



# 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.

<sup>ε1</sup> NOTE—Editorial corrections made throughout in July 2016.

## 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 and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

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

<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.

Current edition approved Oct. 15, 2014. Published February 2015. DOI: 10.1520/D7948-14E01.

<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.

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

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

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 6879 Air quality — Performance characteristics and related concepts for air quality measuring methods

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

ISO 15202-1 Workplace air — Determination of metals and metalloids in airborne particulate matter by inductively coupled plasma atomic emission spectrometry — Part 1: Sampling

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

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 General Terms Specific to This Standard:

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

3.2.2 *chemical agent, n*—any chemical element or compound, on its own or admixed as it occurs in the natural

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

state or as produced by any work activity, whether or not produced intentionally and whether or not commercially available.

3.2.3 *breathing zone, n*—(general definition) space around the worker's face from where he or she takes his or her breath.

**EN 1540 (1)<sup>4</sup>**

(technical definition) hemisphere (generally accepted to be 0.3 m in radius) extending in front of the human face, centred on the midpoint of a line joining the ears; the base of the hemisphere is a plane through this line, the top of the head and the larynx.

**ISO 15202-1**

3.2.4 *exposure (by inhalation), n*—situation in which a chemical agent is present in air which is inhaled by a person.

**EN 1540 (1)**

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

**EN 1540 (1)**

3.2.6 *measurement procedure, n*—set of operations, described specifically, for the sampling and analysis of chemical agents in air.

**EN 1540 (1)**

3.2.6.1 *Discussion*—A measurement procedure usually includes preparation for sampling, sampling, transportation and storage, preparation of samples for analysis and analysis.

3.2.7 *reference period, n*—specified period of time for which the (occupational exposure) limit value of a chemical agent applies.

**EN 1540 (1)**

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

**E1370; ISO 15202-1**

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

**EN 1540 (1)**

3.3 *Definitions of Particle Size Fraction Terms Specific to This Standard:*

3.3.1 *respirable convention, n*—target specification for sampling instruments when the respirable fraction is of interest.

**ISO 7708**

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

**ISO 7708**

3.4 *Definitions of Sampling Terms Specific to This Standard:*

3.4.1 *operating time, n*—period during which a sampling pump can be operated at specified flow rate and back pressure without recharging or replacing the battery.

**ISO 13137**

3.4.2 *personal sampler, n*—sampling device, attached to a person, which collects airborne particles in the breathing zone.

3.4.3 *personal sampling, n*—process of sample collection carried out using a personal sampler.

3.4.4 *(air) sampler, n*—device for collecting chemical agents from the surrounding air.

3.4.4.1 *Discussion*—Air samplers are generally designed for a particular purpose, for example, for sampling airborne particles.

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

3.4.6 *sampling train, n*—apparatus for collecting airborne particles including sampling pump, connecting tubing, respirable size selector, and collection medium (for example, a filter).

3.4.7 *static (area) sampler, n*—sampler, not attached to a person, which collects airborne particles at a particular location.

**EN 1540 (1)**

3.4.8 *static (area) sampling, n*—process of (air) sampling carried out using a static sampler.

**EN 1540 (1)**

3.5 *Definitions of Analytical Terms Specific to This Standard:*

3.5.1 *sample preparation, n*—operations carried out on a sample, after transportation and storage, to prepare it for analysis, including transformation of the sample into a measurable state.

**ISO 24095**

3.5.2 *limit of detection (LOD), n*—lowest reliable mass of crystalline silica that is detected taking into consideration the matrix effects in the sample.

**ISO 24095**

3.5.3 *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**

3.6 *Definitions of Statistical Terms Specific to This Standard:*

3.6.1 *accuracy, n*—closeness of agreement between a test result and the accepted reference value.

**ISO 3534-1**

3.6.2 *analytical recovery, n*—ratio of the mass of analyte measured in a sample to the known mass of analyte in that sample.

**EN 1540 (1)**

3.6.3 *bias, n*—consistent deviation of the results of a measurement process from the true value of the air quality characteristic itself.

**ISO 6879**

3.6.3.1 *Discussion*—Bias is the total systematic error as contrasted to random error. There may be one or more systematic error components contributing to the bias.

3.6.4 *precision, n*—the closeness of agreement of results obtained by applying the method several times under prescribed condition.

**ISO 6879**

3.6.5 *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**

## 4. Summary of Test Method

4.1 Airborne particles are collected by drawing a measured volume of air through a filter 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

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

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 (2 and 3). 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

**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

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 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