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

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



Reference number  
ISO/TS 12025:2012(E)

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## 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of document:

- an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;
- an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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.

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 quantity 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<sup>[1]</sup>. The only current standard, EN 15051:2006<sup>[2]</sup>, 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<sup>[3]</sup>. 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<sup>[4]</sup> 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<sup>[5][6]</sup>.

Aerosols and powders are also generated by tribological abrasive tests<sup>[7]</sup> of nano-composites and paints containing nanoparticles<sup>[8][9]</sup>. 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