



Designation: D7948 – 20

Standard Test Method for Measurement of Respirable Crystalline Silica in Workplace Air by Infrared Spectrometry¹

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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/m³ for a 400 L air sample or 0.02 to 0.25 mg/m³ for a 1000 L air sample, depending on the analytical method.

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

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

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

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

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

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

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2. Referenced Documents

2.1 ASTM Standards:²

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

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

D5337 Practice for Flow Rate Adjustment of Personal Sampling Pumps

D4840 Guide for Sample Chain-of-Custody Procedures

D6061 Practice for Evaluating the Performance of Respirable Aerosol Samplers

E1370 Guide for Air Sampling Strategies for Worker and Workplace Protection

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

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

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

ISO 18158 Workplace air – Terminology

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

3. Terminology

3.1 Definitions:

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

3.2 Definitions of Terms Specific to This Standard:

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.

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

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

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

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

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

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

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

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

4. Summary of Test Method

4.1 Airborne particles are collected by drawing a measured volume of air through a collection substrate/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 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 (1, 2).⁴ 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.

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 cm^{-1} for

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

TABLE 1 Minerals Potentially Encountered and Their Characteristic IR Bands (450–1000 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 |

quartz and 623 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 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. Apparatus

7.1 Sampling and Analytical Equipment:

7.1.1 Sampling Equipment:

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

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

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

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

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

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

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

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

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

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

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

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

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

7.1.2 Analytical Instrumentation:

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

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

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

7.1.3 Ancillary Equipment:

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

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

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

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

7.1.3.5 *Thermometer*, 0°C to 50°C minimum range, with resolution of 1°C or less, for measurement of atmospheric temperature, if required.

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

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

7.1.3.8 *Desiccator*, for dry storage.

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

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

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

7.1.3.12 *Magnetic stirring device*, and stir bars.

7.1.3.13 *Tweezers*.

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

NOTE 6—Examples include quartz and cristobalite NIST SRMs 1878a, 1879a, 2950, 2951, 2958, 2960 and 2957.⁵

8. Sampling Procedure

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

8.2 Preliminary Considerations:

8.2.1 *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 7—Limit values for crystalline silica typically apply to the respirable fraction of airborne particles.

8.2.2 *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:

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

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.

8.2.3 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.

8.2.4 *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 $\pm 5\%$. 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 8—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 9—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.

8.2.5 *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.

8.3 Preparation for Sampling:

8.3.1 *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.

8.3.2 *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 10—Alternatively, commercially available pre-loaded filter cassettes may be used.

NOTE 11—Samplers containing foam substrate may also be used.

8.3.3 *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 12—For impaction devices, this step would not necessarily apply.

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

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

8.3.4.2 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 13—Typical sampling flow rates are ≈ 2 L/min for “low flow rate” personal samplers, ≈ 4 L/min for “higher flow rate” personal samplers, and

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

≈10 L/min for “high flow rate” personal sampling apparatus (9).

NOTE 14—If necessary, allow the sampling pump operating conditions to stabilize before setting the flow rate.

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

8.3.4.4 *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.

8.4 *Sampling Position:*

8.4.1 *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.

8.4.2 *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.

8.5 *Collection of Samples:*

8.5.1 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 15—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.

8.5.2 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.

8.5.3 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.

8.5.4 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.

8.6 *Sample Transportation:*

8.6.1 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.

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

8.6.3 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.

8.6.4 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.

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

9. Analysis

9.1 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 16—The infrared signal due to crystalline silica is particle-size dependent, with smaller particles giving greater signal relative to larger particles (10, 11). Correction techniques can be applied during analysis in efforts to address the particle size dependence of the IR response (11).

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

10. Precision and Bias

10.1 *KBr Disc (Pellet) Method:*

10.1.1 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

TABLE 2 KBr Disc (Pellet) Methods Evaluated

| Method | US NIOSH 7602 (12) | UK MDHS 38 (13) | Spain INSH T MTA/MA-057/A04 (14) | Spain INS IT05 (15) | France AFNOR X43–243 (16) |
|--|--|--|--------------------------------------|--------------------------------------|-----------------------------|
| 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- μ m MCE or 5- μ m PVC | Filter, 37-mm 0.8- μ m MCE or 5- μ m PVC | Filter, 37-mm 0.8- μ m PVC | Filter, 37-mm 5- μ m PVC | Foam, polyurethane |
| Air volume | 400–1000 L; total dust <2 mg | \geq 456 L; total dust \leq 0.7 mg | 300–500 L; total dust <5 mg | 300–500 L; total dust <5 mg | Total dust \approx 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 (μ g quartz) | 10–160 | 5–700 | 10–160 | 3–900 | |
| Estimated MDL ^A (μ g quartz) | 5 | Varies with particle size | 5 | 1 | |
| Precision | CV ^B = 15 % at 30 μ g | CV ^B = 5 % at 50 μ g | CV ^B < 15 % at 30 μ g | CV ^B < 15 % at 30 μ g | |

^A Method detection limit.

^B Coefficient of variation.

(\approx 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 μ g quartz per sample, with estimated method detection limits (MDLs) of 5 μ g or less. Reported precision estimates, in terms of coefficients of variation (CVs), were 15% or less at quartz masses of 30 μ g per sample.

10.1.2 In at least one published method (16), 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 (17), construction sites (18), granite quarries (19), and gold mines (20). Laboratories using the KBr disc method have performed successfully in inter-laboratory proficiency analytical testing programs (21, 22).

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

10.2.1 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- μ m 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 μ g quartz per sample, with estimated method detection limits (MDLs) of 10 μ g or less. Reported precision estimates for these sampling and laboratory methods, in terms of coefficients of variation (CVs), were 10 % or less at quartz masses of 50–500 μ g per sample.

10.2.2 The redeposition IR method has been used extensively to measure respirable quartz in samples of coal mine dust (26–29); 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

TABLE 3 Redeposition Methods Evaluated

| Method | US NIOSH 7603 (23) | US MSHA P-7 (24) | Canada RSST 78 (25) | France AFNOR X43–243 (16) |
|--|--|--|--|----------------------------------|
| 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 (\approx 2 L/min) |
| Sampling medium | Filter, 37-mm 0.8- μ m MCE or 5- μ m PVC | Filter, 37-mm pre-weighted 0.8- μ m MCE or 5- μ m PVC | Filter, 37-mm 5- μ m 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- μ m acrylic copolymer filter | Liquid deposit in 2-PrOH on 0.45- μ m vinyl/acrylic copolymer filter | Liquid deposit in 2-PrOH on 0.45- μ m acrylic copolymer filter | |
| Range (μ g quartz) | 30–250 | 25–250 | 20–400 | |
| Estimated MDL ^A (μ g quartz) | 10 | 3 | 6 | |
| Precision | CV ^B = 9.8 % at 100–500 μ g | CV ^B = 5–10 % at 100–500 μ g | CV ^B < 10 % at 100–500 μ g | |

^A Method detection limit.

^B Coefficient of variation.