

Mineral Oil Residues in Food: Part 5 - How to choose the right sample preparation

▪ Introduction

Mineral oil (MO) residues in food raised public concern due to some elevated concentrations up to several thousand milligrams per kilogram food [1]. Due to the chemical structures two groups of MOs can be differentiated. Mineral oil saturated hydrocarbons (MOSH) consist of linear and branched alkanes, and alkyl-substituted cycloalkanes, whilst mineral oil aromatic hydrocarbons (MOAH) include mainly alkyl-substituted polyaromatic hydrocarbons. Technical grades of mineral contain aromatic hydrocarbons in a concentration range from 15-35%. The determination of MOSH and MOAH in food can be done by an automated LC-GC-FID system for routine analysis. Unfortunately, sample preparation of the “food-matrix” is complex and difficult.

▪ Challenge “Food”- Matrix

The analysis of mineral oil hydrocarbons in food needs for every type of food an adequate sample preparation. The analysis starts with the question, if contaminations only occur on the surface (via migration), or if MOH is also incorporated. Therefore, quantitative extraction of the mineral oil hydrocarbons from the matrix needs varying conditions and strategies. Depending on the matrix, long extraction times are used and extraction efficiency needs to be controlled for every matrix. However, also when extraction is finished, there are several problems. Many food types include a large amount of fat, that needs to be removed prior analysis. Other food types, contain naturally occurring olefins (eg. Squalene, sterenes, carotenoids..), which can interfere with the analysis of the aromatic fraction and need

epoxidation or naturally occurring n-Alkanes that interfere with the MOSH and need to be removed by column chromatography with aluminium oxide.

The following schemes show a summary of possible extraction ways and extract clean up and enrichment for several food types.

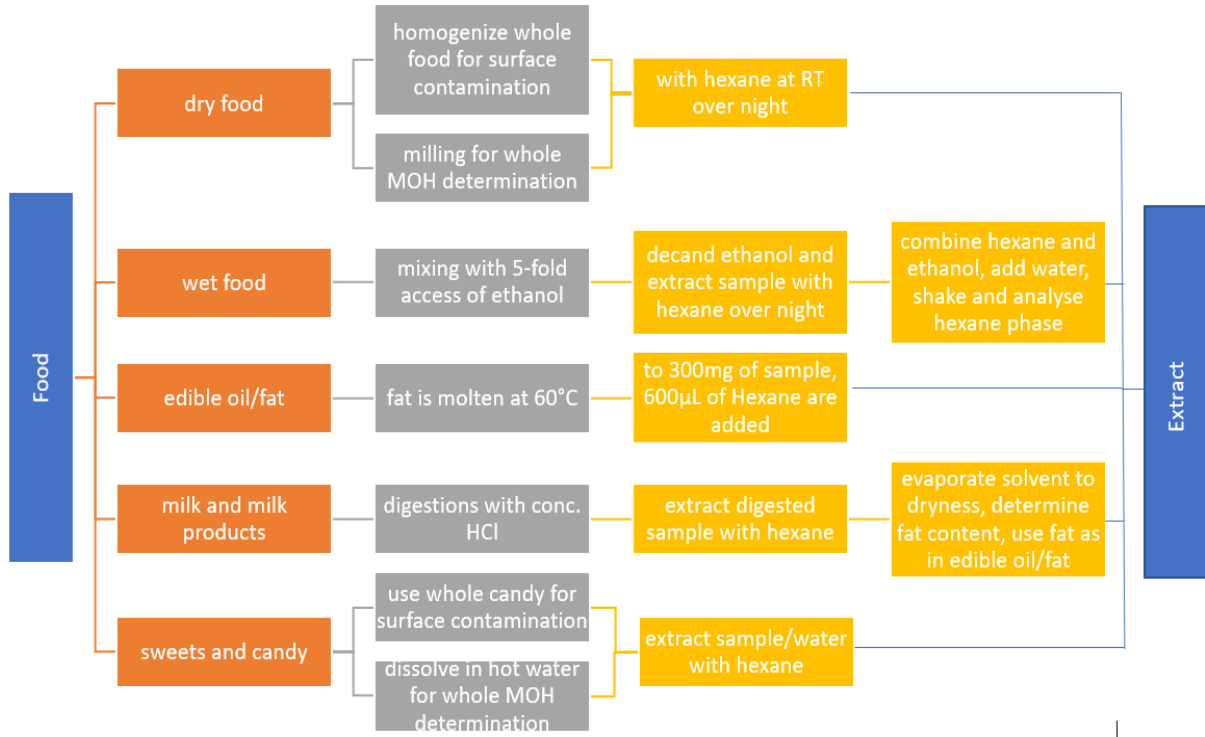
A detailed summary of extraction conditions for different food types can be found in [1,2]. Clean up and enrichment strategies can be found in the Shimadzu application notes #2 and #3 and in [3,4,5].

▪ System Setup

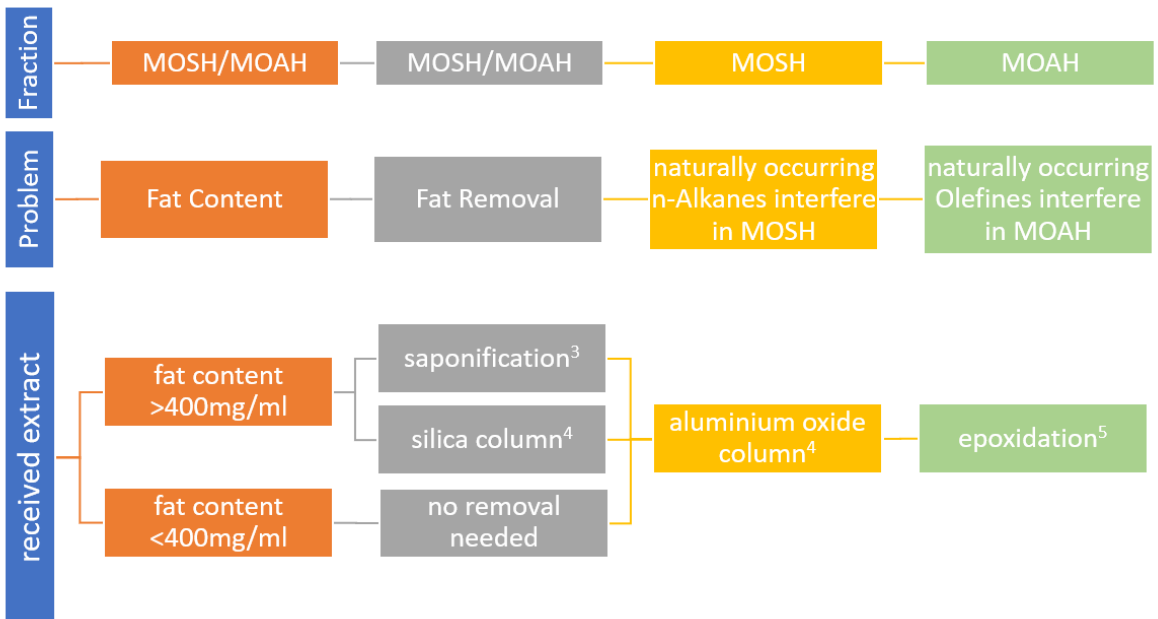
The LC is directly connected to two high temperature GC columns with retention gaps which are installed in one GC oven. MOSH and MOAH fractions are separated on a silica gel programme is started and both fractions are separated simultaneously and detected by FID. Figure 1 shows a typical LC-GC online system based on the Shimadzu LC-20AD solvent delivery pumps, the GC-2030 gas chromatograph and the LC-GC Chronect Interface by Axel Semrau.



Figure 1: LC-GC online system



Scheme 1: Extraction conditions for several food types



Scheme 2: Clean-up of received extract, depending on fraction and interferent; the limit of 400mg/ml refers to an LC column with 20mg fat limit and an injection of 50µL

▪ Conclusion

The analysis of mineral oil hydrocarbons in food is really challenging and needs a wide range of different extraction methods, clean up and enrichment steps.

▪ References

[1] Biedermann M, Grob K. 2012a. On-line coupled high performance liquid chromatography–gas chromatography for the analysis of contamination by mineral oil. Part 1: Method of analysis. J. Chromatogr. A. 1255:56-75.

[2] [BfR] German Federal Institute for Risk Assessment. Messung von Mineralöl – Kohlenwasserstoffen in Lebensmitteln und Verpackungsmaterialien.

<https://mobil.bfr.bund.de/cm/343/messung-von-mineraloel-kohlenwasserstoffen-in-lebensmitteln-und-verpackungsmaterialien.pdf> [14.09.2018]

[3] A. Guinda, A. Lanzón, T. Albi, 1996, 'Differences in Hydrocarbons of Virgin Olive Oils Obtained from Several Olive Varieties', Journal of Agricultural and Food Chemistry, vol. 44, no. 7, pp. 1723-1726.

[4] EN 16995:2017-06: Lebensmittel — Pflanzliche Öle und Lebensmittel auf Basis pflanzlicher Öle — Bestimmung von gesättigten Mineralöl-Kohlenwasserstoffen (MOSH) und aromatischen Mineralöl-Kohlenwasserstoffen (MOAH) mit on-line HPLC-GC-FID.

[5] Marco Nestola, Torsten C. Schmidt, 2017, 'Determination of mineral oil aromatic hydrocarbons in edible oils and fats by online liquid chromatography–gas chromatography–flame ionization detection – Evaluation of automated removal strategies for biogenic olefins', Journal of Chromatography A, vol. 1505, pp. 69-76



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