BS ISO 17780:2015

BSI Standards Publication

Animal and vegetable fats and oils — Determination of aliphatic hydrocarbons in vegetable oils

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National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Animal and vegetable fats and oils — Determination of aliphatic hydrocarbons in vegetable oils

Corp gras d'origines animale et végétale — Détermination des hydrocarbures aliphatiques en corps gras d'origines végétale

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 34, *Food products*, Subcommittee SC 11, *Animal and vegetable fats and oils*.

Introduction

The major saturated hydrocarbons present in vegetable oils are long chain *n*-alkanes, containing more than 21 carbon atoms, and having an odd carbon number preference.[1]

Mineral oils can contain *n*-alkanes with up to 60 carbon atoms with no odd carbon predominance. Chromatograms of mineral oils obtained by this method are characterized by a wide peak due to the presence of a complex mixture of saturated branched and cyclic hydrocarbons. Medium and low viscosity mineral oils are typically characterized by a complex mixture with between C10 and C25 chain length; while high viscosity mineral oils are indicated by a complex mixture with the midpoint around C30 chain length.[2] The Joint FAO/WHO Expert Committee on Food Additives (JECFA) has set several ADIs for mineral oil (2002) dividing low-medium viscosity mineral oils into three different subclasses depending on the point of toxicity. This method does not help to distinguish between different classes.

Chromatograms of diesel oil are characterized by the presence of *n*-alkanes between C10 and C25 chain length with no odd carbon predominance, i.e. both even and odd numbered hydrocarbons are present in relatively equal proportions.

INTERNATIONAL STANDARD ISO 17780:2015(E)

Animal and vegetable fats and oils — Determination of aliphatic hydrocarbons in vegetable oils

1 Scope

This International Standard specifies a method for the determination of saturated aliphatic hydrocarbons from C10 to C56 of natural origin present in vegetable oils, and for detecting the presence of mineral oil and diesel oil.

The method is applicable to all types of crude and refined edible oils and fats, for concentrations of mineral oils from 50 mg/kg to 1 000 mg/kg.

A rapid method for refined and virgin (or cold-pressed) oils is proposed in Annex C. This rapid method is not adapted for crude oils due to a lack of retention of triglycerides observed for some samples.

A method for fat recovery from food samples by soxhlet extraction with a blend of solvents is proposed in Annex D.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 661, *Animal and vegetable fats and oils — Preparation of test sample*

3 Terms and definitions

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

3.1
hydrocarbon contents
sum of saturated aliphatic hydrocarbons, expressed as a mass fraction, determined according to the method specified

3.2
unresolved complex mixture
UCM
complex mixture of saturated hydrocarbons not resolved by gas chromatography, represented by a wide peak, which can be due to a contamination with mineral oil

Note 1 to entry: The width of the peak is approximately 5 min to 15 min depending on gas chromatography conditions,

Note 2 to entry: See relevant chromatograms in Annex A.

3.3
diesel
sum of saturated *n*-alkanes between C10 and C25 chain length, expressed as a mass fraction, determined according to the method

Note 1 to entry: See relevant chromatograms in Annex A.

4 Principle

The saturated aliphatic hydrocarbons of the sample are isolated by liquid chromatography on silica gel impregnated with silver nitrate and determined by capillary gas chromatography with flame ionization detection using an internal standard. From the chromatogram, the area attributed to mineral oil is calculated by the subtraction of sharp peaks due to *n*-alkanes (naturally occurred hydrocarbons) from the total area including the UCM. To indicate diesel contamination, the peak areas of individual hydrocarbons between C10 and C25 chain length are summed and quantified together.

5 Reagents

WARNING — Attention is drawn to national regulations that specify the handling of hazardous substances, and users' obligations thereunder. Technical, organizational and personal safety measures shall be followed.

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

5.1 Silica gel 60[1)], extra pure for column chromatography with particle size between 60 µm and 200 µm (70-230 mesh).

5.2 Water, distilled and cooled down to room temperature.

5.3 Anhydrous sodium sulfate, analytical grade, purity 99 % minimum.

NOTE Sodium sulfate may be replaced by sea sand, washed with *n*-hexane.

5.4 *n*-Hexane, trace organic analysis grade, purity 99 % minimum, residue after evaporation maximum 2 mg/kg.

NOTE 1 Hexane purity may be checked by concentrating 200 ml of *n*-hexane mixed with 2 ml of internal standard solution (5.6) using a rotary evaporator, dissolving the residue in 0,2 ml of *n*-hexane and the analysis of 5 µl by gas chromatography (9.3).

NOTE 2 Hexane may be replaced by isooctane, *n*-heptane or a mixture of alkanes of boiling point 65 °C to 70 °C, as long as the residue after evaporation is maximum 2 mg/kg. Solvents with higher boiling point than *n*-hexane take longer to evaporate. However, they are preferred due to the toxicity of hexane.

5.5 Internal standard: *n*-octadecane (C18), purity 99 % minimum.

n-Octadecane may be replaced by *n*-eicosane (C20). Before choosing one of these two compounds as the internal standard, it should be verified that there is no co-elution with other peaks from the sample to be analysed.

n-Octadecane shall be replaced by naphthalene if the sample is contaminated with a diesel oil, in order to avoid the overlapping of the internal standard peak with the alkane peaks to be quantified.

5.6 Solution of internal standard, mass concentration ρ = 0,04 mg/ml.

As an example, weigh to the nearest mg, approximately 50 mg of *n*-octadecane (5.5) and dilute to 25 ml with *n*-hexane (5.4), and then proceed with a second dilution of this mixture of 1 ml → 50 ml with *n*-hexane. Store this solution at room temperature in order to maintain its stability.

5.7 *n*-Decane (C10), purity 99 % minimum.

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1) Silica gel is available from Merck, reference 7754 or 7734. This reference is an example of a suitable product which is available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

5.8 *n*-Decane solution, mass concentration ρ = 0,04 mg/ml.

As an example, weigh to the nearest mg, approximately 50 mg of *n*-decane and dilute to 25 ml with *n*-hexane (5.4), and then proceed with a second dilution of this mixture of 1 ml → 50 ml with *n*-hexane. Store this solution at room temperature in order to maintain its stability.

5.9 Octatetracontane (C48), purity 99 % minimum. This standard is used to limit the integration of the hump to a certain retention time that will correspond to the retention time of this hydrocarbon.

5.10 Octatetracontane solution, mass concentration approximately ρ = 0,08 mg/ml.

As an example, weigh to the nearest mg approximately 2 mg of octatetracontane (5.9) and dilute to 25 ml of *n*-hexane (5.4). Store this solution at room temperature in order to maintain its stability.

NOTE Solubility of octatetracontane in hexane is limited at room temperature, due to its high melting point. However, the concentration of the solution of octatetracontane does not need to be accurate as it is used only to determine the limit of integration for the mineral oil peak.

5.11 Silver nitrate ($AgNO_3$), analytical grade.

5.12 Silver nitrate aqueous solution, mass concentration ρ = 0,75 g/ml.

As an example, to prepare silver nitrate silica gel for 3 columns, weigh approximately 4,5 g of silver nitrate in 6 ml of distilled water (5.2).

5.13 Carrier gas for gas chromatography, helium or hydrogen.

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

5.15 Alkane standard mixture C10 to C40[2)], solution in non-polar solvent.

5.16 Viscous paraffin and highly liquid paraffin[3)], solution in non-polar solvent.

5.17 Solution of paraffin and *n*-octadecane, mass concentration of paraffin ρ = 0,5 mg/ml, mass concentration of *n*-octadecane ρ = 0,08 mg/ml.

As an example, weigh to the nearest mg, approximately 500 mg of viscous paraffin (5.16) and 80 mg of *n*-octadecane (5.5) and dilute to 10 ml with *n*-hexane (5.4), and then proceed with a second dilution of this mixture of 1 ml → 100 ml with *n*-hexane. Store this solution at room temperature in order to maintain its stability.

6 Apparatus

Usual laboratory apparatus and, in particular, the following.

IMPORTANT — The glassware used for the determination shall be thoroughly cleaned and rinsed with n-hexane (5.4) before use so that it is free from impurities.

2) Alkane standard mixture at 50 mg/l is available from Sigma-Aldrich, reference 68281 (www.sigmaaldrich.com). This reference is an example of suitable products which are available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

3) A viscous paraffin is available from Merck, reference 107160. Highly liquid paraffin is available from Merck, reference 107174. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

6.1 Glass column for chromatography (30 cm to 40 cm length and 15 mm to 20 mm internal diameter), fitted with sintered glass discs and polytetrafluoroethylene (PTFE) stop cock.

NOTE A pad of cotton wool exhaustively extracted with *n*-hexane may be used to replace the sintered glass discs in the glass column.

6.2 Glass rods.

6.3 Round-bottomed flasks, 250 ml and 500 ml capacity.

6.4 Rotary evaporator, with vacuum and a water bath at 35 °C (recommended). Care should be taken to prevent cross contamination. Clean the system thoroughly between determinations.

6.5 Automatic evaporator[4], for 10 ml tube (optional), recommended operating conditions: temperature of water bath = 35 °C, nitrogen pressure = 5 psi.

6.6 Conical glass sample vials, 10 ml capacity.

6.7 Gas chromatograph, suitable for use with capillary column, equipped with an on-column injector or equivalent device, a temperature-programmable oven and a flame ionization detector (FID).

NOTE A programmed temperature vaporization injector (PTV) may also be used.

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

6.9 Capillary column, capable of being programmed up to 400 °C ("high temperature" type) for which the following characteristics are recommended: 100 % dimethylpolysiloxane or 95 % dimethyl/5 % diphenyl polysiloxane stationary phase, length 15 m, internal diameter 0,32 mm or 0,25 mm, film thickness 0,1 µm.

NOTE In order to get a separation between the solvent peak and mineral oil containing short chain hydrocarbons (C10 to C14), a 30 m long capillary column can be used.

6.10 Microsyringe, 5 µl to 10 µl capacity, suitable for on-column injection in gas chromatography.

6.11 Analytical balance, reading accuracy 0,001 g, weighing precision 0,001 g.

6.12 Pasteur pipette, in glass.

Plastic Pasteur pipettes shall be avoided. Polyethylene film shall also be avoided.

7 Sampling

A representative sample should have been sent to the laboratory. It should not have been damaged or changed during transport or storage.

Sampling is not part of the method specified in this International Standard. A recommended sampling method is given in ISO 5555.[3]

4) Zymark TurboVap LV evaporator is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.

8 Preparation of the test sample

Prepare the test sample in accordance with ISO 661.

9 Procedure

9.1 Chromatography column preparation

9.1.1 Preparation of $AgNO_3$ impregnated silica gel

Preparation of the silver nitrate silica gel column (for 3 columns): weigh 45 g of silica gel (5.1) in a 500 ml round-bottomed flask (6.3) protected by aluminium foil. With a Pasteur pipette (6.12), add drop by drop the silver nitrate solution (5.12) shaking continuously. Shake well for 30 min to homogenize. After completing, cover the flask with aluminium foil and allow to stand at room temperature for 12 h before use.

In order to improve homogenization, it is recommended to use an automatic shaker. If no automatic shaker is available, it is possible to put the flask in a rotatory evaporator equipment and rotate for 30 min without vacuum.

NOTE 1 The impregnated silica gel can be stored one week at room temperature in a desiccator, provided the flask is protected with aluminium foil.

NOTE 2 For screening purposes, $AgNO_3$ impregnated silica gel can be replaced by non-impregnated silica gel. The results will be similar or higher than those obtained using silvered silica gel.

NOTE 3 For refined vegetable oils other than refined olive pomace oil, $AgNO_3$ impregnated silica gel can be replaced by non-impregnated silica gel.

9.1.2 Column packing

In a beaker, suspend 18,5 g of silver nitrate impregnated silica gel (9.1.1) in *n*-hexane (5.4). The slurry is introduced onto the column (6.1) containing 40 ml of *n*-hexane and pack the column by tapping it gently using a glass rod (6.2). Add at least 0,5 cm to 1 cm of sodium sulfate (5.3) on top of the $AgNO_3$ - silica gel, and compress the $AgNO_3$ - silica gel bed with a stream of nitrogen. Rinse the $AgNO_3$ - silica gel with another 60 ml of *n*-hexane (5.4) to eliminate impurities in the $AgNO_3$ - silica gel.

The column should be covered with a black paper cylinder or with aluminium foil to avoid oxidation of the silver nitrate.

Elute the solvent until the level of the solvent in the column is about 0,5 cm higher than the $AgNO_3$ - silica gel bed. Put a 250 ml round-bottomed flask (6.3) under the chromatography column.

9.2 Elution of the hydrocarbon fraction

Weigh to the nearest 1 mg, 1 g of the sample in a beaker and add 1 ml of the solution of the internal standard (5.6), transfer the solution to the chromatographic column (9.1.2) with the aid of a Pasteur pipette (6.12) and let the sample penetrate into the stationary phase. Wash the beaker with two portions of 1 ml of *n*-hexane (5.4) and introduce the solution into the column. Elute the hydrocarbon fraction with 55 ml of *n*-hexane (5.4) with a cadence of approximately 15 drops every 10 s, collecting the fraction in a 250 ml flask (6.3). Evaporate most of the solvent up to 1 ml or 2 ml with a rotary evaporator equipped with a water bath set to 35 °C (6.4). Transfer the concentrated solution to a 10 ml conical tube. Concentrate the solvent up to 0,5 ml from the conical tube under a stream of nitrogen, using either a water bath at 35 °C or an automatic evaporator (6.5). Take care that, in both evaporation steps, the residue is not evaporated to dryness to avoid loss of the volatile alkanes.

Adjust the elution volume of *n*-hexane by the analysis of 1 ml solution of paraffin (5.17) and collecting consecutive fractions of 50 ml, 10 ml, and 10 ml, and analysing each one by gas chromatography (GC).

9.3 Gas chromatography

9.3.1 Gas chromatography setup

Install the column (6.9) in the gas chromatograph (6.7) and check the working conditions by injecting the solvent, *n*-hexane (5.4). The baseline should be straight with a small positive drift. If the drift is high, proceed to condition the column, for a negative drift check the connections of the column.

If the column is used for the first time, it is necessary to condition the column by heating it in the column oven using a temperature gradient up to 370 °C (depending on the oven temperature chosen for the analysis) in 4 h. Maintain the temperature for 2 h.

9.3.2 Working conditions for gas chromatography analysis

The following working conditions have proved satisfactory for the analysis:

Column	DB5 HT (15 m long - 0,25 mm internal diameter - 0,10 µm film thickness)
Oven temperature	Initial temperature 60 °C for 3 min, programmed at 12 °C/min to 350 °C, hold for 10 min
Carrier gas	Hydrogen head pressure of 100 kPa
Detector temperature	370 °C
Injection volume	2 µl

NOTE When a programmed temperature vaporization injector is used, the following working conditions have proved to be satisfactory for the analysis: initial temperature 50 °C for 0,5 min; programmed at 300 °C/min to 300 °C, hold for 10 min.

These conditions may be adjusted in accordance with the characteristics of the gas chromatograph apparatus and the column. **However, the oven temperature shall be brought up to 350 °C in order to elute the high molecular weight hydrocarbons**. A temperature ramp of 12 °C/min is a good compromise between a good sensitivity due to a “thinner hump” and a limited baseline drift.

Typical chromatograms are presented in Annex A.

9.3.3 Peak identification

Identify the internal standard *n*-octadecane by injecting 2 µl of the standard solution (5.6). Check the resolution of the *n*-decane (C10) separated from the solvent peak, by injecting 2 µl of the standard solution (5.8). The resolution of the octatetracontane (C48) can be checked by injecting 2 µl of the standard solution (5.10). See the chromatograms in Figures A.1 and A.2.

Inject 2 µl of the alkane standard mixture C10-C40 (5.15) in order to identify the areas to take into consideration for the calculation of C10-C25 alkane concentration (Figure A.1).

In sunflower oils, the major peaks correspond to saturated aliphatic hydrocarbons C27, C29 and C31 (Figure A3).

The resolution of highly liquid paraffin and viscous paraffin (5.16) can be checked by injecting 2 µl of the 0,5 mg/ml standard solution (Figures A.4 and A.5).

A broad peak of about 5 min to 15 min width, depending on the GC conditions, represents a complex mixture of hydrocarbons (UCM) that the chromatography cannot resolve and is attributed to mineral oils (Figure A.6 to Figure A.10).

In case of contamination of vegetable oil with diesel oil, the chromatogram is characterized by the presence of *n*-alkanes between C10 and C25 chain length with no odd carbon predominance (Figure A.11). Naphthalene shall then be used as internal standard.

9.3.4 Performance of the gas chromatography system

Inject 2 µl of the solution of paraffin and *n*-octadecane (5.17) in order to check the performance of the gas chromatography analysis.

Calculate the recovery of the paraffin as follows:

$$R_{HC} = \frac{\sum A_{HC} \cdot C_{IS} \cdot 100}{A_{IS} \cdot C_{HC}}$$

where

$\sum A_{HC}$ is the peak area of paraffin UCM peak (integration from the internal standard peak to the end of the UCM peak - Figure A.4);

A_{IS} is the peak area of the internal standard peak (*n*-octadecane);

C_{IS} is the concentration, in milligrams per millilitres, of *n*-octadecane in the solution of internal standard (5.17);

C_{HC} is the concentration, in milligrams per millilitres, of the paraffin in the solution of internal standard (5.17).

The gas chromatography system is considered as optimized if the recovery of the paraffin is equal to or more than 90 %.

9.4 Procedural blank

A procedural blank sample 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.

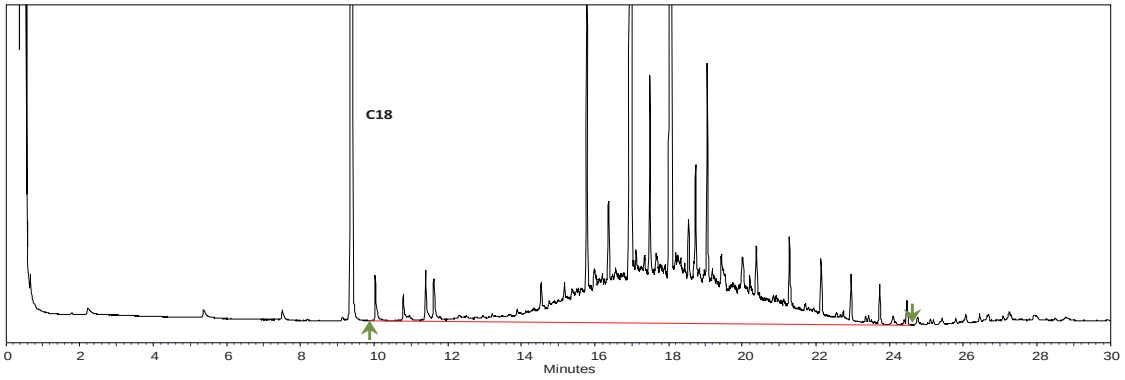
The mineral oil content of the procedural blank shall not exceed the level of **10 mg/kg**. If this level is exceeded, the source of contamination shall be identified and eliminated.

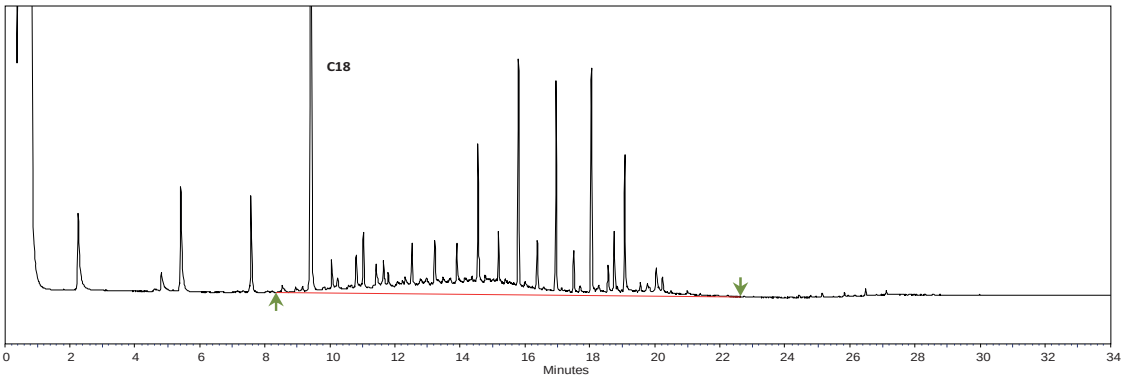
Results shall not be corrected by deduction of the blank content.

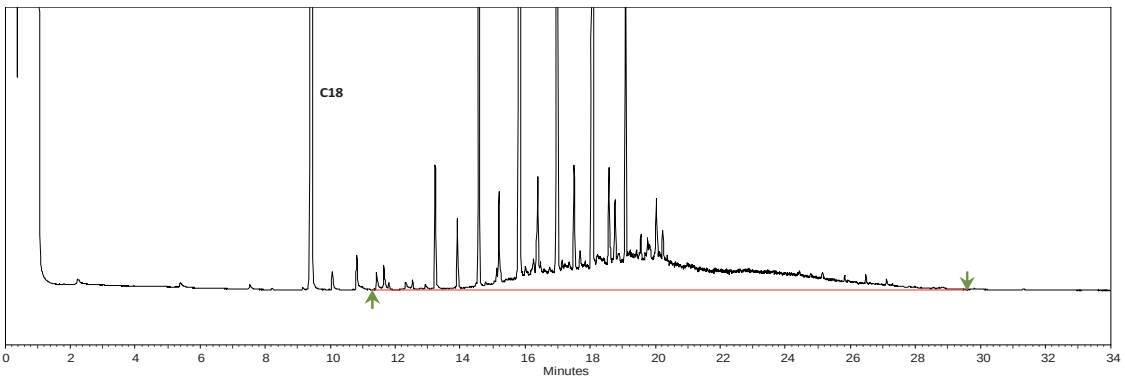
A procedural blank is a blank sample made up of all reagents foreseen for the preparation of a test portion and processed in all respects as a test portion. It consists of 1 ml of internal standard (5.6) transferred to the $AgNO_3$ impregnated silica gel column and analysed as a sample according to the method. The level of contamination is expressed in mg per kg of vegetable oil, considering that the analysis is for 1 g of oil.

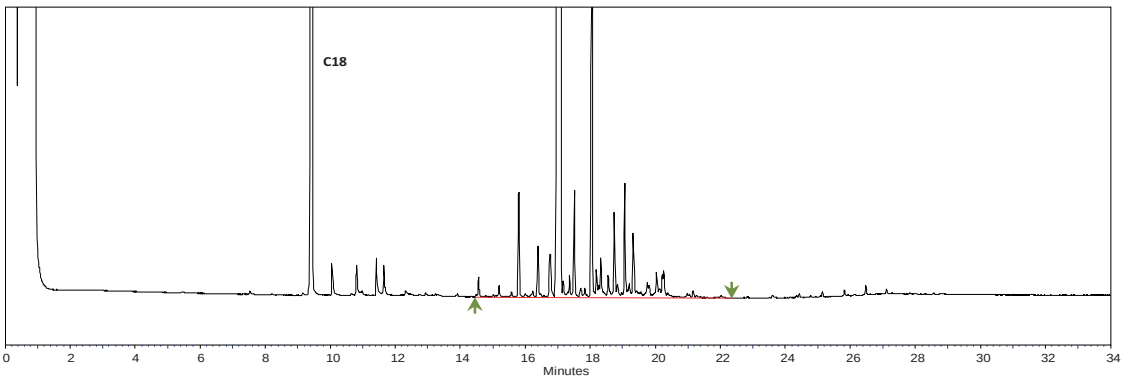
9.5 Quantitative determination

To calculate the content of hydrocarbons in the UCM, first determine the mass fraction of total hydrocarbons (w_{HC1}) by integrating manually the total signal composed of the UCM and the sharp peaks above the UCM from the point that the baseline starts to increase until the baseline at the retention time of octatetracontane (C48). However, depending on the type of mineral oil, the integration of the UCM may begin before the retention time of the internal standard, and it may end after the retention time of the octatetracontane (see Figure 1 for examples of the integration of total hydrocarbon signal; the arrows indicate the beginning and the end of the integration).









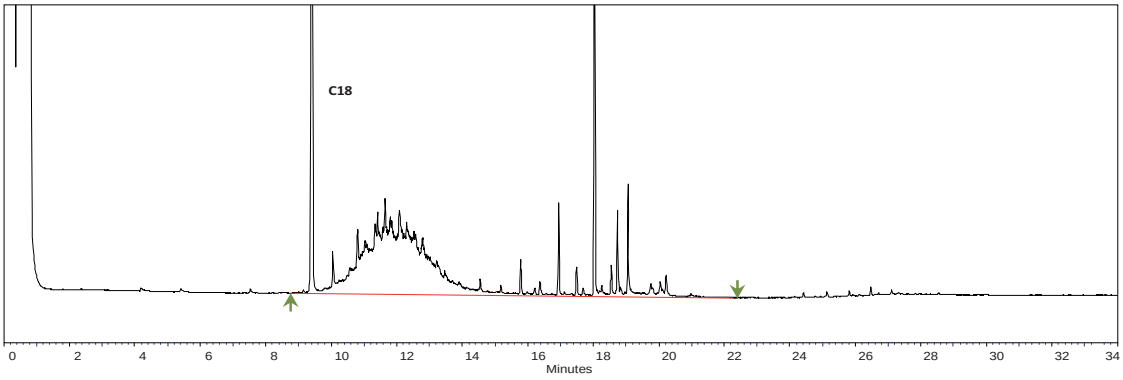
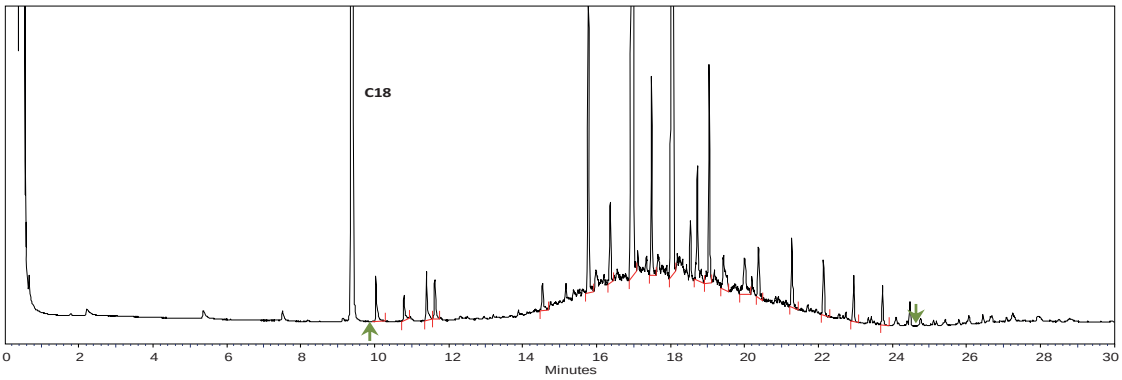
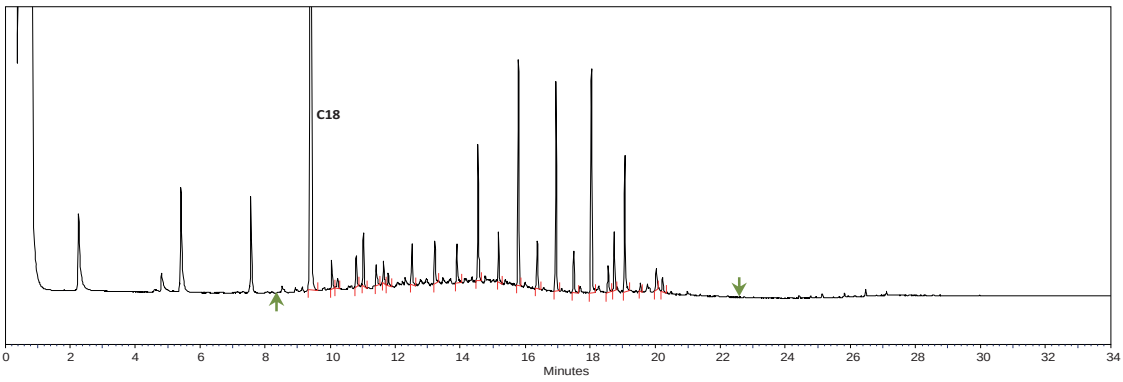
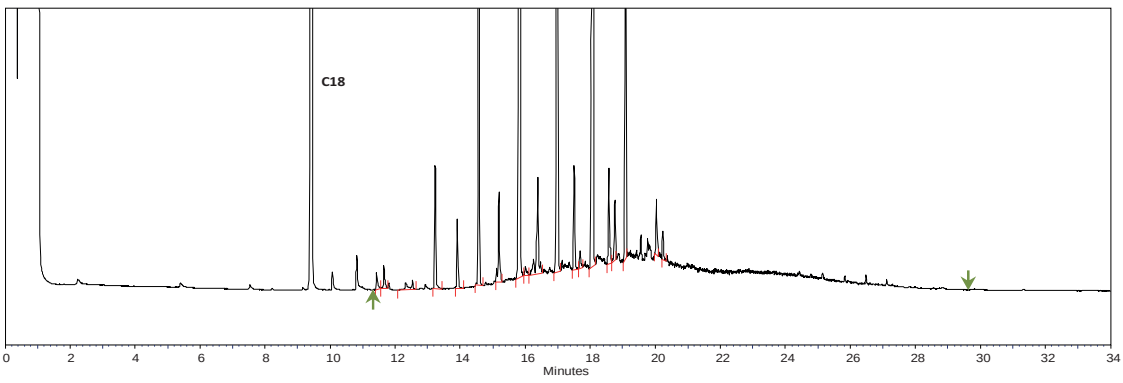


Figure 1 — Examples of integration of the total hydrocarbon signal

Then calculate the mass fraction of hydrocarbons of natural origin (w_{HC2}) re-integrating the chromatogram by tracing manually the valley-to-valley baseline over the UCM profile for all the sharp peaks (see Figure 2 for examples of the integration of natural hydrocarbon signal).







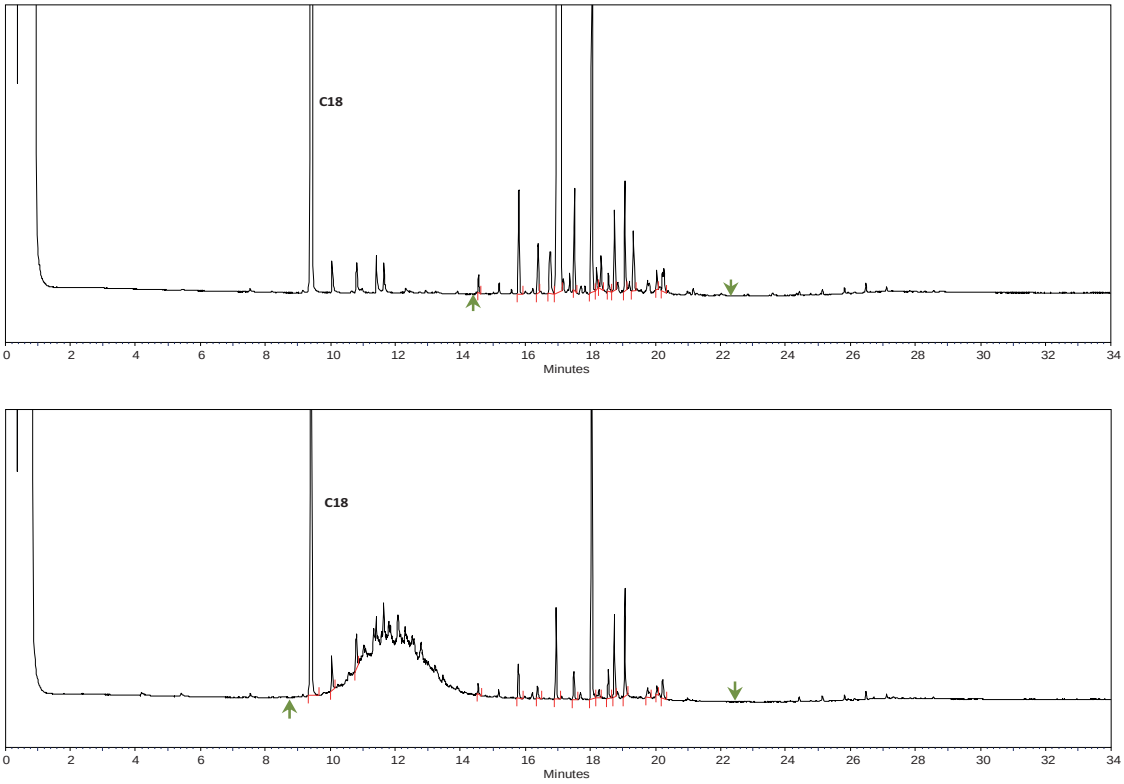


Figure 2 — Examples of the integration of natural hydrocarbon signal

If the *n*-octadecane peak lies on the UCM peak, the area of the standard determined in the second chromatogram (integrated valley-to-valley) (A_{IS}) is subtracted from the total area of the first chromatogram (including the UCM) and the resultant value is A_i.

The mass fraction w_{HC} of the hydrocarbons content, expressed in milligrams per kilogram, is calculated as follows:

$$w_{HC} = \frac{\sum A_i \cdot m_{IS} \cdot 1\,000}{A_{IS} \cdot m}$$

where

$\sum A_i$ is the peak area of all peaks other than the internal standard peak (either the peaks above the UCM only for W_{HC2}, or the UCM + the peaks above the UCM for W_{HC1});

A_{IS} is the peak area of the internal standard peak;

m_{IS} is the mass, in milligrams, of the internal standard in 1 ml of solution (5.6);

m is the mass of the test portion, in grams.

The LOQ of the UCM peak is variable because it depends on the width and height of the peak, which is analyte dependent. The value is subjected to a large error because small variations in the baseline produce great variations in the area. An estimation of the LOQ is 25 mg/kg.

To calculate the concentration of UCM and n-alkanes that elute between C10 to C25, it is necessary to superimpose the chromatogram of the sample and the chromatogram of the alkane standard mixture C10 to C40 and to integrate the part of the UCM which is included in the range C10 to C25. In the standard mixture, the C25 is not present, and hence the retention time should be calculated.

To calculate the concentration of a diesel oil contamination, integration of the *n*-alkanes that elute between C10 to C25 shall be carried out. It is necessary to superimpose the chromatogram of the sample and the chromatogram of the alkane standard mixture C10 to C40, in order to identity the peaks that need to be integrated (Figure A.11).

Validation data for the silver nitrate impregnated silica gel purification are reported in Annex B.

10 Determination of hydrocarbons attributed to mineral origin

The content of hydrocarbons attributed to mineral origin ($W_{\text{MO-C10-56}}$) expressed as a mass fraction in milligrams per kilogram, is calculated as follows:

$$w_{\text{MO}} = w_{HC1} - w_{HC2}$$

The result is expressed in milligrams per kilogram of mineral oil, referred to the internal standard. In case of $w_{\text{MO-C10-25}}$, a special integration till C25 should be carried out.

11 Precision

11.1 Interlaboratory test

Details of an interlaboratory test on the precision of the method are summarized in Annex E. It is possible that the values derived from this interlaboratory test are not applicable to concentration ranges and matrices other than those given.

11.2 Repeatability

The absolute difference between two independent single test results, obtained with this same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will, in not more than 5 % of cases, exceed the repeatability limits, *r*, given in Table E.1, Table E.2 and Table E.3.

11.3 Reproducibility

The absolute difference between two single test results, obtained with this same method on identical test material in different laboratories by different operators using different equipment, will, in not more than 5 % of cases, exceed the reproducibility limits, *R*, given in Table E.1, Table E.2 and Table E.3.

12 Test report

The test report shall specify:

— all information necessary for the complete identification of the sample;

— the sampling method used, if known;

— the test method used, with reference to this International Standard, i.e. ISO 17780;

— the mass(es) of the test portion(s);

— all operating details not specified in this International Standard or regarded as optional, together with details of any incidents that may have influenced the test result(s);

— the test result(s) obtained, or, if the repeatability has been checked, the final quoted result obtained.

Annex A
(informative)

Examples of chromatograms

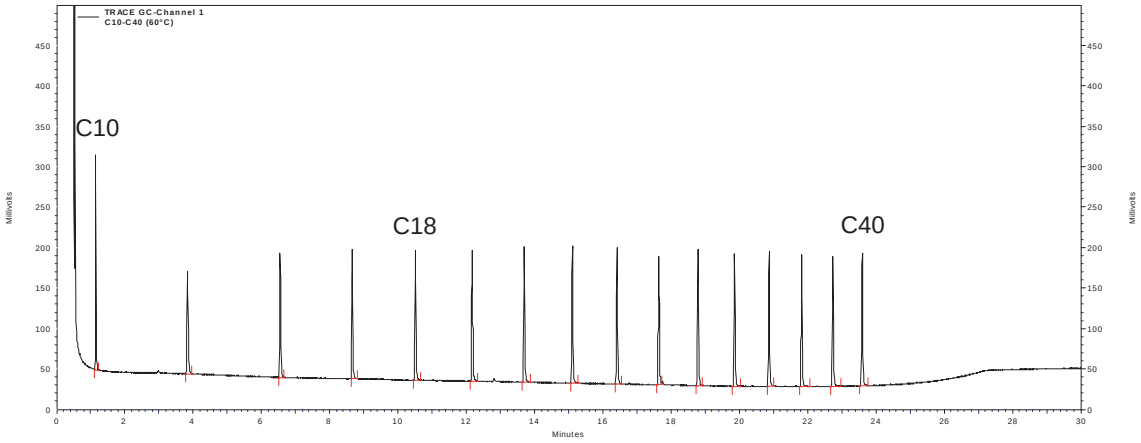


Figure A.1 — GC chromatogram of an alkane standard mixture C_{10} to C_{40}

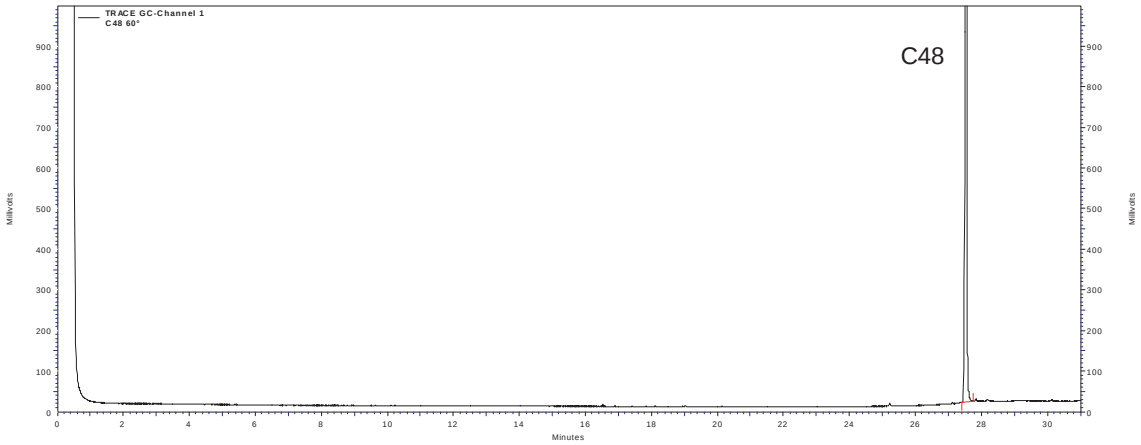


Figure A.2 — GC chromatogram of the hydrocarbon standard: octatetracontane

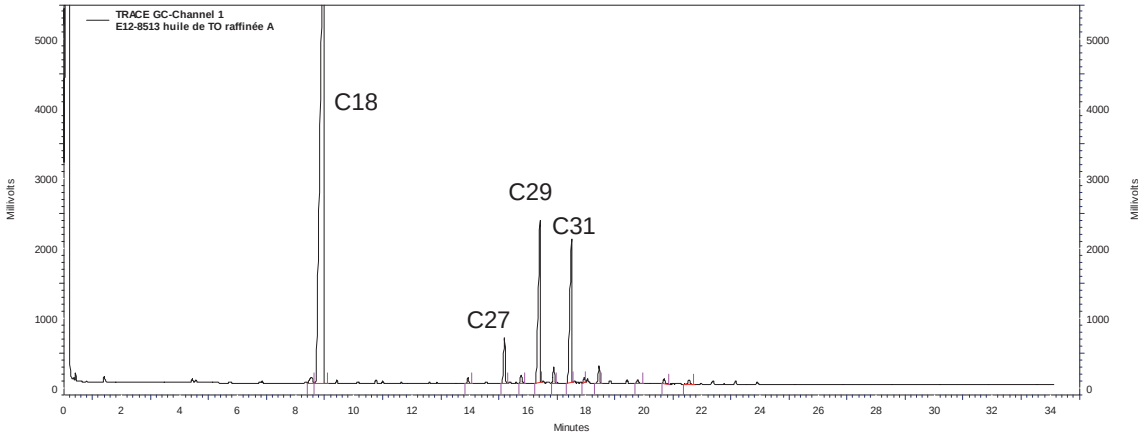


Figure A.3 — GC chromatogram of the aliphatic hydrocarbons fraction of a non-contaminated refined sunflower oil purified with silver nitrate impregnated silica gel — Internal standard: *n*-octadecane

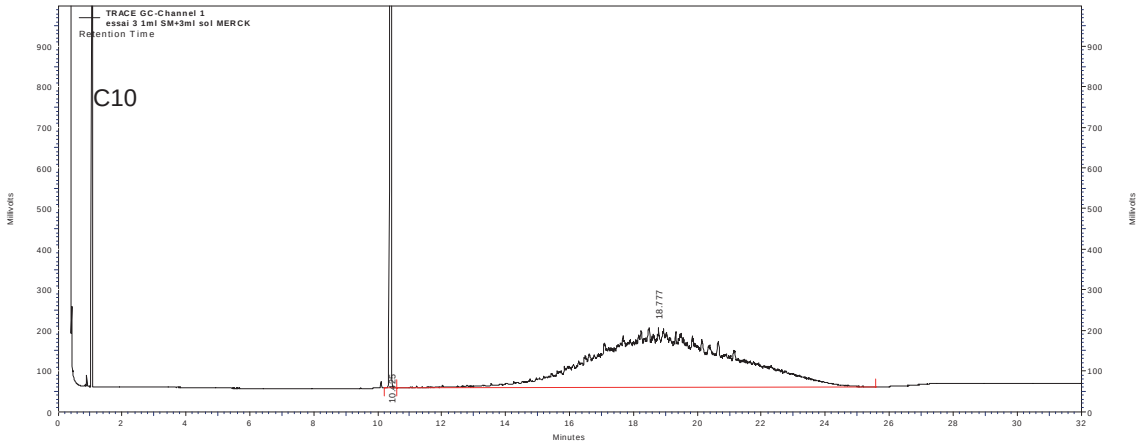


Figure A.4 — GC chromatogram of high viscosity paraffin — Internal standard: *n*-octadecane + *n*-decane

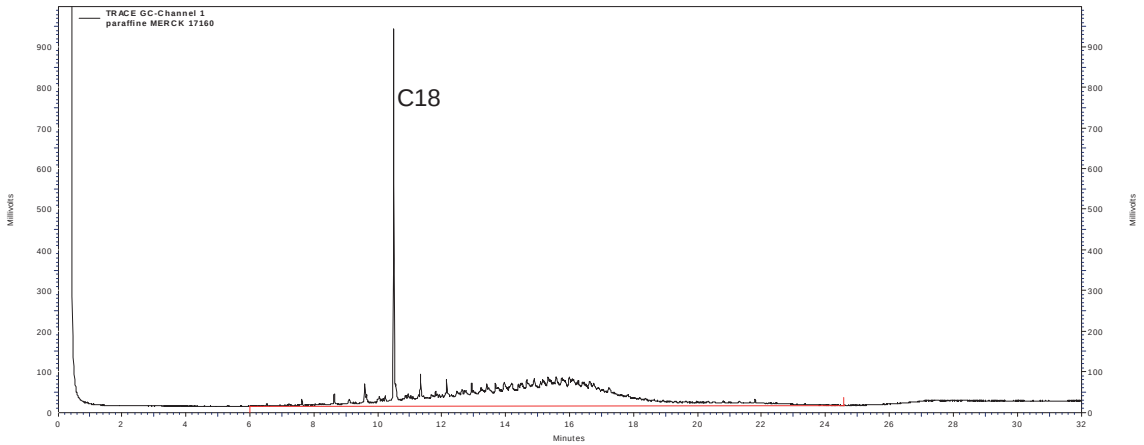
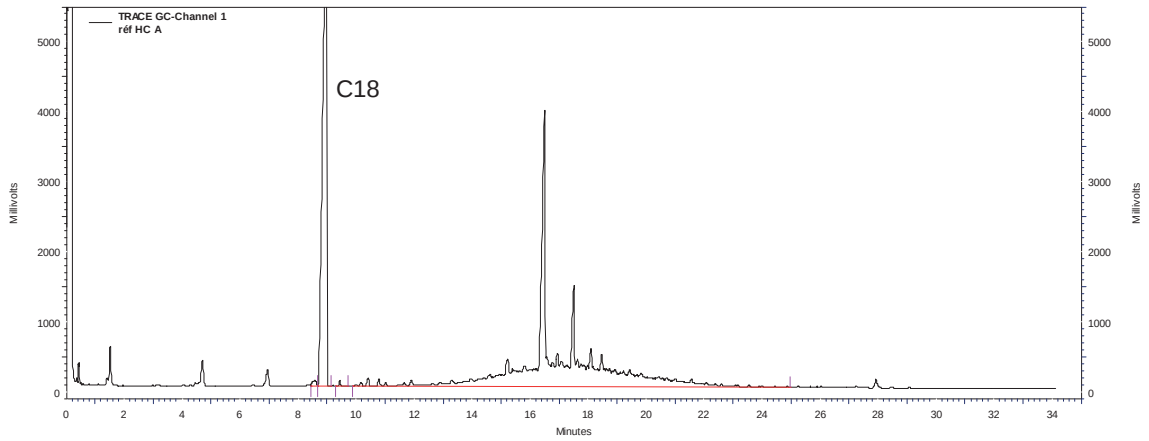
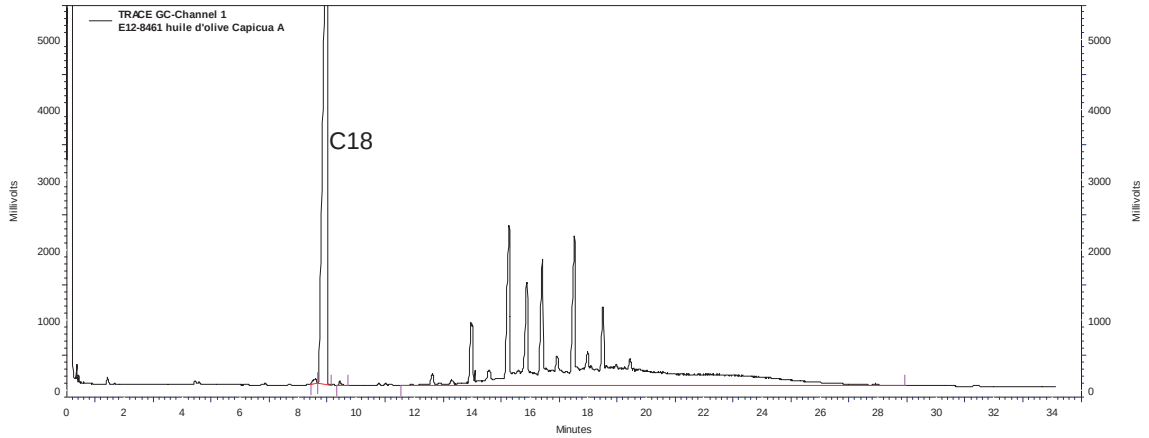


Figure A.5 — GC chromatogram of low viscosity paraffin — Internal standard: *n*-octadecane



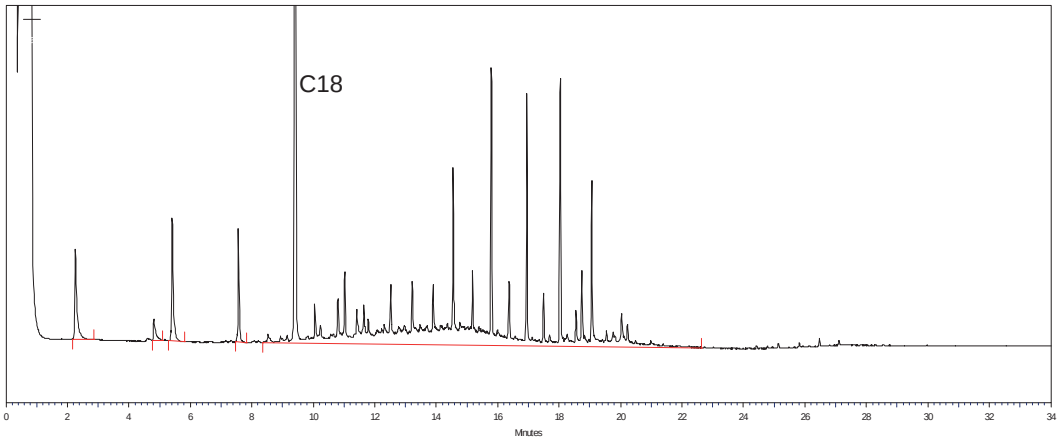
NOTE The baseline was manually traced from 10 min to 25 min to calculate the total hydrocarbon content. Internal standard: *n*-octadecane.

Figure A.6 — GC chromatogram of the aliphatic hydrocarbon fraction of contaminated crude sunflower oil (150 mg/kg) purified with silver nitrate impregnated silica gel



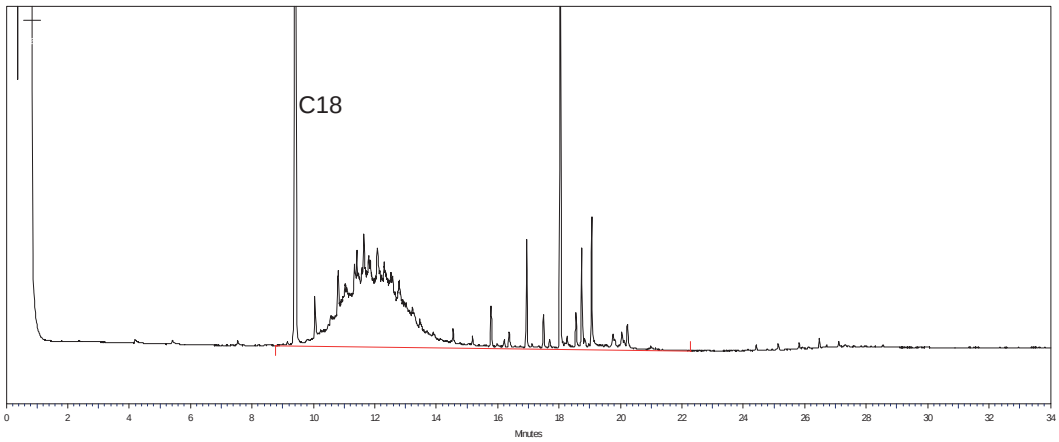
NOTE The baseline was manually traced from 11 min to 28 min to calculate the total hydrocarbon content. Internal standard: *n*-octadecane.

Figure A.7 — GC chromatogram of the aliphatic hydrocarbon fraction of a pomace olive oil purified with silver nitrate impregnated silica gel



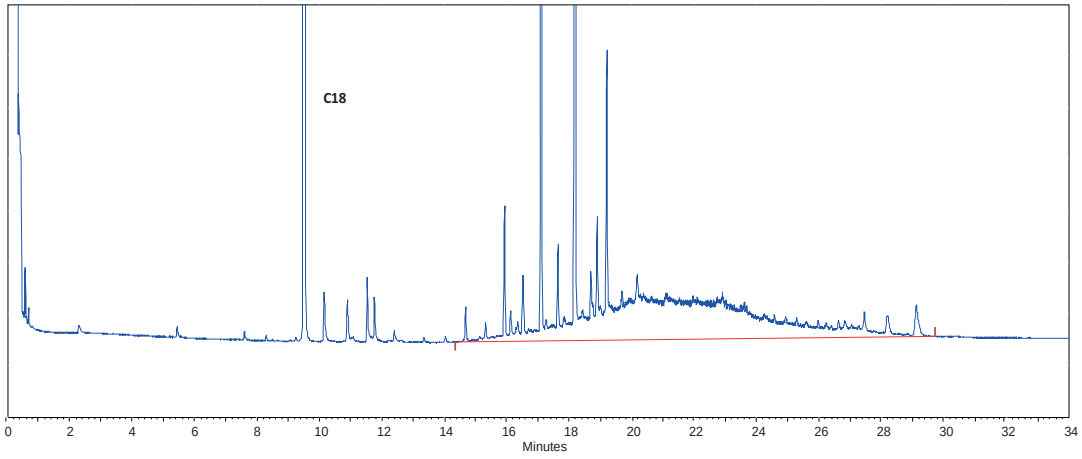
NOTE The baseline was manually traced from 8 min to 23 min to calculate the total hydrocarbon content. Internal standard: *n*-octadecane.

Figure A.8 — GC chromatogram of the aliphatic hydrocarbon fraction of a spiked virgin olive oil (50 mg/kg) purified with silver nitrate impregnated silica gel



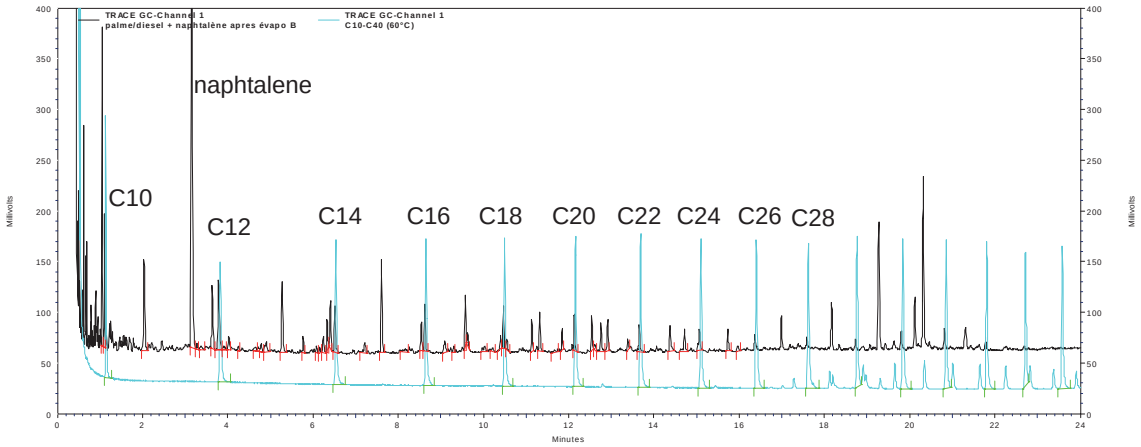
NOTE The baseline was manually traced from 9 min to 22 min to calculate the total hydrocarbon content. Internal standard: *n*-octadecane.

Figure A.9 — GC chromatogram of the aliphatic hydrocarbon fraction of a crude soybean oil (90 mg/kg) purified with silver nitrate impregnated silica gel



NOTE The baseline was manually traced from 9 min to 22 min to calculate the total hydrocarbon content. Internal standard: *n*-octadecane.

Figure A.10 — GC chromatogram of the aliphatic hydrocarbon fraction of a contaminated walnut oil purified with silver nitrate impregnated silica gel.



Key

in black GC chromatogram of the aliphatic hydrocarbon fraction of a crude palm oil contaminated with diesel oil purified with silver nitrate impregnated silica gel - Internal standard: naphthalene.

in blue GC chromatogram of an alkane standard mixture C10-C40.

Figure A.11 — Superposition of two GC chromatograms

Annex B
(informative)

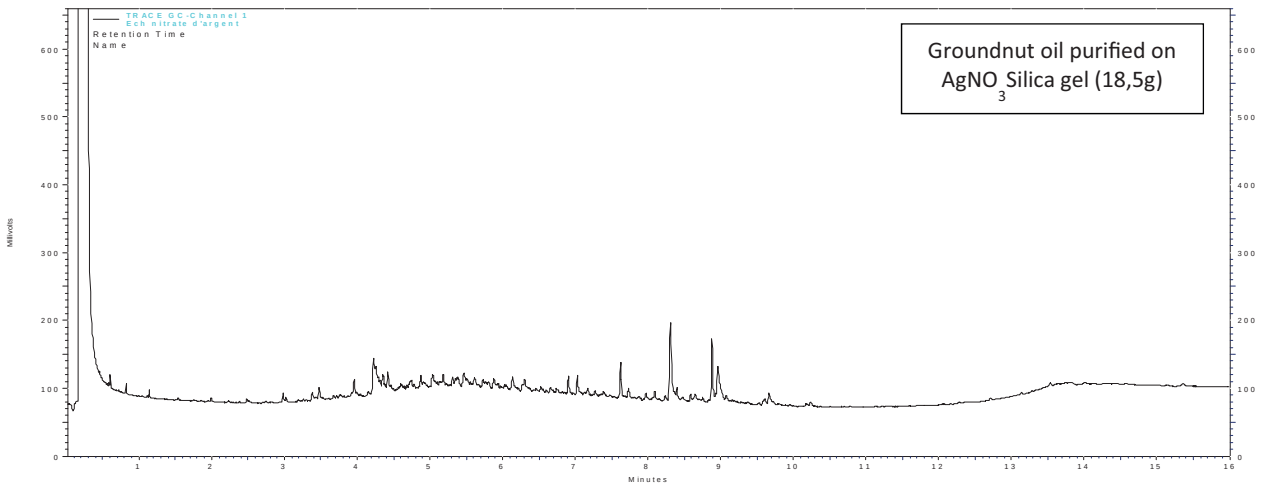
Validation of silver nitrate impregnated silica gel purification

A refined sunflower oil (REF) was spiked with an increasing amount of a viscous paraffin in order to verify the recovery using *n*-octadecane as the internal standard.

Recoveries were calculated for addition of mineral oil content between 10 mg/kg and 1 000 mg/kg and the recoveries were between 97 % and 120 %. For lower concentrations, a slight overestimation of 20 % is noticeable, which corresponds to 3 mg/kg.

Table B.1 — Recovery data for the addition of increasing mineral oil content in vegetable oil

Sample	Mineral oil content (mg/kg)	Recovery (%)
REF	11	
REF + 10 mg/kg	23	120
REF + 20 mg/kg	33	109
REF + 50 mg/kg	65	106
REF + 500 mg/kg	497	97
REF + 1000 mg/kg	1004	99



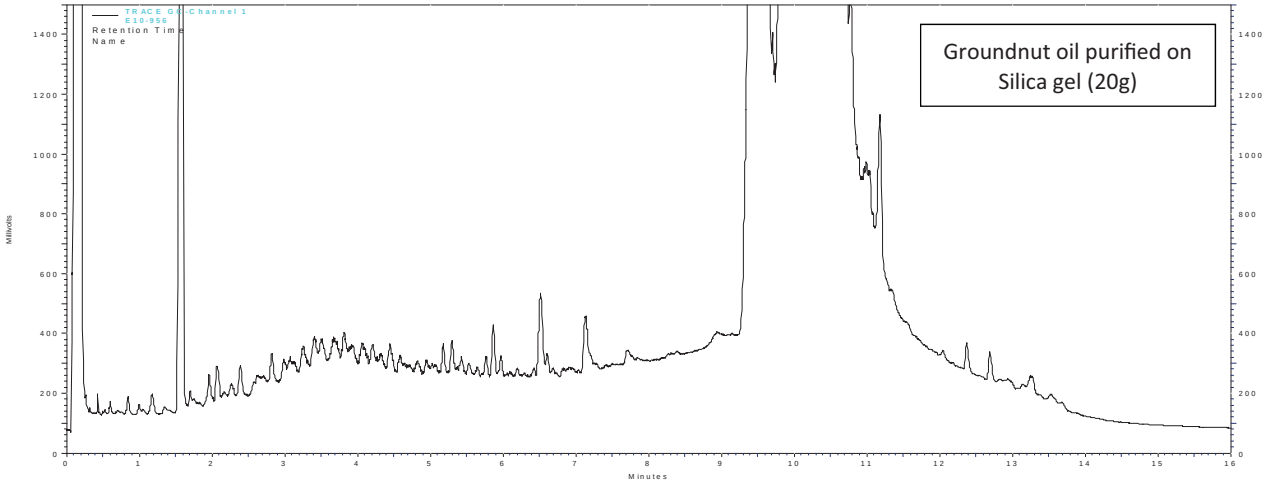


Figure B.1 — Chromatograms of crude vegetable oil for which silver nitrate impregnated silica gel purification is necessary

Annex C
(informative)

Procedure for rapid method

C.1 General

This modification generally can be used for the analysis of refined and virgin oils. For some samples of crude oil, this rapid method may not be satisfactory due to the lack of retention of triglycerides.

C.2 Reagents

C.2.1 Silica Gel 60[5], extra pure for column chromatography with particle size between 60 µm to 200 µm (70 mesh to 230 mesh).

Usually, silica gel can be used directly from the container without any treatment. However, some batches of silica show low activity resulting in poor chromatographic separations. Under these circumstances, the silica gel should be activated by heating for at least 4 h at 500 °C. After heating, place the silica gel in a desiccator to cool down to room temperature and then transfer the silica gel to a stoppered flask. Add 2 % of water and shake until no lumps can be seen and the powder flows freely and keep for 12 h before use.

C.2.2 *n*-Hexane, trace organic analysis grade, purity 99 % minimum, residue after evaporation maximum 2 mg/kg.

NOTE Hexane purity may be checked by concentrating 100 ml of *n*-hexane in a rotary evaporator, dissolving the residue in 0,5 ml of *n*-heptane or *n*-hexane and gas chromatography analysis.

C.2.3 Internal standard *n*-octadecane (C18), purity 99 % minimum.

C.2.4 *n*-Octadecane solution, mass concentration ρ = 0,16 mg/ml.

As an example, weigh to the nearest mg, approximately 40 mg of *n*-octadecane (C.2.3) and dilute to 25 ml with n-hexane (C.2.2), and then proceed with a second dilution of this mixture of 5 ml → 50 ml with *n*-hexane. This solution can be kept in the fridge for one month.

C.2.5 Carrier gas for gas chromatography, helium or hydrogen.

C.2.6 Auxiliary gases for flame ionization detector, hydrogen, air, nitrogen suitable for gas chromatography.

C.3 Apparatus

Usual laboratory apparatus and, in particular, the following.

The glassware used for the determination shall be thoroughly cleaned and rinsed with *n*-hexane (C.2.2) before use so that it is free from impurities.

5) Silica gel is available from Merck, reference 7754 or 7734. This reference is an example of a suitable product which is available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

C.3.1 Empty the glass column for solid phase extraction (SPE)[6], with glass fibre frits, 6 ml volume.

C.3.2 Precision pipette, 0,5 ml and 1 ml.

C.3.3 Volumetric flask, 5 ml.

C.3.4 Glass sample vials, 10 ml capacity.

C.3.5 Muffle furnace, suitable for temperatures up to 400 °C.

C.3.6 Gas chromatograph, suitable for use with capillary column, equipped with an on-column injector or equivalent device, a temperature-programmable oven and a flame ionization detector (FID).

C.3.7 Data acquisition system, with the possibility of manual integration.

C.3.8 Deactivated silica gel capillary pre-column, connected to the GC column, with no stationary phase, 5 m long, 0,53 mm internal diameter, capable of being programmed up to 400 °C ("high temperature" type).

C.3.9 Capillary column, capable of being programmed up to 400 °C ("high temperature" type) for which the following characteristics are advised: 95 % dimethyl/5 % diphenyl polysiloxane stationary phase, length 15 m or 10 m, internal diameter 0,32 mm or 0,25 mm, film thickness 0,1 µm.

C.3.10 Microsyringe, 100 µl capacity, suitable for on-column injection in gas chromatography.

C.3.11 Analytical balance, readability to 0,001 g.

C.3.12 Pasteur pipette, in glass.

Plastic Pasteur pipettes should be avoided. Polyethylene film should also be avoided.

C.4 Procedure

C.4.1 Into a beaker, weigh approximately 2 g of silica gel (C.2.1) and transfer the silica gel into the empty glass column (C.3.1).

C.4.2 Rinse the silica gel with another 2 ml to 3 ml of *n*-hexane (C.2.2).

C.4.3 Elute the solvent until the level of the solvent in the column is less than 0,5 cm higher than the silica gel bed.

C.4.4 Weigh to the nearest 1 mg, approximately 2,5 g of the sample into a 5 ml volumetric flask (C.3.3) and add 1 ml of the 0,16 mg/ml internal standard solution (C.2.4) with a precision pipette (C.3.2) and make up to the mark with *n*-hexane (C.2.2).

C.4.5 Transfer 500 µl of the solution (C.4.4) with a precision pipette (C.3.2) on top of the silica gel, in the SPE column (C.4.1).

---

6) Empty glass columns for solid phase extraction are available from Chromabond - Macherey-Nagel, ref 730172. This reference is an example of suitable products which are available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

C.4.6 Elute the solvent and rinse the SPE column with 4 × 500 µl of *n*-hexane (C.2.2) with a precision pipette (C.3.2) without allowing the silica gel to become dry.

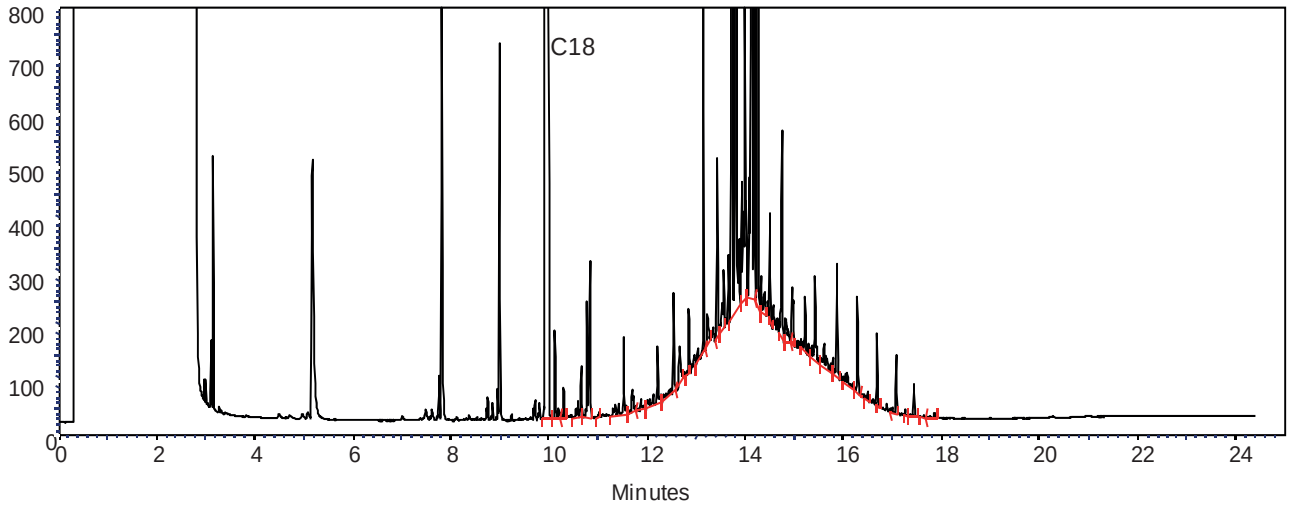
C.4.7 Elute the mineral oil fraction with 3,5 ml of *n*-hexane (C.2.2) in the 10 ml tube (C.3.4).

C.4.8 Analyse the mineral fraction by gas chromatography without any evaporation of the solvent. The chromatographic analysis conditions are to be chosen taking into account the characteristics of the column being used and the type of carrier gas. However, the temperature program of the GC oven must be rapid in order to observe the "hump" formed by the heavy mineral oil elution (see Figure C1 and Figure C2).

An example of analysis conditions is described below:

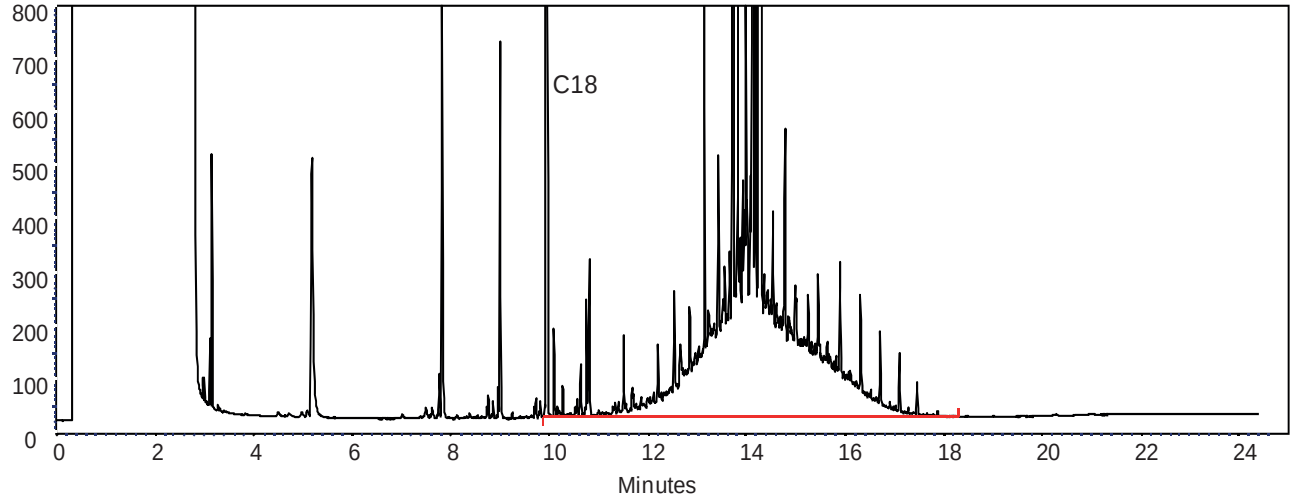
Large volume injection program:	7 s waiting after the introduction of the needle before the injection, speed injection at 5 µl/s, 7 s waiting before taking out the needle of the injector
Column temperature:	65 °C for 7 min, 65 °C up to 350 °C programmed at 25 °C/min, 350 °C up to 370 °C programmed at 5 °C/min, final temperature hold for 10 min
Detector temperature:	380 °C
Carrier gas pressure (hydrogen):	50 KPa
Volume injected:	45 µl of the mineral fraction (C.4.7)

To calculate the content of hydrocarbons of mineral origin, follow the standardized method already described.



NOTE The hydrocarbons of natural origin were determined by manually tracing the valley-to-valley baseline over the UCM profile.

Figure C.1 — GC chromatogram of the aliphatic hydrocarbons fraction of a spiked refined sunflower oil (150 mg/kg)



NOTE The baseline was traced from 10 min to 18 min to calculate the total hydrocarbon content.

Figure C.2 — GC chromatogram of the aliphatic hydrocarbons fraction of a spiked refined sunflower oil (150 mg/kg)

Annex D
(informative)

Fat extraction from food sample

D.1 General

This method specifies a method for fat recovery from a food sample by soxhlet extraction with a blend of solvents. The saturated aliphatic hydrocarbons of a portion of the fat sample are then analysed according to the method specified in this International Standard: isolation by liquid chromatography on silica gel impregnated with silver nitrate and determination by capillary gas chromatography with flame ionization detector using an internal standard.

D.2 Reagents

D.2.1 Celite 545, diatomaceous earth for filtration[7)].

D.2.2 Cotton wool, for laboratory use. In order to remove traces of mineral oil, pads of cotton wool shall be kept in a beaker full of *n*-hexane for one night prior to the extraction.

D.2.3 Cellulose thimble, fitting the extractor, wall thickness of approximately 1 mm. In order to remove traces of mineral oil that are often present in the cellulose thimble, all thimbles shall be kept in a beaker full of *n*-hexane for one night prior to the extraction.

D.2.4 Disodium sulfate, (Na_2SO_4), anhydrous, granular.

D.2.5 *n*-Hexane, trace organic analysis grade, purity 99 % minimum, residue after evaporation maximum 2 mg/kg.

NOTE 1 *n*-Hexane purity may be checked by concentrating 200 ml of *n*-hexane mixed with 1 ml of internal standard solution (C.2.4) using a rotary evaporator, dissolving the residue in 0,2 ml of *n*-hexane and the analysis of 5 µl by gas chromatography.

NOTE 2 *n*-Hexane may be replaced by isooctane, *n*-heptane or an alkane mixture of boiling point 65 °C to 70 °C, as long as the residue after evaporation is maximum 2 mg/kg. Solvents with higher boiling point than *n*-hexane take longer to evaporate. However, they are preferred due to the toxicity of hexane.

D.2.6 Propan-2-ol, trace organic analysis grade, purity 99 % min, residue after evaporation maximum 2 mg/kg.

NOTE Propan-2-ol purity may be checked by concentrating 200 ml of propan-2-ol mixed with 1 ml of internal standard solution (C.2.4) using a rotary evaporator, dissolving the residue in 0,2 ml of *n*-hexane and the analysis of 5 µl by gas chromatography.

D.2.7 Extraction solvent, mixture of *n*-hexane and propan-2-ol, volume concentrations are: σ(*n*-hexane) = 60 ml/100 ml, σ(propan-2-ol) = 40 ml/100 ml.

7) Celite 545 is available from VWR, reference 22552.290 or from Sigma-Aldrich, reference 419931. This reference is an example of suitable products which are available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

D.2.8 Internal standard *n*-octadecane (C18), purity 99 % minimum.

n-Octadecane may be replaced by *n*-eicosane (C20). Before choosing one of these two compounds as the internal standard, it should be verified that there is no co-elution with other peaks from the sample to be analysed.

n-Octadecane shall be replaced by naphthalene if the sample is contaminated with diesel oil, in order to avoid the overlapping of the internal standard peak with the alkane peaks to be quantified.

D.2.9 *n*-Octadecane solution, mass concentration ρ = 0,04 mg/ml.

As an example, weigh to the nearest mg, approximately 50 mg of *n*-octadecane (C.2.3) and dilute to 25 ml with n-hexane (C.2.2), and then proceed with a second dilution of this mixture of 1 ml → 50 ml with *n*-hexane. Store this solution at room temperature in order to maintain its stability.

D.2.10 Glass boiling chips, fat free.

D.3 Apparatus

Usual laboratory apparatus and, in particular, the following.

The glassware used for the determination shall be thoroughly cleaned and rinsed with *n*-hexane (C.2.2) before use so that it is free from impurities.

Plastic Pasteur pipettes shall be avoided. Polyethylene film shall also be avoided.

D.3.1 Precision pipette, 2 ml.

D.3.2 Analytical balance, with a mass resolution of 0,000 1 g.

D.3.3 Soxhlet apparatus, comprising the following: a heating mantle, a round bottom flask of 250 ml capacity, an extractor, a condenser.

Alternatively: **Automated Soxhlet extraction unit**, comprising the following: heating plate, beaker, an extractor and a condenser.

D.3.4 Rotary evaporator, with vacuum and a water bath at 35 °C (recommended). Care should be taken to prevent cross contamination. Clean the system thoroughly between determinations.

D.3.5 Conical glass sample vials, 10 ml capacity.

D.3.6 Spatula, metal.

D.4 Extraction of fat from food sample

D.4.1 Test portion preparation

As a general precaution, all of the sample material received by the laboratory shall be used for obtaining a representative and homogeneous laboratory sample without introducing secondary contamination. In all instances, if the sample has been frozen, allow it to completely thaw before homogenizing and sub-sampling.

Weigh with an analytical balance (D.3.2), 5 g ± 0,05 g of the homogenized test sample, into the Soxhlet thimble (D.3.3). Add 5 g of Celite (D.2.1) and 5 g of disodium sulfate (D.2.4) and mix with a metal spatula (D.3.6). The sample must be topped with a pad of cotton wool (D.2.2) so as to avoid sample losses during recondensation and reflux of the extraction solvent.

D.4.2 Soxhlet extraction

Place the thimble in the Soxhlet extraction chamber; 200 ml of the extraction solvent (D.2.7) shall be added in the 250 ml flask (D.3.3), as well as some boiling chips. The heating temperature and cooling have to be set so to obtain approximately 6 cycles per hour. The extraction time should not be less than 7 h.

For the automated Soxhlet extraction unit: Place the thimble in the Soxhlet extraction chamber; 130 ml of the extraction solvent (D.2.7) shall be added into the extraction beaker (D.3.3), as well as some boiling chips. Adjust the optical sensor to the sample amount. The heating program and cooling have to be set so to obtain approximately 6 cycles per hour. The extraction time should not be less than 7 h.

D.4.3 Evaporation of the solvent for extracts obtained with Soxhlet

The solvent is evaporated with a rotary evaporator (D.3.4) equipped with a water bath set to 35 C to dryness.

Add 2 ml of the *n*-octadecane solution (D.2.9) into the round bottom flask containing the extract solution and homogenize.

D.5 Purification of the fat extract

The method specified in this International Standard is followed by replacing 1 g of sample in 9.2, by 1 ml of the fat extract solution obtained in D.4.3.

Annex E
(informative)

Results of interlaboratory trials

In 2013, a first international collaborative test involving 37 laboratories in 12 countries was carried out on the following eight oil samples (A to H) with different contents. For information, the results on the two foodstuff samples (I and K) are also reported.

A - refined sunflower oil

B - virgin olive oil spiked with a liquid mineral oil at a level of 50 mg/kg

C - crude degummed soybean oil spiked with white oil at a level of 450 mg/kg

D - refined sunflower oil spiked with a viscous mineral oil at a level of 25 mg/kg

E - refined grapeseed oil

F - refined olive pomace oil

G - crude palm oil spiked with diesel oil at a level of 25 mg/kg

H - refined sunflower oil spiked with a viscous mineral oil at a level of 100 mg/kg

I - commercial 82 % fat margarine

K - homemade mayonnaise prepared with refined sunflower oil spiked with a viscous mineral oil at a level of 140 mg/kg

The test was organized by ITERG (France) and the results obtained were subjected to statistical analysis in accordance with ISO 5725-1[4] and ISO 5725-2[5] to give the precision data shown in Table E.1 and Table E.2.

Table E.1 — Summary of statistical results for sample A to sample E

Sample	A	B	C	D	E
Number of participating laboratories, n_P	35	36	36	36	36
Number of laboratories retained after eliminating outliers, n_p	28	31	32	31	31
Number of individual test results of all laboratories on each sample, n_z	56	62	64	62	62
Mean value, (m) mg/kg	**15,8**	**51,8**	**423,3**	**33,2**	**210,3**
Spiking value, mg/kg	-	50	450	25	-
Repeatability standard deviation, s_r, mg/kg	2,6	6,9	16,4	4,2	5,7
Repeatability coefficient of variation, CV(r), %	16,2	13,3	**3,9**	12,6	2,7
Repeatability limit, r, ($2{,}8s_r$), mg/kg	**7,2**	**19,3**	**45,8**	**11,7**	**16,0**
Reproducibility standard deviation, s_R, mg/kg	9,8	18,5	47,2	12,2	28,9
Reproducibility coefficient of variation, CV(R), %	61,9	35,7	11,1	36,6	13,8
Reproducibility limit, R, ($2{,}8s_R$), mg/kg	**27,3**	**51,7**	**132,1**	**34,0**	**81,0**

Table E.2 — Summary of statistical results for sample F to sample K

Sample	F	G	H	I	K
Number of participating laboratories, n_P	36	24	35	21	19
Number of laboratories retained after eliminating outliers, n_p	31	21	30	17	14
Number of individual test results of all laboratories on each sample, n_z	62	42	60	34	28
Mean value, (m) mg/kg	**162,9**	**24,1**	**105,0**	**29,5**	**147,9**
Spiking value, mg/kg	-	25	100	-	140
Repeatability standard deviation, s_r, mg/kg	9,0	3,0	5,5	3,9	8,4
Repeatability coefficient of variation, CV(r), %	5,5	12,3	5,2	13,2	5,7
Repeatability limit, r, ($2{,}8s_r$), mg/kg	**25,1**	**8,3**	**15,3**	**10,9**	**23,4**
Reproducibility standard deviation, s_R, mg/kg	25,3	15,6	16,9	22,7	23,5
Reproducibility coefficient of variation, CV(R), %	15,6	64,7	16,1	77,0	15,9
Reproducibility limit, R, ($2{,}8s_R$), mg/kg	**71,0**	**43,7**	**47,4**	**63,5**	**65,9**

In 2014, a second international collaborative test involving 21 laboratories in nine countries was carried out on the following six oil samples (I to N) with different contents.

I - pomace olive oil

J - refined rapeseed oil

K - 100 mg/kg spiked refined sunflower oil

L - 50 mg/kg spiked virgin olive oil

M - 25 mg/kg spiked refined sunflower oil

N - crude soybean oil

The test was organized by ITERG (France) and the results obtained were subjected to statistical analysis in accordance with ISO 5725-1[4] and ISO 5725-2[5] to give the precision data shown in Table E.3.

Table E.3 — Summary of statistical results for sample I to sample N

Sample	I	J	K	L	M	N
Number of participating laboratories, n_P	20	20	20	20	20	20
Number of laboratories retained after eliminating outliers, n_p	18	18	18	18	17	18
Number of individual test results of all laboratories on each sample, n_z	36	36	36	36	34	36
Mean value, (m) mg/kg	**118,8**	**11,1**	**107,1**	**52,1**	**37,5**	**89,9**
Spiking value, mg/kg	-	-	100	50	25	-
Repeatability standard deviation, s_r, mg/kg	4,4	3,1	3,7	5,5	2,7	5,0
Repeatability coefficient of variation, CV(r), %	3,7	28,1	3,4	10,5	7,2	5,6
Repeatability limit, r, ($2{,}8s_r$), mg/kg	**12,4**	**8,7**	**10,3**	**15,3**	**7,6**	**14,0**
Reproducibility standard deviation, s_R, mg/kg	14,8	8,2	11,2	10,0	9,6	9,2
Reproducibility coefficient of variation, CV(R), %	12,4	73,9	10,4	19,3	25,7	10,3
Reproducibility limit, R, ($2{,}8s_R$), mg/kg	**41,4**	**22,9**	**31,2**	**28,1**	**27,0**	**25,8**

Bibliography

[1] MCGILL A.. MOFFAT C.F., MACKIE P.R., CRUICKSHANK P., The composition and concentration of n-alkanes in retail samples of edible oils. J. Sci. Food Agric. 1993, **61** pp. 357–362

[2] http://www.efsa.europa.eu/en/data/call/datex100806.htm - Call for scientific data on Mineral Oil hydrocarbons in food

[3] ISO 5555, *Animal and vegetable fats and oils — Sampling*

[4] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[5] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*

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