INTERNATIONAL STANDARD

ISO 19087

First edition
2018-07

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

Reference number
ISO 19087:2018(E)

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Published in Switzerland

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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 voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

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

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/

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 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.

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).

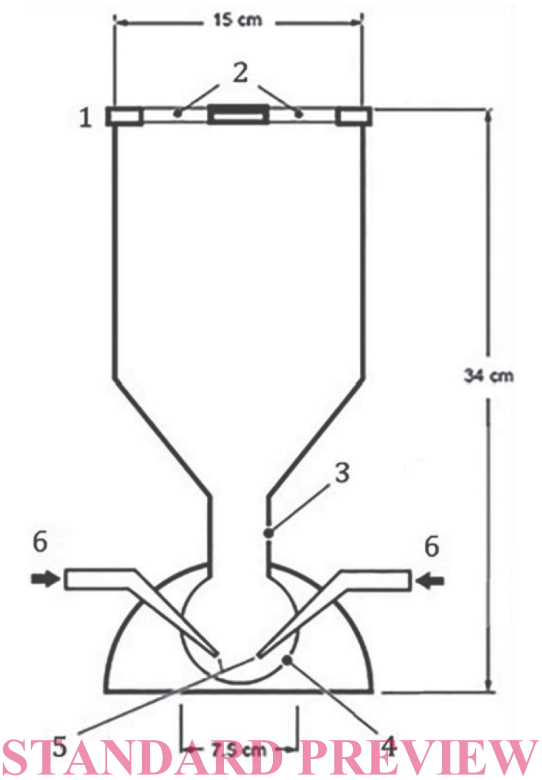
5.2 Equipment for calibration

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.

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.

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.