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Crystalline Silica Analysis: A Comparison of Calibration Materials and Recent Coal Mine Dust Size Distributions

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ABSTRACT: Since 1982 standard calibration materials recommended for respirable crystalline silica analysis by the Mine Safety and Health Administration (MSHA) P7 Infrared Method and the National Institute for Occupational Safety and Health (NIOSH) X-ray Diffraction (XRD) Analytical Method 7500 have undergone minor changes in size distribution. However, a critical assumption has been made that the crystalline silica in ambient mine atmosphere respirable dust samples has also remained essentially unchanged in particle size distribution. The objective of this work, therefore, is to compare recent particle size distributions of underground coal mine dust and the silica component of these dusts with estimated aerodynamic particle size distributions of calibration standard materials MIN-U-SIL 5, Berkeley 5, and SRM 1878 used by two crystalline silica analysis techniques. This work provides resolution to a previously reported discrepancy involving the proper sample dilution for the particle sizing method used.

Dust impactor sampling data for various locations in 13 underground coal mines were collected between 1991 and 1998 and analyzed for the respirable mass median aerodynamic diameters. The data suggest that the MSHA P7 Method will underestimate the silica content of the sample by at most 8.5 % in the median size range of 0.9–3.6 µm and that it is unlikely one would obtain any significant error in the MSHA P7 Method analysis when the method uses Berkeley 5, MIN-U-SIL 5, or SRM 1878 as a calibration standard material.

The results suggest that the NIOSH Analytical Method 7500 would be more appropriate for a dust sample that is representative of the total (no cyclone classifier) rather than the respirable airborne dust, particularly since the mass fraction in the size range below 4 µm is usually a small percentage of the total airborne dust mass. However, the NIOSH Analytical Method 7500 is likely to underestimate the silica content of an airborne respirable dust sample by only 5–10 %. The results of this study also suggest that any changes that may have occurred in the median respirable size of airborne coal mine dust are not significant enough to cause any appreciable error in the current methods used for respirable crystalline silica analysis.

KEYWORDS: silica, coal, analysis, XRD, infrared, calibration materials

Introduction

Crystalline silica (referred to hereafter as silica) dust has long been recognized as an occupational health hazard to the respiratory system responsible for the insidious disease known as silicosis [1–6]. In underground mining this silica dust is created from several sources which generally include, but are not limited to, cutting, drilling, or milling of rock material. For coal mining this rock material may be found as an inherent constituent of the coal, although this is usually not a significant source of silica. The majority of the silica is found in the immediate overlaying or underlaying rock strata which are frequently removed during the coal mining process. Also, significant rock inclusions known as partings occur within the coal seam itself

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and must be removed with the coal. Surface mining operations can also produce significant quantities of airborne respirable silica due to the amount of overburden which must be removed.

Monitoring the respirable coal mine dust exposure for compliance with Federal regulations is mandatory to protect the health of miners [7]. For all coal mines, if the silica content of the dust sample is measured to be 5 % or less, the respirable dust standard is 2.0 mg/m^3. If the silica content exceeds 5 %, the respirable dust standard is reduced according to the formula 10 ÷ % silica [8]. For metal and nonmetal mines, defined as all non-coal producing mines, the respirable dust standard for respirable dust that contains silica (> 1 %) is calculated according to the formula 10 ÷ (% silica + 2). However, there is no technique available for the direct measurement of the respirable silica dust concentration or size distribution in the mining environment. The only means to estimate the respirable silica dust concentration is to perform an analysis of the respirable dust sample collected, which may contain both coal and silica dust as well as other minerals.

Currently, the two fundamental techniques used for routine estimation of the silica content of a respirable dust sample are X-ray diffraction (XRD) and infrared (IR) absorption analysis. The techniques recommended by the National Institute for Occupational Safety and Health (NIOSH) are Method 7603 (crystalline silica in coal mine dust by IR) and Method 7500 (crystalline silica by XRD) [9]. For determination of the silica content of respirable coal mine dust samples, the Mine Safety and Health Administration (MSHA) employs the MSHA P7 Infrared Method, hereafter referred to as P7 [10,11]. The MSHA and NIOSH IR methods are similar but with some differences noted in the sample preparation. Airborne dust samples from metal and nonmetal mines are analyzed by MSHA using MSHA Method P2, which is nearly identical to NIOSH Method 7500.

The results of both XRD and IR techniques have long been recognized to be dependent on particle size [12–15]. However, these techniques are considered reasonably accurate if the silica particle size distribution of the sample approximates the size distribution of the calibration material for the methods and the XRD intensity peak area is used instead of intensity peak height [9]. The criteria under which Methods 7603, 7500, P2, and P7 were developed and implemented were closely scrutinized and impose strict limits on sample requirements and analysis protocol. Methods 7500 and P7 have evolved from the procedures recommended for silica determination of coal mine dust samples in 1982. At that time the product MIN-U-SIL 5[2] was recommended as the standard calibration material, based on this material's particle size distribution closely matching the silica particles found in respirable dust samples from ambient underground mine atmospheres [9,16].

Since 1982, the recommended standard calibration materials have undergone relatively minor changes in size distribution. However, a critical assumption has been made that the silica in ambient mine atmosphere respirable dust samples has also remained essentially unchanged in particle size distribution. This may not be true in some situations since the mining industry has undergone many changes in mining technology, production, and dust control methods. For example, at the time of P7 development and implementation only a small percentage of continuous mining machines in coal mines were equipped with flooded bed scrubbers to collect the dust generated during mining. It is well documented that scrubbers are very efficient in capturing ambient dust particles down to 2 µm aerodynamic diameter [17,18]. Also, among the changes in mining technology is a significant increase in the power delivered by mining machinery. Since mining frequently involves cutting the much harder rock material, which is

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[2] U.S. Silica Company, Berkeley Springs, WV.

usually the major source of silica, it is possible that there has been a change in the amount of milling or grinding and an increase in the actual breakage of this rock material due to these more powerful machines. It would appear reasonable to suspect that some change could occur in the median size of the airborne respirable silica compared to the early 1980s when the MIN-U-SIL 5 material was chosen as the best representation of airborne respirable coal mine silica dust [16].

The objective of this work, therefore, is to compare particle size distributions of respirable underground coal mine dust with estimated aerodynamic particle size distributions of calibration standard materials MIN-U-SIL 5, Berkeley 5, and SRM 1878 by two silica analysis techniques. This was accomplished by evaluating the particle size dependency of the MSHA P7 method and the NIOSH XRD method on size fractions of MIN-U-SIL 10. MIN-U-SIL 10 was chosen to provide a broad size range so that four size fractions could be obtained. This work provides resolution to a previously reported discrepancy involving the proper sample dilution for the particle sizing method used [19]. The discrepancy resulted in an incorrect choice for the sample dilution and the subsequent size dependency relationships for the analytical techniques used.

Materials, Methods, and Analysis

Materials

The work was carried out on size fractions of MIN-U-SIL 10 silica (reported by the manufacturer to be 99.5 % pure), as described and reported previously [19]. The calibration material used for P7 was Berkeley 5 silica, which has the same manufacturer specifications as MIN-U-SIL 5 but which is produced at a different plant, according to the manufacturer. It is noted that the P7 method employed by MSHA at the time of this work used MIN-U-SIL 5 for the calibration material. The P7 procedure used in this work was routinely validated by comparison with analyses performed by MSHA and no discrepancies were ever found. The calibration material for XRD was SRM 1878 produced from MIN-U-SIL 5 by the former National Bureau of Standards and distinct from the currently available and recommended SRM 1878a, prepared by the National Institute for Standards and Technology (NIST).

Particle Sizing

Size distribution based on spherical equivalent diameter (d_{SED}) was performed by an independent laboratory. The size-classified bulk powder silica samples were prepared for size distribution analysis using two different dilutions. The first dilution dispersed the powder in IPA to achieve a suspension concentration of approximately 700 mg/L and stirring for 5 min, sonicating in a 200 Watt ultrasonic bath for 10 min, followed by re-stirring. The second dilution used the same low concentration suspensions that were produced for P7 analysis of the size fractions, which were in the range of 31–38 mg/L, followed by the sonication and stirring process. To examine possible sizing variations due to suspension concentration, the silica sample material in the largest size fraction was analyzed for size over a suspension range of 45–714 mg/L. This was achieved by subsequent dilutions of the 714 mg/L suspension. Analysis of all samples was performed on a Horiba[3] LA-920 particle size analyzer which classifies the particles into 68 size intervals below d_{SED} = 200 µm. For each sample the median d_{SED} was calculated.

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[3] Horiba Instruments, Irvine, CA.

Two groups of gravimetric size distribution data were also reported previously [19]. Group 1 consists of 83 underground samples from locations on 13 continuous mining and longwall mining operations. Group 2 size distribution data consists of 29 samples from underground continuous mining sections as well as from rock material fed to a double roll crusher in a laboratory wind tunnel.

Analysis

Two measures of individual particle size have been used in this study. These are the spherical equivalent diameter (d_{SED}), reported by the optical particle size analyzer, and the mass equivalent aerodynamic diameter (d_{AED}), measured by an aerosol impactor. Since d_{AED} is chosen as the relevant measure of particle size for aerosols sampled by aerodynamic properties, it is necessary to establish a reasonably accurate means to convert values of d_{SED} measured for the size fractions obtained from MIN-U-SIL 10. According to Baron and Willeke [20], it is possible to estimate d_{AED} from the d_{SED}. This conversion has been previously described [19]. In the present work, gravimetric measurement of the median d_{AED} using an inertial impactor for the individual size fractions obtained from MIN-U-SIL 10 was not possible since insufficient material was available. However, gravimetric measurement of median d_{AED} by an impactor and conversion to median d_{SED} for the whole product of MIN-U-SIL 10 was performed. The assumption was made that this procedure applied to the median size of the distribution and was applicable to particles of that size and the individual size fraction distributions of MIN-U-SIL 10 having median sizes $(d_{AED})_i$ and $(d_{SED})_i$.

Prepared laboratory samples of size-classified bulk powder were analyzed for silica content by (1) P7 at the NIOSH Pittsburgh Research Laboratory using a PerkinElmer[4] Spectum GX FTIR spectrometer and (2) the NIOSH XRD method at an independent laboratory participating in the Proficiency Analytical Testing (PAT) program and accredited by the American Industrial Hygiene Association. The P7 and XRD methods used were performed according to the method protocol as specified by MSHA and NIOSH, respectively, with the exception that samples prepared for P7 analysis were from size-classified bulk powder, as described earlier, instead of from an airborne respirable dust sample. XRD samples were prepared from the same sample deposit on which P7 analysis had been performed by low-temperature ashing of the deposit and substrate, followed by redeposition on a silver membrane filter. Silica reported by XRD was based on intensity peak area, as required by method 7500.

Group 2 of impactor samples were analyzed for silica by removing the dust collected on the individual stages and compositing into three groups. These groups consisted of impactor stages 1, 2, and 3 (21.3–9.8 µm); stages 4, 5, and 6 (6.0–1.55 µm); and stages 7, 8, and final (0.93–0.1 µm). Each composite was analyzed for silica according to the NIOSH XRD method at an independent accredited PAT laboratory.

Results and DiscussionBParticle Sizing

The median value $(d_{AED})_{10}$ for MIN-U-SIL 10 was determined to be 3.83 µm from an average of four measurements using Sierra Model 298 9-stage personal sampling impactors. The appropriate median value $(d_{SED})_{10}$ was determined to be 2.97 µm on the Horiba LA-920 particle

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[4] PerkinElmer, Wellesley, MA.

size analyzer. Using $(d_{SED})_{10}$ = 2.97 µm yielded $(d_{AED})_{10}$ = 3.89 µm, good agreement with the value obtained from the gravimetric determination.

Table 1 summarizes median size data for three materials used for calibration of the P7 and the XRD methods. The median d_{SED} value of 1.70 µm for MIN-U-SIL 5 is that reported by the manufacturer. The median d_{SED} values for Berkeley 5 (1.61 µm) and SRM 1878 (1.70 µm) were measured by the same technique as used for the analysis of the MIN-U-SIL 10 size fractions. These values are reported to show the relatively small differences in median size between the original calibration material MIN-U-SIL 5 and two other standard materials used for instrument calibration during these tests. Calculated values of median d_{AED} for the calibration materials listed in Table 1 are reported.

TABLE 1—*Calibration material median size data.*

Calibration materials	d_{SED}[a] µm	d_{AED}[b] µm
MIN-U-SIL 5[c]	1.70[f]	2.23
Berkeley-5[d]	1.61	2.12
SRM 1878[e]	1.70	2.23

[a] Spherical equivalent diameter.
[b] Aerodynamic equivalent diameter.
[c] 1982 recommended calibration standard.
[d] Calibration material used for P7 analysis in this work.
[e] Calibration material used for XRD Method 7500 analysis in this work.
[f] Value reported by manufacturer using sedimentation method.

Table 2 summarizes the fractionated MIN-U-SIL 10 particle size analysis results. Sample weight is the amount of size-classified material dispersed in isopropyl alcohol (IPA) to obtain the particle sizing dilution. The size fractions are categorized by median d_{SED} . Size values have also been expressed in terms of the median d_{AED} as previously described. Each row represents an average particle size based on three measurements of that sample. Repetition for three different samples was performed to obtain the average values in the last two columns of Table 2. The coefficient of variation (CV) for repeatability for all median d_{SED} reported in Table 2 was less than 0.5 %, based on three replicate measurements.

The effect of particle sizing suspension concentration was also investigated. A series of suspension concentrations using the largest size fraction were generated. Beginning with an initial silica/isopropyl alcohol (IPA) concentration of 714 mg/L, four successive factor-of-two dilutions were prepared. Figure 1 shows the results which indicate a significant bias in the particle sizing due to sample concentration. It has been previously reported by this author that it was not known if this bias was due to an inherent suspension concentration dependency in the LA 920 sizing instrument or sample preparation for the dilution tests [19]. The previous work reasoned that, assuming no bias in sample preparation such as differential particle settling, the LA 920 would produce a positive size versus suspension concentration relationship on a purely statistical basis. This would occur due to too few of the larger particles being present in the dilute suspension for proper measurement, resulting in an underestimation of median particle size at the lower dilution concentrations. Therefore, the demonstrated bias would likely have been in sample preparation.

TABLE 2—*Summary particle sizing replication data for MIN-U-SIL 10 size fractions.*

Sample weight, mg	Sizing dilution, mg/L	d_{SED} [a] average, µm	Std.dev. N=3, µm	Pooled Average, µm	d_{AED} [b] average, µm
0.333	33	6.90	0.019	…	…
0.333	33	7.06	0.036	…	…
0.333	33	7.02	0.026	6.99	9.12
0.333	33	6.82	0.004	…	…
0.333	33	6.90	0.046	…	…
0.333	33	6.90	0.022	6.87	8.96
0.328	33	4.16	0.006	…	…
0.328	33	4.17	0.012	…	…
0.328	33	4.05	0.010	4.13	5.40
0.328	33	4.04	0.004	…	…
0.328	33	4.05	0.020	…	…
0.328	33	4.07	0.009	4.05	5.29
0.383	38	2.12	0.002	…	…
0.383	38	2.12	0.004	…	…
0.383	38	2.12	0.003	2.12	2.78
0.383	38	2.02	0.003	…	…
0.383	38	2.02	0.001	…	…
0.383	38	2.07	0.006	2.04	2.68
0.307	31	1.26	0.001	…	…
0.307	31	1.26	0.003	…	…
0.307	31	1.24	0.001	1.25	1.65
0.307	31	1.24	0.004	…	…
0.307	31	1.24	0.002	…	…
0.307	31	1.23	0.003	1.24	1.64

[a] Spherical equivalent diameter.

[b] Aerodynamic equivalent diameter estimate based on average of particle shape factors 1.82 and 1.36.

However, re-examination of the data suggests that the bias is due to suspension concentration effects. Figure 1 also shows the relationship between suspension concentration and the measured laser transmission of the LA 920. The close parallel between the aerodynamic diameter and laser transmission data strongly suggests that increasing the suspension concentration produces secondary scattering interpreted by the instrument as scattering from smaller particles which do not actually exist. This effect has previously been documented [21]. Therefore, only the data based on dilute silica/IPA samples will be presented as well as the result of these changes.

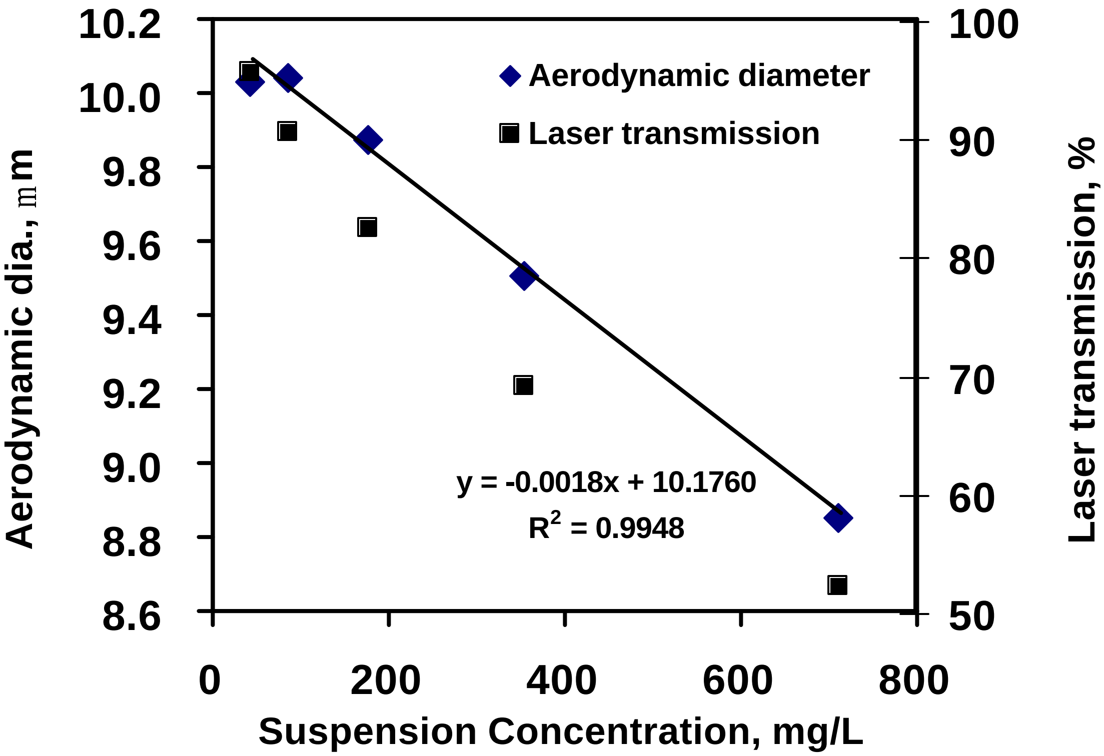

FIG. 1—*Suspension concentration effect on the reported median aerodynamic sizes.*

Results and Discussion--P7

Table 3 summarizes the MIN-U-SIL 10 size fraction analysis results. The average reported mass and standard deviations were determined from triplicate P7 measurements of each sample. Figure 2 shows the silica reported by P7 for three independent samples of each size fraction at the dilute silica suspensions used for particle sizing in the size range of 1.5–10 µm. The error bars for reported silica represent the standard deviation of nine measurements, comprised of triplicate measurements of three samples.

A cubic equation was chosen in this size range, based on previously published data by Foster and Walker [14], to fit the data with an adjusted R-squared value of 0.99. The present analysis closely mimics the results of Foster and Walker's infrared technique as a function of particle size at 799 cm^{-1} wavenumber. It was reported by Dempster and Ritchie [22] that the absorbance decrease below approximately 1.5 µm is due to an amorphous quartz layer on the silica particles which becomes an appreciable volume percentage as the surface area-to-volume ratio increases at smaller particle sizes. Figure 2 also includes a function that represents a particle crystallinity percentage, based on a 0.03 µm amorphous layer on a 100 % crystalline silica particle. The combination of the curves in Fig. 2 provide the reader with some estimate of P7 performance at very small particle sizes.

TABLE 3—*Summary data for P7 infrared and NIOSH 7500 XRD methods for silica on MIN-U-SIL 10 size fractions.*

SILICA P7 REPLICATION				X-RAY: PEAK AREA ANALYSIS ONLY				
Reported mass average, mg	Std. dev. N=3, mg	Silica average, percent	Silica std. dev., percent	Reported mass average, mg	Reported mass average, mg	Silica average, percent	Silica std. dev., percent	d_{AED}[a] average, µm
0.100	0.002	…	…	0.301	…	…	…	…
0.097	0.004		…	0.357	…	…	…	…
0.096	0.004	29.4	1.1	0.327	0.328	98.6	8.4	9.12
0.070	0.004	…	…	0.333	…	…	…	…
0.107	0.001	…	…	0.345	…	…	…	…
0.107	0.004	28.5	5.6	0.342	0.340	102.1	1.9	8.96
0.206	0.006	…	…	0.285	…	…	…	…
0.219	0.003	…	…	0.291	…	…	…	…
0.219	0.018	65.5	3.5	0.301	0.292	89.1	2.5	5.40
0.227	0.004	…	…	0.334	…	…	…	…
0.215	0.011	…	…	0.313	…	…	…	…
0.225	0.012	67.8	3.1	0.291	0.313	95.3	6.6	5.29
0.383	0.016	…	…	0.375	…	…	…	…
0.383	0.009	…	…	0.351	…	…	…	…
0.371	0.019	98.9	3.8	0.334	0.353	92.3	5.4	2.78
0.375	0.008	…	…	0.360	…	…	…	…
0.390	0.016	…	…	0.371	…	…	…	…
0.386	0.015	100.1	3.6	0.378	0.370	96.5	2.4	2.68
0.317	0.008	…	…	0.261	…	…	…	…
0.324	0.007	…	…	0.283	…	…	…	…
0.318	0.009	104.1	2.5	0.250	0.265	86.2	5.5	1.65
0.291	0.006	…	…	0.260	…	…	…	…
0.294	0.017	…	…	0.273	…	…	…	…
0.336	0.004	100.0	7.7	0.254	0.262	85.5	3.2	1.64

[a] Aerodynamic equivalent diameter estimate based on average of particle shape factors 1.82 and 1.36.

Table 3 shows that if the median d_{AED} of the silica particles in dust samples is between 1.6 and 2.8 µm, then P7 provides results which are reasonably accurate. Comparison of the median d_{AED} for the calibration materials shows that they are in close agreement with the P7 size range of accuracy. However, it is seen from Table 3 and Fig. 2 that there is significant underestimation of the reported silica percentage for particle sizes greater than 4 µm. Previous research has shown that MIN-U-SIL 5, MIN-U-SIL 15, and MIN-U-SIL 30 have median d_{AED} values of 1.16, 1.2, and 1.2 µm, respectively, after passing through a 10-mm nylon cyclone [9]. This result occurs because these materials all contain a significant amount of very small material below 5 µm and the cyclone is very effective at uniformly removing the larger, nonrespirable silica particles. This suggests that it is unlikely one would obtain any significant error in P7 analysis of an airborne respirable dust sample which passes a 10-mm cyclone.

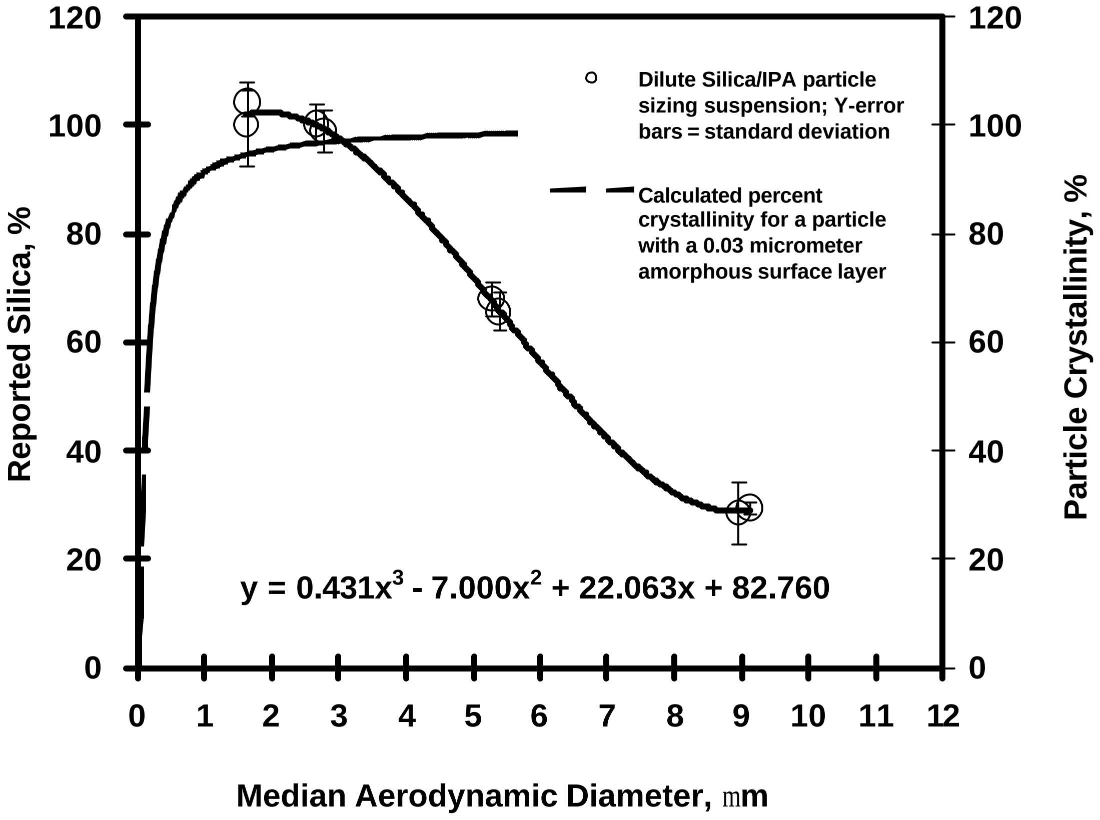

FIG. 2—*Reported silica as determined by the MSHA P7 Method.*

Results and Discussion BXRD Method

Table 3 and Fig. 3 summarize the XRD analysis on the same MIN-U-SIL 10 size fractions which were prepared and analyzed by P7. When compared to Fig. 2, it is immediately obvious in Fig. 3 that XRD yields much less error than P7 in sizes above 4 µm median d_{AED}. However, it is seen from Table 3 and Fig. 3 that there is some underestimation of the reported silica percentage for particle sizes smaller than approximately 2 µm. Although XRD is affected less by particle size than P7 over the size range less than 10 µm, it would appear that this method may underestimate the silica content of an airborne respirable dust sample which passes a 10-mm cyclone. Comparison of the median d_{AED} for the calibration materials shows that they appear to fall in the region where XRD shows some depressed response. The observed decreasing response seen in Fig. 3 is known to occur with XRD analysis [12,23,24]. The work of Edmonds et al. [23] showed the XRD intensity falling to nearly zero in the size range of 1–2 µm for MIN-U-SIL 15 material, possibly indicating a significant fraction of noncrystalline material in this size range. However, Fig. 3 suggests that the MIN-U-SIL 10 material fractionated in the present study does not have a significant fraction of noncrystalline silica in the 1–2 µm size range. XRD analysis would appear to be most appropriate for a dust sample which is representative of the total airborne dust, particularly since the mass fraction in the size range below 4 µm is a small percentage of the total airborne dust mass.

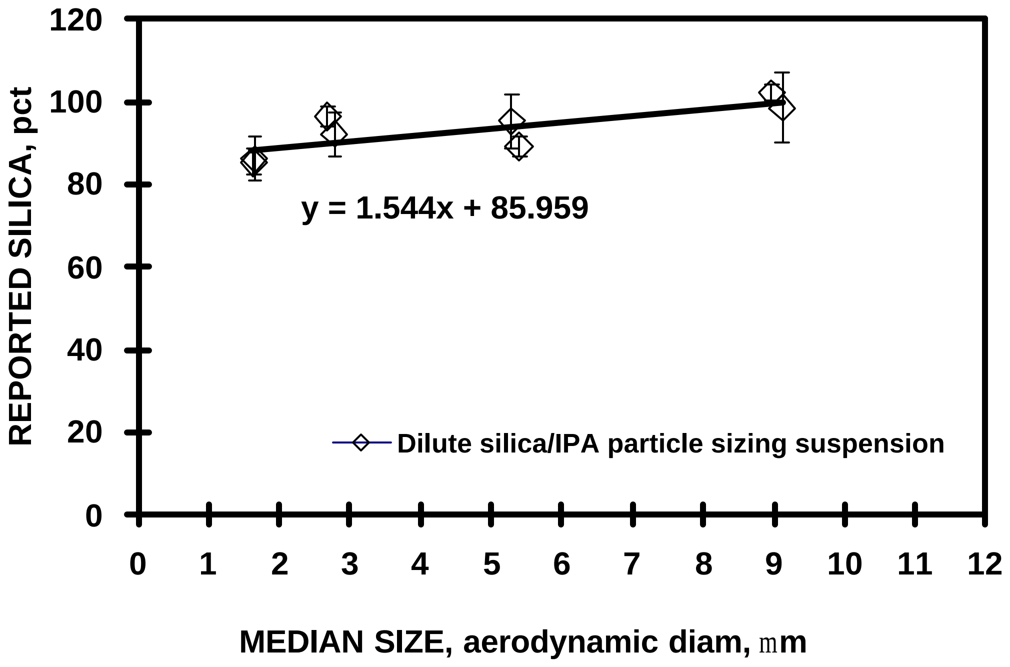

FIG. 3—*NIOSH XRD Analytical Method 7500 results on same samples analyzed by MSHA P7 Method.*

Results and Discussion—Impactor Sampling Data

To investigate a potential change in median size of underground coal mine dust, 83 impactor samples (Group 1) were analyzed for the median d_{AED} of the respirable fraction according to the former ACGIH definition and the individual median d_{AED} values ranged from 0.9 to 3.6 µm [19,25–27]. Using the polynomial regression equation from Fig. 2, P 7 will underestimate the silica content of the sample by at most 8.5 % in the MMAD size range of 0.9–3.6 µm. For the underground respirable size distributions, XRD could underestimate the silica content by 12.7 %. Although it is not known what the median d_{AED} of the silica component for these distributions is, the fact that these are aerodynamic estimates of the respirable fraction suggests that the median d_{AED} of the silica component should be in approximately the same size range.

Group 2 impactor samples (Table 4) were analyzed for total dust and silica content in three size ranges: R_1 = 21.3–9.8 µm, R_2 = 6.0–1.55 µm, and R_3 = 0.93–0.1 µm. Eighteen of twenty-nine samples have a larger percentage of silica in the combined ranges of R_2 and R_3 compared to the total dust in these ranges. However, allowing for an accepted 8 % relative standard deviation error propagation for the silica determination in the combined ranges of R_2 and R_3, 23 of 29 samples have a larger percentage of silica compared to the total dust in these ranges. It is apparent that the largest contributor to this result is the percentage of silica in the size range of 6.0–1.55 µm. Although the values of the median d_{AED} of the dust sample and silica content are not known, it is possible to do a student's t-test for paired two-sample means analysis on the values of (R_2 + R_3) in Table 4. The rationale for this choice is that the median d_{AED} of a respirable dust sample is likely to be in this range. A two-tailed analysis yields $t_{data} = 1.98 < t_{critical} = 2.05$ with 28° of freedom at the 5 % significance level. These results suggest that the median d_{AED} of the dust sample silica content is not statistically different from the median d_{AED} of the total dust sample.

TABLE 4—*Total dust and silica dust weight distribution for composited impactor stages.*

Mine	Sample	Percent of total dust weight				Percent of silica dust weight			
	location	in impactor µm size ranges				in impactor µm size ranges			
		R_1	R_2	R_3	(R_2+R_3)	R_1	R_2	R_3[a]	(R_2+R_3)
		21.3- 9.8	6.0-1.55	0.93- 0.1	6.0-0.1	21.3- 9.8	6.0-1.55	0.93- 0.1	6.0-0.1
N	intake	71.8	17.0	11.2	28	79.8	18.0	2.3	20
	return	89.1	7.1	3.7	11	83.6	14.7	1.7	16
	return	64.3	25.9	9.8	36	33.8	64.4	1.8	66
	lab crusher	76.0	22.3	1.7	24	66.3	32.7	1.1	34
	lab crusher	65.1	30.8	4.1	35	55.3	43.7	1.0	45
	lab crusher	43.0	45.6	11.4	57	31.8	59.9	8.3	68
O	intake	34.2	20.4	45.3	66	24.8	33.1	42.1	75
	return	50.3	38.5	11.2	50	50.7	41.9	7.5	49
	return	49.8	45.7	4.5	50	52.1	44.0	3.9	48
	return	60.2	34.6	5.2	40	55.6	37.7	6.7	44
	lab crusher	61.2	38.8	0.0	39	60.5	39.5	0.0	40
	lab crusher	63.6	36.4	0.0	36	61.1	38.9	0.0	39
P	lab crusher	67.4	32.6	0.0	33	66.8	33.2	0.0	33
	lab crusher	57.2	42.8	0.0	43	54.8	45.2	0.0	45
	lab crusher	57.6	42.4	0.0	42	63.8	36.2	0.0	36
	lab crusher	50.4	49.6	0.0	50	53.8	46.2	0.0	46
	lab crusher	58.1	41.9	0.0	42	69.3	30.7	0.0	31
	lab crusher	57.8	42.2	0.0	42	54.4	45.6	0.0	46
	lab crusher	60.1	39.9	0.0	40	55.0	45.0	0.0	45
Q	intake	39.3	46.4	14.3	61	28.1	56.1	15.8	72
	return	38.2	30.6	31.2	62	19.5	58.5	22.0	81
	return	38.3	38.3	23.3	62	39.2	51.2	9.6	61
	return	40.6	43.0	16.4	59	31.8	59.9	8.3	68
	lab crusher	57.2	38.3	4.5	43	56.1	42.1	1.8	44
	lab crusher	64.6	31.6	3.8	35	68.8	29.6	1.6	31
	lab crusher	61.3	35.6	3.1	39	61.2	36.7	2.1	39
R	lab crusher	73.9	23.9	2.2	26	75.3	20.4	4.3	25
	lab crusher	71.1	24.1	4.8	29	74.3	21.6	4.1	26
	lab crusher	69.8	26.9	3.3	30	76.4	19.7	3.8	24

Note: 23/29 samples have $(R_2+R_3)_{silica}/(R_2+R_3)_{total} \geq 1$ after adjustment for an assumed 8 % relative standard deviation error propagation in the silica determination R_2+R_3.

[a] Values of R_3 reported in Table 3 are 13 % higher than reported by laboratory analysis to correct for the underestimation at 0.5 µm midpoint.

A significant error could be introduced into P7 analysis of a respirable coal sample if the silica dust being sampled contains relatively few particles in the 1–2 µm size range. The result of this would be an increase in the median size of the cycloned material and it is reasonable to ask under what circumstances might this occur. Among the changes in coal mining technology is a significant increase in the power delivered by mining machinery to mine the coal. Since coal mining frequently involves cutting the much harder rock material, which is usually the source of silica, it is possible that there has been a decrease in the amount of grinding and an increase in the actual breakage of this rock material due to these more powerful machines. On this basis, it would appear reasonable to suspect some change in the median size of the airborne respirable silica compared to the early 1980s when the MIN-U-SIL 5 material was chosen as the best representation of airborne respirable silica dust in underground coal mines. However, the results of this work suggest that the median d_{AED} of the respirable silica component of coal mine dust still remains within the size range in which P7 is calibrated. It must be pointed out that the P7 results describe performance only for dust samples that have been classified according to a respirable definition similar to the penetration of a 10 mm cyclone or, at a minimum, samples which have a median d_{AED} in the size range mentioned. Therefore, application of P7 to impactor stage samples that possess 50 % cut points outside of the approximate size range of 0.9–3.6 µm is likely to produce significant underestimation errors due to the particle size sensitivity of P7.

Conclusions

The effect of particle size on the MSHA P7 Infrared Method and NIOSH XRD Analytical Method 7500 has been measured using size fractions of MIN-U-SIL 10 silica. Although the effect of particle size on these measurement techniques has been studied and presented previously, the objective of this work was to compare particle size distributions of underground coal mine dust with the particle size dependency of these two silica analysis techniques using Berkeley 5 and SRM 1878 calibration standard materials.

Impactor sampling data for various locations on continuous mining and longwall mining operations from 13 underground mines were analyzed for the respirable fraction and the resulting median d_{AED}. The individual median d_{AED} ranged from 0.9 to 3.6 µm. It appears that P7 will underestimate the silica content of the sample by at most 8.5 % in the median d_{AED} size range of 0.9–3.6 µm and that it is unlikely one would obtain any significant error in P7 analysis of an airborne respirable dust sample that passes a 10-mm cyclone when the method uses Berkeley 5, MIN-U-SIL 5, or SRM 1878 as a calibration standard material.

It appears that XRD is more appropriate for a dust sample that is representative of the total rather than the respirable airborne dust, particularly since the mass fraction in the size range below 4 µm is a small percentage of the total airborne dust mass. However, the results of this study indicate that XRD may not be as suitable as P7 for respirable dust samples. Comparison of underground respirable size distributions indicates that XRD could underestimate the silica content by as much as 12.7 % if the respirable aerodynamic equivalent diameter is 0.9 µm and 8.5 % if the respirable aerodynamic equivalent diameter is 3.6 µm. Since it is not common to find mine environments with respirable mass median aerodynamic diameters as low as 0.9 µm, the NIOSH Analytical Method 7500 is likely to underestimate the silica content of an airborne respirable dust sample by only 5–10 %.

The results of this study suggest that any changes that may have occurred in the median respirable size of airborne coal mine dust are not enough to cause any appreciable error in the current methods used for respirable crystalline silica analysis. Although the crystalline purity of the standard calibration materials used is not known, the differences reported in this work cannot be attributed to a significant noncrystalline component.

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