Statistical Assessment of Performance Tests for the Analysis of Respirable Crystalline Silica (Quartz) by Commercial Laboratories Using XRD

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1. Introduction

The U.S. Occupational Safety and Health Administration (OSHA) plans to propose a new occupational health standard for respirable crystalline silica (RCS), the most common form of which is quartz dust (e.g., sand).[1] The current permissible exposure limit (PEL) for RCS is equivalent to 100 μg/m^3 averaged over an eight-hour sampling period.[2] OSHA's forthcoming proposal is expected to lower the PEL by a factor of 2, from 100 to 50 μg/m^3 (consistent with recommendations from NIOSH[3]) and possibly to set an action level of 25 μg/m^3 at which various ancillary requirements of the standard would be triggered. Commercial laboratories will analyze the bulk of the RCS samples collected during exposure monitoring of silica-exposed workers. Such samples typically are collected to assess workplace exposures with respect to a benchmark such as the PEL or an action level. It is of great practical interest, therefore, to assess the accuracy, precision, and reliability of analytical results that might be expected from AIHA-accredited commercial laboratories for filters with quartz dust loadings corresponding to RCS exposure concentrations of 100, 50 and 25 μg/m^3.

To help with this assessment, the American Chemistry Council (ACC) Crystalline Silica Panel sponsored an RCS performance testing program. Filters containing three different

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[1] See 78 Fed. Reg. 44284 (July 23, 2013). In the balance of this report, the terms "RCS," "silica," "quartz," and "quartz dust" all refer to respirable crystalline silica and will be used interchangeably.

[2] http://www.cdc.gov/niosh/docs/81-123/pdfs/0553.pdf (accessed July 30, 2013).

[3] http://www.silicosis.com/niosh/ (accessed July 30, 2013).

loadings of respirable quartz dust were sent over a period of several months to five different commercial laboratories for analysis. The design of the program and the results of the analyses are described below, along with a statistical assessment of the findings.

2. Materials and Methods

ACC contracted with the RJ Lee Group (Monroeville, PA) to prepare PVC filters containing different levels of respirable quartz dust loadings for use in a blinded testing program. The filters were sent to five AIHA-accredited commercial laboratories that perform analyses of RCS using x-ray powder diffraction (XRD) methodology. The program was blinded in the sense that the labs were not informed that they were participating in a performance testing program. The filters were submitted with standard chain of custody forms as if they were collected during ordinary workplace monitoring of crystalline silica exposures by commercial customers.

The testing program included three replicate rounds of testing over a period of months, so that precision and accuracy could be assessed on an intra-laboratory as well as an inter-laboratory basis. For each round, three loading levels of respirable quartz (the “reference levels”) were deposited onto new polyvinyl chloride (PVC) filters by the RJ Lee Group. These loadings were the masses of respirable quartz that would be collected during 8-hour monitoring at a sampling rate of 1.7 liters of air/minute when concentrations of respirable quartz dust in air are 25, 50, and 100 $\mu g/m^3$. The resulting reference levels (masses) of quartz dust on the filters were 20, 40 and 80 µg of quartz, respectively.

To assess the impact of other mineral interferences on reported lab results, filters for each reference level of RCS were to be prepared with three different matrices: (1) silica only (Min-U-Sil 5; U.S. Silica Corp., Frederick, MD); (2) silica mixed with respirable kaolin; and (3) silica mixed with respirable soda-feldspar (SRM 99a; NIST). In addition, a blank filter was sent to each lab in each round. Thus, in each replicate round, each of the five labs received 10 filters: 3 pure silica samples, 3 silica/feldspar samples, 3 silica/kaolin samples, and 1 blank filter – for a total of 150 samples (10 filters x 5 labs x 3 rounds). A more complete description of RJ Lee’s filter preparation procedure is contained in Appendix B (letter of May 1, 2012 from Drew R. Van Orden of RJ Lee Group to Jackson Morrill of ACC).

In connection with the mixed matrix filter preparation, there was a misunderstanding between RJ Lee Group and the study's sponsor that resulted in loading 10, 20 and 40 µg of quartz (and equivalent amounts of kaolin or feldspar) on the mixed matrix filters, rather than 20, 40, and 80 µg of quartz.[4] The mixed matrix filters containing only 10 µg of quartz were excluded from the present analysis altogether, since there were no pure quartz filters at that level, and because for almost all those filters (27/30), the labs reported that any quartz present was below the estimated limit of detection (which ranged from 5 µg to 12 µg for the various labs). That left 120 sample results for analysis in this paper.

Initially, RJ Lee attempted to deposit the respirable dust (quartz with or without feldspar or kaolin) onto PVC filters with a 5 µm pore size. However, the mass deposited on these filters was not well retained during filter preparation, perhaps due to loss through the pores, as the Min-U-Sil 5 median particle size is 1.7 µm. To overcome this difficulty, RJ Lee switched to PVC filters with 0.8 µm pores, depositing the minerals suspended in 2-propanol by vacuum aspiration. The filters were then dried and desiccated, placed in standard 2-piece 37-mm sampling cassettes with a unique identification code, and sealed in zip-lock plastic bags, which were boxed and shipped by commercial carrier to Sandler Occupational Medicine Associates (SOMA, Gaithersburg, MD).

For each round of testing, SOMA labeled and shipped the samples with a standard chain of custody form to five commercial laboratories. As previously mentioned, these laboratories were not informed that they were participating in a performance testing program and did not know the reference levels of quartz on the various filters. The selected labs met the following criteria: they accepted samples on a commercial basis; they performed silica analysis by XRD; and they had successfully completed the AIHA-LAP accreditation protocol, as revised in April, 2010. The labs were also located in several different regions of the country. They were asked to analyze the silica mass on each filter and report the results back to SOMA as their client.

During the first round of testing, two laboratories reported difficulty preparing the filters for XRD analysis. These two labs used tetrahydrofuran (THF) as a filter-dissolving agent, and it

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[4] 10, 20, 40 and 80 µg quartz were RJ Lee's target loading levels. For some of the filters, the actual loadings achieved were 1 µg higher or lower than the targeted value.

appears that filters with 0.8 µm pores may have interfered with the dissolution of the filters. By contrast, the three labs that used a muffle furnace to ash the filters before depositing the minerals onto silver membranes did not report any sample preparation problems. Accordingly, the two labs that used the THF process were replaced with labs that used a muffle furnace. The resulting group of five labs employing the muffle furnace preparation procedure was used throughout the remainder of the test program.

Appendix A provides the full data set of study design and results for the 105 filters with quartz loadings at the 20, 40 and 80 µg reference levels and the 15 blanks. In Appendix A:

- Column 1 (“Filter No.”) shows an assigned number for the particular filter.
- Column 2 (“Filter Loading Reference Level (µg Silica)”) shows the reference level (in micrograms of quartz) on the filter.
- Column 3 (“Matrix”) indicates whether the filter contained pure quartz or quartz plus kaolin or feldspar.
- Column 4 (Lab results (µg Silica)”) shows the quantity of crystalline silica (in micrograms) reported by the lab that analyzed the filter. All of the laboratories had minimum detection levels below which silica mass could not be reliably quantified. These detection limits varied among the labs. Where a lab reported the silica mass as being below its detection limit, the notation “0” appears in Column 4.
- Column 5 (“Lab No.”) identifies the laboratory that analyzed the filter. The laboratories are identified here by arbitrarily assigned numbers from 1 to 5.
- Column 6 (“Round No.”) indicates which of the three rounds of testing the filter was in.

The data shown in Appendix A were analyzed by comparing mean reported silica mass on filters from different reference concentrations; and, conversely, by examining the distributions of reference concentrations in different quartiles of the reported silica mass distribution. Variability in reported silica mass for each reference concentration was compared within and among laboratories, using tree-based (CHAID-like analysis) implemented in KnowledgeSeeker™ (Angoss Software Corp., Toronto, Canada), a commercial classification tree and data mining software package to evaluate inter-laboratory differences. All other analyses and comparisons were performed using the Statistica 10.0 (StatSoft, Tulsa, OK) statistics environment.

3. Results and Discussion

Accuracy of Laboratory Results and Ability to Discriminate Between Higher and Lower Concentrations

In the following analysis, filters for which the silica mass was reported to be below the detection limit were excluded, thereby producing higher mean values (hence, better apparent accuracy) and smaller standard deviations (hence, better apparent precision) than would have been the case if these "non-detect" results had been included in the analysis with an assigned value of 0 or one-half the detection limit.

Table 1 shows – for each reference level – the arithmetic mean of the reported silica mass and the standard deviation for all of the silica-only filters having a positive reported value. Figure 1 plots these values for each reference level. As can be seen, the mean reported silica mass increased as the underlying reference level increased. The 95% confidence intervals (vertical bars) around the mean reported levels show that the reference level of 40 μg of silica had mean reported silica values greater than those from the 20 μg reference level and significantly less than those from the 80 μg reference level. However, none of the mean reported silica values for any of the three reference levels came within 30% of the applicable silica reference value: they are all significantly lower, indicating that the accuracy of the analyses was problematic even when the non-detects were excluded. Had they been included, the mean reported values would have fallen further below the respective reference levels, making the accuracy of the results even more problematic.

Table 1. Mean reported silica mass (µg) and standard deviations for silica-only and blank filters having positive reported results

Reference level (micrograms)	Lab results, ug Means	Number of data points N	Lab results, ug Std.Dev.
0	28.85	2	32.74
20	13.36	7	5.21
40	22.93	12	11.22
80	46.91	12	18.31

Figure 1. Mean reported silica mass (µg) on silica-only and blank filters (vertical axis) increased with reference masses of silica dust on filters (horizontal axis).

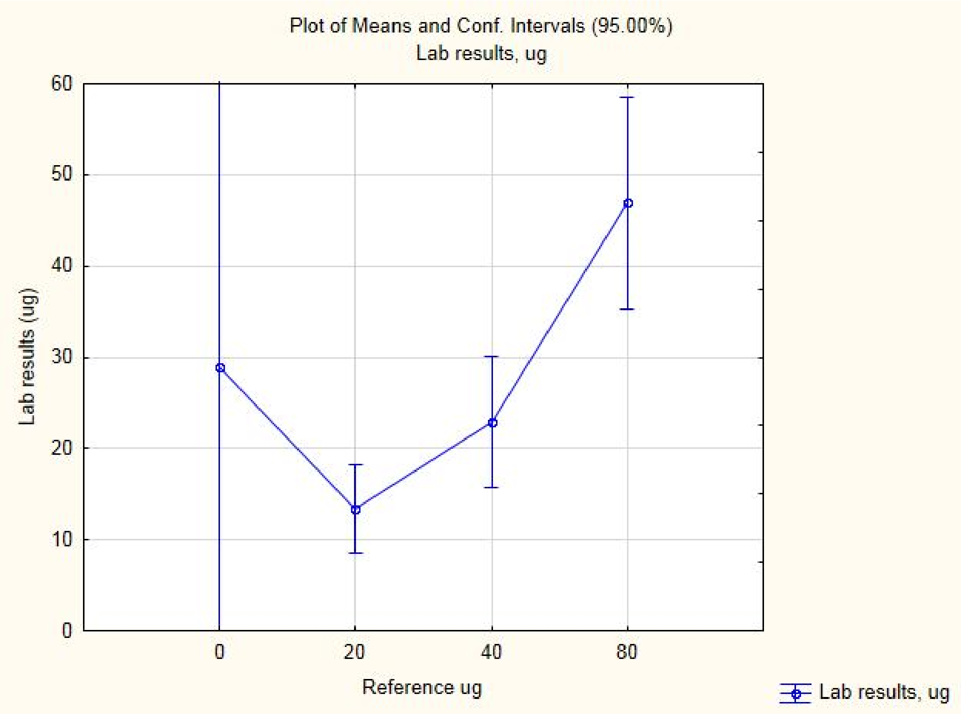

Table 2 shows – for the 20, 40 and 80 µg reference levels – the arithmetic mean of the reported silica mass and the standard deviation for all of the filters (mixed-matrix as well as silica-only) having a positive reported value (i.e., with non-detects excluded). Figure 2 plots these values for each reference level. The results are similar to those in Figure 1, which was limited to the silica-only filters. As in Figure 1, the mean values would be lower (i.e., accuracy would deteriorate) and the standard deviations would be larger (i.e., precision would deteriorate) if the non-detect filters were included. The impact of excluding the non-detects could be significant, as 34% (36 out of 105) of the filters with positive reference level loadings of RCS were reported as non-detects, including 25 of 45 (56%) of the 20 µg reference level filters. Together, Figures 1 and 2 indicate that the accuracy and precision of the reported results leave much to be desired at all three reference levels.

Table 2. Mean reported silica mass (µg) and standard deviations for all 20, 40 and 80 µg reference level filters having positive reported results

Reference level (micrograms)	Lab results, ug Means	Number of data points, N	Lab results, ug Std.Dev.
20	13.33	20	4.50
40	25.04	37	9.08
80	46.91	12	18.31

Figure 2. Mean reported silica mass (µg) on non-blank filters with positive reported results (vertical axis) increased with reference masses of silica dust on filters (horizontal axis).

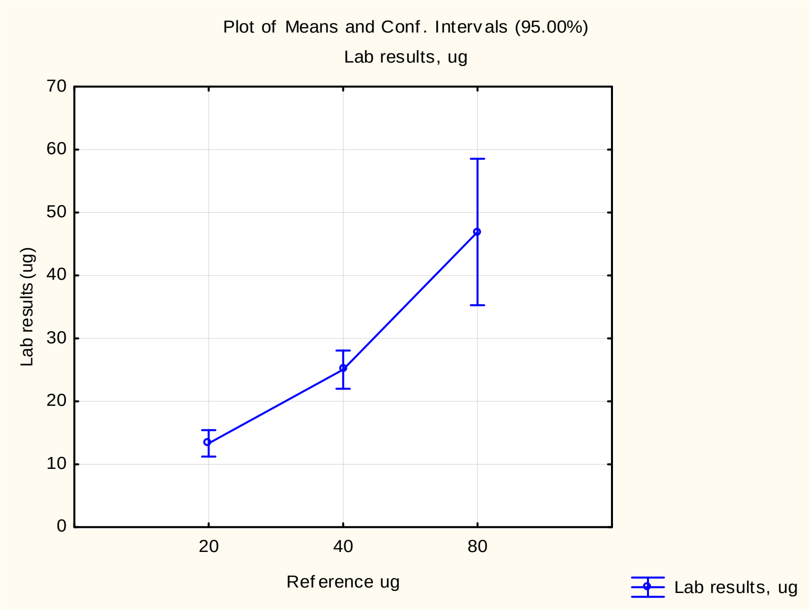

Table 3 presents data for all 71 filters with reported silica mass above the detection limits. The filters are sorted in order of increasing reported silica mass, from 5.1 µg to 66 µg. Among the 10 filters with the lowest reported silica mass above the detection limit, one came from the 0 μg reference level set. This is a false positive, in the sense that positive silica content is identified for a blank filter prepared without any silica. Six of the remaining nine filters with the lowest reported values came from the 20 μg reference level set and three from the 40 μg reference level set. For the ten filters with the highest reported silica loadings, eight (80%) are from the 80 μg reference level, one from the 0 μg reference level, and one from the 40 μg reference level (silica and kaolin matrix). For the top quartile (18 filters with the highest reported silica concentrations), almost half (8) are from the 40 μg reference level set, and one is from the 0 μg reference level set.

These data indicate that the silica mass reported by the labs does not sharply discriminate among different reference levels of actual silica loadings. For example, a reported silica mass in the highest quartile (all of which would be from the 80 μg reference level set if there were perfect agreement between higher reference levels and higher reported silica mass) has about a 50% (9/18) chance of coming from the 40 or 0 μg reference level sets. Conversely, a reported silica mass in the lowest quartile has some chance (about 1/18) of being a false positive and a greater probability (about 4/18, or 22%) of coming from the 40 μg reference level set. Thus, if one wished to identify a threshold for reported silica mass above which one could be confident (e.g., with 95% statistical confidence) that the reported level exceeds some true level – say, 60 μg – these data indicate that such a level of confidence could not be achieved under the conditions of this performance testing protocol.[5] Indeed, among the top 10% of filters (12 out of 120) reported as having the greatest crystalline silica loads, 25% (3 of the 12) come from the 40 μg reference level set, and one is a false positive (a blank with 0 silica load) – indicating that under the conditions of this testing protocol, the laboratory results were not sufficiently accurate to reliably distinguish between concentrations that differ by a factor of 2 (i.e., 80 μg v. 40 μg).

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[5] OSHA standards typically require that employers use a method of monitoring and analysis that has an accuracy of plus or minus 25 percent (+/- 25%) with a confidence level of 95 percent for measurements at airborne concentrations at or above the PEL. See, e.g., 29 CFR § 1910.1028(e)(6) (+/- 25% for Benzene); 29 CFR § 1910.1026(d)(5) (+/-25% for concentrations at or above the action level for Hexavalent Chromium).

Table 3. Filter data sorted by increasing reported silica mass. (Only filters with reported silica content above the detection limit are shown.)

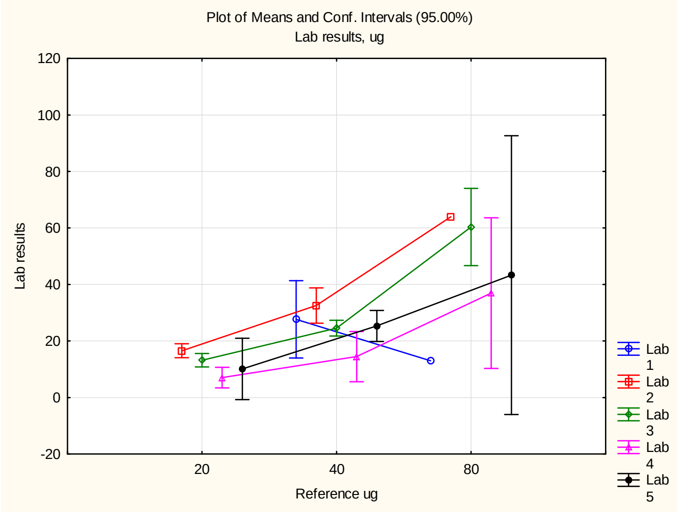
	1 Filter Loading Reference Level (ug Silica)	2 Matrix	3 Lab results (ug silica)	4 Lab No.
1	40	Silica	5.1	4
2	20	Silica + Feldspar	5.4	5
3	20	Silica	5.7	4
4	0	Blank	5.7	5
5	40	Silica + Feldspar	5.7	4
6	20	Silica	6.8	4
7	40	Silica	7.8	4
8	20	Silica + Kaolin	8.6	4
9	20	Silica + Kaolin	11	5
10	20	Silica + Kaolin	11	3
11	20	Silica + Feldspar	12	3
12	20	Silica + Kaolin	12	2
13	80	Silica	13	1
14	20	Silica + Feldspar	13	3
15	20	Silica + Kaolin	14	2
16	20	Silica + Kaolin	14	5
17	20	Silica	14	3
18	40	Silica	14	5
19	20	Silica	14	2
20	20	Silica + Feldspar	14	2
21	40	Silica	14.2	4
22	40	Silica + Feldspar	14.7	4
23	40	Silica + Kaolin	15	2
24	20	Silica	16	3
25	40	Silica + Feldspar	16	5
26	20	Silica + Kaolin	17	2
27	20	Silica	18	2
28	20	Silica	19	2
29	20	Silica + Feldspar	19	2
30	40	Silica	20	3
31	40	Silica + Kaolin	21	3
32	40	Silica	21	5
33	40	Silica + Feldspar	21	3
34	20	Silica + Feldspar	22	2
35	40	Silica + Kaolin	22	1
36	40	Silica + Kaolin	22.8	4

	1 Filter Loading Reference Level (ug Silica)	2 Matrix	3 Lab results (ug silica)	4 Lab No.
37	40	Silica + Kaolin	24	3
38	40	Silica + Kaolin	24	3
39	80	Silica	24.6	4
40	40	Silica	25	3
41	80	Silica	26	5
42	40	Silica + Kaolin	26	5
43	40	Silica + Feldspar	27	5
44	40	Silica + Feldspar	27	3
45	40	Silica + Feldspar	27	5
46	40	Silica	27	2
47	40	Silica + Feldspar	28	3
48	40	Silica + Kaolin	28	1
49	40	Silica + Kaolin	29	5
50	40	Silica	31	3
51	40	Silica + Feldspar	31	2
52	40	Silica + Kaolin	31.1	4
53	40	Silica + Feldspar	32	2
54	40	Silica + Kaolin	32	2
55	40	Silica + Kaolin	33	1
56	40	Silica + Kaolin	33	5
57	40	Silica	35	2
58	40	Silica	35	5
59	80	Silica	39	5
60	40	Silica	40	2
61	40	Silica + Feldspar	40	2
62	40	Silica + Kaolin	41	2
63	80	Silica	42.5	4
64	80	Silica	43.8	4
65	0	Blank	52	2
66	80	Silica	55	3
67	80	Silica	60	3
68	80	Silica	64	2
69	80	Silica	64	2
70	80	Silica	65	5
71	80	Silica	66	3

Variability Among Laboratories

Figure 3 shows the relation between reference levels and reported silica mass for individual laboratories (with non-detects removed).[6] The results from some laboratories (e.g., lab 2) indicate a higher silica loading than others (e.g., lab 4), for every reference level. A filter with a given load of silica particles could easily yield a reported silica mass that varied by a factor of approximately two, depending on which labs provided the analyses. This finding suggests that inter-laboratory variability alone would make it impossible to reliably discriminate among RCS exposure concentrations that differ by a factor of about 2, consistent with the values in Table 3 above. In addition, within individual laboratories, there was substantial overlap among the 95% confidence intervals around mean reported silica mass values for different reference levels. Thus, variability of reported results within individual laboratories was such that reference levels of 20, 40, and 80 μg could not be distinguished reliably from each other.

Figure 3. Individual laboratories differ systematically in reported silica mass at the respective reference levels (data with non-detects removed)



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[6] Of the 21 non-blank filters it analyzed, Lab 1 reported only four values above the detection limit (3 for the 40 μg reference level and 1 for the 80 μg reference level); hence, the plot for Lab 1 has no data point for the 20 μg reference level and no confidence limits for the 80 μg reference level.

Figure 4 below presents an interaction tree, showing in more detail how estimated mean silica mass ("avg") (and their sample standard deviations, "std") depend on specific laboratories. For all non-zero reference levels, the reported silica mass on filters depended significantly on the laboratory that performed the analysis. Laboratory 2 systematically reported silica mass levels about twice that of laboratory 4, for both the 20 and the 40 µg reference levels. These wide ranges indicate that two-fold differences in reference levels could not reliably be distinguished using the reported laboratory results.

Figure 4. Estimated silica mass varied with the laboratory that performed the analysis

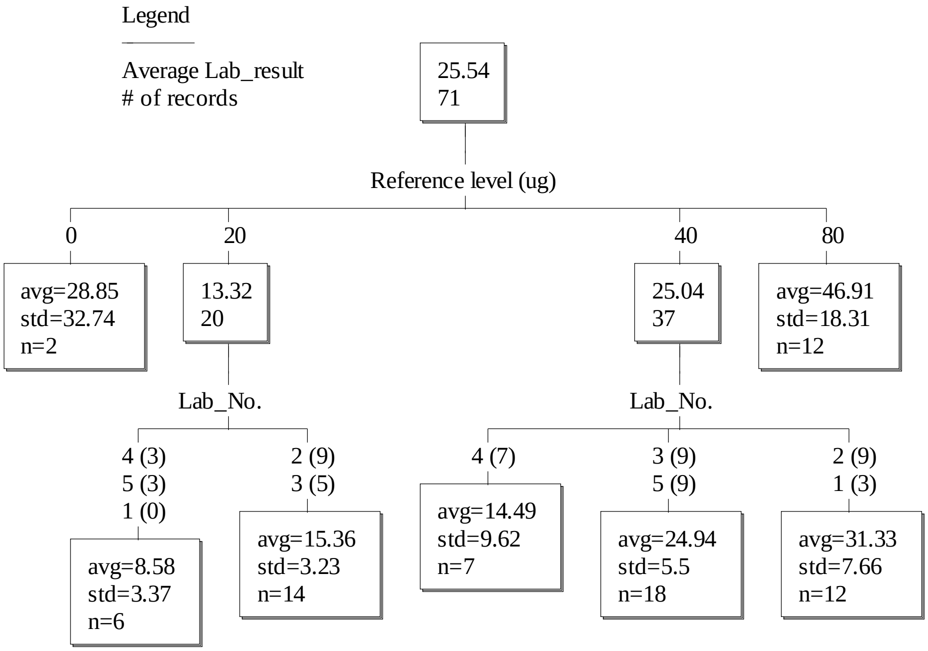

Table 4 shows that different labs have quite different coefficients of variation (ratios of standard deviations to means) for their reported results. Moreover, with the possible exception of Lab 3, the high CV values shown in Table 4 suggest that intra-laboratory precision in these analyses of silica dust is poor – with relative standard deviations ranging from 20% to 66% at the various reference levels.

Table 4. Coefficients of variation (CV) are higher for some labs (e.g., 5) than others (e.g., 3), for all reference levels.

1 Lab No.	2 Filter Loading Reference Level (ug silica)	3 Mean lab results (ug silica)	4 N = number of data points	5 standard deviation	6 CV
1	0		0		
1	20		0		
1	40	27.67	3	5.51	0.20
1	80	13.00	1	0.00	0.00
2	0	52.00	1	0.00	0.00
2	20	16.56	9	3.24	0.20
2	40	32.56	9	8.14	0.25
2	80	64.00	2	0.00	0.00
3	0		0		
3	20	13.20	5	1.92	0.15
3	40	24.56	9	3.64	0.15
3	80	60.33	3	5.51	0.09
4	0		0		
4	20	7.03	3	1.46	0.21
4	40	14.49	7	9.62	0.66
4	80	36.97	3	10.73	0.29
5	0	5.70	1	0.00	0.00
5	20	10.13	3	4.37	0.43
5	40	25.33	9	7.12	0.28
5	80	43.33	3	19.86	0.46

Conclusions

This study examined the performance of five commercial laboratories in analyzing quartz filter loadings of 0, 20, 40 and 80 µg in a blind test design where some of the filters contained only quartz dust, some contained quartz embedded in a mineral matrix with kaolin, and some contained quartz embedded in a mineral matrix with feldspar. The replicate rounds were conducted over a period of months so that results would represent lab performance beyond a single point in time.

The study found that, under the test conditions, the results reported by these commercial laboratories as a group would lack the degree of accuracy and precision sufficient to reliably inform regulatory compliance decisions. That was true even when the filters contained only silica. For 36 of the non-blank filters with 20 or more micrograms of silica (i.e., 36 out of 105 non-blank filters), the laboratories reported non-detected levels of silica. Among all 69 non-blank filters with detected levels of silica (excluding two false-positive blank filters, one of which was reported to have 52 micrograms of silica), the probability that a randomly selected filter from the 40 µg reference group would have a *higher* reported silica level than a randomly selected filter from the 80 µg reference group greatly exceeded the 5% level required for conventional 95% statistical confidence (see Table 3).

Inter-laboratory variability in this performance test program was so high that the reported results could not be used to reliably discriminate among filters prepared to reflect 8-hour exposures to respirable quartz concentrations of 25, 50 and 100 µg/m^3. Moreover, even within a single laboratory, there was enough variability in the reported results so that 2-fold variations in exposure concentrations could not be reliably distinguished.

While the specific conditions of this blinded performance test program may limit the general applicability of these findings, the results point to significant potential shortcomings in the accuracy and precision of analytical results reported for quartz loadings in the neighborhood of 80 µg and below – and they indicate that for a PEL of 50 µg/m^3 and an action level of 25 µg/m^3, measurability problems could make determinations of compliance or non-compliance unreliable.

Appendix A. Full data set

Filter No.	Filter Loading Reference Level (ug Silica)	Matrix	Lab results (ug Silica)	Lab No.	Round No.
1	20	Silica	0	1	1
2	40	Silica	0	1	1
3	80	Silica	13	1	1
4	20	Silica + Kaolin	0	1	1
5	40	Silica + Kaolin	33	1	1
6	20	Silica + Feldspar	0	1	1
7	40	Silica + Feldspar	0	1	1
8	0	Blank	0	1	1
9	20	Silica	19	2	1
10	40	Silica	35	2	1
11	80	Silica	0	2	1
12	20	Silica + Kaolin	14	2	1
13	40	Silica + Kaolin	41	2	1
14	20	Silica + Feldspar	22	2	1
15	40	Silica + Feldspar	32	2	1
16	0	Blank	52	2	1
17	20	Silica	0	5	1
18	40	Silica	35	5	1
19	80	Silica	65	5	1
20	20	Silica + Kaolin	11	5	1
21	40	Silica + Kaolin	33	5	1
22	20	Silica + Feldspar	0	5	1
23	40	Silica + Feldspar	27	5	1
24	0	Blank	0	5	1
25	20	Silica	16	3	1
26	40	Silica	25	3	1
27	80	Silica	66	3	1
28	20	Silica + Kaolin	0	3	1
29	40	Silica + Kaolin	21	3	1
30	20	Silica + Feldspar	13	3	1
31	40	Silica + Feldspar	27	3	1
32	0	Blank	0	3	1
33	20	Silica	6.8	4	1
34	40	Silica	5.1	4	1
35	80	Silica	43.8	4	1
36	20	Silica + Kaolin	0	4	1
37	40	Silica + Kaolin	0	4	1
38	20	Silica + Feldspar	0	4	1
39	40	Silica + Feldspar	0	4	1

40	0	Blank	0	4	1
41	20	Silica	0	5	2
42	40	Silica	21	5	2
43	80	Silica	39	5	2
44	20	Silica + Kaolin	14	5	2
45	40	Silica + Kaolin	29	5	2
46	20	Silica + Feldspar	5.4	5	2
47	40	Silica + Feldspar	16	5	2
48	0	Blank	0	5	2
49	20	Silica	0	1	2
50	40	Silica	0	1	2
51	80	Silica	0	1	2
52	20	Silica + Kaolin	0	1	2
53	40	Silica + Kaolin	22	1	2
54	20	Silica + Feldspar	0	1	2
55	40	Silica + Feldspar	0	1	2
56	0	Blank	0	1	2
57	20	Silica	18	2	2
58	40	Silica	40	2	2
59	80	Silica	64	2	2
60	20	Silica + Kaolin	17	2	2
61	40	Silica + Kaolin	32	2	2
62	20	Silica + Feldspar	19	2	2
63	40	Silica + Feldspar	40	2	2
64	0	Blank	0	2	2
65	20	Silica	5.7	4	2
66	40	Silica	14.2	4	2
67	80	Silica	42.5	4	2
68	20	Silica + Kaolin	8.6	4	2
69	40	Silica + Kaolin	31.1	4	2
70	20	Silica + Feldspar	0	4	2
71	40	Silica + Feldspar	14.7	4	2
72	0	Blank	0	4	2
73	20	Silica	14	3	2
74	40	Silica	31	3	2
75	80	Silica	60	3	2
76	20	Silica + Kaolin	11	3	2
77	40	Silica + Kaolin	24	3	2
78	20	Silica + Feldspar	12	3	2
79	40	Silica + Feldspar	28	3	2
80	0	Blank	0	3	2
81	20	Silica	0	5	3
82	40	Silica	14	5	3

83	80	Silica	26	5	3
84	20	Silica + Kaolin	0	5	3
85	40	Silica + Kaolin	26	5	3
86	20	Silica + Feldspar	0	5	3
87	40	Silica + Feldspar	27	5	3
88	0	Blank	5.7	5	3
89	20	Silica	0	1	3
90	40	Silica	0	1	3
91	80	Silica	0	1	3
92	20	Silica + Kaolin	0	1	3
93	40	Silica + Kaolin	28	1	3
94	20	Silica + Feldspar	0	1	3
95	40	Silica + Feldspar	0	1	3
96	0	Blank	0	1	3
97	20	Silica	14	2	3
98	40	Silica	27	2	3
99	80	Silica	64	2	3
100	20	Silica + Kaolin	12	2	3
101	40	Silica + Kaolin	15	2	3
102	20	Silica + Feldspar	14	2	3
103	40	Silica + Feldspar	31	2	3
104	0	Blank	0	2	3
105	20	Silica	0	4	3
106	40	Silica	7.8	4	3
107	80	Silica	24.6	4	3
108	20	Silica + Kaolin	0	4	3
109	40	Silica + Kaolin	22.8	4	3
110	20	Silica + Feldspar	0	4	3
111	40	Silica + Feldspar	5.7	4	3
112	0	Blank	0	4	3
113	20	Silica	0	3	3
114	40	Silica	20	3	3
115	80	Silica	55	3	3
116	20	Silica + Kaolin	0	3	3
117	40	Silica + Kaolin	24	3	3
118	20	Silica + Feldspar	0	3	3
119	40	Silica + Feldspar	21	3	3
120	0	Blank	0	3	3

Appendix B