SLOVENSKI STANDARD
SIST EN 15188:2007

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Določanje lastnosti samovžiga usedlih plasti prahu

Determination of the spontaneous ignition behaviour of dust accumulations

Bestimmung des Selbstentzündungsverhaltens von Staubschüttungen

Détermination de l'aptitude a l'auto-inflammation des accumulations de poussieres

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13.230	Varstvo pred eksplozijo	Explosion protection

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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

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EUROPEAN COMMITTEE FOR STANDARDIZATION
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Foreword

This document (EN 15188:2007) 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of an identical text or by endorsement, at the latest by February 2008, and conflicting national standards shall be withdrawn at the latest by February 2008.

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For relationship with EU Directive 94/9/EC, 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, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

The self-ignition behaviour of combustible dusts depends on their chemical composition as well as on related substance 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 behind self-heating (or possibly self-ignition) is that the surface molecules of combustible dust particles undergo exothermic reactions with the oxygen in air transported into the void volume between the particles even at normal temperatures. Any heat then released will cause the temperature of the reactive dust-air system to rise, thus accelerating the reaction of additional dust 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 dust, 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 for describing the self-ignition behaviour of a given dust is the determination of the self-ignition temperatures (T_{SI}) of differently-sized bulk volumes of the dust by isoperibol hot storage experiments (storage at constant oven temperatures) in commercially available ovens. The results thus measured reflect the dependence of self-ignition temperatures upon dust volume.

Plotting the logarithms of the volume/surface ratios [lg (V/A)] of differently sized dust deposits versus the reciprocal values of the respective self-ignition temperatures [$1/T_{SI}$ in K^{-1}] or following other evaluation procedures – described in Annex A – one produces straight lines, allowing interpolation, to characterise the self-ignition behaviour of dust deposits of a different scale and of a different bulk geometric shapes (see 5.1). Experience has shown that the spread of slopes of such straight lines determined by different laboratories using differently constructed ovens is fairly large. This is the reason why scale up of those results to industrial scale will lead to non-negligible errors in T_{SI}.

Experience has shown, that it seems necessary to prescribe the installation of a unique inner chamber into the oven, surrounding the dust samples and the thermocouples, with an also prescribed air flow through this chamber. In this way the spread of results should be minimised. Decisions on the design of this inner chamber and on the amount of air flow respectively other test setups leading to comparable results have to be carried out later on.

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 European Standard 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 linear correlation of lg (V/A) versus the reciprocal self-ignition temperature $1/T_{SI}$ (with T_{SI} in K) holds (i.e. not limited to only oxidatively unstable materials).

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

This European Standard should not be applied to dusts like recognised explosives that do not require atmospheric oxygen for combustion, nor to pyrophoric materials.

NOTE Because of regulatory and safety reasons “recognised explosives” are not in the scope of this European Standard. In spite of that, substances which undergo thermal decomposition reactions and which are not “recognised explosives” but behave very similarly to self-ignition processes when they decompose are in the scope. If there are any doubts as to whether the dust is an explosive or not, experts should be consulted.

2 Terms and definitions

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

2.1
self-ignition temperature
T_{SI}
highest temperature at which a given volume of dust just does not ignite

NOTE Self-ignition temperature is expressed in °C.

2.2
oven temperature
arithmetic mean of the measured values of two thermocouples, both freely installed in an oven at half the distance between the wall and the surface of the dust sample

NOTE Oven temperature is expressed in °C.

2.3
sample temperature
temperature measured at the centre of the dust sample using a thermocouple

NOTE Sample temperature is expressed in °C.

2.4
induction time
interval of time between reaching the storage temperature and an ignition

NOTE Induction time is expressed in h.

2.5
ignition
initiation of combustion

[EN 13478:2001, 3.20]

3 Test apparatus

3.1 Sample baskets

The samples have to be loosely filled into mesh wire baskets of different volumes. The baskets have to be open at the top and closed at the bottom. They consist of a narrow-meshed wire net, made of e.g. stainless steel. The width of the mesh has to be chosen in such a way that the dust cannot fall through the mesh, but the diffusion of oxygen from the oven air into the dust sample is not hindered. Recommended shapes of the mesh wire baskets are that of a cylinder with a height to diameter ratio of 1 or that of a cube.

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

NOTE 1 A higher level of certainty is achieved with four different sample volumes.

The smallest volume should normally be in the order of 10 cm^3 and the largest should normally not be smaller than approximately 1 l.

If only a limited amount of sample material is available, even smaller baskets may be used. However it has to be assured that the volume of the basket following in a series exceeds that of the previous one by a factor of 2 at a minimum.

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.

3.2 Oven

Commercially available ovens can be used. They shall have an air inlet opening in the lower section and an air outlet opening in the upper section (see schematic drawing in Figure 1). They should have a useful volume of about 120 l (enabling the installation of an inner chamber into the oven, surrounding the dust samples and the thermocouples, being equipped with an upstream heat exchanger consisting of copper tube coils) and be controllable in a temperature range from 35 °C to 300 °C. This construction enables a prescribed air flow through this chamber. The oven temperature shall be stable within a range of ± 1 % of the respective oven temperature.

Alternative test arrangements can be used to provide the specified test conditions. For example, the test basket can be shielded by placing it inside an additional mesh wire basket. Mechanically ventilated ovens can also be used provided the test basket is shielded (e.g. by using an inner chamber or an additional mesh wire basket).

3.3 Thermocouples

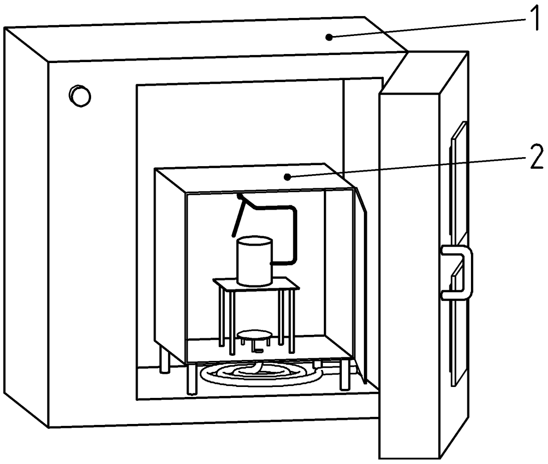
Both for measuring the sample temperature as well as for measuring the oven temperature, sheathed thermocouples with an external diameter of e.g. 1 mm are recommended.

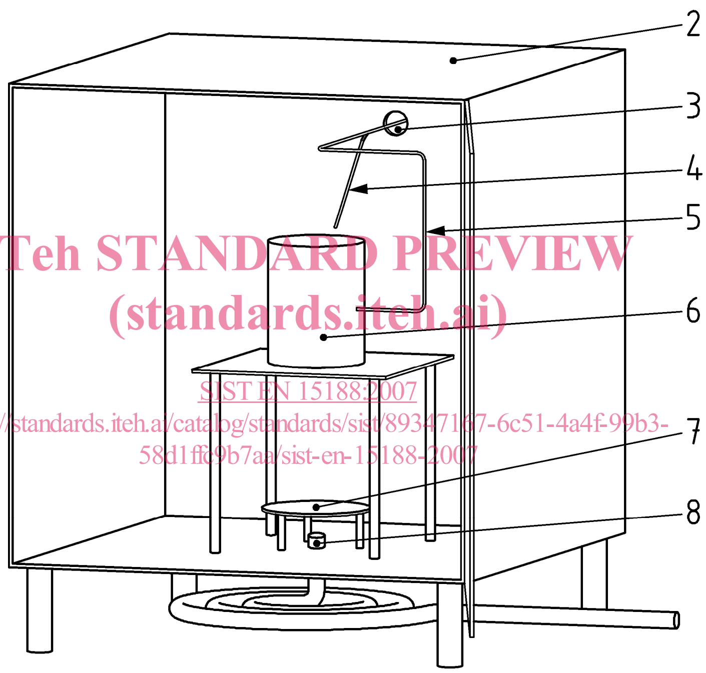
3.4 Temperature recording equipment

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

4 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 dust for each experiment is determined by weighing the baskets as well as the moisture content of the dust before and after filling.





Key

1 heating oven

2 inner chamber (volume ≈ 50 l)

3 air outlet, diameter ≈ 10 mm

4 thermocouple for measuring oven temperature

5 thermocouple for measuring sample temperature

6 wire gauze cylinder with dust sample

7 deflector

8 air inlet (preheated air, adjustable flow rate), diameter ≈ 8 mm

Figure 1 — Suggested experimental setup for hot storage tests

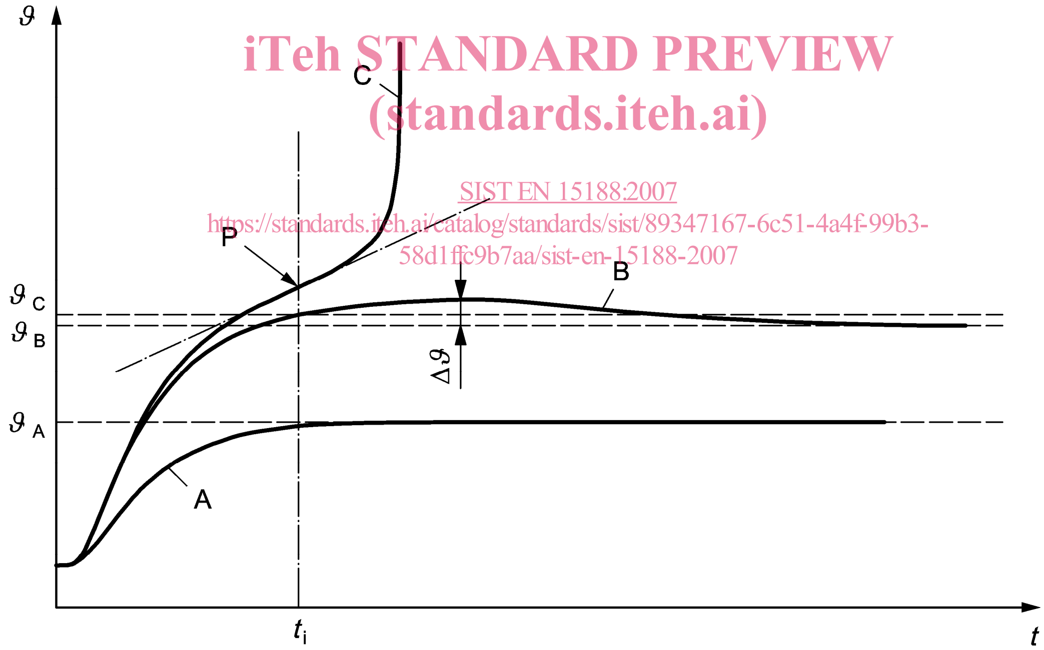
If the results are required to compare different dusts with each other (for the purpose of tabular data compilations), tests shall to be carried out with the dust having passed through a sieve of 250 μm mesh aperture and dried to constant weight at 50° C in a vacuum drying chamber. If necessary, the substance may be ground and/or sieved. All preparation procedures shall be recorded and included in the report, especially when altering the grain size, quoting the grain size distribution of the sample tested.

5 Procedure

5.1 Experimental Procedure

The test basket shall be filled with the dust sample by tapping it several times. Then remove any surplus dust from the upper margin and position the basket at the centre of the oven that has been preheated to the test temperature. 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, each at half the distance between the sample surface and the inner wall of the oven. These two thermocouples are used to measure the oven temperature, corresponding to, in critical cases, the T_{SI}. 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. The temperatures of these three thermocouples shall be recorded continuously over time. 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 2) or if a situation like case C in Figure 2 occurs. Striking features during testing (e.g. production of gases, physical changes to the sample) and mass lost of samples have to be written down.

NOTE Figure 2 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.



Key

ϑ temperature
t duration of the test
P inflection point

Figure 2 — Idealised temperature courses over time in dust samples of the same volume at hot storage temperatures ϑ_A to ϑ_C

When carrying out the tests using the procedure described in this European Standard, precautions to safeguard the health and safety of personnel and other persons shall be taken. Apart from the obvious hazards associated with the handling of the test dust and using equipment at high temperatures, there are two aspects that need special attention:

a) Many dusts will generate hazardous vapours (toxic, corrosive etc.) during decomposition. Adequate extraction should be provided to the area around the oven to capture any vapours emanating from the oven.

b) Some dusts can generate flammable gases or vapours during decomposition in sufficient quantity to create an explosive atmosphere inside the oven. Personnel shall be protected against the effects of such an explosion if this is a potential scenario, by appropriate measures.

5.2 Evaluation of tests

Figure 2 shows idealised temperature curves over time in samples from three hot storage tests of the same volume and the same dust, but at different oven temperatures. The dashed horizontal lines show the oven temperatures (ϑ_A to ϑ_C) of the respective hot storage tests (A, B and C).

If one works at temperatures significantly lower than the T_{SI} the sample temperature will approach asymptotically the oven temperature (curve A).

Higher oven temperatures show noticeable reactions with oxygen in the body of dust. Then sample temperatures temporarily will be higher than oven temperatures. This indicates the beginning of the self-heating processes, without self-ignition of the sample (curve B).

There are two methods to decide whether ignition takes place or not:

a) when the temperature at the centre of the sample rises at least 60 K above its oven temperature;

b) when the temperature at the centre of the sample shows an inflection point, with respect of time, if it occurs above the oven temperature.

Curve B relates to an oven temperature ϑ_B, slightly below the T_{SI}. At its maximum, the sample temperature surpasses the oven temperature by an amount $\Delta\vartheta$ (to be less than 60 K). Thereafter the sample temperature decreases to oven temperature.

Curve C relates to the oven temperature ϑ_C , a value just above the T_{SI}. Heat production in the sample has now reached a point at which it continuously surpasses the heat loss (by heat conduction, convection and radiation). No stationary condition can now occur. After an induction time the temperature of the sample raises rapidly, until self-ignition occurs.

The self-ignition temperature lies between the oven temperatures of curves B and C.

The selection of the hot storage temperatures for the decisive final two tests shall be made in such a way that the oven temperatures of the test just producing ignition (curve C) and that of the test not producing an ignition (curve B) differ by not more than 5 K. When extrapolating the results to larger storage volumes the margin between the final tests shall be smaller or equal to 2 K. The result of T_{SI} measurements is rounded down to the nearest degree.

Figure 3 plots the T_{SI} of many different volumes of a dust as a diagram (lg(V/A) versus $1/T$). The line passing through the T_{SI}-values separates the regions of steady state and unsteady behaviour of the dust volumes. Self-ignition occurs in the region above the curve.

Besides the temperature recordings, the time interval between the positioning of a sample in the oven and the achieving of the storage temperature as well as the complete storage period, should be recorded for every test. Additionally, the time interval between the achieving of the storage temperature and the ignition (induction time, case C), i.e. the achieving of the maximum temperature (case B) should be recorded.