European monitoring of mineral oil

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7th of November 2017

European Commission

Background mineral oil hydrocarbons (MOH)

Potential risk – EFSA (2012)

- **MOH are present in food from various sources – in unknown amounts**
- **Some MOH's are carcinogenic**
- **Some can cause other effects**

The actual occurrence is not clear

- **sources, amounts, exposure**
- **relative occurrence of specific groups**

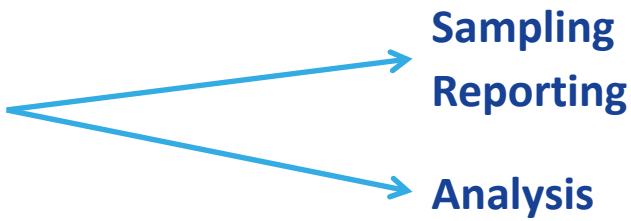
No clear RM approach

- **difficult to justify burden, action levels, etc.**

Monitoring Recommendation

Guidelines – work of Task Force

- Members are appointed by MSs: NRL, OCL
- Guidelines
  - ✓ Set definition of MOH, MOSH, MOAH, POSH
  - ✓ List of authorised MOH food additives and processing aids
  - ✓ Collection of chromatograms of MOH sources
- Getting an overview of MSs' planned monitoring activities
- Develop training for official control laboratories (OCL)
- Develop reference materials for proficiency test



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Recommendation (EU) 2017/84

- Member States should monitor the presence of MOH in food and FCM during 2017-2018.
  - ✓ Active involvement of stakeholders
- Following guidance of EURL
  - ✓ to ensure uniform application of this recommendation
  - ✓ to generate reliable and comparable results
- Food sampling in accordance with the provisions laid down in Commission Regulation (EC) No 333/2007
- Sampling should include a proportionate number of pre-packaged foods
  - ✓ focus on commodities that are closer to the end of the minimum date of durability
- The samples should be analysed as marketed

Recommendation (EU) 2017/84

Foods covered

- animal fat and vegetable oils
- bread and rolls, fine bakery ware
- breakfast cereals and products derived from cereals
- pasta
- grains for human consumption
- confectionery (including chocolate) and cocoa
- fish meat and fish products (canned fish)
- sausages
- ices and desserts
- oilseeds
- pulses, e.g. alfalfa, peas, beans, lentils, soybeans, peanuts
- tree nuts, e.g. almonds, cashews, chestnuts, hazelnuts, walnuts

Possible sources of MOH needs to be investigated

Recommendation (EU) 2017/84

Pre-packed food

- MOH should be determined both in the food and in the FCM if relevant
  - ✓ Particular attention should be paid to the differences between MOSH and MOAH
- Sources of MOH related to storage or processing, should be investigated if relevant
  - ✓ **Focus on relatively warm conditions**
- If MOH detected, collection of data on the FCM
  - ✓ e.g. type and composition of the packaging material, presence of functional barrier, shelf life of the packaged food
  - ✓ carry out further investigations in the establishments of the manufacturers, processors and distributors of food contact materials

Reporting to EFSA

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Guidelines – sampling - Regulation (EC) No 333/2007

Annex part A – Definitions

- **lot**
  - ✓ an identifiable quantity of food delivered at one time and determined to have common characteristics
    - Fish: size shall be comparable
- **sublot**
  - ✓ designated part of a large lot in order to apply the sampling method. Each sublot must be physically separated and identifiable
- **incremental sample**
  - ✓ a quantity of material taken from a single place in the (sub)lot
- **aggregate sample**
  - ✓ combined total of all the incremental samples taken from the (sub)lot
    - aggregate samples are representative of their (sub)lots
- **laboratory sample**
  - ✓ a sample intended for the laboratory

Guidelines – sampling - Regulation (EC) No 333/2007

Annex part B – sampling methods

- Sampling shall be performed by an authorised person
- Each lot or sublot shall be sampled separately
  - ✓ Sample of packaging of the same batch without food if possible
- Precautions shall be taken to avoid any changes which would affect
  - ✓ the levels of contaminants
  - ✓ adversely affect the analytical determination or
  - ✓ make the aggregate samples unrepresentative
    - e.g. containers, use of hand creams by sampling officer
- Incremental samples shall be taken at various places in the lot or sublot
  - ✓ Deviation from such procedure shall be recorded

Guidelines – sampling - Regulation (EC) No 333/2007

Annex part B – sampling methods

- The aggregate sample shall be made up by combining the incremental samples
- Each sample shall be placed in a clean, inert container
  - ✓ Protection against contamination and damage
  - ✓ No loss of analytes by adsorption to the internal wall of the container
  - ✓ No change in composition of the sample during transportation or storage
  - ✓ sample collection tool should be free from mineral oil contamination

Guidelines - sampling - Regulation (EC) No 333/2007

Annex part B – sampling methods

- Each sample shall be placed in a clean, inert container
  - ✓ Food should be sampled in inert containers in respect to mineral oil
  - ✓ Glass containers are preferred
    - Polyolefin sample containers made of e.g. PE and PP may release POSH.
    - Metal sample containers may have a mineral oil film on their surface
    - Paperboard boxes are not suitable even as secondary packaging
  - ✓ Cleanliness container needs to be checked for each new batch
  - ✓ After sampling close with PTFE layered lid or glass stopper
  - ✓ If not available cover glass container first with Al foil before closing with its cap
  - ✓ Al foil needs also be checked for residual mineral oil
  - ✓ Packaged food or FCM should be wrapped in aluminium foil at the point of sampling

Guidelines - sampling - Regulation (EC) No 333/2007

Annex part B – sampling methods

- Each sample shall be sealed at the place of sampling and identified
  - ✓ A record shall be kept of each sampling
  - ✓ Identification of sample
  - ✓ the date and place of sampling
  - ✓ Do not use tape or adhesives (paper/plastic labels) to fix the Al foil that covers the prepacked food
  - ✓ The sample number should be written on the aluminium foil by a marker

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Guidelines - sampling - Regulation (EC) No 333/2007

Annex part B – sampling methods

- Each sample shall be sealed at the place of sampling and identified.
  - ✓ If possible the following information of the food sample should be collected:
    - Article number
    - European Article Number (EAN) code
    - Batch or lot number
    - Country of origin, fill quantity of food
    - Amount of incremental samples
    - Total weight of aggregated food sample
    - Labels (physically or photocopy).
    - History of the food sample e.g. about possible contamination sources during food processing or contact with secondary packaging, transport boxes, jute bags, batching oils

Guidelines - sampling - Regulation (EC) No 333/2007

Annex part B – sampling methods

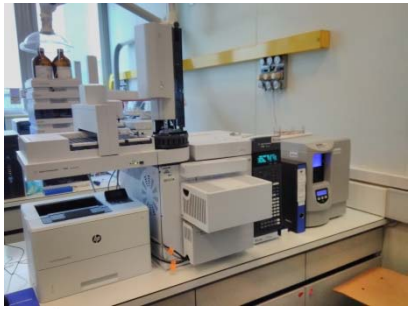
- Each sample shall be sealed at the place of sampling and identified
  - ✓ If possible the following information of the packaged food sample should be collected in addition:
    - Weight of packed food sample
    - Material of the packaging used (e.g. plastic, paper, virgin fibre, recycled paper)
    - Structure of the material of the packaging (e.g. multi-layer material or inner bag incl. further information on the material of layers), presence of a barrier, assembled packaging material)
    - End of the shelf life or the best before date
    - History of the prepacked food sample e.g. about contact with secondary packaging, transport boxes, jute bags

Guidelines - analysis

- Internal and verification standards
- Sample extraction procedures for different foods + FCMs
  - ✓ Animal and vegetable fats and oils: EN ISO 661/prEN16995
  - ✓ From recent literature
- Auxiliary methods:
  - ✓ MOSH: enrichment, elimination long chain n-C_n
  - ✓ MOAH: enrichment, epoxidation olefins
  - ✓ Fat removal by saponification (Moret et al.)

Guidelines - analysis

- Analytical method
  - ✓ Online LC-GC-FID method
    - Biedermann and Grob. J Chrom A (2012) 1255, 56– 75
    - Biedermann et al. J Chrom A (2017) accepted
    - Biedermann et al. J Consum Prot Food Saf (2017)
  - ✓ Manual method
    - Messung von Mineralöl – Kohlenwasserstoffen in Lebensmitteln und Verpackungsmaterialien, BfR-KLZH
    - Fiselier et al. J Chrom A, 1271 (2013) 192– 200
  - ✓ Confirmation: 2D GC-FID/MS
    - Biedermann et al. J Chrom A (2015) 1375, 146–153



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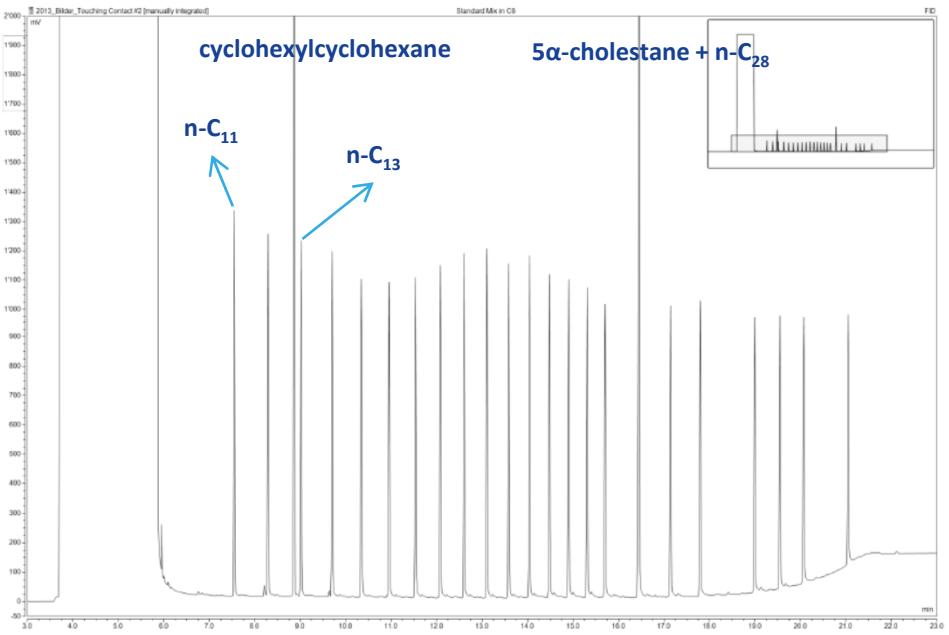
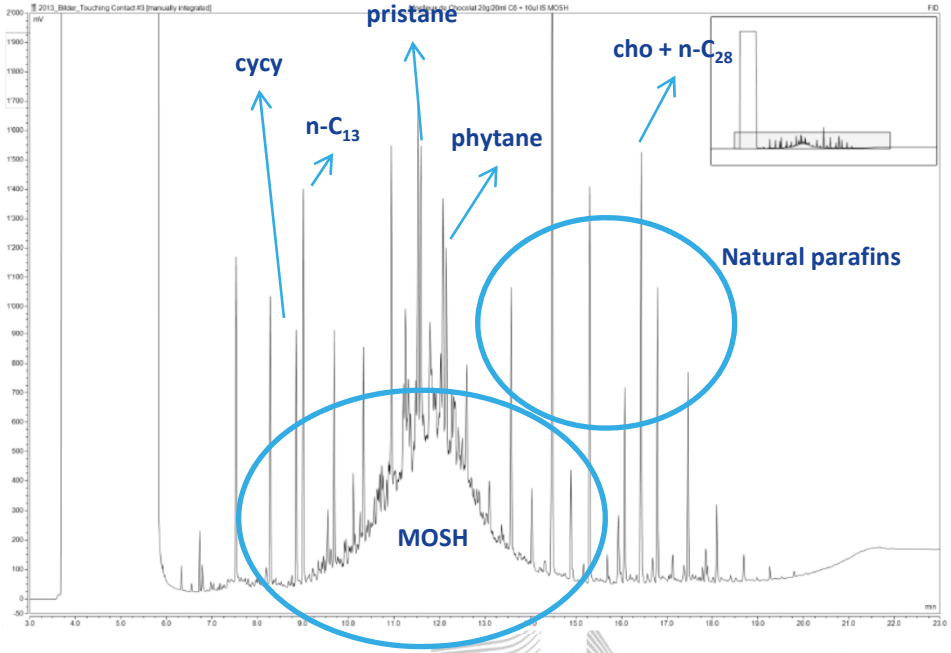
Guidelines - analysis

- Interpretation of chromatograms
  - Biedermann and Grob. J Chrom. A (2012) 1255, 76– 99



MOSH standards + n-C_n



Sample MOSH

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Guidelines - reporting

- Information about food + packaging material if relevant
- Place and date of sampling
- End of the shelf life
- Concentrations (whole mass; mg/kg) + 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_{16}-C_{25}, C_{25}-C_{35}, C_{35}-C_{50}
- Report presence of pristane, phytane, hopanes (MOSH) and diisopropyl naphthalenes (DIPN), dibenzothiophenes (MOAH), POSH, etc.
- start, top and end of all the hump(s) in terms of GC retention of n-C_n
- Integrated HPLC-GC-FID chromatograms of MOSH and MOAH
- Sample preparation methods and deviations to prescribed
- Auxiliary methods used and deviations to prescribed
- Description of strategy to identify source(s)
- + EFSA parameters

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