SLOVENSKI STANDARD
SIST EN 16091:2022

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Nadomešča:
SIST EN 16091:2012

Tekoči naftni proizvodi - Goriva na osnovi srednjih destilatov, metilnih estrov maščobnih kislin (FAME) in mešanic - Ugotavljanje oksidacijske stabilnosti z oksidacijskim preskusom rapidne male skale (RSSOT)

Liquid petroleum products - Middle distillates and fatty acid methyl ester (FAME) fuels and blends - Determination of oxidation stability by rapid small scale oxidation test (RSSOT)

Flüssige Mineralölerzeugnisse - Mitteldestillat- und Fettsäure-Methylester (FAME)-Kraftstoffe und -Mischungen - Bestimmung der Oxidationsstabilität mit beschleunigtem Oxidationsverfahren und kleiner Probenmenge (RSSOT)

Produits pétroliers liquides - Distillats moyens, esters méthyliques d'acides gras (EMAG) et leurs mélanges - Détermination de la stabilité à l'oxydation par méthode d'oxydation accélérée à petite échelle (RSSOT)

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75.160.20	Tekoča goriva	Liquid fuels

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EUROPEAN STANDARD

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Liquid petroleum products - Middle distillates and fatty acid methyl ester (FAME) fuels and blends - Determination of oxidation stability by rapid small scale oxidation test (RSSOT)

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This European Standard was approved by CEN on 19 September 2022.

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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 16091:2022) has been prepared by Technical Committee CEN/TC 19 “Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin”, the secretariat of which is held by NEN.

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of an identical text or by endorsement, at the latest by April 2023, and conflicting national standards shall be withdrawn at the latest by month April 2023.

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 16091:2011.

In comparison with the previous edition EN 16091:2011, the following technical modifications have been made:

a) addition of informative Annex C to include work that has been executed on a modified rapid small scale oxidation method at 120 °C;

b) revision of Clause 8: the performance check of the apparatus and the recommended calibration process have been merged into a single Clause 8.1;

c) Annex on the calibration process has been removed;

d) the document was revised editorially.

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.

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, Türkiye and the United Kingdom.

1 Scope

This document specifies a method for the determination of the oxidation stability of middle distillate fuels, fatty acid methyl ester (FAME) fuel and blends thereof, under accelerated conditions, by measuring the induction period to the specified breakpoint in a reaction vessel charged with the sample and oxygen at 140 °C.

NOTE 1 For the purposes of this document, the term "% (*V*/*V*)" is used to represent the volume fraction (φ).

NOTE 2 The induction period is used as an indication for the resistance of middle distillates, fatty acid methyl ester (FAME) fuels and blends thereof against oxidation. This correlation can vary markedly under different conditions with different FAMEs and diesel fuel blends.

NOTE 3 The presence of ignition improvers can lead to lower oxidation stability results determined by this method. It has for instance been observed that the addition of 2-ethyl hexyl nitrate (2-EHN) can reduce the measured oxidation stability values. See [6] for details.

NOTE 4 For further information on the precision data at a test temperature of 120 °C see Annex C.

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:

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

— IEC Electropedia: available at https://www.electropedia.org/

3.1
breakpoint
point in the pressure-time curve, where the pressure in the vessel has dropped 10 % below the maximum pressure achieved in the current test run

3.2
induction period
time elapsed between starting the heating procedure of the reaction vessel, charged with sample and oxygen, and the breakpoint, measured in minutes

4 Principle

At ambient temperature, a known volume of a sample is placed in a reaction vessel charged with oxygen to a pressure of 700 kPa ± 5 kPa. The reaction vessel is heated to 140 °C. The pressure in the vessel drops as the oxygen is consumed during the oxidation of the sample. The pressure in the vessel is recorded at intervals of 1 s until the breakpoint is reached. The elapsed time from start to the breakpoint is the induction period at the test temperature of 140 °C ± 0,5 °C.

NOTE Work has been executed on the determination of oxidation stability by a modified rapid small-scale oxidation method at 120 °C. The results thereof are given in Annex C.

5 Reagents and materials

5.1 Cleaning solvent, for the removal of oxidation residues from the test vessel, of suitable purity to leave no residue on the apparatus.

NOTE Commercially available ethanol of approximately 95 % (*V*/*V*) purity was found to be suitable.

5.2 Oxygen, extra dry (<5 mg/kg water), commercially available, with a purity of not less than 99,6 %.

5.3 Cleaning tissues, lint-free for cleaning sensitive galvanic coated surfaces without scratching.

5.4 Verification fluid, hydrocarbon fuel, any hydrocarbon fuel with sufficient stability and a known induction period may be used.

NOTE In general, verification fluid with a certified induction period is available from the manufacturer of the apparatus.

6 Apparatus

6.1 Automatically controlled oxidation tester, comprising an oxidation reaction vessel containing:

— test sample cup capable of being rapidly heated;

— pressure sensor capable of measuring pressures of 1 kPa up to 2 000 kPa with a resolution of 1 kPa;

— temperature sensor with a resolution of 0,1 °C;

— pressure and temperature recording at time intervals of 1 s.

The oxidation reaction vessel shall be fitted with filling and relief valves, meant to release the pressure, and a fan to cool the vessel from test to ambient temperature.

The requirements for the apparatus are described in detail in Annex A.

6.2 Pipette, capable of delivering 5,0 ml ± 0,1 ml.

6.3 O-ring seals, see A.6.

7 Sampling and sample handling

Unless otherwise specified, samples shall be taken as described in EN ISO 3170 or EN ISO 3171.

Collect and store samples in an opaque container to minimize exposure to light.

8 Performance check of the apparatus

8.1 Verify the correct operation of the equipment by following the procedure in Clause 10 using the verification fluid (5.4). If the verification measurement result is within the certified range then the pressure and temperature sensor are working correctly, and the reaction vessel is reaching 140 °C ± 0,5 °C within the required time of 270 s ± 30 s.

8.2 If the induction period of the verification fluid does not meet the specified time, repeat 8.1. If it fails a second time, refer to the manufacturer's instructions.

9 Preparation of the apparatus

9.1 Remove the previous test portion by means of a pipette or similar device.

9.2 Remove the used “O-ring” seal and discard.

To avoid contamination of the new test, it is necessary to discard the used “O-ring” seal, because it can be soaked with oxidation products from the previous test.

9.3 Wipe the sample cup, the seal groove and the cover with lint-free cleaning tissue (5.3) soaked with cleaning solvent (5.1) until free of gum or other oxidation residues.

9.4 Allow the test sample cup and cover to dry in air and visually inspect for cleanliness.

NOTE Compressed air is generally unsuitable to accelerate the evaporation of solvent because it can contain traces of oil that can influence the next test.

9.5 Insert a new "O-ring" seal (6.3).

10 Procedure

10.1 Bring the reaction vessel and the sample to be tested to room temperature.

10.2 Using a pipette (6.2), place 5,0 ml ± 0,1 ml of the sample into the reaction vessel sample cup.

10.3 Cover the sample-cup with the screw cap (see A.2) and close the reaction vessel.

10.4 Pre-flush the reaction vessel with oxygen. Introduce oxygen (5.2) into the vessel until a pressure of 700 kPa ± 5 kPa is attained and stabilized over 20 s.

10.5 Immediately after 10.4 start the test by starting the heater.

10.6 The test temperature of 140 °C ± 0,5 °C shall be reached after 270 s ± 30 s.

10.7 If, during the initial 5 min of the test, a steady drop in pressure is observed, discontinue the test and discard the test specimen.

The leakage rate at 140 °C with empty sample cup shall not exceed a value of 2 kPa/h. If the leakage rate shows an increase, check the following components:

— O-ring for damage or residues of samples;

— surface of sample cup for damage; and

— sample cup for sample residues.

Contact the manufacturer to resolve leakage problems from other parts of the instrument.

10.8 The apparatus automatically records the temperature (to the nearest 0,1 °C) and pressure (to the nearest 1 kPa) of the oxidation vessel at time intervals of 1 s.

10.9 The apparatus terminates the test when the pressure readings show a 10 % drop from the maximum observed pressure (breakpoint) and records the time elapsed from the start of the test to the breakpoint (induction period). An example is given in Annex C.

10.10 The apparatus automatically records the average temperature to the nearest 0,1 °C as the temperature of the test.

NOTE The apparatus will automatically switch on the fan to cool the reaction vessel to approximately room temperature. When the apparatus has cooled, the pressure is slowly released, at a rate not exceeding 345 kPa/min.

10.11 When the pressure release has finished, open the apparatus and clean according to Clause 9.

11 Expression of results

Report the induction period expressed in min, rounded to the nearest 0,01 min.

12 Precision

12.1 General

The precision statements were determined from results of interlaboratory tests performed in 2008 and 2009, by statistical examination in accordance with EN ISO 4259:2006 [1]. This method was tested for FAME, B0, B5, B7, B10, and B30 samples, the induction period being in the rage between 22 min and 215 min.

NOTE The interlaboratory testing and the statistical evaluation are detailed in Research Report 2011-404 [6].

12.2 Repeatability, *r*

The difference between two independent results obtained using this method for test material considered to be the same in the same laboratory, by the same operator using the same equipment within short intervals of time, in the normal and correct operation of the method that is expected to be exceeded with an approximate probability of 5 % due to random variation, can be calculated using the following function:

$$r = 0{,}028\,8\,X + 0{,}496\,5 \quad (1)$$

where

X is the mean of the two results expressed in min, rounded to the nearest 0,01 min.

12.3 Reproducibility, *R*

The difference between two independent results obtained using this method for test material considered to be the same in different laboratories, where different laboratory means a different operator, different equipment, different geographic location, and under different supervisory control, in the normal and correct operation of the method that is expected to be exceeded with an approximate probability of 5 % due to random variation, can be calculated using the following function:

$$R = 0{,}086\,3\,X + 1{,}377\,2 \quad (2)$$

where

X is the mean of the two results expressed in min, rounded to the nearest 0,01 min.