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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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

This European Standard was approved by CEN on 9 November 2014.

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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of an identical text or by endorsement, at the latest by June 2015 and conflicting national standards shall be withdrawn at the latest by June 2015.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 iig.
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was pert. 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 $^{\circ}$ 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 (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)

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.