INTERNATIONAL STANDARD

ISO 3679

Fourth edition
2015-03-01

Determination of flash no-flash and flash point — Rapid equilibrium closed cup method

Détermination de l'éclair de type passe/ne passe pas et du point d'éclair — Méthode rapide à l'équilibre en vase clos

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 3679:2015
https://standards.iteh.ai/catalog/standards/sist/d6772554-1050-419b-87ee-08cb221e390e/iso-3679-2015

Reference number
ISO 3679:2015(E)

© ISO 2015

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 3679:2015
https://standards.iteh.ai/catalog/standards/sist/d6772554-1050-419b-87ee-08cb221e390e/iso-3679-2015

COPYRIGHT PROTECTED DOCUMENT

© ISO 2015

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

 © ISO 2015 – All rights reserved

Contents

Page

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 3679:2015
https://standards.iteh.ai/catalog/standards/sist/d6772554-1050-419b-87ee-08cb221e390e/iso-3679-2015

© ISO 2015 – All rights reserved

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

iTeh STANDARD PREVIEW
(standards.iteh.ai)

The committee responsible for this document is ISO/TC 28, *Petroleum products and lubricants*, working Group 9, in conjunction with Technical Committee ISO/TC 35, *Paints and varnishes*, Technical Committee CEN/TC 19, *Gaseous and liquid fuels, lubricants and related products of petroleum, synthetic and biological origin*, and Technical Committee CEN/TC 139, *Paints and varnishes*.

ISO 3679:2015
https://standards.iteh.ai/catalog/standards/sist/d67772554-1050-419b-87ee-08cb221e390e/iso-3679-2015

This fourth edition cancels and replaces ISO 3679:2004 and ISO 3680:2004, which have been technically revised. The main technical changes are:

— incorporation of ISO 3680 flash point technique into the flash/no flash technique as a separate procedure due to the fact that many apparatus on the market combine both tests;

— title change;

— revision of temperature measuring device requirements;

— new precision covering both gas and electric ignition.

 © ISO 2015 – All rights reserved

Introduction

This International Standard is a closed cup equilibrium test method for the determination of the flash/no-flash and flash point of paints, varnishes, binders for paints and varnishes, solvents, adhesives, petroleum, and related products. ISO 1516[1] and ISO 1523[2] are also closed cup equilibrium test methods that are to be considered when selecting a method.

The apparatus specified in this International Standard enables a similar test result to be determined using a more rapid procedure and a smaller test portion (2 ml or 4 ml) than that required in ISO 1516 and ISO 1523. In addition, the apparatus can be made portable to the extent of being suitable for on-site testing in addition to its more normal use in laboratories.

Collaborative work[3] has shown that results obtained by these methods are comparable. The interpretation of flash point results obtained on solvent mixtures containing halogenated hydrocarbons should be considered with caution, as these mixtures can give anomalous results.[4]

A limited study has indicated that some water borne paints can give an elevated flash point when an electric ignitor is used with ISO 3679.

Flash point can be used in shipping, storage, handling, and safety regulations, as a classification property to define "flammable" and "combustible" materials. Precise definition of the classes is given in each particular regulation.

The flash point can indicate the presence of highly volatile material(s) in a relatively non-volatile or non-flammable material and flash point testing can be a preliminary step to other investigations into the composition of unknown materials.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

It is not appropriate for flash point determinations to be carried out on potentially unstable, decomposable, or explosive materials, unless it has been previously established that heating the specified quantity of such materials in contact with the metallic components of the flash point apparatus, within the temperature range required for the method, does not induce decomposition, explosion or other adverse effects.

ISO 3679:2015
https://standards.iteh.ai/catalog/standards/sist/i6772554-1050-410b-87ee-08cb221e390e/iso-3679-2015

The flash point is not a constant physical-chemical property of a material tested. It is a function of the apparatus design, the condition of the apparatus used, and the operational procedure carried out. Flash point can therefore only be defined in terms of a standard test method, and no general valid correlation can be guaranteed between results obtained by different test methods or with test apparatus different from that specified.

ISO/TR 29662[5] (an adoption of CEN/TR 15138[6]) gives useful advice in carrying out flash point tests and interpreting results.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 3679:2015
https://standards.iteh.ai/catalog/standards/sist/d6772554-1050-419b-87ee-08cb221e390e/iso-3679-2015

Determination of flash no-flash and flash point — Rapid equilibrium closed cup method

WARNING — The use of this International Standard can involve hazardous materials, operations, and equipment. This International Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies procedures for flash point tests, within the temperature range of –30 °C to 300 °C, for paints, including water-borne paints, varnishes, binders for paints and varnishes, adhesives, solvents, petroleum, and related products. The procedures are used to determine whether a product will or will not flash at a specified temperature (flash no-flash Procedure A) or the flash point of a sample (Procedure B). When used in conjunction with the flash detector (A.1.6), this International Standard is also suitable to determine the flash point of fatty acid methyl esters (FAME).

2 Normative references

iTeh STANDARD PREVIEW
(standards.iteh.ai)

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.

ISO 3679:2015
https://standards.iteh.ai/catalog/standards/sist/d6772554-1050-419b-87ee-08eb221e390e/iso-3679-2015

ISO 1513, *Paints and varnishes — Examination and preparation of test samples*

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 3171, *Petroleum liquids — Automatic pipeline sampling*

ISO 4259, *Petroleum products — Determination and application of precision data in relation to methods of test*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

3 Terms and definitions

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

3.1
flash point
lowest temperature, as measured in the prescribed manner, of the test portion corrected to a barometric pressure of 101,3 kPa, at which application of an ignition source causes the vapour of the test portion to ignite momentarily and the flame to propagate across the surface of the liquid under the specified conditions of test

3.2
flash no-flash
application of an ignition source at the specified temperature of the test portion, as measured in the prescribed manner, adjusted to a barometric pressure of 101,3 kPa, to determine whether the vapours of the test portion ignite momentarily and a flame propagate across the surface of the liquid under the specified conditions of test

© ISO 2015 – All rights reserved

3.3
fatty acid methyl ester
FAME
fuel comprising mono-alkyl esters of long chain fatty acids derived from vegetable oil or animal fats, designated B100 or biodiesel (100 %)

Note 1 to entry: FAME is specified in specifications such as EN 14214[7] and ASTM D6751.[8]

4 Principle

A test portion of specified volume is introduced into the test cup that is set and maintained at the required test temperature. After a specific time, an ignition source is applied and a determination is made (Procedure A) as to whether or not a flash occurred. In order to determine the actual flash point of the sample, further tests, with fresh test portions at different test cup temperatures, are carried out (Procedure B) until the flash point is determined.

5 Reagents and materials

5.1 Cleaning solvent, for the removal of traces of sample from the test cup and cover.

The choice of solvent will depend upon the previous material tested and the tenacity of the residue. Low volatility aromatic (benzene-free) solvents can be used to remove traces of oil, and mixed solvents can be effective for the removal of gum-type deposits.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

5.2 Reference materials, series of certified reference materials (CRM) and/or secondary working standards (SWS) as described in Annex B.

ISO 3679:2015
https://standards.iteh.ai/catalog/standards/sist/d6772554-1050-419b-87ee-08cb221e390e/iso-3679-2015

5.3 Gas for ignitor and pilot flame, not required if an electric ignitor is used.

Butane, propane, coal gas, or natural gas can be used.

6 Apparatus

6.1 Flash point apparatus, as specified in Annex A.

6.2 Barometer, reading absolute pressure, accurate to ±0,5 kPa and with a resolution of 0,1 kPa.

Barometers pre-corrected to give sea level readings, such as those used at weather stations and airports, shall not be used.

6.3 Heating bath or oven, for warming the sample, if required, and capable of controlling the temperature to an accuracy of ±5 °C.

The oven shall be ventilated and constructed in such a way that it will not cause ignition of any flammable vapours that can be produced when the sample is heated. It is recommended that the oven is of explosion-protected design.

6.4 Cooling bath or freezer (optional), for cooling the samples, if required, and capable of cooling the sample to 10 °C below the expected flash point, and controlling the temperature to an accuracy of ±5 °C.

The bath and oven shall be suitable for use with volatile and flammable materials.

6.5 Draught shield (optional), if required to minimize draughts, a shield fitted at the back and on two sides of the instrument.

© ISO 2015 – All rights reserved

6.6 Cup insert (optional), see Annex C.

For samples that are difficult to remove, a thin metal cup insert can be used but the precision has not been determined.

6.7 Syringes

6.7.1 Syringe, capable of delivering 2,00 ml ± 0,05 ml and equipped with a nozzle suitable for the required test temperature and apparatus.

To enable a 4 ml test portion to be used, this syringe may be used twice.

6.7.2 Syringe, capable of delivering 4,00 ml ± 0,10 ml and equipped with a nozzle suitable for the required test temperature and apparatus.

7 Preparation of apparatus

7.1 General

7.1.1 Select the appropriate instrument for the expected flash point temperature. Follow the manufacturer's instructions for the correct set-up, verification (7.4) and operation of the apparatus, especially the operation and setting of the ignition source.

iTeh STANDARD PREVIEW

7.1.2 The use of a cup insert (6.6) for potentially adherent materials is described in Annex C.

(standards.iteh.ai)

7.1.3 When testing FAME use a 2 ml ± 0,05 ml test portion and a 60 s ± 2 s test time, combined with an electronic thermal flash detector (see A.1.6).

ISO 3679:2015
https://standards.iteh.ai/catalog/standards/sist/d6772554-1050-419b-87ee-08cb221e390e/iso-3679-2015

7.1.4 For sub-ambient test temperatures use Annex D, unless the apparatus has integral test cup cooling facilities.

7.2 Location of apparatus

Support the apparatus (Annex A) on a level and steady surface in a draught-free position.

A draught shield (6.5) is recommended to be used when protection from draughts is not available.

When testing materials which can produce toxic vapours, the apparatus should be located in a fume hood with an individual control of air flow, adjusted such that the vapours are withdrawn without causing air currents around the test cup during the test.

7.3 Cleaning of the test cup assembly and accessories

Clean the test cup cover and its accessories with an appropriate solvent (5.1) to remove traces of gum or residue from the previous test. Wipe dry to remove all traces of solvent.

Follow the manufacturer's instructions for the care and servicing of the instrument, especially regarding electronic ignitors and flash detectors which can be fragile.

A stream of clean dry air, such as compressed air, may be used to remove the last traces of solvent used.

The filler orifice can be cleaned using a suitable cleaning device such as a small brush.

© ISO 2015 – All rights reserved

7.4 Apparatus verification

7.4.1 Verify and correct, if necessary, the reading of the temperature measuring device (see Annex E) at least every 12 months, according to the manufacturer's instructions.

7.4.2 Verify the correct functioning of the apparatus at least once a year, by using a certified reference material (CRM) (5.2) according to Procedure B. The result obtained, after barometric pressure correction (11.2) shall be equal to or less than $R/\sqrt{2}$ from the certified value of the CRM, where R is the reproducibility of the method for petroleum and related products (see 13.3).

It is recommended that more frequent verification checks are made using secondary working standards (SWSs) (5.2).

A recommended procedure for apparatus verification using CRMs and SWSs, and the production of SWSs, is described in Annex B.

7.4.3 The numerical values obtained during the verification check shall not be used to provide a bias statement, nor shall they be used to make any correction to the flash points subsequently determined using the apparatus.

7.4.4 If the instrument fails the verification test, it is recommended that the operator should follow the manufacturers' instructions and check the following, and then repeat the verification check:

a) the cover makes a vapour tight seal with the test cup;

b) the shutter provides a light tight seal;

c) adequate heat transfer paste surrounds the temperature measuring device inserted in the test cup block;

d) the correct operation of the ignition source;

e) the operation of the flash detector (A.1.6) (if fitted);

f) the correct reading of the temperature measuring device.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 3679:2015
https://standards.iteh.ai/catalog/standards/sist/d6772554-1050-419b-87ee-a8c21e390e/iso-3679-2015

8 Sampling

8.1 Unless otherwise specified, obtain samples in accordance with the procedures given in ISO 1513, ISO 15528, ISO 3170, or ISO 3171 or an equivalent National Standard.

8.2 Place sufficient sample volume for testing in a tightly-sealed container made of material appropriate to the liquid being sampled, and for safety purposes, ensure that the sample container is only filled to between 85 % and 95 % of its capacity.

8.3 Store the samples in conditions to minimize vapour loss and pressure build up. Avoid storage of samples at temperatures in excess of 30 °C.

© ISO 2015 – All rights reserved

9 Sample handling

9.1 Petroleum products and fatty acid methyl esters

9.1.1 Subsampling

Cool or adjust the temperature of the sample and its container to at least 10 °C below the first selected test temperature before opening to remove the test portion. If an aliquot of the original sample is to be stored prior to testing fill the container to between 85 % and 95 % of its capacity. Gently mix the subsample to ensure uniformity, so that the loss of volatile components and light ends is minimized.

NOTE Results of flash point tests can be affected if the sample volume falls below 50 % of the container's capacity.

9.1.2 Samples liquid at ambient temperature

If sufficiently fluid, mix samples by gentle hand shaking prior to the removal of the test portion, taking care to minimize the loss of volatile components. Ensure that the sample is at least 10 °C below the selected test temperature before opening to remove the test portion. For mobile materials, mix the sample by gentle shaking. If necessary, heat the sample in its container to a temperature such that the sample can be mixed by gentle shaking or to at least 10 °C below the selected test temperature, whichever is lower. Ensure that high pressures do not develop in the container.

9.1.3 Samples solid or semi-solid at ambient temperature

iTeh STANDARD PREVIEW
(standards.iteh.ai)

If the material under test cannot be made sufficiently fluid to be introduced into the test cup through the orifice by heating in accordance with 9.1.2, transfer the test portion with a solids dispenser or spatula into the test cup while the cover is open. The test portion size can be the mass equivalent of the required volume and the test portion should be spread over the bottom of the test cup as evenly as possible.

ISO 3679:2015
https://standards.iteh.ai/catalog/standards/sist/d6772554-1050-419b-87ee-08cb221a390e/iso-3679-2015

9.1.4 Samples containing dissolved or free water that is not part of the product

If the sample does not contain volatile, low flash point components, the water may be decanted or the sample dehydrated with calcium chloride.

9.2 Paints, varnishes, and related materials

Prepare the samples in accordance with the procedures described in ISO 1513.

10 Procedure

10.1 General

10.1.1 Follow the manufacturer's instructions for setting the test temperature.

10.1.2 When testing fatty acid methyl esters (FAME), a flash detector (A.1.6) shall be used.

10.1.3 Use a new test portion of the sample for each test. Do not apply the ignition source to the test portion more than once. After each test, turn off the pilot and test flames (if used) using the gas control valves, and when the test cup temperature falls to a safe level, remove the test portion and clean the instrument.

10.1.4 Do not confuse the true flash point with the bluish halo that sometimes surrounds the test flame at applications preceding that which causes the actual flash.

© ISO 2015 – All rights reserved

NOTE The optional flash detector (A.1.6) is not affected by the halo, and does not require the operator to closely observe the flash point test.

10.1.5 Record the absolute barometric pressure using a barometer (6.2) in the vicinity of the apparatus at the time of the test.

NOTE It is not considered necessary to correct the barometric pressure reading to 0 °C, although some barometers are designed to make this correction automatically.

10.1.6 Ensure that the test flame size or setting of an electric ignitor is set correctly, as an incorrect setting can significantly affect the test result.

10.2 Procedure A — Flash no-flash test

10.2.1 Inspect the test cup and cover for cleanliness and correct operation, especially with regard to tightness of the cover "O" ring (A.1.1.3), the action of the shutter, the size or intensity of the ignition source, and the position of the ignition source (A.1.2). Clean if necessary (7.3). Put the cover in place and close securely.

10.2.2 Correct the required test temperature for the flash no-flash test according to the absolute barometric pressure as shown in Formula (1). Allow for any known thermometer correction, and then round to the nearest 0,5 °C.

$$t_t = t_s - 0{,}25(101{,}3 - p) \quad (1)$$

iTeh STANDARD PREVIEW
(standards.iteh.ai)

where

t_t is the actual test temperature, in degrees Celsius;

ISO 3679:2015
https://standards.iteh.ai/catalog/standards/sist/d6772554-1050-419b-87ee-0dc022e85906/iso-3679-2015

t_s is the specification or uncorrected target test temperature, in degrees Celsius;

p is the absolute barometric pressure, in kilopascals;

0,25 is a constant with dimensions degrees Celsius per kilopascal;

101,3 is the standard pressure, in kilopascals.

10.2.3 Follow the manufacturer's instructions to set the test temperature and the test time, and select the test portion volume and test time in accordance with Table 1.

Table 1 — Test Conditions

Sample	Test temperatures	Test portion volume	Test time
All except FAME	≤100 °C	2 ml	60 s
All except FAME	>100 °C	4 ml	120 s
FAME	≤300 °C	2 ml	60 s

10.2.4 When the test cup is at the test temperature, fill the appropriate syringe (6.7) with the sample to be tested. Transfer the syringe to the filling orifice, taking care not to lose any sample. Discharge the test portion into the test cup by fully depressing the syringe plunger. Remove the syringe.

10.2.5 Start the test timer. Light the pilot light and adjust the test flame (if used) to conform to the 4 mm gauge.

© ISO 2015 – All rights reserved