TECHNICAL REPORT

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# Petroleum products and other liquids — Guidance for flash point and combustibility testing

*Produits pétroliers et autres liquides — Lignes directrices pour les essais de combustibilité et de point d'éclair*



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# 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 of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.



This document was prepared by Technical Committee ISO/TC 28, *Petroleum and related products, fuels and lubricants from natural or synthetic sources*.



This second edition cancels and replaces the first edition (ISO 29662:2009). The main technical changes compared to the previous edition are as follows:

— the title has been changed;

— combustibility test details have been further added;

— a list of examples of regulations have been added;

— test samples, to include biodiesel, mixtures and samples that form a skin during testing have been added;

— the use of low hazard glass thermometers has been added;

— further details regarding the requirements for barometric corrections have been added;

— Annex A has been added to include temperature ranges for each test method.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

## Introduction

This document was written to assist laboratory managers and technicians, regulators, specification writers and industry in the use, specification and application of flash point and combustibility tests for liquids and semi-solids.

The flash point test can be summarised as a procedure where a test portion is introduced into a temperature-controlled test cup and an ignition source is applied to the vapours produced by the test portion to determine if the vapour / air mixture is flammable or at what temperature the vapour / air mixture is flammable.

Combustibility tests in this document comprise fire point, sustained combustibility and sustained burning tests. These tests can be summarised as a procedure where a test portion is introduced into a temperature-controlled test cup and an ignition source is applied to the vapours produced by the test portion to determine if the vapour / air mixture catches fire and continues to burn.

This document was developed by the Joint ISO/TC 28 - ISO/TC 35 WG9 on flash point methods.



 

# Petroleum products and other liquids — Guidance for flash point and combustibility testing

## 1 Scope

This document establishes an overview of test methods in the field to determine flash point and combustibility of petroleum and related products. It presents advice on application and specification development. This document is not intended to be a comprehensive manual on flash point and combustibility tests, and the interpretation of test results, however it covers the key aspects on these subjects.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1998-1, *Petroleum industry — Terminology — Part 1: Raw materials and products*

ISO 1998-2, *Petroleum industry — Terminology — Part 2: Properties and tests*



## 3 Terms and definitions

For the purposes of this document, the terms and definitions in ISO 1998-1 and ISO 1998-2 and the following apply.



ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

**3.1**
**repeatability**
*r*
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 and in the normal operation of the test method, exceed the given value in only one case in 20

Note 1 to entry: The general description deviates from ISO 4259-1 used in many of the standards dealt with in this document.

**3.2**
**reproducibility**
*R*
difference between two single and independent test results obtained by different operators in different laboratories on identical test material that would, in the long run and in the normal operation of the test method, exceed the given value in only one case in 20

Note 1 to entry: The general description deviates from ISO 4259-1 used in many of the standards dealt with in this document.

 

## 4 Outline of generic definitions and general statements in test methods

**4.1** There are many, slightly different, definitions of flash point, however the following definition is widely used in standard test methods:

The lowest temperature of the test portion, adjusted to account for variations in atmospheric pressure from 101,3 kPa, at which application of an ignition source causes the vapour of the test portion to ignite and the flame to propagate across the surface of the liquid under the specified conditions of test.

**4.2** It is important to realise that the value of the flash point is not a physical constant but it is the result of a flash point test and is dependent on the apparatus and procedure used. This fact is so important that a general statement similar to the following is incorporated into all the main flash point methods:

Flash point values are not a constant physical-chemical property of materials tested. They are 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.

**4.3** Combustibility tests have their own definitions, the following are examples.

— Sustained combustibility: behaviour of a material, under specified test conditions, whereby its vapour can be ignited by an ignition source and, after ignition, sufficient flammable vapour is produced for burning to continue for at least 15 s after the source of ignition has been removed.



— Fire point: lowest temperature of the test portion, adjusted to account for variations in atmospheric pressure from 101,3 kPa, at which application of a test flame causes the vapour of the test portion to ignite and sustain burning for a minimum of 5 s under the specified conditions of test.



— The sustained burning test does not have a formal definition, however, it may be defined as follows: behaviour of a material, under specified test conditions, whereby its vapour can be ignited by an ignition source and, after ignition, sufficient flammable vapour is produced for burning to continue for at least 15 s after the source of ignition has been removed.

NOTE All flash point and combustibility test temperatures are corrected by a formula that compensates if the barometric pressure is not 101,3 kPa.

**4.4** Due to the importance of flash point and combustibility test results for both safety and regulatory purposes, the test method identification should always be included with the test result.

**4.5** In general specific products specifications indicate which standard test method should be employed.

## 5 Brief history

**5.1** The discovery of petroleum and the increased use of flammable distillates in the 19$^{th}$ century, for lighting and heating in place of animal and vegetable oils, led to a large number of explosions and other fire related accidents.

Legislation, such as the UK Petroleum Act in 1862 and the German Petroleum Regulations in 1882, quickly spread around the world and led to the development of many types of test instruments. The following list shows the dates when the major surviving instruments were in a form probably recognisable today:

— 1870 – 1880: Abel closed cup, Pensky-Martens closed cup;

— 1910 – 1920: Tag closed cup, Cleveland open cup.

 

**5.2** Flash point and combustibility tests are key components of transport, safety and health regulations. The examples of such regulations shown below are used in Europe but have numerous equivalents internationally. These regulations have been used in the past to assist in setting specification levels for flash point requirements.

DSD Dangerous substances directive – 2015 replaced by CLP

DPD Dangerous preparations directive – 2015 replaced by CLP

CLP Classification, labelling and packaging

ADR Carriage of dangerous goods by road

GHS Global harmonized system – classification, labelling and packaging

ADN Carriage of dangerous goods by inland waterways

RID Carriage of dangerous goods by rail

## 6 Flash and fire point, and sustained combustion and burning

**6.1** The flash point is essentially the lowest temperature of the liquid or semi-solid at which vapours from a test portion combine with air to give a flammable mixture and 'flash' when an ignition source is applied. Fire point, combustibility and sustained burning tests all use open cup instruments.



**6.2** Fire point can be considered as the lowest temperature of the test portion at which vapour combustion and burning commences when an ignition source is applied and thereafter is continuous and where the heat produced is self-sustaining and supplies enough vapours to combine with air and burn even after the removal of the ignition source.



**6.3** Sustained combustion and burning tests are usually carried out with the test portion at a fixed temperature and tests whether vapour combustion and burning commences when an ignition source is applied and thereafter is continuous and where the heat produced is self-sustaining and supplies enough vapours to combine with air and burn even after the removal of the ignition source.

## 7 Why are flash point and combustibility tests required

The fundamental reason for the requirement of flash point measurements is to assess the safety hazard of a liquid or semi-solid with regard to its flammability and then classify the liquid into a group. The lower the flash point temperature the greater the risk. This classification is then used to warn of a risk and to enable the correct precautions to be taken when using, storing or transporting the liquid.

Specifications quote flash point values for quality control purposes as well as for controlling the flammability risk.

A change in flash point can indicate the presence of potentially dangerous volatile contaminants or the adulteration of one product by another.

Test methods that enable the ability of a liquid to support a sustained combustion flame to be assessed, offer a means of further identifying the hazard of liquids under possible fire conditions for use in safety and health regulation classifications.

# 8 Which test method should be used

## 8.1 First considerations

Firstly, if a specific test method has been specified in a product specification or regulation, then that method should be the first choice. If a number of alternative methods are specified then the choice is influenced by availability and other factors such as sample size requirements, speed of testing or precision. In certain circumstances the choice of the stated referee method is of special importance. Annex A gives an overview of the most common methods and their use in specifications and regulations.

When testing specifically for contamination or contaminants, certain test methods and procedures are more appropriate than others. In general, an equilibrium test method is recommended for testing samples that can contain traces of volatile contaminants.

When selecting a flash point method for incorporation into a product specification or regulation, it is important that the product type is included in the scope of the test method and that the temperature range of the product is covered by the test method. If the product is not included in the scope then the test can be unsuitable for the product or the quoted precision does not apply. Where the scope of a test method is general or not suitable it is recommended to contact an appropriate standardization body for advice.

When testing chemicals, mineral products or corrosive materials, it is recommended to check that the test cup material is suitable and will not produce flammable gases or be damaged by any possible chemical reaction.

The use of the sustained combustibility test is implemented in some safety and health regulations and can be useful for some products to obtain an alternative hazard classification.



## 8.2 Open or closed cup

There are two general classes of flash point tests: open cup and closed cup.



The open cup was initially developed to assess the potential hazards of liquid spillage. In this test, a test portion of the sample is introduced into a cup that is open at the top. An ignition source is passed horizontally over the surface of the liquid, while the cup and liquid are being heated, to test if the vapours 'flash'. If the test is repeated at increasing test portion temperatures a point can be reached when the test portion continues to burn without further application of the ignition source, this is the fire point. The precision of open cup tests is somewhat poorer than closed cup tests as the vapours produced by heating the test portion are free to escape to the atmosphere and are more affected by local conditions in the laboratory. When open cup tests are made at temperatures above ambient temperature, the result is usually higher than a result from a closed cup test due to the reduced concentration of vapours.

The closed cup test contains any vapours produced and essentially simulates the situation where a potential source of ignition is accidentally introduced into a container. In this test, a test portion is introduced into a cup and a close fitting lid is fitted to the top of the cup. The cup and test portion is heated and apertures are then opened in the lid to allow air into the cup and the ignition source to be dipped into the vapours to test for a flash.

The closed cup test predominates in specifications and regulations due to its better precision and ability to detect contaminants.

Fire point, sustained combustion and sustained burning tests outlined in this technical report are all open cup type tests.

## 8.3 Non-equilibrium, equilibrium and rapid equilibrium tests

### 8.3.1 General

These three types of tests and associated instruments are characterised by the level of temperature stabilisation of the test portion and resultant vapours, and by the test portion size and test time.

 

### 8.3.2 Non-equilibrium tests

Test methods such as Pensky-Martens, Tag, Abel and Cleveland are referred to as non-equilibrium tests as the test temperature of the test portion is increased during the test and the temperature of the vapours is not the same (not in equilibrium) as the test portion temperature when the ignition source is dipped at regular intervals into the cup.

This type of test has the advantage that it produces a definitive flash point result. Under normal circumstances, the increasing temperature is not a problem, but when volatile contaminants or components are present the short time between each dip of the ignition source, combined with the rate of temperature increase, does not allow enough time for flammable vapours to evolve and this can cause unreliable results. For this reason, non-equilibrium tests with lower rates of heating usually perform better than those using higher rates of heating, when volatile contaminants or components are present in the test portion.

### 8.3.3 Equilibrium tests

Equilibrium tests are preferred for liquids and semi-solids containing volatile components or contaminants and for confirmatory purposes in regulations as the sample temperature is constant or is increased at a very slow rate. This allows enough time for vapours to build up and for the vapours to be in equilibrium with the test portion before the ignition source is dipped into the cup. The ignition source is dipped in the cup at different test portion temperatures thus resulting in a measurement of a flash point, or the ignition source is dipped only once to carry out a flash no flash test to check conformity with specifications and flammability criteria.

These equilibrium tests use any type of closed cup in a liquid bath and limits the difference of temperature between the test portion and the liquid bath. The liquid bath is specified because it gives a very even temperature distribution on the outside of the test cup thus ensuring that hot spots are not present on the cup surface that could cause the localised increase of flammable vapours and thus a low flash point. Unfortunately, these procedures take a long time to complete.



### 8.3.4 Rapid equilibrium tests

Rapid equilibrium (small scale) tests are not primarily aimed to give the actual flash point of a test portion. The test is a flash no-flash test to determine if the test portion's vapours flash at the test temperature. This is useful for checking conformance against specifications and flammability criteria. The test cup is heated to the test temperature, a small test portion is introduced into the cup, and when the test portion is deemed to be at the test temperature, the ignition source is used to test for a 'flash'. Actual flash point temperature is determined by repeating the flash no flash test at different temperatures with a new test portion. The constant temperature of the test cup ensures that the test portion cannot be overheated and that there is a reasonable time for vapours to build up before the ignition source is applied.

## 8.4 Flash point automation

### 8.4.1 Manual flash point test

For a manual flash point test, the operator is in control throughout the test and ensures that the temperature, stirring and ignition requirements are met throughout the test and determines when and if a flash has occurred. Some semi-auto instruments can assist the operator in detecting a flash or controlling the temperature, but the operator is in control. This is why manual tests are the referee in cases of dispute.

### 8.4.2 Automated flash point testers

Automated flash point testers conform to all the specified requirements of the manual test method such as dimensions, heating rate and flash detection, however the electronics, software and mechanics mimic the manual operations. This can significantly reduce operator time but this does have the