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Ugotavljanje temperature vžiga plinov in hlapov

Determination of the auto ignition temperature of gases and vapours

Bestimmung der Zündtemperatur von Gasen und Dämpfen

Détermination de la température d'auto-allumage des gaz et des vapeurs

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Détermination de la température d'auto-allumage des gaz et des vapeurs

Bestimmung der Zündtemperatur von Gasen und Dämpfen

This European Standard was approved by CEN on 1 August 2005.

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Contents

Foreword

This European Standard (EN 14522:2005) 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of an identical text or by endorsement, at the latest by March 2006, and conflicting national standards shall be withdrawn at the latest by March 2006.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive.

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

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

Introduction

To avoid the hazard of explosion, an appropriate measure is to prevent effective ignition sources. Hot surfaces (heated active or passive) are one of the widespread potential ignition sources. The ignition potential of hot surfaces can be characterized with respect to the flammable substance under use by the auto ignition temperature of the flammable substance.

The auto ignition temperature depends mainly on:

— the properties of the flammable substance;

— oxidiser;

— pressure;

— volume of the test vessel;

— material of the test vessel (hot surface);

— shape of the hot surface (this includes the fact whether the hot surface is surrounded by the cool flammable mixture or the flammable mixture is surrounded by the hot surface);

— flow and turbulence of the mixture;

— inert gas.

Therefore it is necessary to standardize the conditions at which the auto ignition temperature is to be determined.

Auto ignition temperatures as determined according to this European Standard are used first of all for classifying substances and explosion-proof electrical as well as non-electrical equipment into temperature classes. They may be used for designing explosion protection measures when the influence of process conditions is known and taken into account. They may also be element of fire risk assessment.

Because of the influences mentioned above, care shall be taken when applying such results measured under laboratory conditions to industrial applications.

The apparatus and procedure described below is also used for carrying out the ‘Surface ignition test’ in IEC 60601-2-13 ‘Medical electrical equipment – Part 2-13: Particular requirements for the safety of anaesthetic systems’.

1 Scope

This European Standard test method is designed to determine the auto ignition temperature of a flammable gas or vapour in mixture with air, or air/inert gas, at ambient pressure up to 650 °C. It is not suitable to describe the interactions of hot surfaces with explosives.

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 amendments) applies.

ISO 1773, *Laboratory glassware — Narrow-necked boiling flasks*

3 Terms and definitions

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

3.1
auto ignition temperature
T_i
lowest temperature (of a hot surface) at which under specified test conditions an ignition of a flammable gas or flammable vapour in mixture with air or air/inert gas occurs

3.2
ignition delay time
time between the completed injection of the flammable substance and the ignition

NOTE 1 The ignition delay time may vary between fractions of a second and some minutes.

NOTE 2 In literature auto ignition temperature is also referred to as self ignition temperature. In the case of dusts the respective safety characteristic is referred to as minimum ignition temperature.

4 Test method

4.1 Principle

The amount of substance and the temperature of the test vessel, which is filled with air or air/inert gas, are varied to find the lowest temperature (of the hot surface) that causes an ignition.

4.2 Apparatus

4.2.1 General

The test apparatus consists of:

— a test vessel;

— support for the test vessel;

— calibrated measuring thermocouple;

— an electrical hot-air oven;

— metering devices for metering the flammable substance;

— a mirror for observing the ignition;

— timer;

— equipment for cleaning the test vessel.

4.2.2 Test vessel and support

The test vessel shall be a 200 ml narrow-necked Erlenmeyer flask made of borosilicat glass according ISO 1773, or equivalent national standards. It shall be ensured that the inner surface of the bottom is not domed anyway. It shall be equipped with at least one calibrated measuring thermocouple of 1,5 mm maximum diameter, having an accuracy of 0,5 K. The thermocouple(s) shall be mounted with intimate contact to the external surface of the flask at a distance of (25 ± 2) mm to the bottom of the flask (see Annex B). The support for the Erlenmeyer flask shall ensure, that the heat dissipation via the support is as low as possible. If the support is mounted at the neck it shall be ensured that it does not use more than (5 ± 2) mm of the height of the neck of the Erlenmeyer flask (for example see Annex B).

NOTE If there is special need to know exactly the influence of volume on the auto ignition temperature additional experiments in larger (respectively smaller) volumes of the same shape and material may be carried out. Data from literature show a decrease of the auto ignition temperature with increasing volume where at the same time the ignition delay time increases. See Annex E.

4.2.3 Hot air oven and mounting of the vessel

The hot air oven shall be of sufficient dimensions to heat up the test vessel in a uniform manner. It shall be designed in such a way that

1) when the oven is covered with a well fitting lid and after having reached the respective temperature equilibrium

    — the temperature at the position of the measuring thermocouple and the temperature at the position of the centre of the bottom of the Erlenmeyer flask differ not more than 3 K over the whole temperature range and

    — the temperature measured at the position of the measuring thermocouple and the temperature measured at the position half of the height of the Erlenmeyer flask differ not more than 15 K over the whole temperature range;

2) when the oven is equipped with the Erlenmeyer flask filled with air

    — the temperature measured with the measuring thermocouple varies not more than 2 K over a period of 6 min over the whole temperature range.

Care shall be taken that there is no direct contact between the test vessel and the inner walls of the oven. The distance between the inner walls and the Erlenmeyer flask shall be at least 4 mm. The test vessel shall be mounted in such a way that

— it is totally immersed in the oven, whereas the oven should not overlap the Erlenmeyer flask more than 30 mm;

— it is uniformly heated;

— the mixture of air and flammable substance which is generated inside the Erlenmeyer flask by introducing the sample is not affected by the convection inside the oven;

— there is no possibility that the (explosive) mixture of air and flammable substance which is generated inside the Erlenmeyer flask by introducing the sample enters into the oven and

— that it is possible to meter the flammable substances and

— to observe the ignition

(see Annex B for schematic representation).

NOTE Ovens according to IEC 60079-4 or DIN 51794 are suitable ones.

4.2.4 Metering devices

— Gases

The metering device (e.g. flow meter, pump, syringe) shall be designed in such a way that it is possible to meter the gas with an accuracy of 10 % at a rate of (25 ± 5) ml/s. A filling tube that can be introduced into the test vessel shall be connected (movably) to the metering device.

— Liquids

The metering device (e.g. pump, pipette, syringe) shall be designed in such a way that it is possible to meter droplets having a volume of (25 ± 10) µl.

4.2.5 Mirror

A mirror of sufficient dimensions shall be positioned in such a way that it allows to observe the ignition of the flammable gas/air mixture (e.g. a distance of about 25 cm above the opening of the test vessel).

4.2.6 Timer

A timer calibrated in one-second intervals shall be used to determine the ignition delay time.

4.2.7 Equipment for cleaning the test vessel with air

The equipment (e.g. pump, air gun) shall allow the quick and complete cleaning of the test vessel by flushing it with clean air. After the cleaning only pure air shall fill the flask.

4.2.8 Automated apparatus

If automated apparatus are used they shall fulfil all the requirements stated in 4.2.2 to 4.2.7. If the monitoring of the ignition is automated as well, it shall be ensured that all kinds of flames (even the very pale ones and the very small ones) are monitored e.g. by thermocouple and photodiode. For safety reasons additional visual observation should be possible.

4.3 Ignition criterion

Any visible flame observed via the mirror (apparatus in a darkened room) shall be taken as ignition.

NOTE Normally hot flames are observed. Even if they are very pale ones (e.g. hydrogen, methane), these are hot flames. Some substances or mixtures of substances are able to form cool flames. Cool flames are characterized by a very weak luminosity and a small temperature difference (increase) to the unburned mixture. They are the first steps in the case of multi step ignition. They are more likely to appear in rich mixtures than in lean ones and with larger molecules than with smaller ones, as well as in larger volumes than in smaller ones. The apparatus and procedures described here cover to some extent this phenomenon because, especially with larger molecules, that means lower volatile substances inhomogeneous vapour/air mixtures are formed. Inhomogeneous mixtures can be the prerequisite for multi step ignition.

4.4 Sampling, preparation and preservation of test samples

4.4.1 Sampling

For sampling of liquid or gaseous products use the respective national/international standards.

NOTE 1 Respective standards are e.g. EN ISO 3170, EN ISO 3171, EN ISO 15528 etc.

Samples shall not be taken or stored in plastic containers, which allow volatile materials to permeate through the walls and/or react with the container.

In the case of liquid mixtures, the containers shall have a filling amount of more than 80 %.

The sample shall be stored at appropriate temperatures to avoid any change in the composition.

NOTE 2 If the sample consists of a gas mixture that is removed from a container containing a liquid phase, take into account that the composition of the gas and the liquid phase is different. It is recommended to take the test substance from the liquid phase.

4.4.2 Preparation and preservation

The components necessary for the test shall fulfil the following requirements:

Air: The air shall be free of water and oil. If synthetic air is used, it shall be stated in the test report.

Inert gas: The purity of the inert gas or the mixture of inert gases shall correspond to a mole fraction of 99,8 % or better. If a mixture of inert gases is used, the composition of the mixture shall be stated in the test report.

Flammable substance: the flammable substance may be derived from:

— a single substance or a mixture of substances;

— a process sample (of known or unknown composition).

When a single substance or a mixture of substances is used, the purity of each substance shall correspond to a mole fraction of 99,8 % or better. In the case of a mixture of substances or a process sample of known composition the precision of the composition shall be stated in the test report. In the case of a process sample of unknown composition, the source of the sample or the sample shall be defined as well as possible (e.g. process conditions, other physical properties or safety characteristic data).

4.5 Procedure

4.5.1 Metering of the sample

— Gases

The metering device and the connected filling tube are purged sufficiently (at least 10 times the volume) and then filled with the gas. The filling tube is introduced into the test vessel so that the outlet of the tube has a distance of (10 ± 2) mm to the bottom. The required volume is injected into the test vessel at a rate of (25 ± 5) ml per second. The filling tube is then quickly withdrawn from the test vessel.

— Liquids

The required volume shall be injected in portions of (25 ± 10) µl (see 4.2.3) droplets into the centre of the test vessel at a rate of 1 to 2 droplets per second. Care shall be taken to meter the sample in single droplets. Wetting of the side walls of the test vessel shall be avoided. The metering device shall be withdrawn quickly after metering.

NOTE For substances capable to undergo fast pre-reactions at or near the auto ignition temperature (e.g. carbonic acid ethylesters) introducing the liquid by spraying instead of dropping may result in lower auto ignition temperatures (M. Gödde et al.: PTB-Mitt. 108(1998), 437–441).

4.5.2 Determination

4.5.2.1 General considerations

The auto ignition temperature is determined by varying the temperature of the test vessel and the amount of the flammable substance. To reduce the number of necessary trials to a minimum one of the two procedures described below shall be followed.

For many substances the ignition temperature as a function of the amount of flammable substance has a nearly parabolic shape. In such a case it may be suitable to use method P.

If there is no knowledge about the composition of the flammable substance under test or if there is no hint for a parabolic behaviour method S is recommended.

The method used shall be stated in the test report.

Regardless of the method used, it shall be ensured before each test series (step 1 to step 6 method S; step 1 to step 5 method P) that:

— the test vessel is clean, dry, without any residue and any visible alteration of the inner surface;

— the test vessel is flushed completely by clean dry air before each injection of flammable substance;

— before injecting the sample, the temperature of the test vessel is the intended one because cleaning and purging may lower the temperature.

If the auto ignition temperature is to be determined in mixture with air/inert gas, purge the test vessel with the air/inert gas mixture after cleaning (with air) so that the atmosphere inside the Erlenmeyer flask is completely changed, or clean the test vessel with the air/inert gas mixture before each injection.

4.5.2.2 Method S

Carry out the following step 1 to step 7. Step 2 to step 7 shall be carried out at least three times. If the results obtained (step 7) scatter more than 2 % carry out two additional test series.

Note: If there are doubts on the results with respect to the Erlenmeyer flask it may be helpful to carry out one check test with one of the substances mentioned in Annex A chosen according to the respective temperature.

Step 1

Choose a starting temperature above (10 K to 20 K) the estimated auto ignition temperature.

If no appropriate starting temperature can be chosen carry out a preliminary test: Starting from a suitable temperature the test vessel is heated up with a temperature rate of (5 ± 1) K/min. Whilst heating up inject every 20 K, (50 ± 5) ml in case of a gas or 5 droplets in case of a liquid till an ignition occurs after complete injection of the sample. Thereby the test vessel shall be flushed completely by clean dry air before each injection. If no suitable starting temperature can be derived from existing knowledge (e.g. analogy to known samples) start to inject the sample at 80 °C.

Step 2

Heat the test vessel to the ignition temperature found in step 1. Inject (50 ± 5) ml in the case of a gas or 5 droplets in the case of a liquid. If an ignition occurs within 5 min, lower the temperature of the test vessel in intervals of (5 ± 1) K till no ignition occurs within 5 min after introducing the same amount of substance.