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Method ring test
MOSH/MOAH in coconut oil (quantification, advanced, GCxGC-FID)
P2405-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.

Summary

EFSA raised concern for human health based on two scenarios: effects of one- and two-ring MOAH and of three or more ring MOAH.

Consequently, analytical laboratories are challenged to develop robust and reliable analytical methods for quantification of the mono- and diaromatic fraction of MOAH (MDAF) as well as for the quantification of the three- and polyaromatic fraction of MOAH (TPAF).

The aim of this method ring test was to evaluate,

- whether the performance of the inhouse methods for quantification of MOAH and fractions thereof by GCxGC-FID is satisfying, and
- whether the results are reliable and comparable.

No standardised analytical methods are available up to now. The results of the comparably new technique GCxGC-FID are compared to the results of the well-established and more standardised technique LC-GC-FID.

The method ring test as well as the test report consists of three parts:

- Part 1: Analytical results and discussion

  The performance of laboratories is evaluated with respect to their ability to quantify MDAF, TPAF, and total MOAH in a spiked test sample by GCxGC-FID and by their ability to quantify total MOSH and total MOAH by LC-GC-FID.

- Part 2: Applied analytical methods

  Small but important differences in the applied analytical methods might highly influence the outcome. Thus, the laboratories were asked to report details related to the applied analytical methods in a questionnaire. The analytical details support laboratories to identify shortcomings, built the basis for further discussion, and thus help to enhance the quality of the applied methods.

- Part 3: Chromatograms and plots

  Analytical shortcomings can quite often easily be identified using the corresponding chromatograms (LC-GC-FID) resp. plots (GCxGC-FID). The chromatographic separation as well as the extend of the clean-up can highly influence the quantification. The laboratories were thus asked to provide chromatograms and plots to gain insight in the quantification besides the pure numerical values.

A spiked sample of coconut oil is provided as test material. The coconut oil was spiked with a technical white oil and a crude oil.

Ten laboratories across four countries (Germany, Italy, Netherlands, and Switzerland) took part in the test. Nine labs reported results and are considered for evaluation. All nine laboratories reported results related to the quantification by LC-GC-FID, while six labs reported results related to GCxGC-FID.

Besides the pure analytical data, the laboratories were asked to provide comprehensive data related to the applied analytical methods in a questionnaire and chromatograms resp. plots related to the test material.

Analytical results were reported related to:

- MDAF and TPAF by GCxGC-FID,
- total MOAH by GCxGC-FID,
- total MOAH by LC-GC-FID, and
- total MOSH by LC-GC-FID.

The performance of laboratories in the test is evaluated according to

- 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 *comparability* of the results is not applicable due to the high variance of the results and the low number of reported results.

Summary of the spiked levels and the accepted ranges:

Parameter	Spiked level [mg/kg]	Accepted range trueness criterion [mg/kg]
MDAF	2.4	1.6 – 2.9
TPAF	3.7	2.5 – 4.5
Total MOAH	6.1	4.2 – 7.4
Total MOSH	3.4*	3.1 – 5.4

* The target value is the spiked level of 3.4 mg/kg plus the level of MOSH in the blank material of 1.1 mg/kg (assigned value from P2301-MRT).

Summary of the evaluation of the results:

Parameter	Analytical technique	Total number of results	**Trueness:** no. of results, which correspond to recoveries of 70 to 120 % of the spiked level
MDAF	GCxGC-FID	6	6
TPAF	GCxGC-FID	6	1
Total MOAH	GCxGC-FID	7	3
Total MOAH	LC-GC-FID	9	3
Total MOSH	LC-GC-FID	6	4

This method ring test goes beyond the pure statistical evaluation of the data. The reported analytical results are combined with the applied analytical methods and the provided chromatograms and plots.

The summary of the applied analytical methods (part 2 of the report) can support laboratories to improve the quality of the applied analytical method and can build the basis for further discussion and thus for a standardisation of the analytical methods.

The submitted chromatograms and plots of the participants are summarised in part 3 of the report. Typical challenges related to chromatography are discussed in the report. Furthermore, the provided chromatograms allow to draw conclusions on probable problems of individual labs, which might cause over or underestimations of the true values. The chromatograms and plots offer a chance 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?

Up to now, no harmonised or standardised analytical methods are available for quantification of MOAH and fractions thereof by GCxGC-FID.

To summarise the outcome of the method ring test:

- The applied analytical methods are inhouse methods based on different types of sample preparation and measurement.
- An adequate clean-up and enrichment, and an appropriate chromatography is necessary.
- The results are not well comparable between the different labs.
- The reliable quantification of MDAF is feasible, while the quantification of TPAF is challenging.
- Epoxidation as well as low sensitivity might be the main reasons for an underestimation of TPAF and total MOAH.
- 3 out of 7 labs reported satisfying results related to total MOAH by GCxGC-FID.
- The same 3 labs reported satisfying results related to total MOAH by LC-GC-FID.
- 4 out of 6 labs reported satisfying results related to total MOSH by LC-GC-FID.

The overall performance of the labs in this method ring test is dissatisfying. The results of the labs are not comparable, and the overall performance should be improved. A reliable quantification is feasible for experienced laboratories.