Purity Criteria on Food Additives

Bernd Haber, BASF SE

BfR Conference „Spotlight: Food Additives“.
Berlin, 27.11.2024

Agenda

1) Official methods
2) Development of specification requirement
3) MOAHs in food additives
4) Concluding remarks

Agenda

1) **Official methods**

2) Development of specification requirement

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Official methods to test food additives specifications are missing in the EU.

Commission Regulation (EU) No. 231/2012 laying down specifications for food additives listed in Annexes II and III to Regulation (EC) No 1333/2008:

Recital

(3) It is necessary to take into account the specifications and **analytical techniques as set out in the Codex Alimentarius drafted by the Joint FAO/WHO Expert Committee on Food Additives** (hereafter JECFA).

<u>But:</u>

What happens if EU Specifications go beyond JECFA requirements?

EU specifications often go beyond JECFA specifications

E 471 MONO- AND DIGLYCERIDES OF FATTY ACIDS
Reg. (EU) 231/2012

Assay	Content of mono- and di-esters: not less than 70 %
	Content of erucic acid, including erucic acid bound in the mono/diglyceride:
	Not more than 0,2 % (only if added to food for infants and young children)
	Not more than 0,5 % (for all uses except for foods intended for infants and young children)
Description	The product varies from a pale yellow to pale brown oily liquid to a white or slightly off-white hard waxy solid. The solids may be in the form of flakes, powders or small beads.
Identification	
Infrared absorption spectrum	Characteristic of a partial fatty acid ester of a polyol
Test for glycerol	Passes test
Test for fatty acids	Passes test
Solubility	Insoluble in water, soluble in ethanol and toluene at 50 °C
Purity	
Water content	Not more than 2 % (Karl Fischer method)
Acid value	Not more than 6
Free glycerol	Not more than 7 %
Polyglycerols	Not more than 4 % diglycerol and not more than 1 % higher polyglycerols both based on total glycerol content
Arsenic	Not more than 0,1 mg/kg
Lead	Not more than 0,1 mg/kg
Mercury	Not more than 0,1 mg/kg
Cadmium	Not more than 0,1 mg/kg
Sum of 3-monochloropropanediol (3-MCPD) and 3-MCPD fatty acid esters, expressed as 3-MCPD	Not more than 0,75 mg/kg (only if added to food for infants and young children)
	Not more than 2,5 mg/kg (for all uses except for foods intended for infants and young children)
Glycidyl esters of fatty acids, expressed as glycidol	From 30 July 2023 until 30 January 2024, not more than 5 mg/kg if added to food for infants and young children) and not more than 10 mg/kg for all other uses.
	From 30 January 2024, not more than 5 mg/kg for all uses.
Total glycerol	Not less than 16 % and not more than 33 %
Sulphated ash	Not more than 0,5 % determined at 800 ± 25 °C
Soap	—

INS 471 MONO- AND DIGLYCERIDES
JECFA 2000 (55th JECFA meeting)

Assay: Alpha-monoglycerides (Vol. 4): min. 30%

Identification:
- Solubility (Vol. 4)
- IR absorption
- Test for fatty acids (Vol. 4)
- Test for glycerol (Vol. 4)

Purity:
- Water (Vol. 4) (Karl-Fischer method)
- Acid value (Vol. 4)
- Free glycerol (Vol. 4)
- Soap
- Lead (Vol. 4)

New parameters

More details on composition

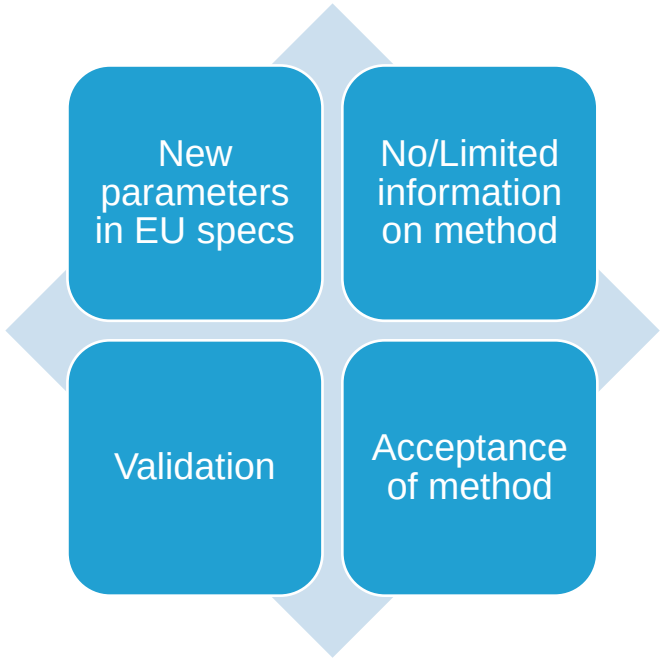
New contaminants

Lower max. levels

Differentiation of general food and infant food use

The EU is front-runner in updating the specifications of food additives and could be the global reference for official methods in the future.

EU Re-evaluation program is creating a broad bunch of changes in specifications of food additives:



- Availability of JECFA methods?
- Actuality of JECFA methods?

- Is the use of JECFA methods binding?
- Alternative methods from pharmacopeial standards or in-house methods?

FAO, Rome 2006

Proposal:

EU Compendium of Official methods

Reference substances

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Regulation (EU) 231/2012 stipulated already more detailed information in food additives specifications.

Commission Regulation (EU) No. 231/2012 laying down specifications for food additives listed in Annexes II and III to Regulation (EC) No 1333/2008:

Recitals

(3) It is necessary to take into account the specifications and analytical techniques as set out in the Codex Alimentarius drafted by the Joint FAO/WHO Expert Committee on Food Additives (hereafter JECFA).

(7) **Detailed information on the production process and starting materials of a food additive** should be included in the specifications to facilitate any future decision pursuant to Article 12 of Regulation (EC) No 1333/2008.

(8) Specifications should not make reference to organoleptic tests related to the taste as it cannot be expected by the control authorities to take the risk to taste a chemical substance.

(10) Specifications should not make reference to the general parameter ‘Heavy metals’ as this parameter does not relate with toxicity, but rather with a generic analytical method. **Parameters related to individual heavy metals are toxicity related and are included in the specifications**.

Evolution of a food additives specification, example sorbic acid (E 200)

Dir 65/66/EEC

	E 200 Sorbic acid
Appearance	White crystalline powder showing no change in colour after heating for 90 mins at 105 °C
Melting range	133–135 °C, after vacuum drying for 4 hours in a sulphuric acid desiccator
Content	Not less than 99%, after vacuum drying for 4 hours in a sulphuric acid desiccator
Volatile substances	Not more than 3%, determined by drying for 24 hours in a sulphuric acid desiccator
Sulphated ash	Not more than 0·2%
Aldehydes	Not more than 0·1% calculated as formaldehyde

Assay
plus 1 identification method
plus 3 purity criteria

Reg. 2024/2597

E 200 SORBIC ACID

Synonyms	
Definition	
Einecs	203-768-7
Chemical name	Sorbic acid; *trans*, *trans*-2,4-Hexadienoic acid
Chemical formula	$C_6H_8O_2$
Molecular weight	112.12
Assay	Content not less than 99 % on the anhydrous basis
Description	Colourless needles or white free flowing crystalline powder, having a slight characteristic odour and showing no change in colour after heating for 90 minutes at 105 °C;
Identification	
Melting range	Between 133 °C and 135 °C, after vacuum drying for four hours in a sulphuric acid desiccator
Spectrometry	A propan-2-ol solution (1 in 4 000 000) shows absorbance maximum at 254 ± 2 nm
Test for double bonds	Passes test
Solubility	Slightly soluble in water, soluble in ethanol.
Purity	
Water content	Not more than 0,5 % (Karl Fischer method)
Sulphated ash	Not more than 0,2 %
Aldehydes	Not more than 0,1 % (as formaldehyde)
Arsenic	Not more than 0,1 mg/kg
Lead	Not more than 0,1 mg/kg
Mercury	Not more than 0,01 mg/kg
Zinc	Not more than 0,1 mg/kg';

Assay
plus 4 identification methods
plus 7 purity criteria
but lowered heavy metal levels

Descriptions in Specifications are very detailed and limit the flexibility of manufacturers.

Example: Draft Rule on Steviol glycosides from fermentation

Definition:
Rebaudioside M from fermentation produced by Yarrowia lipolytica consist of a mixture of steviol glycosides composed of rebaudioside M as the main component, with some rebaudioside D, and smaller amounts of rebaudioside A and rebaudioside B. The manufacturing process comprises two main phases. The first phase involves fermentation of a simple sugar source by a non-toxigenic non-pathogenic strain of Yarrowia lipolytica that has been genetically modified with heterologous genes to overexpress genes which are involved in the synthesis of steviol glycosides to result in the strain VRM (CBS 147477). Removal of biomass by solid-liquid separation and heat treatment is followed by concentration of the steviol glycosides. The second phase involves purification by employing ion exchange chromatography, followed by cristallisation of the steviol glycosides from ethanol, resulting in a final product containing not less than 95% of rebaudiosides M, D, A and B. Viable cells and DNA of Yarrowia lipolytica VRM shall not be detected in the food additive.

Flexibility for producers

Changes in manufacturing process create a potential need for regulatory change.

Process innovations slowed down

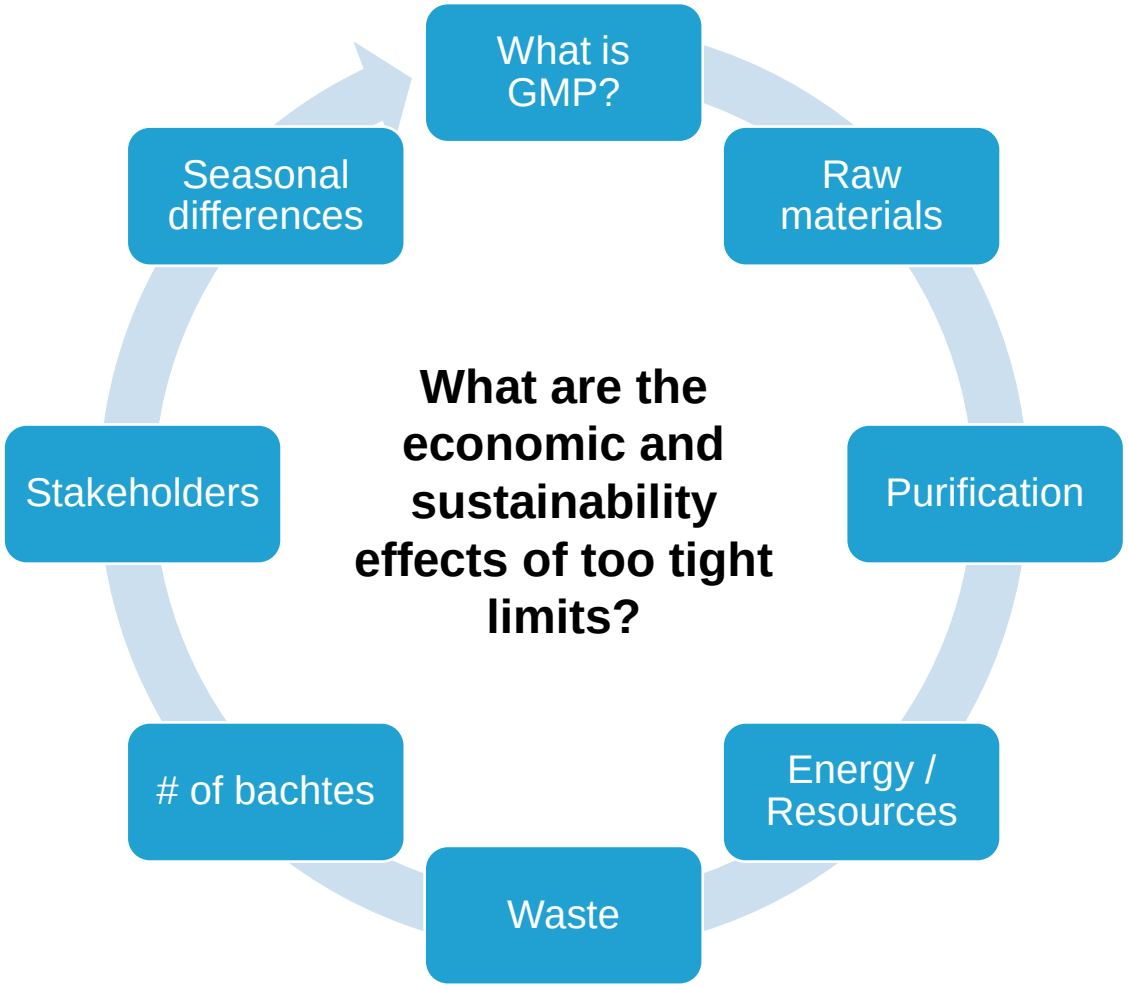
Does a remark in the definition trigger any testing? (e.g. viable cells, DNA)

New thresholds for heavy metals go beyond what is needed to ensure safe use of food additives.

- Heavy metals limits for many food additives in the lower ppm range so far (e.g. Pb 2 ppm)
- New heavy metal limits in specifications of new food additives or in revised specifications are 10-200-fold lower than in the past (e.g. Pb 0.01-0.4 ppm).
- Reasons:
  1. based on scientific assessment (EFSA)
  2. levels achievable by GMP
- Result: heavy metals limits in the range of IF/FOF and below, even for products not intended for IF/FoF.

	Draft amendment			SCoPAFF	
	Locust bean gum (E410)	**Acacia gum (E414)**	**SAOS (E1450)**	**Rebau-dioside M (E960b(i))**	**IF/FoF powder Reg. 2023/915**
Arsenic (mg/kg)	0.1	0.1	0.05 (IF/FoF) / 0.1 (food)	0.01	**0.02**
Lead (mg/kg)	0.4	0.05	0.03 (IF/FoF) / 0.2 (food)	0.01	**0.02**
Mercury (mg/kg)	0.1	0.05	0.05	0.05	-
Cadmium (mg/kg)	0.1	0.05	0.01 (IF/FoF) / 0.1 (food)	0.01	**0.01**
Aluminium (mg/kg)	-	100 (IF/FoF) / 120 (food)	-		-

New heavy metal limits for food additives could create high burden for industry to meet the new requirements.



- Continous improvement is important.
- Still there are many factors affecting product quality.
- Limits should be risk-based and not to the lowest achievable level.
- Little contribution of food additives to the overall intake of heavy metals (esp. Annex III uses).

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MOAH in Food Additives

EFSA 2023:

„Technical specifications of white mineral oils and waxes used as food additives and food packaging materials should be updated, with detailed information about the MOAH content and composition. “

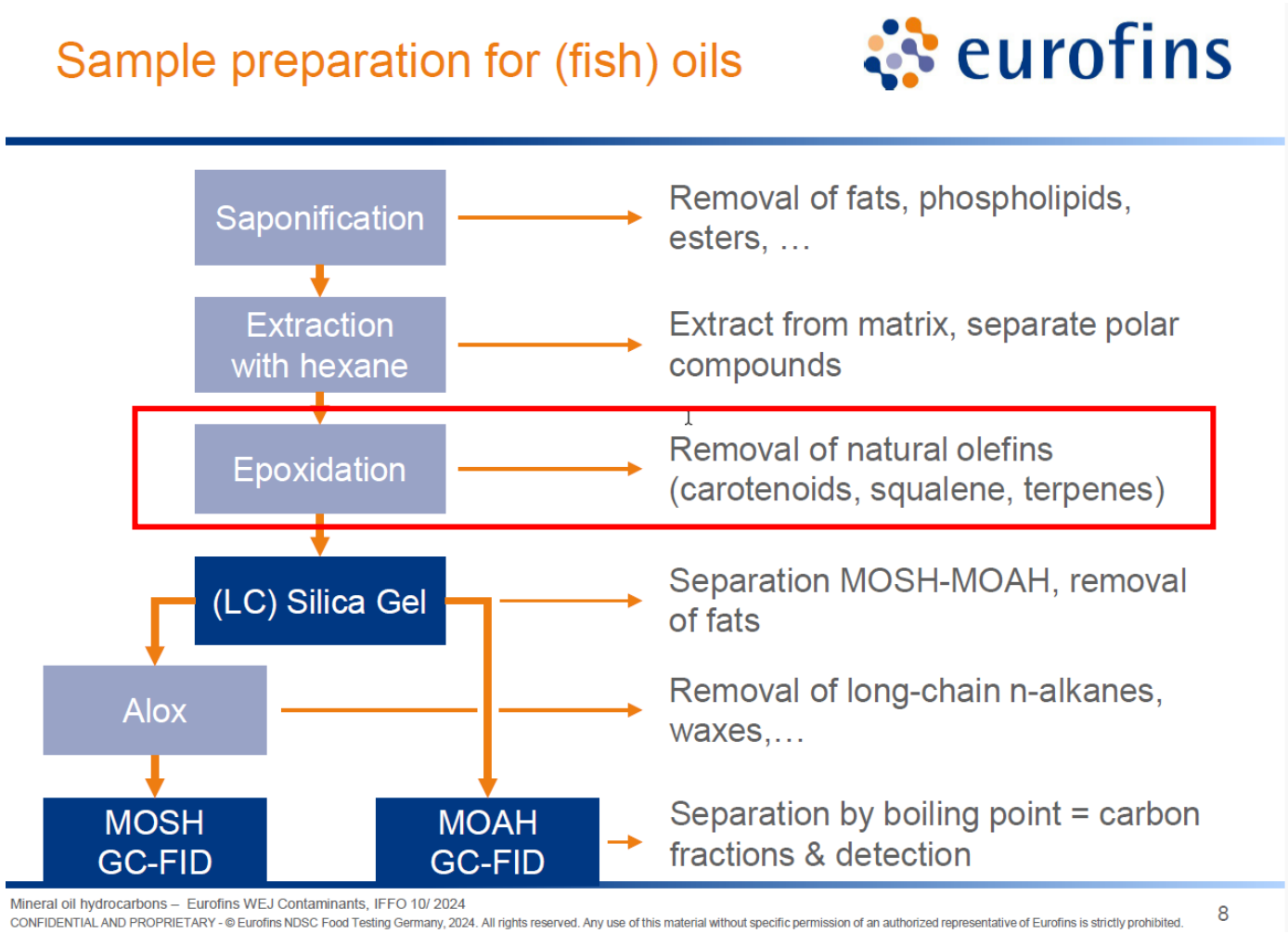
DG SANTÉ proposal / targeted stakeholder consultation (12.2.2024)

- Maximum limit of 2.0 mg/kg for MOAH for all food additives
- Horizontal provision in Reg. (EU) 231/2012 comparable to EtO
- Preferred approach, most pragmatic way.

Discussions on MOSH/MOAH have focussed on foods so far, less on food additives.

Analytical database still limited.

Standard analytical procedure (example fish oils)



- Very similar procedure used for fat-soluble food additives
- Removal of natural olefins by epoxidation is an important step in the sample preparation

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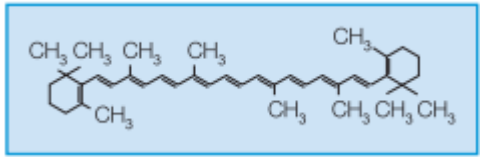
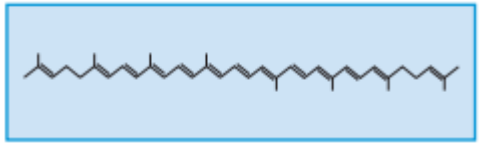
Food additives can be rich in „olefins“. Example: carotenoids

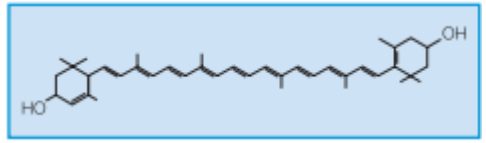
Carotenoids



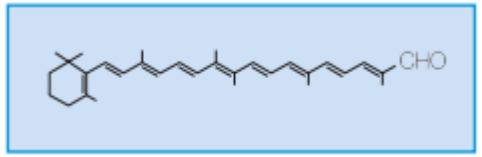
Beta-Carotene / E 160 a (i)



Lycopene / E 160 d



Beta-Apo-8‘-Carotenal / E 160 e



Lutein / E 161 b

E 160 a (i) BETA-CAROTENE

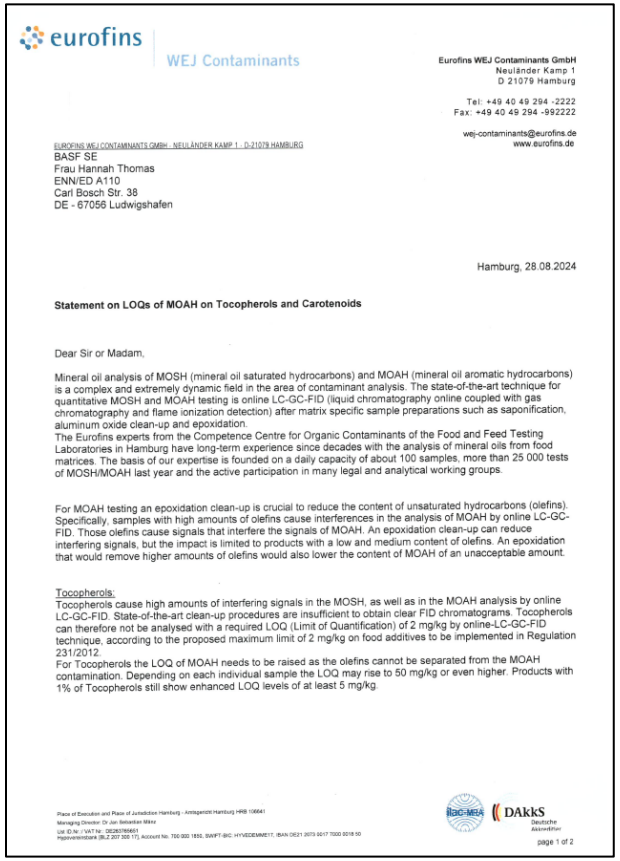
Synonyms	CI Food Orange 5
Definition	These specifications apply predominantly to all trans isomer of beta-carotene together with minor amounts of other carotenoids. Diluted and stabilised preparations may have different trans-cis isomer ratios.
Colour Index No	40800
Einecs	230-636-6
Chemical name	Beta-carotene; beta, beta-carotene
Chemical formula	$C_{40}H_{56}$
Molecular weight	536,88
Assay	Not less than 96 % total colouring matters (expressed as beta-carotene) $E^{1\%}_{1cm}$ 2 500 at approximately by 440 nm to 457 nm in cyclohexane

Olefinic structures are very common in food colours or colouring extracts and determine their coloring principle.

Limitations of state-of-the-art MOAH analysis on carotenoids

Eurofins Statement (28.8.2024):

- Carotenoids cause high amounts of interfering signals
- State-of-the-art clean-up procedures are limited to low and medium amounts of carotenoids
- LOQ of 2 mg/kg cannot be accomplished
- LOQ for pure carotenoids might be rised to 50 mg/kg or even higher
- LOQ for products with 1-25% of carotenoids still enhanced to approx. 3 mg/kg.



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Hamburg, 28.08.2024

Statement on LOQs of MOAH on Tocopherols and Carotenoids

Dear Sir or Madam,

Mineral oil analysis of MOSH (mineral oil saturated hydrocarbons) and MOAH (mineral oil aromatic hydrocarbons) is a complex and extremely dynamic field in the area of contaminant analysis. The state-of-the-art technique for quantitative MOSH and MOAH testing is online LC-GC-FID (liquid chromatography online coupled with gas chromatography and flame ionization detection) after matrix specific sample preparations such as saponification, aluminum oxide clean-up and epoxidation.
The Eurofins experts from the Competence Centre for Organic Contaminants of the Food and Feed Testing Laboratories in Hamburg have long-term experience since decades with the analysis of mineral oils from food matrices. The basis of our expertise is founded on a daily capacity of about 100 samples, more than 25 000 tests of MOSH/MOAH last year and the active participation in many legal and analytical working groups.

For MOAH testing an epoxidation clean-up is crucial to reduce the content of unsaturated hydrocarbons (olefins). Specifically, samples with high amounts of olefins cause interferences in the analysis of MOAH by online LC-GC-FID. Those olefins cause signals that interfere the signals of MOAH. An epoxidation clean-up can reduce interfering signals, but the impact is limited to products with a low and medium content of olefins. An epoxidation that would remove higher amounts of olefins would also lower the content of MOAH of an unacceptable amount.

Tocopherols:
Tocopherols cause high amounts of interfering signals in the MOSH, as well as in the MOAH analysis by online LC-GC-FID. State-of-the-art clean-up procedures are insufficient to obtain clear FID chromatograms. Tocopherols can therefore not be analysed with a required LOQ (Limit of Quantification) of 2 mg/kg by online-LC-GC-FID technique, according to the proposed maximum limit of 2 mg/kg on food additives to be implemented in Regulation 231/2012.
For Tocopherols the LOQ of MOAH needs to be raised as the olefins cannot be separated from the MOAH contamination. Depending on each individual sample the LOQ may rise to 50 mg/kg or even higher. Products with 1% of Tocopherols still show enhanced LOQ levels of at least 5 mg/kg.

Place of Execution and Place of Jurisdiction Hamburg - Amtsgericht Hamburg HRB 106641
Managing Director: Dr Jan Sebastian Mähr
VAT-ID-Nr.: / VAT-Nr.: DE265786651
Hypovereinsbank (BLZ 207 300 17), Account No. 700 000 1850, SWIFT-BIC: HYVEDEMMXXX, IBAN: DE21 2073 0017 7000 0018 50

DAkkS Deutsche Akkreditierungsstelle

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Similar case:

DL-alpha-tocopherol (E 307)

- LOQ (E 307) >> 50 mg/kg
- LOQ (1% Toc.) > 5 mg/kg

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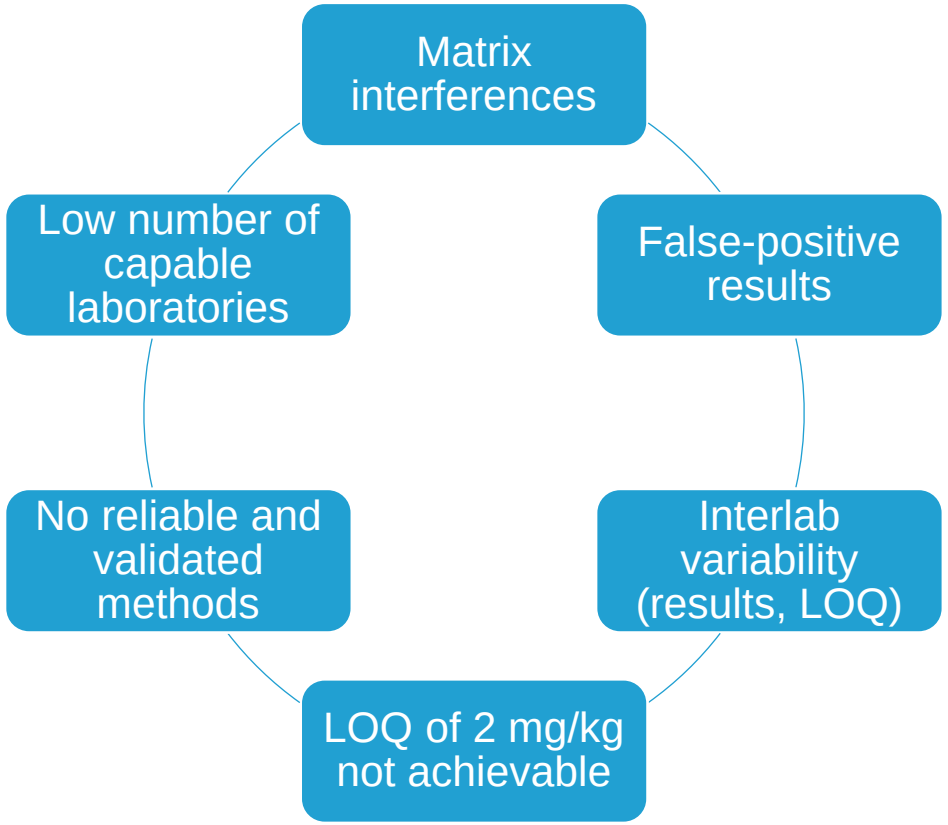
Current analytical methods are not suitable to provide robust data on MOAH for food additives.



MOAH maximum levels for food additives should:

- be risk-based and scientifically justified
- consider the contribution of the food additive to overall MOAH intake
- be built on validated and reliable methods with individual LOQ‘s

Harmonized maximum levels for MOAH for all food additives would be disproportionate and not sustainable.

EFEMA‘s position on the introduction of a horizontal value for MOAH on food additives (based on a letter to DG Santé in July 2025).

- EFSA’s Update of the risk assessment of mineral oil hydrocarbons in food recommends the establishment of maximum limits for certain food additives. **Amending the specifications for all food additives** is in our view **disproportionate**.
- Measuring MOAH in **certain matrices,** like food emulsifiers, can be challenging. This was confirmed by renowned laboratories, who **often propose limits of quantification (LOQs) higher than 2 mg/kg**. This observation is backed-up by evidence and statements from renowned laboratories.
- Considering that the Joint Research Centre (JRC) issued a Guidance documents on the sampling or analysis of MOHs in food and food contact materials, we are of the view that the **development** by JRC or by the European Union Reference Laboratory for Processing Contaminants (EURL-PC) **of a reliable and validated method for food emulsifiers** shall also be considered.
- Even though uncertainties remain, the main source of contamination is to be found in the **raw materials**. As far as emulsifiers are concerned, this is notably case of the **oils sourced outside the EU**, more particularly tropical oils. It seems that MOAH limits above 2 mg/kg could be set for these tropical oils and we would suggest that, should limits be established for food emulsifiers too, the latter **should not be below the limits applicable to these oils**.
- Finally, as DG SANTE organised at the beginning of 2024 a stakeholder forum on mineral oil hydrocarbons (MOHs), where various food sectors could present the outcome of their research and their challenges, we also suggest that a similar forum is also organised for relevant food additives.

A horizontal maximum level for MOAH of 2 mg/kg for all food additives is not justified and also not implementable.

- Food additives cannot be compared to foods and handled in the same manner (different matrices).
- Food additives are concentrated forms of a chemical substance or a group of substances from different origin.
- The potential occurence of MOAH in food additives might be related to an individual manufacturing process and raw materials used and not a general problem of all food additives.
- Specific chemical structures might interfere with available MOAH analytical methods that the proposed LOQ cannot be achieved.
- For many food additives groups analytical methods have to be developed or adapted and individual LOQ have to be established.

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Concluding remarks:

- An EU Compendium of Official Methods for analytical testing of food additives would be desirable, both for industry and for enforcement of the specification requirement.
- Specification requirements on food additives have severely developed in the last years. The changes should be risk-based, reasonable and implementable in routine analysis.
- Introduction of a horizontal maximum level for MOAH for all food additives goes beyond the need and is currently not possible. For several food additive groups the LOQ of 2 mg/kg is not achievable and individual developments on the suitable methods and LOQ‘s are needed.

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