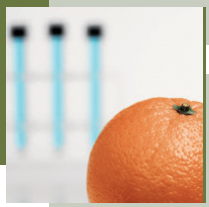

FOOD

Influence of Endogenous n-Alkanes on the Determination of MOSH and MOAH in Food of Plant Origin

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Introduction and Objectives

Mineral oil is a complex mixture containing amongst others different hydrocarbon fractions. One fraction is defined as mineral oil saturated hydrocarbons (MOSH) characterized by both paraffin-like open-chained hydrocarbons generally branched and naphthenic compounds with at least one saturated ring structure. Often a maximum of compounds with 18 to 20 carbon atoms is observed. Another fraction contains mineral oil aromatic hydrocarbons (MOAH) comprising systems of one to four highly alkylated aromatic rings.

MOSH and MOAH were determined in dry food in direct contact to printed paper-based packaging or packaging made of recycling paper. This contamination is caused via vapour space migration from mineral oil-containing inks. Recently the German BfR published a recommendation with maximum levels of mineral oil in food. For MOSH (C10 - C16) a guidance value of 12 ppm was set, for MOSH (C16 - C24) a value of 0.6 ppm was specified. A guidance value for MOAH has not been fixed yet.

Controversely in plant-derived food like tea, herbal infusions and spices endogenous n-alkanes are found in quantities up to 300 ppm. These primarily odd-numbered n-alkanes comprise carbon numbers between C21 and C39 and are localized in epicuticular waxes. Thus the quantification of MOSH compounds at the low levels is highly disturbed in such kind of commodities.

The aim of this project was the development of a modular method for the MOSH/MOAH analysis in all kinds of food and paper-based packaging material displaying a high sensitivity and selectivity for mineral oil hydrocarbons without disturbance by endogenous coextractives.

Experimental

The methodological steps are as follows: after addition of the respective internal standards and marker compounds the mineral oil hydrocarbons were extracted by hexane and hexane/ethanol, respectively.

The separation of the MOSH and MOAH fraction was carried out manually on cartridges with highly activated silica gel. Olefin-containing sample extracts were submitted to an epoxidation step. Interfering endogenous n-alkanes were removed by a basic alumina column cleanup step. A scheme of the analytical methodology is depicted in Figure 1.

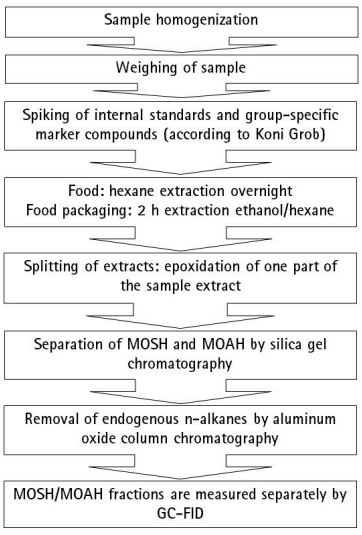

Figure 1: Flow chart of the analysis of MOSH/MOAH in food and paper-based packaging materials.

Table 1: Validation data for the determination of MOSH/MOAH according to DIN ISO 32645

	Food (black tea)			Paper-based packaging materials		
Compound	LOQ [ppm]	CV [%]	Recovery [%]	LOQ [ppm]	CV [%]	Recovery [%]
MOSH C10 - C16	0.12	3.4	92	1.03	3.9	95
MOSH C16 - C24	0.09	3.2	98	0.87	2.4	100
MOAH < C24	0.10	2.5	97	0.79	4.4	102

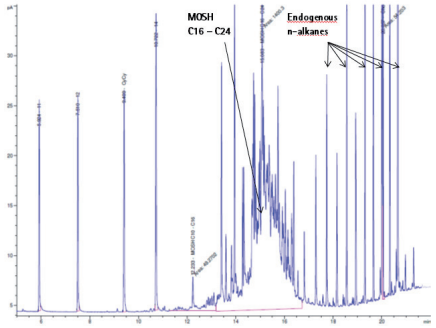

Figure 2: MOSH fraction of a black tea sample, contaminated with 2.3 ppm MOSH (C10 – C16) and 25.6 ppm MOSH (C16 – C24). An endogenous spectrum of odd-numbered n-alkanes (C21 – C35) is disturbing the determination of MOSH compounds.

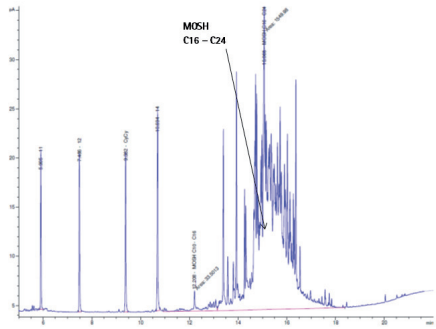

Figure 3: MOSH fraction of a black tea sample after an ALOX column cleanup step. Endogenous odd-numbered n-alkanes (C21 – C35) are completely removed. The isoalkanes of the MOSH fraction are not affected by this cleanup step.

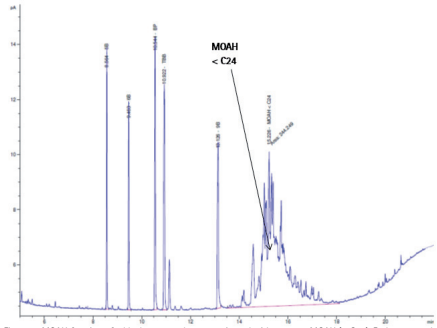

Figure 4: MOAH fraction of a black tea sample, contaminated with 8.9 ppm MOAH (< C24). Endogenous olefinic compounds like terpenoid substances and sterines are completely removed by an epoxidation reaction prior to silica gel column cleanup.

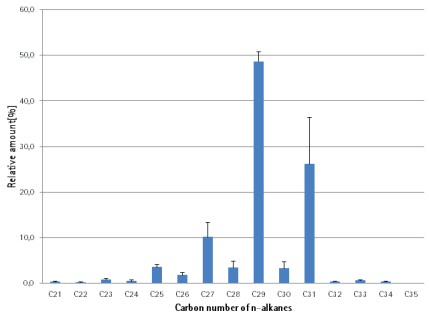

Figure 5: Relative distribution of n-alkanes in black tea samples.

- Endogenous n-alkanes severely interfere the determination of MOSH contamination in plant commodities like tea, herbal infusions and spices.
- A cleanup step by highly activated aluminum oxide selectively removes n-alkanes of carbon numbers > C20 without affecting iso-alkanes predominant in MOSH.
- This additional cleanup step allows the determination of MOSH at sub-ppm levels also in complex plant commodities.
- The amount of MOSH in food samples is not significantly affected by this procedure.