

# **Project-report**

# International test of various canned baby milk products for their content of mineral oil hydrocarbons (MOSH/MOAH)

A project of

foodwatch international with foodwatch Germany, foodwatch Netherlands and foodwatch France

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# **Executive summary**

Within the framework of an international product test, various infant formula and baby milk products packaged in cans were tested for their content of mineral oil hydrocarbons (MOSH/MOAH).

The selected products were purchased in the countries in which foodwatch offices are located: Germany, France and the Netherlands.

# **Method**

In total, 16 different products of infant formulas and baby milk products were analysed for their content of mineral oil hydrocarbons (MOSH/MOAH).

The analytical methods used for the determination (online LC/GC-FID) and verification of the results (GC\*GC-MS) correspond to the recommendations of the Technical Guidance of the European Commission, Joint Research Centre (JRC) for the current EU-wide monitoring for the determination of MOSH/MOAH in foodstuffs.[7] The chemical analysis of the products was carried out in 3 different, accredited laboratories in order to guarantee the highest possible and feasible precision of the analytical results. The verification of the results and the identification of marker compounds was performed in 2 different laboratories.

# **Results**

MOSH/POSH:

In 15 from 16 tested products MOSH/POSH were detected above the limit of quantification of 0.5 mg/kg.

The values range from 0,5 mg/kg up to 8,4 mg MOSH/POSH/kg of product. There are 4 samples in the range of more than 5 mg/kg MOSH/POSH up to 8,4 mg/kg.

# MOAH:

Positive levels of MOAH were found in 8 out of 16 samples (50 %). The levels found ranged from 0,5 mg/kg to 3,0 mg/kg. 8 of the products were not contaminated with MOAH.

The limit of quantification for the used method (Online-LC/GC-FID) was 0.5 mg/kg.

The product selection shows that many manufacturers in France offer their products in metal cans. Therefore 8 samples were selected here. 2 of these products were contaminated with MOAH.



In Germany, only a few manufacturers offer the products in metal cans. Here 4 different products were tested, 3 of them with MOAH contents above 0.5 mg/kg.

In the Netherlands there are only a few manufacturers who package their products in metal cans. However, 3 out of 4 products with positive MOAH contents were noticed here as well.

### **Conclusion/Evaluation of the results**

The results showed that the MOAH percentage in the positively tested products is an indication that they are contaminated with a mineral oil which is insufficiently purified. Food grade mineral oil saturated hydrocarbons (MOSH) (white oils) are reported to contain less than 1 % of MOAH [7].

The analytical verification of the positive results for corresponding marker substances and substance groups was carried out using a technically sophisticated procedure (GC\*GC-TOF). These tests proved the mineral, fossil origin of the found mineral oil contamination.

It is not possible to make a statement about the potential source of the mineral oil contamination. Contamination can occur during the whole production chain, as well as through possible contamination by the packaging material, the cans.

In 8 products (50%) no MOAH could be determined. This means that it is technically possible to produce products without detectable content of MOAH. However, in order to comply with the minimisation principle (ALARA), actions should be taken to ensure that the products concerned meet these requirements, which means that no MOAH should be detectable.

### **Demands**

foodwatch calls on all manufacturers of replacement milk for babies to

- immediately recall publicly and remove contaminated products from the market;

- immediately publish laboratory analyses that show whether their replacement milks are unpolluted or polluted with MOAH;

- prevent any contamination with MOAH mineral oil components suspected of being carcinogenic;

- make a public commitment to sell only food products which do not contain any detectable MOAH.



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### 1. Project target

- Determination of mineral oil hydrocarbons (MOSH/MOAH) in baby milk products, e.g. infant formula and/or follow-on formula, in particular of products packaged in cans
- > Comparison and evaluation of the results of different EU Member States (F, NL and D)
- > Test for mineral oil hydrocarbons in baby milk products packaged in metal cans
  - 8 products from France
  - 4 products from Germany
  - o 4 products from Netherland
- > Comparison and evaluation of the results in the products
- Summary and publication of results

### 2. Introduction

Mineral oils are found in the human environment in many areas, e.g. they have already been found in numerous foods such as rice, pasta, chocolate and edible oils, but can also be found in packaging, children's toys, animal feed and cosmetics. [1]



Packaging materials made of recycled paper can transfer mineral oils to food either through direct contact with the food or in a gaseous phase, i.e. through so called migration.

In addition, mineral oils can also enter foodstuffs at all stages of the processing chain, from harvesting through further processing to packaging. In the environment there is unavoidable, certain background pollution.

But mineral oils are also used specifically by the food industry. For example, as dust binders, lubricants in production machines, as release agents, as polishing agents or in adhesives.

# 3. Health risks

According to the European Food Safety Authority (EFSA) and the German Federal Institute for Risk Assessment (BfR), MOSH and MOAH have different toxicological potential. [1, 2] MOSH can be easily assimilated by the body and accumulate in fatty tissue. In experiments with rats, these led to damage in certain organs. Depending on the chain length and viscosity, MOSH can accumulate in organs of the human body, some fractions are of concern according to the EFSA.

However, only MOSH with a chain length greater than C16 are enriched. [1]

The intake of MOAH should generally be avoided as "a possible carcinogenic potential [...] cannot be excluded" [2].

Since, in addition, no toxicological data are available for evaluation so far, no tolerable intake has been derived to date. [2]

A final risk assessment by EFSA is still pending. However, in January 2017, the European Commission adopted a recommendation for the monitoring of "mineral oil hydrocarbons in foods and materials and articles intended to come into contact with foods". The resulting data will then be made available to EFSA for evaluation. [4]

According to the German Federal Institute for Risk Assessment (BfR), it cannot be ruled out that carcinogenic substances may be present in this fraction of MOAH. The Federal Institute for Risk Assessment (BfR) therefore demands the greatest possible minimisation for MOAH in foodstuffs (ALARA principle: "As Low As Reasonably Achievable"). [1] The opinion of the BfR is also shared by EFSA. [2]

Given the genotoxic and mutagenic nature demonstrated for certain MOAHs, the French Agency for Food, Environmental and Occupational Health & Safety (ANSES) [10] believes that priority should be given to reducing the contamination of food by these compounds. ANSES recommends limiting consumer exposure to MOHs, and to MOAHs in particular, by acting initially on the main sources



of mineral oils in paper and cardboard packaging. ANSES recommends the use of barriers to limit the migration of MOHs from packaging into foods.

In July 2019, a study by the Dutch National Institute for Public Health and the Environment (RIVM) was published, for which new toxicological data since the last EFSA-Opinion in 2012 were evaluated and linked to consumption data. The RIVM concluded that dietary exposure to MOSH had no health effects on the Dutch population. In addition, the migration of MOSH from recycled packaging into food contributes only to a small extent to the overall dietary exposure. The focus should be on exposure to MOAH, as substances contained in this fraction may have a carcinogenic effect. However, this does not mean that all mineral oils containing MOAH are also carcinogenic, but rather that a distinction must be made between the sources. Those containing potentially carcinogenic MOAH compounds should be minimised. [5]

For the risk assessment of MOAH found in food, it is important to distinguish between different sources of MOAH. MOAH derived from crude oils or oils not sufficiently purified, as well as MOAH from combusted (or heated) mineral oils are carcinogenic and therefore, these contaminations should be avoided as much as possible. MOAH from highly refined oils or waxes are of lesser concern.

The risk assessment of MOH is subject to gaps of knowledge. The toxicological relevance of effects shown for MOSH in rats for humans is questioned. For MOAH, with a possible mutagenic and carcinogenic potential, there are no dose response data available. In addition, the characterization of MOH remains a problem, especially for MOAH: some MOAH mixtures may be mutagenic and carcinogenic while others are relatively harmless. A review of the literature published since the 2012 EFSA opinion showed that these gaps in knowledge still form an obstacle for assessing the risks of MOSH and MOAH for human health. The lack of harmonized European or national limits for MOH (MOSH and MOAH) in food products confirms this.[5]

Methods to discriminate between different MOAH in mixtures are currently not available. That is why it is important to distinguish between the different sources of contamination for MOAH, as some contain harmful MOAH, where others do not.[5]

# 4. Sampling

The sampling buying of the products took place in parallel in all three countries in the last week of July and the first week of August 2019.

The determinations and verification of the results are performed from August 2019 to October 2019.

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# 5. Test procedure and analytical methods

Quantitative determination and verification of analytical results is an essential and important part of product testing.

In order to ensure that the product test has the highest possible precision, reproducibility and validity, the quantitative determination of the products was carried out in 3 different laboratories.

This means that <u>for all products with MOAH contents above 0.5 mg/kg</u> (= Limit of Quantification) <u>the determinations were analysed in two further laboratories to verify the MOSH and MOAH results</u>.

The following requirements had to be met by the participating laboratories:

- All participating laboratories must be accredited according to DIN EN ISO/IEC 17025-The participating laboratories all needed to have a substantial experience in the field of the analysis of mineral oil hydrocarbons.
- The quantification of the MOSH/POSH and MOAH should be performed in all laboratories using the Online-LC/GC-FID method. This is the method of choice recommended by the European Commission, Joint Research Centre (JRC) Technical Guidance for the current EU-wide monitoring for the determination of MOSH/MOAH in food. [1,2,7,8,9]
- Sample preparation MOSH fraction: Determination after digestion of product and a) clean up with aluminium oxide or b) without clean up step.
- Sample preparation MOAH fraction: Digestion of the products with subsequent clean up of the extract with epoxidation.

<u>The verification of positive results to determine typical markers and substances for MOSH and</u> <u>MOAH fractions are performed by mass specific detection</u>. For this step the method of choice is two-dimensional gaschromatographic separation and mass specific detection, see [7,8,9]. This verification and identification of markers was performed by 2 laboratories using GC\*GC-TOF.

# 6. Test results

All 16 products were analysed to determine which products are contaminated with mineral oil hydrocarbons. Products with amounts above 2 mg/kg MOSH and 0,5 mg/kg MOAH were analysed in a second repetitive analysis.

To be sure that there is no cross contamination, from all of the products with MOAH contents above 0,5 mg/kg a second container of the same batch was analysed.



In addition, a confirmatory analysis of these products was carried out in 2 further laboratories. For all products with positive MOAH results, the verification of the results and the test for markers was performed in two laboratories using GC\*GC-TOF technology.

The results of laboratory 1 for all 16 selected products are shown in Table 1-3.

Product name	Batch number	Expiration date	MOSH/POSH (C10-C50)	MOAH (C10-C50)
Nestlé Nidal Lait en poudre 1er âge	90720346AC	01.03.2021	5,8 mg/kg	1,2 mg/kg
Nestlé Guigoz Lait bébé en poudre 1 bio	90650017C3	01.09.2020	n.d.	n.d.
Lactalis Célia Lait bébé en poudre 2	800000047	24.09.2020	2,3 mg/kg	n.d.
Lactalis Célia Lait bébé en poudre 1 bio	8000000411	30.04.2020	0,8 mg/kg	n.d.
Vitagermine Baby bioOptima 2	2VT21974	10.02.2021	1,1 mg/kg	n.d.
Hipp Lait pour nourrissons Combiotic 1	1424990	23.12.2019	0,5 mg/kg	n.d.
Danone Blédina Blédilait Croissance + 3	2021.01.27.26	27.01.2021	0,7 mg/kg	n.d.
Danone Gallia Galliagest Croissance 3 Sans lactose	905764 (019079)	19.12.2019	4,0 mg/kg	0,7 mg/kg

### **Test results France**

Table 1: Test results France

### **Test results Germany**

Product name	Batch number	Expiration date	MOSH/POSH (C10-C50)	MOAH (C10-C50)
Novalac Säuglingsmilchnahrung PRE 400g	A59522 75	11.03.2020	3,8 mg/kg	0,5 mg/kg
Nestlé BEBA OPTIPRO PRE 800 g von Geburt an	91120346AA	10/2020	8,4 mg/kg	3,0 mg/kg
Nestlé BEBA OPTIPRO 1 800 g von Geburt an	9098080621	10/2020	5,8 mg/kg	1,9 mg/kg
Nestlé BEBA OPTIPRO 3 800 g ab dem 10. Monat	9108080626	10/2020	1,9 mg/kg	n.d.

Table 2: Test results Germany



### **Test results Netherlands**

Product name	Batch number	Expiration date	MOSH/POSH (C10-C50)	MOAH (C10-C50)
Neolac Biooogisch 1 Volledige zuigenlingenvoeding 0-6 m	11620	15.01.2021	4,3 mg/kg	1,6 mg/kg
Hero Baby nutrasense hypo- allergeen 0-6 maanden	80926-023	26.09.2020	4,1 mg/kg	0,8 mg/kg
Nutrilon Dieetvoeding bij koemelkallergie 1 0-6 maanden	907222 41	22.08.2020	6,1 mg/kg	1,2 mg/kg
Ah zuigelingenmelk 1 STANDAARD 0-6 maanden	30397033	15.04.2021	3,4 mg/kg	n.d.

### Table 3: Test results Netherlands

Notes:

Method: Online-LC/GC-FID;

MOSH: Saponification of sample, cleanup step, removal of natural alkanes with Aluminiumoxide;

MOAH: Saponification, cleanup step and removal of di- and triglycerides after epoxidation

Results calculated to lower bound theory

n.d.= not detected below Limit of Quantification (LOQ) with 0.5 mg/kg  $\,$ 

# 7. Interpretation and discussion

#### MOSH/POSH :

For milk and milk-based products there are currently no agreed acceptable values of contamination published.<sup>1</sup>

<sup>&</sup>lt;sup>1</sup> Due to the persisting lack of legal maximum values all over Europe the German competent authorities together with the food industry published "orientation values for mineral oil hydrocarbons (MOH) in foodstuffs" (April 2019). Dairy products are not covered at all for the time being. But for all food groups covered any verification of MOAH is not accepted. And it appears remarkable for comparison reasons that the sum of MOAH (C10-C50) in 8 out of 16 samples is **above** the "orientation value" of 0.5 mg/kg, i.e. level of quantification for bread and pastries, fine bakery products, cereal products and cereal-based products, cereals.



- All 16 products, however, show a wide range of contamination from MOSH/POSH ranging from non-detectable (below 0,5 mg/kg) to 8.4 mg/kg.
- The products examined here are foodstuffs for particularly vulnerable groups: infants and babies. With regard to maximum acceptable levels of contamination, even stricter standards should be used.

### MOAH:

- For milk and milk-based products there are currently no agreed acceptable values of contamination published.
- The MOAH contents in all 16 products show a wide range of contamination. They vary from non-detectable (below 0,5 mg/kg) to 3,0 mg/kg.
- The products examined here are foodstuffs for particularly vulnerable groups: babies and infants. With regard to maximum acceptable levels of contamination, even stricter standards should be used.

In all products with MOAH contents above 0.5 mg/kg, the percentage of the MOAH fraction to the total mineral oil content was calculated. These proportions are all in the range from 11,6 % MOAH, (Novalac Säuglingsmilchnahrung PRE 400 g) to 27,1 % MOAH (Neolac Biologisch 1).

Food grade mineral oil saturated hydrocarbons (MOSH) (white oils) are reported to contain less than 1 % of MOAH. [7]

Typical marker substances could be identified in the MOSH and MOAH fractions of these products.

### Conclusion:

- The results of the ratio between MOSH and MOAH (MOAH content in whole mineral oil) of the products tested positive for MOAH is an indication that the products are contaminated with a mineral oil which is insufficiently purified.
- The verification of the positively tested products (MOAH above 0,5 mg/kg) by using GC\*GC-TOF all showed typical markers and typical compound classes in both the



MOSH and MOAH fraction. The results for MOAH here should be evaluated according to the ALARA (*As Low As Reasonably Achievable*) principle. It would be good practice from both manufacturers and distributors/retailers of the products to ensure that the concentration of contaminants in foodstuffs is as low as reasonably possible. Only 50 % of the products were able to comply with this principle, as they have a MOAH content below detectable levels (limit of quantification).

- Considering the Limit of Quantification for this method with 0.5 mg MOAH /kg all 8 products with results above this value do not meet the requirement of ALARA.
- As the tested products are foods for particularly vulnerable groups (infants and babies), a discussion on stricter limit values should also be initiated regarding the MOSH results.
- It is not possible to make a statement about the potential source of the mineral oil contamination. Contamination can occur during the whole production chain. It can also come from the packaging, i.e. the cans of the products. However, since no MOAH was found in 50 % of the products, it seems technically feasible to produce.

# 8. Appendix

[1] EFSA Panel on Contaminants in the Food Chain (CONTAM); Scientific Opinion on Mineral Oil Hydrocarbons in Food. EFSA Journal 2012;10(6):2704.

[2] German Federal Institute for Risk Assessment (BfR); Questions and answers on mineral oil components in food - Updated FAQ of the BfR of 12 December 2017. https://www.bfr.bund.de/de/fragen\_und\_antworten\_zu\_mineraloelbestandteilen\_in\_lebensmitt eln-132213.html

[3] German Federal Institute for Risk Assessment (BfR); Recommendation XXXVI. paper, cardboard and paperboard for food contact; 01.09.2017

[4] COMMISSION RECOMMENDATION (EU) 2017/84 of 16 January 2017

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[5] D. Buijtenhuijs, B.M: van den Ven, National Institute for Public Health and the Environment;
Mineral oils in food; a review of ocurence and sources; RIVM Letter report 2019-0048; DOI
10.21945/RIVM-2019-0048

[6]: Benchmark levels for mineral oil hydrocarbons (MOH) in food:



Consumer Protection Consortium of the Federal States, (LAV); German Federation for Food Law and Food Science, (BLL)

Date: April 2019

https://www.lebensmittelverband.de/download/benchmark-levels-moh-in-foods

[7] Bratinova, S., Hoekstra, E. (Editors) JRC Technical Reports (2019) Guidance on sampling, analysis and data reporting for the monitoring of mineral oil hydrocarbons in food and food contact materials. 2019, ISBN 978-92-76-00172-0

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Foodstuffs - 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;

[9] Toolbox German Federation of Food Law and Food Science's (BLL) "Toolbox for Preventing the Transfer of Undesired Mineral Oil Hydrocarbons into Food." 2018

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[10] Opinion of the French Agency for Food, Environmental and Occupational Health & Safety on the migration of mineral oil compounds into food from recycled paper and cardboard packaging (Date : 08/03/2017)

https://www.anses.fr/en/system/files/ESPA2015SA0070EN.pdf