SLOVENSKI STANDARD
SIST EN 17289-1:2021

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Karakterizacija razsutih materialov - Določanje velikostno utežene fine frakcije in deleža kristaliničnega kremena - 1. del: Splošne informacije in izbira preskusnih metod

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

Charakterisierung von Schüttgütern - Bestimmung einer größengewichteten Feinfraktion und des Anteils an kristallinem Quarz - Teil 1: Allgemeine Information und Auswahl der Prüfverfahren

Caractérisation des matériaux en vrac - Détermination de la fraction fine pondérée par taille et de la teneur en silice cristalline - Partie 1 : Informations générales et choix des méthodes d'essai

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

Caractérisation des matériaux en vrac - Détermination de la fraction fine pondérée par taille et de la teneur en silice cristalline - Partie 1 : Informations générales et choix des méthodes d'essai

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European foreword

This document (EN 17289-1:2020) 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of an identical text or by endorsement, at the latest by June 2021, and conflicting national standards shall be withdrawn at the latest by June 2021.

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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, for example, 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 [1] can be used as input for this document. However, the output of this document is not related to the respirable fraction 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 [2].

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).

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 requirements and choice of test method for the determination of the size-weighted fine fraction (SWFF) and the size-weighted fine fraction of crystalline silica (SWFFCS) in bulk materials.

This document gives also guidance on the preparation of the sample and the determination of crystalline silica by X-ray Powder Diffractometry (XRD) and Fourier Transform Infrared Spectroscopy (FT-IR).

NOTE EN 17289-2 specifies a method to calculate the size-weighted fine fraction from a measured particle size distribution and assumes that the particle size distribution of the crystalline silica particles is the same as the other particles present in the bulk material. EN 17289-3 specifies a method using a liquid sedimentation technique to determine the size-weighted fine fraction of crystalline silica. Both methods are based upon a number of limitations and assumptions, which are listed in EN 17289-2 and EN 17289-3, respectively. The method in EN 17289-3 can also be used for other constituents than CS, if investigated and validated.

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 1540, *Workplace exposure — Terminology*

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

EN 17289-3:2020, *Characterization of bulk materials — Determination of a size-weighted fine fraction and crystalline silica content — Part 3: Sedimentation method*

ISO 16258-2:2015, *Workplace air — Analysis of respirable crystalline silica by X-ray diffraction — Part 2: Method by indirect analysis*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 1540 and the following 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

3.1
10th–percentile particle diameter

d_{10}

particle diameter corresponding to 10 % of the cumulative undersize distribution (by volume or by mass)

3.2
90th–percentile particle diameter

d_{90}

particle diameter corresponding to 90 % of the cumulative undersize distribution (by volume or by mass)

3.3
between-samples standard deviation

S_s

standard deviation between the random samples used for homogeneity check

3.4
bulk sample

portion that is representative of the bulk material

3.5
coefficient of variation of the reproducibility

CV_R

ratio of standard deviation to the mean of test results produced under reproducibility conditions, i.e. conditions where test results are obtained with the same method on identical test items in different laboratories with different operators using different equipment

3.6
complex refractive index

$\underline{n}_p$

refractive index of a particle, consisting of a real and an imaginary (absorption) part

Note 1 to entry: The complex refractive index of a particle can be expressed mathematically as

$$\underline{n}_\mathrm{p} = n_\mathrm{p} - \mathrm{i} \times k_\mathrm{p}$$

where

i	is the square root of −1;
k_p	is the positive imaginary (absorption) part of the refractive index of a particle;
n_p	is the positive real part of the refractive index of a particle

[SOURCE: ISO 13320:2020, 3.1.5, [3] modified – Note 2 to entry deleted]

3.7
crystalline silica

SiO_2

silicon dioxide with Si and O orientated in a fixed pattern as opposed to a nonperiodic, random molecular arrangement defined as amorphous

Note 1 to entry: The three most common crystalline forms of silica are quartz, tridymite, and cristobalite.

3.8
equivalent Stokes diameter
equivalent spherical diameter
diameter of a sphere having the same rate of sedimentation and density as the particle for laminar flow in a liquid

3.9
mass fraction of crystalline silica
w_{CS}
mass fraction of crystalline silica in the bulk sample, in percent (%)

3.10
median particle diameter
d_{50}
particle diameter, where 50 % of the particles, by volume or by mass, are smaller than this diameter and 50 % are larger

3.11
mineral phase
homogeneous substance with a well-defined set of physical and chemical properties; it defines a uniquely identifiable mineral

3.12
particle density
ratio of the mass of particles to the volume of the particles, in which closed pores are included in the volume while open pores and volume between particles are excluded

Note 1 to entry: Particle density is typically determined using a pycnometer and has the dimension kg/m³.

3.13
size-weighted fine fraction
SWFF
w_{SWFF}
fraction of the mass of a bulk material as determined by the size and density of the particles and a well-defined probability function, in percent (%)

3.14
size-weighted fine fraction of crystalline silica
SWFFCS
w_{SWFFCS}
fraction of the mass of crystalline silica particles in the SWFF, in percent (%)

3.15
skeletal density
mass of a unit volume of the diatomaceous earth (DE) skeleton, inaccessible to Helium

3.16
standard deviation for proficiency assessment
σ
measure of dispersion used in the assessment of proficiency, based on the available information

[SOURCE: ISO 13528:2015, 3.4 [4]]

3.17
supernatant
column of liquid that is separated from the total sedimentation liquid column which contains the solid particles of interest

Note 1 to entry: See EN 17289-3:2020, Figure A.2.

3.18
z-score
z
standardized measure of laboratory bias, calculated using the assigned value and the standard deviation for proficiency assessment

[SOURCE: ISO 13528:2015, 3.7]

4 Symbols and abbreviations

CS	crystalline silica
DE	diatomaceous earth
LD	laser diffraction
PSD	particle size distribution
FT-IR	Fourier transform infrared spectroscopy
MMAD	mass median aerodynamic diameter
RCS	respirable crystalline silica
RI	refractive index
SWFF	size-weighted fine fraction
SWFFCS	size-weighted fine fraction of crystalline silica
XRD	X-ray powder diffractometry

5 Test methods

There are two ways to determine the SWFF and SWFFCS:

a) by calculation, as specified in EN 17289-2;

b) by sedimentation in a liquid, as specified in EN 17289-3.

The calculation method requires that the aerodynamic particle size distribution of the bulk material is known. When SWFFCS needs to be determined this is often not possible since the PSD of the CS in the sample cannot be determined separately from the rest of the sample. The CS can be finer or coarser than the bulk of the sample. Instead, in this case the sedimentation method shall be used to determine the SWFFCS.

The calculation method is easier and faster to perform. This can be a reason to choose the calculation method over the sedimentation method; the assumption is then made that the size distributions are the same so that SWFFCS can be calculated from the PSD of the whole sample. However, this can only be done after experiments have shown that the results are accurate and consistently equal or higher than the results from sedimentation for that particular bulk material.

The sedimentation method is a good approximation for the determination of SWFF and SWFFCS. However, when samples have a narrow size distribution and a median diameter (d_{50}) in the range from 6 µm to 12 µm (aerodynamic) the method shall not be used since results will be too low. Instead the calculation method shall be applied. This is possible because of the narrow size distribution. In this case the difference in PSD between CS and bulk of the sample is small.

The analytical approach specified in these methods fulfils the expanded uncertainty requirements of EN 482 [5]. Guidance on bias and uncertainties is given in Annex A.

Figure 1 gives a flowchart to assist the user in selecting the appropriate test method for the determination of SWFF and SWFFCS. Both calculation and sedimentation methods are based upon a number of assumptions, which are listed in EN 17289-2 and EN 17289-3, respectively.

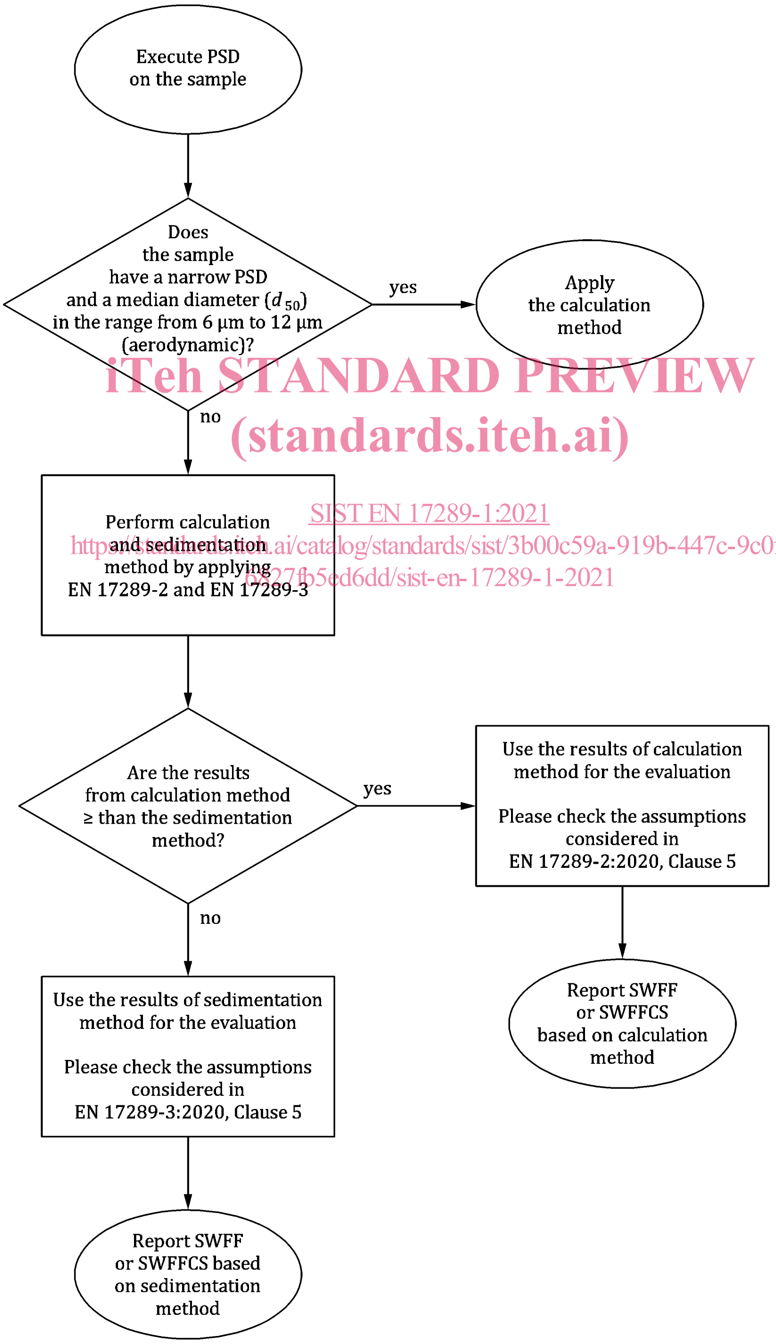

Figure 1 — Selection of test method for SWFF and SWFFCS