FEDIOL
The EU Oil & Proteinmeal Industry

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FEDIOL recommendations
1) on the analytical methods to be used for the analysis of mineral oil hydrocarbons in vegetable oils and fats
2) as regards reporting of the results for such analyses

The vegetable oil and fat sector is highly concerned about analytical methodologies for the determination of mineral oil hydrocarbons (MOH) in vegetable oils and fats. The reliability of the analytical methods is indeed paramount when detecting the possible contamination of its products.

For vegetable oils and fats, **standardized methods**[1] have been developed to analyze mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH). Such standardized methods (which have been evaluated on an inter-laboratory basis and which allow comparison of results between different laboratories) should be favoured today for vegetable oils and fats. Other methods – not necessarily developed for vegetable oil and fat matrices – lead to further uncertainty in data reproducibility. Since the publication of these standards, much progress has been made in mineral oil analysis and methods have been optimized by laboratories.

However, as demonstrated by recent inter-laboratory tests, there is still a **high variability of results** between laboratories, due to a number of analytical challenges (presence of natural or synthetic compounds co-eluting with mineral hydrocarbons, complex processing of results…). While various attempts and promises in achieving very low limits of quantification (LOQ) are made, it should be kept in mind that the lower the quantification limit is, the higher the uncertainty.

The **JRC guidance** for sampling, analysis and data reporting[2] published in February 2019 provides interesting background information and recommendations for the analysis of MOH in food and the reporting of results. Those recommendations have been integrated into this document, along with **additional recommendations** specific to the analysis of vegetable oils and fats, which as recognized by the guidance, represent challenging matrices in terms of methodology and interpretation.

[1] **ISO 17780:2015** - *Animal and vegetable fats and oils. Determination of aliphatic hydrocarbons in vegetable oils* AND **EN 16995:2017** - *Vegetable oils and foodstuff on basis of vegetable oils. Determination of mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) with on-line HPLC-GC-FID analysis*.

[2] S. Bratinova, E. Hoekstra (Editors) *Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials*, Luxembourg: Publications Office of the European Union, 2019 ISBN 978-92-76-00172- 0, doi:10.2760/208879, JRC115694

1. **FEDIOL recommendations on the analytical methods to be used for the analysis of mineral oil hydrocarbons in vegetable oils and fats**

In this context, FEDIOL would like to make the following **recommendations** to laboratories performing MOH analyses for the vegetable oil & fat sector:

- Co-workers should be regularly trained.
- Analytical methods should be based on the **CEN or ISO standards**[1].
- Considering interferences observed in the analysis of mineral oil saturated hydrocarbons (MOSH) & mineral oil aromatic hydrocarbons (MOAH) in vegetable oils (due to the presence of some natural compounds), **additional purification and cleaning steps should be performed systematically**:
    - Additional clean-up, with activated silica or aluminium oxide: this allows to retain long chain natural n-alkanes (usually composed of odd-numbered C in the range C21-C33) which interfere with the quantification of MOSH.
    - Epoxidation: such treatment may be used to eliminate natural olefins (like squalene, sterene or carotenoid) which interfere with the quantification of MOAH. This should be performed systematically for all types of vegetable oils.
- Analytical methods to measure MOSH and MOAH in vegetable oils & fats should fulfill the below **minimum quality criteria**:
    - Usage of the best quality of solvents (especially n-hexane, e.g. "n-hexane for pesticide residue analysis"). When changing solvent quality or supplier, the results of the blank should be evaluated. When having higher blank values, a solvent cleaning procedure should be performed.
    - Heating out glassware for at least two hours, and heating out aluminum oxide and silica gel for 12 hours, at 400°C.
    - Clean LC-GC-System (regular maintenance, GC- and HPLC-column changes, clean HPLC syringe)
    - No direct contact of the extracts with plastics from the vial caps: usage of aluminum foil as barrier between the extract and the cap in order to minimize POSH contamination.
    - Analysis of a blank-sample per analytical serie to monitor if the chemicals and reagents are not contaminated with mineral oil or similar compounds. The blank value should be free of interfering peaks.
    - Daily analysis of a reference material or control sample (monitor several alkane ranges) and putting the values in quality control charts → Stop of analysis when QC-charts limits are exceeded → Root-cause-analysis.
    - Monthly control of alkane ranges retention times using an alkane mix.
    - Automated data transfer of the values into a LIMS (Laboratory Information Management System) in order to reduce transcription errors.
    - Identification and interpretation of MOH quantification under all circumstances should be considered with much caution.
- The analytical methods should aim to fulfill **performance requirements** set by the JRC guidance[2] for the category "Fat/oils".

- Laboratories should regularly participate in professional **proficiency tests**[3] (at least once per year). The need for confidence in laboratory performance is essential for FEDIOL companies. Such confidence can be demonstrated through laboratory participation in proficiency testing schemes. Long term participation and evaluation via proficiency testing usually result in consistent and sustained improvements in laboratory performance.
- GCXGC-ToF-MS is a useful instrument for the characterization of the saturated or aromatic hydrocarbon fractions which may support a more effective root cause analysis with regard to the mineral oil presence. These instruments will play an increasingly important role in the identification of mineral oil input pathways.

2. FEDIOL recommendations as regards reporting of the results for such analyses

Results should be reported as recommended in the JRC guidance[2].

Other important elements to be reported

When analyses show a result above the limit of quantification, **chromatograms** (including chromatogram of the blank) should be made available to FEDIOL members *upon request* (chromatograms give complementary information on the type of mineral oil hydrocarbons at stake and on their possible origin).

Information on the performance of the analytical method is particularly important when analysing results. For that reason, analysis results on MOH presence in vegetable oils and fats provided to FEDIOL members should notably include the following elements:

- Limit of quantification (LOQ) of the analytical method for each type of hydrocarbons (saturated, aromatic).
- Expanded measurement uncertainty (U).
- *Remark*: limit of detection (LOD) of the analytical method for each type of hydrocarbons (saturated, aromatic) may be provided *upon request*

For traceability purpose, the following information should also be reported:

- Internal reference name given to the analytical method by the laboratory (including its version).
- Indication on whether the laboratory is accredited (ISO 17025) for the analytical method used.

[3] Such proficiency tests should be performed on vegetable oil matrices (edible oils/ fats).