European Commission JRC FCM activities

The European Commission's science and knowledge service

Joint Research Centre

European Commission

Mineral oil (MOSH, MOAH) in food & FCM

COMMISSION RECOMMENDATION (EU) 2017/84

of 16 January 2017

on the monitoring of mineral oil hydrocarbons in food and in materials and articles intended to come into contact with food

- **"Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials" is published**

(S. Bratinova, E. Hoekstra (Editors), 2019, ISBN 978-92-76-00172-0, doi:10.2760/208879; https://ec.europa.eu/jrc/en/eurl/food-contact-materials/technical-guidelines)

- **Hands-on training in Ispra**

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SCOPE

This guidance aims to harmonisation of

- Sampling
- Analytical approach
  - Quantification by LC followed by GC-FID
  - Verification by GC-MS and or GCxGC-FID/MS
- Performance criteria
- Reporting

No standard operating procedure

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DESCRIPTION of mineral oil hydrocarbons (MOH)

MOH – hydrocarbons from crude mineral oil or produced from coal, natural gas or biomass (EFSA)

MOSH - alkanes, branched and unbranched (paraffins); cycloalkanes,

alkylated and non-alkylated, mono-, di- and higher ring systems (naphthenes)

MOAH - aromatics (mono-, di- and higher ring systems), mainly alkyl-substituted

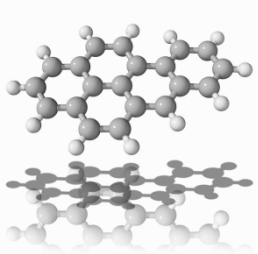
MOH does not include the following hydrocarbons:

- naturally occurring in food: such as *n*-alkanes of odd numbered carbons (from C21 to C35) or natural olefins of terpenic origin (as squalene, steranes or carotenoids)

- such as POH potentially migrating from plastic packaging or synthetic *iso*-paraffins with short and long side chains used e.g. in synthetic lubricants and adhesives

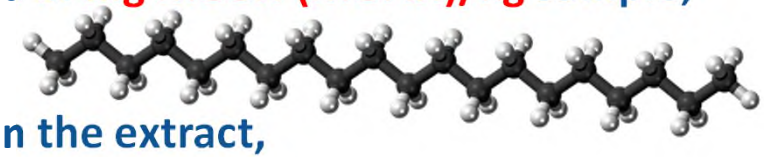
Biedermann M., Grob K. (2012). Journal of Chrom. A 1255, 56
Grob K., Biedermann M., Caramaschi A., Pacciarelli B. (1991) J. High Resolut. Chromatogr. 14, 33
Biedermann M., Grob K. (2012) Journal of Chrom. A 1255, 76

DEFINITION OF THE MEASURANDS



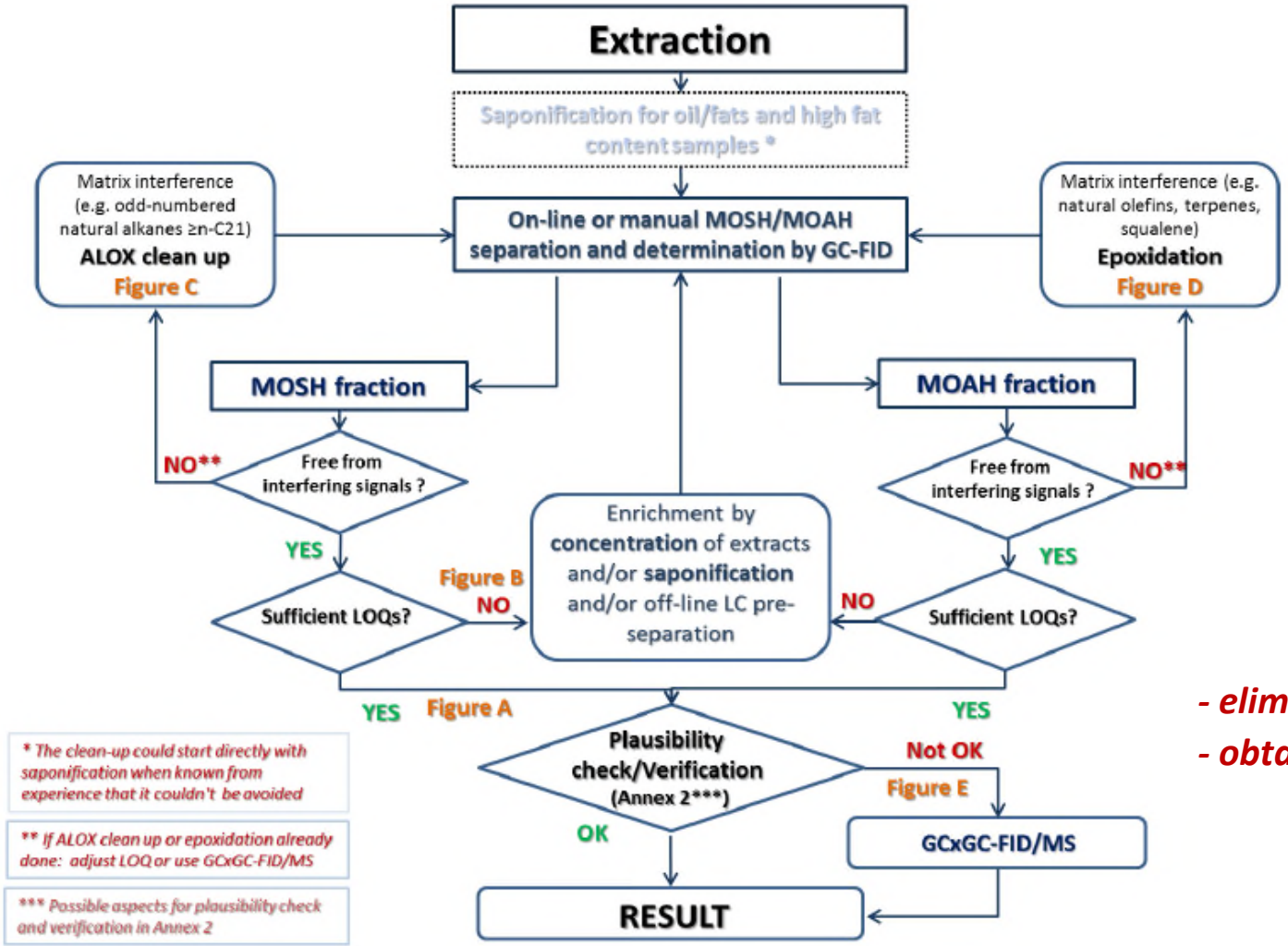
The total MOSH (MOAH) measurand

- total **mass fraction** of MOSH (MOAH), expressed in **mg MOSH (MOAH)/kg** sample,
- after **separation MOSH from MOAH**
- and after **removal** of all possible **interferences** in the extract,



- as quantified by **integration of the whole signal** interval in the GC-FID chromatogram between the retention times of the peak start of ***n*-C10** and the peak end of ***n*-C50,**
- after **subtracting** the identified sharp peaks **not belonging** to MOSH (MOAH)
- and using cyclohexylcyclohexane (MOSH) and 1- or 2-methylnaphthalene (MOAH) as **internal standards** (other hydrocarbon or technique acceptable, if the equivalent result is demonstrated)

Decision tree on the use of auxiliary methods



- *elimination interferences*
- *obtaining requested sensitivity*

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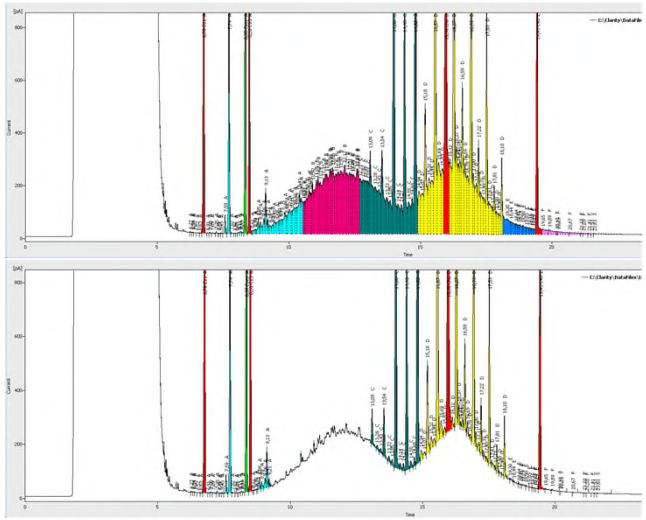
QUANTIFICATION

- **Total MOSH/MOAH content (C_{10}-C_{50}) and**
- **sub-fractions of MOSH and MOAH (C-fractions) defined by the position (R_t) of the elution signals of *n*-alkanes from the GC column.**

The following MOSH and MOAH C-fractions are defined:

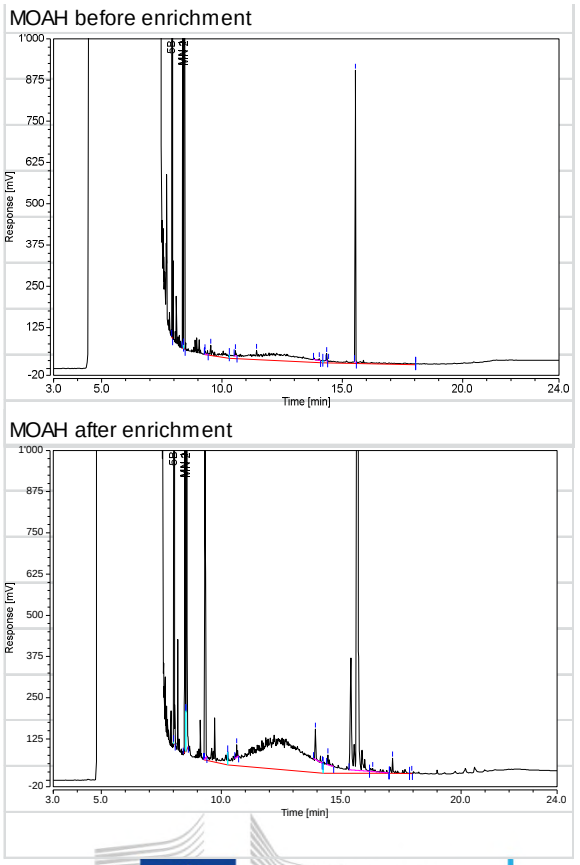


MOSH:	MOAH:
total MOSH	Total MOAH
MOSH ≥n-C_{10} to ≤n-C_{16}	MOAH ≥n-C_{10} to ≤n-C_{16}
MOSH >n-C_{16} to ≤n-C_{20}	MOAH >n-C_{16} to ≤n-C_{25}
MOSH >n-C_{20} to ≤n-C_{25}	MOAH >n-C_{25} to ≤n-C_{35}
MOSH >n-C_{25} to ≤n-C_{35}	MOAH >n-C_{35} to ≤n-C_{50}
MOSH >n-C_{35} to ≤n-C_{40}	
MOSH >n-C_{40} to ≤n-C_{50}	

Performance requirements of the analytical methods

- maximum acceptable LOQ for each C-fraction (LOQ-max)
- target LOQ for each C-fraction (LOQ-t)
- acceptable ranges for recovery (R_{rec})
- intermediate precision (within-laboratory precision)

Categories	Associated foods #	LOQ - max [mg/kg]	LOQ -t [mg/kg]	R_{rec} [%]	interm,. precision [%]
Dry, low-fat content (< 4% fat/oil)	bread and rolls; breakfast cereals; grains for human consumption; pasta, products derived from cereals	0.5	0.1	80 - 110	15
Higher fat/oil content (> 4% fat/oil)	fine bakery ware; confectionery (incl. chocolate) and cocoa; fish meat, fish products (canned fish); oilseeds; pulses; sausages; tree nuts	1	0.2	70 - 120	20
Fat/oils	animal fat (e.g. butter); vegetable oils	2	0.5	70 - 120	20
Paper and Board	reporting only up to C_{35} (extraction optimised up to C_{35})	10	5	80 - 110	10



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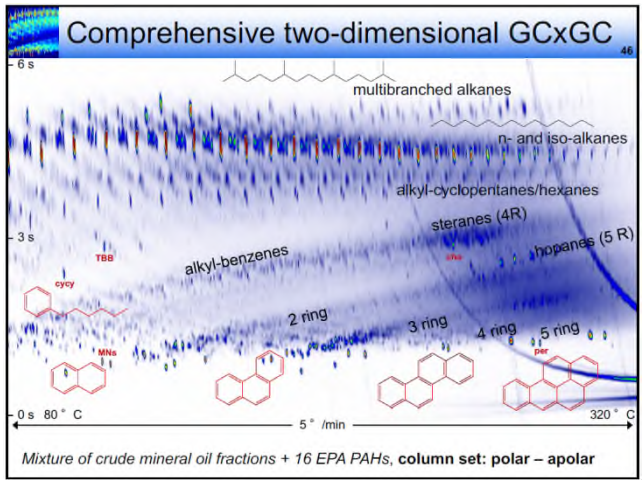
Further MOSH/MOAH CHARACTERISATION

Needed when:

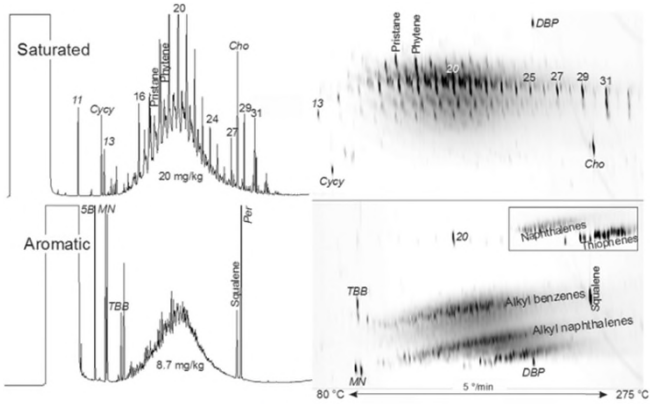
- **interferences suspected even after purification**

GC-MS

GCxGC-FID (MS)



Biedermann M.



Spack L. et al. Food Additives Contam (2017) 34(6), 1052–1071
Biedermann M., K. Grob, Journal of Chromatography A, 1375 (2015) 146–153.
Lommatzsch M. et all, Food Additives & Contaminants: Part A (2016), 33:3
Weber S. et al, A Short Review, 2018, Analytical Chemistry Insights Volume 13: 1–16

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REPORTING

- Information about food + packaging material if relevant
- Place and date of sampling
- End of the shelf life
- Mass fractions ± expanded measurement uncertainty (U) (k=2):
  - *MOSH: total, C_{10}-C_{16}, $C_{16}C_{20}$, C_{20}-C_{25}, C_{25}-C_{35}, C_{35}-C_{40}, C_{40}-C_{50}*
  - *MOAH: total, C_{10}-C_{16}, C_{16}-C_{25}, C_{25}-C_{35}, C_{35}-C_{50}*
  - *diisopropyl naphthalenes (DIPN), POSH, etc.*
- start, top and end of all the hump(s) in terms of GC retention of n-C_n
- Integrated chromatograms of MOSH and MOAH
- Sample preparation methods and deviations from prescribed
- Auxiliary methods used and deviations from prescribed
- Description of strategy to identify source(s)
- EFSA mandatory parameters

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Testing conditions for kitchenware – part 1: plastics

Example

Food Preparation Utensil for Cold/Ambient use (FPU/CA)	FPU/CA1	Utensils use in ambient temperature for short time: Rolling pin, Lattice cutter, Grater, Garlic press, Zester, Vegetable peeler, Apple peeler, Food scales, Apple corer, Apple cutter, Biscuit press, Cherry pitter, Egg separator, Fish scaler, Flour sifter, Herb chopper, Squeezer, Reamer, Mandolin, Wires, Meat tenderiser, Fruit baller, Nutmeg grater, Pastry blender, Mortar and pestle, Roller docker, Pasta cutter, Salad spinner, Julienne peeler, Avocado slicer, Ravioli maker, Vegetable cutter with container, Hamburger press, Coconut scraper, Empanadilla mould type, Meat grinder, Vegetable brush, Cake measuring tape, Cocktail shaker, Coffee measuring spoon

		Use				Sample prep			Test type				Food simulant							Specific Migration Conditions			S/V					Overall Migration conditions	Note
Main Class	Subclass	cold (< 20 °C)	Room Temperature	hot (> 40 °C)	storage (in months)	cut test specimen	intact article	part of it	actual use	article fill	migration cell	total immersion	food	A	B	C	D1	D2	E	time	Temp (°C)	label/instructions	Real	Real (infant/young)	6 (V < 0.5L)	6 (V > 10L)	6 impractical s/v		
	FPU/CA1	x	x			x	x			x		x	x	x	x			x		0.5 h	40		x		x	x	x	3	[1]

[1] OM0 (new): 0.5 h at 40 °C, proposal of futute ammendment of Reg. 10/2011
[2] cf. Table 2 of Annex V
[3] use (10d, 40°C) if equilibrium is reached [cf. Reg. 10/2011 Annex V, Chapter 2 § 2.1.4.e & Amendment 2016/1416]
[4] select test time and temperature according to worst foreseeable condition use (described in the instructions when availllable)
SM, OM: Specific migration, Overall Migration
s/v: surface-to-volume ratio to calculate final migration result

- **Labelling**
- **Intended specific foods**
- **Kitchen small appliances**

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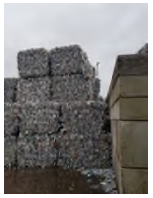
Guidance in draft/needed/identified

- Testing conditions for kitchenware articles in contact with foodstuffs – part 2: Metals
- Migration test design for food contact materials - compiling information from the EN 1186, EN 13130 and the old kitchenware guideline;
- Performance criteria and additional requirements for test methods development in the field of FCM within TF MAM (revision 2009)
- Series of technical guidance on compliance testing of plastic food contact materials:
  - Sampling; Testing when FCM already in contact with food; Verification of compliance testing; Screening; Reporting
- Revision of the MOSH/MOAH Guidance e.g. on procedure for estimation of LOQ
- Harmonisation of extraction techniques for determination of substance in plastic

Monitoring for recycling with focus on PET

Request from DG SANTE for improved understanding of:

- occurrence and sources of contamination from use e.g. acetaldehyde, limonene
- occurrence and sources of incidental contamination e.g. mineral oil
- Implementation of the existing recycling process restrictions such as 5 % limit of non-food post-consumer plastic in collected waste
- Understand the decontamination yield
- Understand the recycling of ABA layers

- Develop a system for collection industrial data before decontamination step and output
- contact with actors → questionnaire
  - Plastic Recyclers Europe
  - European Plastic Converters
  - visits
    - 
    - Circular Economy team JRC.B.5

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Test conditions for bakeware

Previous JRC work

- Comparison of migration test condition of 4 w/v % acetic acid for 24 h @ 22 °C with:
  - ✓ 0.5 w/v % citric acid for 2 h @ 70 °C,
  - ✓ boiling tomato sauce on hotplate 2 h and 6 h

Request from DG SANTE

- Cover the higher temperatures of bakeware
- Cover enamelled metal bakeware

Tests

- Kinetics with 4 w/v % acetic acid @ 22 °C up to 7 days
- Elevated temperature: 95 °C **X** ⇄ **?**
- Different acids as food simulant
- Acidified tomato sauce as food simulant → **Problems with background levels**

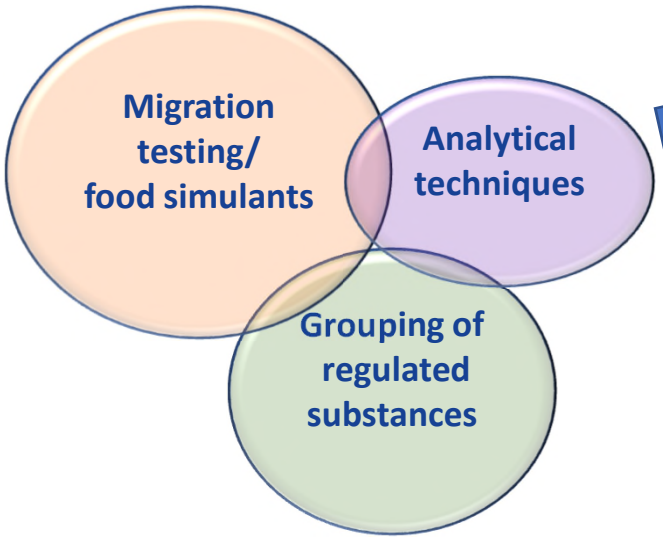
Database of analytical methods and calibrants

- Method often embedded in confidential part
- Violation of Article 20 2(c) of Regulation (EC) No 1935/2004
  - ✓ information related to analytical methods shall not be considered confidential
- Status up to Regulation 2019/1338:

	FCM substances	Calibrant	Method	Calibrant + method
no SML	451	268	48	26
SML=ND	31	28	20	19
SML	272	194	158	126
Group SML	149	103	50	42
SML + group SML	10	7	5	3
Total	893	586	271	210

Development of multi-analyte methods

- Scope: EU regulated substances
- Strategy of grouping e.g. based on chemical structure
- Modular approach
- Existing multi-analyte methods in NRLs



- Evaluation with harmonised performance criteria
- Agreed Operational Procedures.

Working groups → Method validation

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