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Workplace air — Analysis of respirable crystalline silica by Fourier-Transform Infrared spectroscopy

Air des lieux de travail — Mesure de la fraction alvéolaire de la silice cristalline par spectrométrie infrarouge

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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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This document was prepared by Technical Committee ISO/TC 146, *Air quality*, Subcommittee SC 2, *Workplace air*.

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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, their respiratory protection, or the effectiveness of controls. Fourier-Transform Infrared (FTIR) analysis of crystalline silica in a sample of respirable dust collected on a collection substrate is employed in many countries to measure and estimate exposure to RCS. FTIR is able to measure quartz and cristobalite.

This document specifies the analysis procedures for the measurement of RCS through three methods:

a) Direct-on-filter method: a method of analysing RCS directly on the air sample filter. A specific requirement of this method is that the sampler used for the workplace measurements is the same as that used for the preparation of calibration samples.

b) Indirect method by redeposition: a method whereby the dust is recovered from the collection substrate and deposited onto a filter for analysis.

c) Indirect method by potassium bromide (KBr) pellet: a method whereby the dust is recovered from the collection substrate and pressed into a potassium bromide (KBr) pellet for analysis.

Many different types of sampling apparatus are used to collect respirable dust, according to the occupational hygiene convention. This document is designed to accommodate the variety of samplers and collection substrates available to analysts. This document is used in conjunction with ISO 24095 which promotes best practice for these analyses.

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Workplace air — Analysis of respirable crystalline silica by Fourier-Transform Infrared spectroscopy

1 Scope

This document is a standard for the analysis by Fourier-Transform Infrared (FTIR) of respirable crystalline silica (RCS) in samples of air collected on collection substrates (i.e. filters or foams). Three analytical approaches are described for whom the dust from the sample collection substrate is

a) analysed directly on sampled filter,

b) recovered, treated and deposited onto another filter for analysis, or

c) recovered, treated and pressed into a potassium bromide (KBr) pellet for analysis.

This document provides information on the instrumental parameters, the sensitivity of different sampling apparatus, the use of different filters and sample treatment to remove interference. In this document the expression RCS includes the most common polymorphs quartz and cristobalite.

This document excludes the less common polymorphs of crystalline silica, such as tridymite.

Under certain circumstances (i.e. low filter dust loads, low silica content), the analytical approach described in this method cannot fulfil the expanded uncertainty requirements of ISO 20581. Guidance for calculation of uncertainty for measurements of RCS is given in ISO 24095.

2 Normative reference

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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*

EN 13205, *Workplace exposure — Assessment ofsampler performance for measurement of airborne particle concentrations*

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

ISO 18158, *Workplace air — Terminology*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 18158 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at http://www.electropedia.org/

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3.1 General definitions

3.1.1

respirable crystalline silica

RCS

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

3.2 Sampling definitions

3.2.1

analysis filter

suitable filter used to carry out the RCS analysis

Note 1 to entry: For direct-on-filter FTIR analysis the collection substrate is the analysis filter.

Note 2 to entry: For the indirect analysis the dust is removed from the collection substrate and redeposited onto an analysis filter.

3.2.2

collection substrate

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

Note 1 to entry: For the purpose of this document, filters, polyurethane foams and sampling cassettes are examples of collection substrates for airborne particles.

4 Principle

This document provides three Fourier-Transform Infrared (FTIR) analytical approaches for the analysis of RCS in respirable dust collected on a sample collection substrate.

a) Direct-on-filter method:

The respirable dust on the collection substrate (usually a 25 mm diameter filter) is measured without transferring the dust to an analysis filter. The mass of RCS is determined from the FTIR response, calibrated against filters loaded with known amounts of RCS reference material.

b) Indirect method (redeposition):

The dust from the sample collection substrate (i.e. a 37 mm diameter filter or a polyurethane foam) is recovered, treated and deposited on another analysis filter (usually 25 mm diameter or smaller) for analysis by the instrument. The instrument is calibrated by preparing test samples from aliquots of a suspension of a reference material. The mass of RCS is determined from the FTIR response, calibrated against analysis filters loaded with known amounts of RCS reference material.

c) Indirect method (KBr pellet):

The dust from the sample collection substrate (i.e. a 37 mm diameter filter or a polyurethane foam) is recovered, treated and pressed into a KBr pellet for analysis by the instrument. The instrument is calibrated by preparing KBr pellets with known amounts of a reference material. The mass of RCS is determined from the FTIR response, calibrated against pellets 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 FTIR instruments used for the direct-on-filter analysis approach shall be calibrated for the aerosol sampler used to collect the samples.

The suitability of FTIR to determine the RCS concentration in a workplace sample depends on the composition of the dust and potential interferences. If the FTIR method to subtract a reference spectrum from the sample spectrum (refer to 9.3.2) does not lead to a satisfactory baseline profile, then

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the FTIR method is not suitable for the matrix and quantitative FTIR analysis cannot be carried out. As an alternative XRD analytical method should be used. Qualitative X-ray diffraction (XRD) analysis can be carried out prior to FTIR analysis to obtain information about the sample.

5 Apparatus, equipment and reagents

5.1 Sampling equipment

5.1.1 Samplers.

5.1.1.1 The particle size-selection performance of the samplers used shall match the criteria for respirable dust as specified in ISO 7708 according to the test protocol in EN 13205-2.

5.1.1.2 Samplers using a filter or foam pad are suitable for this method.

NOTE Annex B provides information on the performance of different personal respirable samplers that are currently in use.

NOTE Foam pads and silver membrane filters are not suitable for direct-on-filter method.

5.1.1.3 Cassettes holding filters shall be made of conductive material.

5.1.1.4 Each sampler should be labelled with a unique number, in order to identify samplers that start to underperform after long-term use.

5.1.1.5 Samplers (sampling train) shall comply with the requirements for calibration in ISO 13137.

NOTE In some countries there can be exceptions due to national regulations.

5.1.2 Collection substrates and analysis filters.

5.1.2.1 Filters as collection substrates shall be of a diameter suitable for use in the selected sampler and have a capture efficiency for respirable particles as specified in ISO 24095.

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. The collection substrates generally used for the sampling of RCS, and their characteristics, are listed in Annex D.

NOTE Additional information about choice of filter material for quartz measurement in coal mine dust can be found in Reference [24].

5.1.2.3 Filter materials listed in Annex D generally do not contain compounds that interfere with the measurement of quartz and cristobalite. Impurities can be introduced during the filter manufacturing process and background absorbance 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 Variable background has an effect on the readability of absorbance spectra, increasing the limit of detection for RCS. PVC and polypropylene used as analysis filters exhibit the least variability and lowest background levels and thus are useful in situations where low limits of detection are required.

5.1.2.5 Weighing, if required, should be performed following ISO 15767 (see 8.1). Filters shall not be weighed in cassettes as large weight variations have been reported [18]. Reference shall be made to the instructions of the collection substrate manufacturer.

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5.1.2.7 An important property for an analysis filter is that it is transparent to infrared and provides a relatively low background variation near the absorbance for crystalline silica.

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 includes 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 or foam). 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;

d) 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 Equipment for calibration

5.2.1 Dust cloud generator (needed for direct-on-filter analysis).

A device to generate atmospheres of reference material and contain them is needed when following the direct-on-filter analytical approach. An example of such a device is given in Figure 1. 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 6.1.1 to 6.1.6 and 6.1.9. 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).

NOTE An Aerosol generation device can also be useful for preparing samples for indirect FTIR methods.

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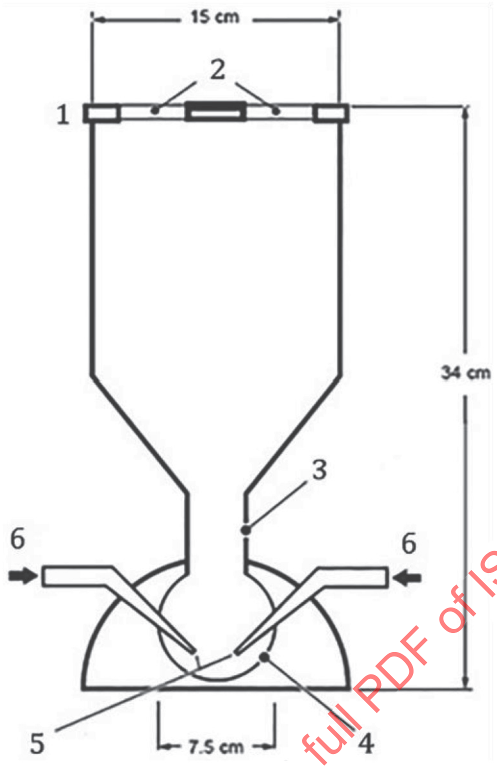
Dimensions in centimetre



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 — Example of an aerosol generation apparatus

5.2.2 Laboratory equipment to prepare suspensions.

For the preparation of calibration samples, suspensions with a defined content of quartz dust can be used. Volumetric flasks of 100 mL and 250 mL, a bath thermostat with a built-in cooling coil, working temperature range shall include 20 °C, microlitre pipettes of variable volumes, with a range of between 50 µL and 1 000 µL, an Erlenmeyer flask and a magnetic stirrer.

5.3 Equipment for the determination of dust concentration

5.3.1 Balance.

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

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5.4 Equipment for sample preparation

5.4.1 Redeposition 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 a fume cupboard to contain dusts, vapours and gases.

5.4.2 KBr pellet laboratory equipment.

Platinum or glazed ceramic crucibles, tongs, a pump to generate vacuum, a boron carbide mortar and pestle, a 50-mm agate or metal microspatula, a laboratory press for preparing KBr pellets, a KBr pellet evacuable die (typically 13 mm), a pump to generate vacuum, a desiccator with silica gel desiccant, a hair brush with anti-static effects (e.g. badger or camel hair) and glassine paper.

5.4.3 Equipment to recover dust from the collection substrate.

A furnace capable of operating at a minimum of 600 °C or a low temperature plasma asher to remove the filter membrane. 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. To prevent reactions occurring between silica and calcium carbonate at high temperatures, the latter may be removed by washing with hydrochloric acid (5.6.2.3).

5.5 Equipment for analysis

5.5.1 Spectrometer.

A FTIR spectrometer with a wavelength range from at least 4 000 cm^{-1} to 400 cm^{-1} and 4 cm^{-1} resolution or better. A suitable sample holder (e.g. a rotatable polarizer mount) is required so that the sample can be rotated in its own plane. This will enable the effect of non-uniform sample deposition to be reduced by taking absorbance measurements at several orientations. If the infrared beam in the instrument has a circular cross-section, rotation is not required.

5.6 Reagents

5.6.1 Direct-on-filter reagents.

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

5.6.2 Redeposition reagents.

5.6.2.1 Suspension.

Deionised water.

2-Propanol.

Ethanol.

5.6.2.2 Filter dissolution.

Tetrahydrofuran (THF).

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

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5.6.2.3 Removal of interferences.

Hydrochloric acid, 0,1 N to 1 N.

5.6.3 KBr pellet reagents.

Potassium bromide (KBr), infrared quality (stored in a desiccator)

1,3-Butanediol

Hydrochloric acid, 0,1 N to 1 N

Sodium hydroxide (2 N)

5.6.4 Reference materials.

It is important to use a reference material in which the purity and crystalline content is well characterized. The material used for calibration shall conform to the recommendations in ISO 24095.

NOTE 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).

NOTE A comparison of the crystallinity of calibration materials for the analysis of respirable quartz and their estimated values can be found in Reference [13].

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6 Sampling

6.1 Sample collection

A general guidance for sampling of respirable aerosol fraction is given in CEN/TR 15230 and ASTM D7948. A guidance specific for the measurement of RCS is given in ISO 24095.

6.1.1 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.

6.1.2 If gravimetric analysis is required, pre-weigh each uniquely identified collection substrate (including a minimum of three blanks) at least to the nearest 0,01 mg, according to ISO 15767 using flat tipped tweezers to avoid contamination and damage.

6.1.3 Load each sampler with a pre-weighed collection substrate, if gravimetric analysis is required, 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.

6.1.4 Ensure each loaded sampler is tested for leaks.

NOTE 1 A leak will change the sampler's performance which will also affect the mass of respirable dust sampled.

NOTE 2 The leak test is used as a benchmark test for proper assembly[8]. Examples for leak tests are: the particle count leak test, performed with an optical counter or a condensation particle counter[8], and the pressure drop leak test, performed with a micro manometer[9].

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

6.1.5 Connect the sampling train and adjust the flow rate to within ±5 % of the required value. The maximum deviation in flow rate (before and after sampling) should not exceed ±5 % of the required value.

6.1.6 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.

NOTE It is helpful to retain one or more unused collection substrate as a field blank from each site. The field blank is used for quality assurance purposes. If the field blank differs significantly from the average laboratory blank, investigate the problem.

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

6.1.8 A minimum sampling time shall be calculated taking into account the limit of quantification on the FTIR method for RCS (see 11.2) and the flow rate of the sampling system, so that compliance with the limit value or other measurement tasks 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[10]. Such losses are not visible to the naked eye, and cannot be quantified by a direct-on-filter method.

6.1.9 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 6.1.5.

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

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) the weight of the respirable dust on the collection substrate in order to determine the recovery after redeposition and to quantify the aliquot for KBr pellet method;

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

6.1.11 The results of a qualitative analysis of a sample of dust representative of the workplace or the activity provides additional information to the analyst to 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. The results from the qualitative scan provides useful information and should be reported to the occupational hygienist (e.g. when sampling a new process or a process where the materials involved in the work activity may change).

6.2 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,

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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 (typically on a 25 mm filter) can be transported by postal service without significant loss, provided that a suitable filter holder and a container designed to prevent damage are used[11].

7 Preparation

7.1 Preparation of calibration samples

Weighing generally is used to quantify the amount of silica in a calibration sample. The accuracy of weighing does not allow the confirmation of mass collected by a filter below the limit of quantification. This may be higher than the lowest concentration of the calibration curve (typical figures: 20 µg to 50 µg). However, it is possible that well-characterized mixtures of silica with other dusts could be employed for this purpose (especially for indirect methods).

NOTE NIST reference quartz and cristobalite on filters can be used as calibration standards in indirect analysis, and this includes masses below 50 µg.

7.1.1 Preparation of direct-on-filter calibration samples

7.1.1.1 Take at least 12 filters for calibration test samples and three filters to be used as laboratory blanks from the same lot and ensure unique identification in a suitable manner.

7.1.1.2 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 filters ensure three consecutive weighing are within 3 µg (1σ).

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

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

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

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

7.1.1.6 Generate aerosols of reference material within the containment apparatus and sample the dust to obtain loaded filters.

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

WARNING — Do not over pressurize the glass chamber.

7.1.1.7 Re-weigh the calibration test filters and the laboratory blanks according to ISO 15767 and determine the mass of reference material on the filter.

7.1.1.8 This method is suitable for the determination of quartz or cristobalite over the range 10 µg to 1 mg on a 25 mm filter. Over this range, there is a linear relationship between the infrared response and the quartz/cristobalite content of the sample.

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7.1.2 Preparation of redeposition calibration samples

7.1.2.1 Prepare at least two suspensions of the analyte by weighing known amounts of the reference material to the nearest 0,001 mg.

7.1.2.2 Calculate the mass of reference material in µg/mL of suspension as an aid to plan the aliquots for the calibration filters.

7.1.2.3 Suspend the reference material 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 become homogenous before withdrawing aliquots.

7.1.2.4 In accordance with ISO 15767, condition 25-mm diameter filters and weigh them to the nearest 0,001 mg. Ensure that three consecutive weighings of the same filter are within 15 µg (1σ). For polycarbonate filters, ensure three consecutive weighings are within 3 µg (1 σ).

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

7.1.2.5 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 reagent.

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.

When using PVC filters, select the rough side of the filter for the redeposition for improved adhesion of the reference material.

7.1.2.6 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 the vacuum on until the filter is dry. Remove the filter and allow it to dry completely. When thoroughly dry, reweigh the filter to determine the mass of deposit.

7.1.2.7 Reweigh the calibration test filters and the laboratory blanks according to ISO 15767 and determine the mass of reference material on the filter.

7.1.3 Preparation of KBr pellet calibration samples

7.1.3.1 Prepare at least two calibration KBr pellets for a minimum of six equidistant concentrations.

7.1.3.2 Ensure that the KBr is dried overnight at 110 °C and kept in a desiccator to prevent the uptake of moisture. A low relative humidity environment will facilitate sample handling when using KBr.

7.1.3.3 Weigh different amounts of reference material accurately to the nearest 0,001 mg and transfer to a crucible. Add the same type of blank collection substrate that was used during sampling in the workplace to the crucible.

7.1.3.4 Alternatively, suspensions with a defined concentration of reference material can be used to prepare calibration standards. A defined quantity of the reference material is suspended in water or ethanol in a 100 mL or 250 mL volumetric flask. Transfer the suspension into an Erlenmeyer flask and stir (magnetic stirrer) at a controlled temperature of 20 °C (bath thermostat). The quantities of suspension necessary for the preparation of the respective calibration standard are taken using a microlitre pipette.

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After drying the loaded filter, the necessary KBr quantity for preparation, as described above, is added to the loaded filter.

NOTE 1 The preparation of the calibration test samples can alternatively be performed according to 7.1.1 or 7.1.2 before proceeding with 7.1.3. To obtain the residual reference material, ash the filters using one of the sample treatment procedures described in 7.2.

NOTE 2 For improved accuracy a suspension of reference material and KBr can be prepared for calibration test samples below 100 µg. For example, a ratio of 1 990 mg of KBr to 10 mg of reference material can be used. KBr can be added additionally if the quantity for a pellet is insufficient.

7.1.3.5 To obtain the residual reference material ash the collection substrates using one of the relevant sample treatment procedures described in 7.2.

7.1.3.6 Weigh 250 mg to 450 mg (accurate to 0,1 mg) KBr and transfer to the crucible with the calibration sample ash or reference material. The mass of the pellets should not deviate by more than 10 % from each other.

7.1.3.7 Mix the sample ash or reference material thoroughly with the KBr using a pestle. Transfer to a mortar to complete mixing if necessary.

7.1.3.8 Quantitatively transfer the mixture to a 13-mm evacuable pellet die and ensure a homogenous distribution in the compression chamber.

7.1.3.9 After inserting the compression tooling into the hydraulic press, the tooling is degassed with the vacuum pump for approximately 1 min and the KBr disk is compressed with a pressure of 80 kN/cm^2 for approximately 1 min.

7.1.3.10 Weigh the finished pellet accurately to the nearest 0,1 mg. Calculate the ratio between the weight of the finished pellet and the weight of KBr that was initially added (usually about 0,98).

7.2 Preparation of the collection substrate for indirect analysis

Analysts using a direct on-air sampling filter analysis approach do not need to prepare the collection substrate and should proceed to Clause 8.

Ensure that all operations involving flammable solvents are performed in a fume cupboard or under air extraction. Some samples contain minerals that may interfere on the analysis for RCS. Refer to some sample treatment strategies outlined in Annex A to remove such interferences. Use the appropriate treatment based on the type of collection substrate.

7.2.1 PVC, MCE and polycarbonate filters

Use either the furnace treatment (7.2.1.1) or plasma asher (7.2.1.2) to ash sampled filter. As an alternative, treatment with tetrahydrofuran (THF) to dissolve the sampled filter can also be chosen (7.2.1.3).

7.2.1.1 Furnace treatment

a) Place each filter into a clean crucible and add 1 ml to 3 ml of 1,3-butanediol to the filter.

NOTE The butanediol is used to improve the control of the heating and combustion process.

b) Slowly heat the crucible in the furnace to a temperature of 450 °C to 600 °C and maintain this temperature for four to six hours.

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

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c) Allow the crucible to cool to room temperature and collect the sample once the filter is ashed.

d) Continue with the procedure in 7.3.1 or 7.4.

7.2.1.2 Plasma ashing

a) The plasma asher shall 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 of the bottle or beaker 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 7.3.2 or 7.4

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

7.2.1.3 Treatment with tetrahydrofuran (THF)

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

If THF is used to treat a sampling filter, a PVC filter cannot be used for redeposition and analysis as the THF will dissolve the filter.

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 by using a vortex mixer, taking care not to spill its contents.

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

e) Continue with the procedure in 7.3.2 using THF as the reagent.

7.2.2 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 7.3.1 or 7.4.

7.2.3 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.

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c) Wash the cassette out by rinsing it with 2 ml to 3 ml of reagent (2-propanol or ethanol) at least twice in the crucible. Rinse in the crucible any items that may have come into contact with the sample, e.g. tongs. 7.2.3

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 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 7.3.1 or 7.4.

7.3 Redeposition onto analysis filter

7.3.1 Crucibles from the furnace

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

b) Weigh, if required, a 25-mm diameter analysis filter to the nearest 0,001 mg.

c) Set up a filtration apparatus with similar dimensions as the one used for the preparation of calibration test filters in 7.1.3.

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

e) Cover the beaker with a watch glass.

f) Ultrasound for 3 min to 5 min.

g) Wash the watch glass in the beaker.

h) Carefully wash contents of crucible into the beaker.

i) Filter the contents of the beaker onto a pre-weighed filter.

j) Allow the filter to dry in air.

k) Reweigh, if required, the filter after conditioning and determine the recovery from the gravimetric weight recorded from the sampling.

7.3.2 Bottles or beakers from a plasma asher

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

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

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

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

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

f) Filter the residue onto an analysis filter with the same dimensions as the calibration test filters.

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7.4 Preparation of KBr pellets

a) A defined amount of KBr is added to the sample. 250 to 450 mg of KBr are added to a maximum of 2 mg of dust in each case.

b) KBr disks should not contain more than 2 mg of dust and not more than 1 mg of quartz.

NOTE Generally a quartz content of considerably less than 1 mg can be expected when using up to 2 mg of dust, as a quartz content of less than 20 % occurs at most workplaces. However, in certain areas (e.g. processing of quartz sand or finely ground quartz sand) the quartz content found in the dust can be even greater than 50 %. In this case an aliquot of the sample has to be prepared.

c) The thickness of the KBr disks for calibration as well as for samples should not deviate by more than 10 % from each other and so the mass of the pellets should be within 10 %.

d) In order to entirely remove the substance from the crucible walls the crucible with the dust and KBr is treated with ultrasound for 15 min.

NOTE If the ashing of cellulose nitrate or MCE sample filters (see 7.2.2) is done with addition of KBr, reaction between the filter material, potassium bromide (KBr) and dust can occur, that can lead to nonsystematic diminished contents of quartz (see details in Reference [20]). Addition of KBr after ashing the filter avoids any reaction.

e) Following transfer into the mixing or grinding tool, the sample mixture is homogenised.

NOTE Using a mill, grinding for more than 5 minutes does not improve results.

f) The KBr quantity required for the preparation of the pressed disk is weighed from the mixture of sample substance and KBr. When filling the mixture into the compression tooling (pellet die) a homogeneous distribution shall be ensured to avoid mass aggregation during compression of the disks. Thus the greatest possible uniformity of the disk thickness is achieved.

g) After inserting the compression tooling into the hydraulic press, the KBr pellet is compressed with a pressure of approximately 80 kN/cm^2 for approx. 1 min.

NOTE The pellet formation can be improved, if the tooling is degassed with the vacuum pump for approx. 1 min prior to compression.

NOTE The compression process and the compression equipment can be checked during initial implementation of the method by running the analysis on a disk four times, having been rotated by approximately 90° in the same direction in the holder. Deviations, large enough to affect the analytical result require the compression process to be optimised. Visible opacity at the edge of the finished disk is a sign of non-optimal compression with a non-uniformly distributed mixture.

h) To ensure good transparency of the pellet to the infrared beam, the sample mixture, the KBr, the milling vessels and compression tooling should be dried at 40 °C in the drying cupboard and stored there between pressing procedures.

If pressed disks are not immediately analysed, they can be stored in the drying cupboard for a short period. The pressed disks shall be ground and pressed again after prolonged storage. Losses in mass shall be taken into consideration, each disk shall be weighed before and after renewed compression.

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8 Analytical procedure

8.1 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. Weighing, if required, shall be performed according to ISO 15767.

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8.2 Fourier-Transform Infrared analysis

8.2.1 Background correction

8.2.1.1 Take the infrared spectra obtained from the calibration samples and subtract the spectrum obtained from a blank sample (either a suitable weight-matched blank filter or KBr pellet without RCS).

8.2.1.2 Alternatively, scan all the blank calibration filters prior to sampling and save the resulting spectra so that each blank filter can be used for its own background correction.

8.2.1.3 Another approach is to analyse a suitable weight-matched blank filter as the "background" prior to the analysis of the calibration filter. In this way only the RCS is analysed on the calibration filter. The background correction can be repeated with three suitable weight-matched blank filters in order to obtain three measurements for one calibration sample.

This is a practical approach when calibrations are prepared for workplace samples that are not prepared by the laboratory. The client may submit a suitable weight-matched blank filter so that the laboratory can use the blank as the "background".

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8.2.2 Measurement

8.2.2.1 Mount each sample (reference, calibration or workplace sample) in turn in the FTIR using a defocused beam so that the beam covers the sample area on the calibration sample. Where the spectrometer has a moveable stage on which to mount the sample holder, it is essential that the stage is placed in the same position for all samples.

It is good practice to take additional measurements by rotating the sample (e.g. 45°, 90° or 180°) to address potential inhomogeneity. The number of additional measurements and the specific rotations have to be determined during method validation.

8.2.2.2 Scan the samples in absorbance mode between a maximum range of 4 000 cm^{-1} and 400 cm^{-1}, using typical settings of 32 scans at 4 cm^{-1} resolution or better.

The number of scans may be optimised for the type of instrument.

8.2.2.3 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 limit value. This is achieved by changing the measurement parameters (number of scans and resolution) to the point at which no further improvement of precision is possible when measuring a filter or KBr pellet with a known mass of RCS. The masses at the limit and half the limit value are typically used. Manufacturers provide a variety of software products and may have different approaches for data collection.

NOTE If limit value is very low, 4 % precision requirement might not be achievable.

8.2.2.4 Monitor the beam intensity periodically through the batch of samples.

NOTE Stability of intensity can controlled by daily measurement of quality control samples.

8.3 Calibration

The analysis of calibration test samples and the evaluation of their spectra shall be done following the same procedure as workplace samples (see 8.2 and Clause 9).

8.3.1 Calibration is performed by preparing calibration test samples with known amounts of RCS and establishing a calibration graph of the intensity of the absorbance peak versus the mass of RCS in the reference material. If calibration samples are collected from an aerosol of reference material use the same equipment intended for workplace measurements.

8.3.2 Generate calibration test samples within a minimum range of approximately 0,1 to 2 times the appropriate limit value for the sampler selected over the sampling period normally used at the workplace:

$$L_{\text{up}} = \rho \times q_V \times t_{\text{samp}} \times 2K_{\text{load}} \tag{1}$$

$$L_{\text{low}} = \rho \times q_V \times t_{\text{samp}} \times 0{,}1K_{\text{load}} \tag{2}$$

where

L_{up} is the upper limit in mg;

L_{low} is the lower limit in mg;

ρ is the limit value , expressed in mg/m^3;

q_V is the flow rate, expressed in l/min/1 000;

t_{samp} is the sampling time, expressed in min;

K_{load} is the loading factor.

The mass of reference material deposited on a collection substrate, 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). The mass value of RCS deposited on a collection substrate, m_{RCS}, is calculated by the mass value of the reference material, obtained by weighing, corrected for its quartz or cristobalite content (crystallinity),

$$m_{\text{RCS}} = m_{\text{RM}} \cdot X_{\text{RM}} \tag{3}$$

where

m_{RCS} is the mass of quartz or cristobalite in mg;

m_{RM} is the mass of reference material in mg;

X_{RM} is the correction factor for quartz/cristobalite content in the reference material expressed as a fraction (e.g. 0,937 means 93,7 %).

A detailed procedure for calibration curve construction is given in the following subclauses. Refer to ISO 11095 for guidance.

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8.3.2.1 For practical purposes, the range between 0,01 mg to 1 mg RCS should be covered by the calibration curve.

8.3.2.2 Plot the baseline corrected peak heights or integral absorbance of the double peak 800 cm^{-1}/780 cm^{-1} against the mass of quartz in mg. Determine the lines of best fit for each plot (which should be linear and pass close to the origin). The slopes of these lines are the response factors (RF) for quartz at 780 cm^{-1} and 800 cm^{-1}. Examine the scatter of the data points from the regression line and re-examine any outliers to determine the cause.

8.3.2.3 For cristobalite, follow the same procedures as described for quartz, but measure the peak at 800 cm^{-1} and 620 cm^{-1}. Plot the baseline corrected peak heights or integral absorbance and determine the lines of best fit and response factors for cristobalite at the two frequencies.

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8.3.2.4 Derive a trend line from the relationship between response and mass of RCS in the calibration sample. 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].

8.3.2.5 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).

9 Evaluation of Fourier-Transform Infrared spectra

9.1 General aspects

9.1.1 Quantitative result is calculated on the most intense absorbance band of RCS that are free from major interferences, according to the calibration function:

$$I_{\mathrm{RCS}^*} = a + b \cdot m_{\mathrm{RCS}} \tag{4}$$

where

I_{RCS^*} is the blank corrected intensity of the quartz or cristobalite absorbance band;

a is the intercept found for the calibration function;

b is the slope found for the calibration function;

m_{RCS} is the mass of quartz or cristobalite in the sample.

9.1.2 Use the band at 800 cm^{-1} and 780 cm^{-1} to determine the amount of quartz. For cristobalite, follow the same procedures as described for quartz, but using the peaks at 800 cm^{-1} and 620 cm^{-1}.

9.2 RCS quantification using peak height

9.2.1 For quartz, draw baselines on the blank corrected spectra tangential to the absorbance minima at approximately 730 cm^{-1} and 830 cm^{-1} on either side of the quartz doublet.

9.2.2 For each spectrum, measure the peak heights from the baseline at 780 cm^{-1} and 800 cm^{-1}, respectively. If more than one spectrum is recorded, calculate the mean peak height at each frequency.

9.2.3 Calculate the amount of quartz on the sample filter, in µg, by dividing the peak height at each frequency by the response factor for that frequency obtained from the calibration standards and then averaging the two results. If the ratio of the peak height at 800 cm^{-1} to that at 780 cm^{-1} is outside the range 1,0 to 1,4, the sample spectrum should be examined for the presence of interferences, in particular cristobalite at 620 cm^{-1} and kaolinite at 915 cm^{-1}.

9.2.4 For cristobalite, follow the same procedures as described for quartz, but measure the peak heights at 800 cm^{-1} and 620 cm^{-1}. The peak at 620 cm^{-1} requires a second baseline to be drawn (again straight and as horizontal as possible) tangential to the trace minima at approximately 650 cm^{-1} and 590 cm^{-1}.

9.2.5 RCS quantification using peak height is ideal for spectra without interferences. However, if mineral interferences are present refer to 9.3 for the evaluation of sample spectra.

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9.3 RCS quantification using peak integral

The following three methods are available to the analyst:

a) determination of the absorption in the range of the analytical bands;

b) subtraction of a reference spectrum;

c) a method that takes the influence of particle size into consideration.

9.3.1 Determination of the absorption in the range of the analytical bands[12][14]

9.3.1.1 The determination of the absorption in the range of the analytical bands is the simplest technique that can be used to determine the mass of RCS in a sample. However, this technique assumes that there are no interferences on the analytical bands used and that the particle size distribution of the sample is similar to that of the reference material used for calibration.

9.3.1.2 This technique may be applied with the other methods listed in this subclause for workplace samples with complex matrices. An evaluation based solely on the determination of the integral absorption should not be carried out. It is advisable to apply this method only in combination with the methods listed in 9.2.2 and/or 9.2.3 in order to be able to identify possible interferences. Spectra subtraction as described in 9.2.2 can give an indication of the actual baseline profile. Generally it can be concluded that in the determination of the quartz or cristobalite mass using integral absorption the influence of the particle size (see 9.2.3) is not taken into consideration. In extreme cases (dust fractions with aerodynamic median diameter of <0,8 µm or >4 µm) this can lead to relative errors of up to ±20 %. Errors of up to ±10 % can be anticipated in dusts containing quartz or cristobalite with particle size distributions that commonly occur at workplaces.

9.3.2 Subtraction of a reference spectrum[20]

9.3.2.1 Subtraction of a quartz or cristobalite reference spectrum

9.3.2.1.1 The quartz or cristobalite mass can be determined by subtracting the absorption spectrum of a calibration standard (with a known reference material weight) from the absorption spectrum of the sample. The subtraction is carried out for the evaluation range of the double band or the analytical band that is used for the determination of the integral intensity (see 9.3.1).

9.3.2.1.2 The subtraction of a reference spectrum should be applied, if the background in the range of the double absorption band of quartz at 800 cm^{-1} and 780 cm^{-1} shows a nonlinear trend (e.g. slope or hump).

NOTE To evaluate the peak integral or peak height, usually the linear background of the undisturbed absorption band is typically drawn approximately between 830 cm^{-1} and 730 cm^{-1}. In the slope position a linear background can be drawn between the tangential points; however, this leads to an underestimation of peak integral or height (Figure 2).

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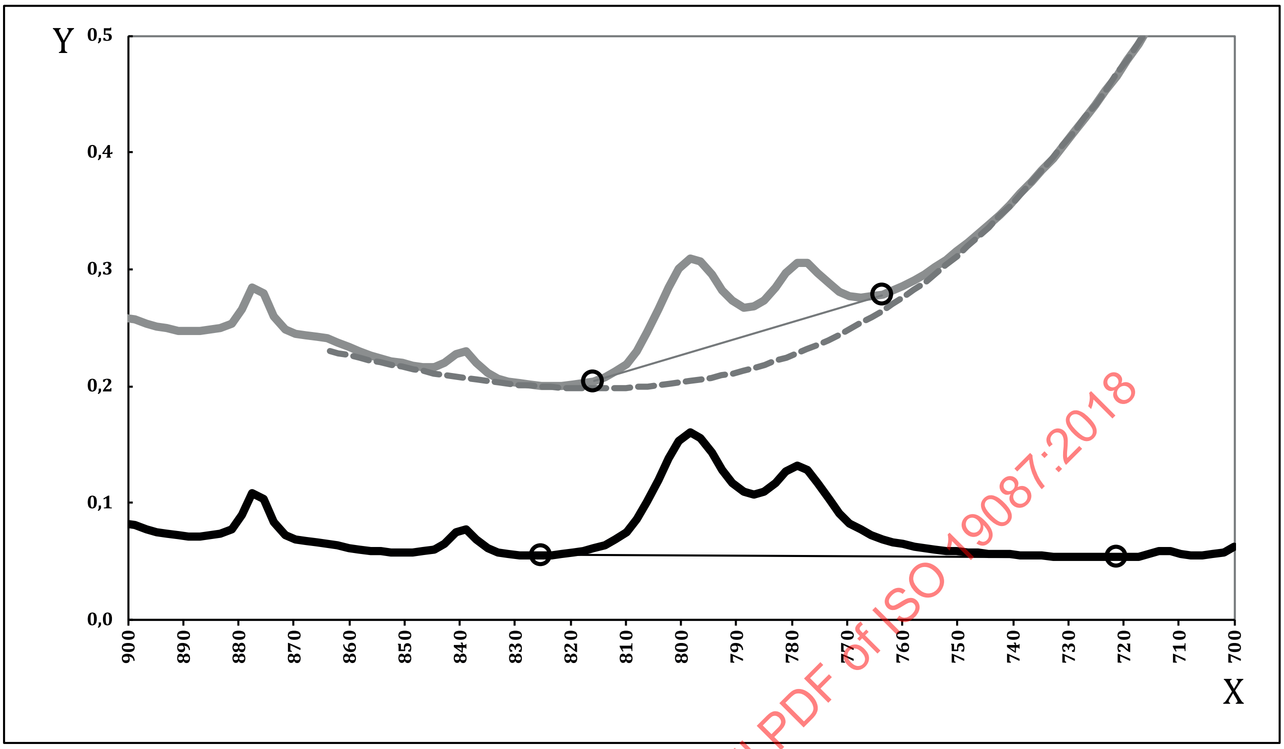

Key

X frequency in cm^{-1}

Y absorbance

1 pure quartz (80 µg)

2 quartz (80 µg; peak in slope position)

3 background by substraction with reference spectrum

4 linear background to evaluate peak integral or height

Figure 2 — Influence of the kind of slope of the background on the determination of the integral of absorbance for quartz

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9.3.2.1.3 The subtraction factor is selected so that the subsequent subtraction result leads to the most plausible baseline profile. When only quartz or cristobalite is present the resulting baseline after subtraction will be equal to the background.

9.3.2.1.4 The subtraction factor shall be optimised around the chosen factor so as to prevent overestimation or underestimation of RCS.

9.3.2.1.5 As quartz or cristobalite from different sources do not always exhibit an identical shape and profile of the absorption bands, standard spectra of different available quartz or cristobalite respirable dusts with known purity can be recorded and used for spectral subtraction. The resulting baseline becomes a doubly curved wave as the position of both of the absorption peaks of the double band of the sample and the standard do not exactly coincide. In this case a different quartz standard should be used in order to be able to determine the subtraction factor optimally.

9.3.2.1.6 The advantage of this evaluation method is that even in samples with strong mineral interferences on the quartz and cristobalite bands, satisfactory quantifications can be carried out. If the application of this method does not lead to satisfactory (plausible) baseline profiles for the subtraction result, then the XRD analytical method should be used (refer to Clause 11). In that case a reliable quantification of the quartz or cristobalite mass is not possible by means of infrared spectroscopy.

9.3.2.2 Subtraction of a reference spectrum from an interfering mineral.

9.3.2.2.1 If a known interference is present in a sample with a simple mineral matrix, prepare a calibration of this mineral as per 8.3. Subtract a known reference spectrum of the known mineral interference as per 9.2.2 from the sample spectrum. The residual spectrum can be used to determine the amount of quartz or cristobalite.

9.3.3 Other evaluation methods

There are other methods available that take the peak height, peak integral and particle size distribution into consideration. Please refer to Annex C for more information.

9.4 Interferences

9.4.1 Two crucial aspects should be considered during the evaluation:

— analytical interference due to associated substances in the dusts;

— dependence of the analysis on the particle size of the dust to be measured.

9.4.2 A main source of error in analysis is the presence of compounds where some absorbance coincides with (or is very close to) quartz or cristobalite. Examples include silicate compounds, kaolinite, muscovite and anorthite. A detailed list of interferences is available in ISO 24095.

9.4.3 Generally, identifying the other components in the dust on a heavily loaded sample assesses the presence of mineral interference.

9.4.4 A qualitative analysis using either X-ray diffraction (XRD) or FTIR can be helpful 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.

10 Calculation of results

10.1 Concentration of RCS

10.1.1 Calculate the volume, V_S, of each air sample, and the product of the nominal sampling flow rate and the sampling time.

10.1.2 Calculate the concentration of RCS, C_{RCS}, as the mass, m_{RCS} (mass of quartz and/or cristobalite), on the filter divided by the air volume, V_S:

$$C_{RCS} = \frac{m_{RCS}}{V_S} \tag{5}$$

where

C_{RCS} is the concentration of quartz and/or cristobalite in mg/m^3;

m_{RCS} is the mass of quartz and/or cristobalite in mg;

V_S is the air volume in m^3.

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11 Performance characteristics

11.1 Limit of detection

11.1.1 The limit of detection and accuracy of the method are dependent upon a number of parameters, for example particle size, other constituents of the sample and the type of spectrophotometer used.

NOTE 1 The limit of detection for RCS is determined as three times the standard deviation of the blank analysis (i.e. blank filter or blank KBr pellet), typically around 10 µg. For a 500 l air sample, this value corresponds to a detection limit of 0,020 mg/m^3.

NOTE 2 A sample-related limit of detection can also be applied where the limit of detection is verified by adding to the analysis filter or KBr pellet a mass of analyte that is not observed on the analytical bands in question. Alternatively, load known 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 perform the measurement on these samples.

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 workplace samples. Analysts should take this into consideration when analysing samples for RCS (ISO 24095).

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11.2 Limits of quantification

11.2.1 The presence of interfering mineral components will raise the limit of quantification for specific analytical bands.

NOTE 1 The limit of quantification for crystalline silica is defined as 10 times the standard deviation of the blank analysis (i.e. blank filter or blank KBr pellet), typically around 30 µg per sample. For a 500 l air sample, this value corresponds to a limit of quantification of around 0,06 mg/m^3.

NOTE 2 The limit of quantification may also be limited by instrumental factors because the masses of RCS when measuring 0,1 of the limit value are close to the limit of detection for some analytical bands when using low flow rate samplers (<4 l/min). This may also raise the limit of quantification beyond the values obtained through traditional estimates.

ISO 20581 states that the maximum expanded uncertainty permitted for sampling and analysis for comparison with limit values between 0,1 to 0,5 times the occupational exposure limit shall be within ±50 % and measurements between 0,5 to 2 times the exposure limit shall be within ±30 %. 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 ISO 20581.

11.3 Uncertainty

The method proposed for estimating uncertainty for the measurement of RCS is described in ISO 24095.

NOTE The expanded uncertainty for the analysis of crystalline silica by FTIR is typically around ±50 % when measuring an airborne concentration in the range 0,02 mg/m^3 to 0,05 mg/m^3 and around ±20 % when measuring an airborne concentration in the range 0,05 mg/m^3 to 0,2 mg/m^3.

11.4 Differences between samplers

11.4.1 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 has a performance that perfectly matches the ideal respirable convention and this causes differences between the masses of respirable dust collected, which will have an impact on the result obtained by FTIR.

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

12 Test report

The test report shall contain at least the following information:

a) details of the method used, with reference to this document, i.e. ISO 19087;

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 reference material 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; information resulting from optional qualitative scan (see 6.1.12);

o) analytical range of the calibration standards;

p) the storage period of the samples;

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

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Annex A
(normative)

Sample treatment strategies for the removal of interferences

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 °C ± 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).

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 – 1 N HCl and wait for 5 min to 8 min. Apply the vacuum to the filter. Dry the filter and follow the relevant procedure in 7.2, 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 – 1 N 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 7.3, replacing the word deionised water for reagent.

A.4 Removal of interferences

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

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Annex B
(informative)

Differences between samplers (cyclones and other types)

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 or practices allow the use of alternative samplers. Most samplers were not designed to conform to the ISO 7708 sampling penetration curve, but their flow-rates can be modified to provide a reasonable match. However, samplers do not match the penetration curve exactly and nor does their performance exactly match that of other sampler types. Even within a sampler type there is a unit-to-unit variation in 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 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 B.1 — Differences of samplers (cyclones and other types) from an average air concentration in calm air[17]

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 %
FSP10	10,2	+13,6 %	±14,1 %	+16,7 %	±9,2 %
PPI2	2,0	+5,6 %	±26,3 %	+12,7 %	±12,5 %
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 %
CIP 10-R	10	−4,6 %	±6,9 %	−6,9 %	±15,7 %
GS3	2,75	−10,5 %	±8,8 %	−9,2 %	±4,8 %
GK2.69	4,2	−17,5 %	±14,9 %	+1,9 %	±7,1 %
IOM with PUF Insert	2,0	−19,0 %	±7,0 %	−17,9 %	±5,8 %
SKC Al cyclone	2,7	−22,5 %	±8,6 %	−14,2 %	±7,9 %
Dorr Oliver*	1,7	−27,2 %	±9,8 %	−22,1 %	±10,2 %

* Some of the samplers were used with a non-conductive cassette but none of the dust collected on the inside walls of the cassette was recovered prior to the analysis.

[#] Flow rate used in the experimental study. Different flow rates may be required for workplace monitoring.

The samplers listed in this table are not exhaustive. The samplers used should comply with the requirements of EN 13205. 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.

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