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Method ring test
MOSH/MOAH in cheese by GCxGC-TOF
P2206-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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08 November 2022

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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 exactly the same way as other participants and the same rules of confidentiality apply.

Summary

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-TOF 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-TOF. Laboratories, especially newcomers in the field of characterisation of MOSH/MOAH by GCxGC-TOF, struggle with the lack of appropriate ring tests for the applied method.

Method ring test P2206-MRT was organised to solve this issue and to get an idea of the state-of-the-art in the identification of subgroups and typical markers of a contamination with MOSH and MOAH in food stuff.

Two samples of cheese were offered as test materials for this purpose:

- Test material 1 was spiked with a crude oil, a paraffin wax, and diisopropyl naphthalene (DIPN).
- Test material 2 was contaminated with PP vessels and adhesives from cheese packings and spiked with a technical white oil and a poly alpha olefin (PAO).

Furthermore, both test materials were spiked with chrysene for homogeneity testing.

The results reporting consisted 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 MOSH and related contaminations in the test materials.
  Subgroups and markers were: n-alkanes, i-alkanes, cyclo-alkanes, hopanes, steranes, phytane, pristane, POSH, PAO, waxes, ROSH, alkylated benzenes, naphthalenes, benzanthracenes, chrysenes, alkylated tetrahydro naphthalenes, alkylated octahydro anthracenes, alkylated dodecahydro benzanthracenes, DIPN, (di-)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. Furthermore, 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. And 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.

- 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

8 labs from Germany, Netherlands, and Switzerland took part in the test. 5 labs reported results and are considered for evaluation.

Test material 1

The laboratories were expected to report:

- Components of crude oils (alkanes, hopanes, steranes, phytane, pristane, alkylated and non-alkylated polycyclic aromatic hydrocarbons (PAH), benzothiophenes, dibenzothiophenes),
- waxes,
- DIPN

The identification of n-/i-/cyclo alkanes, hopanes, steranes, phytane, pristane, alkylated and non-alkylated PAH, DIPN and (di)-benzothiophenes as well as the absence of POSH, PAO, ROSH and ROAH are considered for evaluation. The identification of the spiked wax is difficult, and the results are thus presented for information only.

None of the labs reported false positive results related to POSH, PAO, ROSH, and ROAH.

The overall performance of the labs is as follows:

Test material 1

Sub-group	**n- / i- / cyclo-alkanes**	**hopanes**	**steranes**	**phytane**	**pristane**	**Non-alkylated PAH**	**Alkylated benzenes and alkylated naphthalenes**	**Alkylated octahydro anthracenes**	**Alkylated dodecahydrobenzanthracenes**	**DIPN**	**(Di)-Benzothiophenes**
Correctly identified by	5 out of 5 labs	5 out of 5 labs	5 out of 5 labs	5 out of 5 labs	5 out of 5 labs	5 out of 5 labs	5 out of 5 labs	4 out of 5 labs	3 out of 5 labs	5 out of 5 labs	4 out of 5 labs

Interpretation of the results:

All 5 labs provided an interpretation of potential sources of contamination. The labs reported paperboard and mineral oils correctly as well as the absence of plastic packings and adhesives. Waxes were correctly identified by two labs and jute by 4 out of 5 labs. One lab mentioned food-grade oils as a source of contamination, which is considered incorrect due to the high amounts of aromatic compounds in the oils.

Test material 2

The laboratories were expected to report:

- POSH (cheese was stored in PP-vessels),
- ROSH and ROAH (glues from resealable cheese packings),
- PAO (spiked)
- Hopanes, steranes (white oil, spiked)
- chrysene (added for homogeneity testing of the prepared samples),
- alkylated and non-alkylated PAH of the technical white oil

The results related to pristane and related to the alkylated and non-alkylated PAH are presented for information only and are not considered for evaluation.

The overall performance of the labs was as follows:

Test material 2

Sub-group	n- / i- / cyclo-alkanes	hopanes	steranes	phytane	POSH	PAO	ROSH	ROAH
Correctly identified by	5 out of 5 labs	4 out of 5 labs	3 out of 5 labs	5 out of 5 labs	2 out of 5 labs	5 out of 5 labs	5 out of 5 labs	4 out of 5 labs

Interpretation of the results:

All 5 labs provided an interpretation of potential sources of contamination.
All labs identified adhesives and synthetic lubricants as potential sources of contamination. None of the lab identified a wax in the sample, which is correct. Only two labs identified the contamination with the PP vessels correctly and provided the correct interpretation “plastic packing”. Two labs postulated paperboard as a source of contamination based on the

findings of DIPN. The material was not contaminated/spiked with DIPN during preparation, but it cannot be excluded that the material contains DIPN. The interpretation is thus not considered incorrect. The interpretation “food-grade oils” is considered incorrect due to the high amount of aromatic compounds. One lab identified dibenzothiophenes in the material and provided the interpretation “jute bag”. However, the material was not spiked or contaminated with dibenzothiophenes and the other four labs did not report dibenzothiophenes. Thus, it can be assumed that this is a false positive result as well as a misinterpretation.

Conclusion:

- The labs are able to identify common markers of contamination with MOSH/MOAH and related substances.
- Overall, the results are well comparable.
- The overall quality 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-TOF as well as an exchange of knowledge between the laboratories is desirable in order to improve the overall quality of the analytical data.