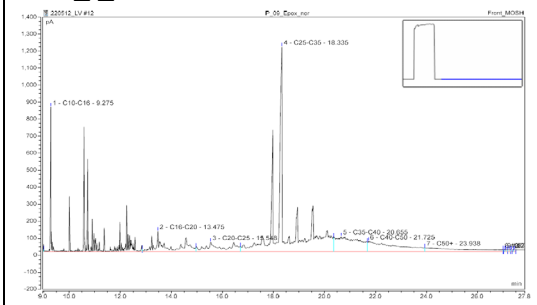
IF03_E_MOSH



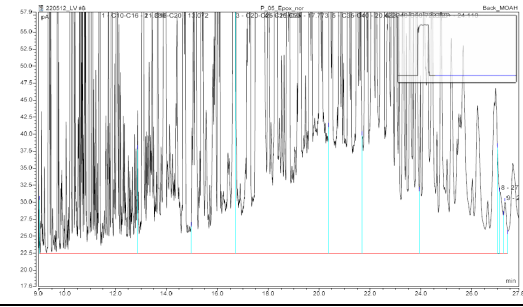
IF03_I_MOAH



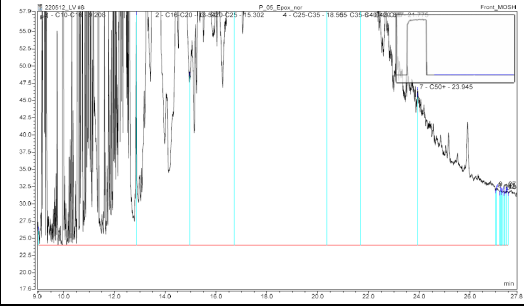
IF03_I_MOSH



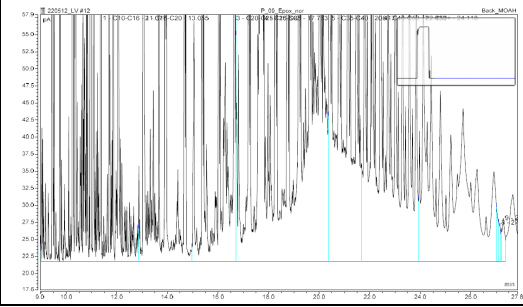
IF03_E_MOAH zoom



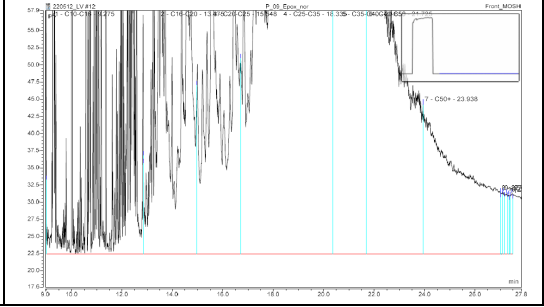
IF03_E_MOSH zoom



IF03_I_MOAH zoom

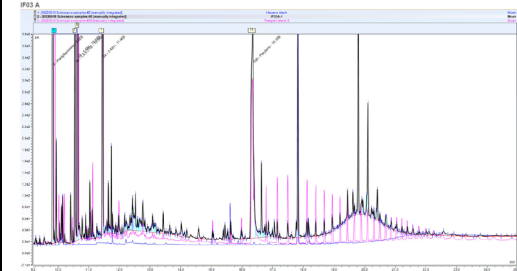


IF03_I_MOSH zoom

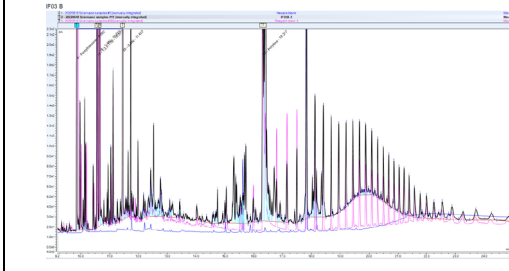


Lab 11

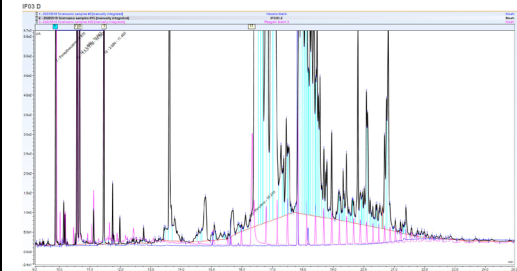
IF03A-1 MOAH



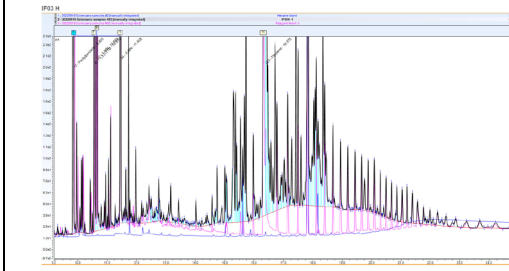
IF03B-2 MOAH



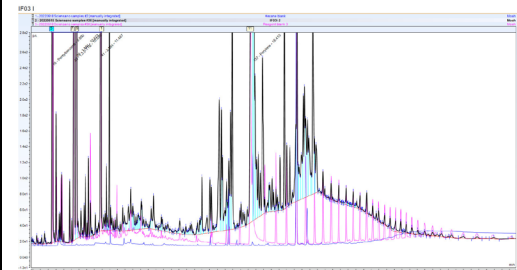
IF03D-2 MOAH



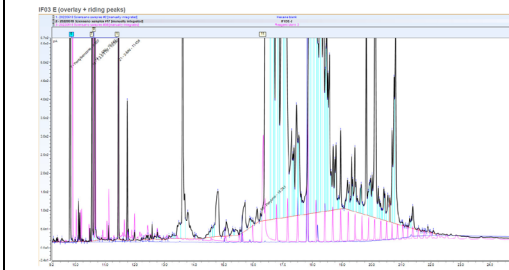
IF03H-1 MOAH



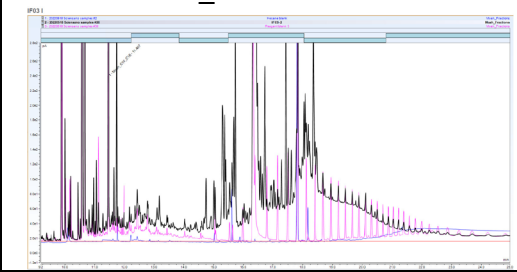
IF03I-2 MOAH



IF03E-2 (overlay+riding peaks) MOAH

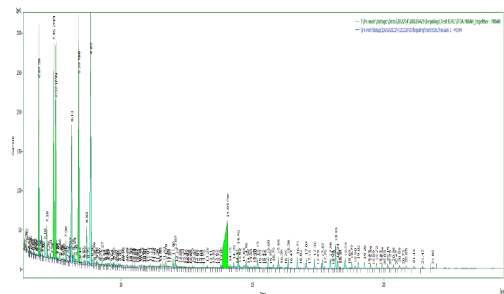


IF03I-2 MOAH_Fractions

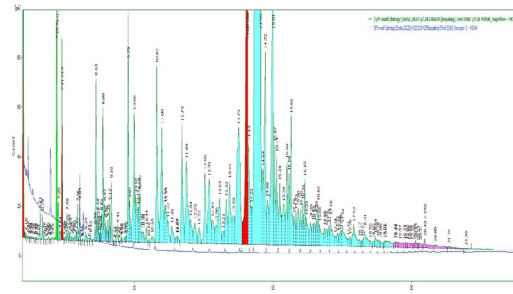


Lab 12

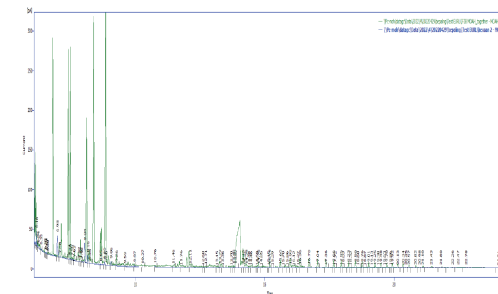
IF03_A_MOAH



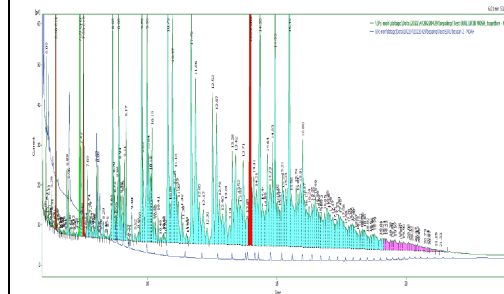
IF03_A_MOSH



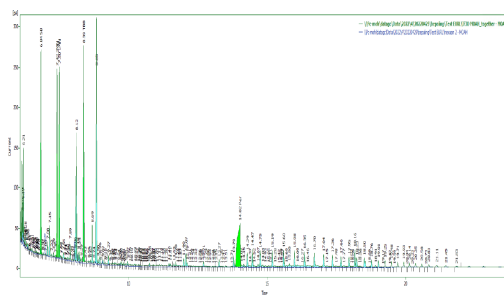
IF03_B_MOAH



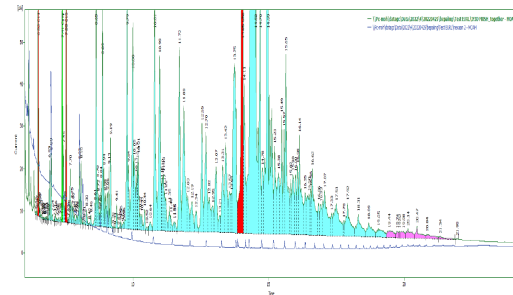
IF03_B_MOSH



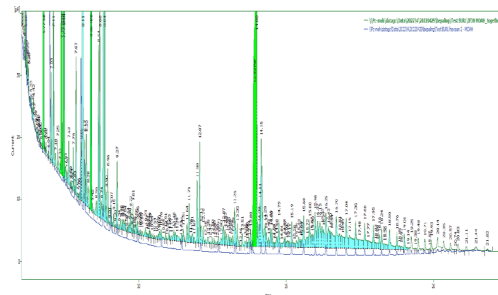
IF03_D_MOAH



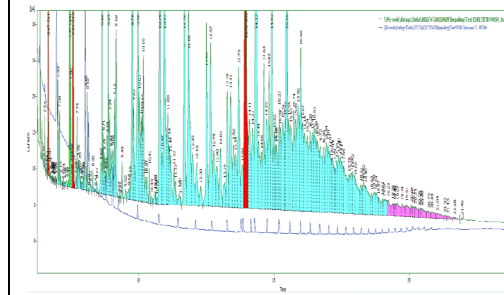
IF03_D_MOSH



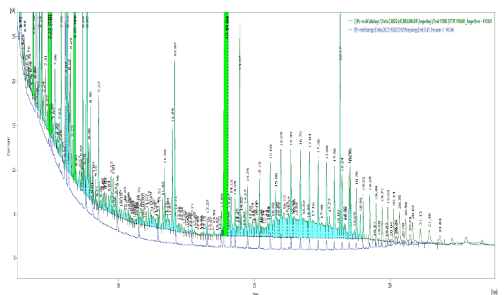
IF03_H_MOAH



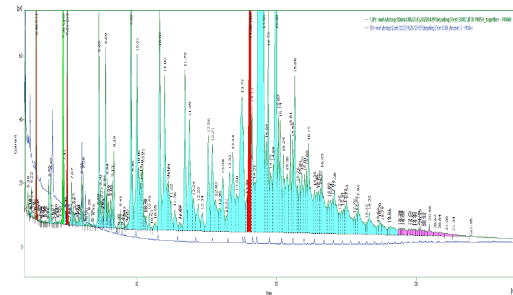
IF03_H_MOSH



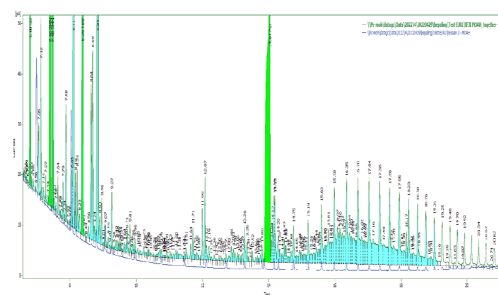
IF03_E_MOAH



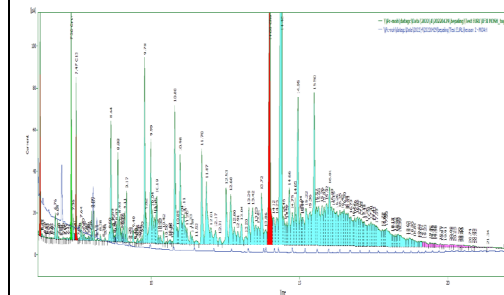
IF03_E_MOSH



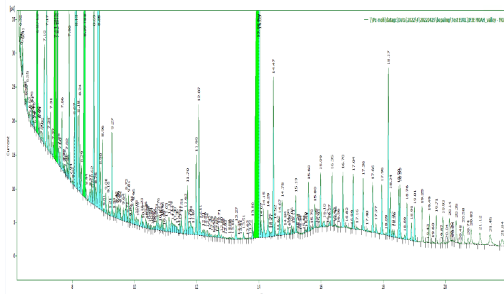
IF03_I_MOAH



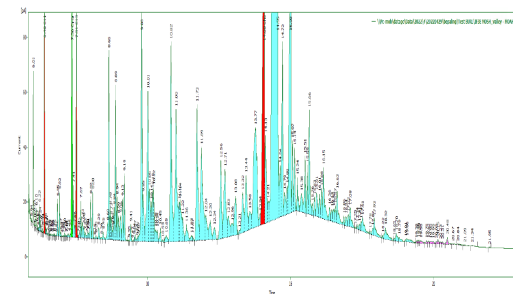
IF03_I_MOSH



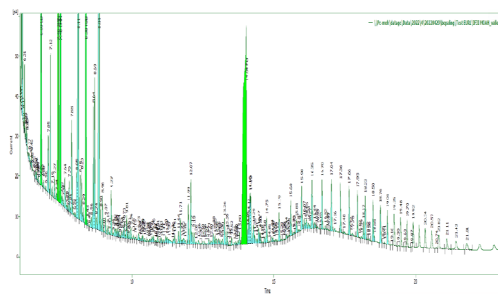
IF03_E_trimmed_MOAH



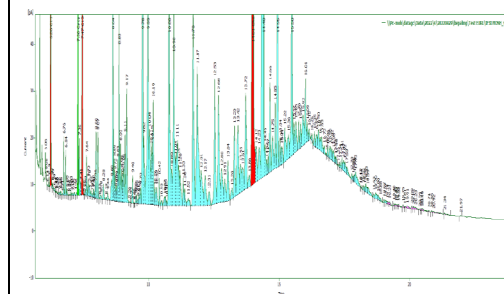
IF03_E_trimmed_MOSH



IF03_I_trimmed_MOAH

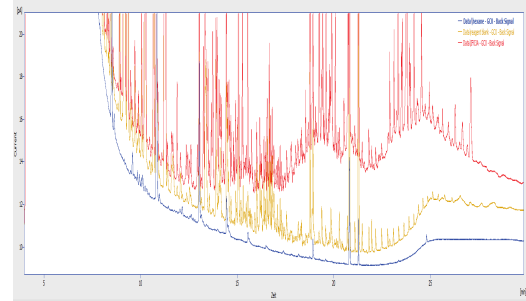


IF03_I_trimmed_MOSH

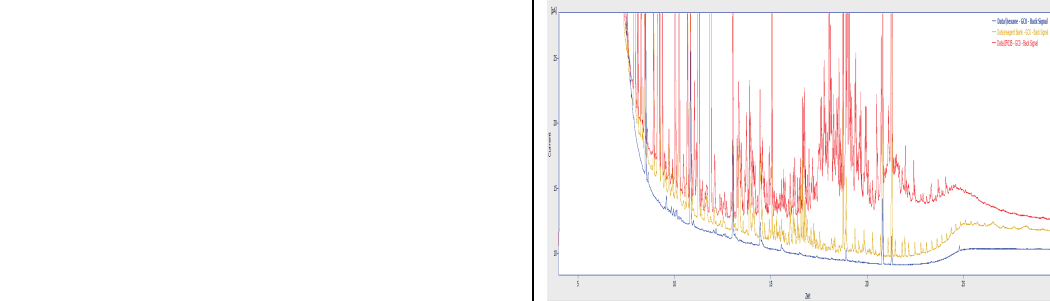


Lab 14

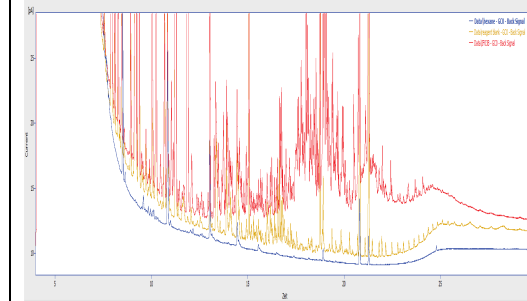
F03_A_MOAH



IF03_A_MOSH



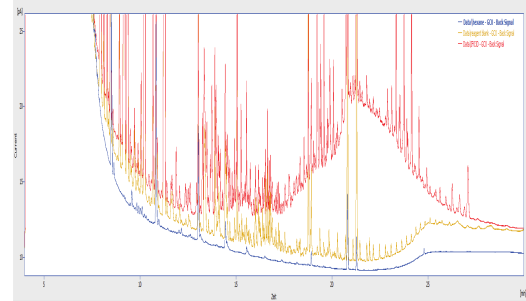
IF03_B_MOAH



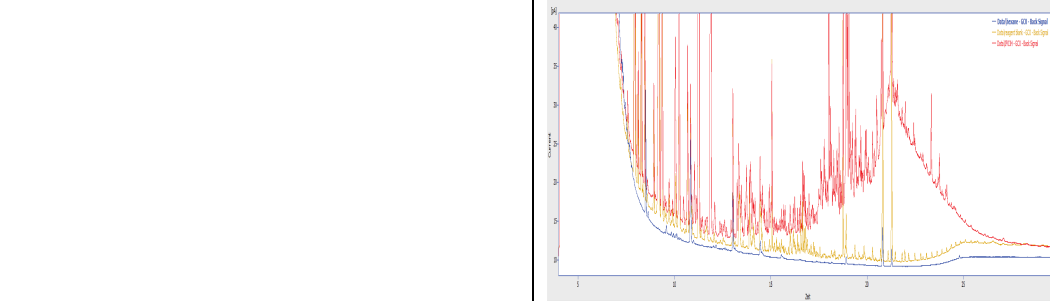
IF03_B_MOSH



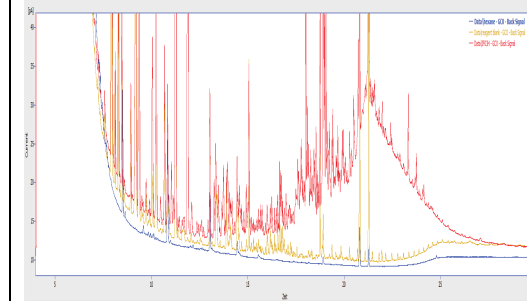
IF03_D_MOAH



IF03_D_MOSH



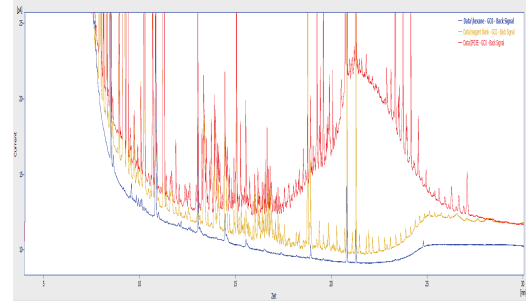
IF03_H_MOAH



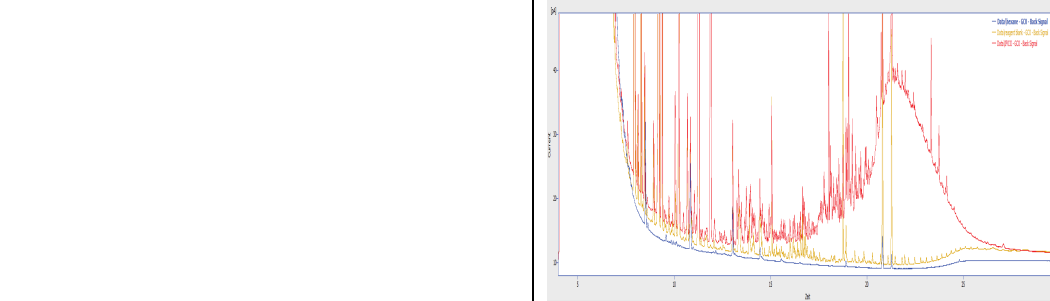
IF03_H_MOSH



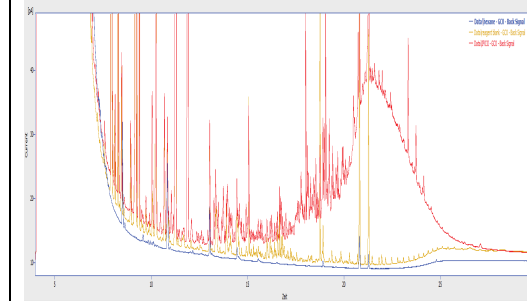
IF03_E_MOAH



IF03_E_MOSH



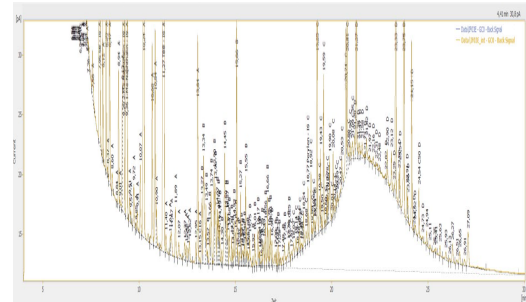
IF03_I_MOAH



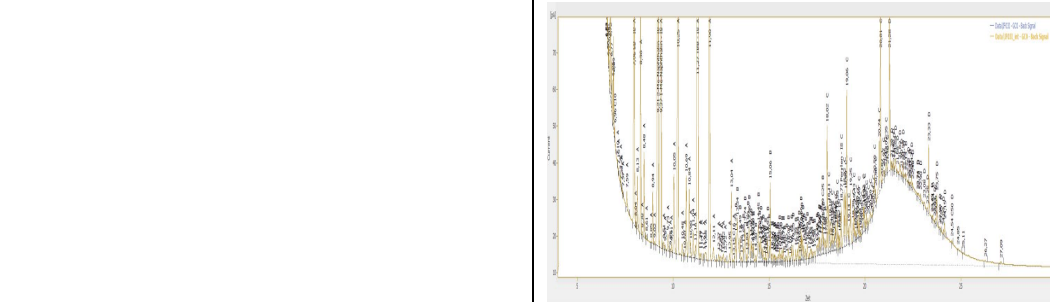
IF03_I_MOSH



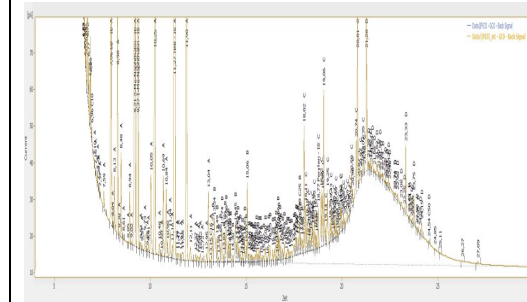
IF03_E_trimmed_MOAH



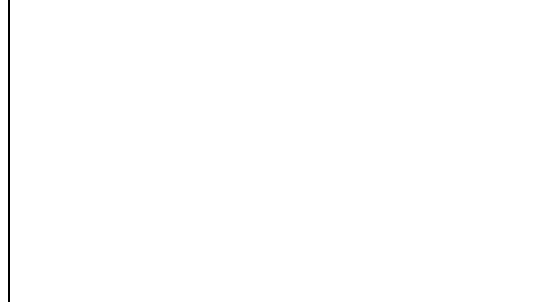
IF03_E_trimmed_MOSH



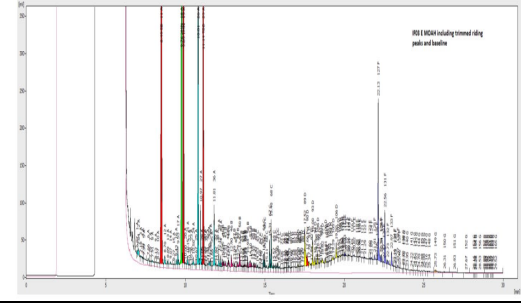
IF03_I_trimmed_MOAH



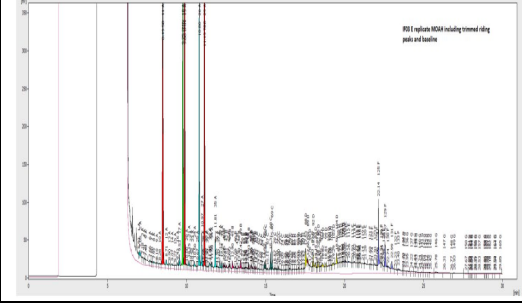
IF03_I_trimmed_MOSH



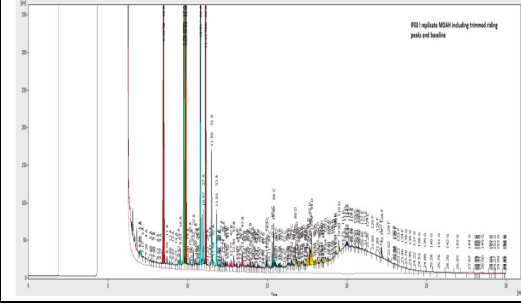
IF03_E_trimmed_MOAH



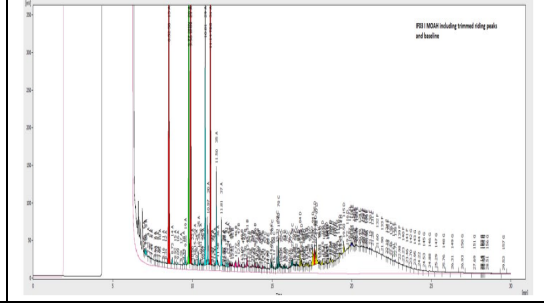
IF03_E_trimmed_MOAH



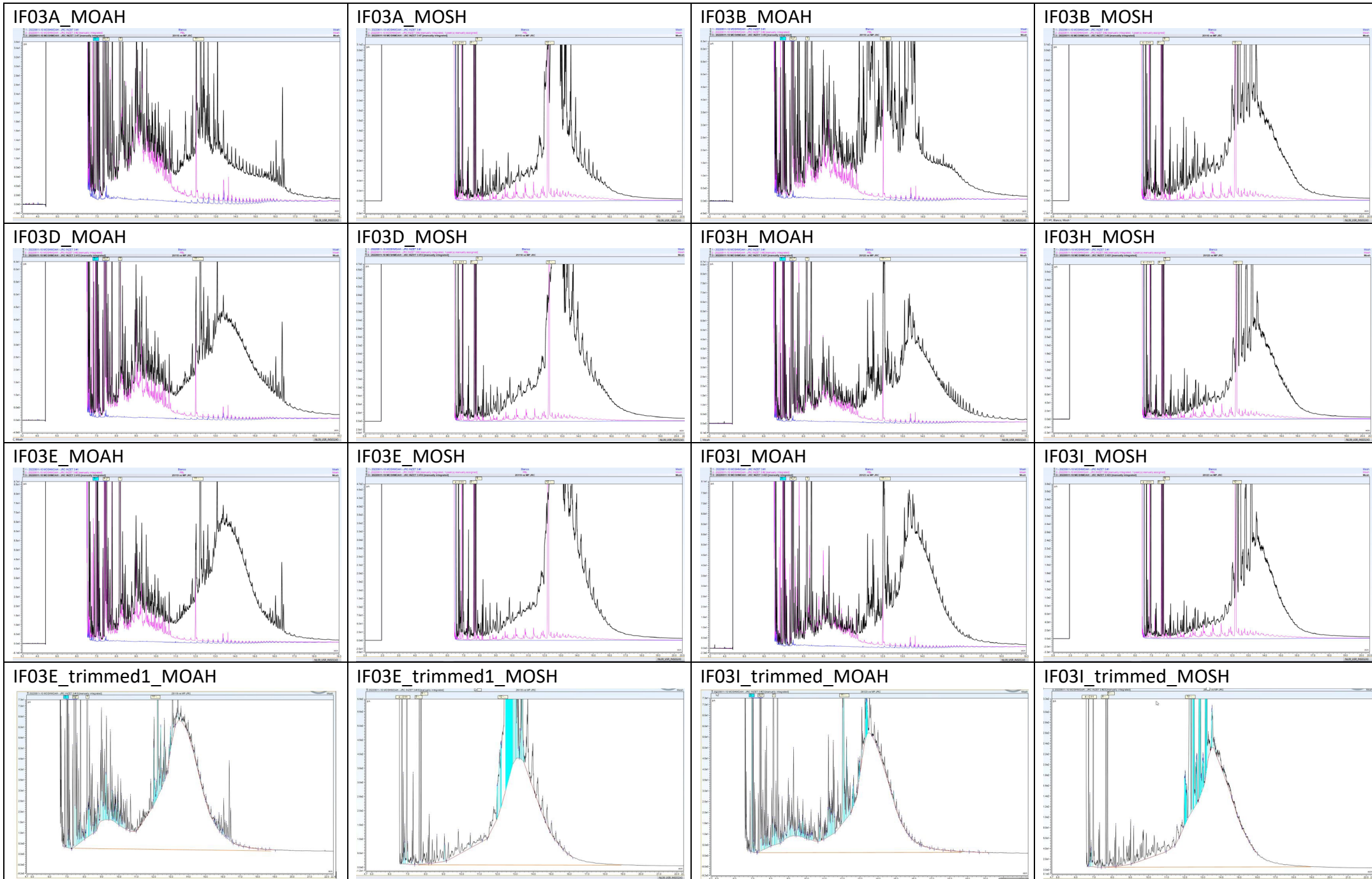
IF03_I_trimmed_MOAH



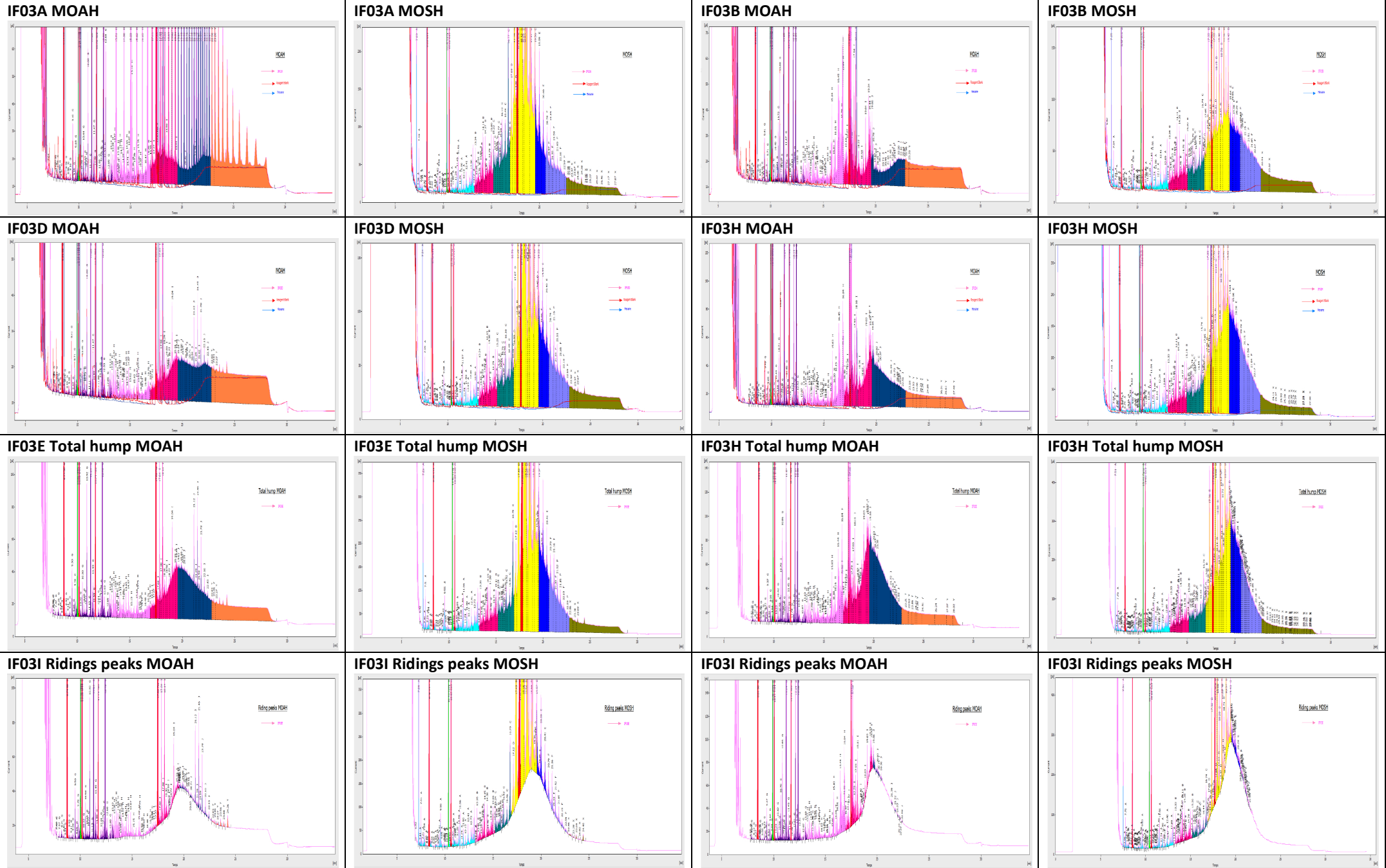
IF03_I_trimmed_MOAH



Lab 16

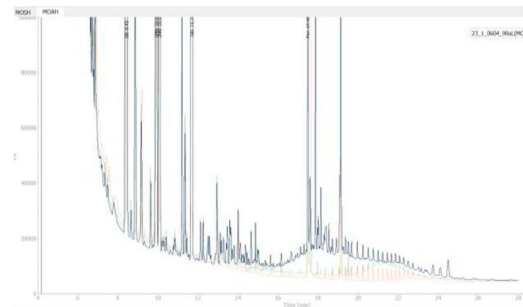


Lab 19

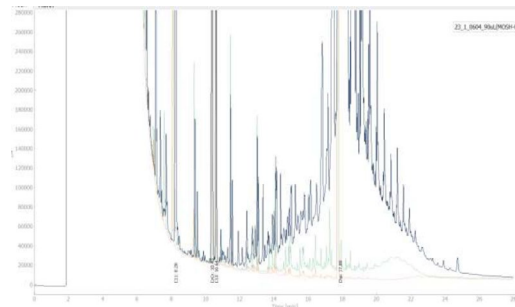


Lab 20

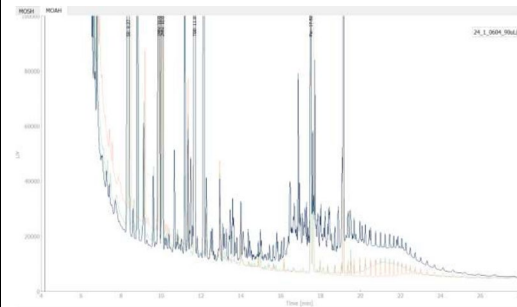
IF03A MOAH



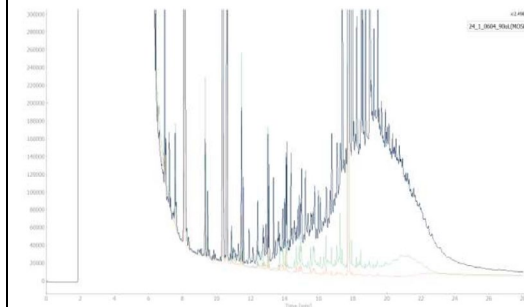
IF03A MOSH



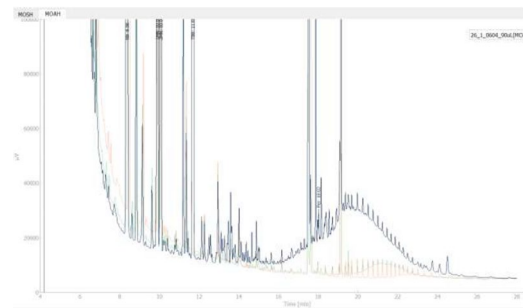
IF03B MOAH



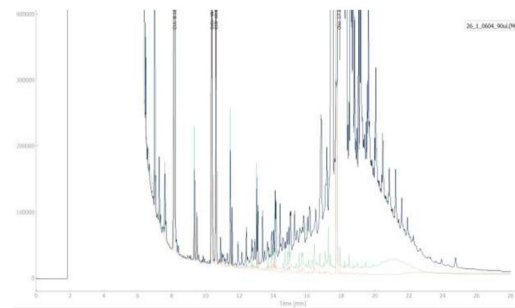
IF03B MOSH



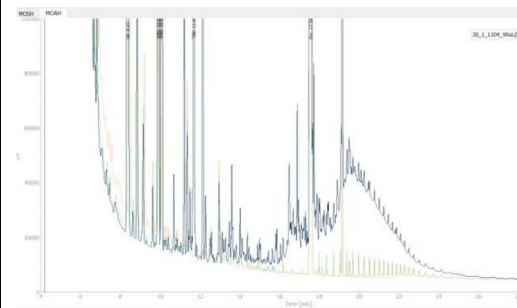
IF03D MOAH



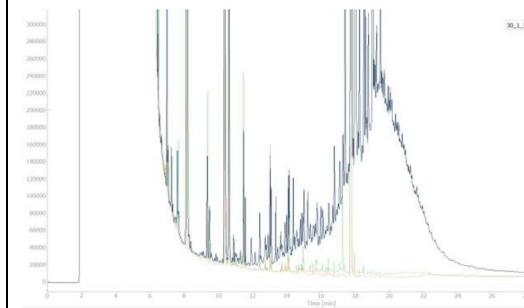
IF03D MOSH



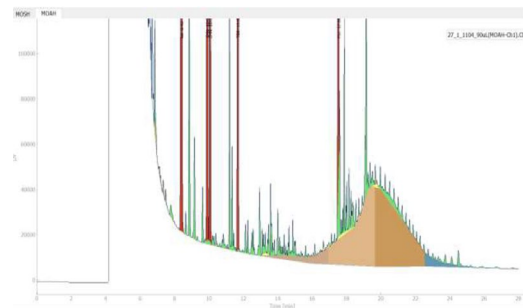
IF03H MOAH



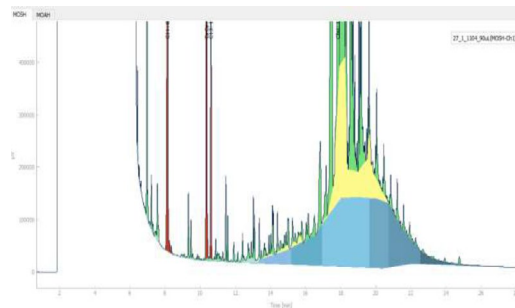
IF03H MOSH



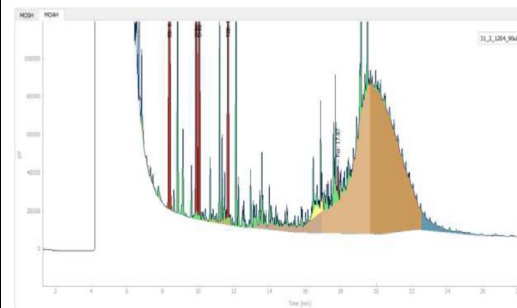
IF03E MOAH



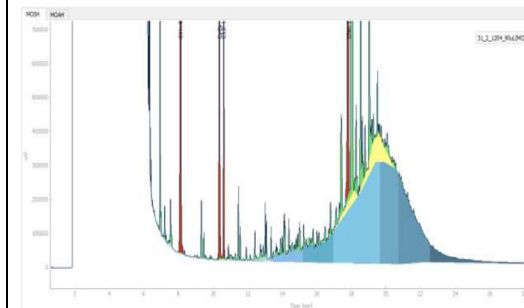
IF03E MOSH



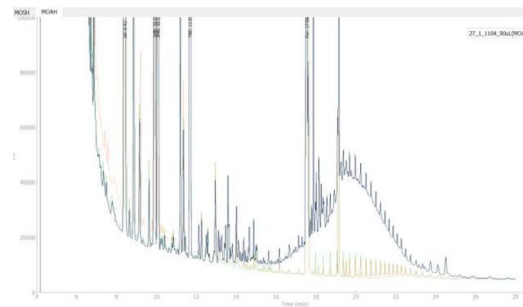
IF03I MOAH



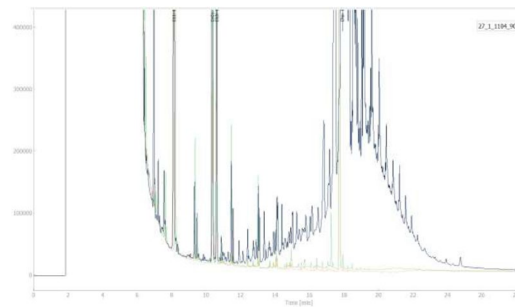
IF03I MOSH



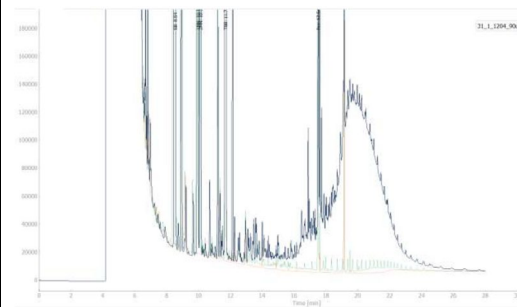
IF03E MOAH trimmed



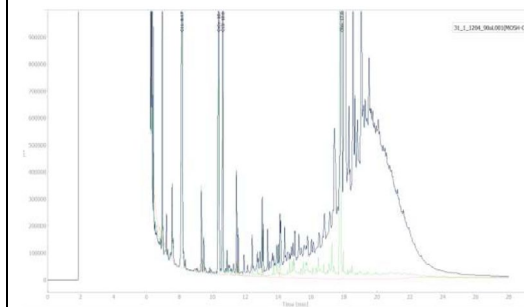
IF03E MOSH trimmed



IF03I MOAH trimmed

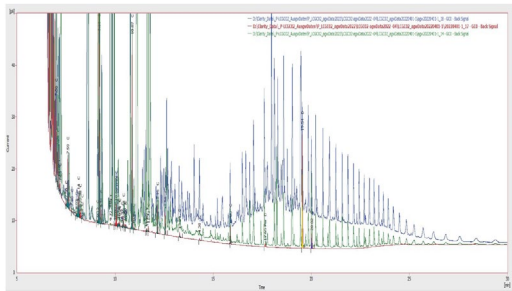


IF03I MOSH trimmed

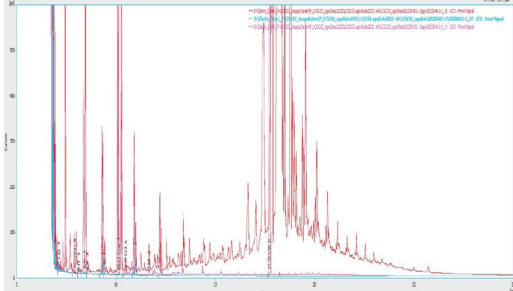


Lab 22

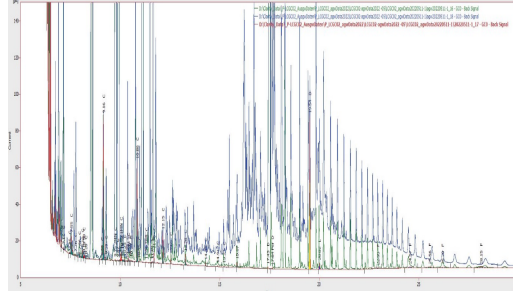
IF03A MOAH



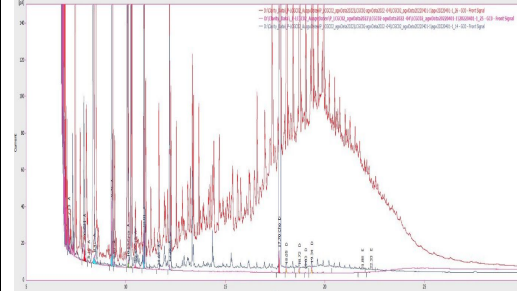
IF03A MOSH



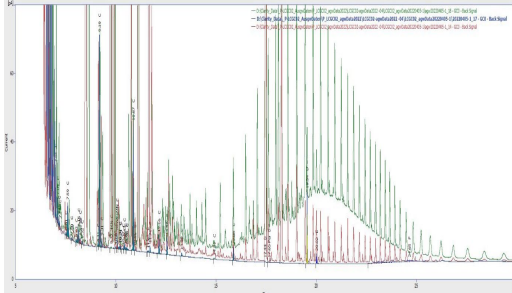
IF03B MOAH



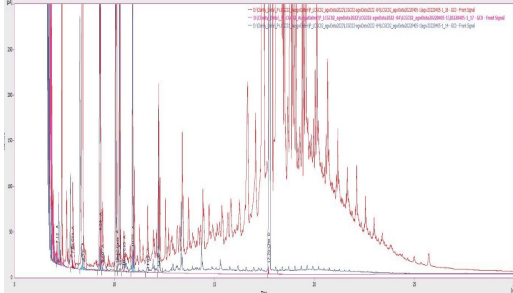
IF03B MOSH



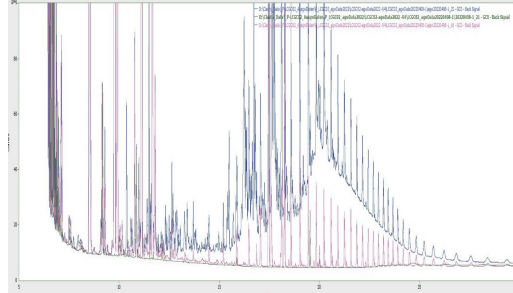
IF03D MOAH



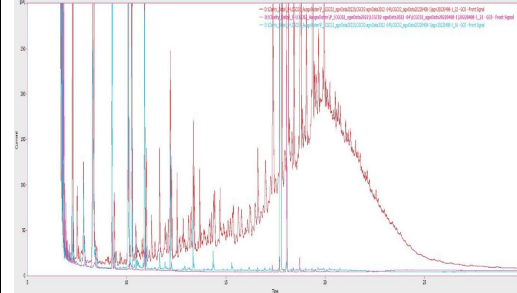
IF03D MOSH



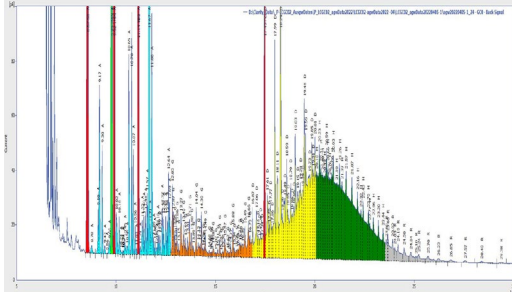
IF03H MOAH



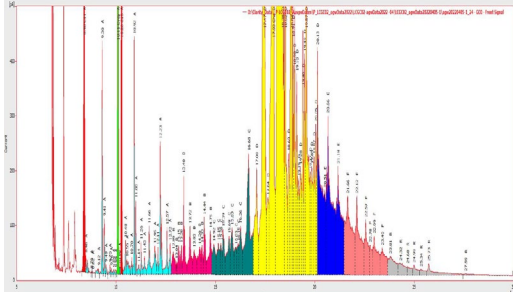
IF03H MOSH



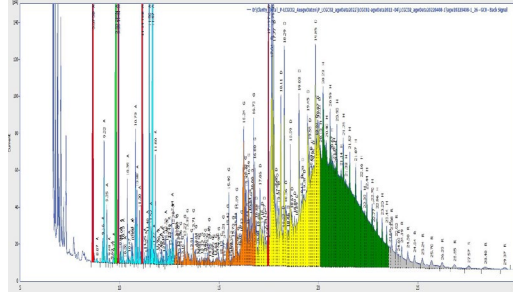
IF03E MOAH



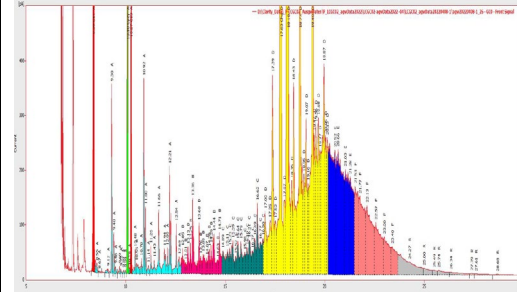
IF03E MOSH



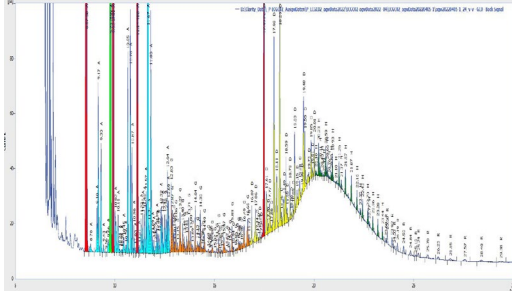
IF03I MOAH



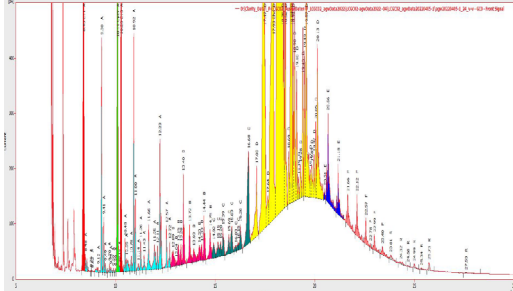
IF03I MOSH



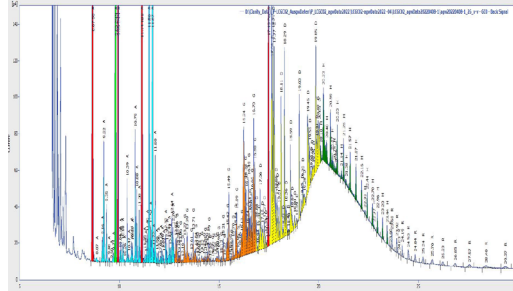
IF03E MOAH trimmed



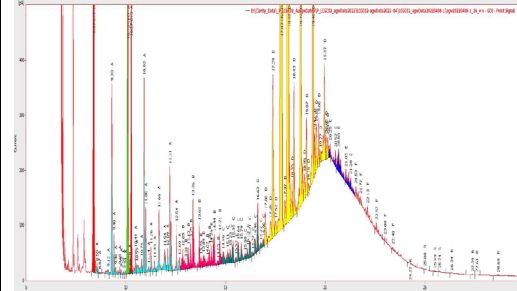
IF03E MOSH trimmed



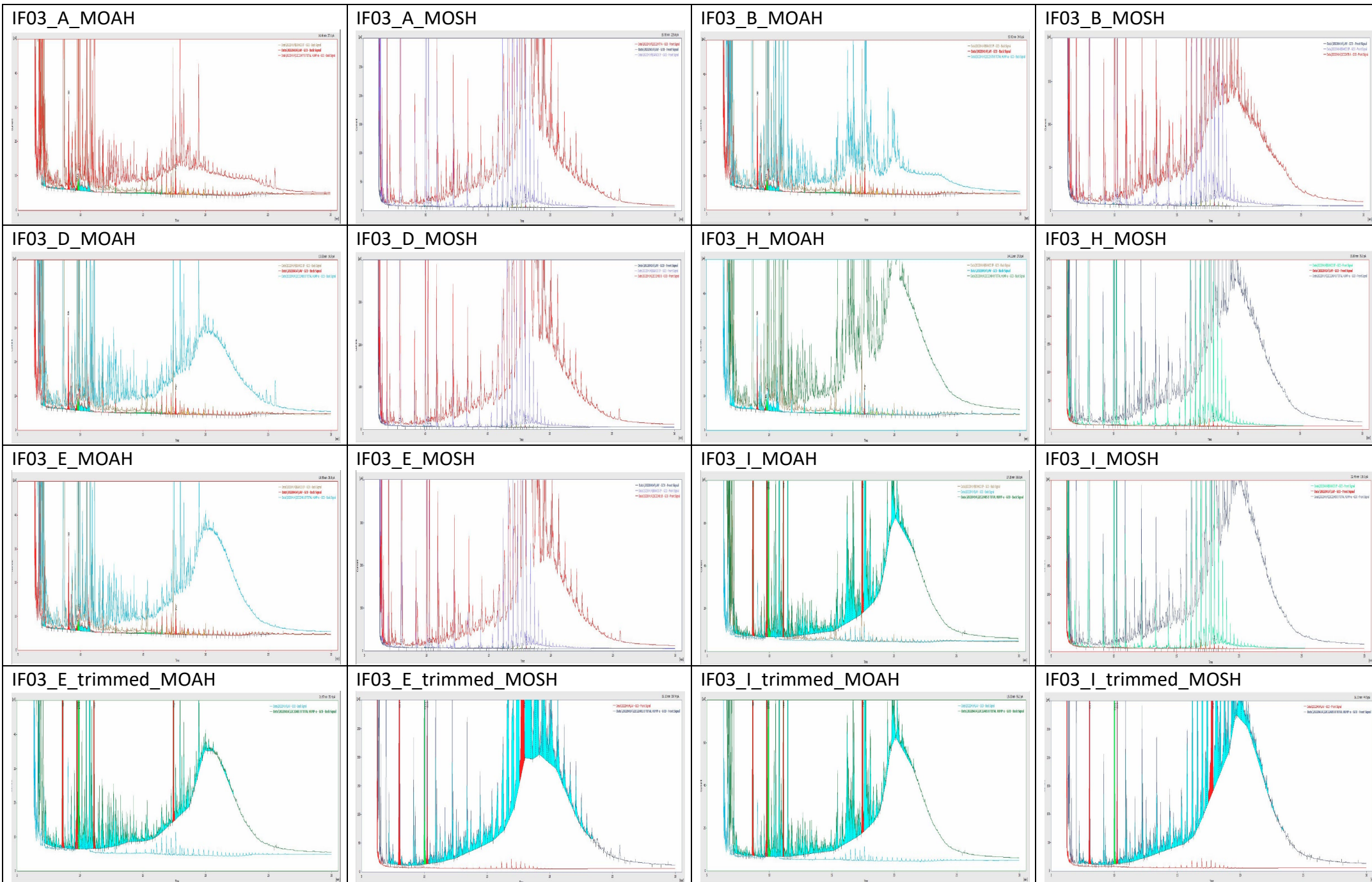
IF03I MOAH trimmed



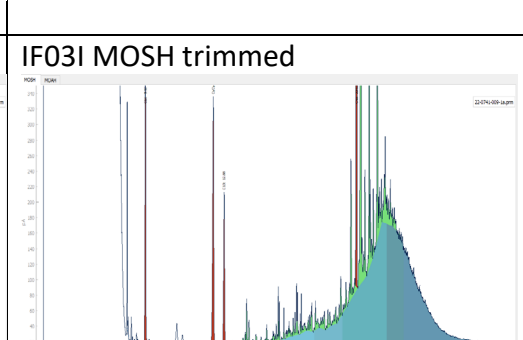
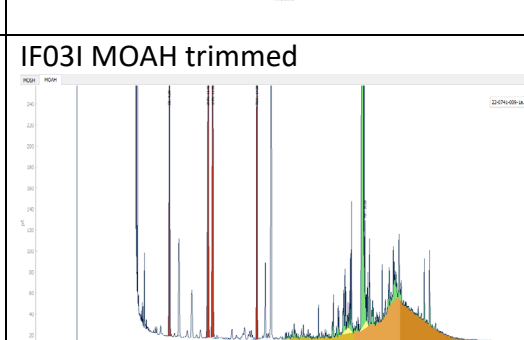
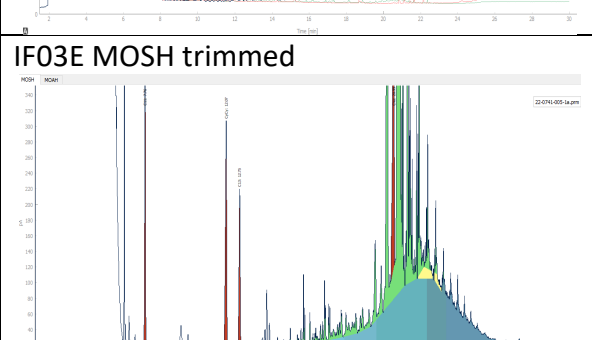
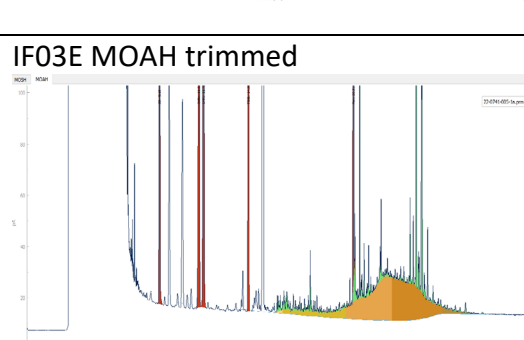
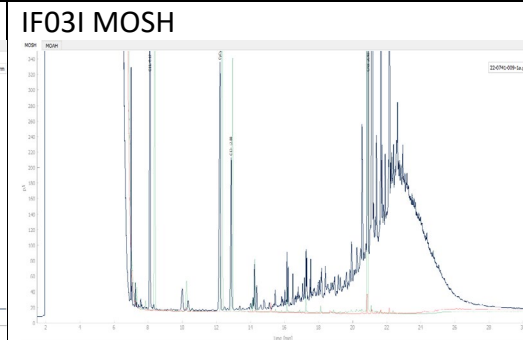
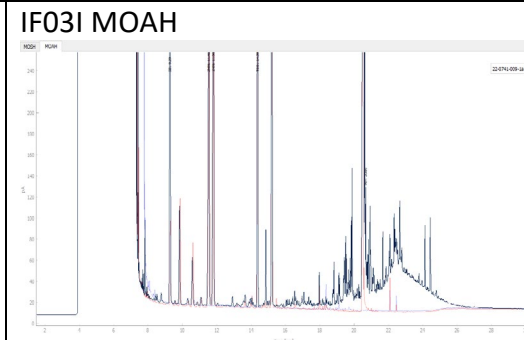
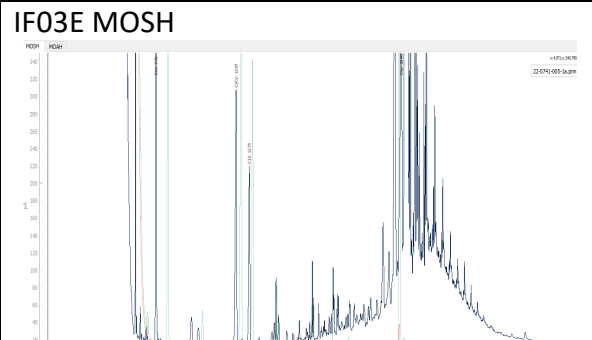
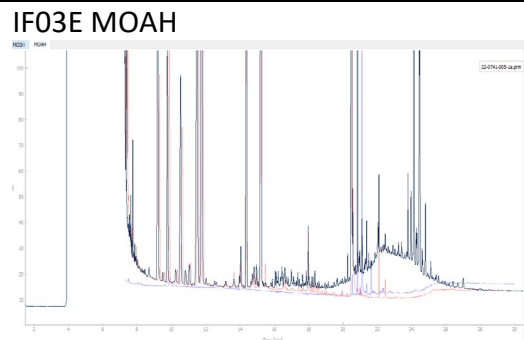
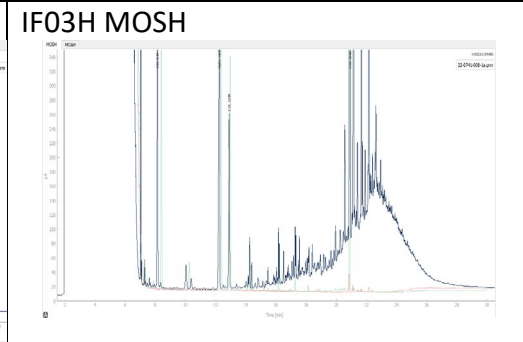
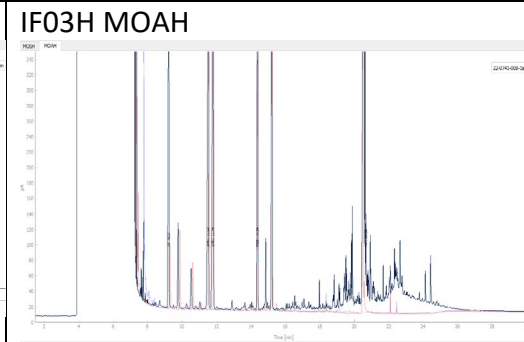
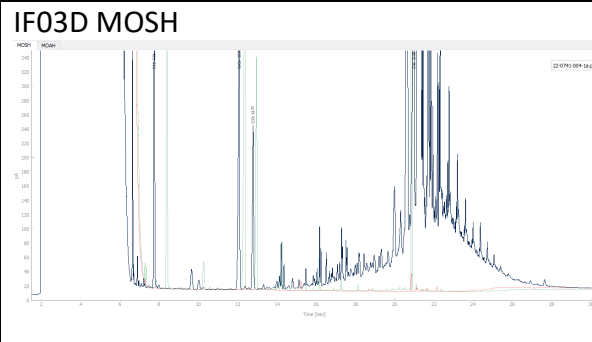
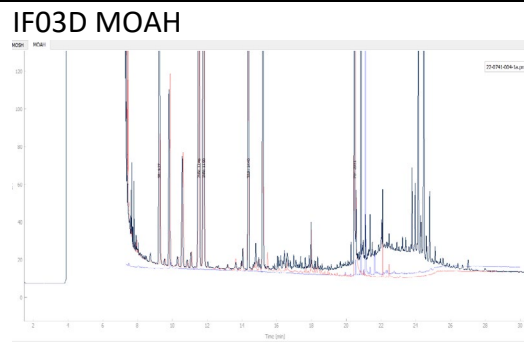
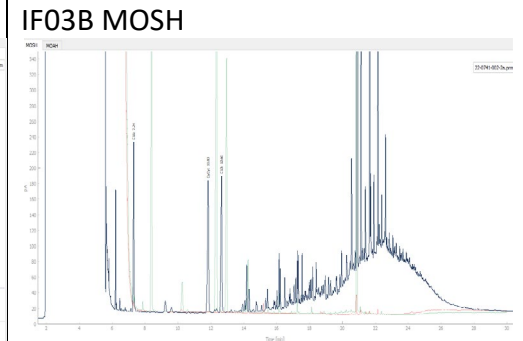
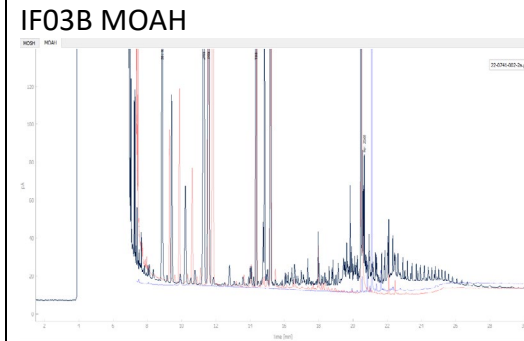
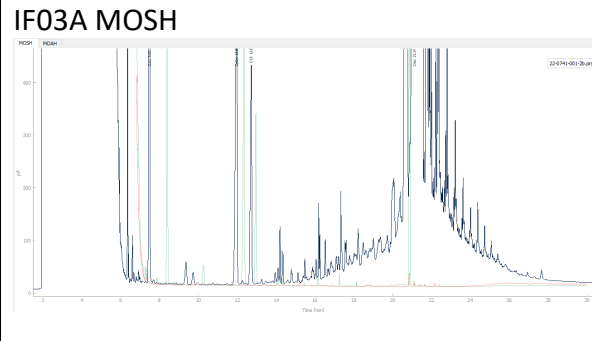
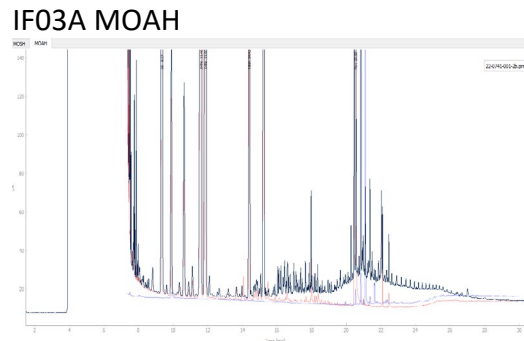
IF03I MOSH trimmed



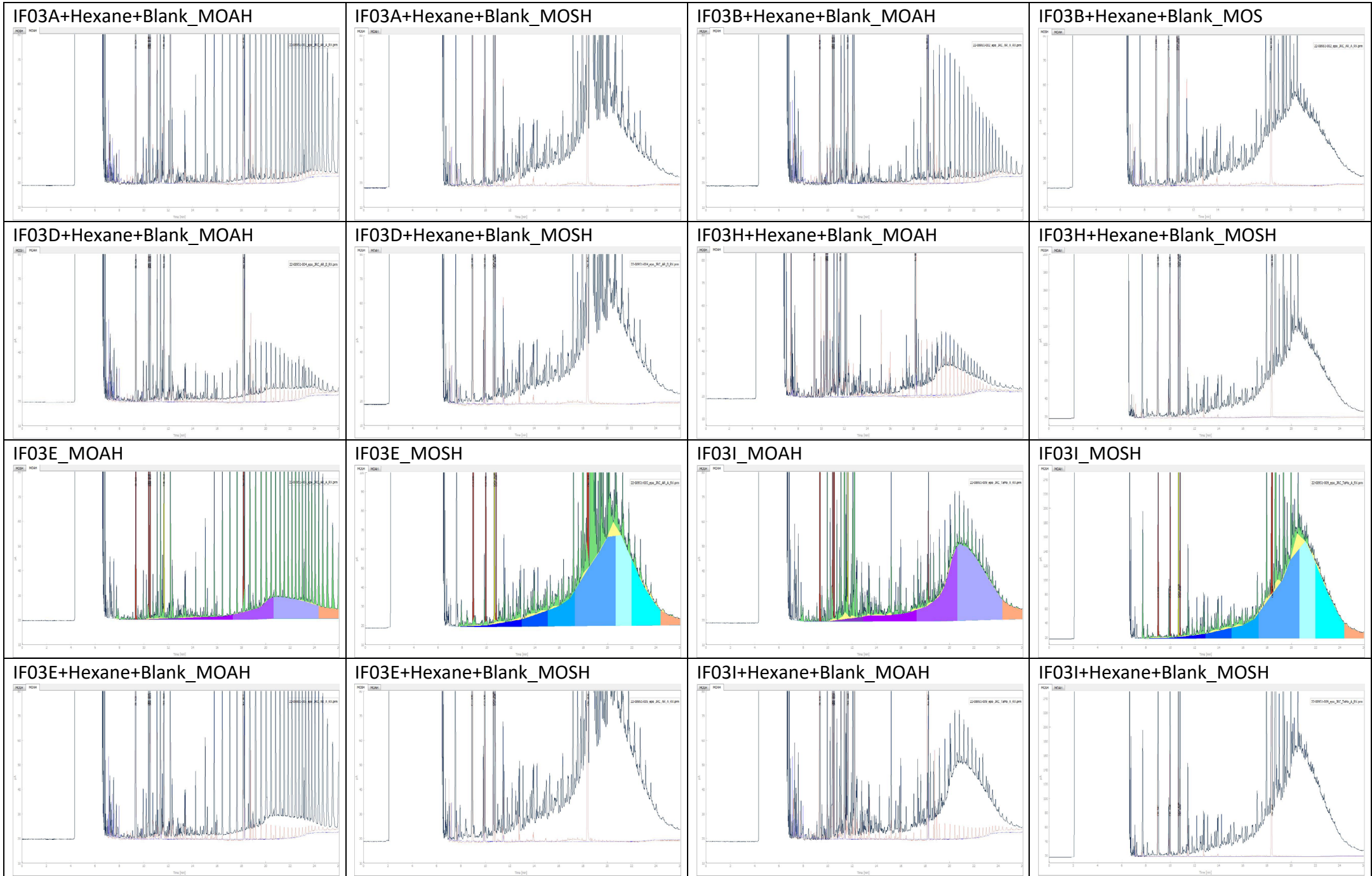
Lab 23



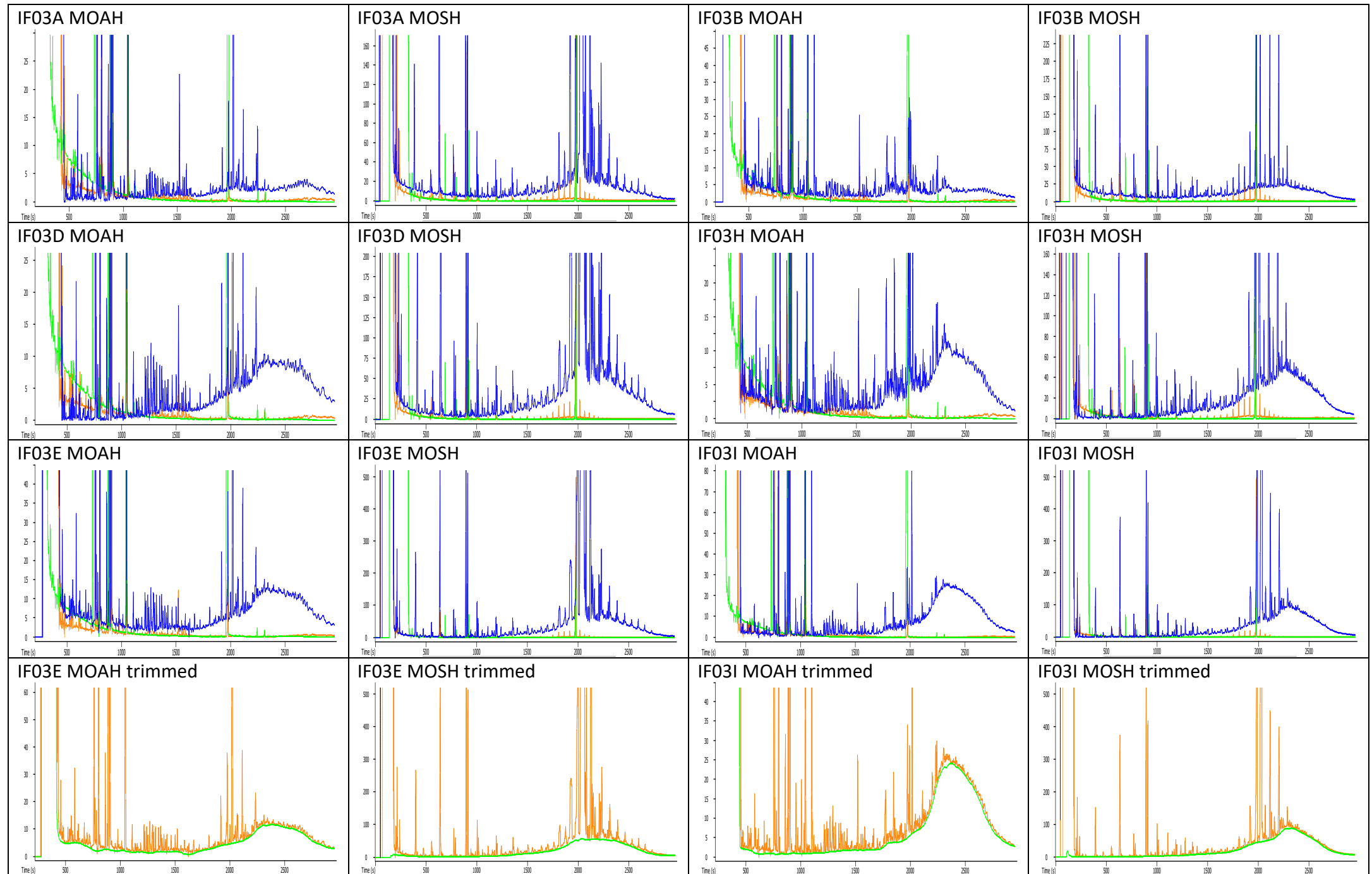
Lab 25a



Lab 28

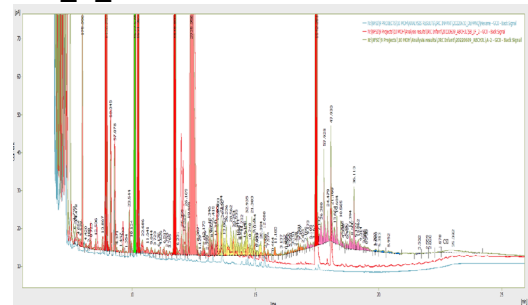


Lab 31

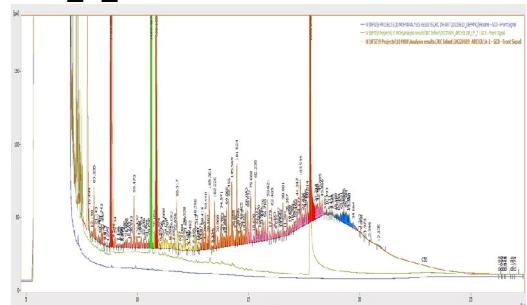


Lab 32

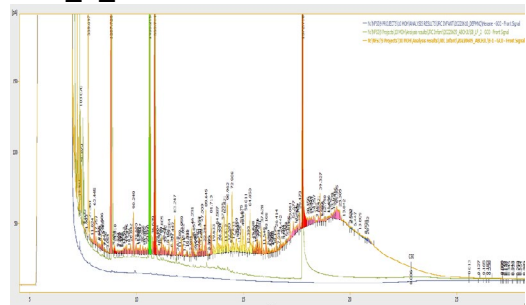
F03_A_MOAH



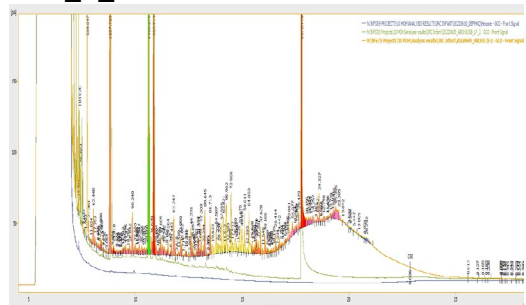
IF03_A_MOSH



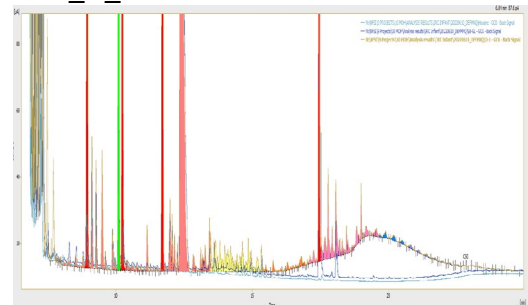
IF03_B_MOAH



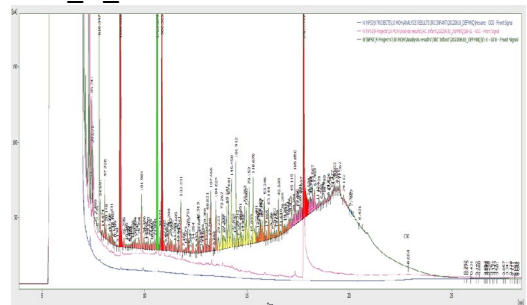
IF03_B_MOSH



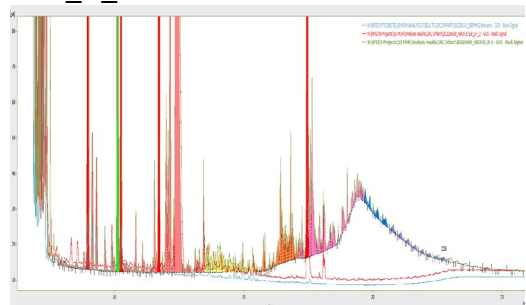
IF03_D_MOAH



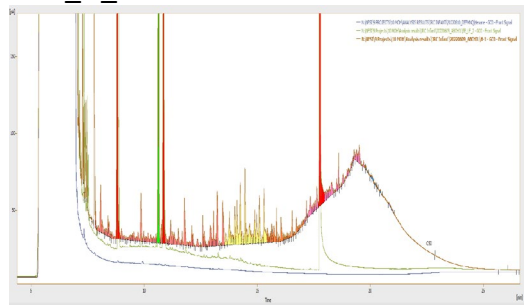
IF03_D_MOSH



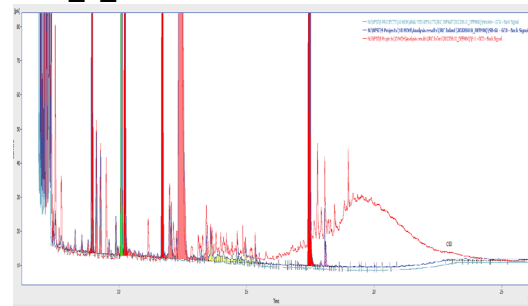
IF03_H_MOAH



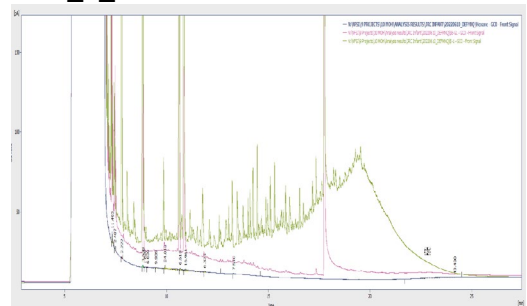
IF03_H_MOSH



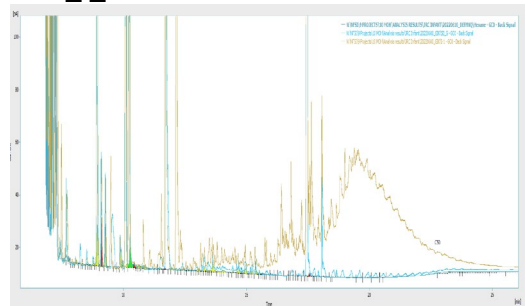
IF03_E_MOAH



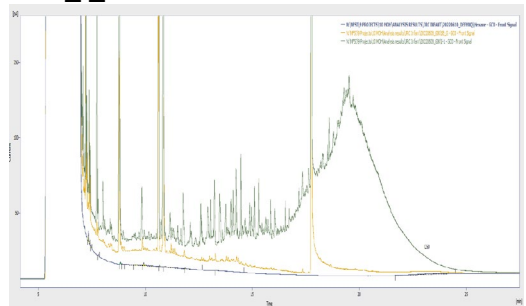
IF03_E_MOSH



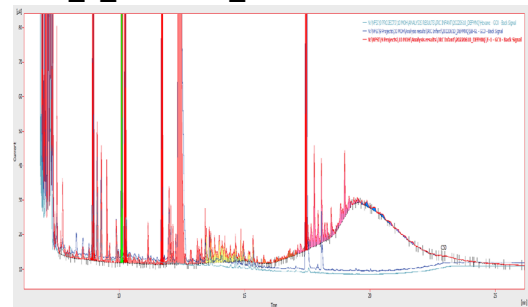
IF03_I_MOAH



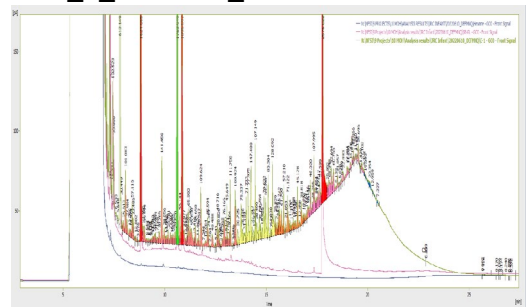
IF03_I_MOSH



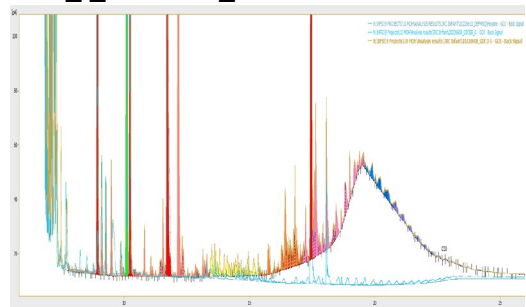
IF03_E_trimmed_MOAH



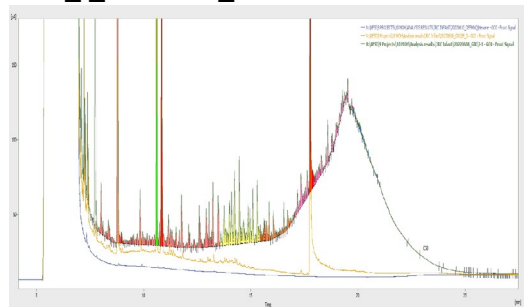
IF03_E_trimmed_MOSH



IF03_I_trimmed_MOAH



IF03_I_trimmed_MOSH



GETTING IN TOUCH WITH THE EU

In person

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

On the phone or by email

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

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

FINDING INFORMATION ABOUT THE EU

Online

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

EU publications

You can download or order free and priced EU publications from EU Bookshop at: <https://publications.europa.eu/en/publications>. Multiple copies of free publications may be obtained by contacting Europe Direct or your local information centre (see https://europa.eu/european-union/contact_en).



The European Commission's science and knowledge service

Joint Research Centre

JRC Mission

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



EU Science Hub
ec.europa.eu/jrc



@EU_ScienceHub



EU Science Hub - Joint Research Centre



EU Science, Research and Innovation



EU Science Hub

