SLOVENSKI STANDARD

SIST EN 14112:2016

01-november-2016

Nadomešča:

SIST EN 14112:2003

Derivati maščob in olj - Metil estri maščobnih kislin (FAME) - Določevanje oksidativne stabilnosti (metoda s pospešeno 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 (Beschleunigte Oxidationsprüfung)

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

Ta slovenski standard je istoveten z: **EN 14112:2016**

ICS:

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

SIST EN 14112:2016 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14112

September 2016

ICS 67.200.10

Supersedes EN 14112:2003

English Version

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

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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 14112:2016 E

Contents

Page

European foreword

This document (EN 14112:2016) 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of an identical text or by endorsement, at the latest by March 2017, and conflicting national standards shall be withdrawn at the latest by March 2017.

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 14112:2003.

Significant changes between this document and EN 14112:2003 are:

a) the limitation of the scope of the method to a maximum induction period of 48 h, reflecting the precision range of the method;

b) indication of a potential alteration of the induction period in the presence of cetane enhancers;

c) editorial changes in order to clarify the test procedure;

d) addition of Clause 2 – Normative references;

e) addition of Clause 11 – Expression of results;

f) background information on the method added as Annex A.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This document is based on EN 14112:2003, which was specifically adapted for the determination of oxidation stability of fatty acid methyl esters (FAME). This method had been developed under CEN/TC 307 (Fats and oils).

The modifications as given in this document address the field experience with this method made since its introduction as a standard test method. Editorial changes are made in order to specify some aspects of the test. Additionally, the cleaning procedure is modified based on field experience.

1 Scope

This European Standard specifies a method for the determination of the oxidation stability of fatty acid methyl esters (FAME) at 110 °C, by means of measuring the induction period up to 48 h.

NOTE 1 EN 15751 [1] describes a similar test method for oxidation stability determination of pure fatty acid methyl esters and of blends of FAME with petroleum-based diesel containing 2 % (V/V) of FAME at minimum.

NOTE 2 The precision statement of this test method was determined in a Round Robin exercise with induction periods up to 8,5 h, thus covering the limit value in EN 14214. Results from precision studies on EN 15751 indicate that the precision statement is valid for induction periods up to 48 h but not for higher values.

NOTE 3 Limited studies on EN 15751 with EHN (2-ethyl hexyl nitrate) on FAME blends indicated that the stability is reduced to an extent which is within the reproducibility of the test method. It is likely that the oxidation stability of pure FAMEs is also reduced in the presence of EHN when EN 14112 is used for testing.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

EN ISO 3171, *Petroleum liquids - Automatic pipeline sampling (ISO 3171)*

3 Terms and definitions

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

3.1
induction period
time which passes between the moment when the measurement is started and the moment when the formation of oxidation products begins to increase rapidly

3.2
oxidation stability
induction period determined according to the procedure specified in this European Standard, expressed in hours

4 Principle

A stream of purified (dried) air is passed through the sample which has been heated to the target temperature which is 110 °C in the usual application of the method. Volatile compounds are formed during the oxidation process. They are, passed together with the air into a flask containing demineralized or distilled water, equipped with a conductivity electrode. The electrode is connected to a measuring and recording device. It indicates the end of the induction period by rapid increase of the conductivity due to the dissociation of volatile carboxylic acids produced during the oxidation process and absorbed in the water. For more details on the background of the method see Annex A.

5 Reagents and materials

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

5.1 Ternary solvent mixture, consisting of methanol/toluene/acetone 1:1:1 (by volume).

5.2 Alkaline laboratory glass cleaning solution.

5.3 2-Propanol.

6 Apparatus

Usual laboratory equipment and glassware, together with the following:

6.1 Device for the determination of oxidation stability, comprising the following parts (see Figures 1 and 2) [1)].

6.1.1 Air filter, comprising a tube fitted with filter paper at the ends and filled with a molecular sieve (6.6), connected to the suction end of a pump.

6.1.2 Gas membrane pump, with an adjustable flow rate of (10 ± 1,0) l/h.

6.1.3 Reaction vessels of borosilicate glass, provided with a sealing cap.

The sealing cap shall be fitted with a gas inlet and outlet tube.

6.1.4 Closed measurement cells, of approximately 150 ml capacity, with an air inlet tube extending to the bottom inside of the vessel. The cell shall have ventilation holes at the top.

6.1.5 Electrodes, for measuring conductivity within a range of 0 µS/cm to 300 µS/cm aligned with the dimensions of the measurement cell (6.1.4).

6.1.6 Measuring and recording apparatus, comprising:

a) an amplifier; and

b) a recorder registering the signal of each of the electrodes (6.1.5).

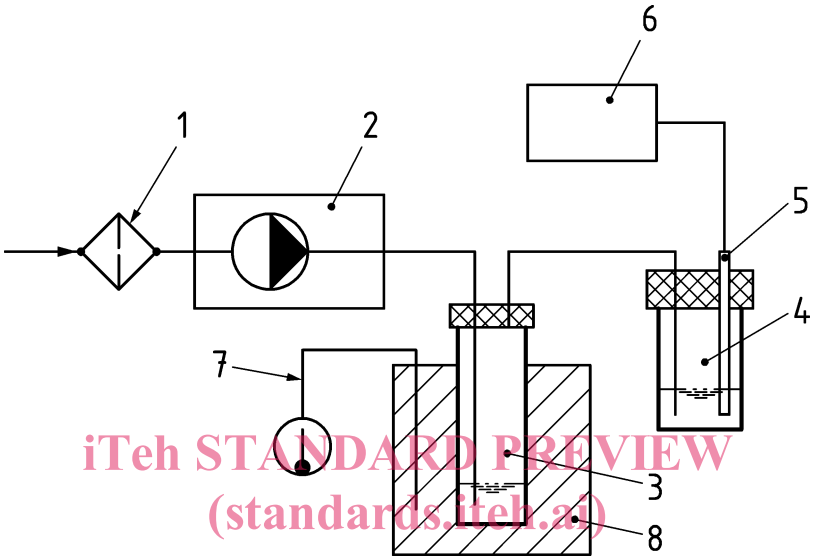
6.1.7 Thyristor and contact thermometer graduated in 0,1 °C **or Pt 100 element** to measure the block temperature, with attachments for relay connection and an adjustable heating element; temperature scale 0 °C to 150 °C.

1) Rancimat is the trade name of a product supplied by Metrohm AG, Herisau, Switzerland; OSI is the trade name of a product supplied by Omnion Inc., Rockland, Massachusetts, USA. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN or CENELEC of the products named. Equivalent products may be used if they can be shown to lead to the same results.

6.1.8 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 (6.1.3) and an aperture for the contact thermometer (6.1.7).

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.

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

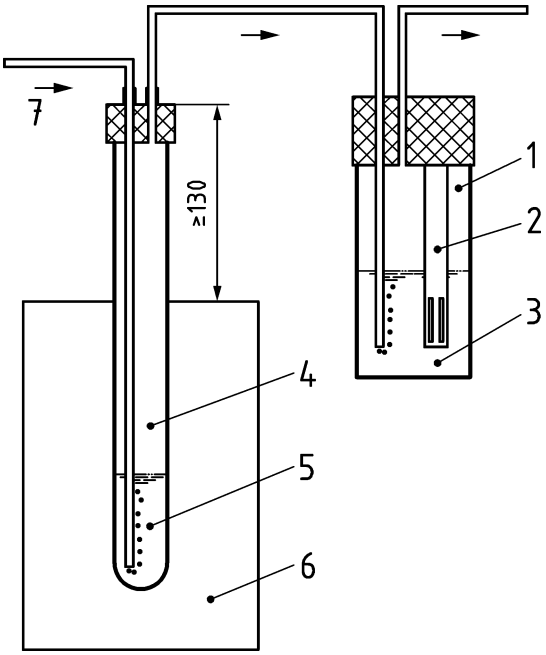


Key

1 air filter (6.1.1)
2 gas membrane pump with flow rate control (6.1.2)
3 reaction vessel (6.1.3)
4 measurement cell (6.1.4)
5 electrode (6.1.5)
6 measuring and recording apparatus (6.1.6)
7 thyristor and contact thermometer (6.1.7)
8 heating block (6.1.8)

Figure 1 — Apparatus

Dimensions in mm



Key

1 measuring vessel
2 electrode
3 distilled/demineralized water
4 reaction vessel
5 sample
6 heating block
7 air inlet

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

6.3 Measuring pipettes and/or measuring cylinders.

6.4 Oven, adjustable to a temperature up to (150 ± 3) °C.

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

6.6 Molecular sieve, with moisture indicator, pore size 0,3 nm, dried in an oven set at 150 °C and cooled down to room temperature in a desiccator before use.

6.7 Balance, capable of weighing with an accuracy of ± 0,1 g or less.

7 Sampling

Unless otherwise specified, sampling shall be conducted according to EN ISO 3170 or EN ISO 3171 and/or in accordance with the requirements of national standards or regulations for the sampling.

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

Store the sample in the dark at about 4 °C and measure it as soon as possible after receipt.

8 Preparation of measurement

8.1 Preparation of test sample

In order to ensure a consistent test condition, all samples shall be treated in the way described below:

— take the required quantity from the centre of the carefully homogenized sample using a pipette;

— analyse the samples immediately after sample preparation.

8.2 Preparation of apparatus

8.2.1 Cleaning procedure

The use of new disposable reaction vessels, air inlet tubes and connecting hoses is recommended in order to save the cleaning procedure.

Sealing caps, measuring cells and electrodes shall be cleaned with 2-Propanol in order to remove organic residues. The connecting hoses should also be washed in the same manner if not replaced.

Rinse with tap water and finally with demineralized or distilled water. Dry the cleaned parts in an oven at 80 °C for at least 2 h. The temperature may not exceed 80 °C due to elastomer stability.

Residual fuel and aging products from the previous experiment and the solvent from the cleaning may remain adsorbed in the elastomers and shall be removed. A drying time of 2 h ensures that all volatile compounds are removed.

In case of reuse, purge the empty reaction vessels and the air inlet tubes at least three times with ternary solvent mixture (5.1) in order to remove residual fuel and adherent organic ageing residues. The last solvent portion should remain colourless.

Rinse with 2-Propanol and tap water. Put the inlet tube into the reaction vessel and fill completely with an aqueous alkaline laboratory cleaning solution.

Store the vessels at room temperature overnight.

Rinse the purified vessels and their inlet tubes thoroughly with tap water and finally with demineralized or distilled water. Dry them in an oven for at least 2 h at 80 °C.

In case of doubt, the cleanliness of the sealing caps and connecting hoses can be checked by running a blank sample under standard test conditions. In this case the conductivity increase shall not exceed 10 µS/cm within 5 h.

8.2.2 Temperature correction

8.2.2.1 General

Any deviation between the temperature of the fuel sample in the test vessel and the temperature of the heating block or the heating bath has a significant impact on the result. In order to ensure that the correct measurement temperature is used, the difference between the temperature of the sample and the temperature of the heating block, ΔT, needs to be determined. For this determination a calibrated external temperature sensor is used.

The temperature correction always needs to be conducted when the test is carried out at a different temperature than before.

8.2.2.2 Procedure

Switch on the heating block and wait until the target temperature is reached and is stable.