



Designation: D7948 – 20

# Standard Test Method for Measurement of Respirable Crystalline Silica in Workplace Air by Infrared Spectrometry<sup>1</sup>

This standard is issued under the fixed designation D7948; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This standard specifies a test method for collection and analysis of samples of airborne particulate matter for measurement of respirable crystalline silica by infrared (IR) spectrometry.

1.2 This test method is applicable to the analysis of crystalline silica (the polymorphs quartz, cristobalite and tridymite) over a working range of 0.025 to 0.4 mg/m<sup>3</sup> for a 400 L air sample or 0.02 to 0.25 mg/m<sup>3</sup> for a 1000 L air sample, depending on the analytical method.

1.3 The methodology is applicable to personal sampling of the respirable fraction of airborne particles and to static (area) sampling.

1.4 This test method describes three different procedures for sample preparation and infrared analysis of airborne crystalline silica samples, which are delineated in **Annex A1 – Annex A3**, respectively: (1) a potassium bromide (KBr) disc IR measurement method, (2) indirect IR analysis after redeposition onto a filter used for measurement, and (3) direct on-filter IR analysis.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.7 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.04 on Workplace Air Quality.

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## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

D4532 Test Method for Respirable Dust in Workplace Atmospheres Using Cyclone Samplers

D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps

D4840 Guide for Sample Chain-of-Custody Procedures

D6061 Practice for Evaluating the Performance of Respirable Aerosol Samplers

E1370 Guide for Air Sampling Strategies for Worker and Workplace Protection

### 2.2 ISO Standards:<sup>3</sup>

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

ISO 3534-1 Statistics — Vocabulary and symbols — Part 1: Probability and general statistical terms in metrology

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

ISO 15767 Workplace atmospheres – Controlling and characterizing errors in weighing collected aerosols

ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories

ISO 18158 Workplace air – Terminology

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

## 3. Terminology

### 3.1 Definitions:

3.1.1 For definitions of terms used in this test method, refer to Terminology D1356.

### 3.2 Definitions of Terms Specific to This Standard:

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

3.2.1 *limit of quantification (LOQ), n*—lowest reliable mass of crystalline silica that is quantifiable taking into consideration the matrix effects in the sample. **ISO 24095**

3.2.2 *limit value, n*—reference figure for concentration of a chemical agent in air. **ISO 18158**

3.2.3 *respirable crystalline silica (RCS), n*—inhaled particles of crystalline silica that penetrate into the unciliated airways. **ISO 24095**

3.2.4 *respirable fraction, n*—mass fraction of inhaled particles penetrating to the unciliated airways. **ISO 7708**

3.2.5 *sampling instrument, n*—device for collecting airborne particles, including the sampler, sampling pump and sampling medium such as a filter.

3.2.6 *time-weighted average (TWA) concentration, n*—concentration of a chemical agent in the atmosphere, averaged over the reference period. **E1370**

3.2.7 *uncertainty (of measurement), n*—parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand. **ISO 3534-1**

3.2.8 *workplace, n*—defined area or areas in which the work activities are carried out. **ISO 18158**

#### 4. Summary of Test Method

4.1 Airborne particles are collected by drawing a measured volume of air through a collection substratefilter mounted in a sampler designed to collect the respirable fraction of airborne particles. After sampling for a specified reference period at a given air sampling flow rate, the sampling substrate (normally a filter) and collected sample are treated to prepare the collected crystalline silica particulate matter for subsequent measurement by infrared (IR) spectrometry. Characteristic IR peaks for crystalline silica are measured and used to determine the mass of crystalline silica in the collected air sample. Three different procedures for sample preparation and infrared analysis of airborne crystalline silica samples are described: (1) a potassium bromide (KBr) disc IR measurement method (after initial filter collection and subsequent sample treatment); (2) indirect IR analysis after redeposition onto a filter used for measurement; and (3) direct on-filter IR analysis. The measurement results can be compared to applicable occupational limit values (OELs) for crystalline silica in respirable airborne particulate samples.

#### 5. Significance and Use

5.1 Respirable crystalline silica is a hazard to the health of workers in many industries who are at risk through exposure by inhalation. Industrial hygienists and other public health professionals need to determine the effectiveness of measures taken to control workers' exposure, and this is generally achieved by taking workplace air measurements. This standard has been published in order to make available a method for making valid exposure measurements for crystalline silica exposures in industry. It will be of benefit to: agencies concerned with health and safety at work; industrial hygienists and other public health professionals; analytical laboratories; industrial users of silica-containing products and their workers, etc.

5.2 This standard specifies a generic sampling and analytical method for measurement of the mass concentration of respirable crystalline silica in workplace air using infrared (IR) spectrometric methods. Several different types of sampling apparatus are used to collect respirable dust, according to the occupational hygiene sampling convention. This standard is designed to accommodate a variety of appropriate samplers and sampling materials that are commercially available.

#### 6. Interferences

6.1 The applicability and performance of the infrared technique(s) used to measure respirable crystalline silica (RCS) is (are) dependent on the ability to address matrix and mineral interferences (ISO 24095). It is necessary to consider the matrix and mineral interferences potentially present in airborne samples, and to take action to minimize these interferences in IR analysis of RCS. Numerous minerals that could be present along with crystalline silica in airborne respirable samples absorb infrared radiation in the spectral region of the quartz absorbance bands at 799  $\text{cm}^{-1}$  and 780  $\text{cm}^{-1}$ , giving rise to positive interference (1, 2).<sup>4</sup> Some of the more frequently encountered of these minerals, along with their characteristic IR frequencies in the range 450–1000  $\text{cm}^{-1}$ , are presented in **Table 1 (2-4)**. Examples of commonly encountered minerals that can interfere with IR analysis include kaolinite, a constituent of clays; muscovite, which is present in micas; and albite, anorthite and orthoclase, which are feldspars.

6.2 Quartz is a common component of soil, rocks, sand, mortar, cement, fluxes, abrasives, glass, porcelain, paints, and brick. Cristobalite is less common and may be a constituent of volcanic rocks and soils; it can be formed in high temperature work such as foundry processes, calcining diatomaceous earth, brick fabrication, ceramic manufacturing and silicon carbide production. Tridymite, which is rarely encountered in workplaces, is present in some volcanic rocks and soils.

6.3 If necessary, quartz and cristobalite can be determined in the presence of other mineral interferences absorbing at  $\approx 800 \text{ cm}^{-1}$  by measurement of the identifying bands at 694  $\text{cm}^{-1}$  for

<sup>4</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

**TABLE 1 Minerals Potentially Encountered and Their Characteristic IR Bands (450–1000  $\text{cm}^{-1}$ )**

Mineral	Major/Interfering Peaks, $\text{cm}^{-1}$	Identifying peaks, $\text{cm}^{-1}$
Quartz	799, 780	694, 512, 467
Cristobalite	798	623, 490
Tridymite	789	617, 476
Amorphous silica	800	464
Kaolinite	795, 754	914, 547, 474
Muscovite	800, 750	535, 481
Mullite	837, 748	556, 468
Pyrophyllite	830, 814	948, 477, 457
Albite	788, 746	726, 652, 598, 470
Montmorillonite	797	918, 668, 526, 470
Daphnite	798, 771	667, 610, 539, 467
Anorthite	760, 730	577, 538, 481
Orthoclase	765, 745, 730	645, 593, 540
Talc	797, 778	668, 641, 620
Vermiculite	810, 755	685, 510

quartz and  $623\text{ cm}^{-1}$  for cristobalite (56). Cristobalite and tridymite absorb at  $\approx 800\text{ cm}^{-1}$ , although they are rarely encountered in practice (tridymite particularly). Kaolinite, which is a common component of coal, can interfere if it is present in appreciable quantities. Calcite, if present at amounts greater than 20 % of total dust loadings, can interfere by reacting with quartz during sample preparation. (Calcite is a prevalent constituent of limestone.) Amorphous silica may interfere if present in large amounts; its interference can be minimized by measuring alternative but less sensitive bands at  $694\text{ cm}^{-1}$  for quartz and  $623\text{ cm}^{-1}$  for cristobalite.

6.4 Besides minerals, matrix interferences from other materials can affect IR analysis. For example, carbonaceous materials are ubiquitous matrix interferants in, for example, coal mines, and iron oxide is a common matrix interferant in, for example, foundries. Numerous background matrix and mineral interferences may be present in airborne dust emanating from construction activities. Various techniques are used in sample preparation and IR measurement in efforts to account for and minimize matrix interferences.

6.5 Standard mixtures of potentially interfering minerals can be prepared using the same sample preparation techniques as for standard crystalline silica samples, and the effect of interference on the IR spectrum can then be assessed and corrected for mathematically. These techniques, which are used to minimize background and mineral interferences to IR measurement, are described in [Annex A1 – Annex A3](#). Generally sample ashing techniques (described in [Annex A1](#) and [Annex A2](#)) are more effective at addressing interferences and matrix effects that might not be adequately accounted for by use of the direct on-filter method ([Annex A3](#)).

6.6 Knowledge of and training in geochemistry and mineralogy is strongly recommended for users of this standard. Although many analytical chemists are familiar with IR spectroscopy (like as applied to organic analysis), mineralogical samples, such as samples containing airborne respirable crystalline silica, require additional knowledge of geochemistry and mineralogy to correctly interpret IR spectra and to account for matrix interferences and mineral transformations.

## 7. Apparatus

### 7.1 *Sampling and Analytical Equipment:*

#### 7.1.1 *Sampling Equipment:*

7.1.1.1 *Respirable samplers*, designed to collect the respirable fraction of airborne particles, for use when the limit values for crystalline silica apply to the respirable fraction of airborne particles (Practice [D6061](#)). Cyclone-type samplers are typically used for personal sampling, although impaction devices are also used ([7, 8](#)).

NOTE 1—Cyclone devices typically use sample collection on filters, although impaction devices may use filters or foams for sample capture.

NOTE 2—As an alternative to cyclones, cascade impactors are often used to characterize the particle size distribution in static (area) sampling.

7.1.1.2 *Filters*, normally composed of polyvinyl chloride (PVC). The filters shall be of a diameter suitable for use with the samplers (typically 37-mm diameter) and shall have a

collection efficiency of not less than 99.5 % for particles with a  $0.3\text{ }\mu\text{m}$  diffusion diameter (ISO 7708).

NOTE 3—Besides PVC, filters comprised of other materials may be suitable, such as mixed cellulose ester (MCE).

NOTE 4—Apart from filters, other types of collection substrates may be suitable, such as foams.

7.1.1.3 *Filter holders*, of appropriate diameter for housing the filters used for sample collection, and preferably comprised of static-dissipative material.

7.1.1.4 *Back-up pads*, to support the filters within the filter holders.

7.1.1.5 *Sampling head holder/connector*, if required, for connecting the cyclone to the filter holder.

7.1.1.6 *Sampling Pumps*—Sampling pumps used shall meet the requirements of ISO 13137.

7.1.1.7 *Flow Meter*, portable, with an accuracy that is sufficient to enable the volumetric flow rate to be measured to within  $\pm 5\%$ . The flow meter calibration, by a provider accredited to ISO/IEC 17025 for such calibrations, shall be traceable to national or international standards (see Practice [D5337](#)). Retain the calibration certificate, including the pressure and temperature at which the calibration was performed, and identifying and performance documentation for the flow meter.

NOTE 5—It is advisable that the flow meter used is capable of measuring the volumetric flow rate to within  $\pm 2\%$  or better.

#### 7.1.2 *Analytical Instrumentation:*

7.1.2.1 Details regarding specific analytical instrumentation and reagents that are required for three different IR sample preparation and analysis procedures are provided in [Annex A1 – Annex A3](#) (KBr disc method, direct on-filter measurement, and indirect redeposition technique, respectively). Use only reagents of analytical grade.

7.1.2.2 *Infrared spectrometer*, double-beam dispersive or Fourier transform device, with  $4\text{ cm}^{-1}$  resolution or better.

7.1.2.3 *Analytical balance*, capable of weighing to the nearest 0.001 mg.

#### 7.1.3 *Ancillary Equipment:*

7.1.3.1 *Flexible tubing*, of a diameter suitable for making a leak-proof connection from the samplers to the sampling pumps.

7.1.3.2 *Belts or harnesses*, to which the sampling pumps can conveniently be fixed for personal sampling (except where the sampling pumps are small enough to fit in workers' pockets).

7.1.3.3 *Flat-tipped forceps*, for loading and unloading filters into samplers.

7.1.3.4 *Filter transport cassettes or similar*, if required, in which to transport samples to the laboratory

7.1.3.5 *Thermometer*,  $0^{\circ}\text{C}$  to  $50^{\circ}\text{C}$  minimum range, with resolution of  $1^{\circ}\text{C}$  or less, for measurement of atmospheric temperature, if required.

7.1.3.6 *Barometer*, suitable for measurement of atmospheric pressure, if required.

7.1.3.7 *Laboratory oven*, for drying (to  $110^{\circ}\text{C}$ ).

7.1.3.8 *Desiccator*, for dry storage.