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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

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National foreword

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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

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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of an identical text or by endorsement, at the latest by June 2015 and conflicting national standards shall be withdrawn at the latest by June 2015.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 (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.

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.

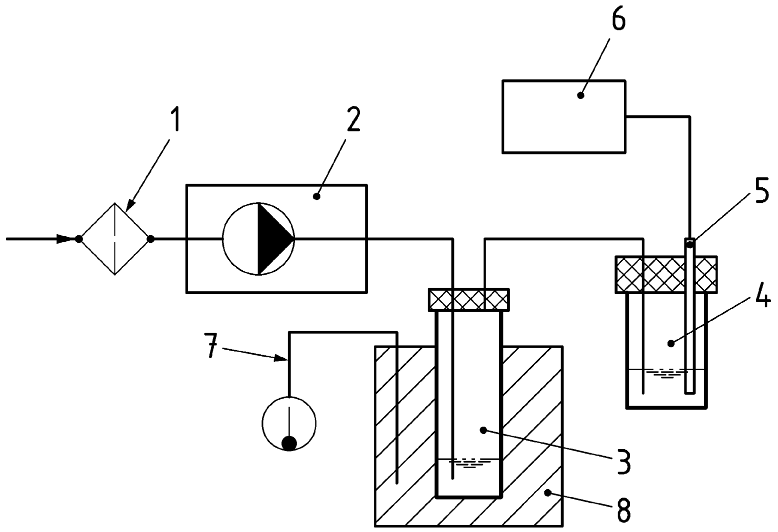
5.3 2-Propanol.

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.

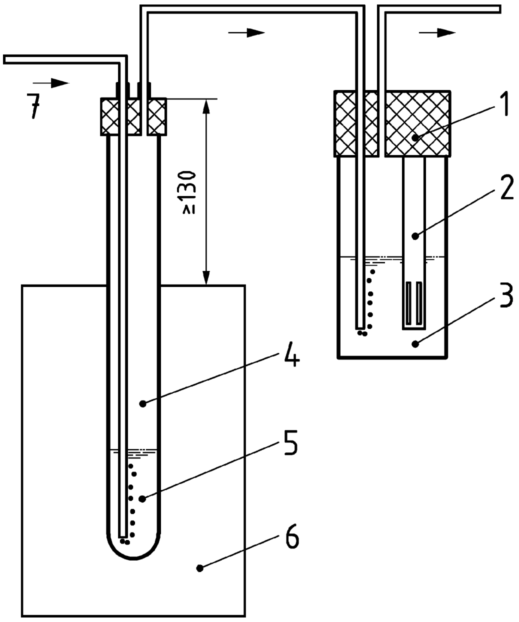
6.1.5 Electrodes for measuring the conductivity within a range from 0 µS/cm to 300 µS/cm aligned with the dimensions of the measurement cell (6.1.4).

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)	5	sample
2	electrode (6.1.5)	6	heating block (6.1.8)
3	distilled/demineralised water	7	air inlet
4	reaction vessel (6.1.3)		

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).

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.

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.

6.7 Balance, capable of weighing with an accuracy of ± 0,1 g or less.

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.

8 Preparation of measurement

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 homogenized 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 save the cleansing procedure and 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 ensures 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 after rinsing.

Rinse with 2-Propanol and tap water. Put the air inlet tube into the reaction vessel and fill completely with an aqueous alkaline laboratory cleaning solution.

Store the vessels at room temperature overnight.

Rinse the cleaned vessels and their air inlet tubes thoroughly with tap water and finally with demineralised or distilled water. Dry them in an oven at 80 °C for at least 2 h.

In case of doubt, the cleanliness of the sealing caps and connecting hoses can be checked by running a blank sample under standard test conditions. In this case the conductivity increase shall not exceed 10 µS/cm within 5 h.

8.2.2 Temperature correction

8.2.2.1 General

Any deviation from temperature of the fuel sample in the reaction vessel from the temperature in the heating block or heating bath has a significant impact on the result. In order to ensure that the correct measurement temperature is used, the difference between the temperature of the sample and the temperature of the heating block, ΔT, needs to be determined. For this determination a calibrated external temperature sensor is used.

The temperature correction always needs to be conducted when the test is carried out at a different temperature than before.

8.2.2.2 Procedure

Switch on the heating block and wait until the target temperature is reached and is stable.

Fill one reaction vessel with 7,5 g thermo-stable oil. Insert the temperature sensor through the sealing cap into the reaction vessel. Use distance clips to keep the sensor away from the air inlet tube. The sensor should touch the bottom of the vessel.

Insert the complete vessel into the heating block and connect the air supply.

If the value of the measured temperature is constant, calculate ΔT:

$$\Delta T = T_{\text{block}} - T_{\text{sensor}} \tag{1}$$

where

ΔT is the temperature difference between heating block and sample;

T_{block} is the temperature of the heating block;

T_{sensor} is the sample temperature in the reaction vessel measured by the sensor.

Adjust the temperature of the heating block according to Formula (2):

$$T_{\text{block}} = T_{\text{target}} + \Delta T \tag{2}$$

where

T_{target} is the intended measurement temperature.

EXAMPLE T_{target} is 120 °C. If a ΔT of +2°C is determined, the temperature of the heating block has to be set to 122 °C.

After this temperature correction the measured temperature in the reaction vessel should be equal to the target temperature.

9 Measurement

9.1 Set up the apparatus and the components as shown in Figure 1. If dedicated equipment is used, follow the manufacturer's instructions.

9.2 Attach the membrane pump (6.1.2) and adjust the airflow to exactly (10 ± 1) l/h. Switch off the pump. Dedicated instruments are usually equipped with automatic flow control.

9.3 Bring the heating block (6.1.8) up to the desired temperature of (120 ± 0,1) °C, using the thyristor and the contact thermometer (6.1.7) or by using an electronic temperature controller. The temperature shall be kept constant (±0,1 °C) during the test period (see also 8.2.2).

If a heating bath (6.1.8) is used, heat to the desired temperature and control the temperature according to the description above.

9.4 Fill the measurement cells (6.1.4) with 60 ml of distilled or demineralized water using a measuring pipette (6.3).

9.5 Check the electrodes (6.1.5) and adjust their signals to the zero axis of the recorder paper, using a calibration potentiometer.

Set the paper feed to 10 mm/h and the measuring frequency to one acquisition per 30 s. Set the measuring value of 200 µS/cm at the maximum result of 100 %.

If it is not possible to adjust the paper feed to 10 mm/h, use 20 mm/h. This shall be reported on the recorder paper.

NOTE Automatic oxidation stability analysers are mostly able to collect the data via PC.

9.6 Weigh (7,5 ± 0,1) g of the conditioned sample (see 8.1) into a reaction vessel (6.1.3) using a pipette (6.3).

9.7 When the test temperature is reached, switch on the membrane pump (6.1.2) and set the air flow to exactly (10 ± 1) l/h. Connect the air inlet tubes and outlet tubes to the reaction vessels and the measurement cells, using the connecting hoses (6.5).

9.8 Place the reaction vessel with the sealing cap (6.1.3) into the corresponding hole in the heating block or into the heating bath (6.1.8).

The preparation steps 9.7 and 9.8 shall be carried out as fast as possible. Then immediately start the automatic data recording or note the start time on the recorder paper.

9.9 The measurement may be terminated:

— when the signal has reached 100 % of the recorded scale, usually 200 µS/cm (see Figure 3, upper diagram), or

— when the conductivity curve flattens again after exceeding the induction period (see Figure 3, lower diagram). Care should be taken to ensure that the test is not terminated before the curve has flattened sufficiently to fit an accurate second tangent.

— after 48 h of testing time.

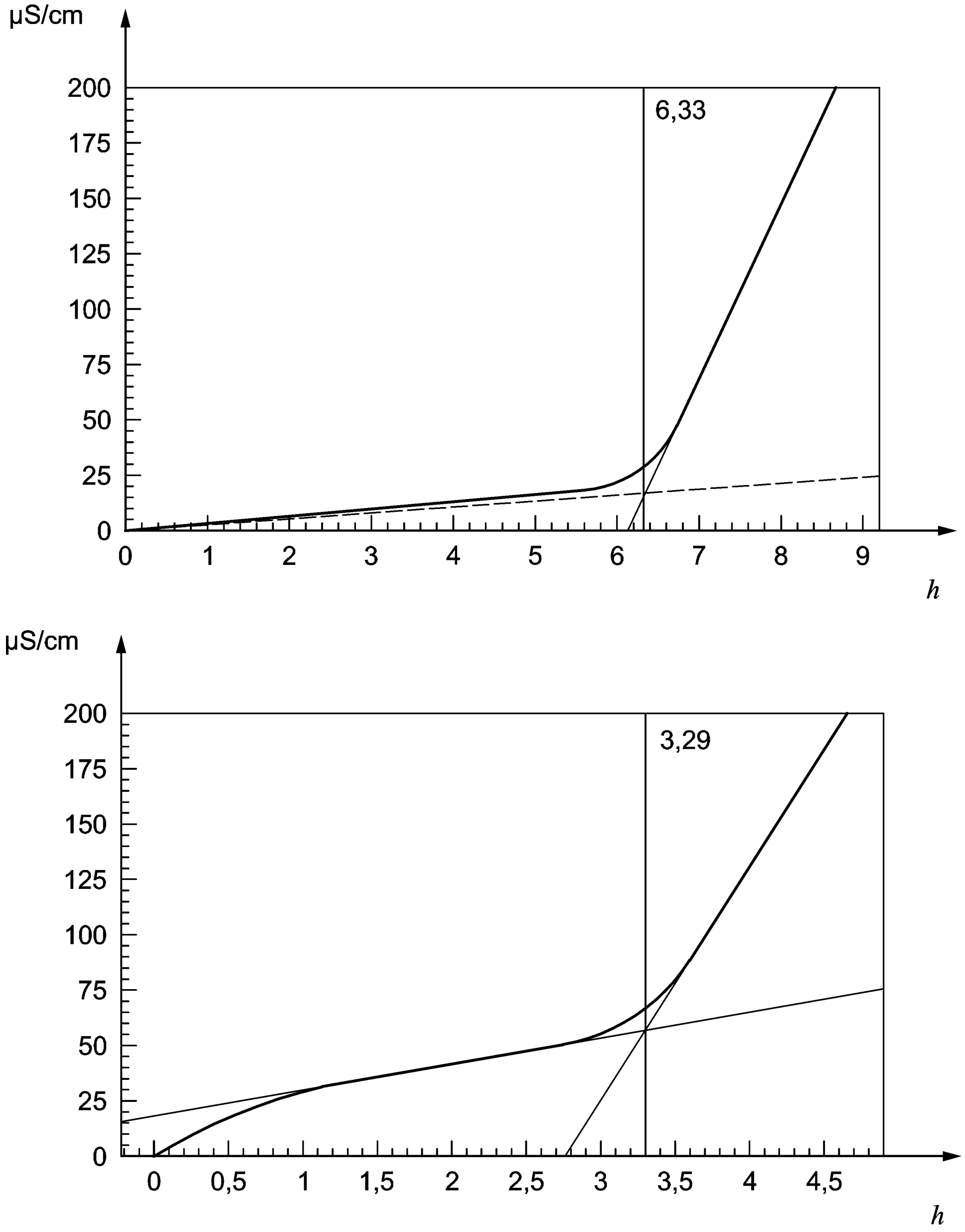


Figure 3 — Measurement termination indications

9.10 During the determination, check the following parameters:

a) The setting of the flow meter. Adjust where necessary in order to ensure a constant flow;

b) The colour of the molecular sieve (6.6) of the air filter. Repeat measurements when the molecular sieve changes colour during the test. It is recommended to exchange the molecular sieve prior to each run.

At temperatures above approximately 25 °C, volatile carboxylic acids can evaporate from the measurement cell. This may lead to a decrease of the conductivity of the aqueous solution, thus causing significant deviations of the conductivity curve (see [4]).

A rapid conductivity increase immediately after starting the test and before reaching the induction period may indicate insufficient cleaning of the sealing caps or connecting hoses (evaporation of residual volatile compounds from the elastomers) (see Figure 4). The cleanliness can be validated according to the procedure given in 8.2.1. Also fuels that contain volatile acids can unexpectedly show a rapid initial conductivity increase (see Figure 5).

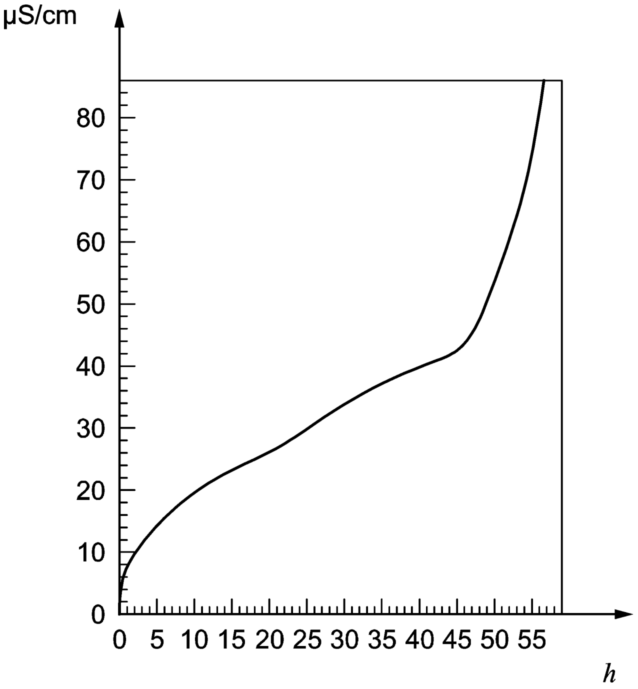


Figure 4 — Indication for insufficient cleaning

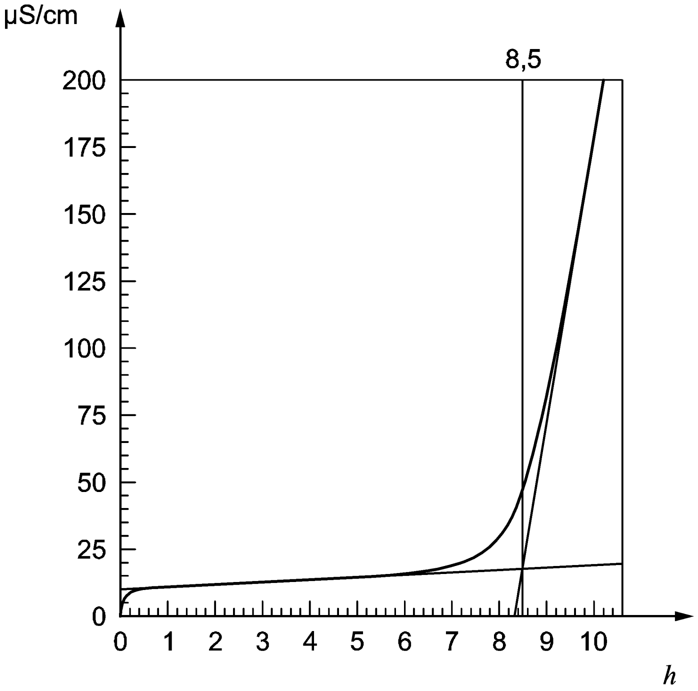


Figure 5 — Indication for rapid initial conductivity increase

10 Calculation and evaluation

10.1 Automatic evaluation

The automatic evaluation as given by the equipment manufacturers may be used if the second derivative of the conductivity curve shows a clear maximum. This is generally the case if diesel/FAME blends with FAME content equal to or higher than 10 % (*V*/*V*) are investigated (see Figure 6, left diagram).

If the second derivative of the conductivity curve is noisy and no clear maximum can be identified, the manual evaluation (10.2) of the conductivity curve itself shall be applied (see Figure 6, right diagram).

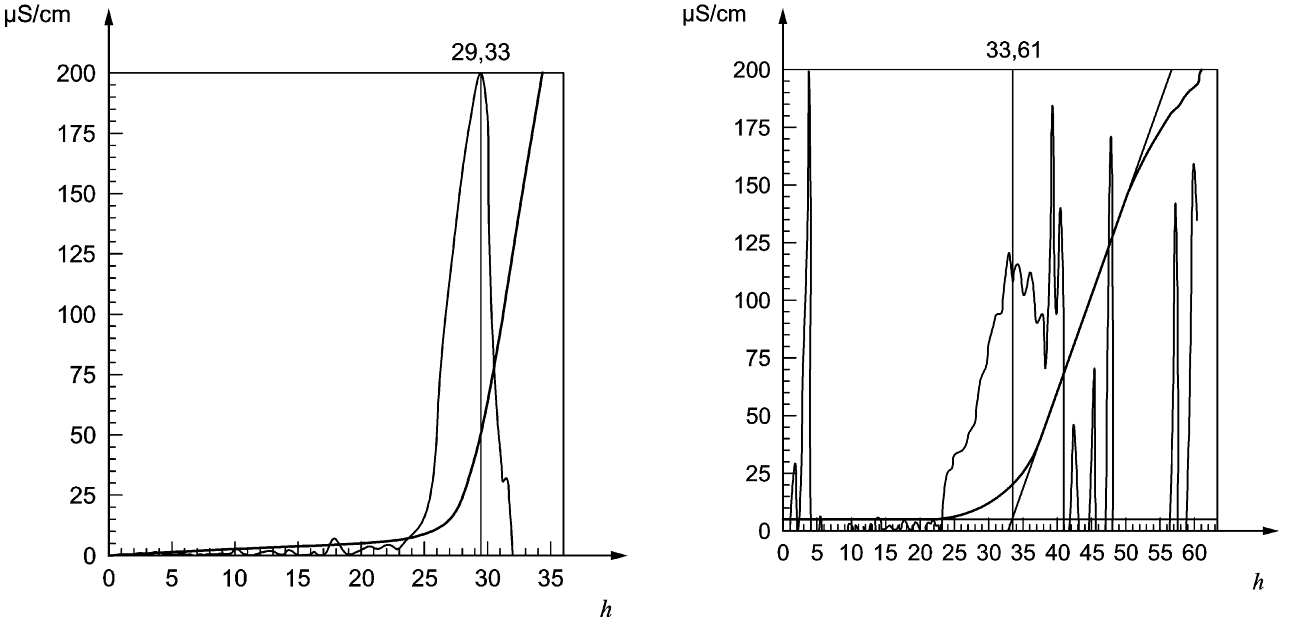


Figure 6 — Evaluation indications

It is recommended to use software settings that permit simultaneous display of the conductivity curve and its second derivative in order to enable the operator to check the automatically calculated value for the induction period.

10.2 Manual evaluation

Set the first tangent to the flattest part of the slowly increasing conductivity curve. Great care shall be taken to fit the best possible tangent line, e.g. by using an enlarged presentation of the original graph. Some instruments supply a zoom-function to accomplish this. The second tangent is set after exceeding the inflexion point at the steepest part of the conductivity curve (see Figure 3).

The induction period is obtained from the intersection point of both tangents.

11 Expression of results

Report the induction period, obtained from 10.1 or 10.2, in hours, rounded to the nearest 0,1 h.

If the induction period exceeds 48 h and the measurement is stopped, the result shall be reported as “> 48 h”.

12 Precision

12.1 General

An interlaboratory study organized in 2011 on a European level with nine participating laboratories, carried out on six samples (B5 to B50), was used to develop the precision statement according to EN ISO 4259 [5].

Results from the precision calculation shall be rounded to the nearest 0,1 h.

12.2 Repeatability, *r*

The difference between two test results, obtained by the same operator with the same apparatus under constant operating conditions on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values calculated from the following equation in absolute value only in one case in twenty, where X represents the mean of the two results:

$$r = 0{,}144\,4 + 0{,}045\,6\,X \qquad (3)$$

12.3 Reproducibility, *R*

The difference between two single and independent test results, obtained by different operators working in different laboratories on identical test material would, in the long run, in the normal and correct operation of the test method, exceed the values calculated from the following equation in absolute value only in one case in twenty, where X represents the mean of the two results:

$$R = 0{,}288\,8 + 0{,}139\,5\,X \qquad (4)$$

13 Test report

The test report shall specify:

a) a reference to this European Standard (i.e. EN 16568:2014);

b) the type and complete identification of the product tested;

c) the sampling method used if known (see Clause 7);

d) the temperature at which the determination was carried out;

e) the test result(s) obtained (see Clause 11), or if the repeatability has been checked, the final quoted result obtained;

f) all operating details not specified in this European Standard, or regarded as optional, together with details of any incidents which may have influenced the test result(s);

g) any deviation, by agreement or otherwise, from the procedure specified;

h) the date of test.

Annex A
(informative)

Background of the method

It has been established that the phenomenon of fuel ageing consists of two consecutive phases, starting with the depletion of the ageing reserve with few chemical changes to the bulk material, followed by the fuel ageing process itself during which the fuel is badly decomposed, forming ageing polymers and acids.

The first phase, the depletion of the ageing reserve, can be accurately monitored by means of accelerated oxidation tests EN 14112 [2] and EN 15751 [1]. Both tests measure the induction period, i.e. how long the fuel is stable under standardized laboratory conditions, and monitor fuel oxidation by tracking conductivity. The conductivity is recorded in a cell equipped with an electrode and filled with water. This cell is constantly flushed with the air that was passed through the fuel sample before. As soon as the fuel oxidizes and the first ageing acids are formed, the air stream carries volatile organic acids into the measurement cell. The end of the induction period is indicated by a strong conductivity increase.

The presence of cetane improvers like 2-ethyl hexyl nitrate (2-EHN) in fuel can influence the level of oxidation stability, even in a disproportionate way depending on the test temperature. For the modifications to EN 15751 as given in this document, the destabilizing effect of 2-EHN was studied at 120 °C. It was visible based on the statistical data gathered but still within the precision range of this test method. A further increase of the oxidation temperature, however, is not recommended.

Bibliography

[1] EN 15751, *Automotive fuels - Fatty acid methyl ester (FAME) fuel and blends with diesel fuel - Determination of oxidation stability by accelerated oxidation method*

[2] EN 14112, *Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of oxidation stability (accelerated oxidation test)*

[3] EN ISO 12205, *Petroleum products - Determination of the oxidation stability of middle-distillate fuels (ISO 12205)*

[4]. DE MAN, J. M., FAN TIE and DE MAN. *L. J. Am. Oil Chem. Soc.* 1987, **64** p. 993

[5] EN ISO 4259, *Petroleum products - Determination and application of precision data in relation to methods of test (ISO 4259)*

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