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**Derivati maščob in olj - Metil estri maščobnih kislin (FAME) - Določevanje  
kislinskega števila**

Fat and oil derivatives - Fatty acid methyl ester (FAME) - Determination of acid value

Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester  
(FAME) - Bestimmung der Säurezahl

Produits dérivés des corps gras - Esters méthyliques d'acides gras (EMAG) -  
Détermination de l'indice d'acide

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Rastlinske in živalske  
maščobe in olja

Animal and vegetable fats  
and oils

**oSIST prEN 14104:2020**

**en,fr,de**



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English Version

**Fat and oil derivatives - Fatty acid methyl ester (FAME) -  
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Produits dérivés des corps gras - Esters méthyliques  
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Ölen - Fettsäure-Methylester (FAME) - Bestimmung  
der Säurezahl

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 307.

If this draft becomes a European Standard, CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
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EUROPÄISCHES KOMITEE FÜR NORMUNG

**CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels**

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## European foreword

This document (prEN 14104:2019) has been prepared by Technical Committee CEN/TC 307 “Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis”, the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 14104:2003.

In comparison with the previous edition, the following technical modifications have been made:

- a) chapter 5 “Reagents” revised for clarification;
- b) automated titration system including optical detection added as alternative;
- c) document revised editorially.

This document has been prepared under mandate M/245 on Fatty Acid Methyl Ester (FAME) given to CEN by the European Commission and the European Free Trade Association.

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## Introduction

This document is based on EN ISO 660 [1] which was specifically adapted for the determination of acid value of fatty acid methyl esters (FAME).

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## 1 Scope

This document specifies a titrimetric method for the determination of acid value in light coloured Fatty Acid Methyl Esters, hereinafter referred as FAME.

It allows the determination of acid value within a range of 0,10 mg KOH/g to 1,00 mg KOH/g.

NOTE For the purposes of this document, the terms “% (m/m)” and “% (V/V)” are used to represent respectively the mass fraction and the volume fraction.

WARNING — The use of this document can involve hazardous materials, operations and equipment. This document does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this document to take appropriate measures to ensure the safety and health of personnel prior to the application of the standard, and to determine the applicability of any other restrictions for this purpose.

## 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 661, *Animal and vegetable fats and oils - Preparation of test sample (ISO 661:2003)*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

## 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:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <http://www.iso.org/obp>

### 3.1

#### acid value

number of milligrams of potassium hydroxide required to neutralise the free fatty acids present in 1 g of FAME, when determined in accordance with the procedure specified in this document

Note 1 to entry: The acid value is expressed in milligrams of potassium hydroxide per gram of sample.

Note 2 to entry: If the sample contains mineral acids these are determined as a part of total acid value. This method does not allow distinguishing between weak (from free fatty acids) and strong (from mineral acids, if present) acidity.

## 4 Principle

A test portion is dissolved in a suitable solvent mixture and titrated with a standardized solution of potassium hydroxide, using phenolphthalein as an indicator in order to detect the titration end point.

## 5 Reagents

Use only reagents of recognized analytical grade and water of grade 3 in accordance with EN ISO 3696.

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**5.1 Toluene.****5.2 Isopropanol.****5.3 Solvent A** for solvent mixture (5.5), **ethanol**, 95 % (V/V) - 97 % (V/V) purity.**5.4 Solvent B** for solvent mixture (5.5), **diethyl ether**, peroxide free, stabilized with butylated hydroxytoluene (BHT).**5.5 Solvent mixture**, mixture of equal volumes of solvent A (5.3) and solvent B (5.4).

Neutralize, just before use, by adding the potassium hydroxide solution (5.2) in the presence of 0,3 ml of phenolphthalein alcoholic solution (5.9) per 100 ml of solvent mixture.

If it is not possible to use diethyl ether, a mixed solvent may be used as follows:

- toluene (5.1) and ethanol (5.3), 1 + 1 mixture by volume;
- toluene (5.1) and isopropanol (5.2), 1 + 1 mixture by volume;

The mixed solvent can be replaced by isopropanol (5.2)

NOTE Larger volumes of solvent mixture and indicator can be necessary for dark-coloured samples.

**5.6 Phthalic acid monopotassium salt**, (>99,5 %) maintained 2 h in an oven at 120 °C and cooled down in a desiccator before use, primary standard quality.**5.7 Benzoic acid**, (>99,5 %) to be dried before use by careful fusion in a platinum crucible at approximately 130 °C, then powdered in a mortar. Store in a desiccator until use, primary standard quality.**5.8 Potassium hydroxide**, standard volumetric solution in ethanol.

$$c(\text{KOH}) = 0,1 \text{ mol/l}$$

The ethanolic potassium hydroxide solution can be replaced by aqueous potassium or sodium hydroxide solutions, but only if the volume of water introduced does not lead to phase separation during titration.

Isopropanol can be used instead of ethanol.

**5.8.1 Preparation of the solution**

Dissolve approximately 6,6 g of potassium hydroxide pellets in ethanol and dilute to 1 000 ml with the same solvent.

The same procedure can be followed when water instead alcohol is used.

Use solution prepared at least 5 days previously and decanted into a brown glass bottle, fitted with a rubber stopper. The solution shall be colourless or straw yellow. If the bottle is connected to the burette, provision shall be made to prevent intake of carbon dioxide, for example by using a tube filled with granular soda lime.

### 5.8.2 Control of the solution concentration

Weigh, to the nearest 0,000 1 g, approx. 180 mg of phthalic acid monopotassium salt (5.6) or approximately 100 mg of benzoic acid (5.7), in a 300 ml flask and dissolve in 100 ml of distilled water. Carry out titration using KOH 0,1 M solution (alcoholic or water solution), using phenolphthalein solution (5.9) as an indicator. At the end point the colour of solution turns to a persisting pink.

Phthalic acid monopotassium salt and benzoic acid dissolve slowly in water: carefully check that the sample is completely dissolved before starting the titration.

The concentration of the potassium hydroxide solution, expressed in moles per litre, (when phthalic acid monopotassium salt is used), is given by:

$$M_{\text{KOH}} = (m_{\text{pH}} / 204,23) \times (1 / V_{\text{KOH}})$$

The concentration of the potassium hydroxide solution, expressed in moles per litre, (when benzoic acid is used), is given by:

$$M_{\text{KOH}} = (m_{\text{benz}} / 122,12) \times (1 / V_{\text{KOH}})$$

where

$m_{\text{pH}}$  is the mass, reported in mg, of phthalic acid monopotassium salt used;

$m_{\text{benz}}$  is the mass, reported in mg, of benzoic acid used;

$V_{\text{KOH}}$  is the volume, in ml, of potassium hydroxide solution used to reach the end point.

NOTE 1 In the above and later mathematical expressions, the symbol represents the numerical values of the quantities.

NOTE 2 Potassium hydroxide standardized solutions are commercially available and suitable for use.

### 5.9 Phenolphthalein, 10 g/l solution in ethanol (5.3).

NOTE Phenolphthalein alcoholic solutions are commercially available and suitable for use.

## 6 Apparatus

**6.1 Burette**, 10 ml capacity, graduated in 0,02 ml subdivisions, EN ISO 385 class A [2].

**6.2 Analytical balance**, capable of weighing with an accuracy of  $\pm 0,001$  g or better

## 7 Sampling

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

It is important that a laboratory receives a sample, which is truly representative and has not been damaged or changed during transportation and storage.

## 8 Preparation of the test sample

Prepare the test sample in accordance with EN ISO 661. The test sample shall not be heated and/or filtered.

## 9 Procedure

### 9.1 Test portion

Weigh approximately 20 g (accuracy  $\pm 0,001$  g) of the test sample into a 250 ml conical flask.

### 9.2 Determination

**9.2.1** Dissolve the test portion (9.1) in 100 ml of previously neutralized solvent mixture (5.5).

After the addition of some drops of phenolphthalein solution (5.9) start the titration whilst stirring the solution, using the potassium hydroxide solution (5.8) to the end point when the addition of a single drop produces a slight, but definite colour change persisting for at least 15 s.

An automated titration system including optical detection to detect the end point may also be used provided it has been validated to meet the precision values specified in Clause 11.

**9.2.2** If the solution becomes turbid during titration, add a sufficient quantity of the mixed solvent (5.5) to give a clear solution.

## 10 Calculation

The acid value  $AV$  is reported as:

$$AV = \frac{56,1 \times V \times c}{m}$$

where

$V$  is the volume, in ml, of the standard volumetric potassium hydroxide solution used;

$c$  is the concentration, in mol/l, of the standard volumetric potassium hydroxide solution used;

$m$  is the mass, in g, of the test portion;

56,1 is the molecular mass of potassium hydroxide.

Results are reported as mg KOH/g sample and shall be rounded to the second decimal digit.

## 11 Precision

### 11.1 Interlaboratory test

Details of interlaboratory test are given in Annex A. The values derived from these tests may not be applicable to concentration ranges and matrices other than those given.

### 11.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, shall not be greater than 0,02 mg KOH/g more than once out of 20 determinations.

### 11.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, shall not be greater than 0,06 mg KOH/g more than once out of 20 determinations.