BS EN 16896:2016

BSI Standards Publication

Petroleum products and related products — Determination of kinematic viscosity — Method by Stabinger type viscosimeter

bsi.

National foreword

This British Standard is the UK implementation of EN 16896:2016.

The UK participation in its preparation was entrusted to Technical Committee PTI/13, Petroleum Testing and Terminology.

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

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

Published by BSI Standards Limited 2016

ISBN 978 0 580 90557 5

ICS 75.160.20

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 November 2016.

Amendments/corrigenda issued since publication

Date	Text affected

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 16896

November 2016

ICS 75.160.20

English Version

Petroleum products and related products - Determination of kinematic viscosity - Method by Stabinger type viscosimeter

Produits pétroliers et produits relatés - Détermination de la viscosité cinématique - Méthode par Viscometer Stabinger

Mineralölerzeugnisse und verwandte Produkte - Bestimmung der kinematischen Viskosität - Verfahren mit dem Viskosimeter nach dem Stabinger-Prinzip

This European Standard was approved by CEN on 27 August 2016.

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

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

Ref. No. EN 16896:2016 E

Contents

Page

European foreword

This document (EN 16896:2016) 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of an identical text or by endorsement, at the latest by May 2017, and conflicting national standards shall be withdrawn at the latest by May 2017.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.

According to the CEN-CENELEC Internal Regulations, the national standards organisations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard specifies a procedure for the determination of kinematic viscosity (ν) at 40 °C in the range from 2 mm²/s to 6 mm²/s by calculation from dynamic viscosity (η) and density (ρ) of middle distillate fuels, fatty acid methyl ester fuels (FAME) and mixtures of these using the Stabinger-type viscosimeter.

The result obtained using the procedure described in this standard depends on the behaviour of the sample. This European Standard should be used predominantly on liquids whose shear stress and shear rate are proportional (Newtonian flow behaviour). However, if the viscosity changes significantly with the shear rate, comparison with other measuring methods is only permissible at similar shear rates.

WARNING — The use of this Standard can involve hazardous materials, operations and equipment. This Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of users of this standard to take appropriate measures to ensure the safety and health of personnel prior to the application of the Standard, and fulfil statutory and regulatory requirements for this purpose.

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 3104, *Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity (ISO 3104)*

EN ISO 3170, *Petroleum liquids - Manual sampling (ISO 3170)*

EN ISO 3171, *Petroleum liquids - Automatic pipeline sampling (ISO 3171)*

EN ISO 12185, *Crude petroleum and petroleum products - Determination of density - Oscillating U-tube method (ISO 12185)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
dynamic viscosity
η
ratio of the applied shear stress to the resulting shear rate of a liquid

3.2
kinematic viscosity
ν
ratio of the dynamic viscosity to the density of a liquid at the same temperature and pressure

Note 1 to entry: The kinematic viscosity is a measure of a liquid's resistance to flow under gravity.

3.3
density
ρ
mass of a substance divided by its volume at a given temperature

3.4
determinability
d
quantitative measure of the variability associated with the same operator in a given laboratory, obtaining successive determined values using the same apparatus for a series of operations leading to a single result, defined as the difference between two such single determined values

4 Principle

A test portion of a sample is introduced into the measuring cells, which are at closely controlled and known temperature. The measuring cells consist of a pair of rotating concentric cylinders and an oscillating U-tube. The dynamic viscosity is determined from the equilibrium rotational speed of the inner cylinder under the influence of the shear stress of the test specimen and an eddy current brake in conjunction with adjustment data. The density is determined by the oscillation frequency of the U-tube in conjunction with adjustment data. The kinematic viscosity is calculated by dividing the dynamic viscosity by the density.

5 Reagents and materials

5.1 Cleaning solvent, able to remove the sample from the measuring cell after the measurement and be completely miscible with all constituents of the sample.

5.2 Drying solvent, highly volatile and miscible with the cleaning solvent, shall be filtered before use and of an appropriate purity so that it does not leave any residues in the instrument.

NOTE 1 A separate drying solvent is not needed if the cleaning solvent also meets the requirements of the drying solvent.

NOTE 2 Commercially available volatile petroleum spirit or cleaner's naphtha of technical grade or better has proven suitable.

5.3 Compressed air, oil-free and filtered with a dew point considerably lower than the lowest measuring cell temperature at which the instrument should be dried.

The pressure should be limited to 100 kPa.

Instead of compressed air it is also possible to use inert gases, e.g. technical nitrogen. The requirements given for compressed air are also valid here.

5.4 Certified reference liquids for viscosity and density, identical to the reference standards referenced in EN ISO 3104 and EN ISO 12185 respectively.

5.5 Reference thermometer and probe, for verification of the temperature calibration.

The measuring uncertainty of the reference thermometer including the probe shall not exceed 0,01 °C. The resolution shall be at least 0,001 °C.

The probe used for the calibration (with an adapter if necessary) shall have a shape which fits the geometry of the viscosity cell. The probe replaces the measuring system (tube and measuring rotor).

6 Apparatus

Usual laboratory apparatus and glassware, together with the following:

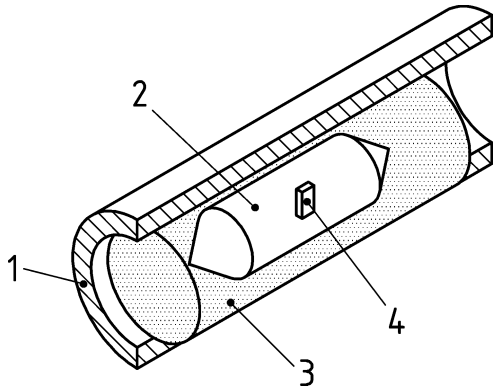
6.1 Stabinger type viscosimeter

6.1.1 Viscosity measurement

The Stabinger type viscosimeter, a concentric rotating viscosimeter contains an outer rotor with an inner rotor (see Figure 1). The small concentric gap between these rotors is filled with sample. The outer rotor is driven at constant speed which makes the inner rotor rotating due to the sample's viscosity. The lightweight inner rotor is centred in the heavier sample due to the centrifugal forces. The equilibrated speed ratio is depending on the driving viscous shear force and the opposing magnetic induction force (eddy current). The dynamic viscosity is a function of the equilibrated speed ratio and adjustment constants. The kinematic viscosity is obtained by dividing the measured dynamic viscosity by the measured density.

6.1.2 Density measurement

The Stabinger type viscosimeter has an integrated density measurement based on the oscillating U-tube principle. The sample filled U-tube is oscillated and the instrument calculates the density from the measured natural frequency of the filled tube using adjustment factors. The viscosity-dependent error of this procedure is corrected using the measured viscosity value.



Key

1	outer rotor (constant speed)	3	sample fluid
2	inner rotor (measured speed)	4	magnet

Figure 1 — Viscosity cell

6.1.3 Temperature control

The Stabinger type viscosimeter has an integrated temperature control which keeps the viscosity and density measurement at the same temperature.

Using Peltier elements, a highly conductive measuring cell block which surrounds the measuring cells is set to the target temperature with a stability of ± 0,005 °C.

The measurement uncertainty of the temperature sensor shall be within ± 0,03°C at 40 °C.

6.1.4 Stability

The instrument automatically ensures the temperature equilibration of the sample by checking the stability of the continuously recorded viscosity and density values by limiting the maximum fluctuation range to ± 0,07 % for the dynamic viscosity and of ± 0,03 kg/m^3 for the density within 60 s.

7 Sampling and sample handling

7.1 Sampling

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 petroleum products.

7.2 Sample handling

For waxy or other samples with high pour point, before drawing the test specimen, heat the sample to the desired temperature, which shall be high enough to dissolve the wax crystals.

8 Calibration and verification

8.1 General

The calibration shall be verified periodically using certified reference standards as described in 5.4.

Due to the measuring range of the viscosity and temperature, more than one calibration fluid can be required. If a reference liquid gives no reference value or if the given reference value is not sufficiently precise for one of the two parameters (viscosity or density) – e.g. a density standard without viscosity values, the affected parameter shall be verified with another suitable reference liquid.

Verify the calibration of the temperature measurement periodically by using a reference thermometer as described in 5.5.

The recommended interval to verify viscosity and density calibration is once a month, for temperature control once a year.

8.2 Verification of calibration

Ensure that the instrument is leak tight and the measuring cells have been cleaned and dried before verification of the calibration is undertaken.

The verification of the calibration should be carried out according to the instrument manufacturer's instructions.

If, despite the correct condition of the instrument, the measured viscosity does not correspond to the certified value with a deviation of less than 0,35 % then the viscosity measurement shall be adjusted according to the instrument manufacturer's instructions.

If, despite the correct condition of the instrument, the measured density does not correspond to the certified value with a deviation of less than 0,001 g/cm^3 then the density measurement shall be adjusted according to the instrument manufacturer's instructions.

If, despite the correct condition of the instrument, the measured temperature does not correspond to the certified value with a deviation of less than 0,03 °C then the temperature measurement shall be adjusted according to the instrument manufacturer's instructions.

9 Apparatus preparation

Ensure that the measuring cells are clean and dry before filling with the sample. The displayed density value of air can be used as an indicator.

Set the test temperature to 40,00 °C. The Stabinger type viscosimeter automatically ensures temperature control and temperature equilibration of the sample.

If the test temperature is below the dew point of the ambient air, a suitable air drying apparatus shall be connected to the air pump inlet of the instrument. When using external compressed air or inert gases ensure that the dew point is lower than the lowest test temperature which can be expected.

Set the determinability limits for viscosity and density to ± 0,1 % and ± 0,2 kg/m^3 respectively.

10 Procedure

10.1 Filling and cleaning

There are three different filling and cleaning procedures which may be used, details are described in 10.2, 10.3 and 10.4.

10.2 Manual filling and cleaning using syringes

10.2.1 Load a sufficient amount of sample to a syringe and remove any air bubbles. Ensure that for the first filling enough sample is used to fill all measuring cells. If sufficient sample is available, it is recommended to fill sample until it is visible in the drain hose. Leave the syringe connected to the instrument during the whole measurement procedure. Typically, a 5 ml syringe entirely filled with sample is enough for a measurement with the viscosimeter.

10.2.2 Switch on the motor for a short time (5 s to 10 s) to ensure that the measuring cells are pre-wetted. In this way any residues are also absorbed into the sample. By subsequently filling at least a further 0,25 ml of sample, the sample in the measuring cell is replaced by fresh sample. If there is enough amount of sample available it is recommended to refill 1 ml.

10.2.3 Start the motor again for the first determination of viscosity and density. The instrument automatically and continuously checks the stability of the measured values until the set criteria are met (6.1.4). Then the instrument requests to refill sample for the next determination. The procedure is the same as for pre-wetting the cells (10.2.2).

10.2.4 If the difference between the determinations is within the set determinability limits (Clause 9), then the values of the last determination shall be reported as valid measured results.

10.2.5 If the difference does not meet the above condition, the instrument requests another filling until the determinability limits or the maximum of 3 repeat measurements has been reached. In the latter case repeat procedure 10.2.1 until the requirement is met.

10.2.6 Remove the sample from the measuring cells and clean the cells with a suitable cleaning solvent. Repeat this procedure until the sample is completely removed. Starting the motor for a short period of time and reading the viscosity of the solvent gives an indication of the quality of the cleaning. If the cleaning solvent is not volatile enough, rinse the cells with a suitable drying solvent and dry the cells with compressed air or by suction. The displayed density value can be used as an indication of the drying.

10.3 Manual filling using sample displacement

10.3.1 The sample volume shall be sufficient to ensure complete displacement of the previous sample. Load the sample to the syringe (typically 25 ml) and remove any air bubbles.

10.3.2 The first filling should be approx. 15 ml. Leave the syringe connected to the instrument during the whole measurement procedure.

10.3.3 Switching on the motor for a short time (5 s to 10 s) ensures that the measuring cells are pre-wetted. In this way any residues are also absorbed into the sample. By subsequently filling a further 2 ml of sample, the sample in the measuring cell is replaced by fresh sample.

10.3.4 After a new start of the motor the first determination of viscosity and density is carried out. The instrument automatically and continuously checks the stability of the measured values until the set criteria are met (6.1.4). Then the instrument requests to refill sample for the next determination.

10.3.5 If the difference between the determinations corresponds with the set determinability limits (Clause 9), then the values of the last determination are given as valid measured result.

10.3.6 If the difference does not meet the above condition, the instrument requests another filling until the determinability limits or the maximum of 3 repeat measurements has been reached. In the latter case repeat procedure 10.3.1 until the requirement is met.

10.3.7 Clean the viscosity measuring cell if necessary (e.g. significant product change, instrument not in use, problems with the determinability limits etc.) according to 10.2.6.

10.4 Automatic filling and cleaning by a sample changer

10.4.1 Before starting the measuring procedure, set appropriate filling, cleaning and drying parameters unless the instrument can adapt these automatically to the sample.

10.4.2 Fill sufficient sample volume into a suitable container for the sample changer and place the container into the magazine. If the sample changer or the sample requires it, close the containers with appropriate covers.

10.4.3 Before starting the measuring cycle, make sure there is sufficient cleaning solvent and drying solvent and enough space in the waste vessel for gathering the sample and solvent.

10.4.4 After starting the measuring cycle, the measuring cells are automatically filled, pre-wetted and the first determination of viscosity and density is carried out. The instrument automatically and continuously checks the stability of the measured values until the set criteria are met (6.1.4). Then the instrument refills sample for the next determination.

10.4.5 If the difference between the determinations is within the set determinability limits (Clause 9), then the values of the last determination are given as valid measured result.

10.4.6 If the difference does not meet the above condition, the instrument fills until the determinability limits or the applicable maximum number of repeat measurements (minimum 3, maximum depending on sample changer) has been reached. In the latter case repeat procedure 10.4.1 until the requirement is met.

10.4.7 After the measurement cycle the cells are cleaned and dried fully automatically and the cycle is repeated for all vessels in the magazines.

11 Calculation

The recorded values are the final results, expressed either as dynamic viscosity in millipascal-seconds or as kinematic viscosity in square millimetre per second and as density in grams per cubic centimetre or kilograms per cubic metre. The calculation of kinematic viscosity is usually performed automatically by the instrument.

12 Expression of results

Express the dynamic (η) and kinematic (ν) viscosity to 4 significant digits, the density (ρ) to the nearest 0,1 kg/m^3, and the test temperature to the nearest 0,01 °C.

13 Precision

13.1 General

This Standard has been developed via a CEN interlaboratory study with 21 laboratories using 12 different samples [1]. The set of samples comprised single FAME types (e.g. palm-kernel and rape seed based), blends of methyl esters, different middle distillates and a mix (50/50) of paraffinic diesel fuel (GTL) and arctic diesel fuel MK1 covering a range of viscosity at 40 °C from 2,61 mm^2/s to 5,50 mm^2/s.

The precision was determined by statistical examination of inter-laboratory test results using EN ISO 4259 [2]. Only the equations for kinematic viscosity are given; for other precision data see [1].

During the ILS all samples were tested according to EN ISO 3104 and to this document. Based on statistical analysis according to EN ISO 4259, no significant bias between the two test methods was found.

13.2 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 value indicated below in only one case in 20.

$$r = 0{,}01\,05 - 0{,}000\,3X \quad (1)$$

where

X is the average of the two results being compared, in mm^2/s.

13.3 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 value indicated below in only one case in 20.

$$R = 0{,}034\,6 + 0{,}005X \quad (2)$$

where

X is the average of the two results being compared, in mm^2/s.

14 Test report

The test report shall contain at least the following information:

a) reference to this European Standard;

b) type and complete identification of the product tested;

c) used method of sampling (see Clause 7);

d) result of the test (see Clause 12);

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

f) date of the test.

Bibliography

[1] RRT 2013-445, Establish Precision Statements for dynamic viscosity and density and the calculation of kinematic viscosity using the Stabinger Viscometer, RRT report, October 2014, to be obtained from the CEN/TC 19 Secretariat, Delft, the Netherland, energy@nen.nl

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

This page deliberately left blank

This page deliberately left blank

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards -based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Copyright in BSI publications

All the content in BSI publications, including British Standards, is the property of and copyrighted by BSI or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use.

Save for the provisions below, you may not transfer, share or disseminate any portion of the standard to any other person. You may not adapt, distribute, commercially exploit, or publicly display the standard or any portion thereof in any manner whatsoever without BSI's prior written consent.

Storing and using standards

Standards purchased in soft copy format:

- A British Standard purchased in soft copy format is licensed to a sole named user for personal or internal company use only.
- The standard may be stored on more than 1 device provided that it is accessible by the sole named user only and that only 1 copy is accessed at any one time.
- A single paper copy may be printed for personal or internal company use only.

Standards purchased in hard copy format:

- A British Standard purchased in hard copy format is for personal or internal company use only.
- It may not be further reproduced – in any format – to create an additional copy. This includes scanning of the document.

If you need more than 1 copy of the document, or if you wish to share the document on an internal network, you can save money by choosing a subscription product (see 'Subscriptions').

Reproducing extracts

For permission to reproduce content from BSI publications contact the BSI Copyright & Licensing team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email subscriptions@bsigroup.com.

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Useful Contacts

Customer Services
Tel: +44 345 086 9001
Email (orders): orders@bsigroup.com
Email (enquiries): cservices@bsigroup.com

Subscriptions
Tel: +44 345 086 9001
Email: subscriptions@bsigroup.com

Knowledge Centre
Tel: +44 20 8996 7004
Email: knowledgecentre@bsigroup.com

Copyright & Licensing
Tel: +44 20 8996 7070
Email: copyright@bsigroup.com

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

bsi.