SLOVENSKI STANDARD
SIST EN 15188:2021

01-april-2021

Nadomešča:
SIST EN 15188:2007

Določanje lastnosti samovžiga usedlih plasti prahu

Determination of the spontaneous ignition behaviour of dust accumulations

Bestimmung des Selbstentzündungsverhaltens von Staubschüttungen

Détermination de l'aptitude à l'auto-inflammation des accumulations de poussières

Ta slovenski standard je istoveten z: EN 15188:2020

ICS:

13.220.40	Sposobnost vžiga in obnašanje materialov in proizvodov pri gorenju	Ignitability and burning behaviour of materials and products
13.230	Varstvo pred eksplozijo	Explosion protection

SIST EN 15188:2021 **en,fr,de**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 15188

December 2020

ICS 13.230

Supersedes EN 15188:2007

English Version

Determination of the spontaneous ignition behaviour of dust accumulations

Détermination de l'aptitude à l'auto-inflammation des accumulations de poussières

Bestimmung des Selbstentzündungsverhaltens von Staubschüttungen

This European Standard was approved by CEN on 18 October 2020.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Ref. No. EN 15188:2020 E

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European foreword

This document (EN 15188:2020) 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of an identical text or by endorsement, at the latest by June 2021, and conflicting national standards shall be withdrawn at the latest by December 2021.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 15188:2007.

This document has been prepared under a standardization request given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive 2014/34/EU.

For relationship with EU Directive 2014/34 EU, see informative Annex ZA, which is an integral part of this document.

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

Introduction

The self-ignition behaviour of dusts and granular materials and their mixtures depends on their chemical composition as well as on related substance and bulk properties. It also depends on the size and geometry of the body of material, and, last but not least on the ambient temperature.

The reason for self-heating (with possible self-ignition) is that the surface molecules of combustible dust or granular materials undergo exothermic reactions with air or other oxidising atmospheres transported into the void volume between the particles even at normal ambient temperatures. Any heat then released will cause the temperature of the reactive system to rise, thus accelerating the reaction of additional molecules with oxygen, etc. A heat balance involving the heat produced inside the bulk (quantity and surface of reactive surface molecules, specific heat producing rate) and the heat loss to the surroundings (heat conductivity and dimension of the bulk, heat transfer coefficient on the outside surface of the bulk and the size of the latter) is decisive as to whether a steady-state temperature is reached at a slightly higher temperature level (the heat loss terms are larger than the heat production term), or whether temperatures in the bulk will continue to rise up to self-ignition of the material, if heat transport away from the system is insufficient (in this case the heat production term is larger than all heat losses).

The experimental basis in this document for describing the self-ignition behaviour of a given dust or granular material is the determination of the self-ignition temperatures (T_{SI}) of differently sized bulk volumes by isoperibolic hot storage experiments (storage at constant oven temperatures) in commercially available ovens. The results thus measured reflect the dependence of self-ignition temperatures upon volume of the accumulation.

Different evaluation procedures – described in Annex A – allow interpolation and extrapolation, to characterize the self-ignition behaviour of deposits of a different scale and of different bulk geometric shapes. Primary method is the evaluation based on the thermal explosion theory according to Frank-Kamenetskii (A.2) and Thomas (A.3).

Interlaboratory tests have shown, that it is necessary to provide prescribed test conditions, e.g. by installation of a mesh wire screen into the oven, surrounding the dust samples and the thermocouples. In this way the spread of results will be minimized. If it is possible based on suitable thermo-analytic test procedures (adiabatic, isothermal or dynamic tests) to derive a reliable formal kinetic model, which describes the heat production of the substance as a function of temperature, then the volume dependency of the self-ignition temperature may be calculated according to the methods described in Annex A.

1 Scope

This document specifies analysis and evaluation procedures for determining self-ignition temperatures (T_{SI}) of combustible dusts or granular materials as a function of volume by hot storage experiments in ovens of constant temperature. The specified test method is applicable to any solid material for which the thermal explosion theory according to A.2 holds (i.e. not limited to only oxidatively unstable materials).

The specified test is applicable to any dust or granular material that reacts primarily with oxygen from the air. For safety reasons, this test is not used with materials mixed with solid/liquid oxidant (e.g. gunpowder, thermites, wood impregnated with liquid oxygen) or materials that could undergo violent non-oxidative reactions (e.g. peroxides, explosives). On a case by case basis, some types of materials undergoing non-oxidative reactions (e.g. non-violent exothermic decomposition reactions) may be however tested provided that additional safety precautions are taken. Where any doubt exists about the existence of hazard due to the properties of the test material (e.g. toxic or explosive), expert advice is sought.

This document is not applicable to the ignition of dust layers or bulk solids under aerated conditions (e.g. as in fluid bed dryer).

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.

EN 1127-1:2019, *Explosive atmospheres - Explosion prevention and protection - Part 1: Basic concepts and methodology*

EN 13237:2012, *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 following terms and definitions given in EN 13237:2012, EN 1127-1:2019 and the following apply.

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

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

3.1
self-ignition temperature
T_{SI}
highest temperature at which a given volume of dust just does not ignite

Note 1 to entry: Self-ignition temperature is expressed in °C.

3.2
oven temperature
arithmetic mean of the measured values of two thermocouples, both freely installed in an oven at a distance of 5 cm to the surface of the dust sample

Note 1 to entry: Oven temperature is expressed in °C.

3.3
sample temperature
temperature measured at the centre of the dust sample using a thermocouple

Note 1 to entry: Sample temperature is expressed in °C.

3.4
induction time
interval of time between reaching the storage temperature and start of ignition (defined by the inflection point, see Figure 3 case C)

Note 1 to entry: Induction time is expressed in h.

3.5
ignition
initiation of combustion

[SOURCE: EN ISO 19353:2019, 3.20]

3.6
bulk density
sample mass divided by the determined volume of the basket

3.7
dust
finely divided solid particles, up to 500 µm in nominal size

3.8
granules
discrete particles larger than 500 µm

4 Test apparatus

4.1 Sample baskets

The samples have to be filled into mesh wire baskets of different volumes. The baskets have to be open at the top and closed at the bottom. They shall consist of two layers of narrow-meshed wire net, made of e.g. stainless steel. The width of the mesh for the inner sample container has to be chosen in such a way that the dust cannot fall through the mesh, but the diffusion of the oven air into the dust sample is not hindered (e.g. mesh opening of 0,05 mm). The outer basket should be made from coarser mesh wire (e.g. mesh opening of 0,5 mm). The outer basket defines the test volume therefore the inner basket should be tightly fitting within, such that it does not distort when inserted. Recommended shapes of the mesh wire baskets are that of a cube, or that of a cylinder with a height to diameter ratio of 1.

To allow an assessment of the self-ignition behaviour of larger sizes of dust accumulations than the laboratory-scale, by extrapolation, at least four mesh wire baskets of different volumes have to be used for the tests.

The smallest volume should normally be in the order of 100 cm^3 and the largest should normally not be smaller than approximately 1 000 cm^3. The volume ratio of the baskets should be approx. 1 : 1,7 : 5: 8; e.g. cubes with edge length of 5 cm, 6 cm, 8,5 cm and 10 cm.

NOTE 1 Larger baskets are acceptable when sufficient material is available.

If only a limited amount of sample material is available, even smaller baskets may be used.

NOTE 2 For the sake of comparing products with respect to their self-ignition behaviour in devices or apparatus, where the sizes of the dust accumulations are limited for the reason of a specific design, often the determination of the self-ignition temperature for a basket of 400 cm^3 or 1 000 cm^3 is sufficient.

4.2 Determination of Basket Volume

The volume of the sample baskets shall be determined by using suitable material consisting of sufficiently small particles of spherical shape having a smooth surface and therefore of a known and stable bulk density, e.g. glass beads with a diameter of approx. 0,3 mm. The baskets are filled with the suitable material; any surplus material from the upper margin has to be removed. The basket is weighed before and after filling. The volume results from the weight of the filled in material with known bulk density (mean value of at least two measurements).

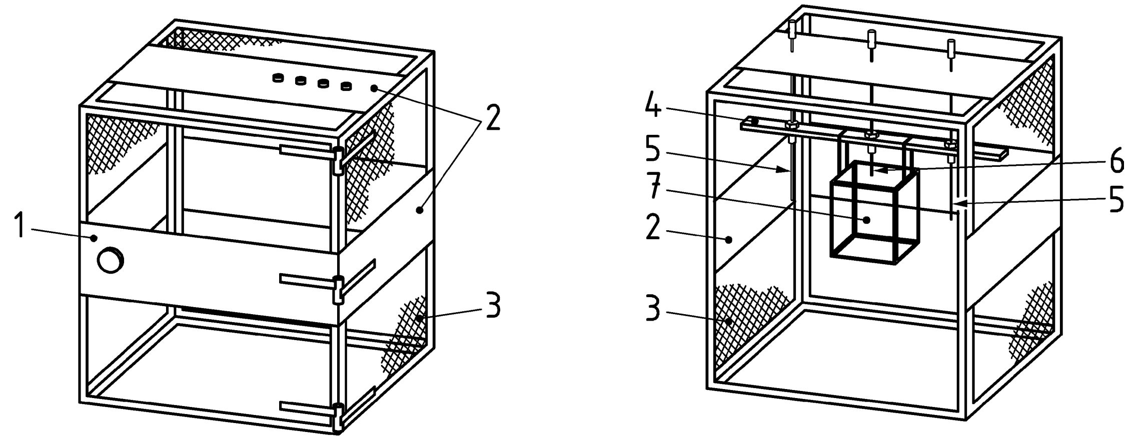
4.3 Oven and test conditions

Commercially available ovens (natural and forced convection) can be used. They shall have an air inlet opening in the lower section and an air outlet opening in the upper section and should be controllable in a temperature range from 35 °C to 300 °C. After reaching the test temperature the oven temperature shall be stable over time within a range of ± 1 K.

The temperature field surrounding the sample shall exhibit maximum spatial temperature differences of 4 K at an oven temperature of 120 °C. To determine the differences the temperature shall be measured on 6 sides of a 10 cm sample cube in a distance of 5 cm, respectively. This measurement shall be performed after installing the set-up and if the set-up (including hot storage oven) is changed. The measurement shall be repeated once a year. Radiative heat transfer across the sample surfaces should be minimized.

To achieve these conditions a mesh wire screen shall be installed into the oven. The minimum distance between the screen and the oven walls shall be 5 cm. The screen consists of mesh wire with mesh opening of e.g. 0,5 mm. The screen shall be equipped with a front door and a sample holder, see Figure 1. The sample baskets shall freely hang in the oven. Additional metal sheets (thickness 0,25 mm to 0,5 mm) can be installed at half height and on top to achieve the above conditions. The width of the sheets shall be chosen in such a way, that the largest sample basket, hanging in the screen, is covered.

For measuring the oven temperature two thermocouples have to be installed on opposite sides at a distance of 5 cm to the sample. The thermocouples have to be re-positioned for sample baskets of differing sizes. The minimum distance between the thermocouples and the wall of the screen is 2,5 cm, see Figure 2.



Key

1 front door

2 metal sheets (optional)

3 mesh wire

4 sample holder

5 thermocouples for measuring oven temperature

6 thermocouple for measuring sample temperature

7 mesh wire container with dust sample

Figure 1 — Mesh wire screen to be installed into the heating oven

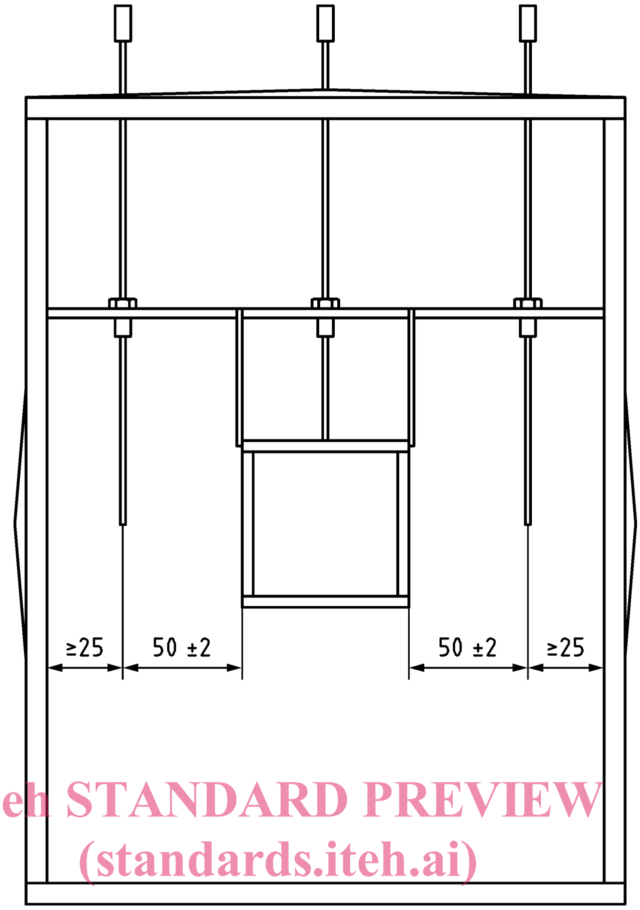


Figure 2 — Position of sample and thermocouples

Alternative test arrangements can be used to provide the specified test conditions. In this case, the uncertainty specified in Clause 8 may be not applicable.

4.4 Thermocouples

Both for measuring the sample temperature as well as for measuring the oven temperature, sheathed thermocouples with an external diameter of e.g. 1 mm are recommended.

4.5 Temperature recording equipment

Appropriate data acquisition may be used for measuring and recording signals of the thermocouples.

5 Preparation of dust samples

To investigate situations occurring in practice a representative sample should be used (produced by the operating conditions of the process). The sample characteristics shall be recorded in the test report.

The bulk density of the sample should be adjusted to the respective practical conditions (if known).

The bulk density shall be determined in the baskets and shall not vary by more than ± 2 % in the test series.

Due to the limited size of the sample baskets used, it may be necessary to remove larger particles from the sample (e.g. fraction > 20 mm)

6 Procedure

6.1 Experimental Procedure

The test basket shall be filled with the dust sample with the settled bulk density. Any surplus dust from the upper margin has to be removed. It shall be checked by weighting if the settled bulk density is reached with an accuracy of ± 2 %. Position the basket at the centre of the oven (see Figure 1) that has been preheated to the test temperature.

NOTE It is also possible to position the basket at the centre of cold oven if the target oven temperature is reached and is stable within 30 min.

The thermocouple for measuring the sample temperature is to be positioned with its hot junction directly at the centre of the sample. The hot junctions of two additional thermocouples on opposite sides will be freely installed in the air space at a distance of 5 cm to the sample (see Figure 2). These two thermocouples are used to measure the oven temperature, corresponding to, in critical cases, the T_{SI}. The temperatures of these three thermocouples shall be recorded continuously over time.

In cases where the sample ignites the oven temperature may increase due to the heat released by the burning sample. To prevent misinterpretation the oven temperature shall be taken at the crossing point (where the temperature of the sample first reaches, and is equal to, the oven temperature) for the evaluation of the test.

The temperature difference of both thermocouples measuring the oven temperature shall not exceed 2 K. If a larger temperature difference is observed the adjustment of the thermocouples and, if necessary, the spatial temperature differences around the sample shall be checked.

The air inlet and air outlet openings of the oven shall be left open during the test to enable fresh air to enter and combustion gases to leave the oven. A sufficient number of hot storage tests – with a fresh dust sample for each test – shall be carried out to determine the highest oven temperature at which no ignition occurs, as well as the lowest oven temperature at which the dust sample showed an ignition for each sample volume chosen. Normally the test can be stopped if the temperature in the sample falls (see case B in Figure 3) or if a situation like case C in Figure 3 occurs. Striking features during testing (e.g. production of gases, physical changes to the sample) and the mass loss from samples shall be written down.

Figure 3 is an idealised one. In some cases, the type B curve is followed by a steep increase of sample temperature after the temperature drop has occurred. Attention should be paid to the fact that this may happen after significantly long periods of time. In such cases, such modified type B curves have to be evaluated as type C ones. This situation may also occur with type A curves.