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BSI Standards Publication

Fat and oil derivatives — Fatty Acid Methyl Esters (FAME) — Determination of oxidation stability (accelerated oxidation test)

National foreword

This British Standard is the UK implementation of EN 14112:2016. It supersedes BS EN 14112:2003 which is withdrawn.

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Produits dérivés des corps gras - Esters méthyliques d'acides gras (EMAG) - Détermination de la stabilité à l'oxydation (Essai d'oxydation accélérée)

Erzeugnisse aus pflanzlichen und tierischen Fetten und Ölen - Fettsäure-Methylester (FAME) - Bestimmung der Oxidationsbeständigkeit (Beschleunigte Oxydationsprüfung)

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (EN 14112:2016) has been prepared by Technical Committee CEN/TC 307 “Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis”, the secretariat of which is held by AFNOR.

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of an identical text or by endorsement, at the latest by March 2017, and conflicting national standards shall be withdrawn at the latest by March 2017.

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

This document supersedes EN 14112:2003.

Significant changes between this document and EN 14112:2003 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) editorial changes in order to clarify the test procedure;

d) addition of Clause 2 – Normative references;

e) addition of Clause 11 – Expression of results;

f) background information on the method added as Annex A.

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Introduction

This document is based on EN 14112:2003, 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).

The modifications as given in this document address the field experience with this method made since its introduction as a standard test method. Editorial changes are made in order to specify some aspects of the test. Additionally, the cleaning procedure is modified based on field experience.

1 Scope

This European Standard specifies a method for the determination of the oxidation stability of fatty acid methyl esters (FAME) at 110 °C, by means of measuring the induction period up to 48 h.

NOTE 1 EN 15751 [1] describes a similar test method for oxidation stability determination of pure fatty acid methyl esters and of blends of FAME with petroleum-based diesel containing 2 % (V/V) of FAME at minimum.

NOTE 2 The precision statement of this test method was determined in a Round Robin exercise with induction periods up to 8,5 h, thus covering the limit value in EN 14214. Results from precision studies on EN 15751 indicate that the precision statement is valid for induction periods up to 48 h but not for higher values.

NOTE 3 Limited studies on EN 15751 with EHN (2-ethyl hexyl nitrate) on FAME blends indicated that the stability is reduced to an extent which is within the reproducibility of the test method. It is likely that the oxidation stability of pure FAMEs is also reduced in the presence of EHN when EN 14112 is used for testing.

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 (dried) 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 demineralized 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 demineralized water.

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) [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 sealing cap shall be fitted with a gas 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 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).

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.

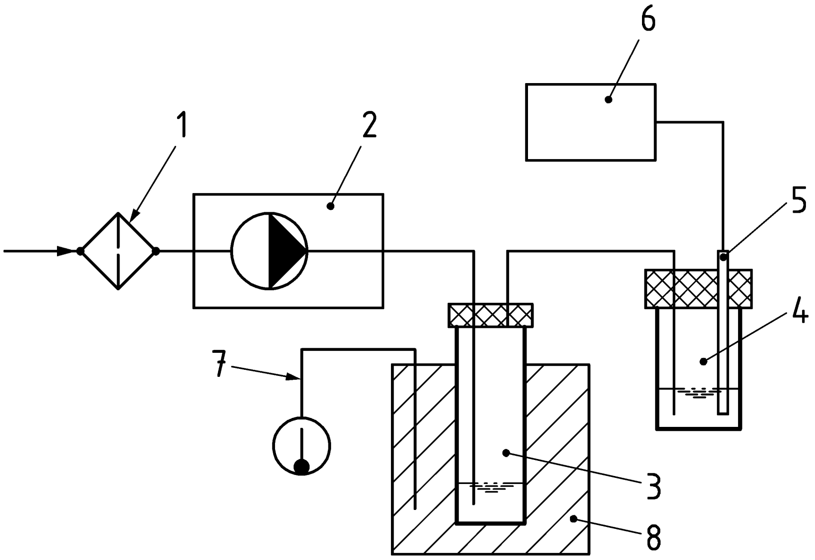
---

[1] Rancimat is the trade name of a product supplied by Metrohm AG, Herisau, Switzerland; OSI is the trade name of a product supplied by Omnion Inc., Rockland, Massachusetts, USA. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN or CENELEC of the products named. Equivalent products may be used if they can be shown to lead to the same results.

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.

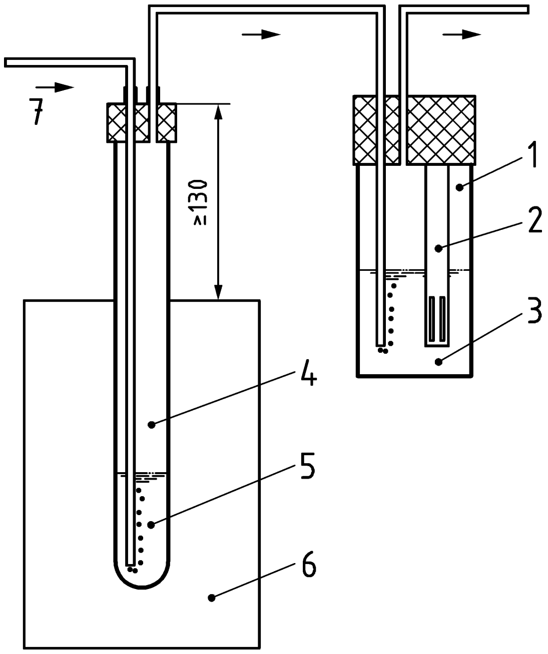


Key

1 air filter (6.1.1)
2 gas membrane 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 — Apparatus

Dimensions in mm



Key

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

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

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.

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 a 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 apparatus

8.2.1 Cleaning procedure

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 demineralized 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.

Residual fuel and aging products from the previous experiment and the solvent from the cleaning may remain adsorbed in the elastomers and shall be removed. A drying time of 2 h ensures that all volatile compounds are removed.

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 demineralized 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 between the temperature of the fuel sample in the test vessel and the temperature of the heating block or the 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 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:

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

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 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 demineralized water using a measuring pipette (6.3).

9.5 Check the electrodes (6.1.5) and adjust their signals using a calibration potentiometer.

9.5.1 The data are recorded by the software of the PC.

9.5.2 If an evaluation using a PC is not possible, proceed as follows: 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.

9.6 Weigh (3,0 ± 0,01) 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).

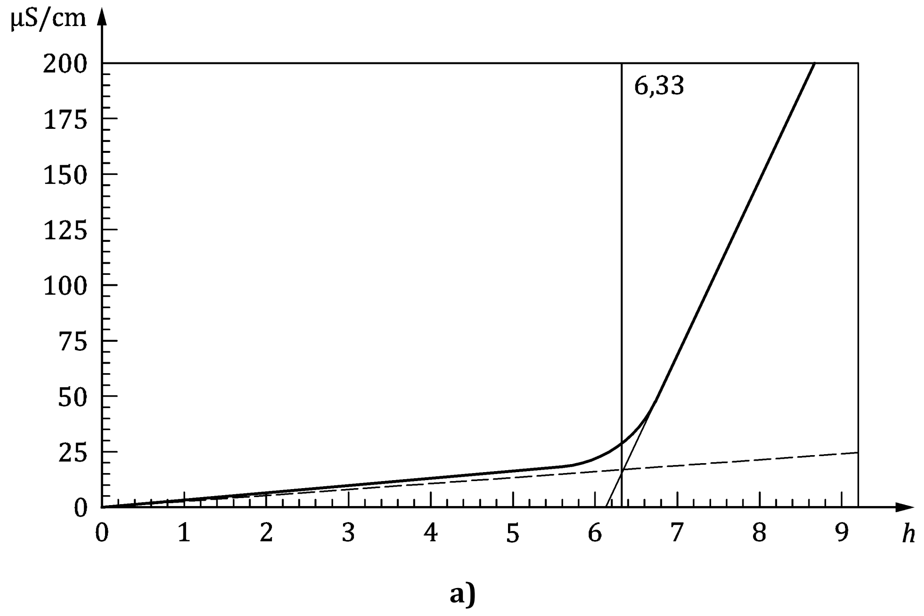
The preparation steps 9.7 and 9.8 shall be carried out as fast as possible. Then immediately start the automatic data recording or note the start time on the recorder paper.

9.9 The measurement may be terminated:

— when the signal has reached 100 % of the recorded scale, usually 200 µS/cm (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; or

— after 48 h of testing time.



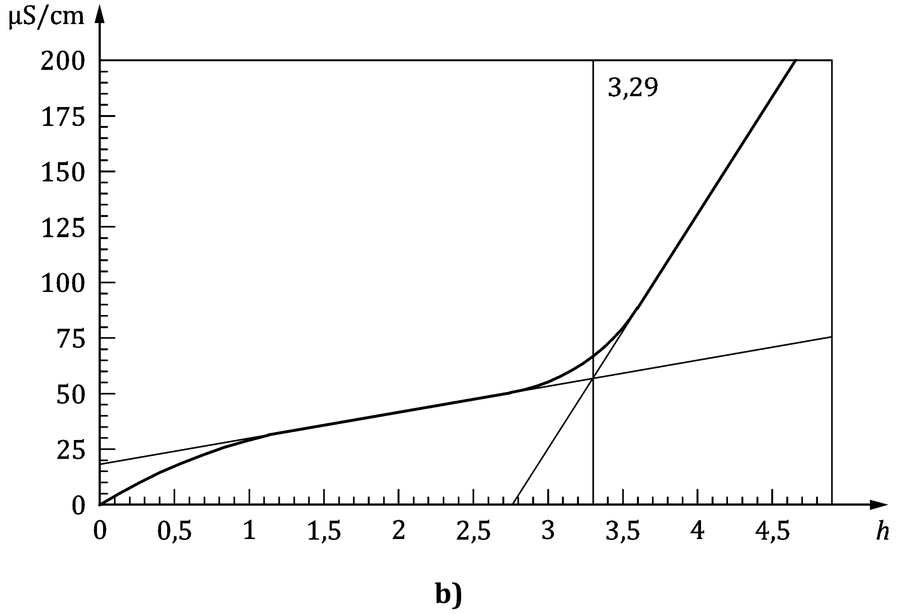


Figure 3 — Measurement termination indications

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.

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 Bibliographical Reference [2]).

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 5). The cleanliness can be validated according to the procedure given in 8.2.1, 1st paragraph. Also fuels that contain volatile acids can unexpectedly show a rapid initial conductivity increase (see Figure 6).

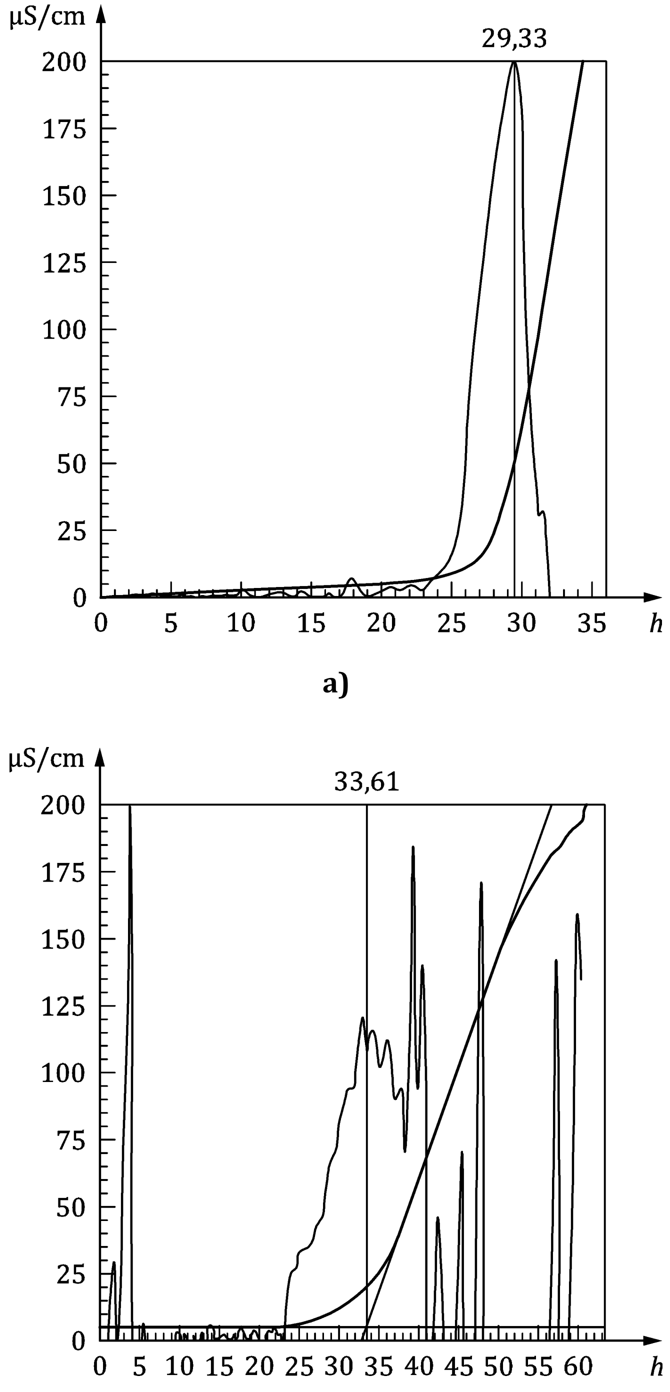
10 Calculation and Evaluation

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 4, upper diagram).

If the second derivative of the conductivity curve is noisy and no clear maximum can be recognized, the manual evaluation (10.2) of the conductivity curve itself shall be applied (see Figure 4, lower diagram).

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.



a)

b)

Figure 4 — Evaluation indications

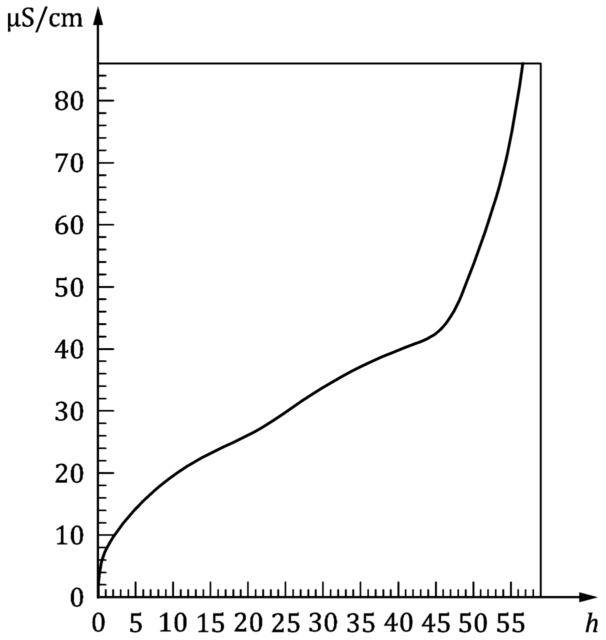


Figure 5 — Indication for insufficient cleaning

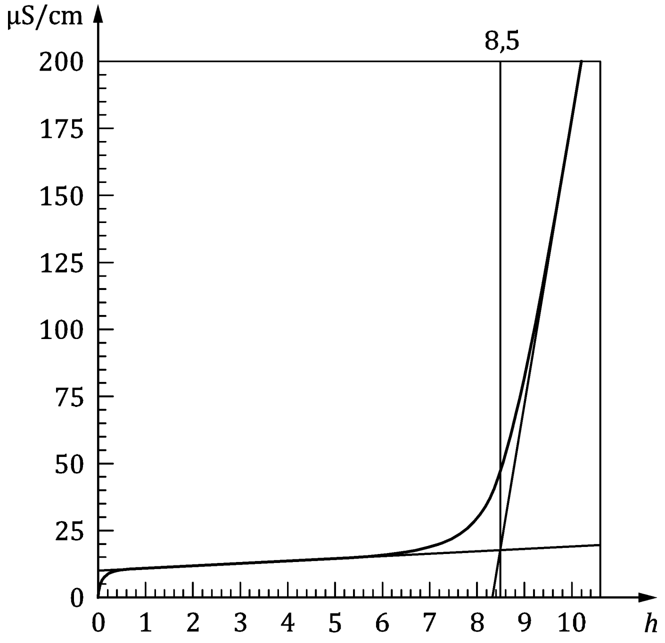


Figure 6 — Indication for rapid initial conductivity increase

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 2000 at European level with the participation of 8 laboratories was conducted on 8 samples at a test temperature of 110 °C and gave the statistical results indicated in Annex B.

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 20.

$$r = 0{,}09 \cdot X + 0{,}16 \tag{3}$$

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 20.

$$R = 0{,}26 \cdot X + 0{,}23 \tag{4}$$

where

X is the mean of the two results.

13 Test report

The test report shall at least specify:

a) a reference to this European standard (i.e. EN 14112:2016);

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 and EN 15751. 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.

Annex B
(informative)

Results of an Interlaboratory Study

A European collaborative study involving 8 laboratories in 5 countries was conducted on 8 samples:

- Sample 1: mixture of rapeseed oil and sunflower oil FAME (not distilled, 1 month old);
- Sample 2: frying oil FAME (not distilled, 1 month old);
- Sample 3: palm oil fraction (C16-C18) FAME (distilled, 1 month old);
- Sample 4: rapeseed oil FAME producer n°3 (not distilled, 1 month old);
- Sample 5: rapeseed oil FAME producer n°2 (distilled, 1 month old);
- Sample 6: sunflower oil FAME (distilled, 2 months old);
- Sample 7: rapeseed oil FAME producer n°1 (not distilled, 2 months old);
- Sample 8: rapeseed oil FAME - storage tank (4 months old).

The test was organized by CEN/TC 307/WG 1 in 2000 and the results obtained were subject to statistical analysis in accordance with EN ISO 4259 to give the precision data shown in Table B.1.

Table B.1 — Thermal oxidation stability

Sample	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
N° of participating laboratories	8	8	8	8	8	8	8	8
N° of participating laboratories after eliminating outliers	7	7	7	6	6	7	6	7
Mean value (h)	**5,42**	**8,48**	**0,92**	**5,64**	**1,20**	**2,90**	**4,87**	**0,97**
Repeatability standard deviation	0,21	0,36	0,09	0,06	0,03	0,31	0,10	0,06
Reproducibility standard deviation	0,36	0,71	0,16	0,44	0,08	0,34	0,63	0,14
Repeatability limit, *r*	0,69	1,22	0,29	0,20	0,11	1,02	0,35	0,20
Reproducibility limit, *R*	1,20	2,39	0,52	1,53	0,29	1,14	2,17	0,46

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