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SLOVENSKI STANDARD
SIST EN 17289-3:2021

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Karakterizacija razsutih materialov - Določanje velikostno utežene fine frakcije in deleža kristaliničnega kremena - 3. del: Metoda sedimentacije

Characterization of bulk materials - Determination of a size-weighted fine fraction and crystalline silica content - Part 3: Sedimentation method

Charakterisierung von Schüttgütern - Bestimmung einer größengewichteten Feinfraktion und des Anteils an kristallinem Quarz - Teil 3: Sedimentationsverfahren

Caractérisation des matériaux en vrac - Détermination de la fraction fine pondérée par taille et de la teneur en silice cristalline - Partie 3 : Méthode par sédimentation

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13.040.30	Kakovost zraka na delovnem mestu	Workplace atmospheres

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Characterization of bulk materials - Determination of a size-weighted fine fraction and crystalline silica content - Part 3: Sedimentation method

Caractérisation des matériaux en vrac - Détermination de la fraction fine pondérée par taille et de la teneur en silice cristalline - Partie 3 : Méthode par sédimentation

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EN 17289-3:2020 (E)

European foreword

This document (EN 17289-3:2020) 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 European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 2021, and conflicting national standards shall be withdrawn at the latest by June 2021.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN shall not be held responsible for identifying any or all such patent rights.

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Introduction

A method was developed in the industrial minerals industry for the purpose of determining the "size-weighted relevant fine fraction" within the bulk material. This document sets out the methods which can be used to measure and calculate the fine fraction of the bulk material and the fine fraction of the crystalline silica, in several types of bulk materials. This information provides additional information to users for their risk assessment and to compare bulk materials. It has been used in the industry and by institutes previously under the acronym SWeRF. EN 17289 (all parts) is based on that industrial method and specifies the analytical methods to determine the difference between materials with coarse quartz and fine quartz, e.g. sands versus flour.

As further activities with the material (intentional or otherwise) can change the particle size distribution, the size-weighted fine fraction can also change. Therefore, the method reports (in terms of the mass fraction in the bulk material in percent) both, the total crystalline silica (CS) and the estimated size-weighted fine fraction of CS.

Conventions as specified in EN 481 can be used as input for this document. However, the output of this document is not related to the respirable fraction at the workplace and cannot be used to replace workplace exposure measurements.

EN 17289 (all parts) specifies two procedures that can be used to estimate the size-weighted fine fraction (SWFF) in bulk materials. It also specifies how the SWFF, once separated, can be further analysed to measure the content of crystalline silica (SWFFCS). The method can be used for comparing the fine fraction in different bulk samples. EN 17289 (all parts) uses the term fine fraction to indicate that it does not analyse airborne particles, but it evaluates the proportion of particles in a bulk material that, based on their particle size, have a potential to be respirable if they were to become airborne.

EN 17289 (all parts) also allows for the size-weighted fine fraction of crystalline silica (SWFFCS) particles in bulk materials to be evaluated in terms of mass fraction in percent, if the fraction separated is subsequently analysed by a suitable method.

In a comparison of similar bulk materials, in which the particle size distribution is the only variable, the SWFF can provide useful information to guide material selection. For example, leaving all other factors aside, a bulk material with a lower SWFF value can pose less of a risk in terms of potential occupational exposure. For the actual exposure at the workplace, the handling etc. of the material, will play a major role.

Concentrations of respirable dust, or respirable crystalline silica (RCS), in the workplace air, resulting from processing and handling of bulk materials, will depend on a wide variety of factors and these concentrations cannot be estimated using SWFF or SWFFCS values. SWFF and SWFFCS values are not intended for workplace exposure assessments as they have no direct relationship with occupational exposure.

The evaluation of bulk materials using SWFF is complementary to determining the dustiness according to EN 15051-1 [1].

The difference between EN 17289 (all parts) and EN 15051-1 is that SWFF quantifies the fine fraction in a bulk material while dustiness quantifies the respirable, thoracic and inhalable dust made airborne from the bulk material after a specific activity (dustiness characterizes the material with relation to the workplace atmosphere when working with the bulk material).

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EN 17289 *Characterization of bulk materials — Determination of a size-weighted fine fraction and crystalline silica content* consists of the following parts:

— *Part 1: General information and choice of test methods;*

— *Part 2: Calculation method;*

— *Part 3: Sedimentation method.*

NOTE   This document is intended for use by laboratory experts who are familiar with FT-IR, XRD methods, PSD measurements and other analytical procedures. It is not the intention of this document to provide instruction in the fundamental analytical techniques.

1 Scope

This document specifies the determination of the size-weighted fine fraction (SWFF) and the size-weighted fine fraction of crystalline silica (SWFFCS) in bulk materials by means of a sedimentation method using a liquid sedimentation technique.

The purpose of this document is to allow users to evaluate bulk materials with regard to their size-weighted fine fraction and crystalline silica content.

NOTE   For preparation of the sample and determination of crystalline silica by X-ray Powder Diffractometry (XRD) or Fourier Transform Infrared Spectroscopy (FT-IR) see EN 17289-1.

Specific methods for the evaluation of SWFF for specific bulk materials are specified in several annexes.

This document is applicable for crystalline silica containing bulk materials which have been fully investigated and validated for the evaluation of the size-weighted fine fraction and crystalline silica.

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 17289-1, *Characterization of bulk materials — Determination of a size-weighted fine fraction and crystalline silica content — Part 1: General information and choice of test methods*

EN 17289-2:2020, *Characterization of bulk materials — Determination of a size-weighted fine fraction and crystalline silica content — Part 2: Calculation method*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 1540 and EN 17289-1 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

CS   crystalline silica

PSD   particle size distribution

SWFF   size-weighted fine fraction

SWFFCS   size-weighted fine fraction of crystalline silica

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5 Assumptions

The sedimentation method is based on the following assumptions:

a) all liquid that is extracted from the sedimentation vessel only comes from the space above the extraction nozzle, and not from the space below the extraction nozzle. If this is not the case this will result in a higher SWFF.

b) the particle size distribution of the bulk material is constant over the particle size range of interest, i.e. from 1 μm to 12 μm (aerodynamic).

A constant particle size distribution is a PSD with a function that has a derivative which is constant, see Formula (1). It is considered constant when classes of the same size contain the same fraction of the total amount.

$$f(d) = \frac{(d - d_{\min})}{(d_{\max} - d_{\min})} \times 100 \tag{1}$$
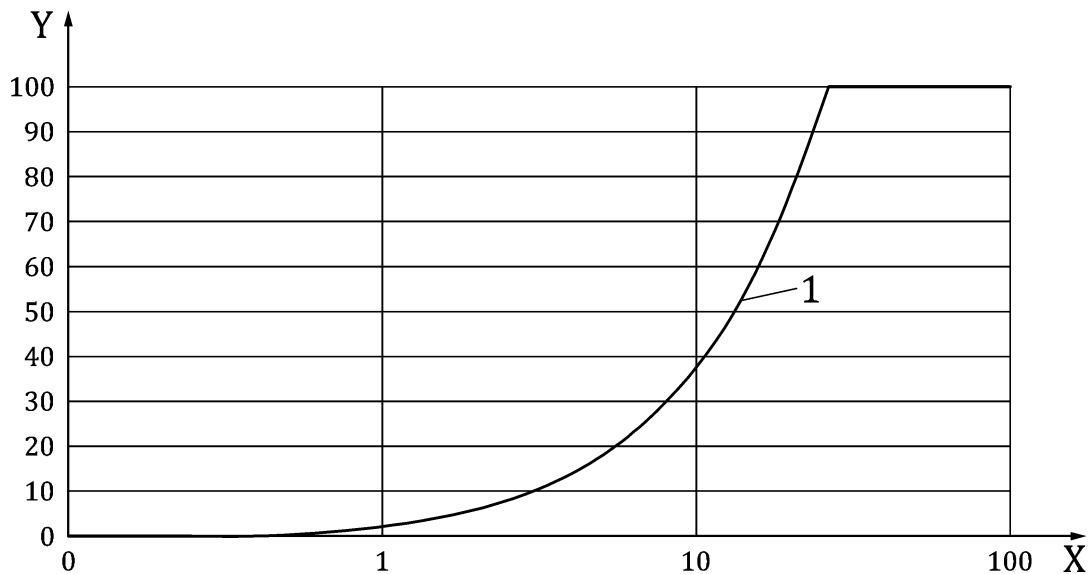
where

$f(d)$ is the cumulative particle size distribution;

d is the particle size, in micrometres (μm);

$d_{\min}$ is the minimum particle size, in micrometres (μm);

$d_{\max}$ is the maximum particle size, in micrometres (μm).

NOTE $d_{\min} \leq d \leq d_{\max}$. See Figure 1 for an example.

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Key

X   particle diameter (μm)

Y   fraction (%)

1   constant cumulative particle size distribution

Figure 1 — Example of a constant cumulative PSD with d_{min} = 0,5 μm and d_{max} = 25 μm

c)   the particle size distribution of the sample does not have a narrow size distribution (relative span $\left(\left[d_{90} - d_{10}\right] / \sqrt{d_{90} \times d_{10}}\right)$ < 1,5) and at the same time, have a $d_{ae,50}$ in the range from 2 μm to 12 μm. Otherwise the error made could be more than 1 % (absolute value).For a relative span less than 1,5 μm and $d_{ae,50}$ in the range from 2 μm to 6 μm, sedimentation overestimates the SWFF.

For a relative span less than 1,5 μm and $d_{ae,50}$ in the range from 6 μm to 12 μm SWFFCS is underestimated. It is therefore recommended in these cases to use the calculation method instead to determine SWFF. The mean reason for determining the SWFF using sedimentation is because, although the PSD of the whole sample is known, the PSD of the crystallin silica is not. The CS can either be in the coarse part of the PSD or in the fine part. However, in the case of a narrow distribution it is likely to assume the CS practically has the same PSD as the whole sample. Since the PSD is so narrow, sample and CS particles are automatically close to each other.

d)   Stokes law is only valid for particles settling in a medium at low Reynolds number. The velocity of a particle settling in a medium is limited by the drag force and this depends on the Reynolds number for that particle. Although the density of liquids is much higher, so is their viscosity so that in the end the difference between the Reynolds number of a particle in air and, for example, in water is only a factor of 5. And although the constants for calculating the drag coefficient of particles depend on the Reynolds number, the variation with Reynolds number within this range is very small and can be neglected. Therefore, the dynamic form factor is assumed to be equal in both air and liquid.

e)   in the material of interest all particles have the same and known particle density.

f)   the sub sample is representative of the bulk material.

If these assumptions are not met, the calculation method shall be used as specified in EN 17289-2.

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6 Determination of SWFF and SWFFCS by sedimentation

6.1 Determination of sedimentation time

The sedimentation time shall be calculated by Formula (2), which is based on Stokes' law and the convention given in EN 481. The derivation of it is given in Annex A.

$$t = h_{sup} \times \frac{18\eta}{\left(\rho_p - \rho_l\right)g} \times \frac{\left(\dfrac{\rho_p}{\rho_0}\right)}{\left(\dfrac{3}{2} \times I_R\right)^2} \tag{2}$$

where

t is the time, in seconds (s), at which the size-weighted particle fraction of the collected supernatant in an optimal way approaches the fraction collected according to the sampling convention of respirable dust (see EN 481);

h_{sup} is the height of the column of supernatant liquid that is extracted at the calculated time, in metres (m);

η is the viscosity of the fluid, in kilograms per metre per second (kg/m·s);

g is the acceleration due to gravity, in metres per square seconds (m/s^2);

ρ_p is the particle density of the particles, in kilograms per cubic metre (kg/m^3);

ρ_l is the density of the liquid medium, in kilograms per cubic metre (kg/m^3);

ρ_0 is the unit density (1 000 kg/m^3 [= 1 g/cm^3]);

I_R is the respirable convention integral: 4,281 × 10^{-6} m.

For determining the SWFF of a sample, the particle density of that sample shall be used for ρ_p.

When SWFFCS is determined, the density of the crystalline silica polymorph of interest (quartz or cristobalite) shall be used.

SWFFCS shall be used when the CS is the constituent of only interest. For the SWFFCS only the sedimentation rate for the quartz (cristobalite etc.) particles are relevant. The fact that the sedimentation time for other particles is not correct is not important since SWFFCS is needed. When the SWFF of material with a different particle density is needed the procedure shall be repeated with a sedimentation time as calculated by Formula (1) with the particle density of that material.

The height h_{sup} to be separated is usually set at 0,1 m, but other heights can be used depending on the nature of the sample. In any case, the height h_{sup} shall be less than half the total height h_{tot} of the sedimentation liquid in the measuring cylinder (see B.4).

6.2 Selection of sedimentation liquid

A suitable sedimentation liquid shall be selected in order to meet the following requirements:

— the particles in the sample shall be completely de-agglomerated;

— the particles in the sample shall not dissolve, swell or disintegrate;

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— the particles in the sample shall not react.

NOTE 1   Water is in most cases a suitable sedimentation liquid.

When necessary, a dispersant or deflocculant additive shall be used in appropriate quantities.

NOTE 2   De-agglomeration is the process where particles are separated from each other without breaking the particles. Disintegration is the process in which particles are broken.

6.3 Sample preparation, sedimentation and SWFF determination

In order to ensure that the sedimentation is performed correctly, the Andreasen pipette method is used [2], [3].

The following steps shall be executed to determine the SWFF or SWFFCS of a sample.

a) take a representative subsample of the bulk sample of approximately 1,0 g;

For coarse bulk material a pre-preparation is required to respect the limitations of the Andreasen pipette method, e.g. sieving.

b) determine the mass m_{tot} of the sample with a precision of 0,001 g;

c) disperse the sample in 50 ml of sedimentation liquid in a 100 ml pre-weighted, dry and clean beaker. The mass of the beaker shall also be determined with a precision of 0,001 g (see also ISO 13317-2);

d) treat the sample in an ultrasonic bath or shaker until completely de-agglomerated;

NOTE 1   The de-agglomeration time will depend on the type of material.

e) if necessary, based on the characterization of the bulk material, add a suitable dispersant or deflocculant to keep the particles from flocculating or coagulating. The use of this product will lead to additional steps specified in 6.4;

For unknown bulk materials a full characterization is needed.

f) pour the dispersed sample in a graduated cylinder with 250 ml volume. Rinse out the sample jar using the sedimentation liquid to ensure that no residue remains. Fill the cylinder up to 250 ml with sedimentation liquid. Seal the open end of the cylinder and shake the contents thoroughly. Then replenish the cylinder up till 250 ml and homogenize;

NOTE 2   The volume of solids is maximum 0,2 % of the volume of the total liquid to ensure unhindered sedimentation of the separate particles.

For some minerals it can be required to use a plunger agitator. With the cylinder in place, agitate. Then remove the plunger to replenish the cylinder up till 250 ml and homogenize.

g) place the cylinder in a location where it is at constant temperature and free from effects that could cause currents in the liquid. The constant temperature can be achieved when using a water bath. Leave to settle for the calculated time (t). Determine the height of the liquid column h_{tot} (m);