SLOVENSKI STANDARD
SIST EN 14111:2022

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Nadomešča:
SIST EN 14111:2003

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

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

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

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67.200.10	Rastlinske in živalske maščobe in olja	Animal and vegetable fats and oils

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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

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

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European foreword

This document (EN 14111:2022) 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of an identical text or by endorsement, at the latest by December 2022, and conflicting national standards shall be withdrawn at the latest by December 2022.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 supersedes EN 14111:2003.

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

— glass weighing scoops should no longer be inserted into the flask.

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

Any feedback and questions on this document should be directed to the users’ national standards body. A complete listing of these bodies can be found on the CEN website.

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Introduction

This document is based on EN ISO 3961 [1] which was specifically adapted for the determination of iodine value of Fatty Acid Methyl Esters (FAME).

1 Scope

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

The precision statement of this test method was determined in a Round Robin exercise with iodine values in the range 111 g iodine/100 g to 129 g iodine/100 g.

The test method is also applicable for lower iodine values; however, the precision statement is not established for iodine values below 111 g iodine/100 g.

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 document, 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)*

EN ISO 3170, *Petroleum liquids - Manual sampling (ISO 3170)*

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

EN ISO 5555, *Animal and vegetable fats and oils - Sampling (ISO 5555)*

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 https://www.electropedia.org/

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

3.1
iodine value
mass of iodine, absorbed by the test portion when determined in accordance with the procedure specified in this document, divided by the mass of the test portion

Note 1 to entry: The iodine value is reported as grams of iodine per 100 g of FAME.

4 Principle

A test portion is dissolved in a suitable solvent mixture 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 recognized 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, freshly prepared at the day of the test.

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. Without added preservatives it should be daily prepared.

5.3 Sodium thiosulfate, standard volumetric solution in water, $c(Na_2S_2O_3 \cdot 5H_2O)$ = 0,1 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. Commercially available Wijs reagent shall be used.

6 Apparatus

Usual laboratory equipment and, in particular:

6.1 (optional) **Glass weighing scoops,** suitable for the test portion.

6.2 Erlenmeyer flasks, 500 ml capacity, fitted with ground glass stoppers and completely dried.

6.3 Precision pipette, 25 ml capacity.

6.4 Burette, 50 ml capacity, graduated in 0,1 ml, ISO 385 class A. Automatic devices with an equivalent precision are allowed.

6.5 Analytical balance, capable of weighing with an accuracy of ± 1 mg or better.

7 Sampling

Unless otherwise specified, sampling shall be conducted according to EN ISO 3170 or EN ISO 5555.

8 Preparation

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 between 0,13 g and 0,15 g (accuracy ± 0,001 g) of the test sample in a flask (6.2).

The use of a glass weighing scoop (6.1) is allowed.

In the case of an iodine value below 100, the mass of the test portion should be adjusted according to EN ISO 3961.

The mass of test portion shall be adjusted when iodine value is below 100 g iodine/100 g.

9.2 Determination

9.2.1 If applicable, transfer the test portion from the weighing scoop to a 500 ml flask (6.2) using 20 ml of solvent (5.4).

Add 25 ml of Wijs reagent (5.5) using a precision pipette (6.3). Insert the stopper, swirl carefully and place the flask in the dark.

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.

A potentiometric determination of the end point is also permissible.

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

10 Calculation

Calculate the iodine value in g of iodine/ 100 g of FAME, using 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 (5.3) 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.

11 Expression of results

Report the iodine value rounded to the nearest 1 g of iodine/100 g.

12 Precision

12.1 General

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.

12.2 Repeatability

The difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, within a short interval of time, under the same test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, shall not be greater than 3 g of iodine/100 g.

12.3 Reproducibility

The difference between two independent results obtained in the normal and correct operation of the same method, for test material considered to be the same, under different test conditions, that is expected to be exceeded with a probability of 5 % due to random variation, shall not be greater than 5 g of iodine/100 g.

13 Test report

The test report shall contain the following information:

a) a reference to this document, i.e. EN 14111:2022;

b) the type and complete identification of the product tested;

c) the method of sampling used;

d) the result of the test (see Clause 11);

e) any deviation, by agreement or otherwise, from the procedures specified;

f) the date of the test.