Facing analytical quality.

Method ring test
MOSH/MOAH in chocolate by GCxGC-TOF
P2101-MRT

Summary

The entire report is available to participants only.

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June 2021,

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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 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, in order 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. Thus, the aim of this method ring test was to evaluate whether the results of different laboratories are comparable and to get a first 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 chocolate were offered as test materials for this purpose:

- Test material 1 was contaminated with a jute bag, a white oil, poly propylene (PP)-vessels, a poly alpha olefin (PAO), a wax, and diisopropyl naphthalene (DIPN).
- Test material 2 was contaminated with a crude oil, and DIPN.

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 blank sample, spiked with internal standards. The labs were asked to highlight all subgroups, which were identified in the contour plots.

Results

11 labs across four countries (Germany, Netherlands, Malaysia, and Switzerland) took part in the test. 10 labs provided results and are considered for evaluation.

Test material 1

The laboratories were expected to report:

- Components of batching oils
- Components of the white oil
- Waxes
- POSH
- PAO
- DIPN
- Chrysenes

n-/i-/cyclo alkanes, POSH, PAO, DIPN and chrysenes are considered for evaluation. The sub-groups of MOSH/MOAH in the batching oil as well as in the white oil are not sufficiently specified resp. the concentration of the batching oils from the jute bag are low and thus difficult to identify. However, all findings are summarised and discussed in the report.

The overall performance of the labs is as follows:

Test material 1

Sub-group	n-alkanes	iso-alkanes	cyclo-alkanes	POSH	PAO	DIPN	chrysenes
Correctly identified by	9 out of 10 labs	10 out of 10 labs	9 out of 10 labs	10 out of 10 labs	9 out of 10 labs	9 out of 10 labs	7 out of 10 labs

The identification of the mineral oil markers hopanes, steranes, phytane, pristane as well as of the spiked wax is more challenging. Due to the low concentration and due to epoxidation of the test material, the markers of the contamination with the jute bag (dibenzothiophenes) were identified by some of the labs only. Some labs had difficulties in reporting the different sub-groups of aromatic compounds, since this is not too common in routine.

Interpretation of the results:

9 out of 10 labs provided an interpretation of potential sources of contamination.
Most of the labs identified paperboard, mineral oils and plastic packings correctly as sources of contamination of test material 1. Food-grade oils were reported by some of the labs, which is not correct due to the high amounts of aromatic compounds in the oils. Waxes were correctly identified as a sources of contamination by two labs. Due to a misinterpretation of cyclo alkanes as ROSH and of alkylated octahydro anthracenes as ROAH, five labs reported a contamination with adhesives, which is not correct.

Test material 2

The laboratories were expected to report:

- Components of the crude oil
- DIPN

The parameters n-/i-/cyclo-alkanes, hopanes, steranes, phytane, pristane, naphthalenes, benzanthracenes, chrysenes, and DIPN, as well as the absence of POSH and PAO are considered for evaluation of test material 2. All further parameters are discussed in detail but not considered for evaluation of the performance of the labs.

The overall performance of the labs was as follows:

Test material 2 - MOSH fraction

Sub-group	n-alkanes	iso-alkanes	cyclo-alkanes	hopanes	steranes	phytane	pristane	POSH	PAO
Correctly identified by	10 out of 10 labs	10 out of 10 labs	10 out of 10 labs	8 out of 10 labs	10 out of 10 labs	9 out of 10 labs	9 out of 10 labs	9 out of 10 labs	10 out of 10 labs

Test material 2 - MOAH fraction

Sub-group	DIPN	naphthalenes	benz-anthracenes	chrysenes
Correctly identified by	10 out of 10 labs	10 out of 10 labs	8 out of 10 labs	9 out of 10 labs

Interpretation of the results:

9 out of 10 labs provided an interpretation of potential sources of contamination.
All 9 labs identified paperboard and mineral oils correctly as sources of contamination. False positive interpretations were provided with respect to adhesives (4 labs), waxes (2 labs), synthetic lubricants (2 labs), plastic packings (2 labs), and food-grade oils (1 lab).

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.
- Interpretation of the findings is challenging, especially in case of overlapping contaminations like cyclo alkanes and waxes with low molecular weights.
- Experience in interpretation is necessary especially with respect to ROSH and ROAH in order to avoid false positive findings.
- 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.