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Ugotavljanje točk eksplozije vnetljivih tekočin

Determination of explosion points of flammable liquids

Bestimmung von Explosionspunkten brennbarer Flüssigkeiten

Détermination des points d'explosion des liquides inflammables

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Determination of explosion points of flammable liquids

Détermination des points d'explosion des liquides inflammables

Bestimmung von Explosionspunkten brennbarer Flüssigkeiten

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of an identical text or by endorsement, at the latest by April 2010, and conflicting national standards shall be withdrawn at the latest by April 2010.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

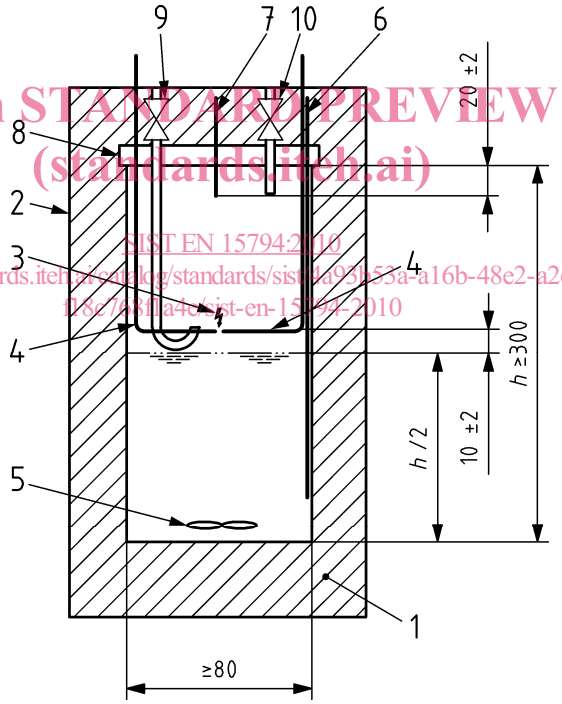
Viscous liquids, liquids which crystallise on cooling, polymerize or separate are to be stored at just above the temperature at which this occurs.

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
2 Test vessel
3 Ignition source (spark)
4 Electrodes
5 Stirrer
6 Thermocouple determining the explosion point and checking the equilibrium
7 Thermocouple for ignition detection and checking equilibrium
8 Tightly fitting lid allowing for pressure relief
9 Inlet valve
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 ± 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 ± 3) °. The distance between the tips shall be (5 mm ± 0,1 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