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Method ring test
MOSH/MOAH in edible oils
(LC-GC-FID and GC×GC-FID)
P2604-MRT

Summary

The entire report is available to participants only.

The method ring test is designed, realised, evaluated, and authorised on behalf of PROOF-ACS GmbH by

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The report is approved by

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DAkkS
Deutsche
Akkreditierungsstelle
D-EP-22211-01-00

PROOF-ACS is a DAkkS accredited proficiency testing provider according to DIN EN ISO 17043:2023 (D-EP-22211-01-00). This method ring test is covered by the scope of accreditation.

PROOF-ACS GmbH does not have any analytical laboratory facilities of its own. Homogeneity testing and stability testing are subcontracted to laboratories, accredited according to DIN EN ISO 17025. The subcontracted laboratory may also participate in the ring tests. If so, the laboratory is treated in the same way as other participants and the same rules of confidentiality apply.

Method ring tests are a highly valuable instruments to gather deep insight into the real challenges of complex analytical methods like the quantification of MOSH and MOAH. Two different techniques, LC-GC-FID and GC×GC-FID are applied for quantification in routine analysis. This method ring test offers the chance to apply and compare both techniques to the same test samples.

The method ring test consists of three parts:

- Part 1: Evaluation of the analytical results

  The performance of laboratories is evaluated with respect to their ability to quantify MOSH and MOAH in two different edible oils, peanut oil, and rice bran oil.

- Part 2: The applied analytical methods

  Details related to the applied analytical methods are summarised and considered for interpretation of the analytical results.

- Part 3: Chromatograms

  The analytical procedure in quantifying MOSH and MOAH is based on the integration of the respective “humps”. The chromatograms of all laboratories are collected and summarised. Conspicuous chromatograms are discussed in the report and are considered for the interpretation of the analytical results. The plots related to the determination by GC×GC-FID are collected and summarised as well.

Peanut oil and rice bran oil are chosen as matrices for the method ring test. Both oils contain a contamination of MOSH at relevant concentration levels. Thus, the oils are provided without additional spiking with MOSH. The peanut oil raw material is free from MOAH, while the rice bran oil raw material is contaminated with MOAH at a relevant concentration level. Consequently, the peanut oil is spiked with MOAH, while the rice bran oil is provided without additional spiking with MOAH.

It is one of the challenges for the labs to trust in their own results and to report the same concentration levels related to the blank material and the test material. An evaluation of the results related to the “blank” material and the “test” material can show the repeatability of the results in the labs if the samples are not spiked, since both materials, the “test material” and the “blank material” are the same material.

13 laboratories across seven countries (Germany, Indonesia, Italy, Malaysia, Netherlands, Thailand, and Spain) took part in the test. All labs reported results and are considered for evaluation.

The laboratories are asked to report analytical results related the test materials and the blank materials. The labs are free to choose both techniques, LC-GC-FID and GC×GC-FID or just the more common LC-GC-FID.

Besides the pure analytical data, the laboratories are asked to provide comprehensive data related to the applied analytical methods in a questionnaire and chromatograms related to the test materials and the blank materials and related to reagent blank samples.

Analytical results are reported related to

- MOSH ≥ n-C10 to ≤ n-C16 (LC-GC-FID)
- MOSH > n-C16 to ≤ n-C20 (LC-GC-FID)
- MOSH > n-C20 to ≤ n-C25 (LC-GC-FID)
- MOSH > n-C25 to ≤ n-C35 (LC-GC-FID)
- MOSH > n-C35 to ≤ n-C40 (LC-GC-FID)
- MOSH > n-C40 to ≤ n-C50 (LC-GC-FID)
- Total MOSH (LC-GC-FID)
- MOAH ≥ n-C10 to ≤ n-C16 (LC-GC-FID)
- MOAH > n-C16 to ≤ n-C25 (LC-GC-FID)
- MOAH > n-C25 to ≤ n-C35 (LC-GC-FID)
- MOAH > n-C35 to ≤ n-C50 (LC-GC-FID)
- Total MOAH (LC-GC-FID)
- Total MOAH (GC×GC-FID)

The labs are asked to report the results related to LC-GC-FID in accordance with the "Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials – 2nd Edition", published by the Joint Research Centre of the European Commission.

According to the guidance document, total MOSH and total MOAH should be determined as follows:

> *„The "total MOSH/MOAH content" (n-C10-C50) is determined by integrating the chromatogram,*
> *- from the retention time of the beginning of the n-C10 peak;*
> *- to the retention time of the end of the n-C50 peak;*
> *- after the trimming of the riding peaks […] above the hump(s); and*
> *- after the subtraction of/adjustment for the reagent blank (baseline).*
>
> *The obtained "corrected hump" should be an unambiguously identified smooth hump" (page 15).*

The results related to total MOSH (LC-GC-FID) and total MOAH (LC-GC-FID and GC×GC-FID) are considered for evaluation. The results related to the individual fractions of MOSH and MOAH are summarised for information only.

The results related to total MOSH in the contaminated blank material peanut oil and total MOSH and total MOAH in the contaminated blank material rice bran oil are evaluated with respect to the comparability criterion for information only.

The raw material (= blank material) peanut oil contains 8.66 mg/kg total MOSH (assigned value), while it is free from total MOAH (< 1.0 mg/kg).

The raw material (= blank material) rice bran oil contains 13.3 mg/kg total MOSH (assigned value), and 6.22 mg/kg of total MOAH.

The raw material rice bran oil is provided as test material without additional spiking, while the raw material peanut oil is spiked with MOAH only.

The performance of laboratories in the test is evaluated according to

- the *comparability* of the results. The evaluation of the comparability is based on the z-score model. The absolute values of z-score should be at least ≤ 2. The comparability criterion is applied to total MOSH and total MOAH related the analysis by LC-GC-FID in both matrices. It is not applicable to the results related to the analysis by GC×GC-FID due to the limited number of reported results.
- the *trueness* of the results. The trueness is expressed as the coverage of the spiked level in %. The coverage should be at least between 70 and 120 % of the spiked level. The trueness criterion is applied to total MOAH by LC-GC-FID and GC×GC-FID in peanut oil only. The levels of total MOSH in both oils and total MOAH in rice bran oil are due to a contamination of the raw material, and the trueness criterion is thus not applicable for evaluation.

The statistical evaluation of the results is summarised in the tables below:

Blank material

Matrix	Parameter	Spiked level [mg/kg]	Assigned value [mg/kg]	Total number of results
Peanut oil	Total MOSH	unspiked	8.66	13
	Total MOAH (LC-GC-FID)	unspiked	< 1.00	12
Rice bran oil	Total MOSH	unspiked	13.3	12
	Total MOAH (LC-GC-FID)	unspiked	6.22	12

Test material

Matrix	Parameter	Spiked level [mg/kg]	Assigned value [mg/kg]	Total number of results	**Comparability:** no. of results, which correspond to |z-score| ≤ 2	**Trueness:** no. of results, which correspond to recoveries of 70 to 120 % of the spiked level
Peanut oil	Total MOSH	unspiked	9.00	13	12	Not applicable
	Total MOAH (LC-GC-FID)	4.1	3.53	13	13	13
	Total MOAH (GC×GC-FID)	4.1	-	5	-	5
Rice bran oil	Total MOSH	unspiked	13.4	12	7	Not applicable
	Total MOAH (LC-GC-FID)	unspiked	6.18	12	10	Not applicable
	Total MOAH (GC×GC-FID)	unspiked	-	-	Not applicable	Not applicable

The standardised analytical method DIN EN ISO 20122:2024 is applied by most of the labs. Effects of increasing standardisation and harmonisation are seen over the last years. Even though the standardised method is now available, the analytical methods still differ in detail. Different approaches and concepts for clean-up are applied by the laboratories. Aluminium oxide, epoxidation, saponification, and/or silica gel are chosen for clean-up depending in the preferences of the labs. However, knowledge and expertise are still needed to avoid false positive and false negative findings as well as incorrect interpretation of the results.

Standardisation is still needed with respect to the conditions of epoxidation. The epoxidation with mCPBA, which was the most common in the past, is outdated and most of the labs apply an epoxidation with performic acid according to Nestola or with performic acid and 1-chlorobutane.

The reverse setup (mid-polar × non-polar) is the most applied setup for quantification by GC×GC-FID. The normal setup (non-polar × mid-polar is less common.

The details related to the applied analytical methods are summarised in part 2 of the report. Part 3 of the report summarises the chromatograms of all participants. Parts 2 and 3 of the report are dedicated to the analytical specialists in the labs. Combining the analytical details to the resulting chromatograms can support the laboratories to improve the quality of the applied analytical methods and to learn from each other.