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Method ring test MOSH/MOAH in rice (quantification, advanced, GCxGC-FID and LC-GC-FID) P2503-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.

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,

a) whether the performance of the inhouse methods for quantification of MOAH and fractions thereof by GCxGC-FID is satisfying, and
b) whether the analysis by GCxGC-FID is comparable to the analysis by the more established and standardised quantification by 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 by GCxGC-FID, as well as 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 rice flour as well as the corresponding unspiked rice flour are provided as test material and blank material. The test material is spiked with a technical white oil and a crude oil.

Twelve laboratories across three countries (Germany, Italy, and Netherlands) took part in the test. All twelve laboratories analysed the materials by LC-GC-FID, while nine labs analysed the materials by GCxGC-FID as well.

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 and the blank 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 *comparability* of the results. The evaluation of the comparability is based on the z-score model. The absolute values of z-scores should be at least ≤ 2. The comparability criterion is applied to MDAF, TPAF, total MOAH, and total MOSH.
- 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 MDAF, TPAF, total MOAH, and total MOSH. The levels of total MOSH in the blank material are considered for evaluation.

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

Blank material

Parameter	Spiked level [mg/kg]	Assigned value [mg/kg]	Total number of results
MDAF	unspiked	<0.3	-
TPAF	unspiked	<0.3	-
Total MOAH (GCxGC-FID)	unspiked	<0.3	-
Total MOAH (LC-GC-FID)	unspiked	<0.3	-
Total MOSH (LC-GC-FID)	unspiked	2.13	12

Test material

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
MDAF	2.4	2.52	9	6	6
TPAF	3.4	3.04	9	5	5
Total MOAH (GCxGC-FID)	5.8	5.01	9	9	7
Total MOAH (LC-GC-FID)	5.8	5.67	12	12	12
Total MOSH (LC-GC-FID)	3.6	4.96*	12	11	8

* The assigned value corresponds to the level of MOSH in the blank material (assigned value 2.1 mg/kg) plus the spiked level of 3.6 mg/kg.

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?

To summarise the outcome of the method ring test:

- The overall performance of the labs improved a lot compared to the previous ring test related to the quantification of MOAH and fractions thereof by GCxGC-FID.
- The results are well comparable between the analysis by GCxGC-FID and LC-GC-FID. The assigned value is slightly higher for LC-GC-FID (5.67 mg/kg) compared to GCxGC-FID (5.01 mg/kg).
- Rice flour is a comparably easy matrix, where less sample clean-up is needed, compared to e.g. fats and oils.
- Four out of nine labs pass the comparability criterion and the trueness criterion for MDAF, TPAF, and total MOAH for the analysis by GCxGC-FID.
- Seven out of twelve labs pass the comparability criterion and the trueness criterion for total MOSH and total MOAH for the analysis by LC-GC-FID.
- Three labs pass the comparability criterion and the trueness criterion for MDAF, TPAF, total MOAH, and total MOSH for both analytical techniques.