

Determination of explosion points of flammable liquids

ICS 75.160.20

National foreword

This British Standard is the UK implementation of EN 15794:2009.

The UK participation in its preparation was entrusted to Technical Committee FSH/23, Fire precautions in industrial and chemical plant.

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

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English Version

Determination of explosion points of flammable liquids

Détermination des points d'explosion des liquides
inflammables

Bestimmung von Explosionspunkten brennbarer
Flüssigkeiten

This European Standard was approved by CEN on 22 September 2009.

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Foreword

This document (EN 15794:2009) has been prepared by Technical Committee CEN/TC 305 "Potentially explosive atmospheres - Explosion prevention and protection", the secretariat of which is held by DIN.

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of an identical text or by endorsement, at the latest by April 2010, and conflicting national standards shall be withdrawn at the latest by April 2010.

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For relationship with EU Directive(s), see informative Annex ZA, which is an integral part of this document.

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Introduction

Flammable liquids can give rise to an explosion hazard as a result of evaporation generating an explosive gas and/or vapour mixture with air. One way of eliminating the explosion hazard is to prevent explosive mixtures of gases and/or vapours with air from being formed. In order to assess the likelihood of an explosive mixture being formed the explosion point of the flammable liquid is required. The explosion point depends mainly on:

- the properties (e.g. explosion limits, vapour pressure, chemical composition including impurities of the flammable liquid);
- pressure;
- size, shape, and percentage fill of the test vessel;
- ignition source (type, energy);
- the criterion for self-propagating combustion.

The explosion point of a liquid is normally lower than its flashpoint. For pure substances the difference can be up to 10 K. In the case of mixtures the difference can be up to 25 K. Some liquids which do not exhibit a flash point may have explosion limits and thus have an explosion point.

To obtain reliable and comparable results it is therefore necessary to standardize the conditions (apparatus and procedure) under which the explosion points are to be determined.

1 Scope

This European Standard specifies a test method to determine the explosion points of flammable liquids in air. This European Standard applies to flammable liquids at atmospheric pressure and at temperatures in the range from - 50 °C to 300 °C.

This European standard must not be applied to explosives or materials which, under the test conditions, are thermally unstable liquids (e.g. polymerizing/oxidizing materials).

2 Normative references

The following referenced documents are indispensable for the application 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.

EN 13237:2003, *Potentially explosive atmospheres – Terms and definitions for equipment and protective systems intended for use in potentially explosive atmospheres*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 13237:2003 apply.

4 Principle of the test method

The test sample is placed in a cylindrical vessel and heated up to a specified temperature. After having reached equilibrium conditions between the liquid phase and the gas phase at the set temperature ignition is initiated using a series of induction sparks. It is observed whether a flame detachment or a temperature rise occurs. The temperature of the test apparatus is raised or lowered stepwise, until just no ignition is observed.

5 Test equipment

5.1 Reagents and Materials

5.1.1 Flammable liquid:

The flammable liquid may be:

- a single liquid or a defined mixture of liquids;
- a process sample (of known or unknown composition).

When a pure liquid or a mixture of defined composition is used, the purity of each liquid shall be 99,8 % mol. or better. In the case of a mixture or a process sample of undefined composition the sample should be characterised so that the origin as well as the related process conditions can be identified.

5.1.2 Sampling and Storage

Sampling should be carried out if possible according to the procedures given in EN ISO 3170, EN ISO 3171, EN ISO 15528 or an equivalent National Standard.

Sufficient sample volume for testing shall be placed in a tightly sealed container appropriate to the material being sampled. At the beginning of the tests, the sample container shall be filled to between 85 % and 95 % of its capacity.

The samples shall be stored in conditions that minimize vapour loss and pressure build up to avoid losing volatile components.

If possible, the sample should be stored in its container either at ambient temperature, or about 5 K below the test starting temperature (expected explosion point), whichever is the lower temperature. The sample shall be maintained at this temperature, or lower, until all tests on the sample are completed.

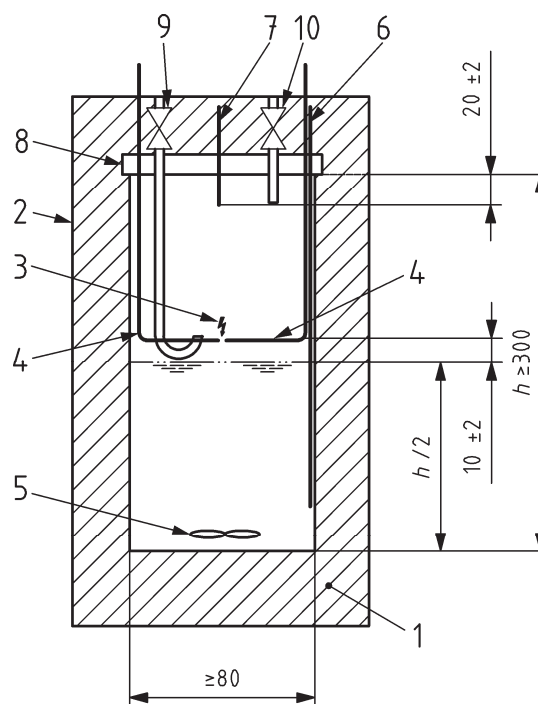
Viscous liquids, liquids which crystallise on cooling, polymerize or separate are to be stored at just above the temperature at which this occurs.

Sample containers are to be kept closed before and after sampling to avoid alterations of the sample (e.g. evaporation of volatile constituents/impurities).

5.2 Apparatus

Apparatus see Figure 1

Dimensions in millimetres



Key

- | | |
|--|---|
| 1 Heating chamber with air circulation and temperature regulator | 6 Thermocouple determining the explosion point and checking the equilibrium |
| 2 Test vessel | 7 Thermocouple for ignition detection and checking equilibrium |
| 3 Ignition source (spark) | 8 Tightly fitting lid allowing for pressure relief |
| 4 Electrodes | 9 Inlet valve |
| 5 Stirrer | 10 Outlet valve |

Figure 1 — Explosion point apparatus; schematic

5.3 Test vessel

The test vessel is an upright cylindrical vessel made of pressure-resistant (10 bar) glass with an inner diameter of between 80 mm to 100 mm and a height of between 300 mm to 500 mm. It is covered with a tightly fitting lid allowing for pressure relief (e.g. smooth metal plate on a ground glass surface).

The electrodes of the ignition source shall be mounted in the lid in a way that the tips are positioned in the centre (10 ± 2) mm above the surface of the liquid, or with a sufficient distance to avoid wetting. The thermocouple for monitoring the gas phase temperature shall be mounted (20 ± 2) mm below the top of the vessel in the vertical axis. If this thermocouple is also used for ignition detection, it has to have a maximum diameter of 0,5 mm and an uncertainty of measurement of 1,0 K. The thermocouple for measuring the temperature of the explosion point should penetrate the liquid phase by at least 20 mm. For purging the vapour phase, an inlet and an outlet valve should be mounted in the lid.

5.4 Heating / cooling chamber

The test vessel is located in a heating/cooling chamber with air circulation, isolated from the ground by a spacer of low thermal conductivity. The chamber should have at least a volume of ten times the test volume and should be greater than 15 l. The chamber should have an air exchange rate of at least ten air changes per h to avoid accumulation of flammable vapours in the chamber. The temperature differences inside the chamber should not exceed 1 K.

The temperature regulating device shall fulfil the following requirements:

- test vessel including the lid has to be heated uniformly so that the temperature distribution inside the empty test vessel does not vary by more than 1,0 K in all directions,
- temperature of the test vessel can be incremented in steps of $1,0 \pm 0,2$ K.

NOTE Fitting the chamber with an explosion proof fan is advisable.

5.5 Ignition source

A series of induction sparks between two electrodes is used as the ignition source.

Stainless steel is a suitable material for the electrodes. The electrodes shall be pointed rods with a maximum diameter of 4 mm. The angle of the tips shall be $(60 \pm 3)^\circ$. The distance between the tips shall be $(5 \text{ mm} \pm 0,1 \text{ mm})$. The electrodes shall be mounted in a gas tight seal, so that it is possible to vary the distance to the liquid. The mounting shall be resistant to heat and the test mixture, and provide adequate electrical resistance to prevent shorting to the test vessel body.

A high voltage transformer, with a root mean square of 13 kV to 16 kV (open circuit voltage) and a short circuit current of 20 mA to 30 mA, shall be used for producing the ignition spark. The primary winding of the high voltage transformer shall be connected to the mains via a timer set to the required discharge time.

The spark discharge time shall be adjusted to 0,2 s. If a spark discharge time of 0,2 s does not result in ignition of the test mixture, the test may be repeated with a spark discharge time of up to 0,5 s.

The power of the induction sparks depends on the gas mixture and its pressure. In air at atmospheric conditions according to calorimetric and electric measurements such a source gives a spark with a power of approximately 10 W.

5.6 Stirrer

The stirrer shall be magnetically driven from the bottom of the vessel, so as not to disturb the flame detachment.

5.7 Barometer

The barometer shall be accurate to 0,1 kPa. Barometers pre-corrected to give sea-level readings shall not be used.

5.8 Safety advice

The safety advice given in Annex A should be followed.

6 Test procedure

6.1 General

When determining the LEP or UEP it is useful to know the closed cup flashpoint of the sample or to estimate the explosion point (Annex B). In the case of an unknown sample composition, it is recommended that the flashpoint is determined first. In the case of samples consisting of or containing compounds having no flashpoint but an explosion range mathematical pre-assessment is recommended if possible. If this is not possible the mixture composition should be analysed to identify the component having the lowest flashpoint. This flashpoint should be used as the starting temperature.

6.2 Details

6.2.1 Step 1

6.2.1.1 Record the ambient pressure in the vicinity of the test equipment.

6.2.1.2 Choose an appropriate starting temperature. When determining the LEP, the first test temperature should be either 5 K (for LEPs below 100 °C) or 10 K (for LEPs above 100 °C) below the flash point or estimated explosion point for pure liquids and at least 15 K below the flash point for mixtures.

When determining the UEP, the first test temperature should be 50 K above the flash point for pure liquids and 75 K above the flash point for mixtures or 10 K above the estimated UEP.

6.2.1.3 If the explosion point is expected to be below ambient temperature cool the sample in its storage container to about 5 K below the expected explosion point and pre-cool the test vessel before adding the sample.

WARNING — Do not heat the sample up to its auto ignition temperature.

6.2.2 Step 2

Fill the test vessel to half of its volume with the test sample. If the explosion point is expected to be above 100 °C the volume expansion of the liquid should be taken into account.

NOTE In particular cases the explosion point may depend on the filling percentage, especially when determining the UEP.

6.2.3 Step 3

The test vessel shall be kept at the test temperature until equilibrium between the gas and liquid phases is achieved. The temperature equilibrium between liquid and gas phase is reached, when the temperatures given by the thermocouple in the gas phase and the liquid phase differ by less than 0,5 K. It may take 30 min or more to get to this temperature equilibrium. After temperature equilibrium is achieved, stir for a further

30 min to ensure concentration equilibrium is attained. When stirring wetting of the electrodes has to be avoided.

When equilibrium conditions are attained an ignition shall be attempted and observed as to whether the criteria for ignition are met or not. The criterion for ignition (self-propagating combustion) is a visually observed upward movement of a flame from the spark gap for at least 100 mm ("flame detachment" examples see Annex C). If a halo reaches the top of the vessel this shall be also counted as an ignition.

Alternatively, a temperature rise of 1 K greater than a reference measurement may be used as the ignition criterion. The temperature rise is measured by the thermocouple in the gas phase ("7" in Figure 1). The reference measurement is conducted under test conditions, in the same experimental setup, but without any liquid in the test vessel (i.e. the entire test contains only air and no flammable vapour or liquid).

NOTE Vapour/air mixtures with a vapour content that lies slightly outside the explosion range may exhibit a luminous phenomenon, which is referred to as a "halo", above but not detached from the spark gap. (see Annex C). The formation of a halo exclusively is not yet considered to be an ignition unless it reaches the top of the vessel.

6.2.4 Step 4

If an ignition is observed when determining the LEP, repeat step 2, decreasing the temperature in 5 K increments until a negative result is obtained. If an ignition is observed when determining the UEP, repeat step 2, increasing the temperature in 5 K increments until a negative result is obtained. The test sample should be replaced with a fresh sample each time an ignition is detected unless the material under test is known to be a single component, pure liquid. If this is the case, the vapour phase requires purging with air after each ignition to ensure sufficient oxygen content for the next ignition trial.

If no ignition is observed, repeat step 3, keeping the same sample in the test vessel, but altering the temperature in 1 K or 2 K increments, depending on the required accuracy, until a positive result is obtained

6.2.5 Step 5

To confirm the result repeat the test with a fresh sample at the nearest no go temperature to where ignition occurred for at least one test. If an ignition occurs step 4 should be repeated in increments of the required accuracy.

Otherwise, the temperature of no ignition in step 5 is recorded as the LEP_o or UEP_o respectively.

7 Expression of results

According to this European standard, all details specified in Clause 9 shall be provided.

The observed results (or mean of the observed results) are corrected to the reference pressure of 101,3 kPa using the following equations.

$$\text{LEP} = \text{LEP}_o + a (p_{\text{ref}} - p) \quad (1)$$

$$\text{UEP} = \text{UEP}_o + a (p_{\text{ref}} - p) \quad (2)$$

where

LEP_o is the observed lower explosion point in °C;

UEP_o is the observed upper explosion point in °C;

LEP is the corrected lower explosion point in °C;

UEP is the corrected upper explosion point in °C;

p is the barometric pressure in kPa;

p_{ref} is 101,3 kPa;

a is 0,25 °C/kPa.

NOTE This equation is strictly correct only within the barometric pressure range 98,0 kPa to 104,7 kPa.

8 Verification of the apparatus

Verification of the used apparatus and test procedure shall be carried out according to Annex D.

9 Test report

The test report shall give the following information:

- a) reference to this European standard;
- b) any deviation to this European standard;
- c) laboratory name, operator and date;
- d) test conditions:
 - barometric pressure;
- e) sample identification:
 - composition, purity and source;
- f) test apparatus, if different from the standard:
 - vessel shape and volume, percentage fill;
- g) LEPo;
- h) LEP;
- i) UEPo;
- j) UEP.

The expanded uncertainty in measurement U of the test procedure is estimated to be 4 K, i.e. twice the uncertainty in the temperature control and temperature gradient in the heating chamber.

An example of an appropriate form that may be used for a test report is given in Annex E.

Annex A (informative)

Safety advice

The following advice should be followed if applicable to the method used:

- a) Ensure the exhaust gases/vapours from the test apparatus are discharged to an area with no ignition sources or they are diluted with air to below the lower explosion limit (LEL).
- b) Ensure the fire risk is reduced as much as possible by minimising the quantities of flammable liquids stored in the test area.
- c) If a spillage occurs in the heating chamber the heating /cooling system should be immediately shut down, but the air circulation system and any exhaust systems kept running.
- d) Use suitable plastic or plastic-coated glass containers in preference to glass containers, as they are less likely to break, or spillages will be contained in the event of breakage.
- e) Take adequate precautions to prevent exposure of personnel to harmful levels of toxic substances and combustion products.
- f) Hot surfaces shall be suitably guarded to minimise the risk of burn injuries.
- g) If mixtures are produced a check should be made that they do not react and they can be stored without change.
- h) The high-voltage connections on the transformer, the supply lines and the electrodes in the test vessel shall be suitably guarded to minimise the risk of electric shock.
- i) Ensure that the liquids under test are thermally stable at all test conditions.
- j) Fit the heating chamber with a spill tray to retain any spilled liquids.

Annex B (informative)

Estimation of explosion points

WARNING — These calculations are only intended to estimate the test start temperature. They should not be used as a conclusive estimate of the explosion points themselves.

B.1 General

Prerequisites for estimating the explosion points are:

- reliable vapour pressure curves (Antoine equations);
- reliable activity coefficients;
- reliable explosion limits.

B.2 Pure substances

Estimate lower and upper explosion points (EP) in °C using Equation (B.1).

$$EP = \frac{B}{A - \log_{10}(\varphi_{\text{lim}} \cdot P_0 \cdot 0,01)} - C_A \quad (\text{B.1})$$

Where

- EP is the explosion point (°C);
 A, B, C_A are the constants of the Antoine equation;
 p is the atmospheric pressure (kPa);
 p_0 1 kPa;
 φ_{lim} is the explosion limit (lower or upper) (vol %).

B.3 Mixtures of flammable components only

Prerequisite: The temperature dependence of the saturated vapour pressure is known for each mixture component.

Estimate explosion points for mixture (EP_{mix}) according to Equation (B.2).

$$\sum_{i=1}^k \gamma_i \cdot x_i \cdot 10^{\frac{B_i(EP_{\text{mix}} - EP_i)}{(EP_{\text{mix}} + C_{Ai})(EP_i + C_{Ai})}} = 1 \quad (\text{B.2})$$

where

- C_{Ai}, B_i are the Antoine equation constants for the i -th component in the liquid phase;
 EP_i are the explosion point of the i -th component (°C);
 k is the number of mixture components;
 x_i is the molar fraction of the i -th component in the liquid phase;
 γ_i is the activity coefficient of the i -th component.

NOTE Experience has shown that Equation (B.2) can underestimate the value of the UEP. Caution needs to be exercised when using this estimate as a starting point for UEP determination.

Annex C (normative)

Criteria for flame detachment

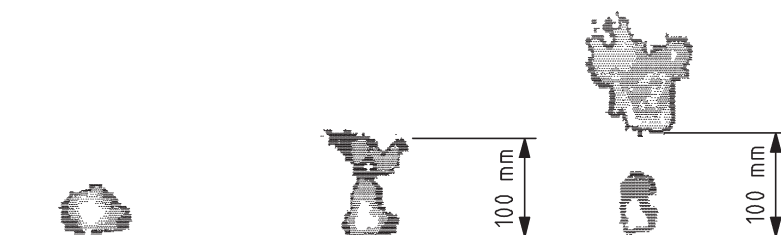


Figure C.1 — Flame detachment

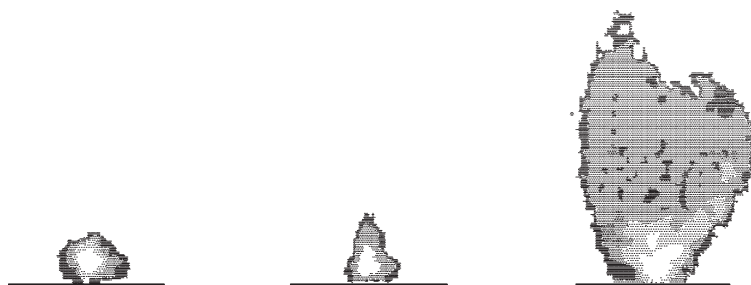


Figure C.2 — Halo (UNDETACHED FLAME)

Annex D (normative)

Verification

This verification procedure shall be used for a new apparatus as well as for checking the performance of existing apparatus. Existing apparatus shall be checked every twelve months, before use if the apparatus has not been used for more than twelve months or whenever parts of the apparatus have been changed or renewed. The verification shall be carried out according to the procedures given in subclause 6.2 with the liquids listed in Table D.1. It is recommended to use all listed liquids for verification of a new apparatus and at least two for checking existing apparatuses. It is not necessary to use acetone if sub-ambient measurement is not required. For verification it is sufficient to fill the test vessel to 10 % of its volume. Verification will be confirmed if the LEP is within the range listed in the table. The purity of the liquids used as a reference liquid has to be at least 99,8 %.

Table D.1 — Data for verification of the apparatus

Liquid	LEP [°C]
Acetone	- 23,0 ± 2,0
m-Xylene	23,0 ± 2,0
n-Octanol	77,5 ± 2,0
n-Hexadecane	122,0 ± 2,0

NOTE Additional special sets of data, obtained with an apparatus, whose verification has been confirmed, may be useful to check the performance of the apparatus.

Annex E (informative)

Example for a form expressing the results¹⁾ Test Report

Name of test laboratory: _____

Determination of LEP [] UEP [] according to

Sample: _____

Purity/ _____

Source: _____

Ambient pressure: _____

Dimensions of test vessel

percentage fill _____

	°C
LEP _o [] UEP _o []	
LEP [] UEP []	

Temperature step width.....

At the temperature stated, the sample has no LEP [] UEP []

Comments:

.....

Date: Operator:.....

Signature:

1) May be copied for reporting the results of the test

Annex ZA (informative)

Relationship between this European Standard and the Essential Requirements of EU Directive 94/9/EC

This European Standard has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association to provide a means of conforming to Essential Requirements of the New Approach Directive 94/9/EC of 23rd March 1994 concerning equipment and protective systems intended for use in potentially explosive atmospheres.

Once this standard is cited in the Official Journal of the European Union under that Directive and has been implemented as a national standard in at least one Member State, compliance with the clauses of this standard given in Table ZA.1 confers, within the limits of the scope of this standard, a presumption of conformity with the corresponding Essential Requirements of that Directive and associated EFTA regulations.

Table ZA.1 — Correspondence between this European Standard and Directive (94/9/EC)

Clause(s)/sub-clause(s) of this EN	Essential Requirements (ERs) of Directive ...	Qualifying remarks/Notes
Clause 4 to Clause 9 and Annexes B and C.	Annex II, Clauses 1.0.1, 1.0.6a; 1.0.6c; 1.2.3, 1.2.9, 2.1.1.1; 2.2.1.1; 2.3.1.1 .	

WARNING — Other requirements and other EU Directives may be applicable to the product(s) falling within the scope of this standard.

Bibliography

- [1] EN ISO 3170, *Petroleum liquids – Manual sampling (ISO 3170:2004)*
- [2] EN ISO 3171, *Petroleum liquids – Automatic pipeline sampling (ISO 3171:1988)*
- [3] EN ISO 15528, *Paints, varnishes and raw materials for paints and varnishes – Sampling (ISO 15528:2000)*

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