SLOVENSKI STANDARD
oSIST prEN 16091:2021

01-junij-2021

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

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

Flüssige Mineralölerzeugnisse - Mitteldestillat- und Fettsäuremethylesterkraftstoffe und Mischungen - Bestimmung der Oxidationsstabilität mit beschleunigtem Verfahren und kleiner Probenmenge

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

Ta slovenski standard je istoveten z: **prEN 16091**

ICS:

75.160.20	Tekoča goriva	Liquid fuels

oSIST prEN 16091:2021 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

DRAFT
prEN 16091

May 2021

ICS 75.160.20

Will supersede EN 16091:2011

English Version

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

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This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee CEN/TC 19.

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European foreword

This document (prEN 16091:2021) 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 document is currently submitted to the CEN Enquiry.

This document will supersede EN 16091:2011.

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

a) A new Annex D was included to include work that has been executed on a modified rapid small scale oxidation method at 120 °C;

b) The document was revised editorially.

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.

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 D.

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 at 140 °C

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 D.

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 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).

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

8.3 Pressure and temperature are critical parameters. Proper calibration of the respective sensors (see 6.1) is therefore important. Recalibrate the tester at least every 12 months for correct temperature and pressure readings.

8.4 In view of general good laboratory practice and harmonization of test methods, calibration as in Annex B is strongly recommended. As the calibration procedure for a pressure sensor is complicated and difficult to be executed by an untrained lab technician, one may (in most cases sufficiently) rely on the verification fluid (5.4).

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 ± 0,1ml 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 Start the heater with no delay between charging with oxygen and starting the test.

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 continuously.

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.

13 Test report

The test report shall contain at least the following information:

a) a reference to this document, i.e. EN 16091;

b) the type and complete identification of the product tested;

c) the result of the test (see Clause 11);

d) any deviation, by agreement or otherwise, from the procedure specified;

e) the date of the test.