SIST

# SLOVENSKI STANDARD
# SIST EN 16568:2015

**01-marec-2015**

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**Goriva za motorna vozila - Metilni estri maščobnih kislin (FAME) goriv in mešanic z dizelskim gorivom - Določevanje oksidacijske 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

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

**Ta slovenski standard je istoveten z:** **EN 16568:2014**

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**ICS:**

75.160.20 Tekoča goriva Liquid fuels

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EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

**EN 16568**

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ICS 75.160.20

English Version

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

Carburants pour automobiles - Esters méthyliques d'acides gras (EMAG) et mélanges avec gazole - Détermination de la stabilité à l'oxydation par méthode d'oxydation plus accélérée à 120 °C

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

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

**CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels**



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# Foreword

This document (EN 16568:2014) 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 June 2015 and conflicting national standards shall be withdrawn at the latest by June 2015.

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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 [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 (2-EHN) at 110 °C and 120 °C was performed.

# 1 Scope

This European Standard specifies a test method for the determination of the oxidation stability at 120 °C of fuels for diesel engines, by means of measuring the induction period of the fuel up to 20 h. The method is applicable to blends of FAME with petroleum-based diesel having a FAME content in the range between 2 % (*V/V*) and 50 % (*V/V*).

NOTE 1 An almost identical 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 The precision of this method was determined using samples with a maximum induction period of approximately 20 h. Higher induction periods are not covered by the precision statement, however, the experience from EN 15751 indicates sufficient precision up to 48 h.

NOTE 3 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.

NOTE 4 For the purposes of this European Standard, the term "% (*V*/*V*)" is used to represent the volume fraction ($\varphi$) of a material.

# 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 (ISO 3170)*



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



EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

# 3 Terms and definitions

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

**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 measurement cell 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.

# 5 Reagents and materials

Use only reagents of recognized analytical grade, and distilled or demineralised water according to EN ISO 3696.

**5.1 Ternary solvent mixture,** consisting of methanol/toluene/acetone 1 : 1 : 1 (by volume).

**5.2 Alkaline laboratory glass cleaning solution.**

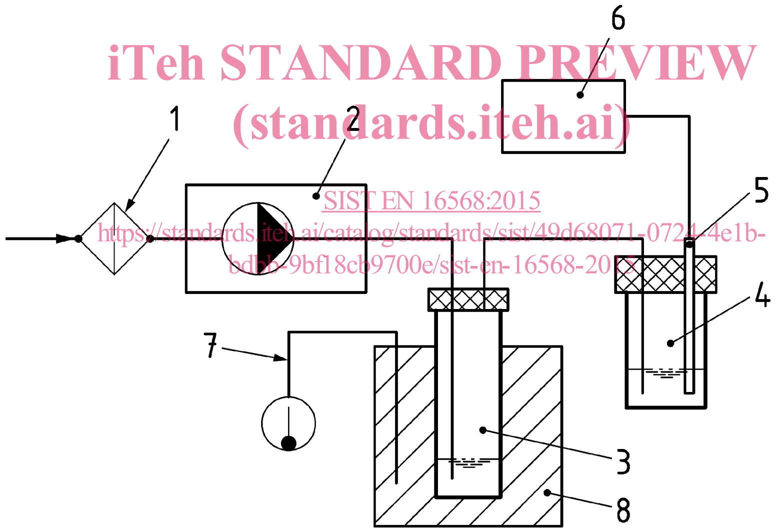
**5.3 2-Propanol.**

# 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 Figure 1 and Figure 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.






**Key**

| | | | |
|---|---|---|---|
| 1 | air filter (6.1.1) | 5 | electrode (6.1.5) |
| 2 | gas membrane pump with flow rate control (6.1.2) | 6 | measuring and recording apparatus (6.1.6) |
| 3 | reaction vessel (6.1.3) | 7 | thyristor and contact thermometer (6.1.7) |
| 4 | measurement cell (6.1.4) | 8 | heating block (6.1.8) |

**Figure 1 — Apparatus**

**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 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 an air 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 the conductivity within a range from 0 µS/cm to 300 µS/cm aligned with the dimensions of the measurement cell (6.1.4).

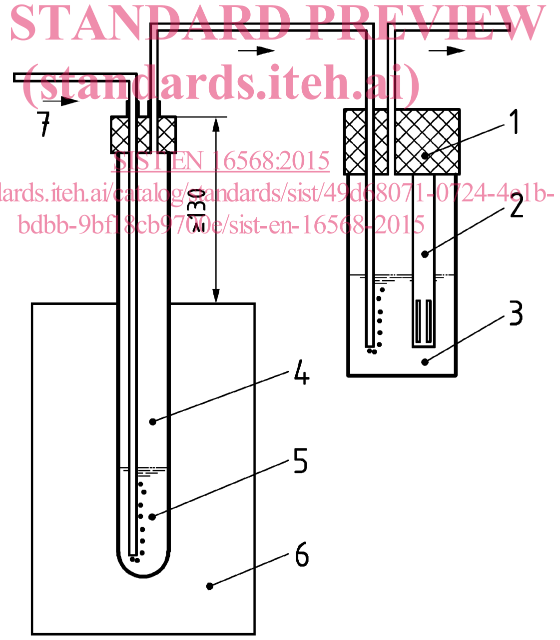
**6.1.6 Measuring and recording apparatus,** comprising of:

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.






**Key**

1 measurement cell (6.1.4)

2 electrode (6.1.5)

3 distilled/demineralised water

4 reaction vessel (6.1.3)

5 sample

6 heating block (6.1.8)

7 air inlet

**Figure 2 — Heating block, reaction vessel and measurement cell**

**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).