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Promoting early exposure monitoring for respirable crystalline silica: Taking the laboratory to the mine site

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Abstract

The exposure to respirable crystalline silica (RCS) in the mining industry is a recognized occupational hazard. The assessment and monitoring of the exposure to RCS is limited by two main factors: (1) variability of the silica percent in the mining dust and (2) lengthy off-site laboratory analysis of collected samples. The monitoring of respirable dust via traditional or real-time techniques is not adequate. A solution for on-site quantification of RCS in dust samples is being investigated by the Office of Mine Safety and Health Research, a division of the National Institute for Occupational Safety and Health. The use of portable Fourier transform infrared analyzers in conjunction with a direct-on-filter analysis approach is proposed. The progress made so far, the necessary steps in progress, and the application of the monitoring solution to a small data set is presented. When developed, the solution will allow operators to estimate RCS immediately after sampling, resulting in timelier monitoring of RCS for self-assessment of compliance at the end of the shift, more effective engineering monitoring, and better evaluation of control technologies.

Keywords

Exposure; field-based monitoring; mining; portable FTIR; respirable crystalline silica (RCS)

The occupational monitoring need

Much has been done in recent years in the area of monitoring exposure to aerosols in occupational environments, specifically in the mining industry, with the goal of obtaining more accurate and timely information. The monitoring solutions developed for respirable coal dust[1, 2] diesel particulate matter,[3] and respirable dust enhanced by video features[4] are aimed at gathering near real-time information of the aerosol concentration levels during the shift. Unfortunately, these solutions do not address the monitoring of respirable crystalline silica (RCS) in the mining environment. The exposure to RCS can lead over time

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to debilitating respiratory diseases affecting the health of workers, such as silicosis[5] and lung cancer.[6, 7]

The approach to occupational exposure monitoring for RCS has not changed substantially in the last few decades and entails collecting a dust sample with a respirable sampler and submitting the sample to an accredited laboratory for analysis using established analytical techniques, infrared, and x-ray diffraction (XRD), that have been tested and assessed extensively.[8] Aside from the cost of analysis, the main limitation of the current approach is the lack of timely information to the industrial hygienist or other responsible person on site. The time between sample collection and interpretation of the results of the analysis at the mine site limits the possibility for occupational health professionals to promptly address overexposures or to request the adjustment of a control technology. In addition, the results could have little or no value in the case of mobile mining operations because the operation has often moved to a new location before exposure results are available, and RCS concentrations might change due to variation in local geology. The use of the aforementioned innovative respirable dust monitoring solutions would not provide useful information on RCS, since they only record aerosol concentration, and the percent of crystalline silica in respirable mine dusts has been found to be variable[9] so a correction factor cannot be applied.

For the above reasons, a solution is needed for on-site quantification of RCS in dust samples, and potential solutions are being investigated by the Office of Mine Safety and Health Research (OMSHR), a division of the National Institute for Occupational Safety and Health (NIOSH). When developed, the solutions will allow operators to estimate RCS immediately after sampling, resulting in timelier monitoring of RCS for self-assessment of compliance at the end of the shift, more effective engineering monitoring, and better evaluation of control technologies.

The pathway to establish a field-based RCS monitoring solution

The quantification of RCS in respirable dust samples at the mine site can be possible only if several challenges are met. The first challenge is the analyzer that will quantify the RCS in the sample using an established analytical protocol. In recent years, several portable Fourier transform infrared spectroscopy (FTIR) instruments became commercially available. They are small, robust, battery-operated, relatively easy to use, and substantially less expensive than benchtop FTIR and XRD instruments. Because FTIR is one of the established analytical techniques for the quantification of RCS in dust samples,[8] these instruments are good candidates to be part of a field-based RCS monitoring solution.

The second challenge is how after collection a sample will be prepared for analysis at the mine site. Two main analytical approaches generally used for the quantification of RCS are indirect and direct methods. Indirect methods entail the preparation of the sample by ashing or dissolution of the filter media used for the collection, before analysis with either XRD or FTIR. In the case of coal mine dust samples, the ashing process also removes the combustible portion of the respirable sample. Direct methods, generally called direct-on-filter (DoF) methods, analyze the sample *as collected* on the air sample filters in the workplace for the analysis.[9, 10] Studies comparing the performance of the two approaches with the same or different analytical technique[11] found some differences, but both approaches are considered adequate for RCS analysis. A DoF method is the logical choice for a field-based RCS monitoring approach since pre-treatment of filters at the workplace is impractical.

To date, the DoF methods have been implemented with filter media no larger than 25 mm in diameter. However, this size of filter media is not used for the collection of respirable dust in U.S. mining environments, where filters with a diameter of 37 mm are more common. Therefore, the 37 mm filters are the initial focus for the new DoF monitoring approach. On the other hand, novel optimized sampling techniques, such as smaller filter sizes, should also be investigated to determine if the advantages outweigh the difficulties associated with introducing new occupational monitoring approaches. For example, high flow rate samplers ($>$4 l min−1) have been recently tested with success[12–14] and they could allow operators to sample for short periods within a working shift to assess potential high exposure tasks. The accuracy of a field-based method is of particular concern, and two likely sources of error are analytical confounders and uneven deposition of RCS on the filtering media. Analytical confounders include other minerals and compounds present in the dust that can affect the accuracy of the quantification of RCS. This effect is a known issue for both established laboratory methods (FTIR and XRD). For samples collected in coal mines, kaolin is a known infrared analytical confounder.[15, 16] In addition, because the DoF techniques do not entail the ashing process, carbonaceous material from different coal seams might affect the infrared spectrum in the region used for the RCS quantification; therefore, this potential matrix effect must be investigated. For samples collected in other mining environments, such as metal mines and sand and gravel operations, the presence of other minerals with potential to confound the FTIR analysis is very likely and an investigation on how they affect the RCS analysis is required. While established methods already address potential analytical confounders in a respirable dust sample collected in a generic occupational environment, a field-based technique must address the confounders present in the specific working site where it will be used.

Finally, because the DoF technique analyzes only a portion of the sample collected on the filter, the quantification of RCS collected on the entire filter can be successful only if deposition across the filter is known. Since deposition is affected greatly by the design of the respirable sampler and sampling cassette, a DoF field-based method will need to account for the sampling system used.

Considering that the field-based RCS monitoring approach will allow mine operators to perform activities generally deputed only to analytical laboratories, the new approach must include procedures for the support of the technique at the mine site—specific protocols, use of blank, and standard samples, and interpretation of data quality need to be developed. While it is not realistic to pursue the level of performance of analytical laboratories, industrial hygienists will have the additional responsibility to periodically assess the performance of their monitoring technique when used at the site.

Progress made so far

Development of the new field-based RCS monitoring approach started with the evaluation of a portable Bruker FTIR spectrometer (Bruker Alpha FTIR) for the estimation of RCS in coal dust samples collected in a controlled calm air chamber.[17] The analytical method selected was FTIR in transmission mode, using the DoF approach. The study helped identify the proper parameters for the DoF-FTIR technique for accurate quantification of RCS in respirable dust samples. The estimation of silica using the DoF-FTIR technique uses a quantification model that correlates the absorption data in a portion of the infrared spectrum characteristic for crystalline silica with the amount of RCS in a sample. The quantification model was created by analyzing high purity RCS (U.S. Silica) calibration samples collected in a calm air dust chamber. The calibration samples were analyzed with the DoF-FTIR technique and with different standard methods, specifically the MSHA infrared P7 method and the NIOSH7500 method based on X-Ray diffraction. The quantification model was found to be independent from the standard method.

The estimation of the RCS in coal dust samples[17] was conducted by measuring the absorption of the region of the infrared spectrum and then applying the quantification model. The presence of kaolin in the samples—a known infrared analytical confounder—was addressed by applying a previously developed correction procedure.[15] Using the developed DoF technique, the portable FTIR was found capable of quantifying RCS in filter samples of different coal dusts at levels as low as 20 μg per filter.

Since the first study, the quantification model has been verified periodically by analyzing high-purity RCS samples with both the DoF-FTIR technique and standard methods. Comparison of the measured RCS in those samples, obtained by the two different approaches, MSHA P7 and NIOSH 7500, showed that the results were not significantly different (alpha = 0.05). In addition, it was shown that even though different accredited laboratories use different crystalline standard silica dusts, the confirmatory analyses proved that the quantification model was not affected.

To move forward with the new monitoring approach, three critical aspects needed to be evaluated: (1) the effect of deposition of the dust on the filter; (2) validation of the DoF method with the portable FTIR instrument; and (3) effect of analytical confounders present in dusts collected in different mining environments. The different response of the infrared and XRD techniques to crystalline silica particles of different size distribution can be of some concern, but this difference was found low for particle sizes typically present in the respirable mine dust.[18, 19]

As described above, the deposition of RCS on sampling filters has a crucial effect on any DoF technique. The deposition pattern for the three different types of 37-mm cassettes generally used in conjunction with the Dorr-Oliver nylon cyclone for monitoring in mining environments was investigated. It was found that the three-piece cassette provides the optimal deposition pattern for a DoF technique entailing FTIR analysis, while the cassette generally used for sampling in coal mines has the most “erratic” and random deposition.[20] The optimal deposition pattern for the three-piece cassette is the result of two factors. The first factor is that the deposition of RCS on the filter is fairly consistent and independent from loading. This result has been further verified for dust samples with a wide range of RCS percent in the dust and for RCS loadings as low as 20 μg. This implies that the estimation of RCS using DoF-FTIR does not need the information of respirable dust mass loading in order to be accurate. The second factor is that when a three-piece cassette is used the RCS is more concentrated in the center of the filter with a radial symmetry. This result has a positive impact on the sensitivity of our approach considering that the DoF-FTIR technique analyzes the center of the filter. Additional work on the RCS deposition pattern has been done using metal mine dust samples, collected with low-volume and high-volume samplers, and showing that the deposition is always radially symmetrical and the deposition pattern is different but consistent for each sampler.[21]

Basic validation of the DoF-FTIR method was conducted with the portable analyzer (Bruker Alpha FTIR). While results are not comprehensive, they reflect the performance of the analytical method with a portable spectrometer. Table 1 provides a synopsis of the tests conducted, the methodology used, and the main finding. The limit of detection and limit of quantification were estimated by using the standard deviation of the measurement of 20 blank PVC filters. This method has been recognized as adequate for the estimation of these two basic method parameters.[22] Repeatability and other variability testing were conducted on respirable dust check samples with a range of RCS from 40–700 μg. The consistency of the measurement over time and with two different portable FTIR analyzers was also assessed. In general, the variability was less than 2% and found to be consistent over the whole mass range. In addition, the analytical method entails the subtraction of the spectrum of a blank PVC filter before the analysis of a dust sample. The effect of variability of the PVC filter was also assessed as 2% by using different blanks when analyzing the same check samples. This information will be the basis for the determination of the precision of the DoF-FTIR method applied to the portable FTIR.

Finally, the primary analytical issue for applying DoF-FTIR to the measurement of silica in a variety of mining environments is that the presence of analytical confounders might affect the estimation of RCS in samples when infrared is used.[23] For coal mines, kaolin is the primary mineral of concern, and the DoF-FTIR technique using respirable dust samples from different coal mines with different levels of kaolin has been recently evaluated and the findings will be summarized in a separate upcoming publication. Efforts are currently in progress for the identification and classification of minerals present in respirable dust samples from selected (non-coal) mining environments that have the potential to confound the FTIR measurement of silica. There is a definite need for a novel RCS exposure monitoring approach for these specific mining environments.[9] Additional work is underway to evaluate various methods to correct for the confounding effect of such minerals. In preparation for the possible presence of analytical confounders, a more comprehensive use of the infrared spectrum has been preliminarily investigated.[24] This investigation found that the partial least square regression approach may reduce the error associated with the estimation of RCS using the DoF-FTIR technique.

Application of the DoF-FTIR technique

The performance of the DoF-FTIR technique was evaluated using respirable dust area samples collected in an operating metal mine and using different evaluation metrics. This mine was chosen because of the absence of known analytical confounders in the samples.

Samples were collected on three different days using a protocol that included the use of Dorr-Oliver nylon cyclones, 37-mm PVC filters in 3-piece styrene/acrylonitrile co-polymer cassettes, and a flow rate of 1.7 lpm. After collection, each sample was analyzed to determine respirable mass by gravimetric analysis and to estimate RCS with the DoF-FTIR technique. The quantification model described above was employed with the assumption that no mineral confounders were present.

When the DoF-FTIR estimation was completed, the filters were sent to an accredited laboratory for the quantification of the RCS with the established NIOSH 7500 method, which was used as a standard. In order to conduct a valid comparison of the DoF-FTIR estimates and NIOSH 7500 results, the laboratory was requested to perform the analysis only on the filter without recovering dust deposited on the wall of the cassettes. The results of the NIOSH 7500 method were used to calculate the percent silica in the respirable dust (%RCS) for each set of samples. The three sets were different in terms of number of samples collected, range of respirable mass collected on the filter, and silica percent in the respirable dust. Table 2 summarizes the general information for each set.

The variation in %RCS as represented in Figure 1 reinforces the need for a field-based silica monitoring solution and highlights the inadequacy of real-time respirable dust monitors to assess the RCS level, since that approach must assume constant silica content in the dust.

The estimation of RCS in each sample using the proposed DoF technique was compared with the results of the standard analysis by using different approaches and metrics, as described below. The comparison was done on each sample, then on each set (resulting from each of the three trips) and finally by combining the sets.

The relationship between the estimation and standard analysis conducted on each sample was first investigated with regression analysis. Subsequently, for each sample the ratio [estimation/NIOSH 7500] = R was calculated and for each set (standalone trip or combined) the ratios were compared to a hypothesis (R = 1) with a Student t-test. In addition, a single-factor ANOVA was used to assess statistical difference among the ratios from each trip. For each set the standard error of the estimate was also calculated. This metric provides a single value characteristic of the error generated by using the DoF-FTIR technique instead of the standard laboratory analysis. Finally, the relative difference associated with the use of the proposed DoF-FTIR technique was calculated for each sample. For each set, the average relative difference and the confidence level (95%) were calculated. In addition, a single-factor ANOVA was used to assess statistical difference among the average relative differences from each trip.

The results obtained with the DoF-FTIR technique and standard analysis are linearly correlated (Table 3). The poorer R^2 for Trip3 in the table is not indicative because the range of RCS was the lowest [17–42 μg]. The analysis of the ratios indicated that the average ratios for each set are not significantly different from 1 (alpha = 0.05). In addition, the ANOVA test showed that the ratios for the three sets are not significantly different (alpha = 0.05)—therefore, the three sets could be combined. The error associated with our estimation was in the range of 15–20 μg; this value will be used in the future to compare the performance of the portable FTIR technique with dusts from different environments.

The relative difference is a more common metric to assess a new technique but it is also greatly affected by the number of samples and analyte levels in the set which cannot be controlled when collecting samples in the workplace. Because of this consideration, it is not surprising that the average for each set is different even if the analysis of the ANOVA did not assess any significant (alpha = 0.05) difference between the sets (Table 3). While the variation is large, especially for lightly loaded samples (Figure 2), the three average relative differences are lower than 10%. When the sets were combined the average relative difference was found to be 3.5% [−3.2–10.3%]. No trend can be detected for the relative differences among the three sets as a function of the amount of RCS in the samples, but a higher scatter in the data is present at RCS levels lower than 25 μg.

Conclusion

The occupational need for a more timely monitoring solution for respirable crystalline silica concentration in mining environments has been presented. NIOSH OMSHR is investigating potential field-based solutions and preliminary progress using a DoF-FTIR method is reported. Several metrics to assess the performance of a new portable analytical technique have been used in the estimation of samples collected in the mill of a metal mine during three different trips. The case study presented here focused on a mine dust with no detectable amounts of other confounding minerals in the dust sample. Current studies are in progress to identify and classify analytical confounders in the respirable dusts present in various non-coal mines in the U.S., with the goal of quantifying the confounding effect and developing appropriate correction strategies.

In addition, OMSHR is exploring the development of features for the new field-based approach that will enhance ease of use at the mine site. The main issue is handling the sample from collection to analysis. A specialized sampling cassette needs to be designed to minimize the potential losses of dust and to optimize the transfer of the sample to the spectrometer.

It is important to mention one intrinsic limitation of field-based DoF-FTIR analysis of RCS: the field-based DoF-FTIR approach cannot directly account for silica dust deposited on the walls of the sampling cassette. While it is known and recently reported that the use of non-conductive cassettes causes significant wall losses in Soo et al.,[25] these are also not accounted for by the analytical methods used by U.S. Mine Safety and Health Administration (MSHA) for the analysis of compliance respirable dust samples collected in coal and non-coal mines. A simple solution to substantially reduce wall losses is to use a conductive cassette and the use of conductive material for the specialized sampling cassette will be explored.

When completely developed and tested, the field-based RCS monitoring solution will increase the potential impact for conducting monitoring activities for assessment of exposure and introduction of engineering dust controls. Industrial hygienists and occupational health professionals in general will have new tools to improve and optimize the monitoring process and overall this novel approach will increase the empowerment and engagement of operators in monitoring RCS at the mine site and ultimately improve the conditions for workers.

While the proposed field-based RCS monitoring solution does not provide real-time information, it has the advantage that it can be used in concert with the traditional sample collection methods and it can be implemented with a single one-time purchase of a portable FTIR analyzer. This would allow for the on-site analysis of multiple mine-wide samples on a daily basis.

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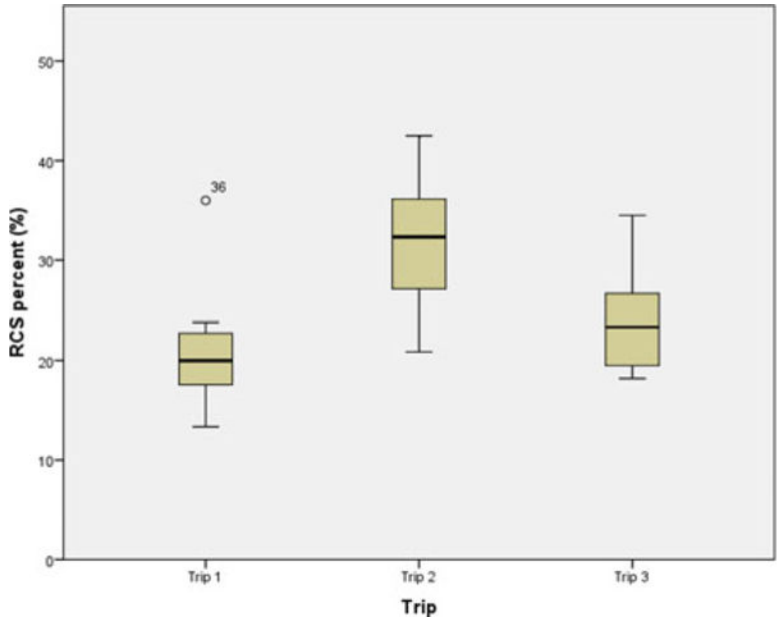

Figure 1.
Boxplot representation of the RCS content (%) in the dust collected in the three different trips to the metal mine.

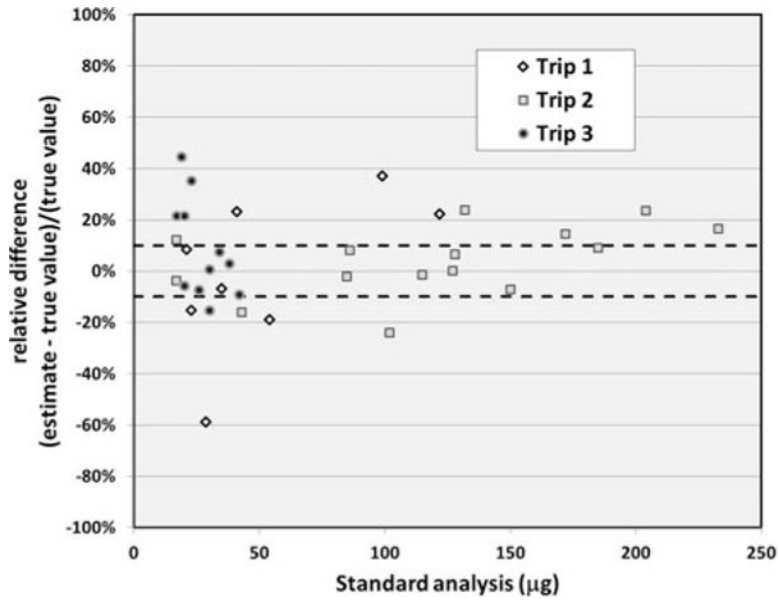

Figure 2.
Relative difference for the estimation of RCS with DoF-FTIR technique and the standard analysis.

Table 1

Synopsis of the basic characteristics of the DoF-FTIR technique for the estimation of RCS in mine dust samples.

	Measure	Value
Limit of detection	3 × Standard Deviation	5 μg
Limit of quantification	10 × Standard Deviation	16 μg
Orientation	Average standard deviation of four analyses with the sample rotated 90 degrees	1.66%
Daily variability	Analysis on the same samples on three different days	0.78%
Intra-instrument variability	Same set analyzed by two instruments	1.65%

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Table 2

General information on the three sets of samples collected in a metal mine to test the DoF-FTIR technique.

	Trip1 8		**Trip2 17**		**Trip3 11**		**Overall 36**	
Number of samples	***Min***	***Max***	***Min***	***Max***	***Min***	***Max***	***Min***	***Max***
Respirable mass range (mg)	97	610	40	736	75	175	40	736
RCS range (μg)	21	122	17	283	17	42	17	283

Table 3

Statistical analysis data of the performance of the DoF-FTIR technique in comparison to the standard NIOSH7500 analysis.

			Trip1	Trip2	Trip3	Overall
Linear Correlation	R2		0.96	0.97	0.72	0.98
	Slope	Lower 99%	0.97	0.98	0.23	1.03
		Upper 99%	1.83	1.28	1.19	1.19
	Intercept	Lower 99%	−43.04	−32.56	−4.03	−12.72
		Upper 99%	11.33	14.42	22.94	5.43
Analysis Ratio	Average		0.99	1.04	1.09	1.04
	STDEV		0.31	0.13	0.19	0.20
Standard Error of the estimate (μg)			18	20	4	16
Relative difference	Average		−1.1%	4.1%	8.8%	3.5%
	Lower 95%		−26.1%	−2.4%	−4.1%	−3.2%
	Upper 95%		23.9%	10.7%	21.8%	10.3%