TECHNICAL SPECIFICATION

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Nanomaterials — Quantification of nano-object release from powders by generation of aerosols

Nanomatériaux — Quantification de la libération de nano-objets par les poudres par production d'aérosols

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 352, *Nanotechnologies*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO/TS 12025:2012), which has been technically revised.

The main changes compared to the previous edition are as follows:

— revised and updated the Introduction and the Bibliography;

— updated 6.4.1 and 6.4.2 and Annex A with regards to the description and selection of the sample treatment procedure in accordance with new European standards.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Industrial powders when subjected to external energy or stress from handling and air flow will release particles entrained in the surrounding air to form aerosols. Aerosols in the nanoscale are more dynamic than micrometre sized particles because of greater sensitivity to physical effects such as Brownian diffusion. Porosity and cohesion of the powder can be much higher than for materials containing larger particles with more resistance to flow and lower volume-specific surface area. Nano-objects in powdered nanostructured materials can dominate relevant properties of the bulk material by particle-particle interactions that form clusters such as agglomerates.

Aerosol release characterization consists of three main stages: generation, transport and measurement. In general, to reduce transport losses and aerosol agglomeration, the distance between generation and measurement should be minimized. Although there are potentially many different approaches[35], the generation of an aerosol is usually physically modelled on different representative scenarios (e.g. to simulate typical manual or machine powder handling processes or worst-case highly energetic dispersion).

This document is only applicable for measuring the release of nano-objects from powders. This allows comparisons of the nano-object release from different powders using the same generation and measurement system. The choice of the measurement method must take into account the characteristics (e.g. time-related dependence) of the generation system and the potential for losses and agglomeration during the transport and entry into the measuring instrumentation. Therefore, this document provides a summary of the generation and measurement methods currently available to assist material scientists and engineers in comparing the nano-object release from different powders.

The quantification of the release of nano-objects from powders described in this document cannot be used as a substitute for dustiness testing or for a health-related risk assessment.

Nanomaterials — Quantification of nano-object release from powders by generation of aerosols

WARNING — The execution of the provisions of this document should be entrusted only to appropriately qualified and experienced people, for whose use it has been produced.

1 Scope

This document describes methods for the quantification of nano-object release from powders as a result of treatment, ranging from handling to high energy dispersion, by measuring aerosols liberated after a defined aerosolization procedure. Particle number concentration and size distribution of the aerosol are measured and the mass concentration is derived. This document provides information on factors to be considered when selecting among the available methods for powder sampling and treatment procedures and specifies minimum requirements for test sample preparation, test protocol development, measuring particle release and reporting data. In order to characterize the full size range of particles generated, the measurement of nano-objects as well as agglomerates and aggregates is adressed in this document.

This document does not include the characterization of particle sizes within the powder. Tribological methods are excluded where direct mechanical friction is applied to grind or abrade the material.

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.

ISO/TS 80004-1:2015, *Nanotechnologies — Vocabulary — Part 1: Core terms*

ISO/TS 80004-2:2015, *Nanotechnologies — Vocabulary — Part 2: Nano-objects*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO/TS 80004-1:2015, ISO/TS 80004-2:2015 and the following apply.

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

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1 General terms

3.1.1
release from powder
transfer of material from a powder to a liquid or gas as a consequence of a disturbance

3.1.2
nano-object number release
n
total number of *nano-objects* (3.2.9), released from a sample as a consequence of a disturbance

3.1.3
nano-object release rate
n_t
total number of *nano-objects* (3.2.9), released per second as a consequence of a disturbance

3.1.4
mass specific nano-object number release
n_m
nano-object number release (3.1.2), divided by the mass of the sample before a disturbance

3.1.5
mass loss specific nano-object number release
$n_{\Delta m}$
nano-object number release (3.1.2), divided by the mass difference of the sample before and after a disturbance

3.1.6
nano-object aerosol number concentration
c_n
number of *nano-objects* (3.2.9) per aerosol volume unit in the sample treatment zone

3.1.7
aerosol volume flow rate
V_t
volume flow rate through the sample treatment zone

3.2 Terms related to particle properties and measurement

3.2.1
aerosol
system of solid or liquid particles suspended in gas

[SOURCE: ISO 15900:2009, 2.1]

3.2.2
equivalent spherical diameter
diameter of a sphere having the same physical properties as the particle in the measurement

Note 1 to entry: Physical properties are, for instance, the same settling velocity or electrolyte solution displacing volume or projection area under a microscope.

Note 2 to entry: The physical property to which the equivalent diameter refers shall be indicated using a suitable subscript, e.g. x_s for equivalent surface area diameter or x_v for equivalent volume diameter.

[SOURCE: ISO/TS 80004-2:2015, A.2.3]

3.2.3
particle size distribution
PSD
cumulative distribution or distribution density of a quantity of particle sizes, represented by *equivalent spherical diameters* (3.2.2) or other linear dimensions

Note 1 to entry: Quantity measures and types of distributions are defined in ISO 9276-1:1998[3].

3.2.4
$PM_{2,5}$
particulate matter smaller than 2,5 µm
mass concentration of fine particulate matter having an aerodynamic diameter less than or equal to a nominal 2,5 micrometres

Note 1 to entry: See Appendix J in Reference [47].

3.2.5
PM_{10}
particulate matter smaller than 10 µm
mass concentration of fine particulate matter having an aerodynamic diameter less than or equal to a nominal 10 micrometres

Note 1 to entry: See Appendix J in Reference [47].

Note 2 to entry: PM_{10} is used for the thoracic fraction as explained in EN 481:1993[15].

3.2.6
condensation particle counter
CPC
instrument that measures the particle number concentration of an *aerosol* (3.2.1) using a condensation effect to increase the size of the aerosolized particles

Note 1 to entry: The sizes of particles detected are usually smaller than several hundred nanometres and larger than a few nanometres.

Note 2 to entry: A CPC is one possible detector for use with a *differential electrical mobility classifier* (3.2.7).

Note 3 to entry: In some cases, a CPC may be called a "condensation nucleus counter (CNC)".

[SOURCE: ISO 15900:2020, 3.8, modified — "using a condensation effect to increase the size of the aerosolized particles" has been added to the definition.]

3.2.7
differential electrical mobility classifier
DEMC
classifier that is able to select *aerosol* (3.2.1) particles according to their electrical mobility and pass them to its exit

Note 1 to entry: A DEMC classifies aerosol particles by balancing the electrical force on each particle with its aerodynamic drag force in an electrical field. Classified particles are in a narrow range of electrical mobility determined by the operating conditions and physical dimensions of the DEMC, while they can have different sizes due to difference in the number of charges that they have.

[SOURCE: ISO 15900:2020, 3.11]

3.2.8
differential mobility analysing system
DMAS
system to measure the size distribution of sub-micrometre *aerosol* (3.2.1) particles consisting of a *differential electrical mobility classifier* (3.2.7), flow meters, a particle detector, interconnecting plumbing, a computer and suitable software

[SOURCE: ISO 15900:2020, 3.12]

3.2.9
nano-object
material with one, two or three external dimensions in the *nanoscale* (3.2.10)

Note 1 to entry: Generic term for all discrete nanoscaled objects.

[SOURCE: ISO/TS 80004-2:2015, 2.2, modified — "discrete piece of" has been deleted from the start of the definition and the Note 1 to entry has been replaced.]

3.2.10
nanoscale
size range approximately from 1 nm to 100 nm

Note 1 to entry: Properties that are not extrapolations from a larger size will typically, but not exclusively, be exhibited in this size range. For such properties, the size limits are considered approximate.

Note 2 to entry: The lower limit in this definition (approximately 1 nm) is introduced to avoid single and small groups of atoms from being designated as *nano-objects* (3.2.9) or elements of nanostructures, which could be implied by the absence of a lower limit.

[SOURCE: ISO/TS 80004-2:2015, 2.1, modified — Note 1 to entry has been replaced and Note 2 to entry has been added.]

3.2.11
agglomerate
collection of loosely bound particles or *aggregates* (3.2.12) or mixtures of the two held together by weak forces where the resulting external surface area is similar to the sum of the surface areas of the individual components

Note 1 to entry: The weak forces, for example, are van der Waals forces or simple physical entanglement.

Note 2 to entry: Agglomerates are secondary particles and the original source particles are primary particles.

[SOURCE: ISO/TS 80004-2:2015, 3.4, modified — "loosely bound particles or aggregates or mixtures of the two held together by weak forces" has replaced "weakly or medium strongly bound particles" the notes to entry have been reworded.]

3.2.12
aggregate
particle comprising strongly bonded or fused particles held together by strong forces where the resulting external surface area is significantly smaller than the sum of calculated surface areas of the individual components

Note 1 to entry: The strong forces, for example, are covalent bonds, or those resulting from sintering or complex physical entanglement.

Note 2 to entry: Aggregates are secondary particles and the original source particles are primary particles.

[SOURCE: ISO/TS 80004-2:2015, 3.5, modified — "held together by strong forces" and "calculated" have been added to the definition and the notes to entry have been reworded.]

3.2.13
dustiness
propensity of materials to produce airborne dust during handling

Note 1 to entry: For the purpose of this document, dustiness is derived from the amount of dust emitted during a standard test procedure.

Note 2 to entry: Dustiness is not an intrinsic property as it depends on how it is measured.

[SOURCE: EN 1540:2011, 2.5.1]

3.2.14
inhalable fraction
mass fraction of total airborne particles which is inhaled through the nose and mouth

Note 1 to entry: The inhalable fraction is specified in EN 481:1993[15].

[SOURCE: EN 1540:2011, 2.3.1.1]

3.2.15
thoracic fraction
mass fraction of inhaled particles penetrating beyond the larynx

Note 1 to entry: The thoracic fraction is specified in EN 481:1993[15].

[SOURCE: EN 1540:2011, 2.3.1.2]

3.2.16
respirable fraction
mass fraction of inhaled particles penetrating to the unciliated airways

Note 1 to entry: The respirable fraction is specified in EN 481:1993[15].

[SOURCE: EN 1540:2011, 2.3.1.3]

4 Symbols

For the purposes of this document, the symbols given in Table 1 apply.

Table 1 — Symbols

Symbol	Quantity	SI unit
n	nano-object number release	dimensionless
n_t	nano-object release rate	s^{-1}
c_n	nano-object aerosol number concentration	m^{-3}
n_m	mass specific nano-object number release	kg^{-1}
$n_{\Delta m}$	mass loss specific nano-object number release, from a treated sample with a mass loss Δm	kg^{-1}
V_t	aerosol volume flow rate	m^3/s^1

5 Factors influencing results of nano-object release from powders

5.1 Test generation method selection

The purpose of the planned test or experimental programme should be carefully defined during the selection of the aerosol generation method.

Selection of the aerosol generation method depends on the following considerations:

a) the powder properties listed in Table 2;

b) the applicability of standardized dustiness test methods, see the EN 15051 series[17][18][19], or of other powder treatment methods to simulate the typical powder handling process in practice[32][34][37] as well as selection of the appropriate treatment parameters.

The outcome of the planned test will be dependent on the experimental conditions selected.

EXAMPLE 1 Determination of the nano-object release of a powder to predict release of nanoparticles during manual and automatic moderate powder handling processes (i.e. weak to moderate dispersion stress) for industrial processing.

EXAMPLE 2 Estimation of nano-object and agglomerate/aggregate release from powder to simulate worst-case scenarios of handling process, where a high energy input or high activation energy is applied to the powder or during the generation of an aerosol for animal inhalation studies. Such high energy input is likely to be used only in fully contained processes to prevent unacceptable exposures to workers.

5.2 Material properties influencing nano-object release from powder

Properties influencing the generation and measurements of aerosolized powders containing nano-objects are summarized in Table 2. Presently, it is not necessarily easy to measure many of these properties; however, they should be considered.

These material-specific properties of powder are relevant to test design (see Clause 6) and data reporting (see Clause 8).

Table 2 — Representative properties influencing nano-object release from powders

Property	Description
Particle size	The value of the particle size depends on the sizing method and the corresponding equivalent diameter (e.g. aerodynamic diameter, electrical mobility diameter, equivalent area diameter). The particle size of primary particles or aggregates will not change during the handling of nanostructured powders. Particle size of agglomerates will change under certain process and handling conditions, for example, shear stress. The measured size distribution of particles will depend on the type of instrument. The instrument can measure aerodynamic or mobility diameters, specific surface areas or other parameters. The exact shape of primary particles will depend on the manufacturing process. Nano-objects can be a small fraction of the total mass for some materials.
Particle shape	Particle shapes are found in a wide range of geometries depending on the material and the process. Agglomerates and aggregates of nano-objects can have a fractal shape. Adhesion forces depend on the particle shape because of the contact area.
Crystallinity	Some powdered materials can exist in various crystalline states or in amorphous form. The fraction of the crystalline phase can vary depending on the particle size.
Hygroscopicity and moisture content	Interaction of the particle with moisture in the air characterized by the relative humidity will affect the cohesion of the particles. Thus, the history of the relative humidity of the environmental conditions used to store the powder can be important. The hydrophobic versus hydrophilic characteristics affect dustiness because as time goes on a hydrophilic nanomaterial such as magnesium oxide will become less dusty as it absorbs water from the air. Some synthetic amorphous silica, on the other hand, can be easily electrostatically charged and is readily aerosolized.
Cohesion	The magnitude of adhesion forces between particles will affect the detachment of particles as force is introduced into the system. Cohesion will affect the porosity between the particles and flowability of the powder. The tendency of the nanopowders to sinter or agglomerate is also a consideration.
Material density	The material density will affect aerosolization. For example, some tungsten oxide has a high density and is not very dusty.
Porosity	Porosity is a measure of the void spaces in a material. This includes the porosity of primary nano-objects, agglomerates and generally the packing density of the bulk powder.
Electrical resistivity	The electrical resistance of the powder affects the ability of the system to dissipate electrical charge.
Triboelectrics	The ability of the material to generate static electricity will affect the forces within the powder.

5.3 Test stages

A schematic overview of the test stages necessary for the quantification of nano-object release from powders is shown in Figure 1. Based on the multitude of factors that influence sample preparation and sample treatment and the current lack of understanding of sample treatment, this document provides requirements on the basic conditions for the aerosol measurement stage.

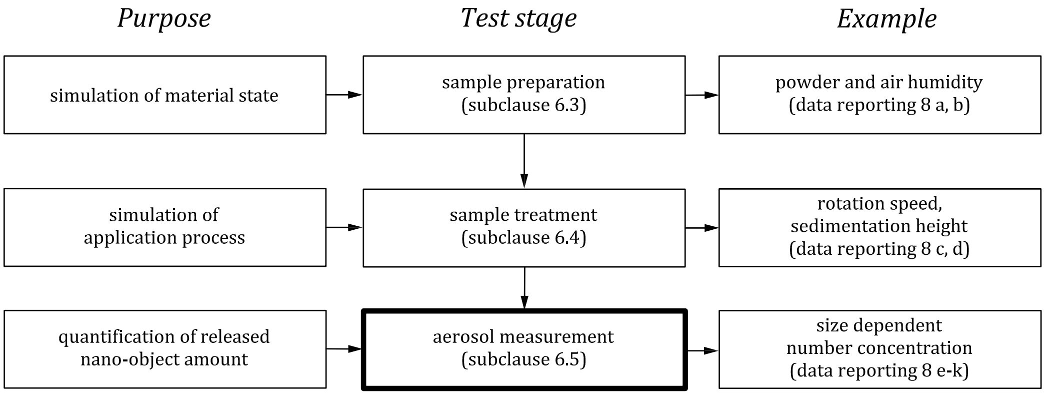

Figure 1 — Schematic overview of test stages for the quantification of nano-object release from powders

Currently, for sample treatment, no one general method can be standardized as a requirement. Nearly all powder studies suffer from incomplete determination of the energy input during sample treatment[38].

For repeatable powder treatment, four methods (rotating drum, continuous drop, small rotating drum and vortex shaker) have been standardized for dustiness measurement of powders containing nano-objects (see Annex B) and further devices are evaluated and recommended in the literature (see Annex C). Annex D adds continuous treatment in technical disagglomeration principles.

6 Test requirements

6.1 General

6.1.1 Process parameters of the sampling procedure and of the measurement procedure shall be selected with regard to the purpose of the test and to relevant material properties from Table 2.

6.1.2 The test protocol shall contain these considerations: the purpose, the procedure parameters and the relevant material properties.

6.1.3 Agreements between the buyer and the seller should include considerations of the process conditions simulated, an ability to relate to standard methods and the objectives of the study.

6.2 Safety assessment

6.2.1 A safety assessment shall be conducted for the materials before beginning the tests. Guidance is given in ISO/TR 13121:2011[4] and ISO/TR 27628:2007[13].

Some nanomaterials can be toxic. The severity of the toxicity can depend on particle composition, size, morphology and other physico-chemical properties of the material.

WARNING — A nanomaterial that is potentially explosive, pyrophoric or sensitive to ignition can present a fire or explosive hazard. Health, safety and environmental control measures shall be implemented to minimize and prevent exposure to airborne nanoparticles and spillage during loading of powders, disposal of used powders and cleaning of equipment.