SLOVENSKI STANDARD
SIST EN 14112:2003

01-november-2003

Derivati maġļ ob in olj - Metil estri maġļ obnih kislin (FAME) - Doloļ evanje oksidativne stabilnosti (metoda s pospeġeno oksidacijo)

Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of oxidation stability (accelerated oxidation test)

Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester (FAME) - Bestimmung der Oxidationsstabilität (beschleunigter Oxidationstest)

Produits dérivés des corps gras - Esters méthyliques d'acides gras (EMAG) - Détermination de la stabilité a l'oxydation (Essai d'oxydation accélérée)

Ta slovenski standard je istoveten z: **EN 14112:2003**

ICS:

67.200.10	Rastlinske in ġivalske maġļ obe in olja	Animal and vegetable fats and oils

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English version

Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of oxidation stability (accelerated oxidation test)

Produits dérivés des corps gras - Esters méthyliques d'acides gras (EMAG) - Détermination de la stabilité à l'oxydation (essai d'oxydation accélérée)

Erzeugnisse aus pflanzlichen und tierischen Fetten un Ölen - Fettsäure-Methylester (FAME) - Bestimmung der Oxidationsbeständigkeit (Beschleunigte Oxydationsprüfung)

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

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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.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (EN 14112:2002) 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of an identical text or by endorsement, at the latest by October 2003, and conflicting national standards shall be withdrawn at the latest by October 2003.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.Annexes A and B are 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 ISO 6886 [1], which was specifically adapted for the determination of oxidation stability of fatty acid methyl esters (FAME).

1 Scope

This European Standard specifies a method for the determination of the oxidation stability of fatty acid methyl esters (FAME) at 110 °C.

2 Terms and definitions

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

2.1
induction period
time which passes between the moment when the measurement is started and the moment when the formation of oxidation products rapidly begins to increase

2.2
oxidation stability
induction period determined according to the procedure specified in this European Standard. Oxidation stability is expressed in hours

3 Principle

A stream of purified air is passed through the sample which has been brought to a specified temperature. The vapours released during the oxidation process, together with the air, are passed into a flask containing water which has been demineralized or distilled and contains an electrode for measuring the conductivity. The electrode is connected to a measuring and recording device. It indicates the end of the induction period when the conductivity begins to increase rapidly. This accelerated increase is caused by the dissociation of volatile carboxylic acids produced during the oxidation process and absorbed in the water.

4 Reagents and materials

Use only reagents of recognised analytical grade, and distilled or demineralized water.

4.1 Molecular sieve, with moisture indicator, pore size 0,3 mm. The molecular sieve should be dried in an oven set at 150 °C and cooled down to room temperature in a desiccator.

4.2 Acetone.

4.3 Alkaline laboratory glass cleaning solution.

4.4 Glycerol.

5 Apparatus

Usual laboratory equipment and, in particular, the following.

5.1 Appliance for the determination of oxidation stability

See Figures 1 and 2 for diagrammatic representations.

NOTE An appliance for determining oxidation stability can be obtained commercially under the trade name Rancimat, model 743, from Methrom AG, Herisau, Switzerland[1)].

1) Rancimat, model 743, is an example of suitable equipment available commercially. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of this equipment.

5.1.1 Air filter, comprising a tube fitted with filter paper at the ends and filled with a molecular sieve (4.1), connected to the suction end of a pump.

5.1.2 Gas diaphragm pump, with an adjustable flow rate of 10 l/h in combination with an apparatus to control the flow rate manually or automatically with a maximum deviation of ± 1,0 l/h from the set value.

5.1.3 Reaction vessels of borosilicate glass, connected to a sealing cap.

The sealing cap shall be fitted with a gas inlet and outlet tube. The cylindrical part of the vessel shall preferably be narrower a few centimetres below the top in order to break any emerging foam. An artificial foam blocker (e.g. glass ring) may also be used for this purpose.

5.1.4 Closed measurement cells, of approximately 150 ml capacity, with a gas inlet tube extending to the bottom inside of the vessel. The cell shall be provided at the top with ventilation holes.

5.1.5 Electrodes, for measuring conductivity with a measuring range of 0 µS/cm to 300 µS/cm aligned with the dimensions of the measurement cell (5.1.4).

5.1.6 Measuring and recording apparatus, comprising:

a) an amplifier ;

b) a recorder for registering the measuring signal of each of the electrodes (5.1.5).

5.1.7 Thyristor and contact thermometer graduated in 0,1 °C **or Pt 100 element** to measure the block temperature, with attachments for relay connection and an adjustable heating element; temperature scale 0 °C to 150 °C.

5.1.8 Heating block, made of cast aluminium, adjustable to a temperature up to (150 ± 0,1) °C. The block shall be provided with holes for the reaction vessels (5.1.3), and an aperture for the contact thermometer (5.1.7).

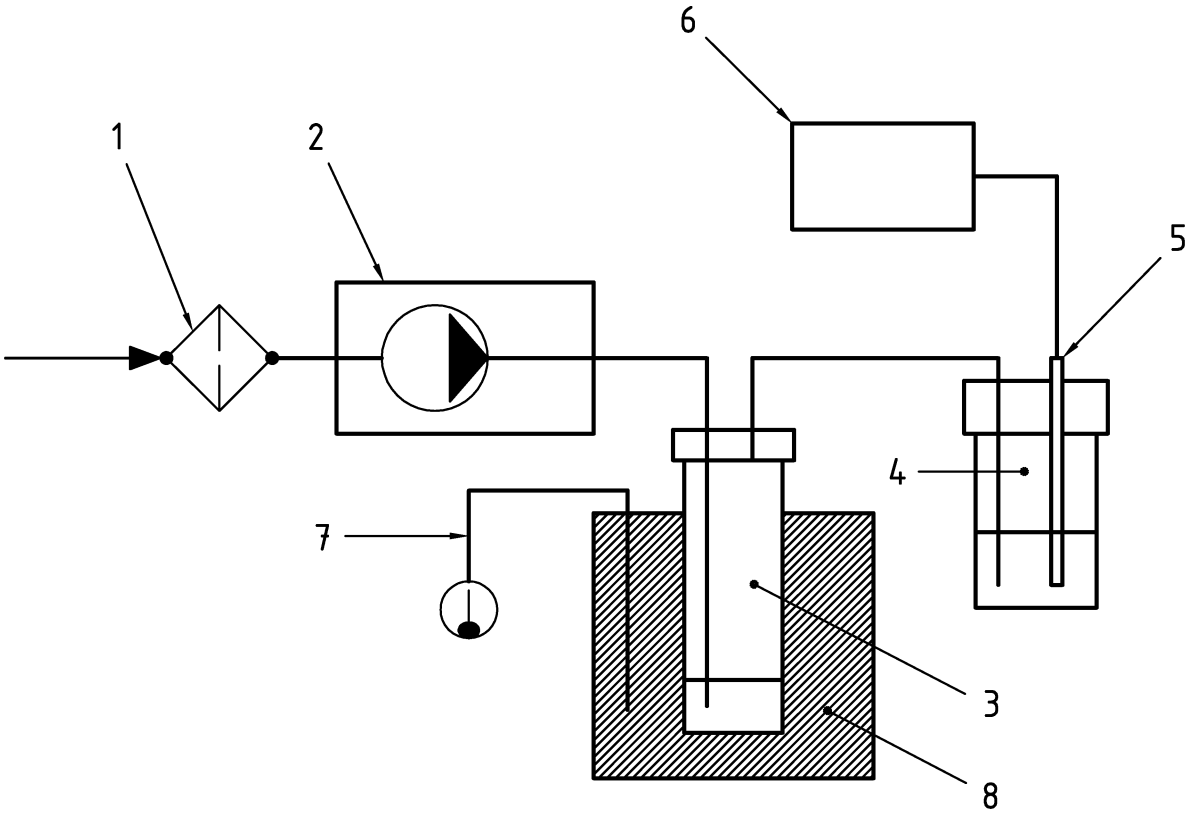
Alternatively, a **heating bath** may be used, filled with oil suitable for temperatures up to 150 °C and adjustable to the nearest 0,1 °C.

5.2 Certified and calibrated Thermometer or Pt100 element, with a temperature range up to 150 °C, graduated in 0,1 °C.

5.3 Measuring pipettes (two), of capacity 50 ml and 5 ml.

5.4 Oven, capable of being maintained up to (150 ± 3) °C.

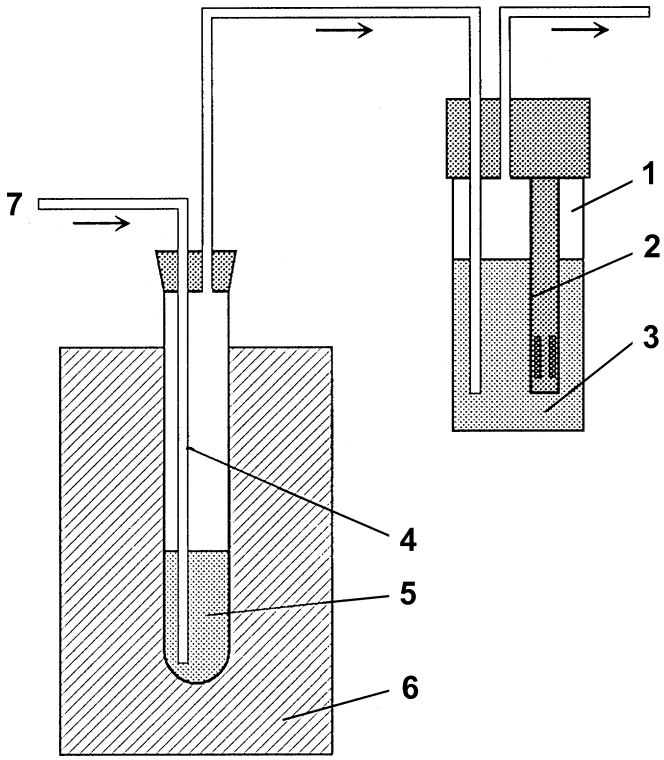
5.5 Connecting hoses, flexible and made of inert material [polytetrafluoroethylene (PTFE) or silicone].



Key

1 Air filter (5.1.1)
2 Gas diaphragm pump with flow rate control (5.1.2)
3 Reaction vessel (5.1.3)
4 Measurement cell (5.1.4)
5 Electrode (5.1.5)
6 Measuring and recording apparatus (5.1.6)
7 Thyristor and contact thermometer (5.1.7)
8 Heating block (5.1.8)

Figure 1 — Diagrammatic representation of the apparatus



Key

1 Measuring vessel
2 Electrode
3 Measuring solution
4 Reaction vessel
5 Sample
6 Heating block
7 Air

Figure 2 — Diagrammatic representation of heating block, reaction vessel and measurement cell

6 Sampling

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

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

Store the sample in the dark at about 4 °C.

7 Preparation of measurement

7.1 Preparation of test sample

In order to prevent the preparation of the test sample from influencing the test result, all handling of the laboratory sample shall be restricted to the steps given below.

Remove the required quantity from the centre of the carefully homogenised sample using a pipette.

NOTE Samples should be analysed immediately after the test sample preparation.