SLOVENSKI STANDARD
oSIST prEN 16568:2013

01-maj-2013

Goriva za motorna vozila - Metilni estri maščobne kisline (FAME) goriv in mešanice z dizelskim gorivom - Ugotavljanje oksidativne stabilnosti z metodo pospešene oksidacije pri 120 °C

Automotive fuels - Fatty acid methyl ester (FAME) fuel and blends with diesel fuel - Determination of oxidation stability by rapidly accelerated oxidation method at 120 °C

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Kraftstoffe für Kraftfahrzeuge - Kraftstoff Fettsäure-Methylester (FAME) und Mischungen mit Dieselkraftstoff - Bestimmung der Oxidationsstabilität mittels beschleunigterem Oxidationsverfahren bei 120 °C

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Carburants automotives - Esters méthyliques d'acides gras (EMAG) et mélanges avec gazole - Détermination de la stabilité à l'oxydation accélérée rapide à 120 °C

Ta slovenski standard je istoveten z: **prEN 16568**

ICS:

75.160.20	Tekoča goriva	Liquid fuels

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EUROPEAN STANDARD

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ICS

English Version

Automotive fuels - Fatty acid methyl ester (FAME) fuel and blends with diesel fuel - Determination of oxidation stability by rapidly accelerated oxidation method at 120 °C

Carburants automotives - Esters méthyliques d'acides gras (EMAG) et mélanges avec gazole - Détermination de la stabilité à l'oxydation accélérée rapide à 120 °C

Kraftstoffe für Kraftfahrzeuge - Kraftstoff Fettsäure-Methylester (FAME) und Mischungen mit Dieselkraftstoff - Bestimmung der Oxidationsstabilität mittels beschleunigterem Oxidationsverfahren bei 120 °C

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 COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Foreword

This document (prEN 16568:2013) 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.

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Introduction

This document is based on EN 15751 [1], which was specifically developed for the determination of oxidation stability of fatty acid methyl ester (FAME) and blended petroleum based diesel fuels. The oxidation stability is assessed by determining the induction period of the fuel. The induction period is a measure for the ageing reserve of the fuel.

The first version of EN 15751 was developed under CEN/TC 19 for a test temperature of 110 °C in order to stay directly comparable to EN 14112:2003 [2] which is used to determine the oxidation stability of pure FAME. The stability of diesel/FAME blends is generally higher compared to pure FAME thus leading to long measuring times. In order to better accommodate the needs of laboratories the idea was raised to increase the reaction temperature to 120 °C. Degradation of the ageing reserve of the fuel follows the Arrhenius law. By increasing the temperature by 10 °C, the reaction rate is doubled cutting in half the induction period.

The modifications to EN 15751, as given in this document, allow the application of this test method for oxidation stability for diesel/FAME blends containing 2 % (*V/V*) of FAME at minimum. This test method is not applicable to pure FAME. Pure FAME was not included in the scope because of reduced ability to differentiate between different qualities when the induction period is reduced by 50 %.

The temperature increase required a new validation for diesel/FAME blends. Blends with up to 50 % (*V/V*) of FAME were selected in order to comprise also high FAME blends which are presently discussed for automotive use. Due to concerns about a potential impact of cetane improvers, an additional study with 2-ethyl hexyl nitrate (EHN) at 110 °C and 120 °C was performed.

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1 Scope

This European Standard specifies a test method for the determination of the oxidation stability of fuels for diesel engines, by means of measuring the induction period of the fuel up to 48 h at 120 °C. The method is applicable to fatty acid methyl esters (FAME) intended for the use as pure biofuel or as a blending component for diesel fuels, and to blends of FAME with petroleum-based diesel containing 2 % (*V/V*) of FAME at minimum.

NOTE 1 A similar test method for oxidation stability at 110 °C is described in EN 15751 [1], which applies to pure FAME and Diesel/FAME blends containing 2 % (*V/V*) of FAME at minimum. Another alternative for distillate fuels is described in EN ISO 12205 [3].

NOTE 2 For induction periods higher than 48 h the precision is not covered by the precision statement of this method. The limit values of the relevant fuel standards are well within the scope of this test method.

The presence of cetane improver can reduce the oxidation stability determined by this test method. Limited studies with 2-ethyl hexyl nitrate (EHN) indicated, however, that the stability is reduced to an extent which is within the precision range of the test method.

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*

EN ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

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3 Terms and definitions

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

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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 air is passed through the sample which has been heated to the specified, elevated temperature. Volatile compounds are formed during the oxidation process. They are passed together with the air into a flask containing demineralised 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.

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5 Reagents and materials

Use only reagents of recognised analytical grade, and distilled or demineralised water [4].

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):

NOTE An instrument for determining the oxidation stability is commercially available under the trade name Rancimat®, (model 743 or higher, from Metrohm AG, Herisau, Switzerland) or OSI® Instrument (from Omnion Inc., Rockland, Massachusetts, USA). These are examples of suitable equipment which are given for the convenience of users of this document. They do not constitute an endorsement by CEN of this equipment.

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) .

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

The length of the reaction vessel depends on the measuring equipment and shall exceed the depth of the oven by at least 130 mm, in order to reduce evaporation losses to a minimum by condensing volatile fuel components at the cold vessel walls outside the oven.

EXAMPLE Total length of the test tube for the Metrohm Rancimat 743 L = 250 mm, for the Omnion OSI Instrument L = 300 mm.

The sealing cap shall be fitted with a gas inlet and outlet tube. A few centimetres below the top, the vessel shall preferably have a slightly reduced inner diameter in order to break any emerging foam. An artificial foam blocker (e.g. glass ring) may also be used for this purpose.

6.1.4 Closed measurement cells of approximately 150 ml capacity, with a gas 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 the conductivity within a range from 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.

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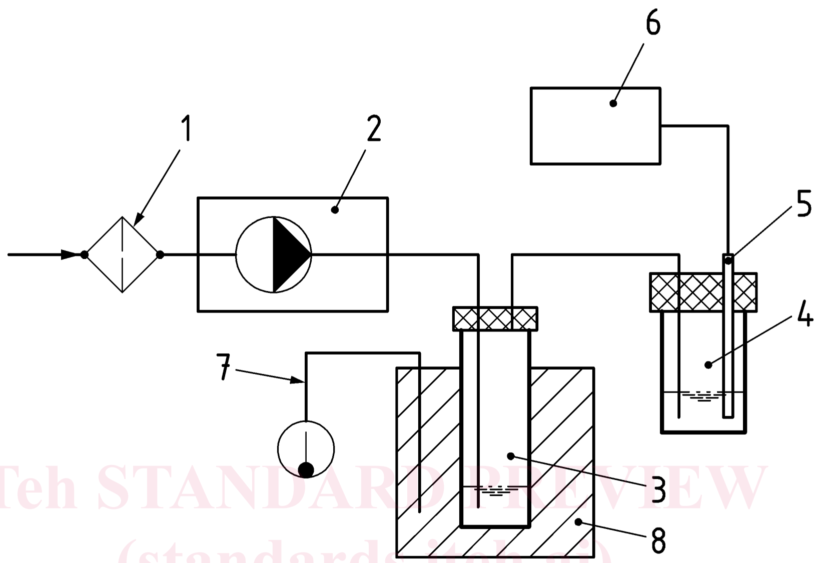
6.1.8 Heating block, made of cast aluminium, adjustable to a temperature up to (150 ± 0,1) °C. The block shall be provided with holes for the reaction vessels (6.1.3), and an aperture for the contact thermometer (6.1.7).

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 Pt 100 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

6.3 Measuring pipettes and/or measuring cylinders.

6.4 Oven, capable of being maintained 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.

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.

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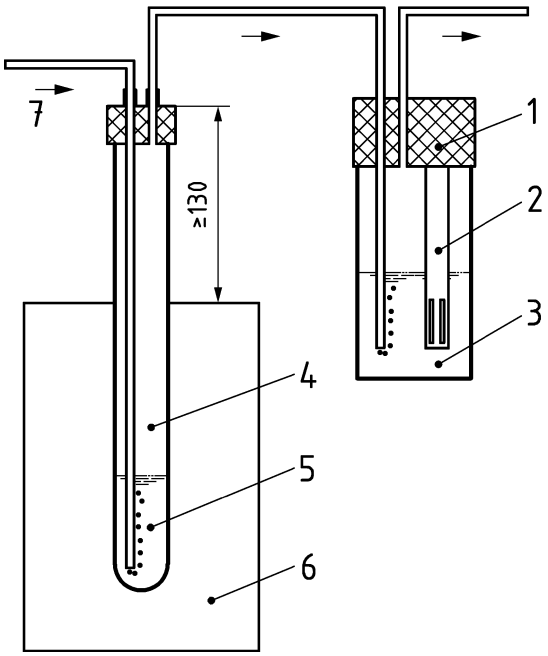

Key

1 Measuring vessel
2 Electrode
3 Distilled/demineralised water
4 Reaction vessel
5 Sample
6 Heating block
7 Air inlet

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Figure 2 — Heating block, reaction vessel and measurement cell

8 Preparation of measurement

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8.1 Preparation of test sample

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

— Take the required quantity from the centre of the carefully homogenised sample using a pipette

— Analyse the samples immediately after sample preparation.

8.2 Preparation of the apparatus

8.2.1 Cleaning procedure

The use of disposable reaction vessels, air inlet tubes and connecting hoses is recommended in order to minimize the impact of remaining impurities.

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 demineralised 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.

NOTE The drying time of at least 2 h assures that solvent adsorbed by the elastomers is removed completely.

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.