TECHNICAL SPECIFICATION

ISO/TS 12025

Second edition
2021-05

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

Reference number
ISO/TS 12025:2021(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

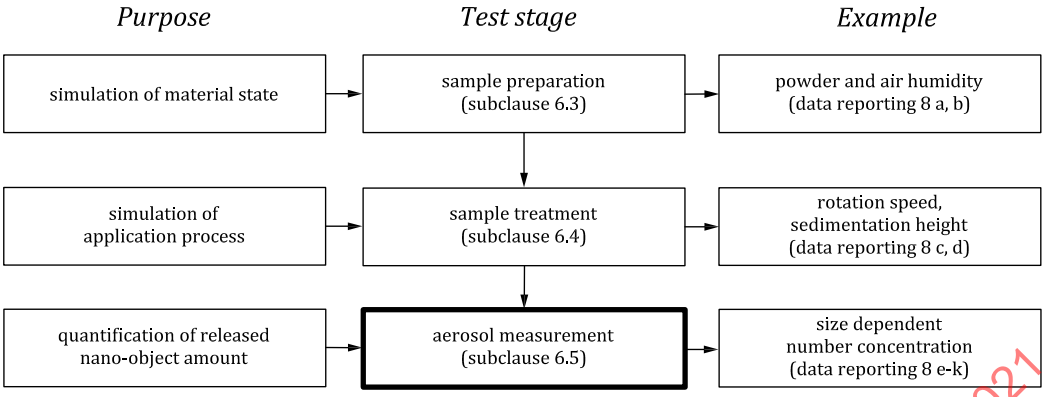


Figure 1 — Schematic overview of test stages for the quantification of nano-object release from powders

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.

6.2.2 Electrical grounding is required to prevent electrostatic charge build-up. Other safety control measures also have to be considered.

Earthing of the dispersion unit shall be carried out to avoid the risk dust explosion.

NOTE Controlled loading of transportation tubes and vessels by precipitation of particles at the beginning of the test can ensure maximum penetration or minimum particle losses.

6.2.3 The tests should be tailored according to the hazard. The following examples are not exhaustive but rather are representative.

EXAMPLE 1 Inert atmospheres are used for some materials and other control measures can be applied (e.g. electrical grounding of equipment, use of antistatic mats and shoes).

EXAMPLE 2 Toxic materials are tested under appropriate controlled conditions (e.g. glove boxes or fume cupboards) or are substituted with a non-toxic or less toxic substance. The substitute material exhibits the significant characteristics of the materials of interest. If the substitute material is tested, it is specified how the equivalence with the toxic material can be ensured.

6.2.4 Differential electrical mobility analysis for aerosol particles can require radioactive sources within the measurement device. The function of the particle charge conditioner is to establish a known size-dependent charge distribution on the sampled aerosol prior to the size classification process. This bipolar ion concentration can be produced either by radioactive ionization of air from radioactive sources or by corona discharge ionization or soft X-ray ionizers.

As stated in ISO 15900:2009[7], the use, transportation and disposal of radioisotopes are regulated by government authorities. Basic international standards and guidelines are, for example, set by commissions of the United Nations such as the International Atomic Energy Agency (IAEA), the International Commission on Radiological Protection (ICRP) and the Agreement concerning the International Carriage of Dangerous Goods by Road (ADR), etc. The licensing, shipping and disposal regulations that govern radioactive sources vary from nation to nation. This document can therefore only advise all users of radioactive material that local, national and international laws and regulations exist and shall be considered.

6.3 Sample preparation

Sample preparation procedures shall be reported, e.g. humidity conditioning of the sample and the test equipment, sample splitting, electrostatic charging and sieving for limiting the maximum agglomerate size of particles.

Guidance on powder sampling, sample splitting and minimum sample size is given in ISO 14488:2007[6]. Additional safety precautions for nanomaterials require the sampling and sample splitting in closed systems or within a fume cupboard.

6.4 Sample treatment

6.4.1 Dustiness generation methods

6.4.1.1 Selection of methods

Methods with controlled levels of applied energy selected to estimate the dustiness related to nanomaterials expected in an industrial or user setting shall be based on established practice as described in 6.4.1. Some selection criteria have been published for dustiness measurement of powders (see Annex A).

6.4.1.2 Reference test methods

The rotating drum, small rotating drum and the continuous drop methods (see Annex B) are three of the four reference test methods for determining health-related dustiness mass fraction and number-based

dustiness. These methods are described in EN 17199-2:2019[21], EN 17199-3:2019[22] and EN 17199-4:2019[23]. The vortex shaker is the fourth method described in EN 17199-5:2019[24]. Further details are given in A.1.

6.4.1.3 Dynamic method

This method uses only milligrams of powdered material per test and is completely self-enclosed. Both of these attributes are useful to evaluate nanoscale materials. The test apparatus consists of a glass chamber, with an aspiration nozzle to disperse milligram quantities of test powder into the chamber, and two samplers within the chamber to collect the dispersed dust[28]. Airflows and sampling times are controlled by the tester, which is connected to a vacuum source. The dust is dispersed by pulling the dust into the glass chamber with a short and rapid application of vacuum (see Annex C). In this test, a high energy input or high activation energy is applied to the powder. This input is adjustable by the inlet orifice. The particle concentration generated is time-dependent.

6.4.2 Dispersing methods for aerosol generation

Continuously operating powder dispersing methods have been developed for a wide range of applications, including generating dust for inhalation studies, filter testing and environmental atmospheres. A number of methods have been used and are tailored to the powder and the application. VDI 3491-3:2018[25] summarizes dispersing methods for solid materials and standardizes five technical realizations, differing in metering, dispersing and state of charge of the aerosol. ISO/TR 19601[8] describes characteristics of such aerosol generation methods, including their advantages and limitations.

An overview of disagglomeration principles is shown in Annex D. One method cannot cover the wide range of different industrial applications of powders containing nano-objects, such as nanostructured powders, and the very different flow properties of powders influencing the metering and mass flow fluctuations of the aerosol. All these methods disperse the complete powder sample with the same energy input and feed all generated aerosol particles to the measurement.

Comparative investigations of the three most widely used air jet dispersers showed the possibility of using the average air velocity in the dispersion zone as the dominating adjustment parameter for nanostructured powders[34].

6.4.3 Sample treatment execution and report

6.4.3.1 The description of the test method shall include specification of sample aerosolization and disagglomeration characteristics:

a) duration of the test and the number of repeat measurements made;

NOTE 1 In general, powder samples before testing are agglomerates (particles touching other particles). The test breaks the cohesive bonds separating particles from the agglomerates. Particles have a tendency to re-agglomerate depending on the particle density in air, the length of the test and the cohesive forces present, such as electrostatic charge. Therefore, the amount of disagglomeration and agglomeration differs with each type of test.

b) type and description of treatment of the powder;

NOTE 2 In the drop test, impact on the bottom coated or uncoated with powder will affect the results.

c) inlet design of the test method.

NOTE 3 In the dynamic method, the inlet diameter can influence the agglomerate acceleration and deceleration within the sampling chamber.

6.4.3.2 The following sample treatment parameters have an influence on the resulting particle release. They shall be kept constant throughout the tests and between tests to achieve reproducibility of the results. For comparison between different tests, they should be quantified.

a) Sample volume and residence time of the sample in the treatment zone. Both sample volume and sample mass shall be recorded. To ensure reproducibility, the volume used shall be a "tamped" volume rather than a "pour" volume. Guidance on how to determine a "tamped" volume is given in ISO 787-11[1] and for a "pour" sample volume in EN 15051-3:2013, Annex B[19].

b) Mechanical energy input to the treatment zone (e.g. air flow and pressure drop).

NOTE Research is needed on the measurement of the force or energy acting directly on the sample or agglomerates, such as local shear stress, resulting from velocity gradients or dynamic impact.

c) Humidity, temperature and ion concentration.

d) Air volume flow through the treatment zone.

e) Particle concentration in the air during treatment (interparticle distances determine the ratio of disagglomeration/agglomeration).

EN 17199-1:2019[20] specifies the conditioning of the powder. For standard testing and inter-comparison, test materials shall be conditioned at a relative humidity (RH) of (50 ± 5) % before testing until they reach a stable mass. For the characterization of the bulk material under workplace conditions, the bulk material shall be sent to the organization performing the dustiness test as placed on the market or as used by the downstream user, in air-tight containers. It shall be tested in the state in which it was received.

The temperature should be kept at a constant 21 °C ± 3 °C (EN 17199-1:2019[20]).

6.4.3.3 The test equipment should be electrically grounded.

6.4.3.4 The repeatability of the aerosolization and disagglomeration process shall be determined over 3 to 10 tests of fresh powder samples. It can be reported as minimum and maximum values in addition to the average values of the particle number release. The repeatability of the measurement shall also be reported as described in 6.5.3.

6.5 Measurement of aerosolized nano-objects

6.5.1 Selection of the measuring method

The harmonization of sampling train and choice of instruments is important to ensure comparable measurement results between generation test methods.

The following performance parameters of aerosol measurement devices define the validity and limits of nano-object release experiments: particle size range, particle concentration range, operating mode, flow rate and the mechanical stress on the measured particles before or in the measurement zone.

The four types of instrument which are available to measure nanoscale aerosols in real-time or near real-time are summarized in Table 3. As can be seen, none of the instruments measure down to 1 nm and all measure greater than 100 nm.

The CPC does not provide size information and only gives a particle number concentration in a certain size range. The other instruments can be used to monitor more specific size distributions depending on their operational capabilities.

The DMAS-type instruments rely on a single classifier so they have to scan the size range selected, which can take around a minute. It can only be used when a constant concentration of particles is generated/released (e.g. continuous drop devices or dispersing methods such as rotating brush generators). The measurement of PSDs with a DMAS is based on particle classification by the electrical mobility

diameter x_{emob}, which depends on its size and its electrical charge, but not on the density (see also E.1). The related number concentrations are measured simultaneously with the CPC in combination.

Electrometer-based mobility particle spectrometers use several classifying pathways to allocated electrometer plates for indirect number counting via electrical charge counting. This method measures electrical mobility distribution of x_{emob} much faster (up to 1 s).

An electrical low pressure impactor (ELPI) also uses allocated electrometer plates for fast measurement of the aerodynamic diameter x_{aero} after a cascade of nozzles. This density-dependent equivalent spherical diameter is relevant, for example, for inhalation exposure (see also E.2).

Table 3 — Typical performance parameters of nano-object aerosol measurement devices

Instrument	Result	Particle size nm	Particle concentration cm^{-3}	Operation mode (scan rate)	Flow rate l/min	Stress, see also Annex F
CPC	Number concentration	3 to 3 000	0 to 1×10^7	Continuous (1 s)	0,03 to 3	Aerosol deflection, capillary flow
DMAS	PSD(x_{emob})	3 to 1 000	1×10^3 to 1×10^7	Intermittent or continuous (30 s to 600 s)	0,3	Inlet-impactor, aerosol deflection
Electrometer-based mobility particle spectrometer: fast mobility particle sizer (FMPS), engine exhaust particle sizer (EEPS)	PSD(x_{emob})	6 to 1 000	1×10^4 to 1×10^8	Continuous (1 s)	10	Inlet cyclone
ELPI/ELPI+	PSD(x_{aero})	7 to 10 000	200 to 2×10^7	Continuous (1 s)	10	Aerodynamic bounce and blow off (if substrates not greased or quantity of dust collected too high)

Additional real-time measurement instruments which can be used to provide more information on the larger particle sizes are listed in Annex E.

Shear stress in the measurement zone can also break-up loose agglomerates. This is estimated in Annex F for considering the measurement of instable agglomerates, e.g. of pyrogenic nanomaterial.

6.5.2 Transport and sampling parameters

The measuring instruments are usually interfaced to the aerosol generation process by the use of sampling tubes. Each instrument will also have its own operational flow rate and inlet geometry and pre-selector (e.g. impactor or cyclone) to exclude particles above a certain size.

Transport and sampling process parameters include the following:

a) The material, length and diameter of sampling tubes. Tubes made of electrically conductive materials are essential for minimizing particle losses due to electrostatic deposition. Furthermore, diffusion, gravity and inertia can cause losses in tubes, which therefore should be as short as possible. However, instruments operate on different flow rates and losses will be different even if the length and size of the tubing is the same. Particle number concentration in the tubes should not be greater than 1 000 000 cm^{-3} to limit particle coagulation (agglomeration).

b) Sampling air flow and dilution ratio.

c) The flow rate in the tube is usually defined by the flow rate of the measurement instrument. Additional filtered dilution air can be needed to reach the required flow rate and/or to reduce the concentration of particles to the requirements of the measurement instrument.

d) Shear stress as a result of aerodynamic pre-classification (at the inlet of the measurement device). Each instrument will have its own operational flow rate and inlet geometry and pre-selector (e.g. impactor or cyclone) to exclude particles above a certain size. These can either exclude or break-up agglomerates of nanoscale particles from the measurement zone. Each instrument excludes different sizes of particles using different methods (see Table 2).

6.5.3 Considerations before testing

Background concentration shall be minimized to improve the detection sensitivity of the engineered nano-objects and nanostructured particles generated from the test. Therefore, the test equipment has to be carefully cleared of most residual particles before the beginning of the measurement by drawing particle-free air through the generator, transport and measuring devices for a sufficient time period. The background readings of the sampling train should be determined before starting the generation of particles. These sometimes need to be subtracted from the test results if the background exceeds > 1 % of any test result.

The tests shall be repeated at least three times and an average calculated.

Repeatability and reliability of aerosol measurements shall be demonstrated first by measuring test aerosols introduced to the treatment zone. Such test aerosols could be generated, for example, by the spraying and drying of solutions of different salt types. The operation of aerosol measurement equipment in field measurements by trained personnel typically has uncertainties in the nanoscale in particle size measurement of about 5 % and in particle number concentration measurement of about 20 %.

6.5.4 Size and concentration measurement results

6.5.4.1 Nano-object number release

The quantity measure “mass” of a particle size fraction depends on the third power of the particle size, i.e. if the particle size decreases by a factor of 10, the particle mass reduces by a factor of 1 000. Thus, the nano-object mass concentration can be too low to be measured alone with current commercially available instruments. Number-based methods are the most sensitive for the smallest size classes in broad PSDs. On the other hand, they count seldom particles in the largest classes with low relative reproducibility (with particle sizes above the nanoscale and therefore not relevant for this document).

The general measurand, the nano-object number release n, is given by Formula (1). If relevant, the dilution ratio has to be taken into account.

$$n = n_t \cdot t = c_n \cdot V_t \cdot t \tag{1}$$

where

n_t is the nano-object release rate;

c_n is the nano-object aerosol number concentration;

t is the measurement time;

V_t is aerosol volume flow rate through the treatment zone.

For variable release concentrations, the second term in Formula (1) can be evaluated as shown in Formula (2):

$$n = V_t \cdot \sum_i c_{n,i} \cdot \Delta t_i \tag{2}$$

where

$c_{n,i}$ is the nano-object aerosol number concentration in an increment of time;

Δt_i is the time increment (i.e. time resolution) of the instrument.

6.5.4.2 Specific nano-object number release

The specific nano-object number release is expressed in two forms, in relation to the sample.

a) The mass specific nano-object number release n_m, as given in Formula (3), expressed in number per kg:

$$n_m = \frac{n}{m} \tag{3}$$

where m is the mass of the test sample.

b) The mass loss specific nano-object number release $n_{\Delta m}$, as given by Formula (4), expressed in number per kg.

$$n_{\Delta m} = \frac{n}{\Delta m} \tag{4}$$

where

n is the nano-object number release;

Δm is the mass difference of the test sample before and after testing.

Particle size range limits and the release characterization of larger particles shall be reported.

6.5.4.3 Size range limits

6.5.4.3.1 General

The nanoscale as applied in this document shall range from 1 nm to 100 nm. It is acknowledged that the resolution of the lower limit is currently technically unfeasible. Also, at the upper limit, different measurement methods can yield different results. The experimental details shall be specified following Clause 8.

6.5.4.3.2 Lower size limit

Nano-objects (more precisely, nanoparticles) with size below about 10 nm require specific sampling and measurement device design because of diffusion losses in tubes or at the inlet of the instruments. Depending on the number-based PSD, the determined particle number concentration can be too low, and particle counters with different lower size limits can give different particle number concentrations.

For example, size measurement by a DMAS is applicable to particle size measurements ranging from approximately 1 nm to 1 µm, see ISO 15900:2009[7]. However, the lower size limit is defined as the diameter at which the instrument counts 50 % of the particles (~3 nm).

6.5.4.3.3 Upper size limit

Because of the geometric definition of nano-objects in ISO/TS 80004-2:2015, as the current best practice, the equivalent diameter measured shall be reported as well as its conversion formula to a geometric dimension of 100 nm (thickness of nanoplates or width of nanofibres) and the necessary assumptions on shape, structure and density of the real nano-objects for this calculation.

The interpretation of equivalent diameters from measurement results of other nano-objects (nanoplates and nanofibres) requires further studies to understand the shape and structure effects on particle sizing. One way of estimating these properties would be to use high resolution electron microscope characterization of the size, shape and porosity of the particles in the powder being investigated in order to identify the dimensions and morphology of the most abundant nano-objects. This information could then be used, with certain assumptions with respect to the physical principles of detection, to relate the signals from the measuring device(s) to the size of nano-objects detected.

Linking nano-object release tests to dustiness tests or other particle release tests in larger size ranges is very useful to enable better interpretation of the results, especially also of the disagglomeration treatment and applied stress. Therefore, simultaneous measurement of larger particle sizes in the sub-micrometre or micrometre size range is recommended.

However, the principle of operation of particle size instrumentation limits the range of particle sizes that can be measured. Therefore, more than one type of instrument is often used with overlapping size ranges. Depending on the material, these size distributions do not always match exactly, because different measuring principles deliver different equivalent diameters.

To calculate one diameter from another, certain material properties need to be known. The assumptions on material properties, relevant for this calculation, shall be reported, for example, the particle density, shape or refractive index.

Furthermore, device-specific effects, for example, coincidence errors and counting efficiency, can reduce the measured particle concentration at the boundaries of a measuring range.

Optical particle spectrometers and optical particle counters, covering only the upper part of the sub 100 nm size range, are standardized in ISO 21501-1:2009[9] and in ISO 21501-4:2018[10].

Laser diffraction in combination with static laser light scattering can be used to measure PSDs (not concentrations) from the nanoscale (depending on the optical properties and the absence of too many larger particles) up to hundreds of micrometres. It also allows the observation of the disagglomeration and is standardized in ISO 13320:2020[5].

6.5.5 Particle size distribution and other characteristic measurement parameters

For conversion of a released particle number into a particle volume or mass, the PSD and the particle shape, porosity and density must be known. The calculation of an effective density of agglomerated particles is very difficult, because porosity, depending on structure parameters such as fractal dimensions must be determined. One method to determine the necessary structure parameters could be a scanning electron microscopy (SEM)/transmission electron microscopy (TEM) image analysis of uncoated (not sputtered) particles, which are sampled in parallel to the aerosol measurement. Compacted apparent density can give an estimate or a proof of calculation.

Particle shape, porosity and density can also be derived from a combination of a DMAS and an aerosol particle mass analyser (centrifuge).

Based on the measured PSD, the overall volume of the released particles, V_n, can be estimated from the numbers n_i in the size classes i, using Formula (5) for summation up to the upper nanoparticle size limit j:

$$V_n = c_{\text{shape}} \cdot \sum_{0}^{j} n_i \cdot V_{\text{particle},i} = c_{\text{shape}} \cdot \sum_{0}^{j} n_i \cdot x_i^3 \tag{5}$$

where

V_n is the overall volume of released particles;

c_{shape} is the particle shape factor (independent from particle size and often only estimated);

$V_{\text{particle},i}$ is the volume of particles in each size increment;

x_i is the diameter of the particle in each size increment.

The mass of the released particles, M_n, due to the handling of the bulk powder can be estimated using Formula (6):

$$M_n = (1-\varepsilon) \cdot \rho_s \cdot V_n \tag{6}$$

where

ρ_s is the solid material density of the particles;

ε is the particle porosity, assumed to be constant as a function of size.

Formula (6) results in a mass of released nanoparticles corresponding to the mass of the handled powder bulk m. Although errors exist when converting number-based measurement data to volume or mass from error amplification resulting from cubing the particle diameter in Formula (5), this is a small part compared to the magnitudes of particle mass loss Δm considered. Remaining uncertainties shall be considered in comparison with the insensitivity of primarily particle mass determining methods, for example, cascade impactors.

Other measurements than released particle number (e.g. released surface area) can be useful for correlation to some effects of particles on health.

7 Requirements for test setups and protocols

The following basic requirements for test setups and protocols shall be regarded.

a) Experimental design should follow established methodology, see ISO/TR 22971:2005[12].

b) Sufficient safety precautions shall be implemented for the range of expected nanomaterials.

c) Accepted methods of sample splitting, such as those mentioned in ISO 14488:2007[6], should be used.

d) Test setups should match the objective of the planned studies.

e) Protocols as written procedures or records shall include:

1) specific characteristics desired from the test;

2) a plan to determine measurement uncertainty;

3) documented data reduction.

8 Test report

For a complete quantification of the nano-object release including the whole aerosol particle release situation, the following shall be reported:

a) identification of the test sample, the source and the composition of powder;

b) the powder storage method and the powder moisture content;

c) details of the testing method and the test conditions (temperature and humidity);

d) any unusual conditions during the tests;

e) raw data as an attachment;

f) the lower limit particle equivalent diameter of the aerosol measurement with regard to diffusion losses within the test setup, and the upper limit diameter regarding sedimentation and inertial deposition;

g) the measured number concentration of nano-objects (smaller than an equivalent diameter relating to a geometric dimension = 100 nm, as defined in ISO/TS 80004-2:2015, see 6.5.4.3.2) as well as the measurement time and the air volume flow rate through the treatment zone and, if appropriate, the dilution ratio for the calculation of the released nano-object number according to Formula (1);

h) the measured total number concentration in the whole particle size range of the measurement device (e.g. < 1 µm) for the calculation of the released sub-micrometre particle number concentration similar to the nano-objects concentration in g);

i) the measured total number concentration of a parallel operated CPC for the calculation of the released total particle number similar to that in g);

j) the tested sample mass in the treatment zone, to which the released nano-object number from g) can be related (see 6.5.2) and, if applicable, the sample mass loss;

k) a reference to this document, i.e. ISO/TS 12025:2021.

The following additional data should be reported:

l) the size distribution with a second method for larger particles from up to 10 µm, and additional mass based equivalent diameter distribution data if available, for example, from a cascade impactor;

m) if applicable, the estimated volume and mass of the released nano-objects in accordance with Formulae (5) and (6);

n) all assumptions regarding material properties, such as the particle density, shape or refractive index used to calculate one diameter from another in g) or to calculate the volume or mass from the diameter in l);

o) for occupational, health and safety studies: the respirable, thoracic and inhalable mass fractions according to EN 481:1993[15].

p) for environmental studies: $PM_{2,5}$ or PM_{10} as defined in 3.2.6 and 3.2.7, respectively.

From this list, the minimum data to report are g), h), i) and j) based on three repetitive tests for a defined size range.

Annex A
(informative)

Considerations for the selection of the sample treatment procedure

A.1 Dustiness reference test methods

The generation of aerosols by industrial activities can be the result of a wide range of different processes, and no single experimental method can simulate all of these. Depending on the powder and the purpose of the testing being carried out, it is necessary to select a generation method that best simulates the process being investigated. However, a wide-ranging test method may be chosen, such as the rotating drum or the continuous drop test, when the main intent of the test is for the ranking of powders in terms of dustiness allowing those which are less dusty to be selected for risk management purposes.

EN 17199-1[20] provides the choice of a dustiness test method amongst four devices: the rotating drum, the small rotating drum and the continuous drop to simulate workplace scenarios. Additionally, the vortex shaker is intended to simulate worst-case scenarios of the handling process with a high energy input to the powder.

The rotating drum, small rotating drum and the continuous drop methods are intended to simulate handling processes, which involve dropping processes of powdery bulk material at some stage. The rotating drum (large and small) and the continuous drop methods differ, however, with respect to the intensity and the duration of treatment of the bulk material. In addition, the rotating drum method drops the bulk materials a number of times, while the continuous drop method drops the bulk material once.

The small rotating drum and the vortex shaker methods require smaller amounts of bulk materials for testing (2 g to 6 g and 0,5 ml per test) compared to the rotating drum and the continuous drop methods (35 ml and 250 ml per test).

The rotating drum, small rotating drum and vortex shaker methods are methods providing time behaviour information of particle number releases. The particle concentrations generated by the rotating drum, small rotating drum and vortex shaker are time-dependent. The continuous drop method rapidly generates a constant concentration of particles after a sharp increase in particle number and the particle concentrations generated are not time-dependent.

The test methods in the EN 17199[20][21][22][23][24] series should enable a comparison of the relative dust release potential of powders.

A.2 Dispersing methods

Nanostructured powders require additional considerations because of specific powder characteristics, such as, for example, high volume specific surface area and nano-object aerosol physics. Some resulting necessary selection and evaluation criteria of aerosol generation methods are:

— adjustable volumetric flow of the air;

— adjustable particle concentration by adjusting the amount of sample introduced into the test;

— adjustable disagglomeration energy input by adjusting, for example, aerosol acceleration in a nozzle;

— independency of energy input adjustment from volumetric flow and particle concentration;

— repeated powder agitation and flow versus singular conversion of powder portions to an aerosol;

— magnitude of tribo-electric charging.

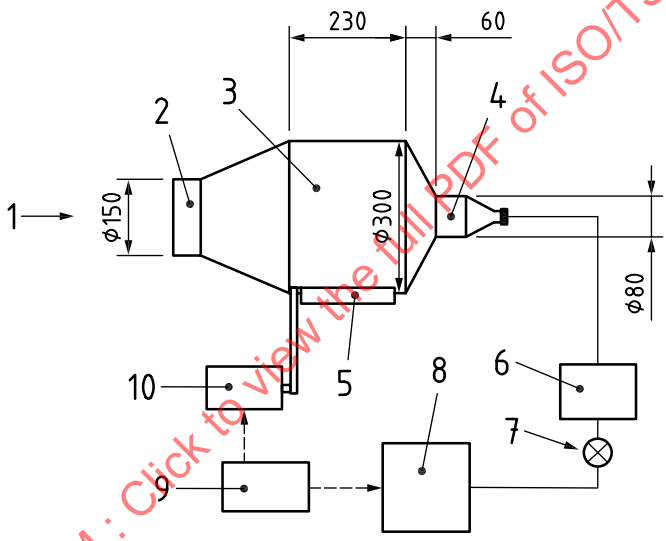
If the complete sample is aerosolized and fed to the disagglomeration zone, then the nano-object number release can be directly related to sample mass. VDI 3491-3[25] standardizes five technical realizations of dispersing methods, differing in metering, dispersing and state of charge of the aerosol. ISO/TR 19601[8] compares their advantages and limitations.

Annex B
(informative)

Dustiness reference test methods

EN 15051-2:2013+A1:2016[18] defines this method as: "The rotating drum method involves the continuous multiple dropping of a sample of the material in a slow horizontal winnowing current of air. The dust released from dropping material is conducted by the airflow to a sampling section where it is separated aerodynamically into the three health-related mass fractions (respirable, inhalable and thoracic fractions, expressed in mg/kg) by a process of horizontal elutriation and inertial impaction in two stages of porous metal foam". The amount of sample required for a test is about 35 cm^3, which should be weighed to the nearest 0,1 g. The rotating drum method is illustrated in Figure B.1.

Dimensions in millimetres



Key

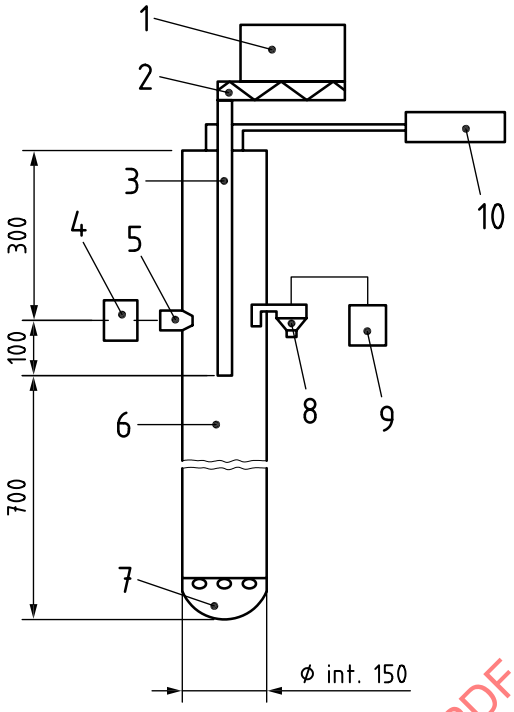
1	air flow	6	mass flow meter
2	inlet stage (protective filter)	7	control valve
3	dust generation section (rotating drum)	8	pump
4	dust collection stage (size-selective foam stages and filter)	9	timer
5	rollers	10	driven motor

NOTE Adapted from the EN 15051 series[17][18][19].

Figure B.1 — Rotating drum method

As a result of the pre-normalization work by Burdett et al.[29], it was concluded that the rotating drum method has the advantage that it can handle a very broad range of materials, including granules and flakes, and that it simulates a broad range of workplace activities. The continuous drop method is illustrated in Figure B.2.

Dimensions in millimetres



Key

1	sample tank	6	backflow pipe
2	metering device	7	collector tank
3	drop pipe	8	sampling head for the respirable aerosol fraction
4	pump for sampling the inhalable aerosol fraction	9	pump for sampling the respirable aerosol fraction
5	sampling head for the inhalable aerosol fraction	10	main flow pump

NOTE Adapted from Reference [47].

Figure B.2 — Continuous drop method

A dual dustiness characterization by rotation tests and single or continuous drop tests should provide data for a much broader range of activities than each method alone[30][43].

Schneider and Jensen[44] have shown by testing manufactured nano-, ultrafine and conventional materials in their dual single drop/rotating drum approach that the dust generation rate changes with time since the start of the rotation (agitation). Some materials release most dust in a brief initial burst. For some the release rate decreases, for others it remains stable, and for yet others it increases with time since the start of the rotation [36][43]. The existence of different types of time-dependent rates of particle release explains in part why a rotating drum and a single/continuous drop method cannot give comparable results for all materials (as also documented in EN 15051-3:2013[19]).

The methods of the EN 15051 series[17][18][19] are accepted standards for micrometre-size particles. Nano-objects and their agglomerates and aggregates (NOAA) cannot be adequately characterized by their mass fraction only.

Based on the results of pre-normative research[47], the EN 15051 series[17][18][19] establishes test methods that measure the dustiness in terms of health-related dustiness mass fraction, number-based dustiness index and number-based emission rate. It also gives guidance on the choice of a test method from four methods: the rotating drum, the continuous drop, the small rotating drum and the vortex shaker.

The continuous drop method generates a time-stable concentration and PSD and therefore allows measurement with a DMAS. All other dustiness methods require measurement methods with high time resolution of about 1 s.

Annex C
(informative)

Dynamic method

C.1 Principle

The closed system design is the most advantageous aspect of this method for tests of harmful substances.

This annex is a summary of a method reported by Boundy, Leith and Polton[28]. The test method is a two-step process:

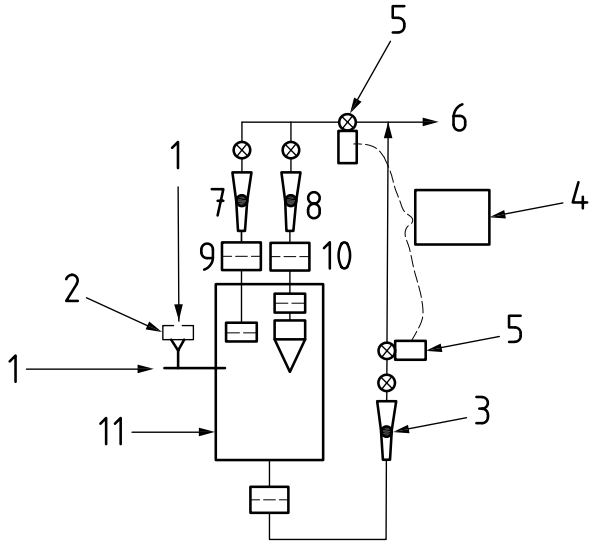
— the powder is injected with a pneumatic disperser into a chamber;

— the chamber is sampled with particle sizing equipment.

The two steps are controlled with timers and solenoid valves to control times and flow rates. The original version was developed to measure the respirable fraction of the dust. Currently, work is in progress to adapt the method to nano-object characterization.

C.2 Equipment

The equipment is shown in Figure C.1.



Key

1	air in	7	flow meter: 2,0 l/m
2	powder injector	8	flow meter: 4,2 l/m
3	flow meter: 53,8 l/m for the first 1,5 s	9	total aerosol mass
4	timer	10	respirable particles
5	solenoid valves	11	chamber: 5,7 l glass jar
6	vacuum source		

NOTE Adapted from Reference [28].

Figure C.1 — Schematic of dynamic device configured to obtain integrated respirable aerosol samples

The equipment in Reference [28] can be described as a representative approach. In practice, the equipment may be modified as long as equivalent performance can be demonstrated. Also, instrumentation to measure airborne nano-objects may be added or substituted for existing components in the existing apparatus.

The chamber is a 5,7 l glass jar selected for transparency, relatively low electrostatic charge build-up and cost. A flat aluminium plate is clamped on the mouth of the jar to provide a seal and support for aerosol sampling equipment. A hole is drilled in the side of the jar to mount a powder injector.

The powder injector consists of:

— a stainless steel tube: 10 cm long, with a 0,44 cm inside diameter and bent at a 90° angle;

— an attached funnel with a removable cap;

— a smaller secondary tube.

The funnel measures 1,0 cm in length and 1,4 cm in diameter, tapering to 0,44 cm, and is attached to one end of the primary tube. The exterior of the funnel holds an O-ring to allow a cap with a central 0,11 cm hole to seal the contents of the funnel. A secondary tube 3 cm long and with a 0,19 cm outside diameter is attached to the primary tube at the base of the right angle. The delivery end of the powder injector is inserted through an O-ring in a 5,0 mm hole centred on one side of the jar. Sampling devices are attached to the aluminium plate.

In the example given in Reference [29], a respirable mass cyclone followed by a filter cassette to collect respirable dust and either an open-face or a closed-face filter cassette to collect total suspended dust is shown. There are two exhaust lines. One is used initially at a flow of 53,8 l/min for 1,5 s and the second

is used at a lower flow to sample from the glass jar. The flow is switched by a timer (4) and two solenoid valves (5) are used to control the flow.

C.3 Procedure

The following steps are followed.

a) The equipment is prepared by cleaning and placing tared filters in the filter holders.

b) Dry powder (5,0 ± 0,1 mg) is placed in the funnel and the cap sealed.

c) The timer is activated causing the dust to be sucked into the jar for 1,5 s and then the jar is sampled for 4 min.

d) The filters are recovered and reweighed.

e) The percentage of respirable dust is determined by the ratio of the respirable dust concentration divided by the total dust suspended concentration.

NOTE If the equipment was modified with a nano-object measuring system, the amount of nano-objects would be calculated.

C.4 Discussion

In Reference [40], the method was found to have good experimental repeatability within duplicate tests for individual dusts and good repeatability between identical apparatus. However, to apply to nano-object testing the method, would need to have different instruments adapted to the method than described in this annex. These instruments can include low pressure cascade impaction, and differential mobility analysis with either electrical or condensation nuclei particle sensing.

About 25 fine and nanoscale materials were tested on a mass basis (in replicate). The tests resulted in a span of about two orders of magnitude for both total and respirable mass, see VDI 3867-5[25].

The advantages of the method are as follows:

— a small quantity of material is used, typically 5 mg;

— the test is rapid and can be conducted in a few minutes;

— the equipment is compact and could be isolated for toxic materials;

— the method is energetic and has a complete dissemination without milling the material.

Accepted methods of sample splitting should be used, see ISO 14488:2007[6].

The limitations of the method are as follows:

— Nanomaterials with very wide size distributions can contain coarse particles that can clog the dust injection system.

NOTE This situation has not occurred in many tests with coarse as well as fine dusts. However, nanomaterials with very coarse fraction need to be screened with a 200 mesh screen in preparation for the test.

— For nano-object measurement, fast responding instruments need to be employed to characterize the decaying concentration. An alternative is to use an aerosol sampler such as a low pressure impactor that will integrate over the pulse.

— It can be difficult to adjust the equipment for conditions to directly simulate industrial dust handling. The method can yield a conservative result, for example, provide a safety factor. Therefore, a

reference dust sometimes needs to be used to compare with other methods. Until now, no example for such a reference dust has been published.

Annex D
(informative)

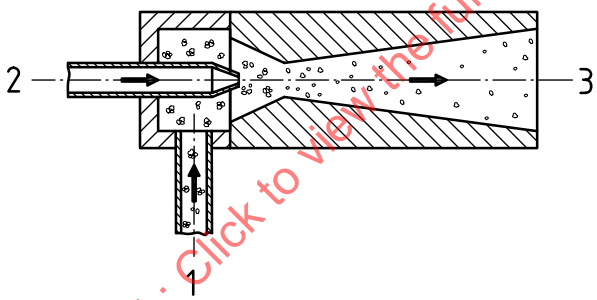
Dispersing methods

The generation of aerosols results in a specific dispersing stress on the particles. Examples are the impact of large agglomerates in a down fall pipe or in a rotating drum (see Annex B). The principles of other dispersing techniques with adjustable energy input and with a strong correlation of intensity, concentration and flow rate are described in References [30] and [41].

VDI 3491-3[25] standardizes five technical realizations of dispersing methods, differing in metering, dispersing and state of charge of the aerosol. Examples are the rotating brush generator, or metering with the aid of a segmented belt or dosage by means of bulk material properties.

ISO/TR 19601[8] compares the advantages and limitations of these five dispersing devices.

Figure D.1 represents a general schematic of an ejector nozzle, which is used with several modifications in a lot of dispersion units. Such nozzles will not tolerate a back pressure and it is difficult to add a scalping cyclone or an impactor. However, the Venturi effect creates a source of vacuum often used to introduce particles. The device in Figure D.1 operates by creating shear forces by introducing energy to the process by air flow.



Key

1 incoming flow particle-laden air
2 incoming flow clean air
3 disagglomerated particle flow

Figure D.1 — Schematic of dispersing nozzle

A modification of this schematic nozzle is standardized in ISO 5011:2020[2] as a so-called “ISO dust injector” and is used in most types of test aerosol generators in VDI 3491-3[25].

To simulate a leak, for example, in a pressurized nanoparticle production or transport line, a critical orifice can be used[46].

Release and exposure of nano-objects were connected to each other by transport and transformation processes and require, therefore, the description/specification of complex exposure scenarios. Within a 2017 published study[33], propagation modelling based on experimentally determined particle release data in accordance with this document was used for exposure estimation and prediction in a defined model room.

The measurement methodology given in 6.5 has been adopted for the determination of experimentally simulated nano-object release from paints, varnishes and pigmented plastic, see ISO 21683[11], to complete the life cycle analysis of such nanomaterials.