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Method ring test
MOSH/MOAH by GCxGC-TOFMS
P2303-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

Dr. Birgit Schindler
Managing Director PROOF-ACS GmbH
Project coordinator

The report was approved by

Dr. Birgit Schindler
cn=Dr. Birgit Schindler,
o=PROOF-ACS GmbH,
ou=Managing Director,
email=proof@proof-acs.de, c=DE
2023.09.22 15:47:41 +02'00'

Dr. Birgit Schindler

Participants with any comments or concerns related to this ring test are invited to contact:

PROOF-ACS GmbH
Gottlieb-Daimler-Str. 1
28237 Bremen
Phone: +49 421 388 928 50
E-mail: proof@proof-acs.de
www.proof-acs.de

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.

Nowadays LC-GC-FID techniques are used for quantification of MOSH/MOAH in food stuff in daily routine. However, the information on the type of MOSH/MOAH, which can be gathered from the analysis by LC-GC-FID is limited and is based on typical patterns of certain contaminations only.

GCxGC-TOFMS is applied if more information is necessary on the different types of structural sub-groups, to draw conclusions on the sources of contamination or to verify the findings of the analysis by LC-GC-FID if results are questionable.

Up to now, there is no harmonised approach established for identification of MOSH/MOAH and related substances by GCxGC-TOFMS. Laboratories, especially newcomers in the field of characterisation of MOSH/MOAH by GCxGC-TOFMS, struggle with the lack of appropriate ring tests for the applied method.

A previous method ring test (P2206-MRT) related to the identification of MOSH and MOAH by GCxGC-TOFMS in cheese was organised by PROOF in 2022. The identification of subgroups and typical markers of a contamination with MOSH and MOAH in food stuff was not well harmonised in 2022. Laboratories were struggling with setting up this new method at that time.

In 2023, two samples of different samples of coconut oils were offered as test materials in this method ring test:

- Test material 1 is a native coconut oil, which is contaminated with POSH and spiked with DIPN and a mixture of different PAH standards.
- Test material 2 is a refined coconut oil, which is spiked with a crude oil, a paraffin wax, a poly alpha olefin (PAO), and benzo[a]anthracene.

Chrysene, which is part of the PAH standard in test material 1 as well as benzo[a]anthracene, which was spiked to test material 2 were used for homogeneity testing of the two test materials.

The method ring test consists of three parts:

- Part 1: Analytical results:

  The laboratories were asked to identify the most popular subgroups as well as typical marker substances of MOSH and MOAH and related contaminations in the test materials.
  Subgroups and markers are: n-alkanes, i-alkanes, multi-branched alkanes, cycloalkanes, hopanes, steranes, phytane, pristane, POSH, PAO, waxes, ROSH, alkylated benzenes, alkylated naphthalenes, alkylated anthracenes, alkylated benzanthracenes, 2- to 6-ring MOAH, DIPN, benzothiophenes, and ROAH.
  The laboratories reported the results as “yes” for compounds, which were identified in the samples resp. “no” for compounds, which were not identified in the sample. Furhtermore, the labs were asked for an interpretation of the analytical results by means of drawing a conclusion on potential sources of contamination based on the reported results.The labs were able to provide any additional information, which they considered useful for the interpretation.

- Part 2: Questionnaire related to the applied analytical techniques:

  The most relevant aspects of the applied analytical techniques were asked for in a questionnaire.

- Part 3: Contour plots:

  The labs were asked to submit contour plots related to the two test materials and related to a procedural blank sample, spiked with internal standards. The labs were asked to highlight all subgroups, which were identified in the contour plots.

Results

11 labs from Germany, Malaysia, Netherlands, and Switzerland took part in the test. 8 labs reported results and are considered for evaluation.

Test material 1

The laboratories were expected to identify

- n-alkanes, i-alkanes, multi-branched alkanes, cycloalkanes,
- POSH
- 2- to 5-ring MOAH, and
- DIPN

and to confirm the absence of

- PAO,
- waxes,
- ROSH,
- benzothiophenes, and
- ROAH.

The overall performance of the labs is as follows:

Test material 1

Sub-group	n- / i- / multi-branched / cyclo-alkanes	POSH	2- to 5-ring MOAH	DIPN	PAO (not present)	Wax (not present)	ROSH (not present)	Benzothiophenes (not present)	ROAH (not present)
Correctly identified by	5 out of 8 labs	8 out of 8 labs	6 out of 8 labs	8 out of 8 labs	8 out of 8 labs	7 out of 8 labs	7 out of 7 labs	7 out of 8 labs	7 out of 7 labs

Interpretation of the results:

All 8 labs provided an interpretation of potential sources of contamination. The labs reported paperboard and plastic packings correctly. Some labs reported jute and mineral oils due to the identified 2- to 5-ring MOAH (PAH), which is not considered incorrect. Two labs reported waxes resp. food-grade oils, which is considered incorrect.

Test material 2

The laboratories were expected to identify:

- n-alkanes, i-alkanes, multi-branched alkanes, cycloalkanes,
- hopanes,
- steranes,
- phytane,
- pristane,
- PAO,
- a wax,
- alkylated MOAH,
- 2- to 4-ring MOAH,

and to confirm the absence of

- POSH,
- ROSH,
- DIPN, and
- ROAH.

The overall performance of the labs was as follows:

Test material 2

One of the labs reported difficulties with the MOSH fraction and did not report results related to parameters of the MOSH fraction.

Sub-group	n- / i- / multi-branched / cyclo-alkanes	hopanes	steranes	phytane	pristane	PAO	wax	Alkylated MOAH	2- to 4-ring MOAH
Correctly identified by	7 out of 7 labs	6 out of 7 labs	6 out of 7 labs	5 out of 7 labs	5 out of 7 labs	6 out of 7 labs	4 out of 7 labs	7 out of 8 labs	8 out of 8 labs

Sub-group	POSH (not present)	ROSH (not present)	DIPN (not present)	ROAH (not present)
Correctly identified by	7 out of 7 labs	7 out of 7 labs	8 out of 8 labs	6 out of 7 labs

Interpretation of the results:

All 8 labs provided an interpretation of potential sources of contamination. The interpretation of one of the labs is limited, because it is based on the results of the MOAH fraction only. All labs identified mineral oils as a potential source of contamination. Synthetic lubricants, jute, and waxes were reported by most of the labs. Two labs reported adhesives. All sources mentioned before are considered correct. A lab reported plastic packings and food-grade oil, which is both considered incorrect.

Conclusion:

- The labs can identify common markers of contamination with MOSH/MOAH and related substances.
- Overall, the results are well comparable but there are differences in the expert level of the labs.
- The overall quality of most of the submitted contour plots is quite satisfying by means of the chromatographic quality as well as by means of the clear presentation of the results.
- There are differences in the sensitivity of the applied analysis, and thus, some of the compounds, which were present at lower concentration levels were not identified by some of the labs.
- Further harmonisation of the analysis of MOSH/MOAH by GCxGC-TOFMS as well as an exchange of knowledge between the laboratories is desirable to improve the overall quality of the analytical data.
- The question whether or not PAH are considered as MOAH should be finally discussed and harmonised.