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**Workplace air — Analysis of  
respirable crystalline silica by X-ray  
diffraction —**

**Part 2:  
Method by indirect analysis**

*Air des lieux de travail — Fraction alvéolaire de la silice cristalline  
par diffraction de rayons X —  
Partie 2: Méthode indirecte d'analyse*

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](http://Foreword - Supplementary information)

The committee responsible for this document is ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace atmospheres*.

ISO 16258 consists of the following parts, under the general title *Workplace Air — Analysis of respirable crystalline silica by X-ray diffraction*:

- *Part 1: Direct-on-filter method*
- *Part 2: Method by indirect analysis*

## Introduction

Respirable crystalline silica (RCS) is a hazard to the health of workers in many industries through exposure by inhalation. Industrial hygienists and other public health professionals need to determine the effectiveness of measures taken to control workers' exposure. The collection of samples of air during a work activity and then measuring the amount of respirable crystalline silica is often done to assess an individual's exposure, the effectiveness of controls or their respiratory protection. X-ray diffraction (XRD) analysis of crystalline silica in a sample of respirable dust collected on a filter is the principle technique employed in many countries to measure and estimate exposure to RCS. X-ray diffraction is able to clearly distinguish the polymorphs of crystalline silica.

This part of ISO 16258 specifies the analysis procedure for the measurement of RCS where the dust is recovered from the collection substrate and deposited onto a filter for analysis. Many different types of sampling apparatus are used to collect respirable dust, according to the occupational hygiene convention. This part of ISO 16258 is designed to accommodate the variety of samplers and collection substrates available to analysts. This part of ISO 16258 is to be used in conjunction with ISO 24095 which promotes best practice for these analyses.

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# Workplace air — Analysis of respirable crystalline silica by X-ray diffraction —

## Part 2: Method by indirect analysis

### 1 Scope

This part of ISO 16258 specifies the analysis of RCS in samples of air collected on collection substrates (i.e. filters or foams) by X-ray diffraction, when using an analytical approach where dust from the sample collection substrate (i.e. filter or foam) is recovered, treated and deposited on another filter for analysis by the instrument. This part of ISO 16258 includes information on the instrumental parameters, sensitivity of different sampling apparatus, the use of different filters, sample treatment to remove interference and correction for absorption effects. In this part of ISO 16258, the expression respirable crystalline silica includes the most common polymorphs quartz and cristobalite. The less common polymorphs of crystalline silica, such as tridymite, are not included within the scope of this part of ISO 16258 because a standard reference material is not available. Under certain circumstances (i.e. low filter dust loads, low silica content), the analytical approach described in this method may not fulfil the expanded uncertainty requirements of EN 482[4]. Guidance for calculation of uncertainty for measurements of RCS is given in ISO 24095.

### 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 7708, *Air quality — Particle size fraction definitions for health-related sampling*

ISO 13137, *Workplace atmospheres — Pumps for personal sampling of chemical and biological agents — Requirements and test methods*

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

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

### 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

#### 3.1 General definitions

##### 3.1.1

##### **airborne particles**

fine matter, in solid or liquid form, dispersed in air

[SOURCE: EN 1540]

Note 1 to entry: Smoke, fume, mist and fog consist of airborne particles.

### 3.1.2

#### **aerosol**

airborne particles and the gas (and vapour) mixture in which they are suspended

[SOURCE: EN 1540]

Note 1 to entry: The airborne particles can be in or out of equilibrium with their own vapours.

### 3.1.3

#### **respirable crystalline silica**

##### **RCS**

inhaled particles of crystalline silica that penetrate into the unciliated airways according to the respirable convention described in ISO 7708

### 3.1.4

#### **exposure (by inhalation)**

situation in which a chemical agent is present in air that is inhaled by a person.

[SOURCE: EN 1540]

### 3.1.5

#### **occupational exposure limit value**

limit of the time-weighted average of the concentration of a chemical agent in the air within the breathing zone of a worker in relation to a specified reference period

[SOURCE: EN 1540]

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Note 1 to entry: Limit values are mostly set for reference periods of 8 hours, but can also be set for shorter periods or concentration excursions. Limit values for airborne particles and mixtures of particles and vapours are given in mg/m<sup>3</sup> or multiples of that for actual environmental conditions (temperature, pressure) at the workplace.

### 3.1.6

#### **workplace**

designated area or areas in which the work activities are carried out

[SOURCE: EN 1540]

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## 3.2 Sampling definitions

### 3.2.1

#### **aerosol sampler**

(airborne) particle sampler

(airborne) particulate sampler)

sampler that is used to transport airborne particles to a collection substrate

[SOURCE: EN 1540]

### 3.2.2

#### **collection substrate**

sampling substrate

collection medium

sampling medium

medium on which airborne chemical and/or biological agents are collected for subsequent analysis

[SOURCE: EN 1540]

Note 1 to entry: Filters, polyurethane foams and sampling cassettes are examples of collection substrates for airborne particles.



**3.2.3****laboratory blank**

unused collection substrate, taken from the same batch used for sampling, that does not leave the laboratory

[SOURCE: EN 1540]

Note 1 to entry: The results from the analysis of laboratory blanks are used to correct sample results for contamination with crystalline silica and/or interferences.

**3.2.4****field blank**

unused collection substrate, taken from the same batch used for sampling, handled in the same way as a collection substrate that is used for sampling, except it is not used for collecting a sample

[SOURCE: EN 1540]

Note 1 to entry: A field blank is transported to the sampling site, loaded in the sampler, where applicable, and returned to the laboratory in the same way as a sample.

Note 2 to entry: The results from the analysis of field blanks are used to identify contamination of the sample arising from handling in the field and during transport.

**3.2.5****breathing zone**

space around the nose and mouth from which a worker's breath is taken

[SOURCE: EN 1540]

Note 1 to entry: Technically the breathing zone corresponds to a hemisphere (generally accepted to be 30 cm in radius) extending in front of the human face, centred on the midpoint of a line joining the ears. The base of the hemisphere is a plane through this line, the top of the head and the larynx. This technical description is not applicable when respiratory protective equipment is used.

**3.2.6****personal sampler**

sampler, attached to a person, that collects gases, vapours or airborne particles in the breathing zone to determine exposure to chemical and/or biological agents

[SOURCE: EN 1540]

**3.2.7****personal sampling**

process of sampling carried out using a personal sampler

[SOURCE: EN 1540]

**3.2.8****sampling train**

apparatus for collecting airborne particles including sampling equipment, pump and connecting tubing

[SOURCE: ISO 24095]

**3.3 Analytical definitions****3.3.1****limit of detection****LOD**

lowest amount of respirable crystalline silica (RCS) that is detectable with a given level of confidence

Note 1 to entry: The limit of detection can be calculated as three times the standard deviation of blank measurements. This represents a probability of 50 % that the analyte will not be detected when it is present at the concentration of the LOD.

Note 2 to entry: The LOD can be used as a threshold value to assert the presence of a substance with a known confidence.

Note 3 to entry: Many analysis procedures require laboratories to calculate an LOD by multiplying the standard deviation of measurements of a number of blank samples (~10) by three. Readers should note that there is some doubt about the relationship between signal and the mass when RCS is measured at very low masses and a specific formula to determine the LOD using statistics based on a normal distribution is not given in this guidance. The test samples used for calibration are not matrix matched and reporting an LOD based on three standard deviations of the background noise may give an optimistic impression of the capability of method when analysing 'real' samples. Analysts should take this into consideration when analysing samples for RCS (ISO 24095).

### 3.3.2 limit of quantification LOQ

lowest reliable mass of an analyte that is quantifiable with a given level of confidence taking into consideration the matrix effects in the sample

[SOURCE: EN 1540]

Note 1 to entry: The limit of quantification can be calculated as 10 times the standard deviation of blank measurements

Note 2 to entry: The value LOQ can be used as a threshold value to ensure measurement of a substance accurately

Note 3 to entry: With LOQ determined from an evaluation experiment based on 10 degrees of freedom, an estimate of an amount at the threshold value LOQ has probability equal to 95 % of falling inside an interval defined as  $\pm 31$  % about the true value, with 95 % confidence in the evaluation

## 3.4 Statistical terms

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### 3.4.1 accuracy

closeness of agreement between a test result and the accepted reference value

[SOURCE: ISO 3534]

### 3.4.2 analytical recovery

ratio of the mass of analyte measured when a sample is analysed to the known mass of analyte in that sample

[SOURCE: ISO 24095]

### 3.4.3 method recovery

ratio of the determined concentration of chemical agent in air to its actual concentration

[SOURCE: EN 1540]

Note 1 to entry: The method recovery incorporates both sampling efficiency and analytical recovery.

### 3.4.4 bias

difference between the expectation of a test result or measurement result and a true value

[SOURCE: ISO 6879]

Note 1 to entry: Bias is the total systematic error as contrasted to random error. There may be one or more systematic error components contributing to the bias. A larger systematic difference from the true value is reflected by a larger bias value.

**3.4.5****precision**

closeness of agreement between independent test/measurement results obtained under stipulated conditions

[SOURCE: ISO 6879]

**3.4.6****true value**

value which characterizes a quantity or quantitative characteristic perfectly defined in the conditions which exist when that quantity or quantitative characteristic is considered

[SOURCE: ISO 3534]

**3.4.7****uncertainty (of measurement)**

parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand

[SOURCE: ISO 3534]

Note 1 to entry: The parameter may be, for example, a standard deviation (or a given multiple of it), or the width of a confidence interval.

Note 2 to entry: Uncertainty of measurement comprises, in general, many components. Some of these components maybe evaluated from the statistical distribution of the results from a series of measurements and can be characterized by standard deviations. The other components, which also can be characterized by standard deviations, are evaluated from assumed probability distributions based on experience or other information. Refer to the ISO Guide 98:2008.

**4 Principle**

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This part of ISO 16258 provides a method for indirect analysis of RCS in respirable dust collected on a filter (usually with a diameter of 37 mm) or foam, dust from the sample collection substrate (i.e. filter or foam) is recovered, treated and deposited on another filter for analysis by the instrument. The instrument is calibrated by preparing test samples from aliquots of a suspension of standard dust. The mass of RCS on the filter is determined from the X-ray response, calibrated against filters loaded with known amounts of standard quartz or cristobalite. Since the volume of air sampled is known the concentration of RCS in the air is readily calculated. If the deposit of sample on the filter is too deep, the X-ray radiation might not penetrate into the whole sample and the radiation may also be absorbed by the sample's matrix. The method assumes the depth of the sample deposit on the filter is so thin that absorption effects are negligible when the dust deposit is less than a critical sample mass. The point at which the absorption becomes significant will depend on the thickness and mass absorption coefficient of the dust on the filter surface. It is generally accepted that samples from most industrial environments will not be significantly affected by absorption. Correction for X-ray absorption is possible by measuring the transmittance of a silver filter or aluminium plate through the dust deposit.

**5 Sampling**

Guidance on the analytical requirements for sampling for RCS is given in ISO 24095. A general guidance for the sampling of respirable aerosol fraction is given in CEN/TR 15230.

**5.1 Sampling equipment****5.1.1 Samplers**

**5.1.1.1** The performance of the samplers used shall match the criteria for respirable dust as specified in ISO 7708.

**5.1.1.2** Samplers that use 25-mm or 37-mm diameter filters as the collection substrate are required. Particle impactors and samplers using a foam pad, such as the CIP 10-R, are suitable for this method.

NOTE [Annex G](#) provides information on the performance of different personal respirable samplers that are currently in use.

**5.1.1.3** Each sampler should be labelled with a unique number, in order to identify samplers that start to under-perform after long-term use.

**5.1.1.4** Samplers shall comply with the manufacturer's requirements for calibration.

NOTE In some countries there might be exceptions due to national regulations, e.g. the CIP 10-R must be calibrated in accordance with the procedure in NF X43 259.[\[22\]](#)

## 5.1.2 Collection substrates

**5.1.2.1** Filters shall be of a diameter suitable for use in the selected sampler and have a capture efficiency for respirable particles of not less than 99 %.

**5.1.2.2** It is important for the analyst to know the composition of the collection substrate used to collect the sample since it has a direct bearing on the analytical approach used to recover the dust. The collection substrates types generally used for the sampling of RCS, and their advantages and disadvantages, are listed in [Table 1](#).

**5.1.2.3** The filter types generally used for the XRD analysis of RCS, and their advantages and disadvantages, are listed in [Table 2](#).

**Table 1 — Dust collection substrates**

Filter Material	Weight stability index <sup>a</sup>	Comments
Polyvinyl chloride (PVC)	HIGH	A filter pore size of 5 µm is usually used
Cellulose nitrate	MEDIUM	Usually used with FSP 10 sampler with a 8 µm pore size
Mixed Esters of Cellulose	LOW	Pore sizes larger than 0,8 µm are used with high flow rate samplers > 4 l/min
Foams Polyurethane	LOW	Used with CIP 10 sampler
<sup>a</sup> Weight stable assuming static elimination.		

**5.1.2.4** Filter materials listed in [Table 1](#) generally do not interfere with the measurement of the major reflections of quartz (101), (100), and (112), and cristobalite (101), (200 and 112), and (102). Impurities can be introduced during the filter manufacturing process and background reflections can increase depending on filter material. Therefore, batches of filters should be regularly tested to detect potential interferences and background levels.

**5.1.2.5** Cristobalite combined reflections (200 and 112) can sometimes be difficult to measure because they are located in the tail at the side of silver reflection (100).

**5.1.2.6** Variable background has an effect on the readability of diffraction peaks, increasing the limit of detection for RCS. Silver filters exhibit the least variability and lowest background levels and thus are useful in situations where low limits of detection are required.