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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 method

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

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (EN 16091:2011) 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of an identical text or by endorsement, at the latest by May 2012, and conflicting national standards shall be withdrawn at the latest by May 2012.

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1 Scope

This European Standard 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 European Standard, 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. It should be recognized, however, that this correlation can vary markedly under different conditions with different FAMEs and diesel fuel blends.

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

2 Normative references

The following referenced documents are indispensable for the application 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 3170:2004)*

EN ISO 3171, *Petroleum liquids — Automatic pipeline sampling (ISO 3171:1988)*

3 Terms and definitions

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

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 ºC

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 ºC. 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 1s until the breakpoint is reached. The elapsed time from start to the breakpoint is the induction period at the test temperature of 140 ºC ±0,5 ºC.

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 approx. 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 (B0).**

NOTE Any hydrocarbon fuel with sufficient stability and a known induction period may be used as verification fluid. 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 ºC;
- pressure and temperature recording of 1 second.

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 and/or in accordance with the requirements of national standards or regulations for the sampling of the product under test.

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 section 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.

NOTE 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.

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.

NOTE To avoid contamination of the new test, it is necessary to discard the used "O-ring" seal, because it may 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 may 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 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 sec.

10.5 Start the heater with no delay between charging with oxygen and starting the test.

10.6 The test temperature of 140 ºC ± 0,5 ºC 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 ºC 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 ºC) 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 ºC 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 developed in Round Robin tests performed in 2008 and 2009, in accordance with EN ISO 4259 [1]. This method was tested for FAME, B0, B5, B7, B10, and B30 samples, the induction period measured being between 22 min and 215 min.

12.2 Repeatability, *r*

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material, would in the long run, in the normal and correct operation of the test method, exceed the values calculated from the following equation only in one case in twenty:

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

where

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

12.3 Reproducibility, *R*

The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material, would in the long term, in the normal and correct operation of the test method, exceed the values calculated from the following equation only in one case in twenty:

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

where

X represents 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 European Standard;

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

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

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

e) the date of the test.

Annex A
(normative)

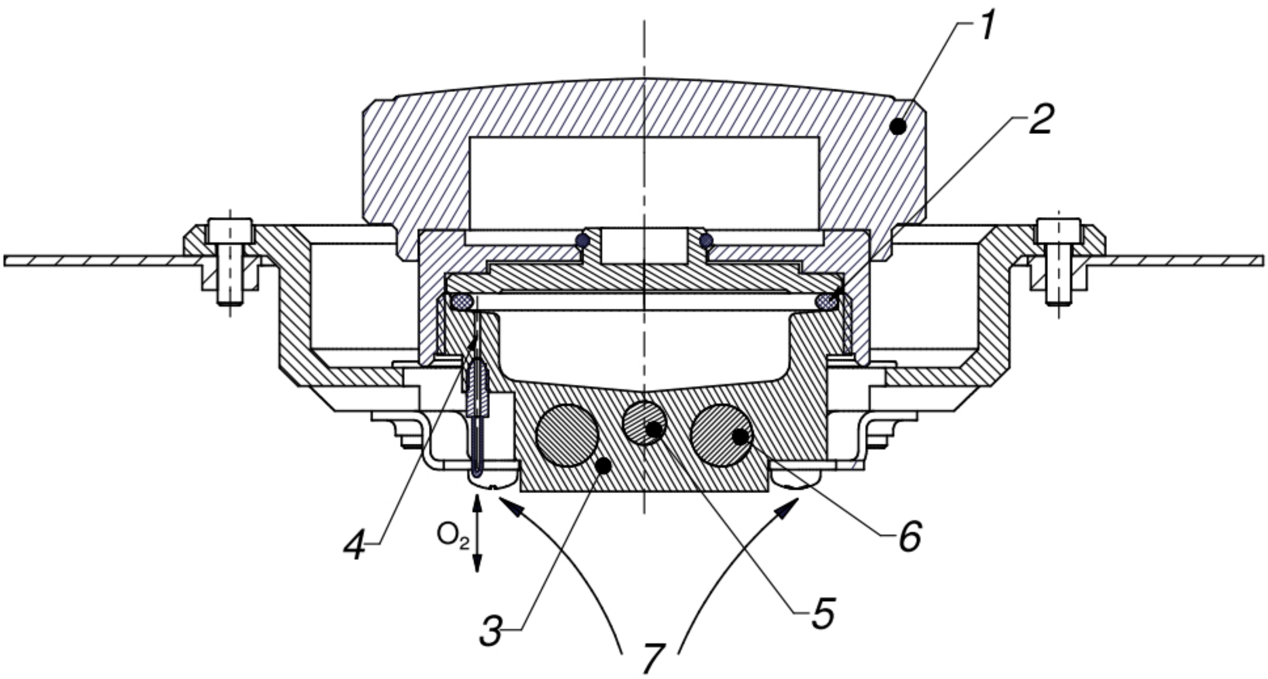
Apparatus description

A.1 General requirements

All equipment in contact with the sample and oxygen shall be made of corrosion-resistant materials and the reaction vessel shall have an over-temperature safeguard.

A.2 Assembly of apparatus

The assembly shall be according to Figure A.1.



Key

1 screw cap (A.4)
2 O-ring seal(A.6)
3 reaction vessel (A.3)
4 oxygen supply (5.2)
5 temperature sensor (A.9)
6 electric heater (A.5)
7 cooling (A.11)

Figure A.1 — Reaction vessel for accelerated oxidation

A.3 Reaction vessel

Inner diameter 47 mm and internal volume without sample of 20 ml. Made of aluminium alloy or other corrosion-resistant metal of similar thermal conductivity, with a cylindrical depression (test cup), and with a hole in the side to accommodate a temperature sensor (A.9).

The interior surface of the test cup is covered with galvanic coating and smoothly polished.

A.4 Screw cap

Made of aluminium alloy or other corrosion-resistant metal of similar thermal conductivity. Interior surface of the screw cap covered with galvanic coating, smoothly polished.

A.5 Electric heater

Power rating 500 W; attached to the bottom of the test cup in a manner that provides efficient transfer of heat. The heater control shall be capable of maintaining the test cup temperature within ± 0,5 ºC for test temperatures up to and including 140 ºC.

A.6 O-ring seals

Made of material being resistant to oxygen, middle distillate fuel components, FAME and heat, typically fluoro-elastomer FPM/FKM (commonly known as Viton®[1]), coated with PTFE.

A.7 Valves

Solenoid with small dead volume and orifice sizes and short cycling rates.

A.8 Pressure sensor

Absolute or relative pressure transducer with a pressure range of 0 kPa to 2 000 kPa and a typical sensitivity of between (1 to 2,5) mV/kPa. Capable of being operated at temperatures up to 150 ºC and calibrated and/or verified against an instrument certified by an authorized certification body.

A.9 Temperature sensor

Platinum thermometer capable of reading up to 200 ºC ± 0,1 ºC, calibrated and/or verified against an instrument certified by an authorized certification body.

A.10 Connecting pipes

Typical inner diameter: 0,5 mm to 1 mm.

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[1] Viton is an example of a suitable product 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 product.

A.11 Cooling fan

Capable of cooling, the reaction vessel from the test temperature to ambient by applying a stream of air to the outside of the reaction vessel.

Annex B
(informative)

Calibration process

B.1 Calibration of temperature indicator.

Calibrate the temperature sensor to the nearest 0,1 ºC according to the manufacturer's instructions.

B.2 Calibration of pressure detector.

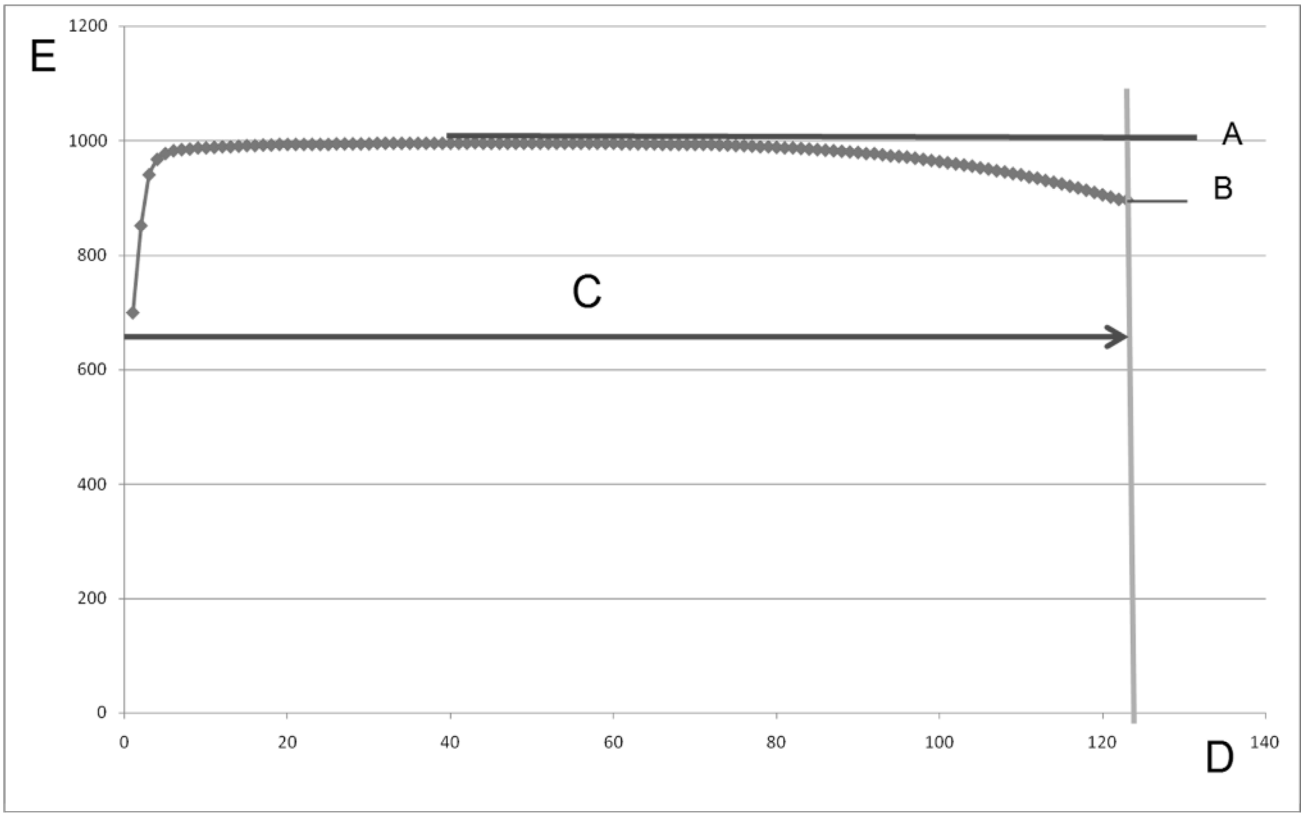
Calibrate the pressure sensor to the nearest 10 kPa according to the manufacturer's instructions.

B.3 Verify, at least every 3 months, that the heater is operating properly and the reaction vessel is reaching 140 ºC ± 0,5 ºC within 270 s ± 30 s.

Annex C
(informative)

Determination of induction period

Figure C.1 presents an example of how the breakpoint and induction period of a sample are determined. The point B indicates the 10% drop from maximum pressure, 0,9 P_{max}, which is the breakpoint and defines the induction period (C).



Key

A maximum observed pressure, P_{max}

B breakpoint

C induction period (min)

D test time (min)

E observed pressure (kPa)

Figure C.1 — Typical pressure/time curve

Bibliography

[1] EN ISO 4259, *Petroleum products — Determination and application of precision data in relation to methods of test (ISO 4259:2006)*

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