



# SLOVENSKI STANDARD SIST EN 14111:2003

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Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of iodine value

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

**iTeh STANDARD PREVIEW**

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

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Ta slovenski standard je istoveten z: **EN 14111:2003**

**ICS:**

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EUROPEAN STANDARD  
NORME EUROPÉENNE  
EUROPÄISCHE NORM

**EN 14111**

April 2003

ICS 67.200.10

English version

## Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of iodine value

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

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

This European Standard was approved by CEN on 2 January 2003.

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. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and United Kingdom.

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EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: rue de Stassart, 36 B-1050 Brussels

**EN 14111:2003 (E)****Foreword**

This document (EN 14111:2003) 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 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 October 2003, and conflicting national standards shall be withdrawn at the latest by October 2003.

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.

Annex A is informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Luxembourg, Malta, Netherlands, Norway, Portugal, Slovakia, Spain, Sweden, Switzerland and the United Kingdom.

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## Introduction

This European Standard is based on the EN ISO 3961 [1] which was specifically adapted for the determination of iodine value of fatty acid methyl esters (FAME).

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## EN 14111:2003 (E)

### 1 Scope

This European Standard specifies a titrimetric method for the determination of iodine value in Fatty Acid Methyl Esters, hereinafter referred as FAME.

### 2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN ISO 661:1995, *Animal and vegetable fats and oils — Preparation of test sample (ISO 661:1989)*.

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*.

### 3 Terms and definitions

For the purposes of this European Standard, the following term and definition apply.

#### 3.1 iodine value

mass of halogen, expressed as iodine, absorbed by the test portion when determined in accordance with the procedure specified in this European Standard, divided by the mass of the test portion

Iodine value is reported as grams of iodine per 100 g of FAME.

### 4 Principle

A test portion is dissolved in a mixed solvent and then Wijs reagent is added. After a specified time, potassium iodide and water are added to the sample and the liberated iodine is titrated using a sodium thiosulfate standardized solution.

### 5 Reagents

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

**5.1 Potassium iodide (KI)**, 100 g/l aqueous solution free from iodate and iodine.

**5.2 Starch solution**

Mix 5 g of soluble starch in 30 ml of water and add to 1 000 ml of boiling water. Boil for 3 min and let stand to cool.

**5.3 Sodium thiosulfate**, standard volumetric solution in water,  $c(\text{Na}_2\text{S}_2\text{O}_3 \bullet 5\text{H}_2\text{O}) = 0,1 \text{ mol/l}$  standardized not more than seven days before use.

**5.4 Solvent**, prepared by mixing equal volumes of cyclohexane and glacial acetic acid.

**5.5 Wijs reagent**, containing iodine monochloride in acetic acid.

The I/Cl ratio of Wijs reagent shall be within the limits  $1,10 \pm 0,1$ .

NOTE Commercially available Wijs reagent can be used.

## 6 Apparatus

Usual laboratory equipment and, in particular:

- 6.1 **Glass weighing scoops**, suitable for the test portion and for inserting into the flask (6.2).
- 6.2 **Conical flasks**, 500 ml capacity, fitted with ground glass stoppers and completely dried.
- 6.3 **Analytical balance**, capable of weighing with an accuracy of  $\pm 0,001$  g or less.

## 7 Sampling

Sampling is not part of the method specified in this European Standard. A recommended sampling method is given in EN ISO 5555 [2].

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

## 8 Preparation of test sample

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

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## 9 Procedure

### 9.1 Test portion

Take approximately 0,13 g to 0,15 g of the test sample, weighed to the nearest 0,001 g in a glass weighing scoop (6.1).

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NOTE In the case of an iodine value below 100, the mass of the test portion should be adjusted according to clause 9 of EN ISO 3961 [1].

### 9.2 Determination

**9.2.1** Place the glass weighing scoop containing the test portion in a 500 ml flask (6.2) and dissolve using 20 ml of solvent (5.4). Add 25 ml of Wijs reagent using a precision pipette. Insert the stopper, swirl carefully and place the flask in the dark.

**CAUTION — Do not use a mouth pipette for the Wijs reagent.**

**9.2.2** Prepare a blank with solvent and reagent as in 9.2.1 but omitting the test portion.

**9.2.3** Leave the flask in the dark for 1 h.

**9.2.4** At the end of the reaction time add 20 ml of potassium iodide solution (5.1) and 150 ml of water.

Titrate with standard sodium thiosulfate solution (5.3) until the yellow colour due to iodine has almost disappeared. Add few drops of the starch solution (5.2) and continue the titration until the blue colour just disappears after very vigorous shaking. Potentiometric determination of end point is also permissible.

**9.2.5** Carry out a blank test using the blank solution (9.2.2) concurrently.

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**10 Calculation - Iodine value**

The iodine value reported in g of iodine/ 100 g of FAME, is given by the following equation:

$$\frac{12,69 \times c \times (V_1 - V_2)}{m}$$

where

$c$  is the exact concentration, in moles per litre, of the standard volumetric sodium thiosulfate solution used;

$V_1$  is the volume, in millilitres, of standard volumetric sodium thiosulfate solution (5.3) used for blank test;

$V_2$  is the volume, in millilitres, of standard volumetric sodium thiosulfate solution (5.3) used for sample titration;

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

Results shall be expressed to the nearest 1 g of iodine/100 g.

**11 Precision****11.1 Interlaboratory test**

Details of interlaboratory tests 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**

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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 3 g of iodine ( $I_2$ )/100 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 5 g of iodine ( $I_2$ )/100 g more than once out of 20 determinations.



## 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 European standard;
- all operating details not specified in this European Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);
- the test result(s) obtained, or if the repeatability has been checked, the final quoted result obtained.

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