SLOVENSKI STANDARD
SIST EN 17517:2022

01-januar-2022

Krma: metode vzorčenja in analize - Določevanje nasičenih ogljikovodikov iz mineralnih olj (MOSH) in aromatskih ogljikovodikov iz mineralnih olj (MOAH) z analizo on-line HPLC-GC-FID

Animal feeding stuffs: Methods of sampling and analysis - Determination of mineral oil saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) with on-line HPLC-GC-FID analysis

Futtermittel: Probenahme- und Untersuchungsverfahren - Bestimmung von mineralölgesättigten Kohlenwasserstoffen (MOSH) und mineralölaromatischen Kohlenwasserstoffen (MOAH) mit Online-Analyse durch HPLC-GC-FID

Aliments pour animaux : Méthodes d'échantillonnage et d'analyse - Détermination des hydrocarbures saturés d'huile minérale (MOSH) et des hydrocarbures aromatiques d'huile minérale (MOAH) par analyse CLHP CG-FID en ligne

Ta slovenski standard je istoveten z: **EN 17517:2021**

ICS:

65.120	Krmila	Animal feeding stuffs

SIST EN 17517:2022 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 17517

October 2021

ICS 65.120

English Version

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This European Standard was approved by CEN on 2 August 2021.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Ref. No. EN 17517:2021 E

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European foreword

This document (EN 17517:2021) has been prepared by Technical Committee CEN/TC 327 “Animal feeding stuffs - Methods of sampling and analysis”, the secretariat of which is held by NEN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2022, and conflicting national standards shall be withdrawn at the latest by April 2022.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document has been prepared under a standardization request given to CEN by the European Commission and the European Free Trade Association.

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Introduction

WARNING — The method described in this document implies the use of reagents that pose a hazard to health. This document does not claim to address all associated safety problems. It is the responsibility of the user of this document to take appropriate measures for the health and safety protection of the personnel prior to use of the standard and to ensure that regulatory and legal requirements are complied with.

1 Scope

This document specifies a method for the determination of saturated and aromatic hydrocarbons (from C10 to C50) in feed. The method has been interlaboratory validated with on-line-HPLC-GC-FID – see [1], [2] and [3]. This method is not intended to be applied to other matrices.

The method can be used for the analysis of mineral oil saturated hydrocarbons (MOSH) and/or mineral oil aromatic hydrocarbons (MOAH).

The method is applicable for feed materials, in particular vegetable oils and other fat rich feed materials, compound feeds and pre-mixtures. It is not applicable to additives or deodistillates.

NOTE 1 The method was not designed for encapsulated matrices.

The method has been tested in an interlaboratory study via the analysis of both naturally contaminated and spiked samples (pre-mixture, soybean meal, sunflower seeds, chicken feed, pig feed, vegetable oil) ranging from 3 mg/kg to 286 mg/kg for MOSH and from 1 mg/kg to 16 mg/kg for MOAH.

According to the results of the interlaboratory study, the method has been proven suitable for MOSH and MOAH mass concentrations, each above 10 mg/kg. However, the method was not fully validated during the collaborative study for the premixture sample due to too low concentrations of MOSH and MOAH. The method was also not fully validated during the collaborative study for the sunflower seeds sample due to a too low concentration of MOAH.

NOTE 2 The conclusions regarding MOAH are based on 4 analyte / matrix combinations while the IUPAC protocol [4] expects this to be a minimum of 5.

In case of suspected interferences from natural sources, the fossil origin of the MOSH and MOAH fraction can be verified by examination of the pattern by GC-MS.

For the determination of MOSH and MOAH in edible fats and oils, another CEN standard is also available: EN 16995. For more information see [5].

Annex C proposes a manual alternative method to on-line HPLC-GC-FID analysis that can be used as a screening method for the determination of MOSH.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 6498, *Animal feeding stuffs - Guidelines for sample preparation (ISO 6498)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
mineral oil saturated hydrocarbons
MOSH
paraffinic (open-chain, usually branched) and naphthenic (cyclic, alkylated) hydrocarbons

3.2
mineral oil aromatic hydrocarbons
MOAH

aromatic mainly alkylated hydrocarbons

3.3
unresolved complex mixture
UCM

complex mixture of saturated or aromatic hydrocarbons not resolved by gas chromatography such as branched paraffins, alkylated naphthenes and alkylated aromatics

4 Principle

The fatty material is extracted from the commodity using organic solvent. After concentration of part of the solvent, the extract is submitted to an epoxidation step. The fractions of MOSH and MOAH are isolated and separated by an HPLC-GC-FID system. MOSH and MOAH fractions are separated on a silica gel column using an *n*-hexane/dichloromethane gradient and each transferred as 450 µl fractions to GC using the Y-interface [6], while triglycerides are kept on the HPLC column. Solvent vapours are discharged via a solvent vapour exit located between the uncoated pre-column and the GC separation column. Volatile components are retained by solvent trapping applying partially concurrent eluent evaporation. High boiling components are spread over the entire length of the flooded zone and refocused by the retention gap technique [2].

The area attributed to mineral oil is calculated by subtraction of peaks due to *n*-alkanes (naturally occurring hydrocarbons), terpenes, squalene and its isomerization products, sterenes and olefins with the structure of carotenoids. MOSH and MOAH are quantitated by internal standard added before analysis. Verification standards are added for monitoring proper HPLC fractionation and GC transfer conditions.

Epoxidation is a purification step that is necessary for the quantification of MOAH. This purification step allows the elimination of olefins like squalene, which elute within the MOAH fraction and interfere with quantification (e.g. olive oil, palm oil). Epoxidation also removes certain olefins co-eluting with the MOSH fraction, therefore epoxidation also may be used as a purification step for the MOSH fraction. The epoxidation step is the best compromise to remove olefins, even though it is not fully quantitative and the efficiency may be sample dependent. Depending on the sample, this reaction may induce the epoxidation of a part of the MOAH or incomplete removal of the interfering olefins.

5 Reagents

WARNING — The method described in this document implies the use of reagents that pose a hazard to health. This document does not claim to address all associated safety problems. It is the responsibility of the user of this document to take appropriate measures for the health and safety protection of the personnel prior to use of the standard and to ensure that regulatory and legal requirements are complied with.

Unless otherwise specified, use only reagents of recognized analytical grade.

5.1 Demineralized water, stored in a glass bottle.

5.2 *n*-Hexane, trace organic analysis grade, for pesticide residue analysis.

n-Hexane purity can be checked by concentrating 30 ml of *n*-hexane mixed with 25 µl of internal standard solution (5.16) and 2 drops of keeper (5.27) using a rotary evaporator, dissolving the residue in 0,2 ml of *n*-hexane and the analysis of 50 µl by on-line-HPLC-GC-FID (6.10). Take care that in the evaporation step

the residue is not evaporated to dryness to avoid loss of volatile hydrocarbons. The signal abundance of the residue after evaporation should not exceed a tenth of the signal abundance obtained at the quantification limit.

5.3 Toluene.

5.4 1,1,2-Trichloroethane.

5.5 Perylene (Per), purity ≥ 99 %.

5.6 *α*-Cholestane (Cho), purity ≥ 97 %.

5.7 *n*-Undecane (*n*-C11), purity ≥ 98 %.

5.8 *n*-Tridecane (*n*-C13), purity ≥ 97 %.

5.9 Tri-tert-butylbenzene (TBB).

5.10 Bicyclohexyl (CyCy), purity ≥ 99 %.

5.11 1-Methylnaphthalene (1-MN), purity ≥ 95 %.

5.12 2-Methylnaphthalene (2-MN), purity ≥ 97 %.

5.13 Pentylbenzene (5-PB), purity ≥ 96 %.

5.14 Stock solutions, mass concentration ρ = 10 mg/ml.

Prepare individual stock solutions by weighing, to the nearest 1 mg, 100 mg of *n*-C11 (5.7), *n*-C13 (5.8), TBB (5.9), CyCy (5.10), 1-MN (5.11), 2-MN (5.12) and 5-PB (5.13) into a 10 ml volumetric flask and dilute to the mark with 1,1,2-trichloroethane (5.4) or toluene (5.3). Store the solutions at room temperature. If crystals precipitate during storage, warm the solution until everything has dissolved.

5.15 Internal standard solution 1 (ISTD1).

Weigh, to the nearest 0,5 mg, 12 mg of Per (5.5) and Cho (5.6) in a volumetric flask of 20 ml (6.21), to which 600 µl of each stock solution (5.14) is added with the exception of *n*-C13, of which 300 µl is added. Fill the volumetric flask up to 20 ml with 1,1,2-trichloroethane (5.4) or toluene (5.3). Resulting mass concentrations are for *n*-C13: ρ = 150 µg/ml, for *n*-C11, TBB, CyCy, 1-MN, 2-MN and 5-PB: ρ = 300 µg/ml and for Per, Cho: ρ = 600 µg/ml.

NOTE This mixture of internal standards is commercially available, ready to use product.[1]

5.16 Internal standard solution 2 (ISTD2).

Dilute the ISTD1 solutions by a factor of 10, e.g. 1 ml filled up to 10 ml with *n*-hexane (5.2). Resulting mass concentrations are for *n*-C13: ρ = 15 µg/ml, for *n*-C11, TBB, CyCy, 1-MN, 2-MN and 5-PB: ρ = 30 µg/ml and for Per, Cho: ρ = 60 µg/ml.

5.17 Chloroperbenzoic acid (CPBA), purity 70 % to 75 %.

[1] Restek Corp. ®, Cat.# 31070 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Other products could be used, if the results are comparable.

5.18 CPBA solution, ρ = 0,2 g/ml in absolute ethanol.

For example 5 g of CPBA (5.17) in 25 ml of absolute ethanol (5.22). The solution can be used for up to one week.

5.19 Carrier gas for gas chromatography, preferably hydrogen, purity ≥ 99,995 %.

5.20 Auxiliary gases for flame ionization detector, hydrogen, air, and nitrogen suitable for gas chromatography.

5.21 Alkane standard mixture C10 to C40, solution of equal concentration in an apolar solvent, ρ = 1 µg/ml.

5.22 Ethanol, absolute.

NOTE The ethanol purity can be checked by concentrating 50 ml of ethanol mixed with 25 µl of internal standard solution (5.16) and 2 drops of keeper (5.27) using a rotary evaporator, dissolving the residue in 0,2 ml of *n*-hexane and the analysis of 50 µl by on-line-HPLC-GC-FID (6.10).

5.23 *n*-Pentacontane (C50), purity ≥ 98 %.

5.24 *n*-Pentacontane (C50) solution in toluene, ρ approximately 10 µg/ml.

Weigh 2 mg of C50 (5.23) in a volumetric flask of 20 ml (6.21) and dilute to the mark with toluene (5.3). Proceed to a second dilution of 1 ml in a 10 ml volumetric flask (6.21). Store the solutions at room temperature.

NOTE 1 Solubility of pentacontane in toluene is limited at room temperature. However, the concentration of the solution of pentacontane does not need to be accurate as it is used only to determine the limit of integration for mineral oil peak.

NOTE 2 It is also possible to use a commercial mixture of *n*-alkanes from C12 to C60 that contains *n*-pentacontane.[2]

5.25 Sodium carbonate solution, ρ = 0,1 g/ml in water (5.1).

5.26 Dichloromethane (DCM), trace organic analysis grade, purity ≥ 99 %.

DCM purity can be checked by concentrating 50 ml of DCM mixed with 25 µl of internal standard solution (5.16) and 2 drops of keeper (5.27) using a rotary evaporator, dissolving the residue in 0,2 ml of *n*-hexane and the analysis of 50 µl by on-line-HPLC-GC-FID (6.10). Take care that in the evaporation step the residue is not evaporated to dryness to avoid loss of volatile hydrocarbons. The signal abundance of the residue after evaporation should not exceed a fifth of the signal abundance obtained at the quantification limit.

5.27 Keeper solvent.

The keeper is a solvent that will not evaporate or evaporate to a lesser degree during the evaporation step, e.g. bis(2-ethylhexyl) maleate. A keeper is used to enhance the recovery of volatile compounds.

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[2] ASTM® D5442 C12-C60 Qualitative Retention Time Mix available by e.g. Supelco® Cat.# 500623 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Other products could be used, if the results are comparable.

6 Apparatus

IMPORTANT — The glassware used for the determination shall be thoroughly cleaned and rinsed with *n*-hexane (5.2) before use so that it is free from impurities.

Usual laboratory apparatus and, in particular, the following. The glassware shall be thoroughly cleaned and rinsed with *n*-hexane (5.2) or baked in an oven before use so that it is free from impurities.

6.1 Mill with stainless-steel rotor or ball mill, capable of reaching particles size ≤ 1 mm.

6.2 Magnetic stirrer.

6.3 Magnetic stir bars.

6.4 Analytical balance, reading accuracy 0,000 1 g.

6.5 Round-bottomed flasks, 250 ml capacity.

6.6 Glass vials with screw caps, 15 ml and 40 ml capacity.

6.7 Centrifuge and centrifuge tubes.

6.8 Automatic evaporator (optional)[3].

6.9 Glass sample vials, volume of 2 ml.

6.10 High performance liquid chromatograph, coupled with gas chromatograph and flame ionization detector (HPLC-GC-FID).

6.11 Data acquisition system, with the possibility of manual integration.

6.12 LC column, 5 µm (250 mm x 2 mm inner diameter (i.d.)) or equivalent.

The silica gel column shall have a capacity to retain 20 mg fat. [4]

6.13 Uncoated precolumn, 10 m x 0,53 mm or equivalent[5].

6.14 Capillary column 1, capable for temperatures up to 350 °C.

The column should have the following characteristics: 100 % dimethylpolysiloxane or 95 % dimethyl / 5 % phenyl methylpolysiloxane stationary phase, a length of 15 m, an internal diameter of 0,32 mm or 0,25 mm and a film thickness 0,10 µm to 0,15 µm or equivalent.

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[3] MicroDancer®, IR-Dancer (e.g. Zinser) or Syncore® Analyst (Büchi) are examples of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of these products. Other products could be used, if the results are comparable.

[4] LiChrospher® Si 60 is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Other products could be used, if the results are comparable.

[5] Hydroguard® MXT® is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Other products could be used, if the results are comparable.

6.15 Capillary column 2, from transfer valve to first Y-piece, fused silica (FS) methyl silicone deactivated (length 1 m, outside diameter (o.d.) 0,27 mm, inner diameter (i.d.) 0,1 mm).

6.16 Capillary column 3, for hydrogen carrier gas, FS methyl silicone (length 1 m, o.d. 360 µm, i.d. 25 µm).

6.17 Capillary column 4, for solvent vapour exit, FS methyl silicone (length 1 m, o.d. 0,68 mm, i.d. 0,53 mm).

The columns given in 6.15, 6.16 and 6.17 have proven to be suitable for the analysis. However these columns can be adjusted in accordance with the characteristics of the HPLC-GC apparatus and the analytical conditions.

6.18 Restriction capillary column, transfer valve and solvent vapor exit, FS uncoated (length 1 m, o.d. 360 µm, i.d. 50 µm).

6.19 Microsyringe, 5 µl to 100 µl capacity, suitable for injection in liquid chromatography.

6.20 Pasteur pipette, glass.

The use of plastic pasteur pipettes and polyethylene film shall be avoided.

6.21 Volumetric flasks, various sizes.

7 Sampling

The sample should be truly representative and not damaged or changed during transport or storage.

Samples should be packed in glass bottles or aluminium foil in order to prevent additional contamination. Plastic and paper packaging are unsuitable.

Sampling is not part of the method specified in this document. A recommended sampling method is given in EN ISO 6497 [7].

8 Preparation of the test sample

Prepare the test sample in accordance with EN ISO 6498.

Grind the laboratory sample (typically 50 g) to a particle size of at least 1 mm in the mill (6.1) in order to ensure representative data. Mix the sample thoroughly.

9 Preparation of the analytical sample

9.1 Fat extraction from feed sample

9.1.1 Fatty material extraction from samples with fat content lower than 30 %

Weigh 5 g of milled sample in a 250 ml round-bottomed flask (6.5), add a magnetic stir bar (6.3). Add 500 µl ISTD2 (5.16) and 100 ml of *n*-hexane (5.2) to the sample and mix for 1 h with a magnetic stirrer (6.2).

Transfer 20 ml of the solvent phase into a 40 ml glass vial (6.6) and wash with 5 ml of demineralized water (5.1). Centrifuge for 2 min at a speed of 2 500 rpm and transfer 10 ml of sample extract to a 15 ml glass vial (6.6) and concentrate the solvent down to 1 ml (triglycerides from the sample act as a keeper) under a stream of nitrogen, using either a water bath at 35 °C or an automatic evaporator (6.8).

9.1.2 Fatty material extraction from sample with fat content higher than 30 %

Weigh 2 g of milled sample in a 250 ml round-bottomed flask (6.5), add a magnetic stir bar (6.3). Add 250 µl ISTD2 (5.16) and 100 ml of *n*-hexane (5.2) to the sample and mix for 1 h with a magnetic stirrer (6.2). Transfer 30 ml of the solvent phase into a 40 ml glass vial (6.6) and wash with 5 ml of demineralized water (5.1). Centrifuge for 2 min at a speed of 2 500 rpm and transfer 20 ml of sample extract to a 40 ml glass vial (6.6) and concentrate the solvent down to 1 ml (triglycerides from the sample act as a keeper) under a stream of nitrogen, using either a water bath at 35 °C or an automatic evaporator (6.8).

9.2 Procedure for fats and fat extracts

9.2.1 Procedure for liquid and solid fats

Weigh, to the nearest 1 mg, 300 mg sample into a 10 ml vial, and fill it up with 600 µl *n*-hexane and add 50 µl ISTD2 (5.16). Shake the vial.

The amount of the added internal standards may be increased, in order to lower the impact of the matrix interferences, if necessary.

The amount of the added internal standards may be decreased, in case low concentrations shall be measured.

Add 500 µl of CPBA ethanolic solution (5.18) and place the vial into an agitator to be shaken for 15 min at a speed of 1 800 rpm at room temperature. Immediately, add 3 ml of sodium carbonate solution (5.25), shake the mixture for 2 min and then centrifuge for 2 min at a speed of 2 500 rpm.

NOTE The collaborative study was performed in 2018. Therefore the epoxidation procedure does not correspond to the one proposed during the Roundtable Workshop on the Determination of MOAH in Infant Formula [11].

Transfer 500 µl of the hexane phase to an autosampler vial. At a maximum, inject 50 µl into the HPLC.

Depending on the level of contamination, the injected volume may be adapted in order to avoid the overloading of the chromatograms.

9.2.2 Procedure for extracted fats

Add 500 µl of CPBA ethanolic solution (5.18) to the sample extract obtained from the fat extraction step (9.1) and place the vial into an agitator to be shaken for 15 min at a speed of 1 800 rpm at room temperature. Immediately add 3 ml of sodium carbonate solution (5.25), shake the mixture for 2 min and then centrifuge for 2 min at a speed of 2 500 rpm.

NOTE The collaborative study was performed in 2018. Therefore the epoxidation procedure does not correspond to the one proposed during the Roundtable Workshop on the Determination of MOAH in Infant Formula [11].

Transfer 500 µl of the hexane phase to an autosampler vial. At a maximum, inject 50 µl into the HPLC.

Depending on the level of contamination, the injected volume may be adapted in order to avoid the overloading of the chromatograms.

9.3 Blank

A procedural blank sample including the fat extraction step (from 9.1.2) should be analysed in order to test the purity of the reagents but also other possible sources of contamination, such as the glassware and the analytical instrument.

The mineral oil content of the procedural blank shall not exceed the level of 3 mg/kg of fat, considering a test portion equal to 5 g of sample. If this level is exceeded, the source of contamination shall be identified and eliminated.