SLOVENSKI STANDARD
SIST EN 14104:2003

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Derivati maġļ ob in olj - Metil estri maġļ obnih kislin (FAME) - Doloļ evanje kislinskega ġtevila

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

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

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

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

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Foreword

This document (EN 14104:2003) 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of an identical text or by endorsement, at the latest by October 2003 and conflicting national standards shall be withdrawn at the latest by October 2003.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 Methylester (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.

Introduction

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

1 Scope

This European Standard specifies one 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.

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, *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
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 European Standard

Acid value is expressed in milligrams of potassium hydroxide per gram of sample.

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

4 Principle

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

5 Reagents

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

5.1 Diethyl ether and 95 % **ethanol**, 1 + 1 mixture by volume.

WARNING Diethyl ether is very flammable and may form explosive peroxides. Use with great caution.

Neutralise, just before use, by adding the potassium hydroxide solution (5.2) in the presence of 0,3 ml of phenolphthalein alcoholic solution (5.3) per 100 ml of solvent mixture.

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

— toluene and 95 % (*V/V*) ethanol, 1 + 1 mixture by volume;

— toluene and 99 % (*V/V*) 2-propanol, 1 + 1 mixture by volume.

The mixed solvent can be replaced by 99 % (*V/V*) 2 propanol.

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

5.2 Potassium hydroxide, standard volumetric solution in ethanol:

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

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.

5.2.1 Preparation of the solution

Dissolve approximately 7 g of potassium hydroxide pellets in ethanol and dilute to 1 000 ml with the same solvent.

NOTE 2 propanol can be used instead of ethanol.

Weigh, to the nearest 0,0002 g, 0,15 g of benzoic acid having minimum purity 99,9 % (*m/m*), or another primary standard, in a 150 ml beaker and dissolve in 50 ml of 4-methylpentan-2-one (5.4). Use a pH meter to follow titration and to detect the end point, start the stirrer and titrate with the potassium hydroxide solution to the equivalence point.

5.2.2 Calculation of the solution concentration

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

$$\frac{1\,000 \times m_0}{122{,}1 \times V_0}$$

where

m_0 is the mass, reported in grams, of benzoic acid used;

V_0 is the volume, in millilitres, of potassium hydroxide solution used to reach the endpoint.

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

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

Use solution prepared at least 5 days previously and decanted into a brown glass bottle, fitted with a rubber stopper provided with a thermometer needed for temperature correction (see 10). 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.

5.3 Phenolphthalein, 10 g/l solution in 95 % Ethanol.

5.4 4-Methylpentan-2-one neutralized just before use by adding the potassium hydroxide solution (5.2) using the pH-meter (6.3).

6 Apparatus

Usual laboratory equipment and, in particular:

6.1 Microburette, 10 ml capacity, graduated in 0,02 ml subdivisions.

6.2 Analytical balance, capable of weighing with an accuracy of ± 0,05 g or less.

6.3 **pH-meter**, equipped with glass and calomel electrodes.

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 a 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.

9 Procedure

9.1 Test portion

Take approximately 20 g of the test sample.

Weigh the test portion into a 250 ml conical flask.

9.2 Determination

9.2.1 Dissolve the test portion (9.1) in 100 ml of previously neutralised solvent mixture (5.1).

Titrate, whilst swirling the solution, with the potassium hydroxide solution (5.2) to the endpoint when the addition of a single drop produces a slight, but definite colour change persisting for at least 15 s.

9.2.2 If the solution becomes turbid during titration, add a sufficient quantity of the mixed solvent (5.1) to give a clear solution.

10 Calculation

The acid value is reported as:

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

where

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

c is the exact concentration, in moles per litre, of the standard volumetric potassium hydroxide solution used;

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

56,1 is the molecular mass of potassium hydroxide.

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

NOTE The concentration of the ethanolic sodium or potassium hydroxide solution varies with temperature and it may be useful to use the following correction:

$$V' = V_t \times [1 - 0{,}0011 \times (t - t_0)]$$

where

V' is the corrected volume, in millilitres, of the standard sodium or potassium hydroxide solution;

V_t is the volume, in millilitres, of the standard sodium or potassium hydroxide solution measured at temperature t;

t is the temperature at which the determination was carried out, in degrees Celsius;

t_0 is the temperature, in degrees Celsius, at which the concentration of the standard sodium or potassium hydroxide solution was determined.

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.

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.