SLOVENSKI STANDARD
SIST EN 15751:2014

01-junij-2014

Nadomešča:

SIST EN 15751:2009

Goriva za motorna vozila - Metilni estri maščobnih kislin (FAME) goriv in mešanice z dizelskim gorivom - Ugotavljanje oksidativne stabilnosti z metodo pospešene oksidacije

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

Kraftstoffe für Kraftfahrzeuge - Fettsäuremethylester (FAME) Kraftstoff und Mischungen mit Dieselkraftstoff - Bestimmung der Oxidationsstabilität mit beschleunigtem Oxidationstest

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

Ta slovenski standard je istoveten z: EN 15751:2014

ICS:

75.160.20 Tekoča goriva Liquid fuels

SIST EN 15751:2014 en,fr,de

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15751

March 2014

ICS 75.160.20

Supersedes EN 15751:2009

English Version

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

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

Kraftstoffe für Kraftfahrzeuge - Kraftstoff Fettsäuremethylester (FAME) und Mischungen mit Dieselkraftstoff - Bestimmung der Oxidationsstabilität (beschleunigtes Oxidationsverfahren)

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Ref. No. EN 15751:2014 E

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Foreword

This document (EN 15751:2014) 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of an identical text or by endorsement, at the latest by September 2014 and conflicting national standards shall be withdrawn at the latest by September 2014.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 15751:2009.

Significant changes between this document and EN 15751:2009 are:

a) the limitation of the scope of the method to a maximum induction period of 48 h, reflecting the precision range of the method,

b) indication of a potential alteration of the induction period in the presence of cetane enhancers,

c) inclusion of the results of a short applicability check on non-petroleum based (such as Fischer-Tropsch synthesis or hydrotreatment process originated) diesel type of fuels (see Introduction),

d) editorial changes in order to clarify the test procedure.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

Introduction

This document is based on EN 14112 [1], which was specifically adapted for the determination of oxidation stability of fatty acid methyl esters (FAME). This method had been developed under CEN/TC 307 (Fats and oils). At the time of development the method was applicable for FAME fuel according to EN 14214 [2], but questions remained on the accuracy towards blends of FAME and diesel fuel.

The modifications to EN 14112 as given in this document, allow application of this test method for oxidation stability for pure FAME and diesel/FAME blends at various levels.

The goal was to have one single test method for FAME fuel, diesel/FAME blends and pure diesel fuels. Although the modifications cover FAME fuel and diesel/FAME blends, CEN/TC 307 decided that it was better to retain EN 14112 for methyl esters and publish a separate standard for all automotive fuel and heating oil applications, as the use of 'diesel and diesel blends' falls out the scope of CEN/TC 307.

While developing the fuels specification for paraffinic diesel fuel, three labs executed a small test on neat fuel and on 7 % (*V/V*) FAME blend based on product originating from both Fischer-Tropsch synthesis and hydrotreatment process. No indications towards a different interaction with the methodology of this document were found, so it was concluded that the stability of these paraffinic diesel fuels can be determined with the test method described in this document. The stability of these products usually is that high that the results do not match the scope of this European Standard.

The modifications required a new validation covering pure FAME, diesel/FAME blends and pure diesel fuels which resulted in the fact that the method is not suitable for pure petroleum-based diesel fuels.

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. 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 diesel fuel containing 2 % (*V/V*) of FAME at minimum.

NOTE 1 EN 14112 [1] describes a similar test method for oxidation stability determination of pure fatty acid methyl esters (see the Introduction to this European Standard).

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.

NOTE 3 The presence of cetane improver can reduce the oxidation stability determined by this test method. Limited studies with EHN (2-ethyl hexyl nitrate) indicated, however, that the stability is reduced to an extent which is within the reproducibility of the test method.

NOTE 4 For the purposes of this European Standard, the term "% (*V/V*)" is used to represent the volume fraction (φ) 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)*

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 target temperature which is 110 °C in the usual application of the method. 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.

5 Reagents and materials

Use only reagents of analytical grade and distilled or demineralised water [3].

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)[1)].

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 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 conductivity within a range of 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).

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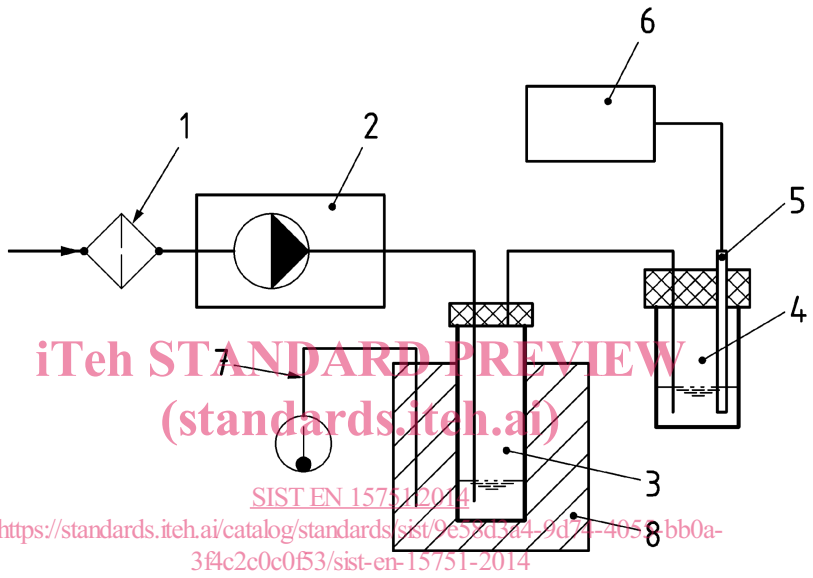
1) These are examples of suitable equipment which are given for the convenience of users of this European Standard. They do not constitute an endorsement by CEN of these products.

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.

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 Pt100 element, with a temperature range up to 150 °C, graduated in 0,1 °C.



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

6.3 Measuring pipettes and/or measuring cylinders

6.4 Oven, adjustable to a temperature 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.