

Industrial Hygiene Report
Control of Airborne Particles in a Lab Setting:

Silica Powder

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Abstract

Herein, a Sentry Air Systems product study of silica particle capture is reported. Due to ongoing discussion of regulations concerning the permissible level of inhalable silica particles in the work place, Sentry Air Systems conducted a study of the efficacy of ductless filter systems for the maintenance of safe breathing levels of this material. A Sentry Air Systems Ductless Containment Hood was used for the containment of a representative silica handling process.

Background

Silicon dioxide (SiO₂) — Silicon and oxygen represent the most abundant elements in the earth's crust and chemically bond to form silica, SiO₂, the most abundant mineral.¹ Pure SiO₂ exists in two crystalline forms, quartz and cristobalite. As a material, silica occupies ubiquitous applications across a range of industries including construction, petroleum, pharmaceuticals and chemicals. While silicon dioxide is chemically inert,² the dispersion of small particles of this substance in the air, known as respirable crystalline silica, represents a serious health hazard. Occupational exposure to respirable silica particles is known to cause silicosis, a chronic lung disease that can be life threatening. In addition to silicosis, uncontrolled occupational exposure to respirable silica may lead to kidney and respiratory diseases, including lung cancer. The growing concern for occupational exposure to respirable crystalline silica has prompted response from European Union regulatory bodies³ and the Occupational Safety and Health Administration (OSHA).⁴ The National Institute for Occupational Safety and Health (NIOSH) prescribes controls for workplace exposure to respirable silica, however, the regulations vary for each industry and permissible exposure limits (PELs) have not changed in 40 years.⁵ Currently, OSHA

¹ House, J. E.; House, K. A. Silicon, Germanium, Tin, and Lead. In *Descriptive Inorganic Chemistry*, 2nd ed.; Elsevier Academic Press: U.S.A., 2010.

² Cotton, F. A.; Wilkinson, G.; Murillo, C. A.; Bochmann, M. The Group 14 Elements: Si, Ge, Sn, Pb. In *Advanced Inorganic Chemistry*, 6th ed.; John Wiley & Sons: U.S.A., 1999.

³ Industrial Minerals Association - Europe. <http://www.crystallinesilica.eu/> (accessed January 10, 2014).

⁴ United States Department of Labor. <https://www.osha.gov/silica/> (accessed January 10, 2014).

⁵ Centers for Disease Control and Prevention. <http://www.cdc.gov/niosh/topics/silica/> (accessed January 10, 2014)

and NIOSH are evaluating existing regulations for control of respirable silica in the work place, and proposals for more uniform, defined engineering and administrative controls are under consideration. Due to the abundance of silica in a range of industrial applications, Sentry Air Systems (SAS) sought to determine the effectiveness of our particle filters for the capture of respirable crystalline silica.

Test Design

Most filter efficiency tests follow the same procedure: Generate a filter “challenge” inside an enclosure and measure “challenge” concentration at the filter inlet, the filter outlet, and in the ambient room air around the test enclosure. Silica particles present a significant challenge in this regard as the solid material absorbs ambient moisture easily and must be constantly agitated to produce airborne particles in significant and/or constant quantities.

For this test, a 5 lb sample of crystalline silica powder (99%, 325 mesh) was subjected to a series of agitation cycles to generate a measurable, constant plume of silica dust particles. An ordered rotation of mechanical operations including containment transfer, sifting, shaking, and whisking were repeated for the duration of the testing period. Replenishment of the silica powder was administered, as necessary, from a stock supply source placed within the containment hood. The agitated silica samples were maintained in relatively similar quantities throughout the testing period to achieve a constant particle “load” within the containment hood.

The test was based on NIOSH Test Method 7500 which specifies a minimum sample volume of 50L and a sampling rate between 1 - 3 L/min. The guidelines were used to determine a target sample volume of 400L, while sampling at 2.5 L/min was chosen to ensure that the samples contained enough analyte to be measured by the analytical lab.

Test Equipment and Setup

The unit under test in this study was an SAS 30-inch Wide Ductless Containment Hood (SS-330-DCH). The SS-330-DCH was configured with a standard carbon pre-filter pad (SS-300-CFP), and a High Efficiency Particulate Absorption (HEPA) filter (SS-300-HF) rated $\leq 99.97\%$ for particle size $\geq 0.3 \mu\text{m}$ for capture of respirable hazardous particulates (Figure 1). This is the standard equipment recommended by SAS for most applications where particles from dust or powders are a concern. The HEPA filter affects particle containment via a combination of mechanisms including interception, impaction, and diffusion.

Air sampling was conducted using SKC-branded personal air samplers (SKC-224-PCXR4), each calibrated with a BIOS International Defender 510 (Defender 510). The flow rate of each sampler was set, measured, and recorded to meet NIOSH and/or OSHA test protocol requirements. The SKC-224-PCXR4 flow rates were recorded both before and after testing and the average of the two values was used. Sample size was determined

by using the timer onboard each SKC-224-PCXR4 air sampler along with their average calibrated flow rates.

Test design dictated that each sampler be setup with a flow rate of about 2.5 L/min for a sample time of about 160 minutes. Adhering to the NIOSH test method 7500, a sample train consisting of a 10 mm cyclone and 5 micrometer PVC membrane were used as sample media.

Test samples were taken at four locations during the test. Definitions of the test points can be found in the table below.

Test Point A (T1A)	Located in the interior of the hood 6 inches below the inlet port.
Test Point B (T1B)	Located on the exterior of the hood, just above the outlet port. <i>Note: The sample taken at T1B was made with the sampling inlet hose perpendicular to exhaust airflow.</i>
Test Point C & D (T1C & T1D)	Located on a shelf in the lab space approximately 5 feet from the test apparatus and about 5 feet off the floor. <i>Note: T1C was taken before the silica load was released and T1D was taken after the silica load was released.</i>
Blank	Consists of a PVC membrane from the same lot number as the other sample membranes and was subjected to the same handling procedures as the other samples, however no dynamic air sampling was performed.

A five pound sample of commercially available silica was used to generate a test plume of dispersed silica particles in the containment hood. To achieve an “equilibrium” concentration of airborne silica particles at the outset of sampling within the hood, a complete agitation cycle was performed prior to activating the T1A & T1B samplers. A noticeable dust plume was generated following the dispensing of the test material into the three aluminum trays, and continued to be visible throughout the experiment. A regular cycle of agitation and transfer was repeated for the duration of the 160 minute testing period. Inspection of the interior of the containment hood at the conclusion of the experiment confirmed a visible film of silica dust coated the acrylic and polycarbonate interior paneling. In addition, electrostatic caking of siliceous dust was prominent throughout the surface of the interior ceiling. While a significant quantity of silica was accumulated on the base of the containment hood, the dynamic quantity under analysis remained sufficient for the duration of the testing period.

The lab space utilized for testing was an air conditioned, non-vented room with approximate dimensions of 12’ W x 12’ L x 8’ H with the SS-330-DCH located atop a 32” long table in one corner of the room. *Note: Ceiling clearance above the unit was approximately 14.5 inches.*

After testing was completed all samples were labeled and couriered to a third party analytical lab for testing. The lab analyzed the PVC membrane samplers for SiO₂ content and reported the results in mg of SiO₂. This information was converted to mg/m³ by calculating the total volume of air sampled at each test point.

Items Naming

T1P1; Large Plastic Container
 T1P2; Aluminum Pan, 13" x 9" x 2"
 T1P3; Aluminum Pan, 13" x 9" x 2"
 T1P4; Aluminum Pan, 13" x 9" x 2"
 T1P5; Metal Whisk
 T1P6; Large Mixing Bowl
 T1P7; Sifter
 TM; Test Material

Procedure

1. Prepare hood for testing. Ensure SS-300-HF is properly installed and sealed in the fan-filter unit.
2. Prepare and label silica containers and equipment according to item naming scheme.
3. Arrange sample trains on the SKC-224-PCXR4 and calibrate with the Defender 510 ensuring that flow rate is +/- 5% of the target flow rate.
4. Place samplers in their respective locations and arrange containers and equipment in the hood.
5. Prior to dispensing the test material, start initial ambient sample T1C and allow full sample to be taken.
6. Pour TM from primary container into T1P1.
7. Using T1P1, pour roughly equal amounts of TM into T1P2, T1P3, & T1P4.
8. Shake each of T1P2, T1P3, & T1P4 for about 20 seconds each. Start sampler at test point T1D and wait 20 minutes.
9. Pour T1P2 into T1P3.
10. Pour T1P3 into T1P4.
11. Sift T1P4 into T1P6 with T1P7.
12. Using T1P5, whisk T1P6 vigorously for two minutes.
13. Pour T1P6 into T1P1.
14. Begin sampling at test points T1A & T1B and repeat steps 7-13 for the duration of the 160 minute testing period.
15. Discontinue sampling at test points T1A & T1B.
16. Begin sampling at test point T1D and continue for 160 minutes.
17. Disassemble all sample trains, cap and label all samples, and pack for shipping.

Data/Findings

	SiO ₂ Industrial Hygiene Test Data		HHH Laboratories Analysis Results		
Test Point	Sample Time (Min.)	Sample Flow Rate (mL/min)			SiO ₂ (mg)
		Pre-test	Post-test	Avg.	
T1A	163	2538.1	2530.9	2534.5	0.39
T1B	163	2458.8	2465.4	2462.1	<0.01
T1C	162	2551.4	2647.2	2599.3	<0.01
T1D	161	2432.8	2556.1	2494.5	<0.01

Calculated Results			
Sample Indicated Concentration			
Test Point	Sample Volume (m ³)	SiO ₂ (mg/m ³)	SiO ₂ (PPM)
T1A	0.41312	0.63	0.26
T1B	0.40132	<0.03	<0.01
T1C	0.42108	<0.03	<0.01
T1D	0.40161	<0.03	<0.01

Indicated Performance
SiO ₂ (% Efficiency)
≥ 95%

Results Summary

HIH Laboratories processed the samples and measured SiO₂ content using X-ray powder diffraction analysis (XRD) monitoring for quartz and cristobalite, the two crystalline forms of SiO₂.⁶ According to the analysis, the detection limit for SiO₂ is 0.01 ppm or 0.03 mg/m³. Therefore, for samples containing negligible quantities of SiO₂, a concentration less than or equal to the detection limit of XRD analysis is assumed.

In evaluating the efficiency of the Sentry Air Systems containment hood, the detection limit of the XRD technique becomes an important consideration. The minimum efficiency rating, within analytical error, is 95%. Although the actual efficiency may be much higher, the prescribed instrumental technique limits ambient background detection to a minimal concentration of 0.01 ppm. Increasing the silicon dioxide test load concentration, or improving the sensitivity of the instrumental technique, would likely result in a significantly improved efficiency rating.

Lab analysis found 0.26 mg of quartz based SiO₂ at test point T1A, which corresponds to an enclosure SiO₂ concentration of about 0.63 mg/m³; XRD data suggests negligible contribution from the cristobalite form of SiO₂, and was not factored in the efficiency calculation. Consideration of the total membrane mass data indicates a total particle load of 0.94 mg, likely due to the presence of solid materials other than SiO₂. For the purposes of the title analysis, only the confirmed SiO₂ species concentration was allowed for determination of the filter efficiency rating. The concentration at the filter outlet port, T1B, reflects only the minimal detection limit of 0.03 mg/m³.

Based on the measured difference in concentration across the filter stack, the indicated filter efficiency for SiO₂ using SAS' HEPA filter media was ≥ 95%. During the course of

⁶ NIOSH method 7500 lists tridymite as another crystalline form of silicon dioxide; however, structural studies suggest tridymite is more likely a solid solution of mineralizer and silica rather than a discrete crystalline species. For reviews of SiO₂ structures, see C. R. Helms and B. E. Deal, eds., *The Physics and Chemistry of SiO₂ and Si-SiO₂ Interface*, 2nd ed., Plenum Press, New York, 1993; E. Philippot, et al., *J. Solid State Chem.* **1996**, 123, 1.

the test the room SiO₂ concentration remained unchanged and below the detection limit of the XRD analysis.

Conclusions and Considerations

Given the results presented here, it is likely that the use of the SS-330-DCH configured with SS-300-HF for work with SiO₂ and related respirable particulates would prove beneficial in reducing operator exposure to respiratory hazards presented by these materials. Further, it is to be expected that use of this equipment may reduce operator exposure to respiratory hazards below what exposure would be expected if no precautions were taken. While the putative efficiency factor is expected to be defined by the HEPA filter rating (99.97% for 0.3 μm particles), the system effectively reduces silicon dioxide particulates to a level comparable to the NIOSH PEL of 50 μg/m³ for respirable SiO₂.⁷

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⁷ NIOSH proposes a revised PEL for respirable silica of 50 μg/m³. The detection limit of XRD does not allow for discerning SiO₂ concentrations below 30 μg/m³. However, within experimental error, the results suggest the revised NIOSH exposure limit is achievable under the described conditions.