Evaluation of Epoxidation Methods for MOAH determination in Edible Oils

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Mineral oil hydrocarbons (MOH) in edible oils continue to be an ongoing topic due to the challenging analysis and the often still considerable contents found. Especially mineral oil aromatic hydrocarbons (MOAH) are of high interest since 3–7 ring MOAH may act as genotoxic carcinogens, as recently underlined again by the European Food Safety Authority (EFSA).[1] To prevent over estimation of MOAH due to biogenic interferences, epoxidation prior to analysis was introduced. Meta-Chlorperbenzoic acid (mCPBA) is the current state of the art epoxidizing agent, manifested in the initial DIN EN 16995 and the new DGF C-VI 22 (20) for improved comparability at lower MOH levels.[2] However a novel approach by Nestola[3] utilizing in situ generated performic acid (PFA) is of great interest, as it seems to show superior performance over mCPBA in removal of biogenic interferences. More Data needs to be generated to compare those methods as already mentioned in the annex of the future ISO standard method. Hence, we systematically evaluate mCPBA and PFA epoxidation strategies in cooperation with routine laboratories to improve the actual DGF standard method.

For a suitable comparison a manual workflow of the PFA epoxidation needed to be established and is now presented for the first time. Sensitivity similar to Nestola's automated PFA epoxidation workflow[3] was achieved without any additional enrichment step and a reduced injection volume of only 90 µL into the LC-GC-FID. Since PFA epoxidation reportedly removes considerable amounts of internal standards, recovery can be an issue. Including the partial and/or complete removal of solvent, the within-laboratory recovery of 80–110% suggested by the second Edition of the JRC Guidance on sampling, analysis and data reporting for the monitoring of MOH in food and food contact materials can be challenging. Especially the PFA epoxidation in chloroform showed substantial losses, besides its superior performance in removal of biogenic interferences. Experiments on the recovery of internal standards will be highlighted and discussed with alternative solvent removal strategies to facilitate the best recovery possible. Additionally, the advantages and disadvantages of a clean-up (silica column) prior to epoxidation are tested in terms of overall performance and system longevity. Benefits using a clean-up column were already observed for challenging samples because of the prevention of the formation of precipates in the final solution. Further evaluation of the LC-GC-FID results are performed via GCxGC-MS to identify and observe the discrimination of critical substance classes e. g. PAH and 3–7 ring MOAH.

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References:

[1] EFSA, Update of the risk assessment of mineral oil hydrocarbons (MOH) in food (Draft for public consultation) (2023).
[2] C. Albert, H.-U. Humpf, L. Brühl, *J. Agric. Food Chem*. (2022), **70,** 33.
[3] M. Nestola, *J. Chromatogr. A* (2022), **1682,** 4634526.