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Standard Test Method for Measurement of Respirable Crystalline Silica in Workplace Air by Infrared Spectrometry[1]

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 (ε) indicates an editorial change since the last revision or reapproval.

ε^1 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^3 for a 400 L air sample or 0.02 to 0.25 mg/m^3 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:*[2]

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:*[3]

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

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[2] 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.

[3] 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)**[4]

(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

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[4] 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 cm^{-1} and 780 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 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.

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

Mineral	Major/Interfering Peaks, cm^{-1}	Identifying peaks, 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

6.3 If necessary, quartz and cristobalite can be determined in the presence of other mineral interferences absorbing at ≈800 cm^{-1} by measurement of the identifying bands at 694 cm^{-1} for quartz and 623 cm^{-1} for cristobalite (**56**). Cristobalite and tridymite absorb at ≈800 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 cm^{-1} for quartz and 623 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. Occupational Exposure Assessment

7.1 The scope of this section pertains to the taking of personal and static (area) samples. Refer to relevant International, European or National Standards (for example, Guide E1370, Ref. (**7**)) for guidance on how to develop an appropriate assessment strategy and for general guidance on measurement strategy.

7.2 *Personal Sampling*—Exposure of workers to crystalline silica shall normally be determined by personal sampling, since the concentration of crystalline silica in the breathing zone is usually higher than their background levels in the workplace.

7.3 *Static (Area) Sampling*—Static (area) sampling may be carried out, if appropriate, to assess the exposure of workers in a situation where personal sampling is not possible; to characterize the background levels of crystalline silica in the workplace in order to give an indication of the efficiency of ventilation; or to provide information on the location and intensity of an emission source.

7.4 *Selection of Measurement Conditions and Measurement Pattern:*

7.4.1 Sampling shall be carried out in such a way as to cause the least possible interference with the worker and the normal performance of the job, and to provide samples that are representative of normal working conditions and that are compatible with the analytical method. The pattern of sampling shall take into consideration practical issues, such as the nature of the measurement task and the frequency and duration of particular work activities.

7.4.2 *Screening Measurements of Variation of Concentration in Time or Space, or Both*—Screening measurements of variation of concentration in time/and or space may be performed to provide information on the likely pattern of concentration of chemical agents. They can be used to identify locations and periods of elevated exposure and to set the duration and frequency of sampling for measurements for comparison with limit values. Emission sources can be located and the effectiveness of ventilation or other technical measures can be estimated (see, for example, Guide E1370; Ref. (**8**)).

7.4.3 *Screening Measurements of Time-Weighted Average Concentration and Worst-Case Measurements*—Screening measurements of time-weighted average concentration may be performed to obtain relatively qualitative information on the exposure level in order to decide whether an exposure problem exists at all and, if so, to appraise its possible seriousness. Such measurements can also be used to determine if the exposure is well below or well above the limit value (see, for example, Guide E1370; Refs.(**8 and 9**)). Screening measurements of time-weighted average concentration are typically carried out in the initial stages of an investigation to assess the effectiveness of control measures. Sampling may be carried out during representative work episodes to obtain clear information about the level and pattern of exposure, or worst-case measurements may be made.

NOTE 1—Screening measurements of time-weighted average concentration made to clearly identify work episodes during which highest exposure occurs are typically referred to as "worst-case measurements" (see Guide E1370).

7.4.4 *Measurements Near an Emission Source*—Measurements may be performed near an emission source to provide information on the location and intensity of the source. In association with other information, they can allow the elimination of a suspected source as a significant contributor to exposure (see Guide E1370; Refs. (**7 and 8**)).

7.4.5 *Measurements for Comparison with Limit Values and Periodic Measurements*—Measurements for comparison with limit values are performed to provide accurate and reliable information on, or allow the prediction of, the time-weighted average concentration of crystalline silica in the air that could be inhaled (see E1370; Refs. (**7 and 8**)). Periodic measurements are performed to determine whether exposure conditions have changed since measurements for comparison with limit values were made, or whether control measures remain effective (see Guide E1370).

NOTE 2—The best estimate of long-term exposure is obtained by taking samples for the entire working period, but this might not be practicable (for instance, because of the possibility of overloading the sampler).

8. Apparatus

8.1 *Sampling and Analytical Equipment:*

8.1.1 *Sampling Equipment:*

8.1.1.1 *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 (**10 and 11**).

NOTE 3—Cyclone devices typically use sample collection on filters, although impaction devices may use filters or foams for sample capture.

NOTE 4—As an alternative to cyclones, cascade impactors are often used to characterize the particle size distribution in static (area) sampling.

8.1.1.2 *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 µm diffusion diameter (ISO 7708).

NOTE 5—Besides PVC, filters comprised of other materials (see ISO 15202-1) may be suitable, such as mixed cellulose ester (MCE).

NOTE 6—Apart from filters, other types of collection substrates may be suitable, such as foams.

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

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

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

8.1.1.6 *Sampling Pumps*—Sampling pumps used should meet the requirements of ISO 13137. Pumps shall have an adjustable flow rate and be capable of maintaining the selected flow rate (from ≈2 L/min and ≈10 L/min for personal sampling pumps, and between ≈5 L/min and ≈40 L/min for high-volume static (area) sampling pumps) to within ±5 % of the nominal value throughout the sampling period. For personal sampling, the pumps shall be capable of being worn by the worker without impeding normal work activity. The pump should have, as a minimum, the following features (**12**):

(1) an automatic control that keeps the volumetric flow rate constant (to within ±0.1 L/min) in the case of a changing back pressure;

(2) Either a malfunction indicator which, following completion of sampling, indicates that the air flow has been reduced or interrupted during sampling; or an automatic cut-out, which stops the pump if the flow rate is reduced or interrupted;

(3) A facility for the adjustment of flow rate, such that it can only be actuated with the aid of a tool (such as a screwdriver) or requires special knowledge for operation (for instance via software), so as to preclude inadvertent readjustment of the flow rate during use;

(4) The pulsation of the flow rate does not exceed 25 %;

(5) A flow rate set within the nominal range does not deviate by more than ±5 % from the initial value under increasing back pressure;

(6) Within the range of ambient temperatures from 5°C to 40°C, the flow rate measured under operating conditions does not deviate by more than ±5 % from the flow rate at 20°C; and

(7) The operating time is at least 2 h, and preferably 8 h; the flow rate does not deviate by more than ±5 % from the initial value during the operating time.

NOTE 7—An integral timer is a highly desirable additional feature.

NOTE 8—A flow-stabilized pump may be required to maintain the flow rate within the specified limits.

8.1.1.7 *Flow Meter*—The flow meter shall be portable and have an accuracy that is sufficient to enable the volumetric flow rate to be measured to within ±5 %. The calibration of the flow meter shall be checked against a primary standard, that is, a flow meter whose accuracy is traceable to national standards. If appropriate, record the atmospheric temperature and pressure at which the calibration of the flow meter was checked. It is advisable that the flow meter used is capable of measuring the volumetric flow rate to within ±2 % or better.

8.1.2 *Analytical Instrumentation:*

8.1.2.1 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.

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

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

8.1.3 *Ancillary Equipment:*

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

8.1.3.2 *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).

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

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

8.1.3.5 *Thermometer*, 0°C to 50°C minimum range, graduated in divisions of 1°C or less, for measurement of atmospheric temperature, if required.

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

8.1.3.7 *Laboratory oven*, for drying (to 110°C).

8.1.3.8 *Desiccator*, for dry storage.

8.1.3.9 *Laboratory glassware, borosilicate*—beakers, bottles, and flasks (etc.) of appropriate volumes and sizes; with stoppers to fit.

8.1.3.10 *Wash bottles*, plastic (for example, polyethylene).

8.1.3.11 *Pipets*, borosilicate or plastic; various sizes as required.

8.1.3.12 *Magnetic stirring device*, and stir bars.

8.1.3.13 *Tweezers*.

8.1.4 *Crystalline silica certified reference materials (CRMs)*—Quartz; cristobalite (plus others as applicable).

NOTE 9—Examples include quartz and cristobalite NIST SRMs 1878a, 1879a, 2950, 2951, 2958, 2960 and 2957.[5]

9. Sampling Procedure

9.1 Sampling of respirable crystalline silica should be carried out in accordance with Test Method D4532.

9.2 *Preliminary Considerations:*

9.2.1 *Selection and Use of Samplers*—Select samplers designed to collect the respirable fraction of airborne particles, as defined in ISO 7708. If possible, the samplers selected should be manufactured from static-dissipative material, since samplers manufactured in non-conducting material have electrostatic properties that can influence representative sampling. Use the samplers at their design flow rate, and in accordance with the instructions provided by the manufacturer, so that they collect the respirable fraction of airborne particles (Test Method D4532).

NOTE 10—Limit values for crystalline silica typically apply to the respirable fraction of airborne particles.

9.2.2 *Sampling Period*—Select a sampling period that is appropriate for the measurement task, but ensure that it is long enough to enable crystalline silica to be measured with acceptable uncertainty at levels of industrial hygiene significance. For example, consider the applicable limit value, and estimate the minimum sampling time required to ensure that the amount collected is above the lower limit of the working range of the analytical method when crystalline silica is present in the test atmosphere at an appropriate multiple of its limit value (for example 0.1 times for an 8 h time-weighted average limit value), using the following equation:

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[5] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

$$t_{\min} = \frac{m_{\min}}{q_v \times 0.1 \times LV} \quad (1)$$

where:

$t_{\min}$ = the minimum sampling time, in minutes;
$m_{\min}$ = the lower limit of the analytical range, in micrograms, for crystalline silica;
q_v = the design flow rate of the sampler, in litres per minute; and
LV = the limit value, in milligrams per cubic metre, for crystalline silica.

9.2.3 When high concentrations of airborne particles are anticipated, select a sampling period that is not so long as to risk overloading the filter with particulate matter.

9.2.4 *Temperature and Pressure Effects*—Refer to the manufacturer's instructions to determine if the indicated volumetric flow rate of the flow meter is dependent upon temperature and pressure. Consider whether the difference between the atmospheric temperature and pressure at the time of calibration of the flow meter and during sampling is likely to be great enough to justify making a correction to take this into account, for example, if the error could be greater than ±5 %. If a correction is necessary, measure and record the atmospheric temperature and pressure at which the calibration of the flow meter was checked and measure and record the atmospheric temperature and pressure at the start and at the end of the sampling period.

NOTE 11—An example of temperature and pressure correction for the indicated volumetric flow rate is given in Appendix X1 for a constant pressure drop, variable area flow meter.

NOTE 12—If too great a correction is required, this could affect the sampler enough to perturb the sampled cut point and penetration curve away from the "ideal" respirable fraction definition.

9.2.5 *Handling of Sample Collection Media*—To minimize the risk of damage or contamination, only handle filters (or foams) using flat-tipped forceps, in a clean area, where the concentration of airborne particles is as low as possible.

9.3 *Preparation for Sampling:*

9.3.1 *Cleaning of Samplers*—Unless disposable samplers are used, clean the samplers before use. Disassemble the samplers, soak in detergent solution, rinse thoroughly with water, wipe with absorptive tissue and allow to them dry before reassembly. Alternatively, use a laboratory washing machine to clean the samplers.

9.3.2 *Loading the Samplers with Filters*—Load clean samplers with filters (pre-weighed to the nearest 0.01 mg, if desired, for gravimetric analysis of sampled dust). Label each sampler so that it can be uniquely identified, and seal with its protective cover or plug to prevent contamination.

NOTE 13—Alternatively, commercially available pre-loaded filter cassettes may be used.

NOTE 14—Samplers containing foam substrate may also be used.

9.3.3 *Attaching the Cyclone to the Sample Substrate Holder*—Connect the cyclone to the sample substrate holder so that the sampling head holder keeps the holder, cyclone and coupling device together rigidly. Ensure that sampled air will enter only at the cyclone inlet.

NOTE 15—For impaction devices, this step would not necessarily apply.

9.3.4 *Setting the Volumetric Flow Rate*—Perform the following in a clean area, where the concentration of airborne particles is low.

9.3.4.1 Connect each loaded respirable sampling apparatus to a sampling pump using flexible tubing, ensuring that no leaks can occur.

9.3.4.2 Remove the protective cover or plug from each sampler, switch on the sampling pump, attach the flow meter to the sampler so that it measures the flow through the sampler inlet orifice(s), and set the required volumetric flow rate to sample the respirable fraction of the aerosol.

NOTE 16—Typical sampling flow rates are ≈2 L/min for "low flow rate" personal samplers, ≈4 L/min for "higher flow rate" personal samplers, and ≈10 L/min for "high flow rate" personal sampling apparatus (**12**).

NOTE 17—If necessary, allow the sampling pump operating conditions to stabilize before setting the flow rate.

9.3.4.3 Switch off the sampling pump and seal the sampler with its protective cover or plug to prevent contamination during transport to the sampling position.

9.3.4.4 *Blanks*—Retain as blanks at least one unused loaded sampler from each batch of twenty prepared, subject to a minimum of three. Treat these in the same manner as those used for sampling with respect to storage and transport to and from the sampling position, but draw no air through the filters.

9.4 *Sampling Position:*

9.4.1 *Personal Sampling*—The sampler shall be positioned in the worker's breathing zone, as close to the mouth and nose as is reasonably practicable, for example, fastened to the worker's lapel. Attach the sampling pump to the worker in a manner that causes minimum inconvenience, for example, to a belt around the waist. Give consideration to whether the nature of the process is likely to result in a significant difference between the actual exposure of the worker and the concentration of airborne particles measured by a sampler mounted on the lapel. If this is the case, make special arrangements to mount the sampler as close as possible to the worker's nose and mouth. When a cyclone is used it needs to remain in an upright position during the duration of sampling.

9.4.2 *Static (Area) Sampling*—If static (area) sampling is carried out to assess the exposure of a worker in a situation where personal sampling is not possible, the sampling position shall be in the immediate vicinity of the worker and at breathing height. If in doubt as to where to place the sampler, the sampling position chosen should be the location where the risk of exposure is considered to be greatest. If static (area) sampling is carried out to characterize the background level of crystalline silica in the workplace, select a sampling position that is sufficiently remote from the work processes, such that results will not be directly affected by airborne particles from emission sources.

9.5 *Collection of Samples:*

9.5.1 When ready to begin sampling, remove the protective cover or plug from the sampler inlet (if applicable) and switch on the sampling pump. Record the time and volumetric flow rate at the start of the sampling period. If the sampling pump is fitted with an integral timer, check that this is reset to zero. If appropriate, measure the atmospheric temperature and pressure at the start of the sampling period using the thermometer and barometer, and record the measured values.

NOTE 18—If the temperature or pressure at the sampling position is different from that where the volumetric flow rate was set, the volumetric flow rate could change and it might need to be re-adjusted before sampling.

9.5.2 At the end of the sampling period, record the time and calculate the duration of the sampling period. Remove the sampler from the workers' lapel, being careful not to invert the cyclone, so as to avoid dust from the grit pot depositing on the filter. The cyclone needs to remain in an upright position after sampling until the filter assembly is removed and capped/plugged.

9.5.3 Check the malfunction indicator or the reading on the integral timer, if fitted, or both, and consider the sample to be invalid if there is evidence that the sampling pump was not operating properly throughout the sampling period (since last checked). Measure the volumetric flow rate at the end of the sampling period using the flow meter, and record the measured value. If appropriate, measure the atmospheric temperature and pressure at the end of the sampling period using the thermometer and barometer, and record the measured values.

9.5.4 Carefully record the sample identity and all relevant sampling data. Calculate the mean volumetric flow rate by averaging the volumetric flow rates at the start and at the end of the sampling period and, if appropriate, calculate the mean atmospheric temperature and pressure. Calculate the volume of air sampled, in litres, at atmospheric temperature and pressure, by multiplying the mean flow rate in litres per minute by the duration of the sampling period in minutes.

9.6 *Sample Transportation:*

9.6.1 For samplers that collect airborne particles on a filter, remove the filter from each sampler, place in a labelled filter transport cassette and close with a lid. Take particular care to prevent the collected sample from becoming dislodged from heavily loaded filters. Alternatively, transport samples to the laboratory in the filter holders in which they were collected.

9.6.2 For samplers that have an internal filter cassette, remove the filter cassette from each sampler and fasten with its lid or transport clip.

9.6.3 For samplers of the disposable cassette type, transport samples (collected on filters or by using inserts) to the laboratory in the samplers in which they were collected.

9.6.4 Transport the samples to the laboratory in a container which has been designed to prevent damage to the samples in transit and which has been labelled to assure proper handling.

9.6.5 Ensure that the documentation which accompanies the samples is suitable for a "chain of custody" to be established (see, for example, Guide D4840).

10. Analysis

10.1 To measure crystalline silica in the collected samples, choose one or more of the procedures described in Annex A1 – Annex A3 (KBr disc (pellet) technique, indirect redeposition method, or direct on-filter measurement, respectively). Carry out analysis in accordance with the selected technique(s), taking into consideration the suspected interferences that may be present in the samples obtained from the occupational setting(s) of interest.

NOTE 19—The infrared signal due to crystalline silica is particle-size dependent, with smaller particles giving greater signal relative to larger particles (**13 and 14**). Correction techniques can be applied during analysis in efforts to address the particle size dependence of the IR response (**14**).

10.2 For further guidance on the applicability of the various IR techniques, refer to Annex A1 – Annex A3.

11. Precision and Bias

11.1 *KBr Disc (Pellet) Method:*

11.1.1 A summary of KBr disc (pellet) method parameters evaluated by various organizations in four countries is presented in Table 2. In most cases, filter collection with low-flow (≈2 L/min) respirable samplers was carried out, with sampling volumes ranging from 300 to 1 000 L. After sample ashing with a plasma asher or muffle furnace, the remaining material was homogenized with potassium bromide and pressed into a KBr pellet (Annex A1). IR spectrophotometric measurements were then performed on the as-prepared KBr discs. By comparing sample IR responses to the signals from similarly-prepared quartz standards (with interference correction), the contents of respirable quartz were measured. Applicable analytical ranges were from 3 to 900 µg quartz per sample, with estimated method detection limits (MDLs) of 5 µg or less. Reported precision estimates, in terms of coefficients of variation (CVs), were 15% or less at quartz masses of 30 µg per sample.

11.1.2 In at least one published method (**15**), a correction technique has been applied in an effort to minimize the bias induced by the particle size dependence of the IR response. The KBr disc method has been used to measure silica in samples of respirable dusts collected from various airborne environments, for example coal mines (**16**), construction sites (**17**), granite quarries (**18**), and gold mines (**19**). Laboratories using the KBr disc method have performed successfully in inter-laboratory proficiency analytical testing programs (**20 and 21**).

11.2 *Redeposition Method (Indirect IR Filter Measurement):*

11.2.1 A summary of redeposition IR method parameters evaluated by four organizations in three countries is presented in Table 3. After sample collection onto filters (or foams) using low-flow respirable samplers, samples were usually subjected to ashing in a plasma asher or in a muffle furnace to remove potential interferences; one laboratory employed a calcination protocol. After sample preparation, all laboratories redeposited the remaining particulate matter onto 0.45-µm acrylic copolymer filters prior to subsequent IR analysis (Annex A2). IR spectrophotometric measurements were then performed on the as-prepared filters. By comparing sample IR responses at (800 and 780 cm^{-1}) to the IR signals from similarly-prepared quartz standards (with interference correction), the contents of respirable quartz were measured. For laboratories that employed filter-based sampling, applicable analytical ranges were from 20 to 400 µg quartz per sample, with estimated method detection limits (MDLs) of 10 µg or less. Reported precision

TABLE 2 KBr Disc (Pellet) Methods Evaluated

Method	US NIOSH 7602 (**22**)	UK MDHS 38 (**23**)	Spain INSHT MTA/MA-057/A04 (**24**)	Spain INS IT05 (**25**)	France AFNOR X43–243 (**15**)
Sampler	10-mm nylon 1.7 L/min; Higgins-Dewell 2.1 L/min	Higgins-Dewell 1.9 L/min	10-mm nylon 1.7 L/min	Casella 1.9 L/min	CIP 10-R 10 L/min
Sampling medium	Filter, 37-mm 0.8-µm MCE or 5-µm PVC	Filter, 37-mm 0.8-µm MCE or 5-µm PVC	Filter, 37-mm 0.8-µm PVC	Filter, 37-mm 5-µm PVC	Foam, polyurethane
Air volume	400–1000 L; total dust <2 mg	≥456 L; total dust ≤0.7 mg	300–500 L; total dust <5 mg	300–500 L; total dust <5 mg	Total dust ≈0.5 mg
Sample preparation	RF Plasma or Muffle furnace	Muffle furnace	Muffle furnace	Muffle furnace	Calcination filter or foam
Calibration	Quartz in KBr	Generated quartz on PVC; then KBr	Quartz in KBr	Quartz in KBr	
Range (µg quartz)†	10–160	5–700	10–160	3–900	
Estimated MDL[A] (µg quartz)	5	Varies with particle size	5	1	
Precision	CV[B] = 15 % at 30 µg	CV[B] = 5 % at 50 µg	CV[B] < 15 % at 30 µg	CV[B] < 15 % at 30 µg	

† Editorially corrected.

[A] Method detection limit.

[B] Coefficient of variation.

TABLE 3 Redeposition Methods Evaluated

Method	US NIOSH 7603 (**26**)†	US MSHA P-7(**27**)	Canada RSST 78(**28**)	France AFNOR X43–243 (**15**)
Sampler	10-mm nylon 1.7 L/min; Higgins-Dewell 2.1 L/min	10-mm nylon 1.7 L/min (+ correction factor)	10-mm nylon 1.7 L/min	10-mm nylon (≈2 L/min)
Sampling medium	Filter, 37-mm 0.8-µm MCE or 5-µm PVC	Filter, 37-mm pre-weighted 0.8-µm MCE or 5-µm PVC	Filter, 37-mm 5-µm PVC	Foam, polyurethane
Air volume	300–1000 L; total dust <2 mg		800 L	Total dust <0.5 mg
Filter preparation	RF Plasma or Muffle furnace	RF Plasma	RF Plasma	Calcination filter or foam
Calibration	Liquid deposit in 2-PrOH on 0.45-µm acrylic copolymer filter	Liquid deposit in 2-PrOH on 0.45-µm vinyl/acrylic copolymer filter	Liquid deposit in 2-PrOH on 0.45-µm acrylic copolymer filter	
Range (µg quartz)†	30–250	25–250	20–400	
Estimated MDL[A] (µg quartz)	10	3	6	
Precision	CV[B] = 9.8 % at 100–500 µg	CV[B] = 5–10 % at 100–500 µg	CV[B] < 10 % at 100–500 µg	

† Editorially corrected.

[A] Method detection limit.

[B] Coefficient of variation.

TABLE 4 Direct On-Filter Methods Evaluated

Method	UK MDHS 37 (**36**)	UK MDHS 101 (**37**)
Sampler	Higgins Dewell 2.2 L/min	Higgins Dewell 2.2 L/min
Sampling filter	37-mm, 0.8 µm MCE or 5-µm PVC	37-mm 5-µm PVC
Air volume	≥456 L; total dust <1 mg	500 L
Calibration	Generated quartz dust on PVC filter	Generated quartz dust on PVC filter
Range (µg quartz)†	10–1000	10–1000
Estimated MDL[A]	Varies with particle size	3 µg
Precision	CV[B] = 5 % at 50 µg	CV[B] < 5 % at 50–200 µg

† Editorially corrected.

[A] Method detection limit.

[B] Coefficient of variation.

estimates for these sampling and laboratory methods, in terms of coefficients of variation (CVs), were 10 % or less at quartz masses of 50–500 µg per sample.

11.2.2 The redeposition IR method has been used extensively to measure respirable quartz in samples of coal mine dust (**29-32**); generally quartz is the only polymorph of crystalline silica that is found in coal. The filter redeposition technique has also been applied to the determination of airborne respirable quartz in a foundry (**33**). Laboratories using the redeposition method have performed successfully in an inter-laboratory proficiency analytical testing program (**21**). The redeposition IR technique has been shown to be effective for quartz measurement using high-flow as well as low-flow respirable samplers (**34**).

11.3 *Direct On-Filter Method:*

11.3.1 An overall summary of the evaluation of the direct on-filter IR measurement of respirable crystalline silica (Annex A3) is presented in Table 4. After sampling onto PVC filters (normally) using low-flow rate cyclones, direct on-filter measurements of crystalline silica in the filter deposits were carried out over a range of sample loadings. Calibration was performed using filters prepared with generated atmospheres of quartz in an aerosol chamber. Software techniques were used to subtract interfering bands from the IR spectrum of the samples. While reported method detection limits (MDLs) of ≈3 µg per sample compare favorably with MDLs of indirect IR methods (see 11.1 and 11.2), it is noted that matrix and mineral interferences can be substantial, especially with highly-loaded samples of variable particle size (**35**). Reported precision as the coefficient of variation (CV) is 5 % or less for respirable quartz masses of 50–200 µg per sample.

11.3.2 The direct on-filter technique has been used to measure quartz in samples from a variety of worksites and activities including foundries (**4, 38**), mining (**39-35**), construction (**39**), ceramics operations (**4, 38**), fabrication of building materials (**4, 39**), etc. Bias due to re-radiation effects observed in heavily loaded samples when using older dispersive IR devices can be corrected for (**41**); alternatively, the use of an FTIR instrument, which is not so affected, is recommended (**37**). Laboratories using the direct on-filter IR method have performed successfully in an inter-laboratory proficiency analytical testing program (**20**).

12. Records

12.1 *Sampling Information:*

12.1.1 At a minimum, the following information on the sampling protocol shall be recorded:

12.1.1.1 A statement to indicate the confidentiality of the information supplied, if appropriate;

12.1.1.2 A complete identification of the air sample, including the date of sampling, the place of sampling, the type of sample (personal or static), either the identity of the individual whose breathing zone was sampled (or other personal identifier) or the location at which the general occupational environment was sampled (for a static sample), a brief description of the work activities that were carried out during the sampling period, and a unique sample identification code;

12.1.1.3 The make, type, and diameter of filter (or other sampling media) used;

12.1.1.4 The make and type of sampler used, including information about the target size fraction of airborne particles that the sampler is designed to collect;

12.1.1.5 The make and type of sampling pump used, and its identification;

12.1.1.6 The make and type of flow meter used, the primary standard against which the calibration of the flow meter was checked, the range of flow rates over which the calibration of the flow meter was checked, and the atmospheric temperature and pressure at which the calibration of the flow meter was checked, if appropriate;

12.1.1.7 The time at the start and at the end of the sampling period, and the duration of the sampling period in minutes;

12.1.1.8 The mean flow rate during the sampling period, in litres per minute;

12.1.1.9 The mean atmospheric temperature and pressure during the sampling period, if appropriate;

12.1.1.10 The volume of air sampled, in litres, at ambient conditions; and

12.1.1.11 The name of the person who collected the sample.

12.2 *Analytical Information:*

12.2.1 At a minimum, the following information about the analytical method shall be recorded:

12.2.1.1 The unique sample identification code(s);

12.2.1.2 The type(s) of filter/sampling medium prepared and analyzed;

12.2.1.3 The type of sample preparation method used (ashing technique, if applicable);

12.2.1.4 The IR method used for analysis (KBr disc technique, redeposition method, or direct on-filter technique);

12.2.1.5 The IR instrument used (make/model, dispersive or FTIR device);

12.2.1.6 Quality control protocols that were followed; and

12.2.1.7 Analytical results for crystalline silica, in desired units (for example, mg/m^3 or % by weight).

13. Keywords

13.1 air monitoring; infrared; respirable crystalline silica; samplers; sampling and analysis; workplace atmospheres

ANNEXES

(Mandatory Information)

A1. MEASUREMENT OF CRYSTALLINE SILICA BY IR SPECTROMETRY – KBr DISC (PELLET) METHOD

A1.1 Scope

A1.1.1 Annex A1 specifies a method for the measurement of crystalline silica in samples of respirable airborne dust, whereby the collected particulate matter is removed from the filter and introduced into a potassium bromide (KBr) matrix. The KBr matrix containing the collected particles is then pressed into a disc (pellet) under high pressure. Crystalline silica within the KBr disc is then measured using IR spectrometry.

NOTE A1.1—This procedure is also applicable to samples collected on foams.

A1.1.2 Interferences from amorphous silica, kaolinite and other silicates are minimized to the extent possible in the sample preparation and analysis procedure. A technique for removing calcite interference is also described.

A1.1.3 The method is suitable for the measurement of crystalline silica over the range 5 g to 700 g (0.025 to 0.4 mg/m^3) for a 400-L air sample.

A1.2 Principle

A1.2.1 Samples of particulate matter collected onto filters using respirable sampling devices are removed from the filters and ashed to remove interferences such as iron oxide and carbonaceous materials. This ashed material is then introduced into a KBr matrix and pressed into a disc using a high-pressure press. Each KBr disc is then placed into the sample beam of an infrared spectrometer, and the IR absorbance is measured at characteristic frequencies for crystalline silica. The measured magnitude of each absorbance is compared with measurements at the same wavenumbers on certified reference material (CRM) crystalline silica samples of similar particle size.

Note A1.2—The procedure described is also amenable to samples collected using foams.

A1.3 Analytical Reagents and Equipment

A1.3.1 *Reagents and Materials:*

A1.3.1.1 Potassium bromide (KBr), infrared quality

A1.3.1.2 Hydrochloric acid (HCl), 9 % w/w. Add 25 ml concentrated HCl (37 % w/w) to 70 ml deionized water, cool to room temperature, and bring to 100 ml with deionized water.

A1.3.1.3 Calibration stock standard, 0.5 % w/w. Accurately weigh and thoroughly mix 5 g KBr (dried overnight at 100–110°C) with 25 mg quartz, cristobalite, or tridymite CRM. Store in a glass bottle placed in a desiccator.

A1.3.1.4 Ethanol, 95 %, for cleaning sample handling equipment.

A1.3.1.5 Isopropanol.

A1.3.1.6 Kaolinite.

A1.3.1.7 Desiccant (for example, drierite).

A1.3.2 *Equipment:*

A1.3.2.1 Ashing device—Low-temperature (RF plasma) asher and aluminium (Al) weighing dishes; or muffle furnace and porcelain or platinum crucibles.

A1.3.2.2 Mortar and pestle.

A1.3.2.3 Micro-spatula, 50-mm agate or mullite metal.

A1.3.2.4 Forceps, non-serrate, non-magnetic.

A1.3.2.5 Membrane filtration apparatus, for 37-mm diameter filters, with polyvinyl chloride (PVC) filters to fit, and filtration funnel with clamps.

A1.3.2.6 Laboratory press, for preparing KBr discs (to at least 55 680 kPa pressure (10 tons per 13-mm diameter disc)), with 13-mm KBr disc (pellet) die (evacuable).

A1.3.2.7 Vacuum pump, for connecting to laboratory press.

A1.4 Sample Preparation

A1.4.1 Remove each filter sample from each sampler and, if necessary for gravimetric analysis, weigh the filter to the nearest 0.01 mg in accordance with ISO 15767. The total sample weight W_t, if measured, is the difference between the weights of the pre-weighed filter and the loaded filter after sampling.

Note A1.3—A modification of the procedure described below can be used to prepare foam samples for IR analysis.

A1.4.2 *Sample Ashing*—Use one of the following techniques (that is, either low-temperature asher or muffle furnace) to ash the samples and blanks.

A1.4.2.1 *Low-Temperature (RF Plasma) Asher*—Place the sample in a labelled aluminium (Al) weighing dish that was previously rinsed with distilled water and then ethanol and subsequently air dried. Place each Al dish in the low temperature asher so that sample exposure to the plasma is optimized. Ash the sample in accordance with the manufacturer's instructions. Then carefully bring the asher to atmospheric temperature and pressure and remove each Al dish. Place the Al dishes in a desiccator for at least one hour.

A1.4.2.2 *Muffle Furnace*—For samples expected to contain significant amounts of calcite (>20 % of total particulate loading), wash the filters with 9 % HCl and proceed to A1.4.2.2.1. Otherwise skip and proceed to A1.4.2.2.2.

A1.4.2.2.1 Place a 37-mm diameter, 5-m pore size polyvinyl chloride (PVC) filter in the membrane filtration apparatus. Remove the sample filter from the sampler and place it atop the PVC filtration filter. Clamp the filtration funnel so as to completely expose the dust deposit on the sample filter. Introduce 10 ml of 9 % HCl and 5 ml of isopropanol and allow to stand for 5 min. Apply vacuum and carefully filter the acid and alcohol in the funnel. Wash and vacuum the filter with three successive 10-ml portions of distilled water. Turn off the vacuum.

A1.4.2.2.2 Place each sample and blank into a porcelain or platinum crucible and loosely cover. Introduce the crucibles into the muffle furnace and ash for at least 2 h at 600°C (800°C if graphite is suspected in the sample). Allow the furnace to come to atmospheric temperature and pressure and remove each crucible. Place the crucibles in a desiccator for at least one hour.

A1.4.3 *Preparation of KBr Discs (Pellets):*

A1.4.3.1 Remove the ashed samples from the desiccator and weigh to the nearest 0.1 mg. Add 250–300 mg KBr (weighed to the nearest 0.1 mg and dried overnight at 100–110°C) to each ashed sample.

A1.4.3.2 Mix each ashed sample and KBr thoroughly with a microspatula. Transfer the entire mixture to the mortar and grind with the pestle.

A1.4.3.3 Quantitatively transfer the ground mixture to a 13-mm evacuable KBr disc (pellet) die and place into the laboratory press (connected to a vacuum pump). Applying vacuum, compress the die at 55 680 kPa (10 tons per 13-mm diameter KBr disc) in the press for 5 min, then release the vacuum. Remove the compressed KBr disc from the press and weigh to the nearest 0.1 mg. Store each KBr disc in a desiccator until ready for analysis.

Note A1.4—KBr absorbs moisture from the atmosphere and is therefore stored in a desiccator to prevent this.

A1.5 Calibration Test Samples

A1.5.1 Prepare at least five working standard KBr discs as follows:

A1.5.1.1 To weighing vessels, weigh (to the nearest 0.001 mg) 5–100 mg portions of calibration stock standard containing 0.5 % CRM quartz (and cristobalite and/or tridymite, if desired) in KBr. If kaolinite is suspected in samples, also weigh 10–200 mg kaolinite (A1.3.1.6). Quantitatively transfer the weighed amounts of each material mixture to a mortar.

Note A1.5—Addition of kaolinite is especially important if its presence is suspected and if the samples were low-temperature ashed. The presence of cristobalite is rare but, if its presence is suspected, this polymorph can be measured independently of (or along with) quartz.

A1.5.1.2 Add an accurately weighed portion (200–250 mg) of KBr and quantitatively transfer to the mortar containing the weighed amounts of CRMs and grind with the pestle.

A1.5.1.3 Quantitatively transfer the ground mixture to a 13-mm evacuable KBr disc (pellet) die and place into the laboratory press (connected to a vacuum pump). Applying vacuum, compress the die at 55 680 kPa (10 tons/13-mm diameter KBr disc) in the press for 5 min, then release the vacuum. Remove the compressed KBr disc from the press and weigh to the nearest 0.1 mg. Store each working standard KBr disc in a desiccator until ready for use in calibration.

A1.6 Calibration of Infrared Spectrometer

A1.6.1 Set the infrared spectrometer to absorbance mode and to appropriate settings for quantitative analysis (for example, 4 cm^{-1} resolution or better for FTIR). Following manufacturer's instructions, check the instrument to ensure acceptable normal response.

A1.6.2 Obtain infrared spectra for each working standard KBr disc (including media blanks) between 600 and 950 cm^{-1}, and determine the absorbance crystalline silica at 800 cm^{-1}. If kaolinite was added to the working standards, measure its absorbance at 915 cm^{-1}.

NOTE A1.6—Another possibility is to measure the integrated area of the analyte peak; for example, the area beneath the quartz doublet at 800 and 780 cm^{-1} and baseline can be obtained and used to prepare the calibration graph.

A1.6.3 Rotate each working standard KBr disc by 45° and re-measure. Repeat KBr disc rotation and measurement twice more until four scans are obtained for each working standard KBr disc. Average the absorbance values from the four scans for each working standard KBr disc at 800 cm^{-1} (and 915 cm^{-1}, if applicable).

A1.6.4 If necessary (that is, where kaolinite is present in working standards), correct for measurements at 800 cm^{-1} for kaolinite interference.

A1.6.5 Plot the average absorbance at 800 cm^{-1} versus g of crystalline silica in each working standard KBr disc.

A1.6.6 If necessary (that is, where significant mineral interferences at ≈800 cm^{-1} are apparent in samples), measure and plot the IR intensities of the peaks at 695^{-1} cm for quartz (and at 625 cm^{-1} for cristobalite, if its presence is suspected).

A1.7 Measurement

A1.7.1 Set the infrared spectrometer to absorbance mode and to appropriate settings for quantitative analysis (for example, 4 cm^{-1} resolution or better for FTIR).

A1.7.2 Obtain infrared spectra for each sample KBr disc between 600 and 950 cm^{-1} and determine the absorbance of crystalline silica at 800 cm^{-1}. If kaolinite is present in samples that were ashed at low temperature, as indicated by a band at 915 cm^{-1}, also determine its absorbance.

A1.7.3 If samples show significant interference at ≈800 cm^{-1} from other minerals, measure the absorbance of quartz at 695 cm^{-1} (or cristobalite at 625 cm^{-1}, if applicable, or both).

A1.7.4 Rotate each sample KBr disc by 45° and re-measure. Repeat KBr disc rotation and measurement twice more until four scans are obtained for each sample KBr disc. Average the absorbance values for each sample KBr disc at 800 cm^{-1} (and 915 cm^{-1}, 695 cm^{-1} and 625 cm^{-1}, if applicable).

A1.7.5 If necessary (such as if kaolinite is present in samples ashed at low temperatures, as indicated by a band at 915 cm^{-1}), correct for measurements at 800 cm^{-1} for kaolinite interference. A similar protocol should be followed for any other suspected interfering minerals (Table 1) that were not eliminated during the ashing process.

A1.8 Calculations

A1.8.1 Using the calibration graph (prepared from working standard KBr discs), determine the weight W_s of crystalline silica in g for each sample, using the absorbance values obtained at 800 cm^{-1} (and corrected for kaolinite and/or other interferences, if necessary). If necessary (notably where there is significant mineral interference observed ≈800 cm^{-1}), determine Ws based on absorbance values for quartz at 695 cm^{-1} (or for cristobalite at 625 cm^{-1}, if necessary, or both).

A1.8.2 For each sample, calculate the concentration of crystalline silica, C (mg/m^3), in the volume of air sampled, V (litres):

$$C = [W_s / V], \text{ mg/m}^3 \qquad (A1.1)$$

A1.8.3 If applicable, calculate the percentage of crystalline silica (% Q) in the sample by dividing by the total sample weight, W_t:

$$\% \ Q = [W_s / W_t] \times 100 \qquad (A1.2)$$

A2. MEASUREMENT OF CRYSTALLINE SILICA BY IR SPECTROMETRY – INDIRECT REDEPOSITION METHOD

A2.1 Scope

A2.1.1 Annex A2 specifies a method for the measurement of crystalline silica in samples of respirable airborne dust, whereby the collected particulate matter is removed from the collection filter (ashed) and then redeposited onto a membrane filter for subsequent IR measurement.

NOTE A2.1—This procedure is also applicable to samples collected on foams

A2.1.2 Interference from kaolinite and calcite are minimized by using the sample preparation procedures described.

A2.1.3 The working range is 0.02 to 0.25 mg/m^3 for a 1000-L sample.

A2.2 Principle

A2.2.1 Samples of particulate matter collected onto filters using respirable sampling devices are removed from the filters and ashed to remove interferences such as iron oxide and carbonaceous materials. This material remaining after ashing is then redeposited onto an acrylic copolymer membrane filter. Each membrane filter is then placed into a sample holder, and the IR absorbance is measured at characteristic frequencies for crystalline silica. The measured magnitude of each absorbance is compared with measurements at the same IR frequency range on filter samples of certified reference material (CRM) crystalline silica of similar particle size.

NOTE A2.2—The procedure described is also amenable to samples collected using foams.

A2.3 Analytical Reagents and Equipment

A2.3.1 *Reagents and Materials:*

A2.3.1.1 2-propanol, reagent grade.

A2.3.1.2 Ethanol, 95 %, for cleaning sample handling equipment.

A2.3.1.3 Quartz calibration stock solution, 15 g/ml. Add 7.5 mg dried quartz CRM to 300 ml 2-propanol and bring to 500 ml with 2-propanol. Sonicate to mix.

A2.3.1.4 Kaolinite calibration stock solution, 100 g/ml. Add 50 mg dried kaolinite 300 ml 2-propanol and dilute to 500 ml with 2-propanol. Sonicate to mix.

NOTE A2.3—Not required if muffle furnace is used to ash samples.

A2.3.1.5 Hydrochloric acid solution, 9 % w/w. Add 25 ml concentrated HCl (37 % w/w) to 70 ml deionized water, cool to room temperature, and bring to 100 ml with distilled water.

NOTE A2.4—Required if calcite is suspected and where samples are ashed with a muffle furnace.

A2.3.1.6 Oxygen, purified.

A2.3.2 *Equipment and Supplies:*

A2.3.2.1 Ultrasonic bath, for homogenizing stock solutions.

A2.3.2.2 Filters, for calibration standards and redeposition: 47-mm diameter, 0.45-µm pore size vinyl chloride-acrylonitrile copolymer membrane.

A2.3.2.3 Ashing device: Low-temperature (RF plasma) asher and aluminium (Al) weighing dishes; or muffle furnace and porcelain or platinum crucibles.

A2.3.2.4 Filtration apparatus, for redeposition onto copolymer membrane filters: glass filtration funnel with Bakelite base and fritted support and with a 1.0-cm internal diameter neck; side-arm vacuum flask; and clamps to ensure leak-proof seals.

A2.3.2.5 Glass fiber filters, 25-mm diameter, for back-up during filtration.

A2.3.2.6 Membrane filtration apparatus, for 37-mm diameter filters, with polyvinyl chloride (PVC) filters to fit, and filtration funnel with clamps. (For removal of calcite, which is required only if using a muffle furnace for sample ashing.)

A2.4 Sample Preparation

A2.4.1 Remove each filter sample from samplers and, if necessary for gravimetric analysis, weigh the filter to the nearest 0.01 mg. The total sample weight W_t, if measured, is the difference between the weights of the pre-weighed filter and the loaded filter after sampling.

NOTE A2.5—A modification of the procedure described below can be used to prepare foam samples for IR analysis (**14**).

A2.4.2 *Sample Ashing*—Use one of the following techniques (that is, either low-temperature asher or muffle furnace) to ash samples and blanks.

A2.4.2.1 *Low-Temperature (RF Plasma) Asher*—Place the sample in a labelled aluminium (Al) weighing dish that was previously rinsed with distilled water and then ethanol and subsequently air dried. Place each Al dish in the low temperature asher so that sample exposure to the plasma is optimized. Ash the sample in accordance with manufacturer's instructions. Carefully bring the asher to atmospheric temperature and pressure and remove each Al dish. Place the Al dishes in a desiccator for at least one hour.

A2.4.2.2 *Muffle Furnace*—For samples expected to contain significant amounts of calcite (>20 % of total particulate loading), wash the filters with 9 % HCl and proceed to A2.4.2.2.1. Otherwise skip and proceed to A2.4.2.2.2.

A2.4.2.2.1 Place a 37-mm diameter, 5-µm pore size polyvinyl chloride (PVC) filter in the membrane filtration apparatus. Remove the sample filter from the sampler and place it atop the PVC filtration filter. Clamp the filtration funnel so as to completely expose the dust deposit on the sample filter. Introduce 10 ml of 9 % HCl and 5 ml of isopropanol and allow to stand for 5 min. Apply vacuum and carefully filter the acid and alcohol in the funnel. Wash and vacuum the filter with three successive 10-ml portions of distilled water. Turn off vacuum.

A2.4.2.2.2 Place each sample and blank into a porcelain or platinum crucible and loosely cover. Introduce crucibles into the muffle furnace and ash for at least 2 h at 600°C (or 800°C is graphite is suspected in the sample). Allow the furnace to come to atmospheric temperature and pressure and remove each crucible. Place the crucibles in a desiccator for at least one hour.

A2.4.3 *Redeposition:*

A2.4.3.1 The following is carried out using the filtration apparatus with the 1.0-cm funnel.

A2.4.3.1.1 While applying slight vacuum, place a 25-mm glass fiber filter on the fritted base of the funnel. Cut a 47-mm acrylic copolymer filter in half. Superimpose one half over the other, glossy sides down, and place on the glass fiber filter. (The lower half of this filter serves as a blank and is used in the reference beam of the infrared spectrometer). Position filter funnel, apply clamp and turn off vacuum.

A2.4.3.1.2 Add several millilitres 2-propanol to the funnel. Check that the funnel is securely and uniformly clamped.

A2.4.3.1.3 Place sample beakers into ultrasonic bath for at least 30 sec to ensure homogeneous dispersion.

A2.4.3.1.4 For each sample, remove beaker, wipe excess water from the outside, transfer slurry to the filtration funnel and reapply vacuum. During filtration, rinse the beaker twice with 2-propanol to remove all dust and add rinsates to funnel. Control the filtration rate to keep the liquid near the funnel top during rinsing to avoid disturbing the deposit.

A2.4.3.1.5 When the depth of liquid in the funnel reaches about 4 cm above the filter, gently rinse the inside of funnel with 2-propanol and complete filtration. Remove the clamp and lift off the funnel, taking care not to disturb and deposit. Release the vacuum.

A2.4.3.1.6 Delineate the deposit area, such as by marking around the circumference using a pencil or scriber. (This is especially important for standards or light-colored samples.) Place the acrylic copolymer filter halves in petri dishes and allow to air dry.

A2.5 Calibration Test Samples

A2.5.1 Prepare at least five working standard filters as follows:

A2.5.1.1 Place the flask containing the quartz calibration stock solution (and kaolinite calibration stock solution, if necessary) in an ultrasonic bath for 30 to 45 min to homogenize the dispersion.

A2.5.1.2 Move the flask to a magnetic stirrer and stir slowly while the flask cools to room temperature. Continue to stir slowly while preparing standards so as to maintain homogenized suspensions.

A2.5.1.3 Mount a 47-mm acrylic copolymer filter in the filtration apparatus in the same manner used to redeposit the samples. Add 5 ml 2-propanol to the funnel.

A2.5.1.4 Withdraw an aliquot of the quartz suspension containing the quartz calibration stock solution from the center of the flask. Draw liquid to the mark but do not attempt to adjust volume by draining pipet. Carefully wipe the outside of the pipet, and then drain the suspension into the filter funnel. Rinse down the inside wall of the pipet with a few millilitre of 2-propanol, draining the washings into the filter funnel. Apply vacuum to complete the filtration. Prepare quartz working standard filters to cover the range 10 to 250 g per filter.

A2.5.1.5 Carry an additional set of these standards and media blanks through all sample preparation steps monitor for contamination and losses.

A2.5.1.6 If samples were ashed in a low-temperature asher, prepare at least five kaolinite standard filters in the range of 100 to 600 g per filter in the same manner as for quartz filter working standards.

A2.6 Calibration of Infrared Spectrometer

A2.6.1 Set the infrared spectrometer to absorbance mode and to appropriate settings for quantitative analysis (for example, 4 cm^{-1} resolution or better for FTIR). Following manufacturer's instructions, check the instrument to ensure acceptable normal response.

A2.6.2 Obtain infrared spectra for each quartz working standard filter and blank filter between 600 and 950 cm^{-1}. Construct a calibration graph of IR absorbance at 800 cm^{-1} versus g quartz per filter. This graph should be linear and, with blank correction, should pass through the origin.

A2.6.3 The quartz calibration curve shall be prepared before analyzing samples as a check on the preparation of uniform deposits. Repeatability shall be <10 % on replicate standards with more than 25 g quartz.

A2.6.4 If necessary (that is, where samples were ashed using a low-temperature asher), obtain infrared spectra for each kaolinite working standard filter, and use the magnitudes of the observed kaolinite peaks at 615 cm^{-1} and ≈800 cm^{-1} as a function of kaolinite loading (g per filter) to correct for the absorbance of the quartz IR peak at 800 cm^{-1}.

NOTE A2.6—Kaolinite correction calibration curves are required when samples are ashed in a low temperature asher. Since kaolinite is not destroyed under these circumstances and has as interfering peak at ≈800 cm^{-1}, for a correction must be performed.

A2.7 Measurement

A2.7.1 Set the infrared spectrometer to absorbance mode and to appropriate settings for quantitative analysis (for example, 4 cm^{-1} resolution or better for FTIR).

A2.7.2 Obtain infrared spectra between 600 and 950 cm^{-1} for each sample that was redeposited onto acrylic copolymer filters, determine the absorbance of crystalline silica at 800 cm^{-1}.

A2.7.3 If kaolinite is present in samples that were ashed at low temperature, as indicated by a band at 915 cm^{-1}, also determine its absorbance. For each sample, use the kaolinite correction calibration curve to adjust the magnitude of the measured quartz peak at 800 cm^{-1}. A similar protocol should be followed for any other suspected interfering minerals (Table 1) that were not eliminated during the ashing process.

A2.7.4 If samples show significant interference at ≈800 cm^{-1} from other minerals present, measure the absorbance of quartz at 695 cm^{-1} (or cristobalite at 625 cm^{-1}, if desired, or both).

A2.8 Calculations

A2.8.1 Using the calibration graph (prepared from working standard filters), determine the weight W_s of crystalline silica in g for each sample, using the absorbance values obtained at 800 cm^{-1} (and corrected for kaolinite or other interferences, if necessary, or both). If necessary (that is, where there is significant mineral interference observed ≈800 cm^{-1}), determine Ws based on absorbance values for quartz at 695 cm^{-1} (and for cristobalite at 625 cm^{-1}, if necessary).

A2.8.2 For each sample, calculate the concentration of crystalline silica, C (mg/m^3), in the volume of air sampled, V (litres):

$$C = [W_s / V], \text{ mg/m}^3 \tag{A2.1}$$

A2.8.3 If applicable, calculate the percentage of crystalline silica (%Q) in the sample by dividing by the total sample weight, W_t:

$$\%\ Q = [W_s / W_t] \times 100 \tag{A2.2}$$

A3. MEASUREMENT OF CRYSTALLINE SILICA BY IR SPECTROMETRY – DIRECT ON-FILTER METHOD

A3.1 Scope

A3.1.1 Annex A3 specifies a method for the measurement of crystalline silica in samples of respirable airborne dust, whereby the collected particulate matter on the filter is analyzed directly using IR spectrometry.

A3.1.2 The method detection limit (MDL) and the accuracy of the method are dependent upon a number of parameters, for example: particle size, loading level, interfering mineral constituents, and type of IR instrument used.

A3.1.3 The working range is 0.02 to 0.25 mg/m^3 for a 1000-L sample.

A3.2 Principle

A3.2.1 A sample of respirable dust is collected on a membrane filter by the use of a respirable dust sampler. The collected particulate material is then subjected to direct on-filter IR measurement at characteristic frequencies for crystalline silica. The measured magnitude of each absorbance is compared with measurements at the same wavenumbers on filters prepared using certified reference material (CRM) crystalline silica samples of similar particle size.

A3.3 Equipment and Materials

A3.3.1 Filtration apparatus, for preparation of crystalline silica working standards on membrane filters: glass filtration funnel with Bakelite base and fritted support and with a 1.0-cm internal diameter neck; side-arm vacuum flask; and clamps to ensure leak-proof seals.

A3.3.2 Filters, PVC or acrylic copolymer membrane, to fit in the filtration funnel: substrate medium for crystalline silica working standards.

NOTE A3.1—Alternatively, an aerosol chamber can be used for preparation of crystalline silica calibration standards from test atmospheres.

A3.3.3 Quartz calibration stock solution, 15 g/ml. Add 7.5 mg dried quartz CRM to 300 ml 2-propanol and bring to 500 ml with 2-propanol. Sonicate to mix.

A3.3.4 Kaolinite calibration stock solution, 100 g/ml. Add 50 mg dried kaolinite 300 ml 2-propanol and dilute to 500 ml with 2-propanol. Sonicate to mix.

A3.3.5 Ultrasonic bath, for homogenizing stock solutions.

A3.3.6 2-propanol, reagent grade

A3.4 Calibration Test Samples

A3.4.1 Prepare at least five working standard filters as follows:

A3.4.1.1 Place the flask containing the quartz calibration stock solution (and kaolinite calibration stock solution, if required) in an ultrasonic bath for 30 to 45 min to homogenize the dispersion.

A3.4.1.2 Move the flask to a magnetic stirrer and stir slowly while the flask cools to room temperature. Continue to stir slowly while preparing standards so as to maintain homogenized suspensions.

A3.4.1.3 Mount a PVC or acrylic copolymer filter in the filtration apparatus. Add 5 mL 2-propanol to the funnel.

A3.4.1.4 Withdraw an aliquot of the quartz suspension containing the quartz calibration stock solution from the center of the flask. Draw liquid to the mark but do not attempt to adjust volume by draining pipet. Carefully wipe the outside of the pipet, and then drain the suspension into the filter funnel. Rinse down the inside wall of the pipet with a few millilitre of 2-propanol, draining the washings into the filter funnel. Apply vacuum to complete the filtration. Prepare quartz working standard filters to cover the range 10 to 250 g per filter.

A3.4.1.5 Prepare at least five kaolinite working standard filters in the range of 100 to 600 g per filter in the same manner as for quartz filter working standards.

A3.4.1.6 Carry these standards and media blanks through all sample preparation steps monitor for contamination and losses.

A3.5 Calibration of Infrared Spectrometer

A3.5.1 Set the infrared spectrometer to absorbance mode and to appropriate settings for quantitative analysis (that is, 4 cm^{-1} resolution or better for FTIR). Following manufacturer's instructions, check the instrument to ensure acceptable normal response.

A3.5.2 Obtain infrared spectra for each quartz working standard filter and blank filter between 600 and 950 cm^{-1}. Construct a calibration graph of IR absorbance at 800 cm^{-1} versus g quartz per filter. This graph should be linear and, if blank-corrected, should pass through the origin.

A3.5.3 The quartz calibration curve shall be prepared before analyzing samples as a check on the preparation of uniform deposits. Repeatability shall be <10 % on replicate standards with more than 40 g quartz.

A3.5.4 Obtain infrared spectra for each kaolinite working standard filter, and use the magnitudes of the observed kaolinite peaks at 615 cm^{-1} and ≈800 cm^{-1} as a function of kaolinite loading (g per filter) to correct for the absorbance of the quartz IR peak at 800 cm^{-1}.

A3.6 Measurement

A3.6.1 Set the infrared spectrometer to absorbance mode and to appropriate settings for quantitative analysis (for example, 4 cm^{-1} resolution or better for FTIR).

A3.6.2 Obtain infrared spectra between 600 and 950 cm^{-1} for each sample filter, and determine the absorbance of crystalline silica at 800 cm^{-1}. Sample deposition onto the filter surface is often uneven, thus the IR measurement technique should take this consideration into account, either by measuring the center of the filter or by taking multiple measurements covering the entire filter surface (and, if necessary, applying an algorithm to obtain an average IR response for multiple measurements).

A3.6.3 If kaolinite is present in samples, as indicated by a band at 915 cm^{-1}, also determine its absorbance. For each sample, use the kaolinite correction calibration curve to adjust the magnitude of the measured quartz peak at 800 cm^{-1}. A similar protocol should be followed for any other suspected interfering minerals (Table 1).

A3.6.4 If samples show significant interference at ≈800 cm^{-1} from other minerals present in the sample, measure the absorbance of quartz at 695 cm^{-1} (or cristobalite at 625 cm-1, if desired, or both).

A3.7 Calculations

A3.7.1 Using the calibration graph (prepared from working standard filters), determine the weight W_s of crystalline silica in g for each sample, using the absorbance values obtained at 800 cm^{-1} (and corrected for kaolinite or other minerals, if necessary, or both). If necessary (notably where there is significant mineral interference observed at ≈800 cm^{-1}), determine W_s based on absorbance values for quartz at 695 cm^{-1} (and for cristobalite at 625 cm^{-1}, if necessary).

A3.7.2 For each sample, calculate the concentration of crystalline silica, C (mg/m^3), in the volume of air sampled, V (litres):

$$C = [W_s / V], \text{ mg/m}^3 \tag{A3.1}$$

APPENDIX

(Nonmandatory Information)

X1. TEMPERATURE AND PRESSURE CORRECTION FOR THE INDICATED VOLUMETRIC FLOW RATE

X1.1 Bubble flow meters are preferred for measuring the volumetric flow rate because the readings they give are independent of temperature and pressure. For other flow meters, it might be necessary to apply a correction to the indicated volumetric flow rate if the temperature and pressure at the time of measurement are different to when the calibration of the flow meter was checked.

X1.2 A typical example of the need for a temperature and pressure correction is when a constant pressure drop, variable area, flow meter is used to measure the volumetric flow rate. In this instance, the following expression should be used to calculate the air sample volume:

$$V_{corr} = q_V \times t \times \sqrt{\frac{p_1 \times T_2}{p_2 \times T_1}} \tag{X1.1}$$

where:

V_{corr} = the corrected air sample volume, in litres;
q_V = the mean volumetric flow rate, in litres per minute;
t = the duration of the sampling period, in minutes;
p_1 = the atmospheric pressure, in kilopascals (kPa), during calibration of the flow meter;
p_2 = the mean atmospheric pressure, in kPa, during the sampling period;
T_1 = the temperature, in kelvin, during calibration of the flow meter; and
T_2 = the mean temperature, in kelvin, during the sampling period.

X1.3 Other flow meters can also require correction for variation in temperature and pressure. The operating instructions provided by the manufacturer should be consulted for information about any necessary correction.

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