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

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The UK participation in its preparation was entrusted to Technical Committee NTI/1, Nanotechnologies.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Foreword

The text of ISO/TS 12025:2012 has been prepared by Technical Committee ISO/TC 229 “Nanotechnologies” of the International Organization for Standardization (ISO) and has been taken over as CEN ISO/TS 12025:2015 by Technical Committee CEN/TC 352 “Nanotechnologies” the secretariat of which is held by AFNOR.

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Endorsement notice

The text of ISO/TS 12025:2012 has been approved by CEN as CEN ISO/TS 12025:2015 without any modification.

Contents

Page

Foreword ... **iv**

Introduction ... **v**

1 Scope ... **1**

2 Normative references ... **1**

3 Terms, definitions and abbreviated terms ... **1**
3.1 General terms ... 1
3.2 Terms related to particle properties and measurement ... 2

4 Symbols ... **4**

5 Factors influencing results of nano-object release from powders ... **5**
5.1 Test method selection ... 5
5.2 Material properties influencing nano-object release from powder ... 5
5.3 Test stages ... 6

6 Test requirements ... **7**
6.1 General ... 7
6.2 Safety assessment ... 7
6.3 Sample preparation ... 8
6.4 Sample treatment ... 8
6.5 Measurement of aerosolized nano-objects ... 10

7 Requirements for test setups and protocols ... **14**

8 Data reporting ... **15**

Annex A (informative) **Considerations for the selection of the treatment procedure** ... **16**

Annex B (informative) **Rotating drum and continuous drop methods** ... **18**

Annex C (informative) **Vortex shaker method** ... **21**

Annex D (informative) **Dynamic method** ... **23**

Annex E (informative) **Disagglomeration principles** ... **26**

Annex F (informative) **Selection of the nano-object measuring method** ... **27**

Bibliography ... **30**

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.

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ISO/TS 12025 was prepared by Technical Committee ISO/TC 229, *Nanotechnologies*.

Introduction

The emissions or release of nano-objects into the surrounding air from powdered nanostructured materials resulting from handling is an important consideration in the design and operation of many industrial processes. Released nano-objects may affect human health and the environment, depending on the nature and quanitity of the nanomaterial. It is therefore important to obtain data about the propensity of nanomaterials to release nano-objects, thereby allowing exposure to be evaluated, controlled and minimised.

Three main target groups of experts for the evaluation of the release of nano-objects from powdered nanostructured materials are:

— material scientists and engineers, who design safe nanomaterials and safe nanomaterial handling processes;

— occupational, health and safety specialists;

— environmental specialists, who need exposure data in addition to toxicity data for risk assessment of manufactured nanomaterials (see A.2) and who collect dustiness data (gravimetric as well as particle concentration and particle size information).

The propensity of nanomaterials to release nano-objects into the air is determined by test methods devised to apply energy to a sample to stress the intra-particle bonds. This stressing induces abrasion, erosion or comminution, which causes dissemination of the particles into the gaseous phase, i.e. generation of aerosols allowing quantification with aerosol instrumentation.

Methods to measure the release of nano-objects from nanomaterials may include dustiness testing methods but basic differences from conventional dustiness methods should be considered. The high variability of the flow properties of powders and the influence of the test setup should also be considered. Conventional dustiness methods for micrometre size particles estimate the amount of dust generated in terms of dust mass fraction or dustiness indices. The methods of aerosol generation for the determination of the dustiness of powders containing primary particles of less than 10 µm in diameter have been found to produce very dissimilar results.

There are a large number of possible combinations of different approaches for the design of dustiness methods[1]. The only current standard, EN 15051:2006[2], selected two methods: the rotating drum method and continuous drop method. The measured values are the inhalable, thoracic or respirable mass fractions, expressed in mg/kg.

Definitions of the inhalable, thoracic and respirable fractions can be found in EN 481[3]. Aerodynamic diameters of 100 µm, 10 µm and 4 µm are the upper limits of the corresponding size fractions. These mass fractions, which are relevant for inhalation, can be added as measurands in measurement of aerosolised nano-objects to characterize the complete particle release scenario.

Schneider and Jensen[4] described approaches using particle size distributions by number to relate exposure from nano-objects in the indoor environment to source strengths resulting from the release of nano-objects during the handling of nanostructured powders. They concluded that dustiness testing combined with online size distribution measurements provides insight into the state of agglomeration of particles released during handling of bulk powder materials.

Furthermore, the evaluation of the release of nano-objects from powdered nanostructured materials requires additional methods and measurands compared to the methods assessing the dustiness of powders. Particle number concentration and size distribution are other measurands necessary for quantifying the release of nano-objects.

Aerosols of nano-objects 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 those containing larger particles with more resistance to flow and lower volume-specific surface area. Nano-objects in powdered materials can dominate relevant properties of the bulk material by particle-particle interactions that form clusters like agglomerates. There is still a lack of understanding

in the characterization of these secondary nanostructured particles, consisting of primary nano-objects. It has been shown for fumed silica, as an example, that the resulting aerosol particle size distribution depends strongly upon the conditions involved in the different measuring methods[5][6].

Aerosols and powders are also generated by tribological abrasive tests[7] of nano-composites and paints containing nanoparticles[8][9]. Such abrasion tests are not addressed by this Technical Specification. However, the measurement methodology of these publications has been proven for the quantification of nano-object release from wear powders by generation of aerosols.

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 Technical Specification provides methodology 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. In addition to information in terms of mass, the aerosol is characterized for particle concentrations and size distributions. This Technical Specification provides information on factors to be considered when selecting from 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 recommended in this Technical Specification.

This Technical Specification 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 referenced documents are indispensable for the application of this document. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO/TS 27687:2008, *Nanotechnologies — Terminology and definitions for nano-objects — nanoparticle, nanofibre and nanoplate*

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

3 Terms, definitions and abbreviated terms

For the purposes of this document, the terms and definitions given in ISO/TS 27687 and ISO/TS 80004-1 and the following apply.

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, released from a sample as a consequence of a disturbance

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

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

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

3.1.6
nano-object aerosol number concentration
c_n
number of nano-objects 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

[ISO 15900:2009, definition 2.1]

3.2.2
intraparticle porosity
ratio of the volume of open pores internal to the particle to the total volume occupied by the solid

[ISO 15901-1:2005, definition 3.9]

3.2.3
interparticle porosity
ratio of the volume of space between particles in a powder to the apparent volume of the particles or powder

[ISO 15901-1:2005, definition 3.10]

3.2.4
equivalent spherical diameter
diameter of a sphere that produces a response by a given particle-sizing instrument, that is equivalent to the response produced by the particle being measured

NOTE 1 The physical property to which the equivalent diameter refers is indicated using a suitable subscript (ISO 9276-1:1998).

NOTE 2 For discrete-particle-counting, light-scattering instruments, the equivalent optical diameter is used.

NOTE 3 For inertial instruments, the aerodynamic diameter is used. Aerodynamic diameter is the diameter of a sphere of density 1 000 kg m^{-3} that has the same settling velocity as the irregular particle.

NOTE 4 [ISO/TS 27687:2008, definition A.3.3]

3.2.5
particle size distribution
PSD
cumulative distribution or distribution density of a quantity of particle sizes, represented by equivalent spherical diameters or other linear dimensions

NOTE Quantity measures and types of distributions are defined in ISO 9276-1:1998.

3.2.6
particulate matter smaller 2,5 µm
PM2,5
mass concentration of fine particulate matter having an aerodynamic diameter less than or equal to a nominal 2.5 micrometres ($PM_{2,5}$)

NOTE See Reference [10].

3.2.7
particulate matter smaller 10 µm
PM10
mass concentration of fine particulate matter having an aerodynamic diameter less than or equal to a nominal 10 micrometres (PM_{10})

NOTE 1 See Reference [11].

NOTE 2 PM10 is used for the thoracic fraction as explained in EN 481:1993.

3.2.8
condensation particle counter
CPC
instrument that measures the particle number concentration of an aerosol using a condensation effect to increase the size of the aerosolised particles

NOTE 1 The sizes of particles detected are usually smaller than several hundred nanometres and larger than a few nanometres.

NOTE 2 A CPC is one possible detector for use with a DEMC.

NOTE 3 In some cases, a condensation particle counter may be called a condensation nucleus counter (CNC).

NOTE 4 Adapted from ISO 15900:2009, definition 2.5.

3.2.9
differential electrical mobility classifier
DEMC
classifier that is able to select aerosol particles according to their electrical mobility and pass them to its exit

NOTE 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.

3.2.10
differential mobility analysing system
DMAS
system to measure the size distribution of sub-micrometre aerosol particles consisting of a DEMC, flow meters, a particle detector, interconnecting plumbing, a computer and suitable software

NOTE [ISO 15900:2009, definition 2.8]

3.2.11
nano-object
material with one, two or three external dimensions in the nanoscale

NOTE 1 Generic term for all discrete nanoscale objects.

NOTE 2 [ISO/TS 27687:2008, definition 2.2]

3.2.12
nanoscale
size range from approximately 1 nm to 100 nm

NOTE 1 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 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 or elements of nanostructures, which might be implied by the absence of a lower limit.

NOTE 3 [ISO/TS 27687:2008, definition 2.1]

3.2.13
agglomerate
collection of loosely bound particles or aggregates 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 The weak forces, for example, are van der Waals forces or simple physical entanglement.

NOTE 2 Agglomerates are secondary particles and the original source particles are primary particles.

NOTE 3 Adapted from ISO/TS 27687:2008, definition 3.2.

3.2.14
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 The strong forces, for example, are covalent bonds, or those resulting from sintering or complex physical entanglement.

NOTE 2 Aggregates are secondary particles and the original source particles are primary particles.

NOTE 3 Adapted from ISO/TS 27687:2008, definition 3.3.

3.2.15
dustiness
propensity of materials to produce airborne dust during handling

NOTE 1 For the purposes of this document, dustiness is derived from the amount of dust emitted during a standard test procedure.

NOTE 2 [EN 15051:2006, definition 3.4]

4 Symbols

For the purposes of this document, the following symbols apply:

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 method selection

The purpose of the planned test or experimental program should be carefully defined during selection.

Selection of the test method depends on the following considerations:

a) powder properties listed in Table 1;

b) applicability of standardized dustiness test methods[2] or of other powder treatment methods to simulate the typical powder handling process in practice 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 and of the dustiness of a powder to predict release of particles during handling in typical industrial processes.

EXAMPLE 2 Estimation of nano-object and agglomerate/aggregate release from powder during very high energy testing.

5.2 Material properties influencing nano-object release from powder

Properties influencing generation and measurements of aerosolized powders containing nano-objects are summarized in Table 1. Presently, many of these properties might not be easily measured, however, they should be considered.

Table 1 — Representative properties influencing nano-object release from powders

Property	Description
Particle size	Fundamental property. 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. Therefore it may behave like a process parameter. The measured size distribution of particles will depend on the type of instrument. The instrument might 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 may 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 may have a fractal shape. Adhesion forces depend on the particle shape because of the contact geometry.
Crystallinity	Some powdered materials can exist in various crystalline states or in amorphous form. The fraction of the crystalline phase may vary depending on the particle size.
Hygroscopicity	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 may 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, for example, 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 flow ability 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.

These material-specific properties of powder are considered in the test design in Clause 6 or in the data reporting in Clause 8, respectively.

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 Technical Specification provides normative content on basic conditions for the aerosol measurement stage.

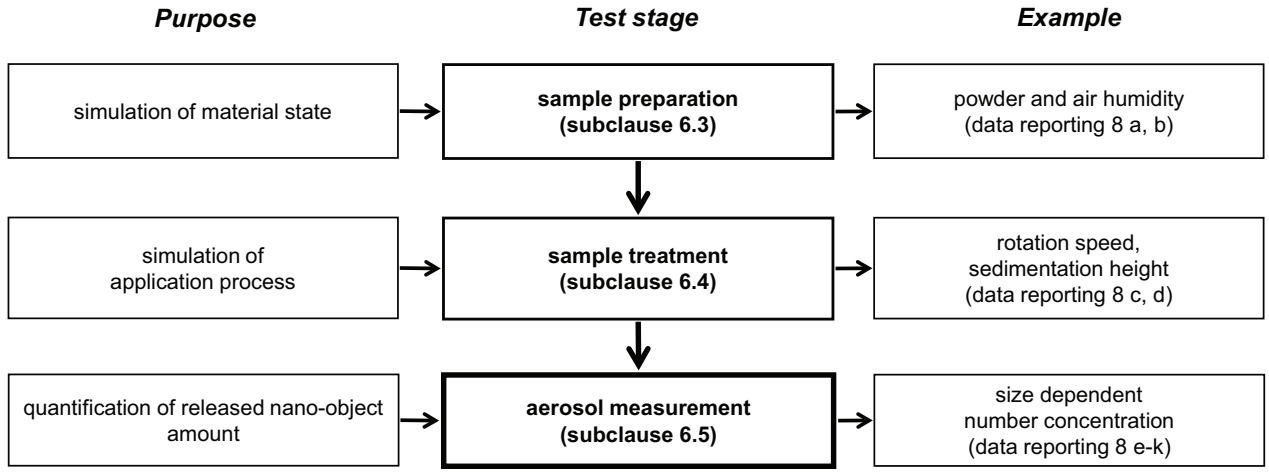


Figure 1 — Schematic overview of test stages for the quantification of nano-object release from powders

Currently, for sample treatment no one general method can be normatively standardized. Nearly all powder studies suffer from incomplete determination of the energy input during sample treatment[12]. For repeatable powder treatment, two devices have been standardized for dustiness measurement (see Annex B) and further devices are tested and recommended in literature (see Annexes C and D). Annex E 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 1.

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 buyer and seller should include considerations of the process conditions simulated, 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[47] and ISO/TR 27628[13].

NOTE 1 Some nanomaterials might be toxic. The severity of the toxicity might depend on particle composition, size, morphology and other physico-chemical properties of the material.

NOTE 2 A nanomaterial that is potentially explosive, pyrophoric or sensitive to ignition might present a fire or explosive hazard.

6.2.2 Electrical earthing shall be considered to prevent electrostatic charge build-up.

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 may need to be used for some materials and other control measures to be applied (e.g. electrical earthing of equipment; use of antistatic mats and shoes).

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

6.2.4 Differential electrical mobility analysis for aerosol particles may 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. ISO 15900 points out that 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 like IAEA, ICRP, ADR, etc. The licensing, shipping and disposal regulations that govern radioactive sources vary from nation to nation. This Technical Specification can therefore only advise all users of radioactive material that local, national and international laws and regulations should be considered and followed.

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 maximum agglomerate size of particles.

Guidance on powder sampling, sample splitting and minimum sample size is given in ISO 14488. 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 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 the established practice described in 6.4.1. Some selection criteria were published for dustiness measurement of micrometre sized powders (see Annex A).

6.4.1.2 Reference test methods

The rotating drum method (see Annex B) is one of the two reference test methods for determining a dustiness index described in EN 15051:2006. The dust is generated by a multiple continuous dropping process of powder at low speed and is intended to simulate general handling processes, which involve continuous dropping processes.

The continuous drop reference method (see Annex B) intends to simulate dust generation processes where there are continuous falling operations (e.g. conveying, discharging, filling) and where dust is liberated by winnowing during falling. It differs from the rotating drum method in that the bulk material is dropped only once, but continuously.

6.4.1.3 Vortex shaker method

In the vortex shaker method (see Annex C) a powder is placed in a glass test tube and agitated using a laboratory vortex shaker while a continuous flow of gas (typically air) is supplied to the tube. The particles released from the powder are carried out of the tube by the air flow and delivered to measurement instruments that determine size and/or concentration of the released particles. This technique does not require a large

amount of sample for testing. Typically the amount of sample is in the range from a few milligrams to a few hundreds of milligrams. The method needs to be checked for the specific powder for stability.

6.4.1.4 Dynamic method

This method utilizes 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. 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 D).

6.4.2 Dispersion methods

Powder dispersion 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 dust and the application.

An overview of disagglomeration principles is shown in Annex E. One method cannot cover the wide range of different industrial applications of powders containing nano-objects, like nanostructured powders, and the very different flow properties of powders.

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 test and number of runs;

NOTE 1 In a rotating drum or vortex shaker, agglomeration of fractal powders can occur because of repeated powder disturbance.

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 may 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, 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 'tap' volume rather than a 'pour' volume. Guidance on how to obtain a constant sample volume is given in EN 15051:2006, C.3.

b) Mechanical energy input to the treatment zone (air flow pressure drop or other energy input).

NOTE 1 Research is needed on the measurement of the force or energy acting directly on the sample or agglomerates, like local shear stress, resulting from velocity gradients or dynamic impact.

c) Humidity, temperature and ion concentration.

NOTE 2 All material is to be tested under the same humidity-controlled environment. The dustiness of a powder tested under a 50 % humidity atmosphere will be different from the dustiness of the same powder tested under a 30 % or 80 % humidity atmosphere. Humidity inside the test equipment should be 50 % ± 10 %.

NOTE 3 Temperature also should be kept constant 21 °C ± 3 °C.

d) Air volume flow through the treatment zone.

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

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 three to 10 tests of fresh powder samples and 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 6.6.6.

6.5 Measurement of aerosolized nano-objects

6.5.1 Transport and sampling parameters

Transport and sampling process parameters are:

a) material, length and diameter of sampling tubes;

NOTE Tubes made of electrically conductive materials are recommended for minimizing particle losses due to electrostatic deposition. Furthermore diffusion, gravity and inertia can cause losses in tubes, which should therefore 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.

b) sampling air flow, dilution ratio;

c) shear stress as a result of aerodynamic pre-classification (at the inlet of the measurement device);

d) shear stress within a measurement device (for instance of aerodynamic sample focusing);

e) particle number concentration in the tubes should not be greater than 1 000 000 cm^{-3} to limit particle coagulation (agglomeration).

6.5.2 Size and concentration measurement results

6.5.2.1 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 may 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 particle size distributions.

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.

$$n = n_t \cdot t = c_n \cdot V_t \cdot t \quad (1)$$

where

n_t is the nano-object release rate;

c_n is the measured nano-objects number concentration;

t is the measurement time;

V_t is the air volume flow rate through the treatment zone.

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

$$n = V_{\mathrm{t}} \cdot \sum_{i} c_{\mathrm{n},i} \cdot \Delta t_i \quad (2)$$

where

$c_{n,i}$ is the measured nano-objects in an increment of time;

Δt_i is the time increment.

6.5.2.2 Specific nano-object number release

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

a) mass specific release n_{m} is given in Formula (3), expressed in number per kg

$$n_m = \frac{n}{m} \quad (3)$$

where m is the mass of the test sample;

or

b) mass loss specific release $n_{\Delta \mathrm{m}}$ given by Formula (4), expressed in number per kg

$$n_{\Delta m} = \frac{n}{\Delta m} \quad (4)$$

where

n is 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.

6.5.2.3 Size range limits

The nanoscale as applied in this Technical Specification 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 may yield different results. The experimental details shall be specified in accordance with Clause 8.

6.5.2.3.1 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 particle size distribution, the determined particle number concentration might be too low, and particle counters with different lower size limit may give different particle number concentrations.

Size measurement by differential electrical mobility analysis for aerosol particles is applicable to particle size measurements ranging from approximately 1 nm to 1 µm (ISO 15900). The lower size limit is defined as the diameter at which the instrument counts 50 % of the particles.

6.5.2.3.2 Upper size limit

Differential electrical mobility analysis for aerosol particles covers the particle size range from 3 nm to 1 µm. Truncation of the number-based particle size distribution at 100 nm mobility equivalent diameter and summation of the contents of the smaller size classes delivers the nano-particle release number by calculation with the particle concentration and aerosol flow rate.

Because of the geometric definition of nano-objects in ISO/TS 27687 as the current best practice, the equivalent diameter measured shall be reported as well as its conversion equation 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) actually requires further studies to understand 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 with 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 may not 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, e.g. 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 standardised in ISO 21501-1[14] and in ISO 21501-4[15].

Laser diffraction in combination with static laser light scattering can be used to measures particle size distributions (not concentrations) from the nanoscale (depending on the optical properties and the absence of too many larger particles) up to hundreds of micrometres. It allows also the observation of the disagglomeration and is standardised in ISO 13320[16].

6.5.3 Particle size distribution and other characteristic measurement parameters

For conversion of a released particle number into a particle volume or mass, the particle size distribution and the particle shape, porosity and density must be known. The calculation of an effective density of agglomerated particles is very difficult because structure parameters like fractal dimensions resulting in porosity must be determined. One method to determine the necessary structure parameters could be an SEM/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.

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

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

where

V_n is the overall volume of released particles;

c_{shape} is the particle shape factor;

$V_{particle,i}$ 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 powder bulk can be calculated using Formula (6) estimating the density of the particles estimated from the particle porosity ε and the solid material density:

$$M_n = \varepsilon \cdot \rho_s \cdot V_n \quad (6)$$

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.

Some important characteristic measurement parameters to consider are:

a) Formula (6) results in a mass of released nano-particles 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 factor compared to the magnitudes of particle mass loss Δm considered. Remaining uncertainties shall be considered in comparison to the insensitivity of primarily particle mass determining methods, e.g. cascade impactors.

b) Measurements other than released particle number (e.g. released surface area) might be useful for correlation to some particle effects like health mechanisms.

Further important characteristic measurement parameters to consider are:

— Variations in the generated particle number concentration and particle size in short duration scale. [For scanning measurement methods like DMAS, it is important that the aerosol concentration under study is stable over the scan time (0,5 min to 3 min) and that the maximum particle number concentration to be measured does not exceed 100 000 cm^{-3}. Typically, a useable size distribution of a sample at a concentration of approximately 10 000 cm^{-3} can be obtained once every three minutes. Averaging of five to 10 scans can often be used to yield data for moderately unstable or lower concentration samples.]

— Particle size resolved concentration distribution. (In principle, size distributions may be integrated to obtain a measurement of total concentration. However, the comparability of this measurement to a direct measurement using a CPC and other DMAS instruments is often poor. Therefore parallel measurement for correction of the integrated particle number of DMAS by CPC result is recommended here.)

— Concentration of sub-micrometre and micrometre particles > 100 nm up to 1 µm or 2,5 µm or 10 µm or 30 µm (e.g. PM10 measurement may be compared to the thoracic fraction obtained from conventional dustiness testing or workplace measurements).

— Measurements other than particle number concentration, such as total particle surface area concentration. (These may be useful for toxicologists for correlation to potential health effects of nano-objects. Total particle surface area concentration can be calculated from number-based particle size distribution and the particle number concentration or can be measured also by electrostatic methods directly. In both cases, particle shape or morphology shall be taken into account via an appropriate correction factor.)

— Background concentration. (It shall be minimized to improve the detection sensitivity of the engineered nano-objects and nano-structured 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 device for a sufficient time period.)

6.5.4 Selection of the measuring method

The following performance parameters of aerosol measurement devices define 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. Mechanical stress can occur if the instrument has regions of high speed flow that may de-agglomerate the particles.

Further considerations for the selection of the measuring method, like time resolution down to one second for non-stationary release processes and other performance parameters of actual aerosol measurement devices, are listed in Annex F. These considerations may require the use of electrometer-based spectrometers instead of the differential mobility analysing system to ensure the necessary time resolution.

Annex F distinguishes in two tables between number-based nano-object aerosol measurement devices and other aerosol measurement devices that complement performance.

6.5.5 Repeatability and uncertainties

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

Repeatability can be reported as the coefficient of variation in the size classes. The number of repetitions shall be also reported. Because of the variability in measurements at the smaller size scales, uncertainties should be reported for particles in the following size < 10 nm; 10 nm to 25 nm; 25 nm to 50 nm; 50 nm to 75 nm; and 75 nm to 100 nm, 100 nm to 300 nm, 300 nm to 1 000 nm. Comparable log scale binning with five classes from 10 nm to 100 nm can also be used. Especially high diffusion losses require specific sampling and measurement device design in the size range smaller than 10 nm.

7 Requirements for test setups and protocols

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

a) Experimental design should follow established methodology (see ISO/TR 22971).

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

c) Accepted methods of sample splitting, such as those given in ISO 14488, 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) plan to determine measurement uncertainty;

3) documented data reduction.

8 Data reporting

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

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

b) the powder storage method and powder and air humidity;

c) details of the testing method;

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 diameter or dimension = 100 nm, as defined in ISO/TS 27687:2008, 6.6.3.1) as well as the measurement time and the air volume flow rate through the treatment zone and, if appropriate, 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. the < 1 µm) for the calculation of the released sub-micrometre particle number similar to g);

i) the measured total number concentration of a parallel operated CPC for the calculation of the released total particle number similar to 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 ISO/TS 12025:2012.

8.2 The following additional data should be reported:

a) 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 e.g. from cascade impactor;

b) if applicable, the calculated volume and mass of the released nano-objects according to Formulae (5) and (6);

c) 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);

d) for occupational, health and safety studies: the respirable, thoracic and inhalable mass fractions according to EN 481; for environmental studies: PM2.5 or PM10 as defined in 3.2.6 or 3.2.7;

e) 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 treatment procedure

A.1 General

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 purpose of the testing being carried out, it is necessary to select a generation method that best simulates the process being investigated.

The suitability of a given dustiness test method to simulate actual activities has been proposed by Higman as quoted in Petavratzi et al.[17] and is shown in Table A.1. Furthermore Gill et al.[18] describe a broad range of test methods.

Table A.1 — Suitability of a given test method to simulate actual activities[17]

	Continuous drop	Rotating drum	Fluidized bed[a]
Dustiness ranking	G	G	G
Effectiveness of dedusting	R	G	G
Effects of attrition	U	R	R
Dust generation in:			
Screening — classification	R	R	G
Mixing, granulation, coating	U	G	R
Pneumatic drying, conveying	U	G	G
Loading–unloading	G	R	R
Mechanical conveying — elevating	R	G	R
Bagging	G	R	G
Vechicle movement	G	G	G
Losses from stockpiles	R	R	G

G Good results can be expected

U Unsatisfactory results

R Reasonable results should be obtained with care

[a] Vortex shaker method might be considered.

Nanostructured powders require additional considerations because of specific powder characteristics, e.g. 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 e.g. 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 dissaglomeration zone, then the nano-object number release can be directly related to sample mass. Such methods are currently under development[19][20].

A.2 Application of methods

Nano-object work place exposure evaluation protocols and a data summary are summarized in Reference [12]. Nearly all powder studies summarized in Reference [22] suffer from incomplete determination of the energy input during sample treatment[23][24][25][26][27][28][29]. Some powder drop tests were affected by the material properties, e.g. flowability, themselves. Furthermore repeated treatment of powders in rotating drum or fluidized bed can generate secondary agglomerates. In this case the number of released particles rapidly decreases during the test[28][29]. Fast measuring devices may help to avoid this problem[23].

The single-drop and the rotating drum part of the dustiness test using 6 g of test material gave very reproducible results both in terms of amount and size distribution of the generated particles[21]. A single drop of 6 g powder released a total particle number between 10 nm and 10 µm of $1{,}3 \cdot 10^7$ from fumed silica and $20{,}6 \cdot 10^7$ from ultrafine titania.

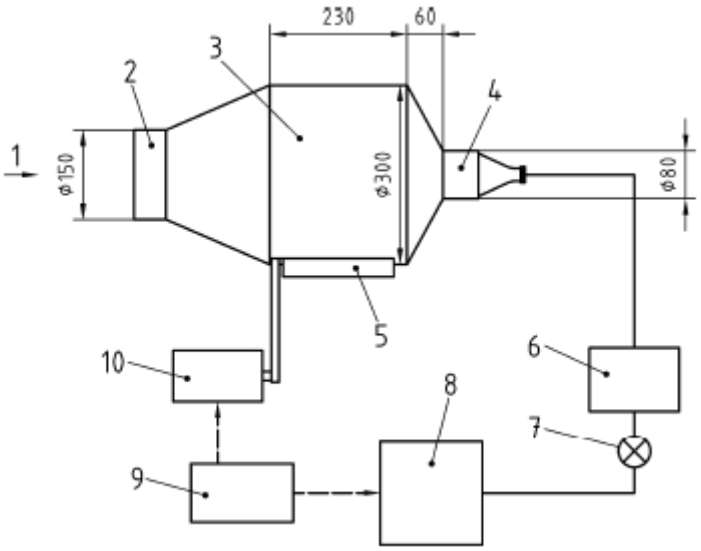
Annex B
(informative)

Rotating drum and continuous drop methods

EN 15051 defines this method: "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 mass health-related 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.

In EN 15051:2006, Annex D, "Reference bulk materials", seven materials covering the expected range of dustiness have been chosen for the equivalence test. These materials have been characterized using both reference test methods, shown in Figures B.1 and B.2 respectively. The dustiness classifications obtained using the standard reference test methods are given in EN 15051:2006, Tables D.1 and D.2.

Dimensions in millimetres



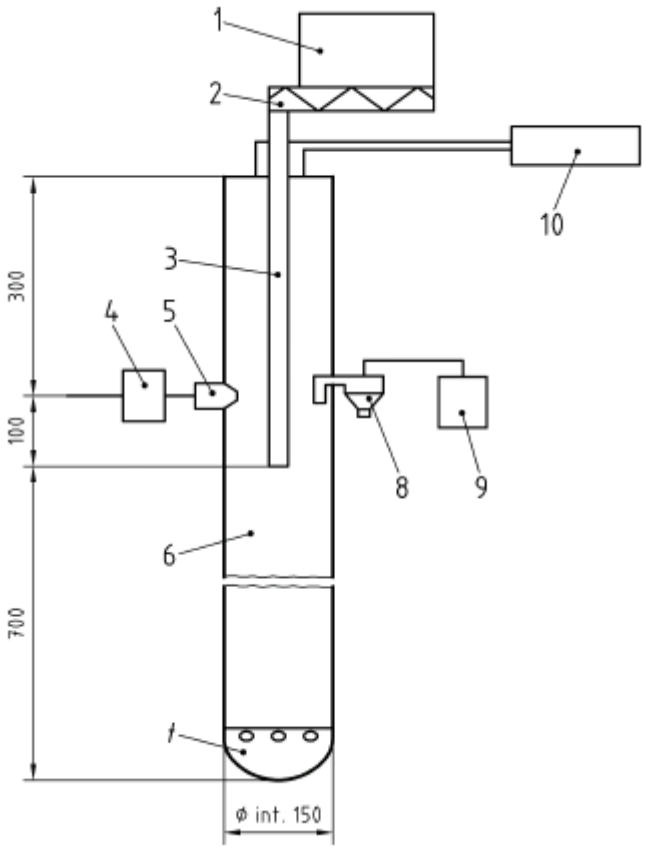
Key

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

Figure B.1 — Rotating drum method, adapted from EN 15051

As a result of the pre-normalization work by Burdett et al.[30] 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.

Dimensions in millimetres



Key

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

Figure B.2 — Continuous drop method, adapted from EN 15051

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[31][17].

Schneider and Jensen[23] 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 start of 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 start of rotation[17][21]. The existence of different types of time dependent rate of release of particles

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).

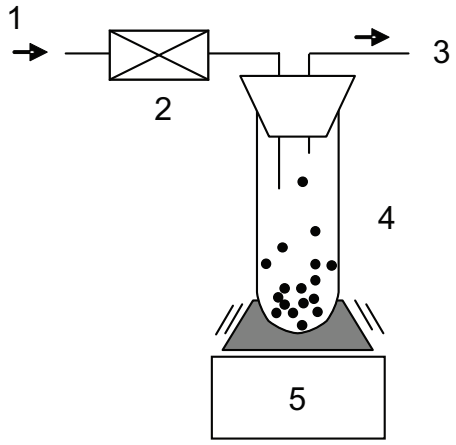
Annex C
(informative)

Vortex shaker method

This annex describes the method that utilizes a vortex shaker[32][33] for applying energy to the sample powder. In a typical setup, approximately 1 cm^3 (or less) of sample powder is placed in a glass test tube (e.g. a tube having an outer diameter of 25 mm and a length of 20 cm[33]) and agitated using a laboratory vortex shaker, with passing an air flow (e.g. Five $L \cdot min^{-1}$[33]) through the test tube (Figure C.1). The powder may be mixed with or without beads. Aerosol particles released continuously during the agitation are measured by aerosol measurement instruments (such as a differential mobility analysing system, DMAS, an aerodynamic spectrometer, and an optical particle counter) and/or collected by aerosol samplers (such as a filtration-based sampler and a cascade impactor).

Because of the motion of the shaker, the sample powder moves up along the wall and then falls down while it forms a steady eddy in the test tube. This causes collisions between powder particles by themselves and also between power particles and the inner surface of the test tube, making it a mock process involving dropping and collision. In addition, since an air flow is supplied, it can be considered to partially reflect the aerosolization caused by a stream of air.

The advantages of this method are that it is easy, simple, and compact, is constructed from generic lab items, and can continuously generate particles (several tens of minutes or more) with a small amount (approximately 1 cm^3 or less) of sample powder. In addition, it is advantageous that, after a test, the sample tested can easily be replaced with the next sample which is in another glass test tube, and be stored as it is in the glass test tube. This is especially useful for handling toxic or expensive materials.



Key

1 air in
2 HEPA filter
3 aerosol outlet to instruments
4 test tube
5 vortex shaker

Figure C.1 — Schematic of a typical setup of the vortex shaker method, adapted from Reference [33]

Other examples use a mixing impeller[31][34] instead of the shaker. One can adjust the disagglomeration intensity, concentration and flow rate, which would be beneficial in a sense that one disperser can simulate various disagglomeration/wear processes of different disagglomeration intensities. Such

powder mixing devices can generate in high porosity powders like carbon black, fumed titania, alumina or silica, large agglomerates by powder shearing like in the rotating drum.

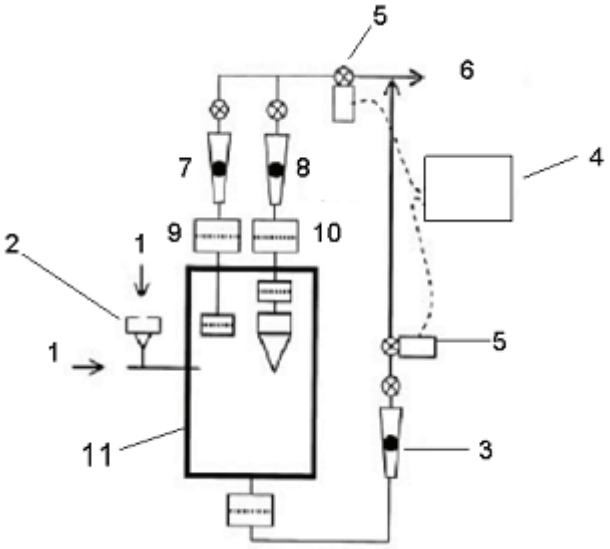
Annex D
(informative)

Dynamic method

D.1 Principle

This annex is a summary of a method reported by Boundy, Leith and Polton[19]. The test method is a two step process: 1) the powder is injected with a pneumatic disperser into a chamber and 2) 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.

D.2 Equipment



Key

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

Figure D.1 — Schematic of dynamic device configured to obtain integrated respirable aerosol samples, adapted from Reference [19]

The equipment in Reference [19] 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, 0,44 cm inside diameter bent at a 90° angle, an attached funnel with a removable cap and a smaller secondary tube. The funnel measures 1,0 cm in length, 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 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 attached to the aluminium plate.

In the example in Reference [19], 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 $Lmin^{-1}$ 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.

D.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.

D.4 Discussion

In Reference [35] 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 might include low pressure cascade impaction, 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[36].

The advantages of the method are

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

NOTE Accepted methods of sample splitting should be used [ISO 14488].

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

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

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

Limitations of the method are as follows:

— Nanomaterials with very wide size distributions might contain coarse particles that might 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.

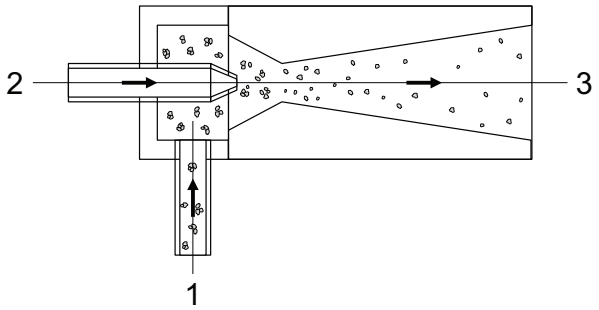
— It might be difficult to adjust the equipment for conditions to directly simulate industrial dust handling. The method might yield a conservative result e.g. provide a safety factor. Therefore a reference dust might need to be used to compare to other methods. Until now no example for such a reference dust has been published.

Annex E
(informative)

Disagglomeration principles

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 [31] and [34].

Figure E.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 E.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 E.1 — Schematic of dispersing nozzle

Another approach is to apply the energy acoustically to disagglomerate the powder[37][38][39]. The acoustic energy level can be controlled to obtain desired aerosol characteristics.

To simulate a leak i.e. in a pressurized nanoparticle production or transport line, a critical orifice can be used[40].

Annex F
(informative)

Selection of the nano-object measuring method

F.1 Methods and performance parameters of actual aerosol measurement devices

Aerosol measurement methods for nano-object measurement and for larger concomitant particles are explained in 6.5.3. The selection of the measurement method depends on performance parameters, which can be found in Tables F.1 and F.2 for actual aerosol measurement devices. For instance shear stress occurs at aerodynamic classification or focusing zones and may change the agglomerate size to be measured significantly.

Aerosol measurement devices for nano-object measurement and for larger particles can be subdivided in condensation particle counter, differential mobility analysing system, electrometer based mobility particle spectrometer, light scattering aerosol spectrometer, optical particle counter, aerodynamic spectrometer and measurement devices for mass concentration of larger specified aerodynamic diameters (e.g. PM2.5, PM10).

These measurement methods are described in detail in References [41] and [42].

Tables F.1 and F.2 add practical performance parameters by an additional column with estimated stress conditions on the particles during the measurement.

Table F.1 — Performance parameters of nano-object aerosol measurement devices

Instrument	Result	Particle size	Particle concentration	Operation mode	Flow rate	Stress
[-]	[-]	[nm]	[cm^{-3}]	[-]	[l/min]	[-]
Condensation particle counter CPC	Number concentration	3- 3 000	$0 – 1 \times 10^7$	Continuous (1 s)	0,03 – 3	Aerosol deflection, capillary flow
Differential mobility analysing system DMAS	PSD	3 –1 000	$1 \times 10^3 – 1 \times 10^7$	Intermittent (30 s – 600 s)	0,3	Inlet-impactor, aerosol deflection
Electrometer based mobility particle spectrometer	PSD	6 –1000	$1 \times 10^4 – 1 \times 10^8$	Continuous (1 s)	10	Cyclone

Table F.2 — Performance complementing other aerosol measurement devices

Instrument	Result	Particle size	Particle concentration	Operation mode	Flow rate	Stress
[-]	[-]	[nm]	[cm^{-3}]	[-]	[l/min]	[-]
Light scattering aerosol spectrometer (high sensitivity)	PSD	90 – 20000 (60 – 1 000)	$0,01 – 1 \times 10^5$ ($0 – 1,8 \times 10^4$)	Continuous	0,01 – 0,1	Aerodynamic focusing
Optical particle counter	Number concentration in few classes	50 – 10 000	$0 – 1 \times 10^5$	Continuous	0,03 – 3	Aerodynamic focusing

Table F.2 *(continued)*

Instrument	Result	Particle size	Particle concentration	Operation mode	Flow rate	Stress
[-]	[-]	[nm]	[cm^{-3}]	[-]	[l/min]	[-]
Aerodynamic spectrometer	PSD	370 – 20 000	0,001 – 1 × 10^4	Continuous (1 s – 18 s)	1,0 – 5,0	Aerodynamic acceleration
Electrometer based low-pressure cascade impactor	PSD, charge concentration	7 – 10 000	n.d.	Continous (5s -)	10, 30	Aerodynamic, bounce, blow off
Filtration based collection device	Mass concentration smaller than specified aerodynamic diameters (e.g. PM2,5, PM10)	< 30 000	n.d.	Non-continuous	10 – 1 × 10^{-3}	Aerosol deflection

Basic components of the DMAS are the focus of standardization in ISO/TC24, SC4, ISO 15900 and ISO 27891. A precondition for accurate measurement with DMAS is a time of 30 s with constant particle concentration and size distribution for polydispersed size distribution (3 min for monodispersed).

For dynamic methods, up to 1 s time resolution can be achieved with an electrometer based mobility particle spectrometer, consisting of electrical mobility classification and simultaneous concentration measurement with several electrometers. A minimum volumetric flow rate of 10 $l \cdot min^{-1}$ at the inlet is required, which could disagglomerate nanostructured particles after a low energy aerosolization and disagglomeration procedure before.

An electrometer based low-pressure cascade impactor or micro-orifice cascade impactor would apply highest shear stress by aerodynamic classification in small orifices. A further issue when using cascade impactors for unstable agglomerate measurement is bounce and blow-off at the collection stage. A tacky or an oil covered surface of the impaction stages can reduce the blow-off effect.

To check if the unknown stress in the measuring device distorts the test result, the stress in the aerosolization and disagglomeration procedure should be varied in small steps. If the change in stress results in a change in particle size and concentration, it may be assumed that the stress in the measurement device is not the dominant factor disturbing the measurements.

F.2 Influence of instrument measuring principle on equivalent diameter

The measured value of particle size differs when the measurement method is different. Aerodynamic diameter is the most relevant equivalent particle diameter for inhalation exposure in the size range above approximately 100 nm. Therefore, it is preferred that sizes are measured by aerodynamic methods or are converted to aerodynamic diameter when sizes are obtained in other forms such as mobility- or optical-equivalent diameters.

NOTE Aerodynamic diameter is the diameter of a sphere of density 1000 kg m^{-3} that has the same settling velocity as the irregular particle. Electrical mobility equivalent diameter can be calculated from the migration velocity dependent on the strength of the electrical field, the mechanical mobility and the number of charges per particle. Optical equivalent diameter can be determined from the scattered light intensity of an instrument, calibrated e.g. with polystyrene spheres.

For example, a polystyrene sphere of 100 nm diameter with a density of about 1000 kg m^{-3} represents the upper limit for the nanoparticle definition as well as for the ultrafine particle definition that addresses aerodynamic or mobility equivalent diameters. Porous particles have smaller equivalent optical and aerodynamic diameters than non-porous particles. Non-porous particles with higher density than polystyrene have larger aerodynamic equivalent diameters.

However, size measurement by or conversion to aerodynamic diameter is not always possible due to the limitation of available instruments or lack of information for conversion, especially at sizes below 100 nm. In addition, mobility-equivalent diameter is a more relevant size in the size range of less than approximately 100 nm because thermal deposition due to Brownian motion of particles is a more dominant deposition mechanism in the respiratory system in this size range. Therefore, the reported size does not have to be aerodynamic. The interpretation of equivalent diameters from measurement results of nanoplates and nanofibres actually requires further studies to consider extreme shape and structure influences.

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