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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

Reference number
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Foreword

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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 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.

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[7]. 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]

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^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.

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 ±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 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

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	Weigh stability index[a]	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

[a] 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.

5.1.2.7 Weighing should be performed following ISO 15767. Filters shall not be weighed in cassettes as large weight variations have been reported.[21] Reference shall be made to the instructions of the collection substrate manufacturer.

5.1.2.8 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.

5.1.2.9 A silver filter used for sampling allows an effective correction for absorption in case a heavily loaded sample is analysed (Annex D). 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 2).

5.1.2.10 High values for back pressure can compromise the sampling time, when the sampling of a complete 8-h shift is desired. Table 2 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]

5.1.2.11 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 2 — Filter materials for XRD analysis

Filter Material (pore size)	SAMPLING	WEIGHING	XRD ANALYSIS								
	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[8]. 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 collection substrate (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 collection substrate, 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 mass of respirable dust sampled.

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]

NOTE 3 The leak test is not applicable to the CIP 10-R

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 collection substrate 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 (8.3) and the flow rate of the sampling system, so that compliance with the OELV can be reliably assessed.

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.

Care should be taken not to overload the collection substrate.

NOTE Potential sample losses within the sampler, i.e. sample entering the sampler but not carried through to the collection substrate, 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 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 collection substrate;

c) the unique identifier of each sample;

d) the volume of the air sampled;

e) weight of the respirable dust on the collection substrate in order to determine the recovery after redeposition;

f) 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 Instrument

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

For the preparation of calibration samples a microbalance capable of weighing ±1 µg or better over the range 0 g to 5 g is required. An electrostatic eliminator is needed when weighing collection substrates. For the weighing of foams from the CIP 10-R sampler a balance with an analytical sensitivity of 10 µg with an operational range between 0 g to 20 g is required. Weighing should be performed according to ISO 15767.

6.1.3 Laboratory equipment

Platinum or glazed ceramic crucibles, beakers, tongs, calibrated pipettes, ultrasonic bath, magnetic stirrer, apparatus to filter a sample onto a 25-mm diameter filter, a pump to generate vacuum, and fume cupboard to contain dusts, vapours and gases.

6.1.4 Equipment to recover dust from the air sample filter

Furnace capable of operating at a minimum of 600 °C (to remove the filter membrane) or a low temperature plasma asher. Tetrahydrofuran (THF) can be used for the dissolution of PVC filters instead of a plasma asher or furnace.

NOTE To remove interfering substances temperatures of up to 1 000 °C may be required.

6.1.5 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.

6.1.6 Reagents

6.1.6.1 Suspension

Deionised water

2-Propanol

Ethanol

6.1.6.2 Filter dissolution

Tetrahydrofuran (THF)

1,3-Butanediol (if using cellulose nitrate air sample filters)

6.1.6.3 Removal of interferences

0,1 N to 1 N hydrochloric acid

6.1.7 Drift correction sample

An aluminium plate or other suitable robust material must 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 B.

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 C.

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 from aliquots of a suspension of standard RCS. The amount of RCS deposited is determined by 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 D), 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 D).

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

6.4.1 Prepare at least two suspensions of the analyte by weighing mg amounts of the standard material to the nearest 0,01 mg.

6.4.2 Calculate the mass of silica in µg/mL of suspension as an aid to plan the aliquots for the calibration filters.

6.4.3 Suspend the powder in the reagent with an ultrasonic probe or bath for a minimum of 3 min. Immediately move the suspension to a magnetic stirrer (without heat), add a stirring bar and allow the suspension to return to become homogenous before withdrawing aliquots.

6.4.4 In accordance with ISO 15767, condition 25-mm diameter 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 (1 σ).

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

6.4.5 Mount the filter on the filtration apparatus. Place 2 ml to 3 ml of reagent on the filter. Turn off the stirrer and shake the suspension by hand. Immediately withdraw an aliquot from about the centre of the suspension and transfer it from the pipette to the filter funnel. Eject the volume from the pipette into the filter funnel. Wash the sides of the funnel several times with small amounts of solvent.

NOTE 1 The diameter of the filter funnel and suction area below the filter will have an influence on the area of deposition of the sample and hence the sensitivity of the calibration. Filtering apparatus can vary in both respects and it's important to use apparatus with similar dimensions.

NOTE 2 When using PVC filters, select the rough side of the filter for the redeposition.

6.4.6 Apply vacuum and rapidly filter the suspension. Do not wash down the sides of the funnel after the deposit is in place since this will rearrange the material on the analysis filter. Leave vacuum on until filter is dry. Remove the filter and allow it to dry. When thoroughly dry, reweigh the filter to determine the mass of deposit.

6.4.7 Prepare a series of calibration filters to obtain a minimum range of approximately 0,1 to 2 times the appropriate OELV for the collection substrate selected over the sampling period normally used at the workplace:

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

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

6.4.8 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).

6.4.9 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}:

$$M_{RCS} = M_{RM} \cdot X_{RM} \quad (3)$$

6.4.10 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

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

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

6.4.13 If the mass of dust redeposited 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 D).

6.4.14 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.[14]

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

NOTE 3 Polycarbonate filters provide very flat surfaces and may cause preferred orientation for the 26,6 degree quartz reflection, which results in a curved, rather than a straight line relationship between X-ray response and the mass measured at low (<200 µg) masses. A method used to determine preferred orientation is to add 1,5 mg of calcium fluoride (CaF_2) to each calibration standard to simulate the deposition of a 'real' sample.

6.5 Sample treatment of collection substrate

Ensure that all operations involving flammable solvents are performed in a fume cupboard or under air extraction. Take the sample collected on the air filter and remove interferences. Refer to some sample treatment strategies outlined in Annex A. Then use the appropriate treatment based on type of collection substrate.

— For PVC and MCE, filters follow the instructions in 6.5.1.

— For cellulose nitrate filters, follow the instructions in 6.5.2.

— For polyurethane foams, follow the instructions in 6.5.3.

6.5.1 PVC and MCE filters

Use either the furnace treatment (6.5.1.1) or plasma asher (6.5.1.2) to ash sampled filter. As .an alternative, treatment with tetrahydrofuran to dissolve sampled filter can also be chosen (6.5.1.3).

6.5.1.1 Furnace treatment

a) Place each filter into a clean crucible and heat in a furnace at 450 °C to 600 °C for a minimum of four hours.

b) If graphite is present refer to Annex A for sample treatment strategies.

c) Collect the sample once the filter is ashed and allow the crucible to cool to room temperature.

d) Continue with the procedure in 6.6.1.

6.5.1.2 Plasma ashing

a) The plasma asher must have a minimum power rating of 3 W and a restricted air intake to ensure that the sample dust is not lost when the vacuum is removed.

b) Place the air sample filter into a clean glass bottle or beaker.

c) Cover the open end with perforated foil to allow the gasses used for the plasma to circulate and to restrict any potential dust losses.

d) Ash the sample to ensure the residue from the air sample filter is minimised.

e) Continue with the procedure in 6.6.2.

NOTE Plasma ashing will leave a small residue from the filter ~0,1 %.

6.5.1.3 Treatment with tetrahydrofuran (THF)

a) Place the PVC filter in a small thin beaker, or centrifuge tube, or glass bottle with a lid.

b) In a fume cupboard, and while taking precautions to protect the hands from contact with the solvent, add 8 ml to 10 ml of THF.

c) Cover the end of the receptacle and agitate the solvent, either by hand, or buy using a vortex mixer, taking care not to spill its contents.

d) Then place in an ultrasonic bath until the filter is dissolved.

e) Continue with the procedure in 6.6.2 using THF as the reagent.

6.5.2 Cellulose nitrate filters

a) Place the filter carefully into a clean crucible.

b) In a fume cupboard, wet the filter with a small amount of 1,3-butanediol.

c) Place in a furnace under an air extraction hood at room temperature.

d) Do not heat the furnace before introducing the samples. Wear safety glasses.

e) Heat slowly from room temperature to 450 °C to 600 °C for a minimum of four hours.

f) Collect the sample and allow the crucible to cool to room temperature.

g) Continue with the procedure in 6.6.1.

6.5.3 Polyurethane foams

a) Place an empty crucible in a muffle furnace at 450°C to 600 °C for 10 min to 15 min, allow to cool in a desiccator for a minimum of four hours and then weigh. Record the weight (M_1) accurate to the nearest 0,01 mg.

b) Remove the foam from the cassette and place into the cleaned pre-weighed crucible.

c) Wash the cassette out by rinsing it with 2 ml to 3 ml of reagent (2-propanol or ethanol) at least twice into the crucible. Rinse into the crucible any items that may have come into contact with the sample, e.g. tongs.

d) Ensure the foam is covered with reagent.

e) Ignite the reagent in the crucible.

f) When the foam is melted place it in a furnace at 200 °C to 300 °C for 30 min and then progressively raise the temperature to 550 °C to 650 °C for a minimum of three hours.

g) Allow the crucible to cool in a desiccator and then reweigh the crucible after conditioning in a weighing room (M_2) accurate to the nearest 0,01 mg.

h) The residue is calculated as $M_2 - M_1$. Record and make a note of any low recovery.

i) Continue with the procedure in 6.6.1

6.6 Redeposition onto analysis filter

6.6.1 Crucibles from the furnace

6.6.1.1 Let the crucibles cool in an area free from possible contamination.

6.6.1.2 Weigh a 25-mm diameter analysis filter to the nearest µg.

6.6.1.3 Set up a filtration apparatus suitable for use with 25-mm diameter filters.

6.6.1.4 Place the crucible containing the sample residue into a beaker and cover with sufficient reagent (3 ml to 5 ml).

6.6.1.5 Cover the beaker with a watch glass.

6.6.1.6 Ultrasound for 3 min to 5 min.

6.6.1.7 Wash watch glass into the beaker.

6.6.1.8 Carefully wash contents of crucible into the beaker

6.6.1.9 Filter contents of beaker on to a pre-weighed filter.

6.6.1.10 Allow the filter to dry in air.

6.6.1.11 Reweigh the filter after conditioning and determine the recovery from the gravimetric weight recorded from the sampling.

6.6.1.12 Analyse the sample following the procedure in 6.7.

6.6.2 Bottles or beakers from a plasma asher

6.6.2.1 Remove foil or perforated lid from the top of the glass bottle or beaker.

6.6.2.2 If soluble components are present refer to Annex A for sample treatment strategies.

6.6.2.3 Add an amount of reagent to the bottle and carefully wash the sides.

6.6.2.4 Do not spray solvent directly onto the sample as this may aerosolise the dust sample.

6.6.2.5 Put the lid on the bottle or a watch glass on top of the beaker and ultrasound for about 5 min to 10 min.

6.6.2.6 Filter the residue onto a 25-mm diameter filter.

6.6.2.7 Analyse the sample following the procedure in 6.7

6.7 Sample analysis

6.7.1 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.

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

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

6.7.4 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 D).

7 Calculation

7.1 Gravimetric analysis

The mass of dust, M_{dust}, on a collection substrate or redeposition filter (i.e. medium) is calculated as:

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

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

$$\Delta M_{blank} = M_{blank\ after} - M_{blank\ before} \quad (5)$$

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 the equation:

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

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 D.

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 shall 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

$$R_A \geq 3 \cdot \sigma_{Rs} \quad (9)$$

where R_A is the count rate of the analyte reflection and σ_{Rs} 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 E.

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[7] 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, National Institute of Safety and Health (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 % should 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 F provides charts giving the equations for the relationship between the combined sampling and analysis uncertainty when measured on calibration samples. The equations 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 G compares the performance of a selection of samplers used for the measurement of respirable crystalline silica in 2010.

8.6 Differences between analytical approaches

An alternative analytical approach is available when using a sampler with a 25-mm diameter air-sampling filter, where the sample is analysed without any treatment to recover the dust and place it on an analysis filter (refer to ISO 16258-1). Differences exist in the way the instrument is calibrated and the need to use the sampler involved in the sampling exercise to prepare calibration samples. Taking into consideration the uncertainty of the sampling and the analysis (when using the same sampler), for most matrices, the difference in results between analytical approaches is not significant.

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 16258-2;

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
(normative)

Sample treatment strategies

The removal of interference is not always needed (e.g. when the interference is a trace component).

A.1 Clay minerals

Kaolin is a common interference in mineral samples, which can decompose in temperatures as low as 450 °C. For other kaolinite minerals like nacrite and dickite the temperature for decomposition may be as high as 650 ± 50 °C, although it might require a temperature of 800 °C for their complete removal. Care should be taken when heating quartz to temperatures higher than 800 °C for any length of time. Although the conversion temperature from quartz to cristobalite is about 1 100 °C, partial conversion is observed in some samples in temperatures above 800 °C. Some clay samples from industrial sources may also contain calcite. Calcite should be removed with dilute acid following paragraph A.3 before heating samples in a furnace. The formation of wollastonite from the reaction with calcite and silica can occur at temperatures as low at 500 °C.

Use a furnace temperature within ±50 °C appropriate for the clay matrix.

A.2 Coal and similar carbon based materials

Some carbon based components (e.g. anthracite or graphite) and organic matter can be removed by ashing in a furnace at about 800 ± 50 °C. However, the heating process can potentially oxidize other components in the sample (e.g. iron) and add to the complexity of the analysis.

A.3 Removal of acid soluble components

If necessary, calcite, dolomite, gypsum, iron and its oxides can be removed with the use of dilute hydrochloric acid (HCl). Iron has a high absorption for copper X-ray radiation so its removal is beneficial if the sample is mostly hematite, magnetite or iron metal.

A.3.1 Calcite, gypsum, dolomite, and other similar minerals

Find an unused air-sampling filter of a diameter suitable for use with filtration apparatus (25 mm).

Set up the filtration equipment with the 25 mm filter.

Place the air sample filter in the funnel of the filtration apparatus.

Add 0,1 – 1N HCl and wait for 5min to 8 min.

Apply the vacuum to the filter.

Dry the filter and follow the relevant procedure in 6.5 replacing the word water for solvent.

A.3.2 Iron and iron compounds

Treat the air sample filter in a furnace or plasma asher to remove the air sample filter and allow the crucible or bottle to cool.

Add 5 ml to 10 ml of 0,1 – 1N HCl.

Allow to cool at a temperature of 50 °C to 60 °C for a suitable length of time (approximately 2 h).

Redeposit the sample following 6.6 replacing the word deionised water for reagent.

Annex B
(informative)

Example instrumental conditions

Table B.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 Type: Shape: No. of reflections:	none	none	Diff. Beam Curved graphite Asymmetric Flat 1	Diff. Beam Curved graphite Asymmetric Flat 1
Detector Type: Active length (°)	Solid state pixcel detector 3,347	Solid state array detector 2,12	Point detector	Point detector

Annex C
(informative)

Data collection parameters

Table C.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 D
(normative)

Correction 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 D.1, for copper radiation, when a sample is redeposited and a deposition diameter on the filter is 15 mm for example. In this 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 0.9 mg. To apply to a specific sampler it is recommended to perform your own tests following the procedure in Mecchia et al.[19]

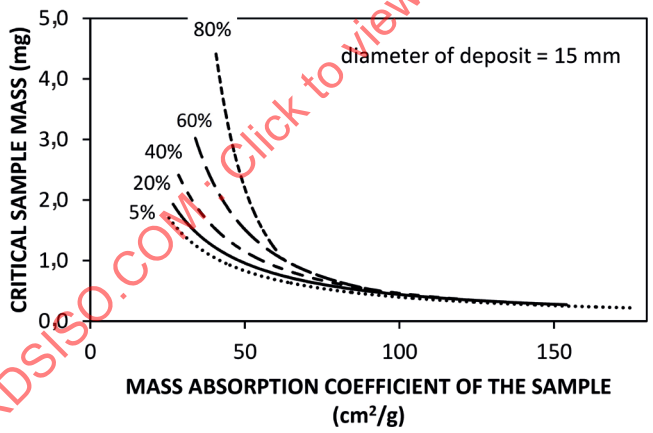

Figure D.1 — Critical sample mass which causes an underestimation of 10 % in the determination of quartz concentration, for a wide range of mass absorption coefficient of the sample (copper radiation). Five percentages of quartz in the mixture are represented

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.