SLOVENSKI STANDARD
SIST EN 15751:2009

01-september-2009

Goriva za motorna vozila - Metilni estri maġļ obne kisline (FAME) goriv in meġanice z dizelskim gorivom - Doloļ evanje oksidativne stabilnosti z metodo s pospeġeno oksidacijo

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

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

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

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75.160.20	Tekoļ a goriva	Liquid fuels

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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 gazole - Détermination de la stabilité à l'oxydation par méthode d'oxydation accélérée

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Foreword

This document (EN 15751:2009) 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of an identical text or by endorsement, at the latest by December 2009, and conflicting national standards shall be withdrawn at the latest by December 2009.

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Introduction

This document is based on EN 14112:2003 [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.

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 determining the oxidation stability of fuels for diesel engines. The method is applicable to fatty acid methyl esters (FAME) intended for use as pure biofuel or as a blending component for diesel fuels, and to blends of FAME and petroleum-based diesel containing 2 volume percentage of FAME at minimum.

NOTE 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). EN ISO 12205 [3] describes a test method that is applicable to pure petroleum-based diesel.

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 European Standard, 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 rapidly begins to increase

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 brought to a specified temperature. The vapours released during the oxidation process, together with the air, are passed into a flask containing water which has been demineralised or distilled and contains an electrode for measuring the conductivity. The electrode is connected to a measuring and recording device. It indicates the end of the induction period when the conductivity begins to increase rapidly. This accelerated increase is caused by 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 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 Appliance for the determination of oxidation stability, consisting of the following (see Figures 1 and 2 for diagrammatic representations):

NOTE An apparatus for determining oxidation stability can be obtained commercially 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 and are given for the convenience of users of this document. It does 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 diaphragm pump, with an adjustable flow rate of 10 l/h in combination with an apparatus to control the flow rate manually or automatically with a maximum deviation of ± 1,0 l/h from the set value.

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 stick out of the oven at least for 130 mm. Condensing volatile fuel components at the cold vessel walls outside the oven reduces evaporation losses to a minimum.

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. The cylindrical part of the vessel shall preferably be narrower a few centimetres below the top 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 be provided at the top with ventilation holes.

6.1.5 Electrodes, for measuring conductivity with a measuring 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 of:

a) an amplifier, and

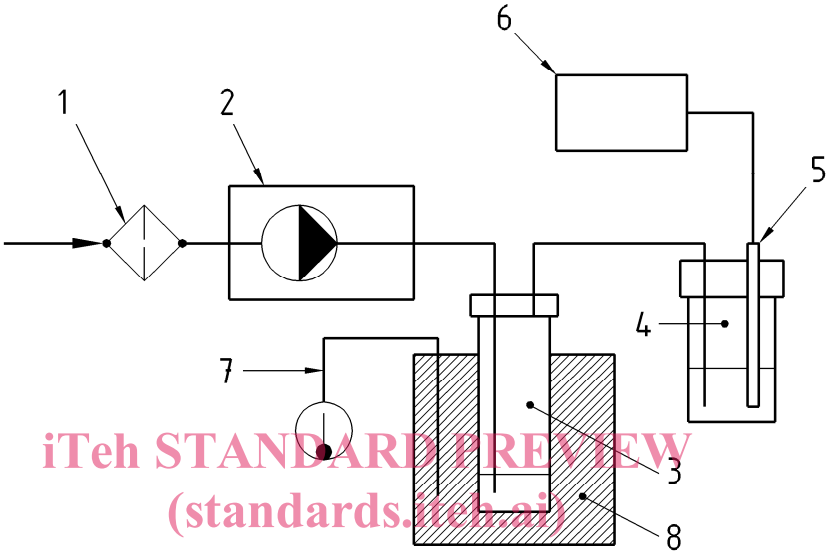
b) a recorder for registering the measuring signal of each of the electrodes (6.1.5).

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.

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)
2 Gas diaphragm 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 — Diagrammatic representation of the 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 in the commodity specification, 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.

It is important that the laboratory receive a sample which is truly representative and has not been damaged or changed during transport and storage.