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

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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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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of an identical text or by endorsement, at the latest by September 2014 and conflicting national standards shall be withdrawn at the latest by September 2014.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.

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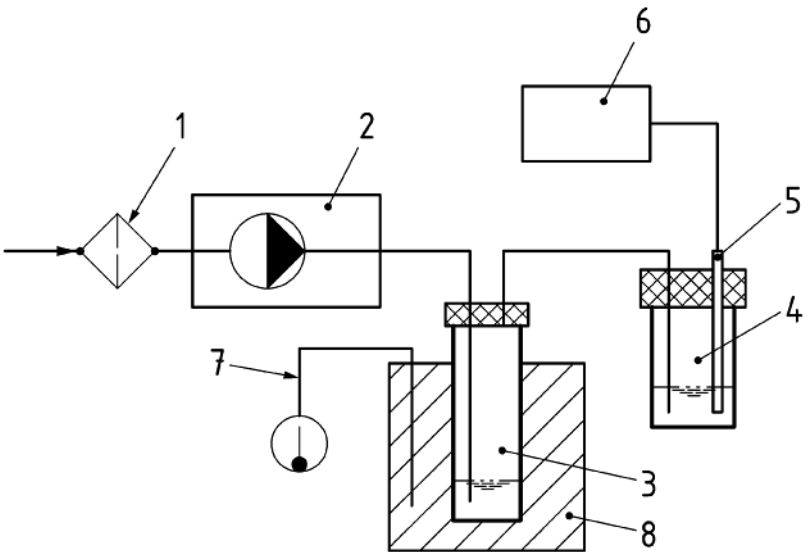
1) These are examples of suitable equipment which are given for the convenience of users of this European Standard. They do not constitute an endorsement by CEN of these products.

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.

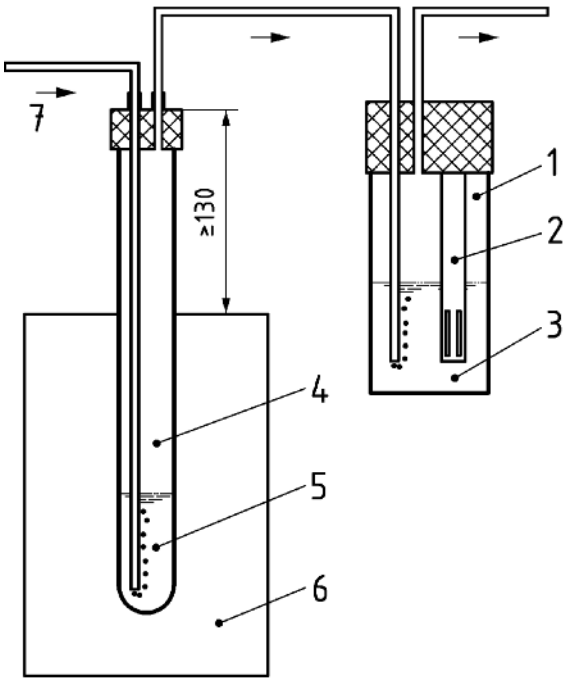
7 Sampling

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.

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.

Dimensions in millimetres



Key

1	measuring vessel	5	sample
2	electrode	6	heating block
3	distilled/demineralised water	7	air inlet
4	reaction vessel		

Figure 2 — Diagrammatic representation of heating block, reaction vessel and measurement cell

8 Preparation of measurement

8.1 Preparation of test sample

In order to ensure a consistent test condition, all samples shall be treated in the way described below:

— Take the required quantity from the centre of the carefully homogenised sample using a pipette.

— Analyse the samples immediately after sample preparation.

8.2 Preparation of apparatus

8.2.1 Cleaning procedure

NOTE 1 The use of new disposable reaction vessels, air inlet tubes and connecting hoses is recommended in order to save the cleaning procedure.

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 2 The drying time of at least 2 h assures 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.

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

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 the test temperature in the test vessel 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 5 g thermo-stable oil. Insert the temperature sensor through the cap into the reaction vessel. Use distance clips to keep the sensor away from the air inlet. 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:

$$\Delta T = T_{\text{block}} - T_{\text{sensor}} \quad (1)$$

where

ΔT is the temperature difference between heating block and sample;

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

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

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

$$T_{block} = T_{target} + \Delta T \quad (2)$$

where

T_{target} is the intended measurement temperature.

EXAMPLE T_{target} is 110 °C. If a ΔT of +2 °C is determined, the temperature of the heating block has to be set to 112 °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 as shown in Figure 1. If commercially available equipment is used, follow the manufacturer's instructions.

9.2 Attach the membrane pump (6.1.2) and adjust the air flow 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) to a temperature such that the required test temperature (usually 110 °C, but see 8.2.2) is reached in the test tube(s), 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 8.2.2.

9.4 Fill the measurement cells (6.1.4) with 60 ml of distilled or demineralised 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 might be able to collect the data via a computer system.

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

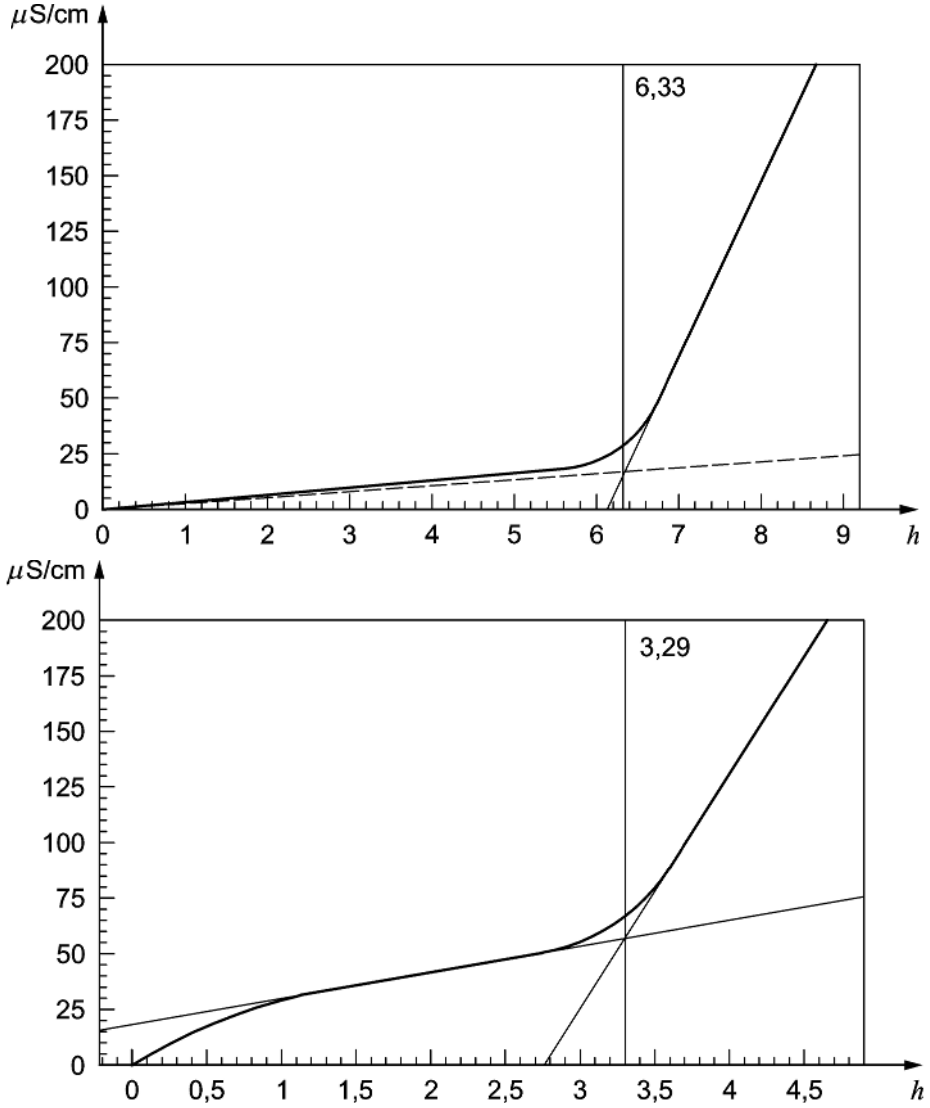
9.8 Place the reaction vessel with the sealing cap (6.1.3) into the corresponding hole in the heating block or into the heating bath (6.1.8).

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, or

— when the curve levels after reaching the inflexion point (see Figure 3), or after 48 h of testing time.



Key

X-axis time (h)

Y-axis signal (µS/cm)

Figure 3 — Measurement termination indications

Care should be taken to not stop the test too early to ensure the calculation of an accurate second tangent.

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.

NOTE 1 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 [6]).

NOTE 2 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 Note 1 in 8.2.1. Also fuels that contain volatile acids can unexpectedly show a rapid initial conductivity increase (see Figure 5).

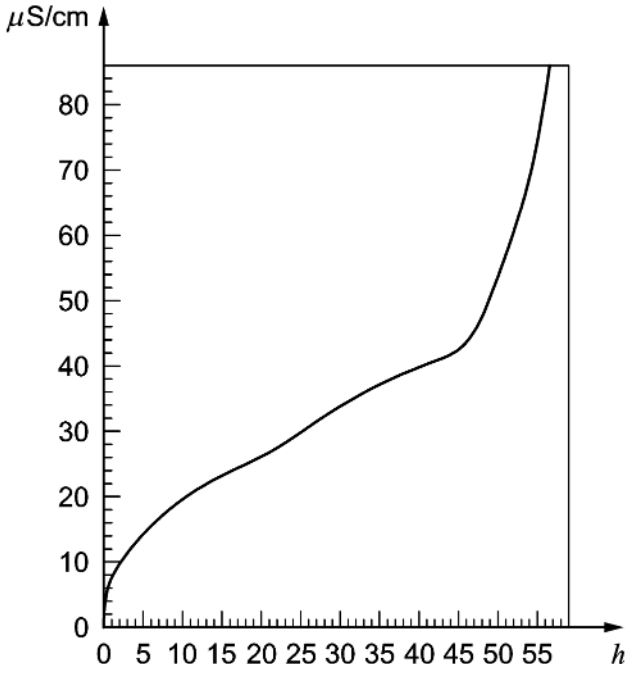


Figure 4 — Indication for insufficient cleaning

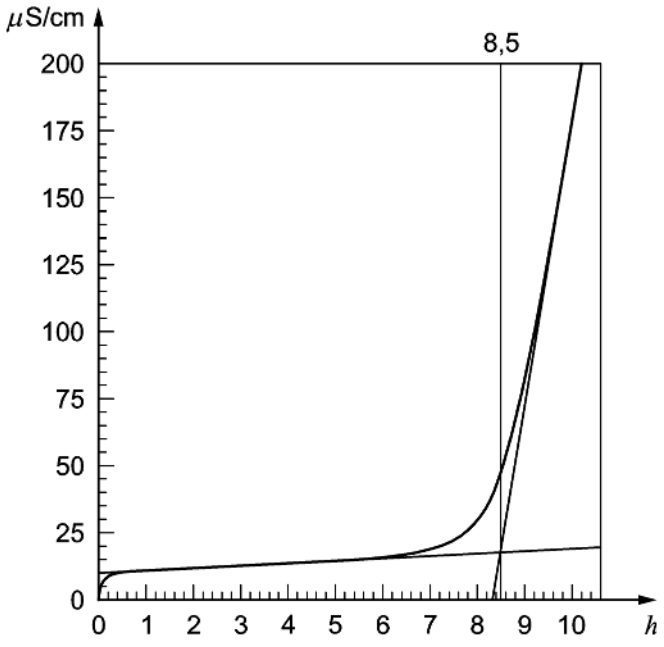


Figure 5 — Indication for rapid initial conductivity increase

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 pure FAME and diesel/FAME blends with a FAME content equals or higher than 10 % (*V/V*) are investigated (see Figure 6, upper diagram).

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

NOTE Software settings are recommended 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.

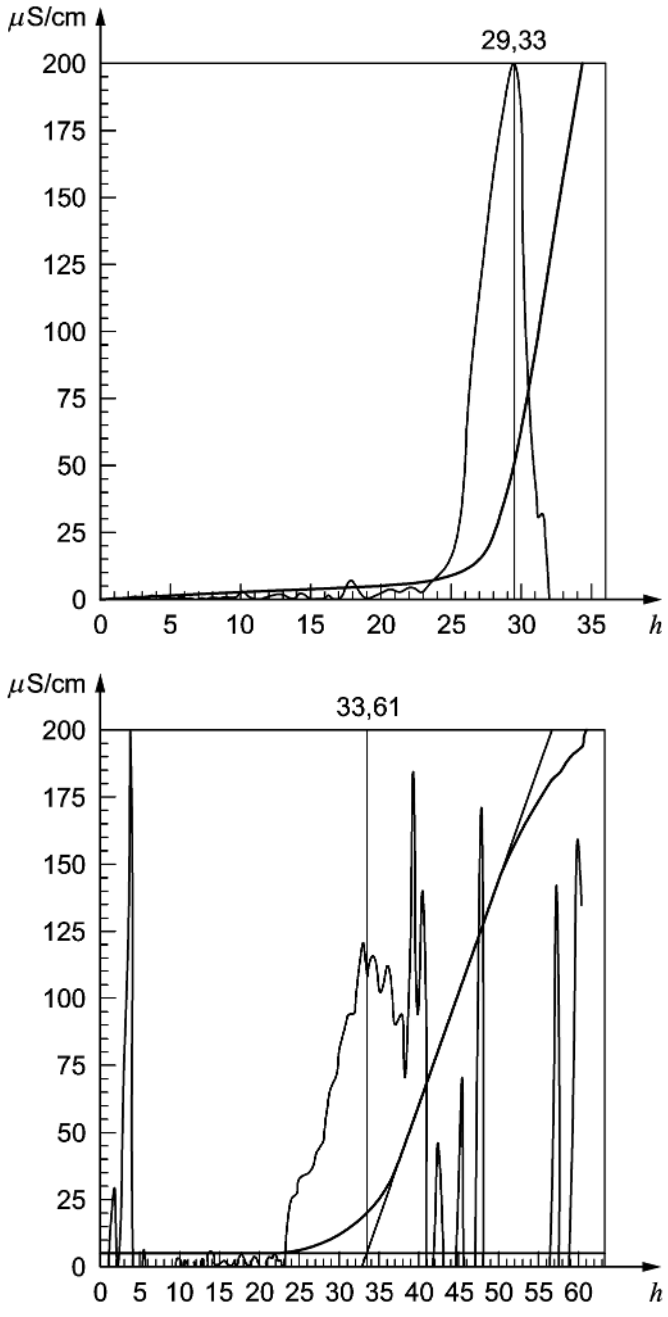


Figure 6 — Evaluation indications

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 and 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 test organized in 2007 at European level with the participation of 12 laboratories was carried out on 10 samples and gave the precision, derived from statistical analysis by EN ISO 4259 [4]. See Introduction for further work on paraffinic diesel fuel types.

Results from the calculation of precision estimates used 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 formula in absolute value only in one case in twenty.

$$r = 0{,}220\ 27 + 0{,}043\ 44\ X \tag{3}$$

where

X is the mean of the two results.

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 formula in absolute value only in one case in twenty.

$$R = 0{,}372\ 69 + 0{,}190\ 38\ X \tag{4}$$

where

X is the mean of the two results.

13 Test report

The test report shall specify:

a) a reference to this European Standard (i.e. EN 15751);

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

In the method described in this European Standard, the oxidation process is split in two phases.

a) The first phase (the induction period) is characterised by slow reaction of oxygen during which peroxides are formed.

b) The second phase is characterised by rapid reaction in which peroxides are not only formed but these peroxides are then dissociated under the influence of the high temperature. During this reaction, products such as aldehydes, ketones and short chain carboxylic acids are formed.

The method described in this European Standard is a conductometric determination of volatile carboxylic acids (mainly formic acid and acetic acid) produced during oxidation. The procedure was published in 1974 [7].

An automated potentiometric determination method was published in 1972 [8] and the method was standardized by ISO/TC 34/SC 11 as ISO 6886 in 1996 (later revised in 2006, [5]).

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[2] EN 14214:2003, *Automotive fuels - Fatty acid methyl esters (FAME) for diesel engines - Requirements and test methods*

[3] EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696)*

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