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

Part 1: Direct-on-filter method

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National foreword

This British Standard is the UK implementation of ISO 16258-1:2015.

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

Part 1:
Direct-on-filter method

Air des lieux de travail — Fraction alvéolaire de la silice cristalline par diffraction de rayons X —

Partie 1: Méthode directe d'analyse sur filtre

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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).

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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

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 are 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 procedure for a method of analysing RCS directly on the air sample filter. A specific requirement of this method is that the filter from the sampling apparatus has a diameter of 25 mm. The method also discusses optimum instrumental parameters, for various types of detectors in common use at the time of publication. This part of ISO 16258 is to be used in conjunction with ISO 24095 which promotes best practice for these analyses.

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

Part 1:
Direct-on-filter method

1 Scope

This part of ISO 16258 specifies the analysis of respirable crystalline silica (RCS) in samples of air collected on 25 mm-filters by X-ray diffraction, when using an analytical approach where the dust on the air sample filter is directly analysed by the instrument. This part of ISO 16258 includes information on the instrumental parameters, sensitivity of different sampling apparatus, uses of different filters and correction for absorption effects. In this part of ISO 16258, the expression RCS 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.[5] 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
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
OELV
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]

Note 1 to entry: Limit values are mostly set for reference periods of 8 h, 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^3 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]

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.

Note 2 to entry: For the purpose of this part of ISO 16258, the collection substrate is a filter.

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 an analyte 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 ±31 % about the true value, with 95 % confidence in the evaluation.

3.4 Statistical terms

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 may be 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

This part of ISO 16258 provides a method for direct on-filter analysis of RCS in respirable dust collected on a 25-mm filter, when limited or no sample preparation is desired. The mass of RCS on the filter is determined from the X-ray response, calibrated against filters loaded with known amounts of RCS reference material. Since the volume of air sampled is known the concentration of RCS in the air is readily calculated. Because the different aerosol samplers for respirable dust deposit the sample over the surface of the filter in different ways, the X-ray diffraction instruments used for the direct on-filter analysis approach shall be calibrated for the aerosol sampler used to collect the samples. 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 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 diameter filters as the collection medium are required. Particle impactors and samplers using a foam pad, such as the CIP 10, are not suitable for this method.

NOTE 1 Although calibrations have been tried using 37 mm diameter filters, these are not successful because of the variability caused by, the distribution of dust over the surface of the filter.

NOTE 2 Annex F provides information on the performance of different personal respirable samplers that require a 25-mm filter for sampling and 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.

5.1.2 Filters

5.1.2.1 Filters shall be 25 mm in diameter 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 filter used to collect the sample since it has a direct bearing on the analytical approach used. The filter types generally used for the sampling and XRD direct-on-filter analysis of RCS, and their advantages and disadvantages, are listed in Table 1.

5.1.2.3 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.

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

5.1.2.5 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.

5.1.2.6 Weighing should be performed following ISO 15767. Filters shall not be weighed in cassettes as large weight variations have been reported.[21]

5.1.2.7 Silver and mixed esters of cellulose filters are rigid and easy to handle when weighing and loading the sampler. PVC and especially polycarbonate filters are flexible and require careful handling.

5.1.2.8 A silver filter used for sampling allows an effective correction for absorption in case a heavily loaded sample is analysed (Annex C). When an organic filter is used for sampling and analysis, an effective correction for absorption can still be carried out, by measuring the intensity of a reflection of the underlying metallic base, if the material has a high or medium transparency to X-ray radiation (Table 1).

5.1.2.9 High values for back pressure can compromise the sampling time, when the sampling of a complete 8-h shift is desired. Table 1 indicates that if the given filters are used, then pumps that cannot cope with the back pressure shown in the table are not likely to achieve or maintain a flow rate of 2,2 l/min.[10]

5.1.2.10 This part of ISO 16258 describes a non-destructive technique, and samples can be re-analysed at a later date. Organic filters are ideal for storage. Silver filters oxidize in air, forming a dark-coloured oxide layer. To prevent oxidation, store silver filter samples in airtight container.

Table 1 — Filter materials for direct-on-filter-analysis

	SAMPLING	WEIGHING	XRD ANALYSIS								
Filter Material (pore size)	Back pressure at 2,2 l/min (in kPa)	Weigh stability[a]	Interfering Reflections						Background Fluctuation		Mass Absorption Correction
			Quartz			Cristobalite			Quartz		Filter Transparency To X-Rays
			101	100	112	101	200	102	101	100	
Silver (0,8 µm)	1,7	HIGH	no	no	no	no	yes	yes	LOW	LOW	No transparency
Polyvinylchloride (PVC) (5 µm)	0,5	HIGH	no	no	no	no	no	no	LOW	LOW	LOW
Mixed Esters of Cellulose (MCE) (0,8 µm)	3,2	LOW	no	no	no	no	no	no	HIGH	HIGH	MEDIUM
Polycarbonate (0,8 µm)	unknown	HIGH	no	no	no	no	no	no	LOW	HIGH	HIGH

[a] Weight stable assuming static elimination.

5.1.3 Sampling pumps

Sampling pumps shall comply with the requirements of ISO 13137.

5.1.4 Flow meters

Flow meters shall comply with the requirements of ISO 13137.

5.1.5 Other equipment required

Other equipment required for sampling include the following:

a) belts or harnesses to which the sampling pumps can conveniently be fixed;

b) flexible tubing, to connect the sampler to the sampling pump;

c) a means to transport the samples from the workplace to the laboratory, which minimises the possibility of accidental transfers of collected dust to or from the collection substrate (filter). Transportation will usually require caps or covers for the samplers, filter cassettes or other substrates, as detailed in the manufacturer's instructions for use of the instruments.

NOTE A thermometer (readable to 1 °C) and a barometer (readable to 0,1 kPa), to measure atmospheric temperature and pressure for flow rate correction, when the temperature and pressure at the time of use differ from the conditions under which the flow meter was calibrated (ISO 24095).

5.2 Sample collection

A general guidance for sampling of respirable aerosol fraction is given in CEN/TR 15230.[7] A guidance specific for the measurement of RCS is given in ISO 24095.

5.2.1 Clean the samplers before use to prevent contamination from previous work. Dismantle the parts that come into contact with the dust (referring to the manufacturer's instructions when necessary), soak the samplers in detergent, ultrasound to remove the fine dust and rinse in water. Allow time for the apparatus to dry before reassembly.

5.2.2 Pre-weigh each uniquely identified filter (including a minimum of three blanks) using flat tipped tweezers to avoid contamination and damage at least to the nearest 0,01 mg, according to ISO 15767.

5.2.3 Load each sampler with a pre-weighed filter, and connect each loaded sampler to a sampling pump.

NOTE For some sampler types a different procedure could be required; refer to the manufacturer's instructions.

5.2.4 Ensure each loaded sampler is tested for leaks. The leak test is used as a benchmark test for proper assembly.[11]

NOTE 1 A leak will change the sampler's performance which will also affect the XRD response.

NOTE 2 Two leak tests are available to assess bypass leakage: the particle count leak test, performed with an optical counter or a condensation particle counter,[11] and the pressure drop leak test, performed with a micro manometer.[12]

5.2.5 Connect the sampling train and set the flow rate to the value specified for the sampler to within ±2,5 %. The maximum deviation in flow rate (before and after sampling) should not exceed 5 %.

5.2.6 For personal sampling in the workplace, attach the sampler to the worker within his or her breathing zone, attach the pump to a belt or harness and connect it to the sampler by a length of flexible tubing, without impeding the comfort of the worker or his or her activity.

5.2.7 From each site, retain a minimum of one unused filter in a sampler, as a field blank.

5.2.8 To begin sampling, switch on the pump and record the time.

5.2.9 A minimum sampling time shall be calculated taking into account the limit of quantification on the X-ray method for RCS (see 8.3) and the flow rate of the sampling system, so that compliance with the OELV can be reliably assessed.

NOTE 1 For compliance testing the full working shift should be sampled, when possible. Longer sampling times improve the measurement precision of all samplers unless overloading occurs.

NOTE 2 Care should be taken not to overload the filter.

NOTE 3 Potential sample losses within the sampler, i.e. sample entering the sampler but not carried through to the filter, has been observed to occur in field sampling, with large variability.[13] Such losses are not visible to the naked eye, and cannot be quantified by a direct on filter method.

5.2.10 At the end of the sampling period, switch off the pump, record the time, and calculate the duration of the sampling period. Verify the volumetric flow rate of the sampling train according to point 5.2.5.

5.2.11 Record the relevant details of the sample collection. The details needed by the laboratory analyst are the following:

a) the type of sampler used to collect the sample;

b) the type of sampling filter;

c) the unique identifier of each sample;

d) the volume of the air sampled;

e) information about the industrial process that may aid evaluation of the results.

5.2.12 Ideally, a sample of dust representative of the workplace or the activity is needed to help the analyst evaluate potential interference. In the absence of information about the materials involved in the industrial process, a heavily loaded filter sample or a settled dust sample can provide sufficient qualitative information. This qualitative scan provides useful information to the occupational hygienist (e.g. when sampling a new process or a process where the materials involved in the work activity may change).

5.3 Transport

Care should be taken to transport aerosol samplers in their upright position, to avoid the possible deposition of dust onto the air sample filter from the grit pot. Cassettes in some samplers can be used to securely transport the filters. Losses of sample can occur, if pressure is applied to the surface of the dust collected on a filter, especially during the transfer of the filter from a sampler cassette or sample container. For example, sample losses can occur if the sample surface comes into contact with tweezers, O-ring seals, or the edge of the sampler. Filters can become charged during sampling and may attract themselves to these items. Losses of dust from the filter surface or found in the cassette shall be noted on the analyst's report.

NOTE A filter load up to 4 mg of the respirable fraction can be transported by postal service without significant loss, provided that a suitable filter holder and a container designed to prevent damage are used.[14]

6 Analytical procedure

6.1 Apparatus and equipment

6.1.1 X-ray diffraction system

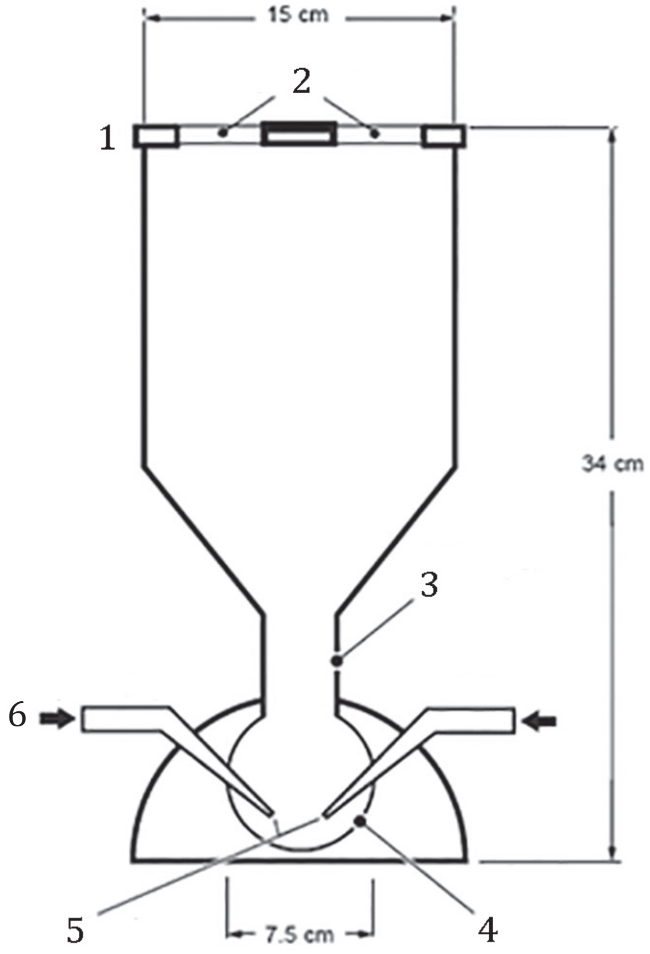
An X-ray powder diffractometer with reflection geometry is needed. Diffractometers with Bragg-Brentano semi-focusing geometry with a copper or cobalt target are commonly used.

6.1.2 Balance

A microbalance capable of weighing ±1 µg or better, over the range 0 g to 5 g is required for the preparation of calibration test samples. An electrostatic eliminator is needed when weighing filters.

6.1.3 Dust cloud generator

A device to generate atmospheres of standard dust and contain them is needed. An example of such a device is given in Figure 1[15]. This device is constructed from borosilicate glass with a lid made from acrylic glass. An aerosol of dust is generated into the upper cylindrical chamber by applying a short burst of pressurized air to a dust contained in a bowl at the bottom. Sampling equipment shall be prepared following 5.2.1 to 5.2.6 and 5.2.10. Samplers are fitted at the top of the device. To avoid agglomerations and charge interactions between the dust and the filter inside the aerosol sampler it is recommended that the device be earthed (grounded).



Key

1	acrylic glass lid
2	holes for cyclone samplers
3	B29/32 cone and socket joint
4	bowl for sample
5	orifice diameter 1 mm
6	compressed air at approximately 50 psi

Figure 1 — Aerosol generation apparatus

6.1.4 Standard reference materials

It is important to use a calibration dust standard in which the purity and crystallinity is well characterized. The United States National Institute of Science and Technology (NIST) have developed Standard Reference Materials (SRM) for respirable quartz (1878 series) and for respirable cristobalite (1879 series). Work by Stacey et al. specifies different reference materials for quartz analysis and their estimated values.[16] The material used for calibration shall conform to the recommendations in ISO 24095.

6.1.5 Reagents

Reagents are not normally required for the direct on filter analysis method.

6.1.6 Drift correction sample

An aluminium plate or other suitable robust material shall be used to correct for the drift in radiation intensity over time. The reference alumina plate NIST SRM 1976b is frequently used.

6.2 Gravimetric analysis for respirable dust

This method assumes that gravimetric measurement for respirable dust is performed before the sample is analysed for RCS and the value recorded. This is important since knowledge about the mass of dust on the filter helps the analyst evaluate the quality of their results and ascertain the potential need for correction due to sample X-ray absorption effects. Some national methods require the determination of the percentage of crystalline silica in the respirable dust. Weighing shall be performed according to ISO 15767.

6.3 X-ray diffraction analysis

6.3.1 Instrumental parameters

The instrument should be optimised for intensity rather than resolution. Ideally, the slits and mask should be designed to allow the radiation of the majority of the filter sample. For example, a good choice would be to set the automatic slits to irradiate 18 mm of the sample area. Broad focus tubes are recommended because they distribute a larger flux/unit area over the filter sample. Power settings of at least 1,6 kW are used although higher power settings give better sensitivity. Examples of instrumental parameters are given in Annex A.

6.3.2 Scan parameters

The scan parameters shall be optimised to achieve a minimum instrumental precision of 4 % or better on a sample with a mass of RCS representative of the OELV. This is achieved by changing the measurement parameters (counting time and step size) to the point at which no further improvement of precision is possible when measuring a filter with a known mass of RCS. The masses at the limit and half the OELV are typically used. Manufacturers provide a variety of software products and may have different approaches for data collection. Generally, a 2 °2θ range is collected around each XRD reflection, although some quantification programs may require a larger scan range. Examples of data collection parameters are given in Annex B.

6.4 Calibration curve

Calibration is performed by preparing calibration test filters loaded with known amounts of RCS and establishing a calibration graph of intensity of the peak versus mass of reference RCS material. The dust shall be deposited on the filters in essentially the same manner as for the analyte samples, by sampling an aerosol of RCS in a dust cloud generation chamber, using the same equipment as stated in 5.2.1 to 5.2.6 and 5.2.10. The amount of RCS deposited is determined from pre- and post-deposition weights, corrected for the crystallinity of the reference material used to load the calibration test filters. For thin loading, up to a critical value for the mass of dust (Annex C), the X-ray intensity response is linear with respect to the mass present on the filter within the area of the filter irradiated by the incident X-ray beam. For greater loading, X-ray absorption can no longer be considered negligible and a specific correction is required (Annex C).

A detailed procedure for calibration curve construction is given in the following sections. Refer to ISO 11095[2] for guidance.

6.4.1 Take at least 12 filters and 3 filters to be used as laboratory blanks, from the same lot and ensure unique identification in a suitable manner.

6.4.2 In accordance with ISO 15767, condition the filters and weigh them to the nearest 0,001 mg. Ensure that three consecutive weighing of the same filter are within 15 µg (1σ). For polycarbonate and silver filters, ensure three consecutive weighing are within 3 µg (1σ).

NOTE A major contribution to the uncertainty of the calibration is the precision (see Reference [1]) of weighing the filters.

6.4.3 The apparatus used to generate any dust aerosol (Figure 1) shall be placed inside a fume cupboard to prevent any potential exposure.

6.4.4 Put a pre-weighed filter into the sampler to be used for the sample collection.

6.4.5 Connect the sampler to the pump. The pump is usually outside the containment apparatus connected by a tube to the sampler.

6.4.6 Generate aerosols of standard dust within the containment apparatus and sample the dust to obtain filters loaded within a minimum range of approximately 0,1 to 2 times the appropriate OELV for the sampler selected over the sampling period normally used at the workplace:

$$Upper\ Limit = OELV\ in \frac{mg}{m^3} \times flow\,rate\,in \frac{l/min}{1000} \times sampling\,time\,in\,min \times loading\ factor\ of\ 2 \tag{1}$$

$$Lower\ Limit = OELV\ in \frac{mg}{m^3} \times flow\,rate\,in \frac{l/min}{1000} \times sampling\,time\,in\,min \times loading\ factor\ of\ 0{,}1 \tag{2}$$

NOTE 1 Filters with a low mass of RCS (<50 µg) are difficult to prepare with good precision and accuracy.

NOTE 2 Caution, do not over pressurize the glass chamber.

6.4.7 Re-weigh the calibration test filters and the laboratory blanks. The mass of reference material deposited on a filter, M_{RM}, is calculated as the difference between pre- and post-weighing, corrected for weight instability. The correction is effected by subtracting the average blank mass change from the mass change of the active samples (ISO 15767).

6.4.8 The mass value of RCS deposited on a filter, M_{RCS}, is calculated by the mass value of the reference material, M_{RM} obtained by weighing, corrected for its crystallinity, X_{RM}

$$M_{RCS} = M_{RM} \cdot X_{RM} \tag{3}$$

6.4.9 Mount each filter in turn in the diffractometer, measure the diffraction intensity of the three most intense reflections of RCS on the calibration test filters

6.4.10 Measure the X-ray intensity drift correction monitor periodically through the batch of samples.

6.4.11 The average area intensity, I_M^0 of a reflection of the drift correction sample shall be recorded and later used for drift correction.

6.4.12 If the mass of dust deposited on the filter exceeds the critical mass, measure the intensity of a reflection from the reference material chosen for absorption correction, with and without the sample in place (Annex C).

6.4.13 Using the intensity value for each reflection (corrected for absorption if necessary), derive a trend line from the relationship between response and mass RCS loaded on the filter. The basic assumption for the construction of the calibration curve is that the calibration function is linear, therefore slope and intercept of the straight line shall be determined.[2]

NOTE 1 Force the trend line through zero or use a regression weighted for the precision of counting. A regression with an intercept can be applied if results are reproducible, e.g. with proficiency test or reference samples.

NOTE 2 Reasons for an intercept significantly different from zero include: the instrumental parameters do not permit the illumination of the whole sample; the peak fitting software has inappropriately introduced a bias

6.5 Sample treatment

No treatment of the sample is usually required before analysis. It is possible to heat the dust on a silver filter in a furnace at 600 °C to remove some organics and clay components. Heating the dust on the silver filter will change the °2θ position and intensity of the silver reflection used for absorption correction

6.6 Sample analysis

6.6.1 A qualitative analysis should be performed on at least one of the samples from each workplace or activity performed to identify any potential interference. Care should be taken not to assume all the samples have the same mineral constituents. For example, stonemasons in the same workplace may work on different stones.

6.6.2 For quantitative analysis, scan as a minimum three major reflections for RCS on each sample, and collect the integrated area for each reflection.

6.6.3 Measure the X-ray intensity drift correction monitor periodically through the batch of samples

6.6.4 If the mass of dust deposited on the filter exceeds the critical mass, measure the intensity of a reflection from the reference material chosen for absorption correction, with and without the sample in place (Annex C).

7 Calculation

7.1 Gravimetric analysis

The mass of dust, M_{dust} on a filter is calculated as:

$$M_{dust} = \left(M_{after\ sampling} - M_{filter} \right) - \overline{\Delta M_{blanks}} \tag{4}$$

where M_{filter}, is the weight of the filter before sampling, $M_{after\ sampling}$, is the weight of filter after sampling, and $\overline{\Delta M_{blanks}}$, is the average blank weight change between after $M_{blank\ after}$ and before $M_{blank\ before}$ sampling:

$$\Delta M_{blank} = M_{blank\ after} - M_{blank\ before} \tag{5}$$

7.2 X-ray diffraction analysis

7.2.1 Calculate the area intensity, I_{RCS} of each XRD reflection of RCS measured in the sample. The background positions shall be chosen consistently and shall be free of interferences.

7.2.2 Correct the area intensity of each XRD reflection of RCS, I_{RCS} for the drift of tube intensity from the original calibration following Formula (6):

$$I_{RCS*} = I_{RCS} \cdot \left(I_M^0 / I_M \right) \tag{6}$$

where I_M is the average area intensity of a reflection of the monitor taken the day of sample analysis, I_M^0 is the average area intensity of a reflection of the monitor when the calibration standards were first measured, and I_{RCS*} ,is the area intensity of the RCS reflection in the sample corrected for the drift of tube intensity.

7.2.3 If the mass of dust deposited on the filter exceeds the critical mass, correct the intensity of each XRD reflection of quartz or cristobalite for absorption, following the procedure given in Annex C.

7.2.4 A main source of error in analysis is the presence of crystalline compounds where some reflections coincide with (or are very close to) quartz or cristobalite. Examples include calcium silicate, biotite, montmorillonite, kaolinite, muscovite or wollastonite. A detailed list of interferences is available in ISO 24095.

7.2.5 Generally, identifying the crystalline components in the dust on a heavily loaded filter assesses the presence of interference. Patterns in the software programs used with X-ray diffraction instruments will indicate if one of the reflection lines of a pattern of another crystalline component coincides with a measurement reflection of the analyte.

7.2.6 Ratios for the intensity of the measured reflections relative to the primary reflection for quartz or cristobalite should be compared with those obtained in the calibration samples. These ratios, recorded in terms of percent relative to the most intense reflection, can be checked to confirm the presence of interferences. Relative ratios that are published in the International Committee on Diffraction Data (ICDD) files should be used as a guideline only. Laboratory calculated ratios and the use of automatic slits in X-ray diffraction instruments may produce relative ratios that are slightly different from those published. The confirmation of the presence of interference can also be determined by assessing the intensity of each measurement with the relative magnitude of the slopes of the calibration trend lines for the measured reflections.

NOTE 1 A difference of more than 10 % to 15 % in area intensities is considered significant.

NOTE 2 The analyst is to check the intensity values produced by the instrument, when values that are inconsistent with other reflections are obtained or the measured value is low, with software that displays the scans.

7.2.7 Quantitative result is calculated on the most intense X-ray reflections of RCS that are free from major interferences, according to the calibration function:

$$I_{RCS*} = a + b \cdot M_{RCS} \tag{7}$$

where I_{RCS*} is the corrected intensity of the RCS reflections, a and b are the intercept and the slope found for the calibration function, and M_{RCS} is the mass of RCS in the sample.

7.3 Concentration of RCS

7.3.1 Calculate the volume, V_S, of each air sample, and the product of the average measured sampling flow rate and the sampling time.

7.3.2 Calculate the concentration of RCS, C_{RCS}, as the mass, M_{RCS}, on the filter divided by the air volume, V_S:

$$C_{RCS} = \frac{M_{RCS}}{V_S} \tag{8}$$

7.3.3 Calculate the concentration of RCS by adding the concentration of all present crystalline polymorphs.

8 Performance characteristics

8.1 Limit of detection

8.1.1 Limit of detection for each X-ray reflection in terms of mass measured on the filter is dependent on the type of instrument, its operating parameters, other components within the sample and the background on the analysis filter.

8.1.2 The use of repeated measurements of blank filters to determine the limit of detection does not necessarily provide a good estimate because the matrix masks the noise from the analysis filter in many real samples. The procedure recommended in this method is to calculate the standard deviation of the count rate of the background over the appropriate scan range and the limit of detection is found when

$$R_A \geq 3 \cdot \sigma_{Rs} \tag{9}$$

where R_A is the count rate of the analyte reflection and σ_{R_s} is the standard deviation of the count rate of background.

Typical limits of detection on calibration test samples containing pure quartz for each measurable X-ray reflection for copper radiation and are listed in Annex D.

NOTE A sample related limit of detection can also be applied where the limit of detection is verified by adding to the analysis filter a mass of analyte that is not observed on a scan or to load typical quantities of the matrix of interest (e.g. 200 µg to 500 µg of calcite) when pure samples of the matrix free from silica contamination are available and to perform measurement on these samples.

8.2 Minimum detectable value

A different performance characteristic of the measurement process is given by the minimum detectable value, determined for each X-ray reflection in terms of mass of RCS measured on the filter, according to ISO 11843.[4] The minimum detectable value shall be calculated using the same calibration data used for routine analysis.

8.3 Limits of quantification

Traditionally, multiplying the standard deviation of measurements obtained by measuring backgrounds on blank filters by 10 sets the limit of quantification. However, the presence of interfering crystalline components will raise the limit of quantification for specific analyte reflections.

NOTE 1 The limit of quantification may also be limited by instrumental factors because the masses analysed on filters, when measuring 0.1 of the OELV, are close to the limit of detection for some XRD reflections when using low flow rate samplers (< 4 l/min). This may also raise the limit of quantification beyond the values obtained through traditional estimates.

NOTE 2 In Europe, EN 482[5] states the maximum uncertainty permitted for sampling and analysis of occupational hygiene measurements between 0,1 to 0,5 times the exposure limit should be within ±50 % and measurements between 0,5 to 2 times the exposure limit should be within ±30 %. In the USA, NIOSH stipulates an expanded accuracy requirement of ±25 %, and the method bias not greater than 10 % for laboratory-based methods.[17] Any measurement with an estimated expanded uncertainty for sampling and analysis greater than 50 % is to be considered as a measurement that is not suitable for any purpose according to EN 482.

8.4 Uncertainty

The method proposed for estimating uncertainty for the measurement of RCS is described in ISO 24095. Annex E provides charts giving the equations for the relationship between the combined sampling and analysis uncertainty when measured on calibration samples. The formulae provide the percentage expanded uncertainty for a measured mass over the analytical range 20 µg to 300 µg.

8.5 Differences between samplers

8.5.1 Samplers can collect significantly different masses of material when sampling the same aerosol, even though most are designed to have a performance that conforms to the ISO 7708 definition for respirable dust. No available sampler for the respirable fraction has a performance that perfectly matches the ideal deposition curve and this causes differences between the masses collected of respirable dust, which will have an impact on the result obtained by X-ray diffraction.

8.5.2 Annex F compares the performance of a selection of samplers used for the measurement of respirable crystalline silica in 2010.

8.6 Differences between analytical approaches

8.6.1 The alternative analytical procedure is to recover the dust from the air sample filter and deposit it onto another filter for analysis (refer to ISO 16258-2). This approach is often used with samplers with larger diameter filters (>25 mm) or when the sampling medium is foam and is termed the indirect analysis approach. Differences exist in the way the instrument is calibrated and there is a need to take into consideration the potential for loss of sample during recovery of dust from the air sample filter. However, when care is taken, the difference in results between the analytical approaches is not significant.

8.6.2 It is not apparent if any advantage exists between the two methods. If PVC filters are used as an air sample filter and a calibration exists for the indirect approach it is possible to use the sample preparation procedures in ISO 16258-2 to verify results.

9 Test report

The test report shall contain at least the following information (ISO 24095):

a) details of the method used, with reference to this part of ISO 16258, i.e. ISO 16258-1;

b) all information necessary for the complete identification of the sample;

c) the condition of the sampling medium such as damage, overloading and any residual dust in the container used for transport;

d) the dust standard used for the calibration;

e) the mass, in micrograms, of analyte;

f) the estimated expanded uncertainty;

g) the LOD and if appropriate the LOQ;

h) the name of the analyst;

i) the name and address of the laboratory;

j) the signature of the laboratory manager or a person delegated by the laboratory manager;

k) the date of the analysis;

l) the name and the model of the instrument used for the analysis;

m) identification of the report;

n) the interferences present during the analysis of the sample;

o) analytical range of the calibration standards;

p) the storage period of the samples;

q) all operating details not specified in this part of ISO 16258, or regarded as optional, together with details of any incident that may have influenced the results.

Annex A
(informative)

Example instrumental conditions

Table A.1 — Examples of instrumental conditions for Bragg-Brentano diffractometers with solid state detectors.

X-ray tube Anode material: Wavelength type: Kα1 (Å) Kα2 (Å)	Cu Kα1 1,540 598 1,544 426			
Voltage (kV):	40	50	40	50
Current (mA):	40	45	40	45
Focus type:	Broad Line	Broad Line	Long Fine Line	Broad Line
Length (mm):	12	12	12	12
Width (mm)	2,0	2,0	0,4	2,0
Incident beam path Radius (mm)	240	240	173	173
Soller slit Opening (rad.)	0,04	0,04	none	0,04
Divergence slit Type: Height (mm):	Fixed 1/2°	Automatic 18	Fixed 1°	Fixed 1°
Mask Width (mm)	15	20	none	none
Sample spinning movement Rotation time (s)	1	1	1	1
Diffracted beam path Radius (mm)	240	240	173	173
Anti-scatter slit Type: Height (mm):	Fixed 1°	Automatic 18	Fixed 0,2 mm	Fixed 0,3 mm
Monochromator	none	none	Diff. Beam Curved graphite	Diff. Beam Curved graphite
Type:			Asymmetric	Asymmetric
Shape:			Flat	Flat
No. of reflections:			1	1
Detector Type: Active length (°)	Solid state pixcel detector 3,347	Solid state array detector 2,12	Point detector	Point detector

Annex B
(informative)

Data collection parameters

Table B.1 — Data collection parameters

	Proportional/Scintillation Detector	Array Detector
Qualitative scan	From 6 to 65 °2θ at 1 °2θ/min	From 6 to 65 °2θ in 0,02 °2θ steps for a minimum of 15 s/step
Silver Reference, Ag(111) at 38,2 °2θ or Aluminium plate, Al(111) at 38,5 °2θ	From 37,2 ± 0,3 °2θ to 39,5 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 4 s/step	From 37,2 ± 0,3 °2θ to 39,5 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 120 s/step
Quartz (101) at 26,6 °2θ Relative intensity≈100	From 25,6 ± 0,3 °2θ to 27,6 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 8 s/step	From 25,6 ± 0,3 °2θ to 27,6 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 180 s/step
Quartz (100) at 20,9 °2θ Relative intensity≈20	From 19,9 ± 0,3 °2θ to 21,9 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 12 s/step	From 19,9 ± 0,3 °2θ to 21,9 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 240 s/step
Quartz (112) at 50,2 °2θ Relative intensity≈12	From 49,2 ± 0,3 °2θ to 51,2 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 12 s/step	From 49,2 ± 0,3 °2θ to 51,2 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 240 s/step
Cristobalite (101) at 22,0 °2θ Relative intensity≈100	From 21,0 ± 0,3 °2θ to 23,0 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 12 s/step	From 21,0 ± 0,3 °2θ to 23,0 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 240 s/step
Cristobalite (200) at 36,1 °2θ Relative intensity≈12 and Cristobalite (112) at 36,4 °2θ Relative intensity≈5	From 35,1 ± 0,3 °2θ to 37,4 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 12 s/step	From 35,1 ± 0,3 °2θ to 37,4 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 240 s/step
Cristobalite (102) at 31,4 °2θ Relative intensity≈9	From 30,4 ± 0,3 °2θ to 32,4 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 12 s/step	From 30,4 ± 0,3 °2θ to 32,4 ± 0,3 °2θ in 0,02 °2θ steps for a minimum of 240 s/step

Annex C
(normative)

Corrections for absorption

When there is a mass of material deposited on the analysis filter greater than the critical mass the result obtained by the instrument needs correction for the absorption of the copper radiation from the sample matrix. The reduction in the intensity of the X-ray reflections of a plate sample is used to measure absorption.[18]

A correction can be applied to the measurement of RCS to compensate for X-ray absorption effects when the loading is very high or contains a significant proportion of an element that absorbs the radiation employed for analysis. The "critical mass for a sample", m_{cr}, is the value for which the mass calculated by XRD without the correction for absorption deviates from the theoretically "true" mass by a certain set threshold amount. This method sets the threshold at a deviation of 10 %. For a given radiation wavelength (typically, CuKα), the critical sample mass for a weight fraction of quartz, depends on the area of deposition on the filter and the mass absorption coefficient of the sample. A practical guide to check the "critical sample mass" for a wide range of possible sample characteristics is provided in Figure C.1, for copper radiation, if a cyclone sampler is used for sampling (collection diameter on the filter 18 or 21 mm for example). In these cases a mass absorption coefficient of 100 cm^2/g, which represents a highly absorbing sample, requires the application of correction for absorption when the dust mass on the filter is more than 1,0 mg (deposition diameter: 21 mm) or 0,5mg (deposition diameter: 18 mm). To apply to a specific sampler it is recommended to perform your own tests following the procedure in Mecchia et al.[19]

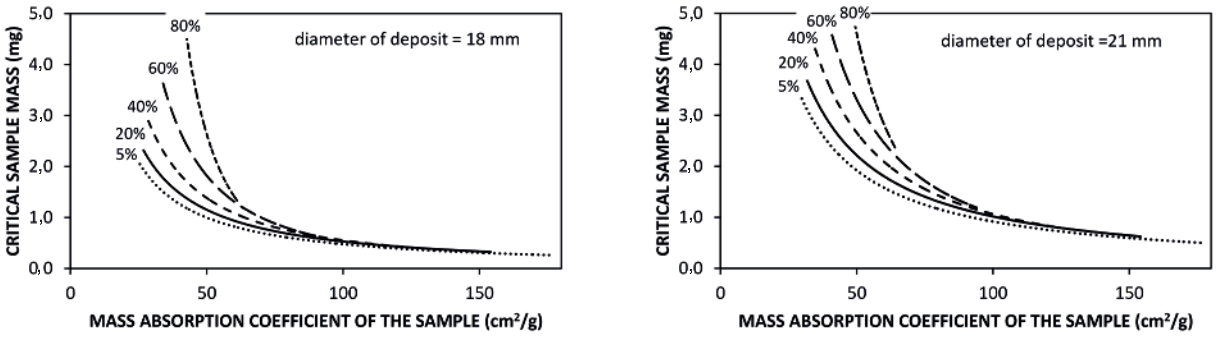


Figure C.1 — Critical sample mass which causes an underestimation of 10 % in the determination of quartz concentration, for a wide range of mass absorption coefficient of the sample (copper radiation). Five percentages of quartz in the mixture are represented.

Where absorption correction is required, either one or both of the X-ray reflections for silver (111 or 200) are measured on silver filters, before sampling or sample analysis. The main reflections from an aluminium or silicon plate can also be used to correct for absorption

When a silver filter is used as an analysis filter, the average intensity from five blank filters from the same batch can be measured as an initial intensity not affected by absorption.

Where a polycarbonate filter is used as the analysis filter, it is placed on top of a silver filter or other material used as a plate and the intensity from the reflections of silver (or the metal used as backing plate) is measured through the sample and filter. This is possible because polycarbonate does not significantly absorb copper X-rays.

Where a PVC filter is used the plate reflections through each filter should be measured before sampling because this filter type does absorb X-rays significantly.

The intensity of the measurement, adjusted for instrumental drift I_{RCS^*} is corrected using the Formula (C.1). In this example, silver is used to estimate the reduction in intensity:

$$I_{RCS^{**}} = I_{RCS^*}\left[-R\frac{\ln T}{\left(1-T^{R}\right)}\right] \tag{C.1}$$

where

R is the ratio

$$\frac{\sin\theta_{Ag}}{\sin\theta_{i}} \tag{C.2}$$

where $\sin\theta_{Ag}$ is the sine of the angle in θ of the silver line used for correction and $\sin\theta_{i}$ is the sine of the angle in θ of the X-ray diffraction reflection measured for the analyte.

$\ln T$ is the natural logarithm of the transmittance

$$\frac{I_{Ag}}{I_{Ag}^{0}} \tag{C.3}$$

where I_{Ag} is the measured intensity of the silver reflection through the sample and I_{Ag}^{0} is either the intensity of the silver reflection before the dust is placed on top or the average intensity of five blank silver filters from the same batch as the silver filter used in the correction.

The mass represented by the corrected for absorption intensity, $I_{RCS^{**}}$ is then calculated by applying the calibration factors.

An example for a deposit on a polycarbonate filter with an aluminium backing plate used for the correction is given below.

The intensity of a measurement of quartz for the (100) reflection (20,9 °2θ peak) was obtained as I_{RCS^*} 45,6 counts per second (cps) and the ratio of the intensity of an aluminium plate measured before and after the sampling was 0,863.

Therefore, $\ln T$ = −0,1473.

The $\sin\theta$ for the aluminium reflection at 38,5 °2θ is 0,329 and the $\sin\theta$ for the quartz (100) reflection at 20,9 °2θ is 0,18.

Therefore, R is 1,82, so, $I_{RCS^{**}} = 45{,}6\cdot\left[\frac{-1{,}82\cdot\left(-0{,}1473\right)}{1-0{,}863^{1{,}82}}\right]$

and, $I_{RCS^{**}} = 45{,}6\cdot 1{,}140 = 52{,}0\ \text{cps}$.

Annex D
(informative)

Range of typical detection limits

Table D.1 — Range of typical detection limits

Quartz			
XRD reflection	100	101	112
Range (μg)	10 to 20	3 to 9	15 to 25
Cristobalite			
XRD reflection	101	102	200 and 112
Range (μg)	5 to 15	50 to 70	35 to 60

These typical limits of detection are consensus values obtained from various international laboratories.

Annex E
(informative)

Typical expanded uncertainty for the direct-on-filter method

The example in Figure E.1 obtained from reproducibility data assumes the contribution to the uncertainty from sampling for an 8 h sample is consistently 10,1 % across the analytical range.

These data were obtained from an X-ray diffraction instrument using automatic slits irradiating an area of 18 mm and may be different when using fixed slits.

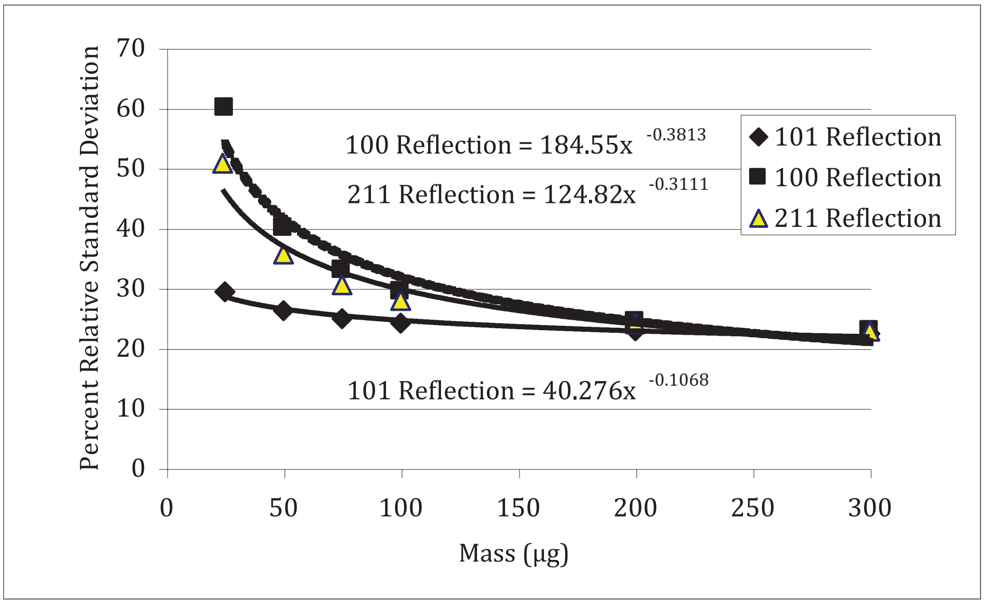


Figure E.1 — The expanded uncertainty (including sampling with a SIMPEDS cyclone) for the 3 principle reflections of quartz on silver filters

The uncertainty was determined according ISO 24095.

Annex F
(informative)

Differences between samplers

It is not uncommon, even within the same company, to discover that several different sampler types were used to assess the same task. Some national methods mention a range of samplers and, although each sampler is designed to conform to ISO 7708 sampling penetration curve for respirable dust, and many have a close match, none have the same performance. As differences in performance affect the mass of dust collected by the sampler this can cause differences in the mass of analyte measured and hence results for RCS obtained by laboratories. The table below compares the performance of respirable samplers in common use with direct-on-filter methods for the analysis of RCS when sampling dust in a calm air chamber with low wind speeds (0,3 m/s or less). The differences are related to the consensus value of all the samplers in the test run. The results are based on the sampling of ultra-fine and medium Arizona road dust, which contains a significant percentage of quartz. Generally, the performance of samplers is dependent on wind speed and the median and the standard deviation of the particle size range. Therefore these results are indicative.

Table F.1 — Differences of samplers from an average air concentration in calm air [20]

Variable	Flow Rate (L/min)	Difference	95 % Confidence level	Difference	95 % Confidence level
Sampler		Medium ARD		Ultra-fine ARD	
SKC Conductive	2,2	+34,0 %	±11,0 %	+24,3 %	±11,4 %
SKC "non corrosive"	2,2	+5,5 %	±13,1 %	+3,1 %	±8,1 %
Envirocon GX-1	2,2	+4,0 %	±7,8 %	+1,5 %	±8,0 %
SIMPEDS	2,2	+3,5 %	±9,2 %	−6,1 %	±4,7 %
BCIRA	2,2	−0,6 %	±10,2 %	−3,6 %	±8,0 %
GS3	2,75	−10,5 %	±8,8 %	−9,2 %	±4,8 %
IOM with PUF Insert	2,0	−19,0 %	±7,0 %	−17,9 %	±5,8 %

The samplers listed in this table are not exhaustive. The samplers should be used with 25 mm filters and should comply with the requirements of ISO 7708. These results are applicable to the flow rates specified by the supplier of the sampler and may be different if another flow rate is used. Please note that these results are applicable to calm air conditions used in the test and varying performances may be obtained in workplaces due to different particle size distributions and environmental conditions.

Please refer to the ASTM Special Technical Publication (STP) 1565 *Silica and Associated Respirable Mineral Particles* for publications on the performance of respirable dust samplers.

The information provided does not constitute an endorsement by ISO of any of the products mentioned.

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