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Characterization of bulk materials - Determination of a sizeweighted 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

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Characterization of bulk materials - Determination of a sizeweighted 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

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Contents

Page

European foreword 3
Introduction 4
1 Scope 6
2 Normative references 6
3 Terms and definitions 6
4 Symbols and abbreviations 9
5 Test methods 9
6 Guidelines for the determination of crystalline silica 11
6.1 Preparation of sample to be analysed 11
6.2 Sample preparation for further analysis by XRD and FT-IR 11
7 Test report 11
Annex A (informative) Round robin test to establish a SWFF reference sample 13
A.1 General 13
A.2 Test material 13
A.3 Evaluation and appraisal method 14
A.4 Results 15
Annex B (informative) Determination of CS in bulk samples using X-Ray diffraction (XRD) or FT-IR spectroscopy 17
B.1 General 17
B.2 Preparation of bulk samples for determination of CS using XRD 17
B.2.1 General 17
B.2.2 Method 1: Bulk method 17
B.2.2.1 General 17
B.2.2.2 Preparation 18
B.2.2.3 Measurement 18
B.2.2.4 Calculation 18
B.2.3 Method 2: Deposition method 19
B.3 Preparation of bulk samples for determination of CS using FT-IR 19
B.3.1 Identification of the interfering minerals 19
B.3.2 Interferences removal 20
B.3.3 Standards and sample preparation 20
B.3.4 Operating conditions 20
B.3.5 Measurement 20
B.3.6 Determination of the quartz content 21
Bibliography 23

European foreword

This document (prEN 17289-1:2019) 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.

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 method provides the necessary information to the users and allows them to compare bulk materials, by measuring the fine fraction, in order for them to select the safest 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 describes 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) might change the particle size distribution, the size weighted fine fraction might also change. Therefore, the method reports (in terms of the mass percentages in the bulk material) both, the total CS and the estimated size weighted fine fraction of CS.

Conventions as described in EN 481 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 for workplace exposure measurements.

EN 17289 (all parts) describes two procedures that can be used to estimate the size weighted fine fraction (SWFF) in bulk materials. It also describes how the SWFF, once separated, can be further analysed to measure the content of crystalline silica ($SWFF_{cs}$). 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 ($SWFF_{cs}$) 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 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 $SWFF_{cs}$ values. SWFF and $SWFF_{cs}$ values are not to be used for occupational exposure assessments as they have no 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 (it 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*.

Part 1 gives information on how to choose the most appropriate method as well as a guideline for the determination of crystalline silica. A calculation method based on particle size distribution is described in Part 2. Part 3 describes a method using sedimentation.

1 Scope

This document specifies the 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 ($SWFF_{CS}$) in bulk materials.

This document gives also guidance on the preparation of the sample and determination of crystalline silica by XRD and FT-IR.

NOTE prEN 17289-2:2019 specifies a method to estimate 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. prEN 17289-3:2019 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 prEN 17289-2:2019 and prEN 17289-3:2019, respectively. The method in prEN 17289-3:2019 can also be used for other constituents, if investigated and validated.

This document is applicable for bulk materials which have been fully investigated and validated. The criteria for the materials are described in prEN 17289-2:2019 and prEN 17289-3:2019. This includes industrial minerals which can contain crystalline silica such as quartz, clay, kaolin, talc, feldspar, mica, cristobalite, vermiculite, diatomaceous earth, barite, andalusite, iron ore, chromite etc.

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 1540:2011, *Workplace exposure - Terminology*

prEN 17289-2:2019, *Characterization of bulk materials – Determination of a size-weighted fine fraction and crystalline silica content — Part 2: Calculation method*

prEN 17289-3:2019, *Characterization of bulk materials – Determination of a size-weighted fine fraction and crystalline silica content — Part 3: Sedimentation method*

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

ISO 24095, *Workplace air — Guidance for the measurement of respirable crystalline silica*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 1540:2011 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
between-samples standard deviation
Ss
standard deviation between the random samples used for homogeneity check

3.2
bulk density
ratio of the mass of a quantity of dry granular material to the total volume of its grains, including the volume of any closed pores within the grains

[SOURCE: ISO 836: 2001[3]]

3.3
bulk sample
portion that is representative of the bulk material

3.4
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

[SOURCE: ISO 5725-1[4]]

3.5
complex refractive index
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

$$n_p = m_p - i \times k_p$$

where

i is the square root of −1;

k_p is the positive imaginary (absorption) part of the refractive index of a particle;

m_p is the positive real part of the refractive index of a particle

[SOURCE: ISO 13320] [5]

3.6
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.

3.7
D_{10}
particle diameter corresponding to 10 % of the cumulative undersize distribution (by volume or by mass)

[SOURCE: ISO 13320]

3.8
D_{50} median particle diameter
particle diameter, where 50 % of the particles – by volume or by mass – are smaller than this diameter and 50 % are larger

[SOURCE: Adapted from ISO 13320]

3.9
D_{90}
particle diameter corresponding to 90 % of the cumulative undersize distribution (by volume or by mass)

[SOURCE: ISO 13320]

3.10
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

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

3.12
relative density
ratio of the density of a substance to the density of a given reference material

3.13
size weighted fine fraction
SWFF
proportion of mass of particles in a bulk material below a well-defined probability function

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[6]]

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 prEN 17289-3:2018, Figure A.2.

3.18
$SWFF_{CS}$
proportion of crystalline silica particles in a bulk material below a well-defined probability function

3.19
z-score
z
standardized measure of laboratory bias, calculated using the assigned value and the standard deviation for proficiency assessment

[SOURCE: EN ISO/IEC 17043[7]]

4 Symbols and abbreviations

CS	Crystalline Silica
DE	Diatomaceous Earth
PSD	Particle Size Distribution
FT-IR	Fourier Transform Infrared Spectroscopy
XRD	X-ray Powder Diffractometry
SWFF	Size Weighted Fine Fraction
$SWFF_{CS}$	Size Weighted Fine Fraction of crystalline silica

5 Test methods

There are two ways to determine the SWFF and $SWFF_{CS}$:

a) by calculation, as described in prEN 17289-2:2019;

b) by sedimentation in a liquid, as described in prEN 17289-3:2019.

The calculation method requires that the aerodynamic particle size distribution of the bulk material is known. When $SWFF_{cs}$ 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 $SWFF_{cs}$.

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 $SWFF_{cs}$ 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 $SWFF_{cs}$. 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.

NOTE The method given in prEN 17289-2:2018 (first measuring the particle size distribution and thereafter calculate the SWFF based on this size distribution), overestimates the SWFF if the laser diffraction measurements are accurate, otherwise the bias is unknown. For non-spherical particles, the liquid sedimentation method (see prEN 17289-3:2018) overestimates the SWFF.