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Secretariat:

Size Weighted Respirable Fraction – SweRF

and

Size Weighted Respirable Fraction of Crystalline Silica - SWeRFcs

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Foreword

This document has been prepared by the Industrial Minerals Association, IMA-Europe.

This document is currently submitted to the Primary Questionnaire procedure.

1 Scope

This standard specifies a method for evaluating the size weighted respirable fraction in terms of weight percent, which is a means of expressing the content of respirable particles in bulk materials. The method can also be extended to give a means of expressing the content of respirable crystalline silica particles. The method can be used for comparing the potential health hazard of different bulk samples.

2 Normative references

EN 481:1994 Workplace atmospheres - Size fraction definitions for measurement of airborne particles.

3 Terms and definitions

In this European standard, the terms and definitions given in EN 481 apply. In addition, for the purpose of this European standard, the following terms and definitions apply.

3.1
SWeRF
Size Weighted Respirable Fraction, a measurement of the content of respirable particles in bulk samples, taking into account the particle size distribution and the probability for particles reaching the alveoli.

3.2
SWeRF_{cs}
Size Weighted Respirable Fraction of Crystalline Silica, calculated by multiplying the SWeRF of a sample by the crystalline silica content of the sample.

4 Methods for determination of SWeRF and SWeRF_{cs}

4.1 Introduction

SWeRF and SWeRF_{cs} can be evaluated in two possible ways; either by calculation or by sedimentation.

The guidance given in appendix A1 explains the context in which each method can be used.

4.2 Determination of SWeRF and SWeRF_{cs} by calculation.

The SWeRF of a sample is calculated by first determining its particle size distribution. A size weighting is then applied, based on the probability function given in EN 481, i.e. the probability function for particles reaching the alveoli when inhaled. See formula 1.

$$SWeRF = \int_{x=0}^{x=\infty} f(psd)_x \times P(x) dx \quad (1)$$

$f(psd)_x$ = particle size fraction for aerodynamic diameter x .

$P(x)$ = probability of reaching the alveoli for particles of aerodynamic diameter x , according to EN 481

x = aerodynamic diameter = $D \times \sqrt{SG}$, where D is the physical diameter and SG is the specific gravity.

The $SWeRF_{cs}$ is calculated by multiplying the $SWeRF$ of the sample with the crystalline silica content of the sample, see formula 2. It is assumed that the Particle Size Distribution of the sample and of its Crystalline Silica content are the same.

$$SWeRF_{cs} = SWeRF \times \varphi_{cs} \quad (2)$$

φ_{cs} = mass fraction of crystalline silica in the sample

- NOTE The content of crystalline silica of the sample can be determined using techniques such as X-ray Powder Diffraction (XRD) or Infrared Spectroscopy (IR). The methods are derived from the standards used to determine crystalline silica in respirable airborne dusts, e.g. MDHS 1010.

4.3 Determination of $SWeRF$ and $SWeRF_{cs}$ by Sedimentation

4.3.1 Determination of sedimentation time

The time of sedimentation can be calculated using the following equation (see formula 3), which is based on Stokes' law and the convention described in EN 481.

$$t = h \times \frac{18 \times \eta}{(\rho_s - \rho_l) \times g} \times \frac{4}{9} \times \frac{SG_s}{\left(\int_{x=0}^{x=\infty} (EN481) dx \right)^2} \quad (3)$$

t = time (s) at which separation of the particles by sedimentation in the liquid equals separation according to EN 481.

h = height (m) of the column of the supernatant liquid that is extracted after time = t.

η = dynamic viscosity of the liquid (kg/ms)

g = acceleration due to gravity (m/s²)

ρ_s = density of the solid particles (kg/m³)

ρ_l = density of the liquid medium (kg/m³)

SG_s = specific gravity of the solid particles (water = 1)

x = aerodynamic diameter (μ m)

$$\int_{x=0}^{x=\infty} (EN 481) dx = 4,281 (\mu\text{m}) \quad (\text{Integral of Probability Function EN 481})$$

For determining the $SWeRF$ of a sample, the density of this sample should be used for ρ_s . When $SWeRF_{cs}$ is determined, the density of crystalline silica (quartz or cristobalite) should be used.

- NOTE the height h to be decanted is usually set at 0,01 m, but other heights may be used depending on the nature of the sample. In any case, the height h should be less than half the total height H of the sedimentation liquid in the measuring cylinder (see step 7 in clause 5.3).

4.3.2. Selection of Sedimentation Liquid

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

- The particles in the sample must be completely deagglomerated.
- The particles in the sample should not dissolve, swell or disintegrate.
- The particles in the sample should not react.
- NOTE Water in most cases is a suitable sedimentation liquid.
- NOTE When necessary, a dispersant or deflocculant additive may be used in appropriate quantities (see guidance in annex A).

4.3.3. Sample Preparation, Sedimentation and SWeRF Determination

The following steps should be executed to determine the SWeRF or SWeRFcs of a sample:

- 1) Take a sample of the material to be analysed of approx. 0.005 kg.
- 2) Determine the weight M of the sample to be analysed with a precision of 0.001 g.
- 3) Disperse the sample in 50 ml of sedimentation liquid in a 100 ml pre-weighed, dry and clean beaker. The weight of the beaker should also be determined with a precision of 0.001 g.
- 4) Treat the sample in an ultrasonic bath or a shaker until completely deagglomerated.
- NOTE The deagglomeration time will depend on the type of material, see for further information annex A.
- 5) If necessary, add a suitable dispersant or deflocculant to keep the particles from flocculating or coagulating. See for further information Annex A.
- 6) Pour the dispersed sample in a 250 ml measuring cylinder. 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 homogenise.
- NOTE The volume of solids should be maximum 1 % of the volume of the total liquid to ensure unhindered sedimentation of the separate particles.
- 7) Place the cylinder in a location where it is at constant temperature and free from effects that could cause currents in the liquid and leave to settle for the calculated time (t). Determine the depth of the liquid column H (m).
- 8) From the cylinder, after calculated time (t), lower a pipette to an insertion depth of h below the surface, draw the volume of the supernatant (Vt) above the tip of the pipette with a siphon or pipette and transfer into the pre-weighed beaker.

Make sure that, whilst using the pipette, the tip of the pipette remains in the same place in the cylinder; do not insert it deeper as the level descends.

- NOTE Placing a clip on the pipette and attaching it to the edge of the cylinder will make it easier to keep the pipette in the right place.
- 9) Place the beaker containing the supernatant on a hot plate to evaporate the liquid and heat gently until dry. Then place the beaker in the oven at 103 °C for one hour and transfer into a desiccator, leaving it to cool down to ambient temperature .
 - 10) Re-weigh the beaker to within 0.001 gram accuracy and note the post-weight. Determine the weight of the residue m by subtracting the weight of the pre-weighed beaker (see step 3).

- NOTE The above procedure should initially be repeated twice in order to check the reproducibility of the sedimentation method for SWeRF determination.

11) Determine the SWeRF of the sample using equation 4.

$$SWeRF = \frac{H \times m}{h \times M} (\%) \quad (4)$$

12) The SWeRFcs of the sample may be calculated by using equation 5.

$$SWeRFcs = \frac{H \times m}{h \times M} \times \varphi_{cs} (\%) \quad (5)$$

Annex A

Guidance on the Use of the Calculation and Sedimentation Methods

Informative

A.1 Recommendations on Using the Calculation and/or Sedimentation Method

There are two ways to determine the SWeRF and SWeRF_{CS}:

1. the calculation method through a simple Excel sheet,
2. the sedimentation method in a liquid.

Experiments have been done on quartz sand and flours, feldspar, limestone, leucophyllite, clay and diatomaceous earth and it has been shown that:

- For quartz flours and sand, the calculation method can be applied directly and provides an accurate result which is consistent with the sedimentation method.
- For some minerals the calculation method may provide an overestimation of the SWeRF_{CS}, in which case the sedimentation method will give a more accurate figure. This is the case when the PSD (Particle Size Distribution) of the CS in the material is significantly coarser than the PSD of the bulk material.
- For the sedimentation method, more investigation is needed, for some minerals, to find a suitable liquid and/or dispersant agent.

A.2 Recommendations for Using the Sedimentation Method

In order to get reliable results from the sedimentation test, the particles in the sample must be completely deagglomerated. If necessary, a suitable dispersant or deflocculant should be added to keep the particles from flocculating or coagulating.

In this case, to adjust the weight of the samples when an additive has been added, a blank cylinder must be prepared by adding only the dispersant to the liquid. Also, three blank beakers must be prepared. These beakers should be treated the same as described in steps 3 and 4 of clause 4.3.3. Determine the weight of the residual additive by taking the average of the residue weight in the 3 blank beakers. Deduct this average weight of residual additive from the mass of the sample in step 10.

The three blank beakers should also be used as controls to check that the additive does not interfere with subsequent IR or XRD measurement.

A.3 Guidelines for the Determination of Respirable Crystalline Silica in Bulk Samples

Currently, the two fundamental techniques used for routine estimation of the silica content of a respirable dust sample are X-ray diffraction (XRD) and infrared (IR) absorption analysis. An ISO standard on determination of crystalline silica in bulk material has been published in 2009 (ISO 24095 Workplace air - Guidance for the measurement of respirable crystalline silica).

Respirable Crystalline silica can be quantified in air monitoring and bulk samples

- If they are uncontaminated with minerals and/or materials yielding overlapping and / or
- interfering peaks with the principal crystalline silica reflections

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- If standard reference materials equivalent in peak half-width (crystallinity) and particle size distribution are available for comparison with the mineral or material sample under study.

The choice of analytical technique depends largely on other materials present on the sample which may interfere in the analysis (overlapping, matrix effects, high mass absorption coefficient, etc.).

- Examples of interfering minerals: Quartz interferes with cristobalite and cristobalite with quartz
- Other minerals, interfering on XRD pattern, with primary Quartz peak: Aluminium phosphate, biotite, albite, microcline, muscovite, orthoclase, sanidine, sillimanite, wollastonite.
- Interfering on IR Quartz spectra: albite, anorthite, kaolinite, muscovite, orthoclase, ripidolite.
- Minerals interfering with cristobalite: albite, anorthite, calcite, cordierite, kaolinite, muscovite.

All analytical methods for crystalline silica analysis (XRD and IR) are particle size dependent. It is well known that as particle size increases the response of XRD increases while the response of IR decreases.

Crystalline silica methods require calibration standards of known purity, specific particle size and distribution and sample-to-sample homogeneity. Ideally the particle size distribution of the calibration standard would closely match that of the samples being analysed.

The following recommendations apply whether using XRD or IR analysis:

- Periodic phase identification of the minerals or material under investigation (by a mineralogist or a petrologist or other suitable methods) from representative bulk samples.
- If possible, remove the interfering phases:
 - by treatment of the sample with dilute hydrochloric acid (metals, oxides, carbonates, etc) and/or fused potassium bisulphate (most silicates),
 - by ashing.
- Choose the more appropriate technique accordingly.
- Standard reference samples should have ideally the crystallinity and the same particle size distribution as the samples under consideration.
- Standard reference samples and samples must be prepared in the same manner.
- Use rotating sample holder to maximise random orientation of particles.
- Select a quartz (or cristobalite) line free from interference from other phases. Note that sensitivity is reduced if secondary peaks are used.
- The crystalline silica content of the samples under study should be in the same range as those used for the calibration curve.

Specific recommendations when using XRD technique:

- Monitor and correct for the gradual decline in X Ray tube emission.
- Use preferably the peak area instead of intensity peak height.