SLOVENSKI STANDARD
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[Not translated]

Workplace exposure - Measurement of dustiness of bulk materials that contain or release nano-objects or submicrometer particles - Part 4: Small rotating drum method

Exposition am Arbeitsplatz - Messung des Staubungsverhaltens von Schüttgütern, die Nanoobjekte oder Submikrometerpartikel enthalten oder freisetzen - Teil 4: Verfahren mit kleiner rotierender Trommel

Exposition sur les lieux de travail - Mesurage du pouvoir de resuspension des matériaux en vrac contenant des nano-objets et leurs agrégats et agglomérats - Partie 4: Méthode impliquant l'utilisation d'un petit tambour rotatif

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

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European foreword

This document (prEN 17199-4:2017) has been prepared by Technical Committee CEN/TC 137 “Assessment of workplace exposure to chemical and biological agents”, the secretariat of which is held by DIN.

This document is currently submitted to the CEN Enquiry.

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

Introduction

Dustiness characteristics in terms of mass-based respirable dustiness levels, particle number generation rate and size distribution provide users (e.g. manufacturers, producers, occupational hygienists and workers) information on the ability of the powder to release fine dust during handling and its dust and dustiness characteristics [4], [5], [6]. Whereas the respirable dustiness index corresponds to the measure used in most of the current exposure limits, the integrated number- and size-distribution information as well as the kinetics of dust release can be of great help to the manufacturers of bulk materials to improve their products and users to select the most appropriate material considering their use and risk management measures. [7] gives an example on how a suite of dustiness data can applied for selection of most suitable material in a production.

This European Standard gives details of the design and operation of the small rotating drum test method that characterizes the respirable dustiness of solid bulk materials including nanomaterials in terms of particle number, particle size distribution, dustiness kinetics, particle morphology and chemical composition.

The small rotating drum method has been designed to represent general bulk material handling processes, including processes where bulk material is tipped, poured, mixed, scooped, dropped or similar; either mechanical or by hand.

The small rotating drum method presented here differs from the rotating drum method, the continuous drop method and the vortex shaker method presented in prEN 17199-2:2017 [1], prEN 17199-3:2017 [2] and prEN 17199-5:2017 [3], respectively. The rotating drum and small rotating drum perform, both, repeated pouring or agitation of the same sample nanomaterial. The continuous drop method simulates continuous feed of a nanomaterial while the vortex shaker method simulates vigorous agitation of a nanomaterial.

This European Standard was developed based on the results of pre-normative research [8]. This project investigated the dustiness of ten bulk materials including nine bulk nanomaterials with the intention to test as wide a range of bulk nanomaterials as possible in terms of magnitude of dustiness, chemical composition and primary particle size-distribution as indicated by a high range in specific surface area Subsequently, the sampling line was optimized to improve dust transmission to avoid significant losses in the system and make the sampling closer to the efficiency in the prototype by [4] and EN 15051-2 [9].

1 Scope

This European Standard provides the methodology for measuring and characterizing the dustiness of bulk materials that contain or release nano-objects or submicrometer particles, under standard and reproducible conditions and specifies for that purpose the small rotating drum method.

In addition, this European Standard specifies the selection of instruments and devices and the procedures for calculating and presenting the results. It also gives guidelines on the evaluation and reporting of the data.

The methodology described in this European Standard enables

a) the measurement of the respirable dustiness mass fraction,

b) the measurement of the number-based dustiness index of respirable particles in the size range from about 10 nm to 1 000 nm,

c) the measurement of the number-based size distribution of the released aerosol in the size range from about 10 nm to 10 µm,

d) the quantification of the initial dustiness emission rate and the time to reach 50 % of the total particle number released during testing, and

e) the characterization of the aerosol from its particle size distribution and the morphology and chemical composition of its particles.

This European Standard is applicable to the testing of a wide range of bulk materials including powders, granules or pellets containing or releasing nano-objects or submicrometer particles in either unbound, bound uncoated and coated forms.

NOTE 1 Currently no number based classification scheme in terms of particle number and emission rate has been established for powder dustiness. Eventually, when a large number of measurement data has been obtained, the intention is to revise the European Standard and to introduce such a classification scheme, if applicable.

NOTE 2 The small rotating drum method has been applied to test the dustiness of a range of materials including nanoparticle oxides, nanoflakes, organoclays, clays, carbon black, graphite, carbon nanotubes, organic pigments, and pharmaceutical active ingredients. The method has thereby been proven to enable testing of a many different materials that can contain nanomaterials as the main component.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 481, *Workplace atmospheres - Size fraction definitions for measurement of airborne particles*

EN 1540, *Workplace exposure - Terminology*

EN 13205-2, *Workplace exposure - Assessment of sampler performance for measurement of airborne particle concentrations - Part 2: Laboratory performance test based on determination of sampling efficiency*

EN 15051-1, *Workplace exposure - Measurement of the dustiness of bulk materials - Part 1: Requirements and choice of test methods* prEN 17199-1:2017, *Workplace exposure - Measurement of dustiness of bulk materials that contain or release nano-objects or submicrometer particles - Part 1: Requirements and choice of test methods*

EN 16897, *Workplace exposure - Characterization of ultrafine aerosols/nanoaerosols - Determination of number concentration using condensation particle counters*

ISO 15767, *Workplace atmospheres - Controlling and characterizing uncertainty in weighing collected aerosols*

ISO 27891, *Aerosol particle number concentration - Calibration of condensation particle counters*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 1540, EN 15051-1 and prEN 17199-1:2017 apply.

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

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp

4 Symbols and abbreviations

CPC	Condensation Particle Counter
d_{50}	a lower particle size at which the counting or sampling efficiency is 50 %
ELPI® [1)]	Electrical Low Pressure Impactor
EM	Electron Microscopy
FTIR	Fourier Transform Infra-Red Spectroscopy
GC	Gas Chromatography
HEPA	High Efficiency Particulate Arrestance
HPLC	High Performance Liquid Chromatography
ICP	Inductive Coupled Plasma
ID	Inner Diameter
LOQ	Limit Of Quantification
MS	Mass Spectrometry
PGR	Particle number Generation Rate
Raman	Raman Spectroscopy

1) ELPI® is the trade name or trademark of a product supplied by Dekati. This information is given for the convenience of users of this European Standard and does not constitute an endorsement by CEN of the product named. Equivalent products may be used if they can be shown to lead to the same results.

RH	Relative Humidity
SEM	Scanning Electron Microscopy
SRD	Small Rotating Drum
TEM	Transmission Electron Microscopy
XRF	X-ray Fluorescence

5 Principle

The small rotating drum method described in this European Standard measures the dustiness of bulk materials containing or releasing nano-objects in terms of

— respirable mass fraction,

— number-based dustiness indices,

— number-based emission rates, and

— time period to generate 50 % of the emitted particle numbers.

In addition, this European Standard describes the procedures by which the aerosols can be further characterized in terms of their particle size distributions and the morphology and chemical composition of their airborne particles.

The sampling for the purpose of and the execution of qualitative or quantitative analysis of the morphology and chemical composition of the collected airborne nanostructured particles are described. Performing these analyses is optional but can provide confirmation of the sizes of the particles generated and complementary information to the real-time instruments.

Table 1 provides

— an overview of the different measurands,

— information on whether determining these measurands is mandatory or not, and

— the aerosol instruments and sampling devices needed to determine a measurand.

Table 1 — Measurands, aerosol instruments/sampling devices and associated recommendations for the small rotating drum method

Measurand	Method/Device specific to measurand	Mandatory/optional
Mass-based respirable dustiness index (mg/kg)	Inline sampling of dust on PTFE or PVC filter using a pre-separator cyclone living up to sampling convention for respirable dust.	mandatory
		optional Filters may be analysed chemically after weighing using e.g., XRF, ICP-MS, GC-MS, HPLC-MS, FTIR, and Raman spectrometry depending on needs and suitability of the sample.
Number-based dustiness index of respirable particles in the size range from about 10 nm to 1 000 nm (1/mg)	Condensation Particle Counter (CPC) – alcohol-based	mandatory
Number-based average emission rate of respirable particles in the size range from about 10 nm to 1 000 nm (1/mg.s)	Condensation Particle Counter (CPC) – alcohol-based	mandatory
Number-based initial dustiness kinetics considering the number of particles released in the size range from about 10 nm and 1 000 nm ($1/s^2$)	Condensation Particle Counter (CPC) – alcohol-based	mandatory
Time-based dustiness kinetics assessed as the time required to generate 50 % of the total number of particles released in the size-range from about 10 nm to 1 000 nm (s)	Condensation Particle Counter (CPC) – alcohol-based	mandatory
Number of modes of the time-averaged particle number-based size distribution as $dN/d\log D_p$	Direct reading number-based electrical low pressure cascade impactor measuring particles from about 10 nm to 10 000 nm	mandatory
Equivalent modal diameters of the time-averaged particle number-based size distribution as $dN/d\log D_p$		mandatory
Number of modes of the time-averaged mass-based size distribution as $dM/d\log D_p$	Cascade impactors	optional
Equivalent modal diameters of the time-averaged mass-based size distribution as $dM/d\log D_p$		optional
Morphology characterization of the nano-objects, the particles, agglomerates and aggregates	E.g., a TEM-grid holder equipped with porous carbon film TEM-grid	optional Particles on TEM-grids may be analysed by transmission (TEM) or scanning (SEM) electron microscopy

6 Equipment

6.1 General

Figure 1 gives a schematic example for a small rotating drum set-up which is configured with a bypass tube to bypass the test atmosphere while preparing and cleaning the small rotating drum.

6.2 Test apparatus

The usual laboratory apparatus and, in particular, the following.

6.2.1 Small rotating drum, consisting of the components described in detail in 6.2.2 to 6.2.12.

The small drum consists of a cylindrical part with a radius of 8,15 cm and a length of 23 cm and two 45° truncated conical ends with a centre depth of 6,3 cm (see Figure 1). These dimensions give a total volume of about 5,675 l. The cylindrical part of the drum contains three powder lifter vanes (2 cm × 22,5 cm) placed 120° apart. The inner surfaces shall be polished to reach an arithmetical mean roughness profile of 0,19 µm, which can be obtained by vibratory finishing. The drum is rotated driven by a cogwheel belt connected to a programmable electrical engine.

The volume-flow-balance shall be as follows:

— Q_E is 10 l/min to the ELPI®;

— $Q_A = Q_{B1} + Q_{B2} + Q_C$;

— $Q_C + Q_D = Q_E$ = 10 l/min.

If measurement systems and sampling systems with different volume flows are applied, the entire sampling train shall be redesigned to allow isokinetic (or at least near-isokinetic) sampling. See also 6.2.4. Mass flow controllers should be used to ensure a stable volume flow of humidified air to deliver Q_A and Q_D.