

Method ring test MOSH/MOAH by GC×GC-TOF-MS P2606-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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Summary

Nowadays LC-GC-FID and/or GCxGC-FID techniques are used for quantification of MOSH/MOAH in food stuff in daily routine.

GCxGC-TOFMS is applied if more information is necessary on the different types of structural sub-groups, and to draw conclusions on the sources of contamination.

Work is in progress to set up a harmonised approach for identification of MOSH/MOAH and related substances by GCxGC-TOFMS.

Method ring test were organised by PROOF-ACS in 2022 (P2206-MRT, cheese) and 2023 (P2303-MRT, coconut oil) related to the identification of MOSH and MOAH by GCxGC-TOFMS. The identification of subgroups and typical markers of a contamination with MOSH and MOAH in food stuff was not well harmonised in at that time. Laboratories were struggling with gathering experience with the new analytical technique and the applied method.

This method ring test consists of three parts:

- Part 1: Analytical results:

The laboratories are 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 benzantracenes, 1- to 6-ring MOAH, DIPN, benzothiophenes, and ROAH.

The laboratories reported the results as “yes” for compounds, which are identified in the samples resp. “no” for compounds, which are not identified in the sample. Furthermore, the labs are asked for an interpretation of the analytical results by means of drawing a conclusion on potential sources of contamination based on the reported results.

- Part 2: Questionnaire:

The most relevant aspects of the applied analytical instruments and analytical methods are asked for in a questionnaire.

- Part 3: Contour plots:

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

Two different test materials are offered in P2606-MRT:

- Test material 1 is a sample of cocoa butter, which is spiked with a crude oil standard and a paraffin wax, and contains a contamination with POSH and DIPN.
- Test material 2 is a sample of dried beans, offered as bean flour, which is spiked with a lubricant oil and a process oil, a poly alpha olefin (PAO), and benzo[a]anthracene (for homogeneity testing), and which contains a contamination with mineral oil components (phytane, pristane) as well as a contamination with POSH.

Results

7 labs from Germany, and Netherlands took part in the test. All 7 labs reported results and are considered for evaluation.

Test material 1 - cocoa butter

The laboratories are expected to identify

- n-alkanes, i-alkanes, multi-branched alkanes, cycloalkanes,
- hopanes,
- steranes,
- phytane,
- pristane,
- POSH,
- wax
- Alkylated benzenes, alkylated naphthalenes, alkylated anthracenes, alkylated benzantracenes,
- 1- to 6-ring MOAH,
- DIPN, and
- benzothiophenes,

and to confirm the absence of

- PAO,
- ROSH, and
- ROAH.

The overall performance of the labs is as follows:

Test material 1 - cocoa butter

Sub-group	n- / i- / multi-branched / cyclo-alkanes	Hopanes and steranes	Phytane and pristane	POSH	Wax
Correctly identified by	7 out of 7 labs	6 out of 7 labs	7 out of 7 labs	5 out of 7 labs	5 out of 7 labs

Sub-group	Alkylated benzenes, naphthalenes, and anthracenes	Alkylated benzantracenes	1-ring MOAH	2- and 3-ring MOAH	4-ring MOAH	5- and 6-ring MOAH	DIPN	Benzothiophenes
Correctly identified by	7 out of 7 labs	5 out of 7 labs	6 out of 7 labs	7 out of 7 labs	5 out of 7 labs	4 out of 7 labs	5 out of 7 labs	4 out of 7 labs

Sub-group	PAO	ROSH	ROAH
Absence confirmed by	7 out of 7 labs	7 out of 7 labs	7 out of 7 labs

Interpretation of the results:

6 out of 7 labs provided an interpretation of potential sources of contamination. Most of the labs reported mineral oils, plastic packing, paperboard, and waxes correctly.

Test material 2 - dried beans

The laboratories are expected to identify:

- n-alkanes, i-alkanes, multi-branched alkanes, cycloalkanes,
- hopanes,
- steranes,
- phytane,
- pristane,
- POSH,
- PAO,
- Alkylated benzenes, naphthalenes, anthracenes, and benzantracenes, and
- 1- to 6-ring MOAH,

and to confirm the absence of

- waxes,
- ROSH, and
- ROAH.

DIPN and benzothiophenes are not considered for evaluation.

The overall performance of the labs is as follows:

Test material 2 - dried beans

Sub-group	n- / i- / multi-branched- alkanes	Cycloalkanes	Hopanes	Steranes	Phytane and pristane	POSH	PAO
Correctly identified by	7 out of 7 labs	5 out of 7 labs	6 out of 7 labs	5 out of 7 labs	7 out of 7 labs	4 out of 7 labs	7 out of 7 labs

Sub-group	Alkylated benzenes, naphthalenes, anthracenes	Alkylated benzanthracenes	1-ring MOAH	2-ring MOAH	3-ring MOAH	4-ring MOAH	5- and 6-ring MOAH
Correctly identified by	6 out of 7 labs	3 out of 7 labs	6 out of 7 labs	7 out of 7 labs	6 out of 7 labs	5 out of 7 labs	3 out of 7 labs

Sub-group	Wax	ROSH	ROAH
Absence confirmed by	7 out of 7 labs	7 out of 7 labs	7 out of 7 labs

Interpretation of the results:

6 out of 7 labs provided an interpretation of potential sources of contamination. Most of the labs identified the spiked compounds correctly as due to synthetic lubricants and mineral oils, while 3 labs failed for the plastic packing due to false negative results related to POSH.

Conclusion:

- The labs can identify common markers of contamination with MOSH/MOAH and related substances.
- The results are comparable on an overall basis, 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 are 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.