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Method ring test
MOSH/MOAH in edible oils (quantification)
P2501-MRT

Summary

The entire report is available to participants only.

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

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The report was 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 17043:2010 (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.

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 samples of edible oils.

- 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 edible oils rapeseed oil and soy bean oil are chosen as matrices for the method ring test. Both samples are provided unspiked (blank material) as well as spiked with MOSH and MOAH. Both unspiked oils contain low levels of MOSH, while both oils are free from MOAH.

15 laboratories across six countries (China, France, Germany, Malaysia, Netherlands, and Spain) took part in the test. 13 labs reported results and are considered for evaluation.

The laboratories were asked to report analytical results related the test materials and the blank materials. Besides the pure analytical data, the laboratories were 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 were reported related to the fractions

- MOSH ≥ n-C10 to ≤ n-C16
- MOSH > n-C16 to ≤ n-C20
- MOSH > n-C20 to ≤ n-C25
- MOSH > n-C25 to ≤ n-C35
- MOSH > n-C35 to ≤ n-C40
- MOSH > n-C40 to ≤ n-C50
- Total MOSH
- MOAH ≥ n-C10 to ≤ n-C16
- MOAH > n-C16 to ≤ n-C25
- MOAH > n-C25 to ≤ n-C35
- MOAH > n-C35 to ≤ n-C50
- Total MOAH

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 and total MOAH 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 blank materials are evaluated with respect to the comparability criterion for information only.

The raw material (= blank material) rapeseed oil contains 1.85 mg/kg of total MOSH, while the blank material soy bean oil contains 2.66 mg/kg of total MOSH. Both unspiked oils are free from MOAH (< 1 mg/kg). The levels of MOSH in the blank materials are considered for evaluation of the results related to the test materials with respect to the trueness criterion.

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 to both matrices.
- 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 MOSH and total MOAH related to both matrices. The levels of MOSH in the blank material are considered 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
Rapeseed oil	Total MOSH	unspiked	1.85	12
	Total MOAH	unspiked	<1	12
Soy bean oil	Total MOSH	unspiked	2.66	12
	Total MOAH	unspiked	<1	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
Rapeseed oil	Total MOSH	10	12.2	13	12	12*
	Total MOAH	4.8	4.45	13	12	12
Soy bean oil	Total MOSH	17	18.7	13	11	12*
	Total MOAH	7.7	7.74	13	10	10

* The levels of MOSH in the blank material are considered for evaluation.

A standardised analytical method (DIN EN ISO 20122:2024) is now available for quantification of MOSH and MOAH in edible oils. Effects of increasing standardisation and harmonisation were seen over the last years. The results of the laboratories became more and more comparable. Even though the standardised method is 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. Applied methods are based on epoxidation with mCPBA or performic acid. And different solvents are used during epoxidation. The conditions of epoxidation as well as the reagents might result in highly differing results.

In common proficiency tests, the statistical evaluation is limited to the comparability of the results. However, the comparability is just a first step, especially in case of challenging analytical methods. Much deeper insights are possible if the trueness criterion is applied, and if the information related to the applied analytical methods is combined with the provided chromatograms for evaluation.
The summary of the applied analytical methods (part 2 of the report) can support laboratories to improve the quality of the applied analytical method e.g. the choice of the most suitable conditions for epoxidation. Furthermore, the method details can build the basis for further discussion and thus for a standardisation of the analytical methods related to MOSH and MOAH.

The submitted chromatograms of all participants are summarised in part 3 of the report. The provided chromatograms allow for a deep insight in the challenges of quantifying MOSH and MOAH. The chromatograms thus offer a chance to each laboratory to compare the own outcome of the analytical methods to those of other laboratories on the market. Is the chromatography in line with the state-of-the-art or does it need an improvement?

Some of the major challenges by means of the analytical methods and chromatography to be solved are:

- The choice of a suitable method for clean-up (e.g. aluminium oxide, saponification, epoxidation).
- An adequate application of the clean-up and thus a satisfying removal of interfering substances.
- A sufficient sensitivity (e.g. by sufficient pre-concentration).
- An adequate identification and interpretation of interferences.

Expert knowledge is indispensable for a correct interpretation of the resulting chromatograms. The laboratories must be able to identify interferences to avoid misinterpretation and thus overestimation of the true values of MOSH and MOAH.

If the labs are experienced and sophisticated analytical methods are correctly applied, a reliable, comparable, and true quantification of MOSH and MOAH is possible.