SLOVENSKI STANDARD
SIST EN 14112:2021

01-januar-2021

Nadomešča:
SIST EN 14112:2016

Derivati maščob in olj - Metilni 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 accélérée)

Ta slovenski standard je istoveten z: **EN 14112:2020**

ICS:

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

SIST EN 14112:2021 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 14112

November 2020

ICS 67.200.10

Supersedes EN 14112:2016

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 und Ölen - Fettsäure-Methylester (FAME) - Bestimmung der Oxidationsstabilität (beschleunigte Oxidationsprüfung)

This European Standard was approved by CEN on 25 October 2020.

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 CEN-CENELEC 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 CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.

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

CEN-CENELEC Management Centre: Rue de la Science 23, B-1040 Brussels

Ref. No. EN 14112:2020 E

Contents

Page

European foreword

This document (EN 14112:2020) 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 May 2021, and conflicting national standards shall be withdrawn at the latest by May 2021.

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 14112:2016.

Significant changes between this document and EN 14112:2016 are:

— change of Figure 2, removal of dimension between air inlet and heating block;

— introduction removed;

— document revised editorially.

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, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Republic of North Macedonia, Romania, Serbia, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This document 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.

For induction periods higher than 8,5 h the precision is not covered by the precision statement of this method.

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 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.

NOTE 3 For the purposes of this document, the term "% (*V*/*V*)" is used to represent the volume fraction.

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 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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

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 document, 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, and 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 Chemicals

Use only chemicals 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).

---

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 document 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.

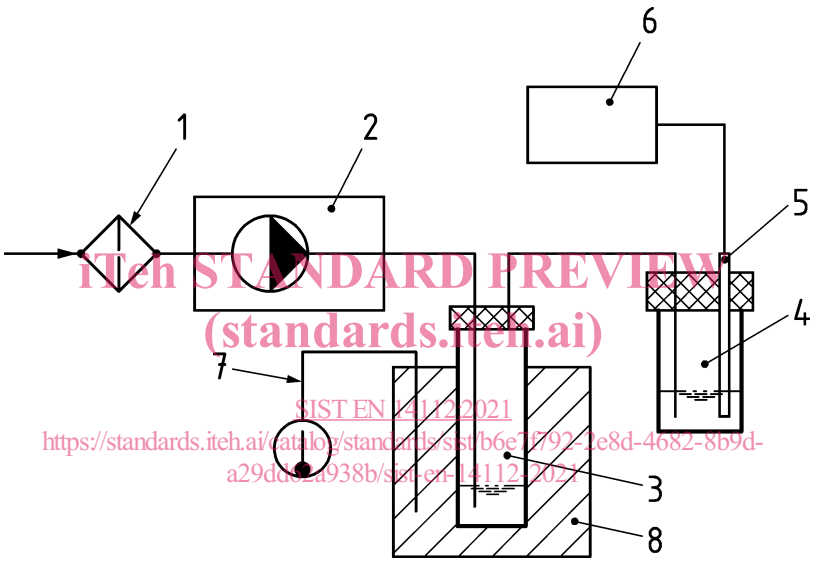
6.1.7 Contact thermometer graduated in 0,1 °C **or Pt 100 element** to measure the block temperature, with attachments for relay connection and an adjustable heating element; temperature scale 0 °C to 150 °C.

6.1.8 Heating source, 6.1.8.1 or 6.1.8.2

6.1.8.1 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).

6.1.8.2 Heating bath, filled with oil suitable for temperatures up to 150 °C, and adjustable to the nearest 0,1 °C.

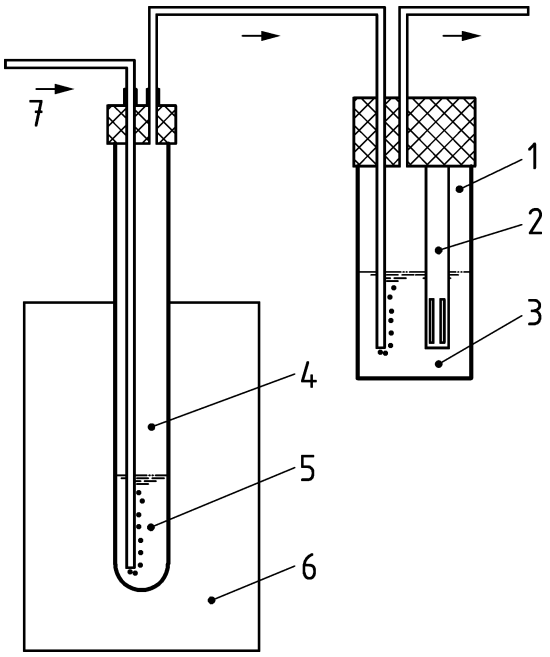
6.2 Certified and calibrated thermometer or Pt100 element, with a temperature range up to 150 °C, graduated in 0,1 °C.



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 contact thermometer or Pt 100 element (6.1.7)
8 heating block (6.1.8.1)

Figure 1 — Schematic overview of apparatus



Key

1 measurement cell (6.1.4)
2 electrode (6.1.5)
3 distilled/demineralized water
4 reaction vessel (6.1.3)
5 sample
6 heating block (6.1.8.1)
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 should be 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. Ensure that the container is filled up to the top in order to minimize contact with air.

Store the sample in the dark and measure it as soon as possible after receipt. For long time storage it is recommended to store the samples at 4 °C.

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:

— homogenize the sample carefully;

— 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 (5.3) 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 (5.3) and tap water. Put the inlet tube into the reaction vessel and fill completely with an aqueous alkaline laboratory cleaning solution (5.2).

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 test without any 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.

Fill one reaction vessel with 5 g thermo-stable oil. Insert the temperature sensor through the cap into the reaction vessel. Use distance clips to keep the sensor away from the air inlet. The sensor should touch the bottom of the vessel.

Insert the complete vessel into the heating block and connect the air supply.

If the value of the measured temperature is constant, calculate ΔT:

$$\Delta T = T_{\text{block}} - T_{\text{sensor}} \quad (1)$$

where

ΔT is the temperature difference between heating block and sample;

T_{block} is the temperature of the heating block;

T_{sensor} is the sample temperature in the reaction vessel measured by the sensor.

Adjust the temperature of the heating block according to Formula (2):

$$T_{\text{block}} = T_{\text{target}} + \Delta T \quad (2)$$

where

T_{target} is the intended measurement temperature.

EXAMPLE T_{target} is 110 °C. If a ΔT of + 2 °C is determined, the temperature of the heating block has to be set to 112 °C.

After this temperature correction, the measured temperature in the reaction vessel should be equal to the target temperature.

9 Measurement

9.1 Set up the apparatus as shown in Figure 1. If commercially available equipment is used, follow the manufacturer's instructions.

9.2 Attach the membrane pump (6.1.2) and adjust the air flow to (10 ± 1) l/h. Switch off the pump. Dedicated instruments are usually equipped with automatic flow control.

9.3 Bring the heating block (6.1.8.1) to a temperature such that the required test temperature (usually 110 °C, but see 8.2.2) is reached in the test tube(s), using the contact thermometer (6.1.7) or by using an electronic temperature controller.

If a heating bath (6.1.8.2) is used, heat to the desired temperature and control the temperature according to 8.2.2.

9.4 Fill the measurement cells (6.1.4) with 60 ml of distilled or demineralized water using a measuring pipette or a cylinder (6.3).

9.5 If recommended by the manufacturer, check the electrodes (6.1.5) and adjust their signals using a calibration potentiometer.

9.5.1 Automatic instruments record and process the data.