SLOVENSKI STANDARD
kSIST-TS FprCEN/TS 17274:2018

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Nanotehnologija - Smernice za določanje protokolov za eksplozivnost in vnetljivost praškov, ki vsebujejo nanomateriale (za transport, ravnanje z njimi in shranjevanje)

Nanotechnologies - Guidelines for determining protocols for the explosivity and flammability of powders containing nano-objects (for transport, handling and storage)

Nanotechnologien - Leitfaden für Protokolle zur Bestimmung des Brand- und Explosionsverhaltens von Pulvern, die Nano-Objekte beinhalten (für Transport, Handhabung und Lagerung)

Nanotechnologies - Lignes directrices pour la détermination de protocoles pour l'explosivité et l'inflammabilité de poudre contenant des nano-objets (pour le transport, la manipulation et le stockage) central

Ta slovenski standard je istoveten z: **FprCEN/TS 17274**

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13.230	Varstvo pred eksplozijo	Explosion protection

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Nanotechnologies - Guidelines for determining protocols for the explosivity and flammability of powders containing nano-objects (for transport, handling and storage)

Nanotechnologies - Lignes directrices pour la détermination de protocoles pour l'explosivité et l'inflammabilité de poudre contenant des nano-objets (pour le transport, la manipulation et le stockage) central

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This draft Technical Specification is submitted to CEN members for Vote. It has been drawn up by the Technical Committee CEN/TC 352.

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Recipients of this draft are invited to submit, with their comments, notification of any relevant patent rights of which they are aware and to provide supporting documentation.

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

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Contents

Page

European foreword

This document (FprCEN/TS 17274:2018) has been prepared by Technical Committee CEN/TC 352 "Nanotechnologies", the secretariat of which is held by AFNOR.

This document is currently submitted to the Vote on TS.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

1 Scope

This document provides protocol guidelines for determining explosivity and flammability characteristics of powders containing manufactured nano-objects. These explosivity and flammability characteristics are needed for safety data sheets for safe storage, handling and transport of any powder.

In particular, this document will provide protocol guidelines concerning:

— the determination of flammability characteristics of powders containing nano-objects with regard to sensitivity to ignition sources;

— the ability of a powder containing nano-objects to generate an explosive atmosphere and the assessment of its explosion characteristics.

This document is not suitable for use with recognized explosives, such as gunpowder and dynamite, explosives which do not require oxygen for combustion, or substances or mixtures of substances which may under some circumstances behave in a similar manner. Where any doubt exists about the existence of hazard due to explosive properties, it is best to seek expert advice.

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 14034-1:2004+A1:2011, *Determination of explosion characteristics of dust clouds - Part 1: Determination of the maximum explosion pressure pmax of dust clouds*

EN 14034-2:2006+A1:2011, *Determination of explosion characteristics of dust clouds - Part 2: Determination of the maximum rate of explosion pressure rise (dp/dt)max of dust clouds*

EN 14034-3:2006+A1:2011, *Determination of explosion characteristics of dust clouds - Part 3: Determination of the lower explosion limit LEL of dust clouds*

EN 14034-4:2004+A1:2011, *Determination of explosion characteristics of dust clouds - Part 4: Determination of the limiting oxygen concentration LOC of dust clouds*

EN ISO/IEC 80079-20-2:2016, *Explosive atmospheres - Part 20-2: Material characteristics - Combustible dusts test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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 http://www.iso.org/obp

3.1
powder
assembly of discrete particles usually less than 1 mm in size

[SOURCE: EN ISO 3252:2000, 1001]

3.2
nano-object
discrete piece of material with one, two or three external dimensions in the *nanoscale*

Note 1 to entry: The second and third external dimensions are orthogonal to the first dimension and to each other.

[SOURCE: CEN ISO/TS 80004-1:2015, 2.5]

3.3
powder containing nano-objects
powder containing a specific relative amount of nano-objects in number, or displaying a specific surface area per volume, above a specific threshold value

3.4
smouldering temperature
describes the flammability behaviour of a flat dust layer on a hot surface. It is defined as the lowest temperature of a heated, free-standing surface which is capable of igniting a powder containing nano-objects

3.5
cloud
solids dispersed in a gaseous phase

3.6
flammable powder containing nano-objects
combustible powder containing nano-objects which ignites and burns, under effective ignition sources (see Clause 6)

Note 1 to entry: For the purpose of this document, the flammability characteristics of a powder containing nano-objects will be viewed as its sensitivity to thermal ignition sources.

3.7
pyrophoric solid
solid substance or mixture which, even in small quantities, is liable to ignite after coming into contact with air at ambient conditions.

Note 1 to entry: For the purpose of this document, pyrophoric powder containing nano-objects are considered to be flammable and may yield explosive atmospheres.

3.8
combustible powder
powder able to undergo an exothermic oxidation reaction with air when ignited

3.9
explosive atmosphere containing nano-objects
combustible powder containing nano-objects which may form an explosive mixture with air at atmospheric conditions

3.10
explosivity of an atmosphere containing nano-objects
aptitude of an explosive atmosphere containing nano-objects to explode.

Note 1 to entry: For the purpose of this document, the explosivity of a combustible powder will reside in its ability to form an explosive atmosphere in which a combustion reaction can propagate

3.11 Flammability characteristics

3.11.1
minimum ignition temperature (MIT) in layer of a powder containing nano-objects
lowest temperature of a hot surface at which ignition occurs in air in a layer of combustible powder containing nano-objects under specified test conditions

3.11.2
minimum ignition temperature (MIT) in cloud of a powder containing nano-objects
lowest temperature of a hot surface on which the most ignitable mixture of dispersed combustible nano-objects and air is ignited under specified test conditions

3.11.3
self-ignition temperature of accumulations of powders containing nano-objects
highest temperature of a given volume of an accumulation of a powder containing nano-objects which does not ignite

3.12 Explosion characteristics

3.12.1
minimum ignition energy (MIE) of an atmosphere containing nano-objects
smallest electrical energy stored in a capacitor, which is sufficient enough to ignite an explosive mixture in its most critical composition when discharged via a spark gap

3.12.2
lower explosion limit
LEL
highest concentration at which an explosion is not detected in three successive tests (see Clause 7)

3.12.3
initial pressure
P_i
pressure inside the explosion vessel at the moment of ignition

3.12.4
initial air temperature
T_i
temperature inside the explosion vessel at the moment of ignition

3.12.5
explosion pressure
p_{ex}
highest over pressure relative to the initial pressure occurring in a closed vessel during the explosion of a specific mixture of flammable powder containing nano-objects with air or air and inert substances determined under specified test conditions

3.12.6
maximum explosion pressure
p_{max}
maximum explosion overpressure (pressure above initial pressure conditions) during explosions of all explosive atmospheres in the explosion range of a combustible substance in a closed vessel under specified test conditions and ambient atmospheric conditions

3.12.7
maximum rate of pressure rise
$(dp/dt)_{max}$
maximum rate of explosion pressure rise is the maximum value of the pressure rise per unit time during explosions of all explosive atmospheres in the explosion range of a combustible substance in a closed vessel under specified test conditions and standard atmospheric conditions

3.12.8
normalized maximum rate of pressure rise
K_{st}, K_{max}
K_{st} and K_{max} are equivalent and defined as the maximum rate of pressure rise normalized for a container size of 1 m^3 (see 7.3.4.3)

Note 1 to entry: In K_{st}, st (staub) means dust in German.

3.12.9
limiting oxygen concentration
LOC
highest oxygen concentration in a mixture of a combustible dust and air and an inert gas, in which an explosion will not occur.

4 Safe handling of powders containing nano-objects

Preventive and protective safety measures, described as for example in ISO/TR 12885:2008 should be taken in carrying out all the tests discussed in this document. It is nevertheless advised to avoid using pyrophoric powders in the explosivity and flammability tests. Usual fire and explosion safety measures should be taken.

In the following, various tests of characterization related to the flammability and explosivity of powders containing nano-objects are presented. The choice of the tests to be conducted will depend upon the specific application of the user.

5 Preparation and characterization of samples

5.1 Receipt of sample

For very reactive powders containing nano-objects, samples can be received in suitable containers of small sizes (e.g.: 100 g/200 g), prepared under partial vacuum conditions or under argon or any inert preferably nontoxic fluid that can easily be separated from the solids.

The type of package used to store and transport the powder should be reported since it may alter the product specification and modify the explosivity and flammability characteristics of the powder containing nano-objects.

To be most representative of the material being used in the process, standardized sampling procedures should be followed, as the one described, for example, in the ISO 10725:2000. If no standardized sampling procedures are followed, the sample provider should mention that no sampling procedures were followed in providing the sample.

5.2 Characterization of the sample

The powder containing nano-objects shall be at least characterized in terms of particle size distribution and moisture content as:

— particle size distribution ($d_{10\%}$, median $d_{50\%}$, $d_{90\%}$) by any suitable methods in terms of volume, number (e.g. CEN ISO/TS 12025:2015, ISO 13320:2009, CEN/TS 17010:2016);

— moisture content (e.g. EN 14346:2006).

NOTE Additional characteristics may prove useful for interpretation of test results. These may include:

— nano-object size and shape of constituent or primary particles, possibly by TEM or SEM methods (e.g. CEN ISO/TS 17200:2015, ISO 13322-1:2014 and ISO 13322-2:2006);

— specific Surface Area, possibly by BET method (e.g. ISO 9277:2010).

These techniques may help assess whether the powder containing nano-objects exceeds a given threshold in terms of number size distribution or specific surface area per volume (e.g. 50 % in number size distribution or 60 m^2/cm^3 in specific surface area per volume). For such typical cases, increased vigilance on safety measures in handling and testing reactive powders should be given (see Clause 4).

Each method used for sample characterization should be mentioned in the report (e.g. use of Electron microscopy for particle size distribution measurements, protocol for measuring moisture content,…).

5.3 Preparation of sample

If it is not possible to test the sample as received (for example if the particle sizes are too large to be tested), or if the sample is to be tested under most conservative conditions, it may then be necessary to prepare specifically the sample for testing. This may include:

— Drying:

Drying should be performed without significantly decreasing the reactivity of the material.

— Sieving and/or milling/grinding (< 63 µm object size, to remove large objects):

If sieving does not yield the desired results, milling/grinding should be considered.

If milling, grinding or sieving is required, the samples should be prepared in a safe manner.

The sample preparation steps should be reported because the sample explosivity/flammability characteristics may be altered during these procedures. Pre-conditioning status of the sample received (e.g. use of inert gas) should be mentioned. If the samples are tested as received, the test report should mention it.

NOTE Sample preparation such as grinding and sieving, or drying may alter the material characteristics. For mixtures of powders, sample preparation can result in changes to the sample composition, which in turn can result in changes to the experimentally determined parameters.

Characterization of the powder to be tested shall be reported.

6 Flammability characteristics - Test methods to characterize the sensitivity to ignition sources

6.1 Test for pyrophoricity of a powder containing nano-objects

6.1.1 Test Equipment

Inert (non electrically charged) spoon, non combustible support.

6.1.2 Test Procedure and reporting results

A sufficient amount of the test substance (e.g.: 1 ml to 2 ml) is dropped from a height of 1 m through air onto a non-combustible support. It is observed whether the substance starts burning during the fall or within 5 min after the complete fall.

Six trials are made. A substance is considered as "pyrophoric" if ignition occurs at least in one trial. In this case, there is no need to further test flammability characterisitics.

6.2 Flammability characteristics in layers and accumulations

6.2.1 Burning tests

6.2.1.1 General

The aim of the test is to determine if and to what extent a fire started by an external source will propagate in a layer consisting of a powder containing nano-objects.

Two tests should be considered. The first one is a screening test so as to qualify the burning behaviour of the powder, and the second one is to quantify the degree of flammability (burning rate) of the powder.

6.2.1.2 Determination of the burning behaviour as a screening procedure

6.2.1.2.1 Test equipment

Ceramic plate, electrically heated glowing platinum wire at approximately 1 000 °C (diameter 1 mm, length 86 mm, intensity 30 A).

6.2.1.2.2 Test procedure and reporting results

At room temperature, the prepared sample (approximately 5 cm^3) is deposited on the ceramic plate as a strip approximately 2 cm wide and 4 cm long (Annex A, Figure A.1). One tries to ignite, within 15 s to 30 s, the sample from one end of the strip with the glowing platinum wire. In case of no ignition with the platinum wire, one can use a gas burner (minimum diameter 5 mm, at least 1 000 °C).

This screening test is carried out in a laminar flow hood. The burning characteristics are observed and rated as follows (Combustibility Indices 1 to 6, as follows):

1) No ignition

2) Extinction after short burning

3) Local burning or glowing, limited spread of the flame

4) Smouldering fire or slow flameless decomposition

5) Complete combustion of the heap

6) Very fast combustion or fast flameless decomposition

NOTE For metallic powders, ignition with a gas burner is advised.

6.2.1.3 Determination of burning rate of a layer of a powder containing nano-objects

6.2.1.3.1 Test equipment

Mould, gas flame micro burner or electrically heated glowing platinum wire (1 000 °C), placed into a laminar flow fume hood.

6.2.1.3.2 Test procedure and reporting results

Using a mould, a powder train of 250 mm length, 20 mm width and 10 mm height (Annex A, Figure A.2) is produced on an impervious, low heat-conducting base plate. The powder train is ignited at one end with the glowing platinum wire. In case of no ignition with the platinum wire, one can use a gas burner

(minimum diameter 5 mm, at least 1 000 °C). The ignition time is generally set up to 2 min, and up to 5 min for metal powders.

In a preliminary test, it is observed, whether the combustion propagates over a distance of 200 mm within 4 minutes. If the time is longer than 4 minutes (and 40 minutes for metal powders), the substance is considered as not "highly flammable solid". No further testing is required. In all other cases, the burning rate is determined by measuring the combustion time for over a distance of 100 mm after an initial burning distance of 80 mm from the ignition point.

A substance is classified a "highly flammable solid" if the combustion time over 100 mm is less than 45 s (or 10 min for metal powders) in one of six trials.

6.2.2 Determination of MIT of a layer of powder containing nano-objects

6.2.2.1 Test equipment

The apparatus consists of a heated plate and a powder ring (A suitable system is given in Annex A, Figure A.3).

Electrically heated circular plate made out of stainless steel, diameter 200 mm, thickness approximately 20 mm, electric power 2 kW to 3 kW, temperature controller and temperature recorder for the plate and sample temperature.

6.2.2.2 Test procedure and reporting results

For the determination of the minimum ignition temperature a layer of 5 mm thickness and with a diameter of 10 cm is prepared on a heating plate. Then it is exposed during at least 30 min (within a 2 h limit) to a constant elevated temperature of the plate. The Minimum Ignition Temperature (MIT) of the layer is the lowest temperature of the plate, at which the layer ignites, smoulders or self-heats to a temperature of 450 °C or 250 °C above the temperature of the plate. If none of these three conditions are met during four trials of at least 30 min (within a 2 h limit) each, the result of the plate temperature test is considered negative and the test is carried out at higher temperatures. In practice, the lowest temperature rounded down to an even 10 °C which will just result in a smouldering appearance or ignition will be defined as the MIT of the layer.

For further details, refer to EN ISO/IEC 80079-20-2:2016.

6.2.3 Determination of the spontaneous ignition behaviour of accumulations of powder containing nano-objects

NOTE These are powders which, in contact with air, are liable to self-heating.

6.2.3.1 Screening test procedure for self-ignition temperature and reporting results

6.2.3.1.1 Test equipment

Oven

A temperature-programmed laboratory oven (volume about 2 l) fitted with natural air circulation and explosion relief system (A suitable equipment is given in Annex A, Figure A.4).

Wire mesh cube

Stainless steel wire mesh basket.

6.2.3.1.2 Test procedure and reporting of the results

The size of the wire mesh has to be chosen in such a way that the powder cannot fall through the mesh and the diffusion of oxygen from the oven air should not be hindered. This test can only be applicable if the powder can be kept in the basket during the experiment.