



Respirable Coal Mine Dust in the Vicinity of a Roof Bolter: an Inter-laboratory Study to Compare Wet Versus Dry Dust Collection Systems

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Abstract

Among underground coal miners, roof bolter operators are generally considered to have some of the highest risks for hazardous respirable dust exposure. This is because bolting requires drilling into roof strata that can often be a source of silica and silicate dust, which are associated with occupational lung diseases. However, little is known about the variability of dust characteristics (e.g., mineralogy constituents, particle size) in the vicinity of the bolter—or when specific dust controls are applied. As part of a prior NIOSH study, respirable dust samples were collected during several different events in standardized locations around an active roof bolter, and personal samples were also collected from the operator during the cleanout of the bolter's dust collection system when it was equipped with a novel wet dust box versus a traditional dry box. Those samples were made available for follow-up analysis by scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM–EDX), as well as direct-on-filter Fourier transform infrared spectroscopy (FTIR). Results showed variability in dust constituents and particle sizes at locations around the roof bolter, and indicated some event-to-event differences in dust sources. Additionally, compared to the dry dust box, the wet dust box appeared to reduce (by 41–82%) the relative silica and silicate content in the respirable dust to which the operator was exposed during cleanout. Furthermore, an inter-laboratory comparison demonstrated the reproducibility of a standardized direct-on-filter FTIR method for estimating quartz mass (i.e., the predominant form of crystalline silica) in respirable coal mine dust samples. However, for constituent analysis by SEM–EDX, differences observed between results from two independent labs indicate that standardization of the analytical protocol is necessary to enable comparability of results.

Keywords Roof bolter · Respirable dust · Dust collection box · SEM–EDX · Silica

1 Introduction

1.1 Background

Starting in the late 1990s, the prevalence of occupational lung disease among US coal miners began to rise

precipitously, especially in central Appalachia, and rates remain high [1, 2, 3]. To fully explain this trend, significant attention has been drawn to the characteristics of respirable coal mine dust (RCMD) that may be responsible [4]. Properties such as the size and shape of particles, which impact lung deposition [5, 6, 7, 8], and their chemistry, which affects lung response and toxicity [9, 10, 11, 12], are important considerations for assessing RCMD exposure. Indeed, studies have suggested that certain dust constituents, especially fine silica and perhaps silicates, have played a key role in the resurgence of lung disease among US coal miners [13, 14]. Among the various occupations in underground coal mines, roof bolter operators are well known for having a relatively high exposure risk in regard to RCMD [15, 16]. Recent studies have shown that dust in the general vicinity of active roof bolting is often finer and contains higher concentrations of silica and silicate content than those in other

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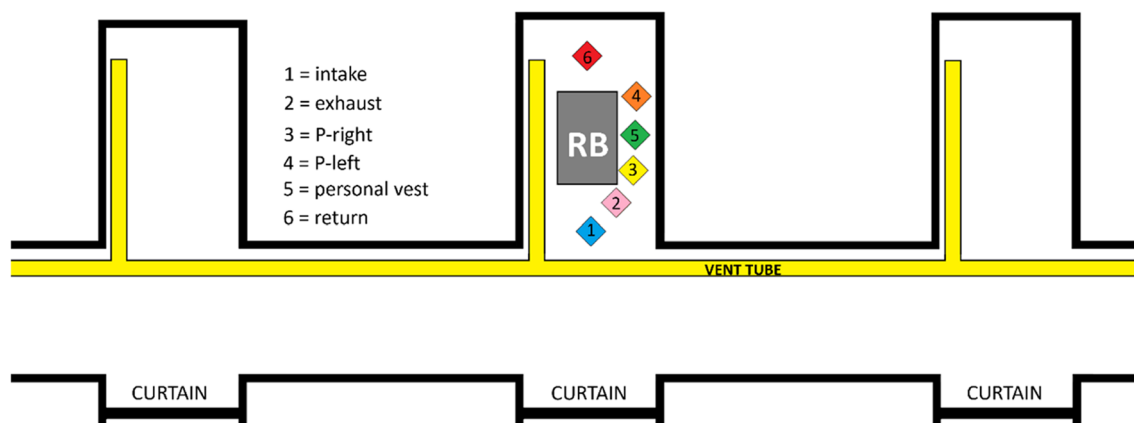


Fig. 1 Standardized sampling locations used by NIOSH to evaluate dust concentrations during the use of a wet dust collection box (versus a traditional dry box) on a roof bolter (adapted from [27])

locations of coal mines [17]. This is because dust in the roof bolter location is predominately sourced from the roof rock strata that sits above the target coal seam [18, 19].

Since roof bolting became standard practice in underground coal mines in the USA, dust generated from drilling (i.e., necessary to install the bolt) has been recognized as a health hazard and addressed through the application of water and the use of dust collectors [20]. To reduce roof bolter operators' exposure to respirable dust, a dust collection system is typically used on the roof bolter. The system essentially uses a vacuum to draw dusty air at the rock-drill interface through the hollow drill steel and then a pre-cleaner cyclone. Coarse particles in the airstream are deposited onto the mine floor, while fine airborne particles are directed into a "box," and air in the box is cleaned by a high-efficiency filter before exhausting back into the mine [21, 22]. Traditionally, this is a dry process—and while it is generally effective for capturing dust directly from the drilling process, roof bolter operators can still be exposed to an above-normal concentration of RCMD. Such exposures can happen during the periods when the dust collection system is operating less efficiently or when the roof bolter is working downwind of active mining (i.e., normally by the continuous miner).

Relatively high exposures can also happen inadvertently when the roof bolter operator is emptying the dust collection box [21, 23, 24]. Dust box cleanout typically involves opening the box and emptying the collected dust onto the mine floor, which can lead to a rapid re-aerosolization that results in a relatively brief but higher concentration of dust exposure. Results obtained from a previous study indicated that respirable dust levels during the box cleanout may be between 6 and 14 mg/m³ [25]. One solution to this problem has been to use dust collector bags within the dust collection box [21]. This is analogous to the use of a vacuum cleaner bag within a rigid receptacle: when the bag is full, it can be removed without spilling the contents. While the dry dust

bag approach is simple and generally effective, there are several drawbacks including (a) the periodic need to replace and handle it for disposal and (b) the possibility that the bag can rupture. Goodman and Organiscak observed dust from the outside of the bags being reintroduced to the air stream when the bags were flexed during handling [25]. As an alternative approach to the dry dust collection system, a wet system has been considered [26, 27, 28].

1.2 Summary of NIOSH Field Research on Wet Dust Collection System for a Roof Bolter

For a recent case study published by Reed et al. [27], researchers at the National Institute of Occupational Safety and Health (NIOSH) partnered with Blue Mountain Energy's Deserado Mine to test a wet dust collection system on a JH Fletcher model CHDDR dual-boom roof bolter. The wet system was designed as a modification to a standard dry dust collection system. It maintained the use of the vacuum pump to pull dust from drilling, but eliminated the pre-cleaner; instead, all material was wetted by a nozzle spray as it entered the dust collection box and was then deposited as a sludge inside the dust box. A drain with a rotary valve was installed in the bottom of the box. When drilling was completed, the rotary valve was opened to allow the sludge to empty, depositing the sludge onto the mine floor. The box exhaust air was still filtered through the final filter.

For the Reed et al. [27] study, the sampling plan included pairs of respirable dust samples collected during four separate events (i.e., shifts) in six standardized locations (Fig. 1). Two of the events corresponded to the use of the wet dust box on the roof bolter, and the other two corresponded to the use of the dry dust box. Five of the locations were sampled during active roof bolting: in the intake air upwind of the roof bolter, pre-cleaner for the left (P-left) and right (P-right) bolters on the machine, at the exhaust of the dust collection

system, and in the return air coming off the bolter. For the particular roof bolter studied, the pre-cleaners and dust boxes for both booms were located on the right side of the machine, and the pre-cleaner sampling locations were placed at the dust box doors for the respective boom in an attempt to sample any dust emanating from the pre-cleaners. During testing of the wet box, the pre-cleaners were removed from the bolting machine, but the P-left and P-right samplers were placed in the same locations as for the dry box testing. The sixth location was on the vest of the roof bolter operator, and these samples were collected only during the time when the operator was cleaning out the dust collection system.

With the intent to compare respirable dust mass concentrations (mg/m^3) and crystalline silica mass content (%) in each sampling location for each event, Reed et al. [27] collected filter samples via standard coal mine dust personal sampling units (CMDPSU); these consist of an air pump (operated at 2.0 L/min) and 10-mm nylon cyclone to collect the respirable particles onto a 37-mm polyvinyl chloride filter (PVC, 5 μm pore size) inside a three-piece cassette. Unfortunately, a problem with the measurement of sample filter pre-weights prevented the intended dust analysis. However, the samples were analyzed for quartz¹ mass by Fourier transform infrared spectroscopy (FTIR) per the non-destructive direct-on-filter method outlined by Chubb and Cauda [29]. This yielded an estimate of crystalline silica mass (μg) but since the total sample weight was uncertain, the mass percentage could not be determined. Though not reported by Reed et al. [27], the FTIR data was archived by at NIOSH and, importantly, the pairs of filter samples were preserved.

In each location, Reed et al. [27] also used a light scattering particle monitor (Thermo Fisher pDR-1000) to log time-series data [27]. Typically, this data is calibrated to mass concentration post hoc using the time-weighted average mass concentration determined from a collected filter sample [30]. Due to the problem mentioned with filter pre-weight measurements, the pDR data could not be calibrated. Nonetheless, Reed et al. [27] were able to use the data to study changes in the relative dust concentration between sampling events for a given standardized location (i.e., because the same pDR unit was always used in the same location). As expected, they found that the dust collection system type (i.e., wet versus dry) did not seem to influence the respirable dust concentration in the vicinity of the active roof bolter. However, the operator's vest samples showed that use of the wet system reduced the exposure by an average of 60% during dust box cleanout.

In a second NIOSH study of the roof bolter wet dust collection system, Reed et al. [28] reported similar results

(i.e., reduction in respirable dust exposure by 71–88% when cleaning out the wet versus dry collection system). They were also able to show that respirable quartz exposure, specifically, was reduced during dust box cleanout when using the wet versus dry collection system. Notably, in the second study, there was no issue with obtaining filter weight measurements; so, quartz analysis was performed by the standard NIOSH Method 7603, which is destructive. Thus, no dust samples were preserved.

1.3 Research Objectives

While the Reed et al. studies [27, 28] demonstrated the potential benefits of the novel wet dust collection system for reducing roof bolter operator exposure to respirable dust in terms of mass concentration, detailed dust characterization was not part of the original study designs. Increasingly, however, there is significant interest in understanding the whole composition of respirable coal mine dust—including the distribution of various constituents and particle sizes—and the effects of different dust sources and engineering controls on such characteristics [4]. Accordingly, there is interest in the development and application of standardized analytical methods for dust characterization.

The current work leverages availability of preserved dust samples from the first study by Reed et al. [27] to both investigate respirable dust characteristics and to explore analytical methods. Specifically, the first research objective was to evaluate the variability in dust constituents and particle sizes as a function of sampling location (per Fig. 1), event, and the type of dust collection system installed on the roof bolter (i.e., wet or dry). For this, dust analysis was performed using scanning electron microscopy with energy dispersive X-ray (SEM–EDX). SEM–EDX is a common technique for analysis of particles in the micron size range and has been applied in numerous studies of respirable coal mine dust [19, 31, 32, 33, 34, 35, 36, 37, 38, 39, 40, 41, 42, 43]. While methodologies have been developed and standardized internally within some research groups [17, 44, 45], wider standardization does not yet exist, and comparisons have not been done across different labs using their own methods to gauge the similarity of results. Since the preserved dust samples from the Reed et al. [27] study were collected in pairs (i.e., duplicates), their availability for follow-up work presented an added opportunity for intra-laboratory comparison. Thus, the second research objective of the current work was to compare results of respirable coal mine dust characterization by SEM–EDX between two academic laboratories using their own, internally developed, methods. Moreover, given that the available dust samples had already been analyzed by NIOSH using the standardized direct-on-filter FTIR method (per [29]), and one of the academic labs represented here is also equipped to perform this analysis, another opportunity

¹ In coal mines, quartz is typically the dominant form of respirable crystalline silica.

Table 1 Summary of respirable dust samples selected for analysis by each lab. F denotes samples that were analyzed by FTIR. For SEM–EDX analysis, D denotes samples that were analyzed directly on the sample filter; R denotes samples from which dust was recovered and redeposited prior to analysis. (N/A denotes that a sample was not analyzed)

Lab	Sampling event	Date*	Intake	Return	P-Left	P-Right	Exhaust	Vest
VT	1	Aug 23	N/A	F, R	N/A	F, R	F, R	F, R
	2	Aug 24	N/A	F, R	N/A	F, R	F, R	F, R
	3	Aug 29	N/A	F, R	N/A	F, R	F, R	F, R
	4	Aug 30	N/A	F, R	N/A	F, R	F, R	R
MTU	1	Aug 23	N/A	R	R	R	R	R
	2	Aug 24	N/A	D	D+R	N/A	D	D
	3	Aug 29	D	D	D+R	D	D	D
	4	Aug 30	D	N/A	D	D	D	N/A

*Sampling date per [27]

for intra-laboratory comparison was recognized. Thus, a third objective of this work was to compare the quartz mass results determined using direct-on-filter FTIR between the NIOSH and academic laboratories. This method is relatively new and has not been widely adopted in the field, but it is of significant interest for enabling end-of-shift silica monitoring [4].

2 Materials and Methods

A total of 48 respirable dust samples (representing 24 pairs of duplicates) collected by NIOSH for the Reed et al. [27] study were made available for the current work. The samples were split (one from each pair) between two laboratories represented by the authors: Virginia Tech (VT) and Michigan Technological University (MTU). Each lab performed its own SEM–EDX analysis on a subset of the received samples, and results were compared on the basis of numerous variables as shown in Table 1. The VT lab, while using a redeposition protocol for SEM-EX analysis, also performed direct-on-filter FTIR analysis of received samples to compare with earlier results from NIOSH on quartz mass. The MTU lab performed both direct-on-filter analysis on PVC filters using the SEM/EDX analysis and a redeposition protocol onto polycarbonate (PC) filters followed by an SEM/EDX analysis. No FTIR analyses were performed by MTU.

2.1 FTIR Method

As noted, the respirable dust samples had been collected on PVC filters, which are appropriate for direct-on-filter FTIR analysis [29]. To enable an inter-laboratory comparison (NIOSH to VT), the VT lab performed the same FTIR analysis on 23 of the 24 received sample filters (one filter was prepared for SEM–EDX analysis before it could be routed for FTIR). The FTIR analysis was completed using a Bruker Optics ALPHA II FTIR Spectrometer (Billerica, Massachusetts, USA). To analyze a filter, it was carefully

removed from its original cassette and placed inside a four-piece cassette that was designed for the direct-on-filter FTIR; the four-piece cassette fits in a special cradle that enables precise alignment in the FTIR instrument during scanning. The absorbance spectra (4000 to 400 cm^{-1}) were recorded from 16 scans of a 6-mm spot on the filter center using the Bruker's OPUS software (version 8.2.28), and then blank corrected (i.e., by subtracting the spectra generated on a blank PVC filter). Then, crystalline silica mass was determined using the publicly available FAST software developed by NIOSH [46], which includes a correction for the possible interference of kaolinite in the sample [38, 47, 48, 49, 50].

2.2 VT SEM–EDX Method

A total of 16 samples were selected for SEM–EDX analysis in the VT lab (Table 1). Dust was first recovered from the PVC filters and redeposited onto 47-mm track-etched polycarbonate filters (PC, 0.4 μm pore size). This is because the fibrous nature of PVC filters might hinder particle identification or classification under the microscope. Dust recovery and redeposition were performed using the following procedure: (1) each PVC filter was placed in a glass tube and submerged in isopropyl alcohol (IPA); (2) the tube was placed in an ultrasonic bath at 30 $^{\circ}\text{C}$ for three minutes to dislodge particles from the filter; (3) the PVC filter was removed from the tube; (4) the IPA and suspended dust were poured into a vacuum filtration unit to redeposit the particles onto a clean PC filter and allowed to dry completely; and (5) a 9-mm circular subsection was cut at the center of the filter and mounted onto an aluminum SEM stub and sputter coated with Au/Pd for analysis.

For SEM–EDX analysis, the VT lab utilized an FEI Quanta 600 FEG environmental scanning microscope (Hillsboro, OR, USA) with a Bruker Quantax 400 EDX spectroscope (Ewing, NJ, USA). The Bruker's Esprit software (Version 1.9.4) was used to run an automated routine on each prepared sample stub using the backscatter electron detector and settings shown in Table 2. Per Johann-Essex et al.,

Table 2 SEM–EDX parameters and constituent classes used by the VT and MTU labs

Operating parameters			Constituent classes	
Parameter	VT	MTU	VT	MTU
Accelerating voltage	15 kV (imaging, EDX)	15 kV (imaging), 12 or 9 kV (EDX)	Carbonaceous (C) Mixed carbonaceous (MC)	Coal
Spot size	5.5 μm	6.0 μm	Aluminosilicates-kaolinite (ASK)	Silicates
Magnification	1000×	2000×	Aluminosilicates-other (ASO)	
Particle length	0.9–10 μm	0.9–10 μm	Other silicates (SLO)	
Field area	14,025 μm ²	2127 μm ²	Silica	Silica
Max particles per field	50	60	Carbonates	Carbonates
Min fields per sample	10	8	Heavy minerals (HM)	Others
Target particles per sample	500	400	Others	

Table 3 Particle classification criteria based on normalized atomic % values derived from SEM–EDX analysis. The VT lab method included elements C, O, Ca, Mg, Al, Si, Ti, and Fe, and the MTU lab method included C, O, Ca, Mg, Al and Si

Constituent class	VT Lab							MTU Lab						
	C	O	Ca	Mg	Al	Si	Ti	Fe	C	O	Ca	Mg	Al	Si
Coal	≥75	<29	≤0.50	≤0.50	<0.35	<0.35	≤0.60	≤0.60	>70	-	<3	<3	<1	<1
Silicates	-	-	-	-	≥0.35	≥0.35	-	-	-	-	<3	<3	>1	>1
Silica	-	-	-	-	-	≥0.33	-	-	-	-	<3	<3	<1	>5
Carbonates	<88	>9	>0.50	>0.50							>3	>3	<1	<1
Others	Does not fit another class							Does not fit another class						

the routine scans multiple fields of view that are spaced to ensure data collection across the sample stub area [45]. To achieve a target of 500 total particles analyzed per sample, the routine limits analyzed particles per field to 50, such that at least 10 fields are scanned. In each field, particles are identified by contrast (i.e., against the smooth PC filter background), and then, data are collected for up to 50 particles (moving from left to right and top to bottom) with diameter between 0.9 and 10 μm. For each particle, the routine recorded length, width, and projected area (i.e., used to compute a projected area diameter, PAD). EDX spectra was also captured, and normalized atomic % values for eight elements (C, O, Al, Si, Ca, Mg, Fe, Ti) were used to classify it into one of nine mineralogy constituent classes (Table 2). Classification criteria were previously published by [17]; for this study, some classes were collapsed to enable direct comparisons with data from the MTU lab (see Table 2). The classification criteria for the collapsed bins are shown in Table 3.

2.3 MTU SEM–EDX Method

The MTU laboratory performed SEM–EDX analysis on the samples shown in Table 1 using two approaches: (1) particles were analyzed directly on the PVC filter and (2) particles were analyzed following dust recovery from the PVC filter and

redeposition onto a PC filter. For the direct analysis, two 8-mm square subsections were cut from the PVC filter (i.e., one from the center and one from the edge). Both were mounted on an aluminum stub and sputter coated with Pt, then analyzed with a Philips XL40 Environmental Scanning Electron Microscope (Houghton, MI) using the parameters given in Table 2. This analysis was conducted manually (rather than with a computer-controlled routine), and was limited to particles having a diameter greater than approximately 0.9 μm to enable comparison with the VT lab results. (It is noted however that individual particle sizes were not recorded during the analysis). Images for particle identification were obtained using the backscatter electron detector (at 15 kV), and EDX analysis to classify individual particles was accomplished using the secondary electron detector (at 9–12 kV). (The use of low accelerating voltage for the EDX analysis was to avoid interference of reflected signals from the filter substrate and nearby dust particles.) The MTU lab used normalized atomic % values for six elements (C, O, Al, Si, Ca, Mg) to classify each particle into one of the five constituent classes shown in Table 2. The classification criteria are shown in Table 3. For samples that were subjected to dust recovery and redeposition onto PC filter prior to SEM–EDX analysis, the MTU lab approach was similar (but not identical) to that of the VT lab: A quarter of the original PVC sample filter was added to a clean glass beaker filled with 5 mL of IPA, and the beaker was sonicated for about 5 s. Then,

the PVC filter was removed from the beaker and a Luer-lock syringe was used to take a 2-mL aliquot of the dust-loaded IPA suspension. The suspension was filtered using a syringe-filter assembly containing a clean 10-mm PC filter (0.2 µm pore size). The PC filter was dried overnight and prepared for SEM–EDX analysis by mounting it on an aluminum stub and coating it with Pt. The SEM–EDX analysis protocol for the redeposited dust samples was the same as described above for the direct analysis on the original PVC filters.

3 Results and Discussion

The approach to sample selection for SEM–EDX analysis was in consideration of the aforementioned research objectives. Related to the first objective, the aim was to evaluate variability in dust characteristics in the vicinity of the roof bolter, spatially (i.e., between standardized locations), temporally (i.e., between sampling events), and with respect to the dust collection system (i.e., wet versus dry dust box). For this, samples from 16 of the 24 total pairs were selected for SEM–EDX analysis using the VT lab’s method (Table 1). This method has been developed and improved over several iterations [40, 44, 45], and was recently used to characterize respirable dust from 25 coal mines [17, 51]. The 16 samples represent the same four locations sampled during each of the four events: the bolter dust collection system pre-cleaner for the right-side bolter (p-right), the collector exhaust, the return air from the bolter, and the personal (vest) sample from the operator during dust box cleanout. Since the wet dust collection system was tested during events 1 and 2, whereas the dry system was tested during events 3 and 4, it is also possible to compare results based on the specific collection system. (It is noted that samples from the bolter intake airway and the left-side pre-cleaner location (p-left) were not selected for the VT lab’s SEM–EDX.)

The MTU lab analyzed a total of 19 samples by SEM–EDX (Table 1). Related to the second research objective, 13 of these were from sample pairs also analyzed by the VT lab (comparison of results is discussed in Sect. 3.3 below), and the other six were from the bolter intake and p-left locations, which further enabled some understanding of the dust characteristics in these sampling locations. While the majority of the MTU lab’s analysis was conducted directly on a portion of the sample filter, it is noted that for two samples, the MTU lab also analyzed dust particles that were recovered and redeposited from another portion of the filter.

3.1 Dust Constituents and Size Distributions by Location, Event, and Dust Collection System

Table 4 and Fig. 2a show the constituent distribution of particles (number %) observed in the 16 samples analyzed by the VT lab, and Table 5 and Fig. 2b show these results for the

19 samples analyzed by the MTU lab. Across the three locations analyzed around the roof bolter (i.e., p-right, exhaust, return) during all sampling events, coal was the most dominant dust constituent—accounting for about 43–72% of all particles per VT results, and 29–94% per MTU results. Some coal dust might have been generated by the roof bolter activity, and some might have entered the roof bolter area with the intake air due to upwind activities (e.g., if the continuous miner was operating upwind the bolter). Carbonates—which could be sourced from dolomite-rich roof rock or application of limestone rock dust product in the mine—were also present to some extent (3–32% per VT, and 2–26% per MTU) in samples from all three locations around the roof bolter. The balance of the dust particles were silicates (4–18% per VT, and 2–55% per MTU) and silica (5–20% per VT, and 0–15% per MTU), which are expected to be sourced from the roof bolting activity, and possibly activities upwind of the roof bolter area. Samples analyzed from the intake location showed high coal (77% for event 3 and 70% for event 4 per MTU), with minor percentages of silicates, silica, and carbonates. Notably, no consistent trend was observed when comparing samples analyzed by the MTU lab at the p-left versus p-right locations.

While variability between the three locations around the bolter is clear for all four sampling events, there does not appear to be a consistent trend. For example, during events 1 and 3, the sample from the bolter’s dust collection system exhaust had somewhat less silicates (5% and 9% were observed by VT; 2% and 12% by MTU) and silica (5% and 6% observed by VT; 0% and 2% by MTU) than the samples from the p-right and return locations; however, this was not the case for events 2 and 4. This is most likely due to variability in local air circulation conditions and dust sources between the sampling locations from event to event. Notably, the MTU analysis that was conducted directly on the PVC filters also showed variability between locations—but again, it was not consistent from event to event. On the other hand, the carbonates and silicate + silica contents suggest an overall variability in dust sources based on the specific sampling event. In events 1 and 4, the carbonate content was relatively high (18–32% observed by VT; 3–26% by MTU²) in all three locations analyzed around the bolter, while the silicate + silica content was relatively low (10–24% observed by VT; 2–43% by MTU). However, the opposite was generally observed for events 2 and 3, wherein carbonates were relatively low (3–15% observed by VT; 2–9% for MTU) and silicates + silica were higher (15–38% observed by VT; 14–69% by MTU). These event-to-event variations could

² Event 1 carbonates as analyzed by MTU were lower than VT carbonates, ranging 3–6% MTU versus 18–20% VT. No significant explanation for the large difference can be provided.

Table 4 Summary of results for samples analyzed by the VT lab. For the SEM–EDX analysis, dust constituent distributions are shown on the basis of number %, and the overall D10, D50, and D90 particle sizes are shown. It is noted that all samples were recovered and re-deposited for SEM–EDX analysis (R). The table also shows results of the direct-on-filter FTIR analysis for both the VT and NIOSH labs.

It is noted that 23 of the 24 samples received by VT were analyzed by FTIR; however, SEM–EDX analysis was limited to just 16 of the samples. (N/A denotes that a sample was not analyzed. <LOD denotes FTIR-derived silica mass was below the limit of detection based on corrected integrated spectral peak area for quartz)

Sampling event	Location (SEM analysis)	SEM results								FTIR results	
		Dust constituents (number %)					Particle Size			Quartz mass (µg)	
		Coal	Silicates	Silica	Carbonates	Other	D10 (nm)	D50 (nm)	D90 (nm)	VT	NIOSH
Event 1 (wet box)	Intake (R)	N/A								<LOD	<LOD
	Return (R)	53	10	12	20	1	963	1330	2392	6.62	6.46
	p-right (R)	62	6	11	20	1	963	1232	2249	<LOD	<LOD
	p-left (R)	N/A								3.82	4.51
	Exhaust (R)	69	5	5	18	0	963	1197	2268	<LOD	<LOD
	Vest (R)	85	4	3	8	0	918	1161	3047	<LOD	<LOD
Event 2 (wet box)	Intake (R)	N/A								<LOD	<LOD
	Return (R)	57	13	17	9	2	963	1330	2424	4.56	5.30
	p-right (R)	60	7	15	15	1	963	1314	2528	<LOD	<LOD
	p-left (R)	N/A								<LOD	<LOD
	Exhaust (R)	58	18	20	3	0	918	1143	1813	26.89	28.68
	Vest (R)	60	9	15	14	1	963	1298	2464	<LOD	<LOD
Event 3 (dry box)	Intake (R)	N/A								<LOD	<LOD
	Return (R)	57	14	16	10	0	963	1377	2903	19.09	19.83
	p-right (R)	61	14	11	11	0	963	1642	4247	6.21	8.09
	p-left (R)	N/A								16.00	12.19
	Exhaust (R)	72	9	6	13	1	963	1480	3031	<LOD	<LOD
	Vest (R)	55	17	23	4	1	1006	1536	2677	15.78	<LOD
Event 4 (dry box)	Intake (R)	N/A								<LOD	<LOD
	Return (R)	63	4	10	22	1	918	1232	2053	<LOD	<LOD
	p-right (R)	43	10	14	32	1	1006	1466	3306	4.89	6.73
	p-left (R)	N/A								<LOD	<LOD
	Exhaust (R)	56	8	10	24	1	963	1346	2429	<LOD	<LOD
	Vest (R)	53	19	22	5	1	963	1392	2548	N/A	<LOD

be due to differences in roof rock geology from location to location. Other variations could be due to rock dust from rock dusting at the mine site and differences in the bolter activity (e.g., drilling depth, rotational pressure, and total drilling time).

With respect to the wet versus dry dust collection system, no discernable trends were observed in dust constituents between the three locations analyzed around the roof bolter. This is consistent with expectations since both systems should pull the respirable dust fraction into the containment box, exhaust from the box is through a high-efficiency filter, and, in the case of the wet system, the sludge discharged from the box during operation should result in little re-entrainment of dust particles from the mine floor. Indeed, neither the Reed et al. [27] study from which the samples used here originated nor the Reed et al. [28] follow-up study of the wet dust collection system, indicated any consistent

differences in dust concentrations around the operating roof bolter that were attributable to the dust collection system type.

However, the work by Reed et al. [27] did indicate that the wet system (compared to the dry) yielded lower dust concentrations for the roof bolter operator during dust box cleanout (i.e., 0.475 versus 1.188 mg/m³, respectively) [27]. Here, the SEM–EDX results for the operator’s vest samples can provide further insights: When the operator was cleaning out the dry dust box (events 3 and 4), the vest samples contained relatively more silicates + silica (40% for event 3 and 41% for event 4 were observed by VT; 44% for event 3 by MTU) than did the samples collected around the operating roof bolter just preceding box cleanout, i.e., the return, p-right, and exhaust samples for the same event (15–30% for event 3 and 14–24% for event 4 were observed by VT; 14–33% for event 3 by MTU). In other words, the silicate

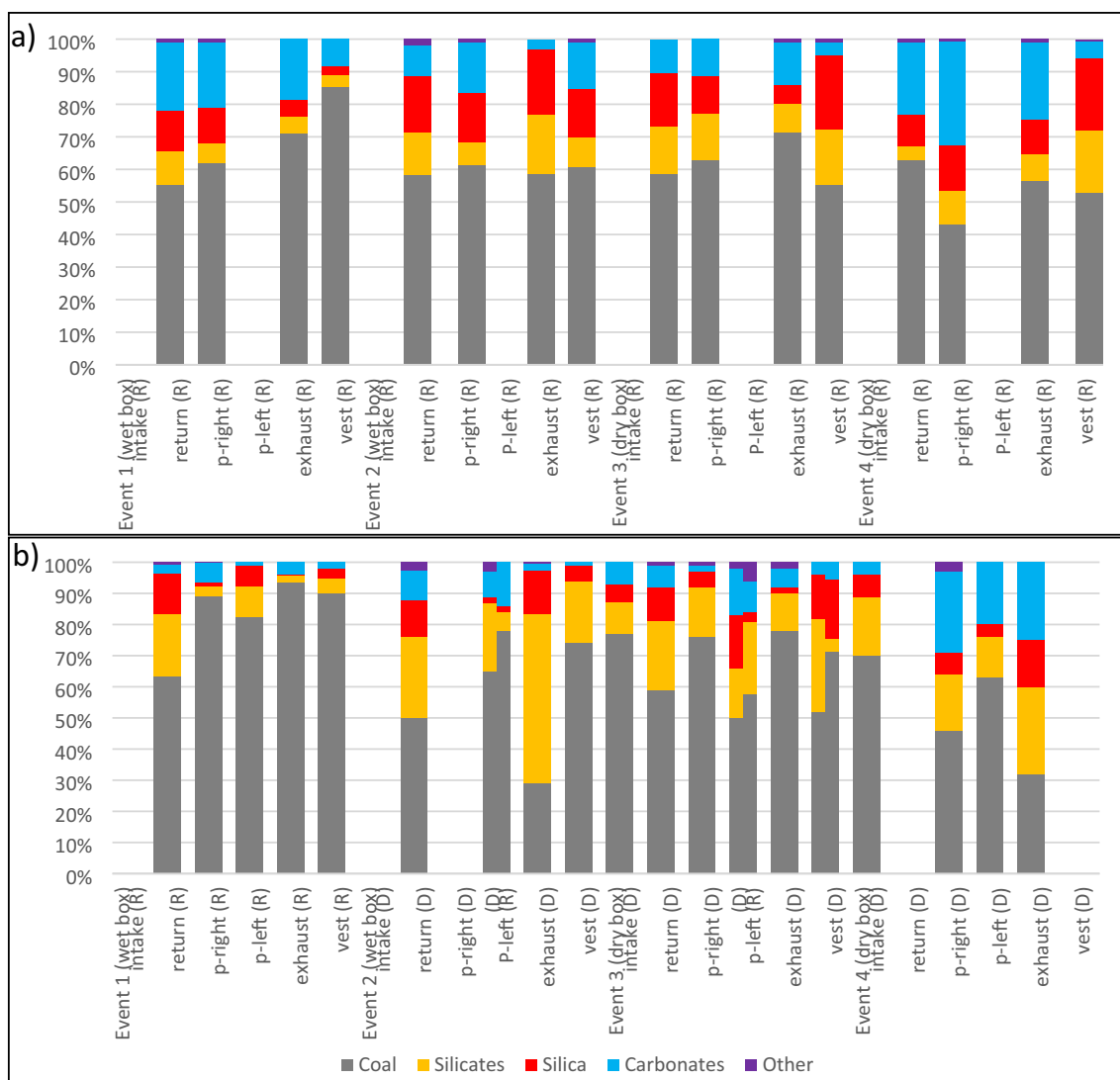


Fig. 2 Constituent distribution (on the basis of particle number %) of dust particles analyzed by SEM–EDX observed by **a**) the VT lab and **b**) the MTU labs. The analysis method is noted for each sample (R = recovered dust, D = direct-on-PVC sample filter)

and silica content appeared to be concentrated in the respirable dust generated during the dry box cleanout. However, when the operator was cleaning out the wet dust box (events 1 and 2), the vest samples generally contained relatively less silicates + silica than the samples collected around the operating roof bolter. In addition, when compared to the dry dust box, the wet box appeared to reduce the relative silicate + silica content in the operator’s vest sample during the box cleanout (i.e., from 40 and 41% for the dry box to 7 and 24% for the wet box as observed by VT; from 44% for the dry box to 8% and 25% for the wet box by MTU). Considering the results from both labs, this represents a reduction of about 41–82%. Taken together with the Reed et al. [27] results, this suggests that not only did the wet dust collection system reduce the respirable dust concentration, but it also reduced the relative hazard of the dust based on contained

constituents. As noted earlier, silica is considered the most hazardous of typical RCMD constituents, and there is evidence that silicates may also play a role in occupational lung disease in coal miners [13, 52, 53].

For context, in the second study of the wet dust collection system by NIOSH, [28] reported that silica (quartz) content was reduced in the operator’s samples during the cleanout of the wet box as compared to the dry box—consistent with the findings presented here. Moreover, according to Reed et al. [27], the wet dust collection box seems to accumulate less material than the dry box during bolting activities. This lesser accumulation is due to the frequent dumping of sludge material through the drain (by opening the rotary valve) in the time between bolting rows in the entry. This frequent dumping means that it requires a less frequent cleanout. In addition, once the entire entry is bolted, the wet box can

Table 5 Summary of results for samples analyzed by the MTU lab using SEM–EDX. Dust constituent distributions are shown on the basis of number %. It is noted that samples from Event 1 were recovered and redeposited for analysis (R), while samples from events 2 to 4 were analyzed by the direct-on-PVC filter method (D); for two samples (i.e., p-left in events 2 and 3), both recovered and direct analysis were conducted. It is noted that 19 of the 48 samples received by MTU were analyzed. (N/A denotes that the sample was not analyzed)

Sampling Event	Location (SEM analysis)	SEM results				
		Dust constituents (number %)				
		Coal	Silicates	Silica	Carbonates	Other
Event 1 (wet box)	Intake (R)	N/A				
	Return (R)	63	20	13	3	1
	p-right (R)	89	3	1	6	0
	p-left (R)	83	10	7	1	0
	Exhaust (R)	94	2	0	4	0
	Vest (R)	90	5	3	2	0
Event 2 (wet box)	Intake (D)	N/A				
	Return (D)	50	26	12	9	3
	p-right (D)	N/A				
	p-left (D)	65	22	2	8	3
	p-left (R)	78	6	2	14	0
	Exhaust (D)	29	55	14	2	0
Event 3 (dry box)	Intake (D)	77	10	6	7	0
	Return (D)	59	22	11	7	1
	p-right (D)	76	16	5	2	1
	p-left (D)	50	16	17	15	2
	p-left (R)	57	23	3	10	6
	Exhaust (D)	78	12	2	6	2
Event 4 (dry box)	Intake (D)	52	30	14	4	0
	Return (D)	70	19	7	4	0
	p-right (D)	N/A				
	p-left (D)	46	18	7	26	3
	p-left (D)	63	13	4	20	0
	Exhaust (D)	32	28	15	25	0

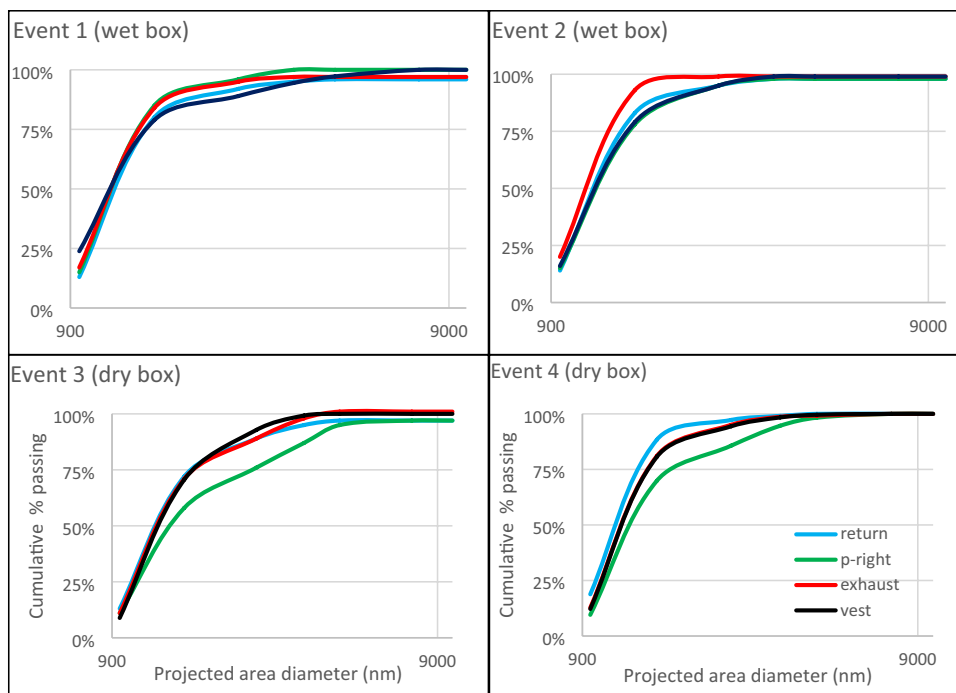
be emptied through the rotary valve in the drain to avoid frequent cleanout with a hose (i.e., as was tested in the Reed et al. [27] and [28] studies). These factors should further decrease the potential for hazardous exposure to respirable silica dust.

The SEM–EDX work performed by the VT lab also enabled analysis of particle size. (As stated above, the MTU lab did not record particle size data during SEM–EDX work, which was conducted manually.) For all 16 samples analyzed by the VT lab, Fig. 3 shows the overall particle size distributions, and Table 4 provides the D10, D50, and D90 particle sizes, which represent the size at which 10, 50, and 90% of the particles are finer. Similar to variability in dust constituents, there appears to be some event-to-event variability in terms of size. For instance, samples from event 3 generally had somewhat coarser particles than those from other events, which can be visualized in Fig. 3 and observed by comparing the D50 and D90 sizes in Table 4. Moreover, samples from the bolter’s dust collection system exhaust had relatively fine particles, which fits with expectations given

that the exhaust was filtered. And samples from the p-right location had relatively coarse particles when the pre-cleaner was in use (i.e., during the dry dust collection system testing in events 3 and 4). As mentioned earlier, the role of the pre-cleaner is to remove coarse dust from the airstream headed into the dust collection box [23], so relatively coarser dust (even in the respirable fraction) makes sense in this location—and has in fact been observed in prior studies [22, 54].

Regarding the operator’s vest samples, Fig. 3 shows that dust during the wet box cleanout was slightly coarser than dust in the three locations around the roof bolter during operation, whereas for the dry box cleanout, this was not necessarily the case. Given that finer particles are generally considered to pose greater hazards, this could suggest yet another advantage of the wet dust collection system. That said, it should be reiterated that the SEM–EDX analysis conducted for this study did not examine submicron particles, and the overall size distribution for the vest samples across all four events was fairly similar. Other factors—aside from the use of the wet versus the dry dust collection

Fig. 3 Overall particle size distributions determined from the VT lab SEM–EDX analysis. Each plot shows results for the return, p-right, exhaust, and vest samples from a given sampling event



system—such as the variability in the depth of drilling and drill bit sharpness could lead to the generation of different dust concentrations and particle sizes being deposited in the dust collection box during each sampling event [55]. Thus, additional testing to reduce event-to-event variability would be valuable to further evaluate the influence of the wet (versus dry) dust collection system on particle size during box cleanout.

3.2 Interlaboratory Comparisons

As mentioned, the second and third objectives of the current work were focused on comparison of dust analysis results obtained by different labs.

3.2.1 Results Based on SEM–EDX Analysis

A comparison of the results obtained by the MTU lab versus the VT lab are shown in Fig. 4. In total, there were 13 pairs of results that could be compared: nine pairs from events 2–4 for which MTU performed direct analysis of particles on the original PVC sample filter and VT performed analysis on recovered dust particles, and four pairs for which both labs performed analysis on recovered dust particles.

While the relationship between the two lab results shown by the trendlines in Fig. 4 are not particularly strong, they do demonstrate fair agreement between the MTU and VT labs in terms of relative abundance of primary dust constituents. For example, both labs found that the majority of dust particles in most samples were coal with the remainder of particles

divided between the other primary classes (i.e., silicates, silica and carbonates). The trendline slopes shown in Fig. 4 indicate that, in general, the MTU lab tended to classify more particles

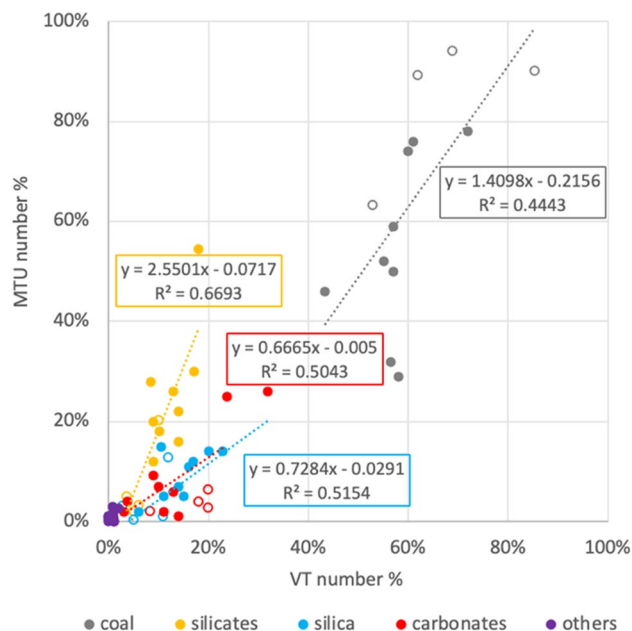


Fig. 4 Plots showing comparisons of the different dust constituents from samples analyzed by both VT and MTU labs. (It is noted that the plot only contains results from exhaust, p-right, return and vest since VT did not analyze the intake and p-left samples.) From each dust constituent plot, the filled circles represent direct MTU-recovered VT data points, while the open circles represent the recovered MTU-recovered VT data points

as coal and silicates than did the VT lab (i.e., the slope is 1.4098 and 2.5501 for coal and silicates, respectively). The opposite trend is seen for silica and carbonates (i.e., slope of 0.7284 and 0.6665 for silica and carbonates, respectively). Additional statistical analysis (*t*-test, correlation, etc.) were not conducted for the SEM–EDX results. The subsequent discussion concerning the variability of results are reasons for not conducting these additional statistical analyses.

It is important to note that even if two labs gather elemental data on the same particle, the use of different SEM–EDX instrumentation, settings, and routines could lead to some variability in the results. However, here, the main differences between the MTU and VT results are probably related to two other factors. First, analysis was conducted on pairs of samples—with each lab analyzing one sample from a given pair—rather than on the same samples. While paired samples of respirable coal mine dust are expected to be similar in terms of dust constituents [41], it is of course possible (even probable) that two samples collected side by side are not identical. Second, the two labs utilized different sample preparation and particle classification criteria.

To elaborate on particle classification, each lab took a similar approach to applying classification rules, whereby a particle's elemental content was compared to set thresholds for each constituent class (e.g., see Table 3 for thresholds from both labs). For instance, for both labs' criteria, coal particles are characterized by “minimal” content of analyzed elements other than C and O. However, the definition of “minimal” varied between the two labs such that the MTU coal classification allowed more Al, Si, Ca, and/or Mg content in the coal class than did the VT classification. This helps explain the observed tendency of the MTU lab to find more coal particles in most samples. On the other hand, for the carbonates class, the MTU criteria was more restrictive than the VT criteria, which helps explain the tendency of the MTU lab to find less carbonates. For the silicate and silica classes, both labs essentially split Si-bearing particles based on their Al content, but the MTU criteria for silica was again more restrictive than the VT criteria. Thus, the MTU lab tended to find less silica but more silicates than the VT lab.

Aside from differences in particle classification, and any inherent differences between the samples in each pair, differences in sample preparation between the two labs are also worth mentioning. While the VT lab only performed SEM–EDX analysis on recovered dust particles, most of the MTU analysis was directly on the surface of the original PVC filters. For direct-on-filter SEM–EDX analysis, sample collection is generally preferred on a smooth filter substrate, but this was not part of the original design of the Reed et al. [27] study during which these samples were collected—thus, both approaches utilized here have their drawbacks. While recovery and redeposition of dust can be subject to some losses, contamination, and/or deagglomeration of particles,

analysis directly on a PVC filter can be subject to bias with respect to particles that are on the filter surface versus those that embedded in the fibrous structure of the filter. Therefore, the direct analysis probably included somewhat coarser particles since finer particles could have been effectively hidden. Conversely, the dust recovery procedure (i.e., including sonication in IPA) may yield a somewhat finer size distribution due to deagglomeration of particles [56]. Both of these factors might influence the apparent distribution of dust constituents. The MTU lab conducted direct and recovered dust analysis on two samples (i.e., from the p-left location in events 2 and 3). The direct analysis indicated somewhat lower abundance of coal particles, but higher silicates + silica, compared to the recovered dust analysis (see Table 5). This might mean that the coal dust particles were relatively fine, and therefore more easily embedded in the PVC. Moreover, it is possible that directly analyzed coal particles were influenced to some extent by associated mineral particles. For example, a coal particle that is agglomerated with silicates might have been classified as a silicate particle during the direct analysis, but deagglomeration could enable the coal particle to be classified as such during the recovered dust analysis.

3.2.2 Results Based on FTIR Analysis

NIOSH had already analyzed the 48 filter samples available for this study using direct-on-filter FTIR to estimate silica mass (i.e., as quartz). That analysis was repeated on the samples received by the VT lab (i.e., 23 of 24 total samples) following the same analytical method (i.e., as described by Chubb & Cauda [29]). Figure 5 compares the NIOSH and VT results (tabulated in Table 4) for all samples ($n=8$) with quartz above the limit of detection (LOD). Especially considering the shipment, handling, and storage time of samples, the relationship between lab results appears very good, from the review of the trendline in Fig. 5 (i.e., slope of 0.9792 with minimal intercept). This demonstrates very good reproducibility of quartz measurement in respirable coal mine dust by the direct-on-filter FTIR method (and FAST software). It is noted that a direct comparison of the FTIR (i.e., μg of quartz) to SEM–EDX data (i.e., number % of silica) is not possible since the total sample weights could not be determined. Additionally, conducting a two-sample *t*-test (assuming equal variances) at a 95% confidence indicated no significant difference in the mean results for the NIOSH versus VT analysis ($p=0.90$).

Per Table 4, of the 8 samples with NIOSH and VT FTIR results $>$ LOD, all were from the p-right, p-left, or return locations except for one from the exhaust location. Notably, this trend was also observed for the additional NIOSH results for the 24 samples not provided to the VT lab (data not shown). This trend makes sense given the data reported

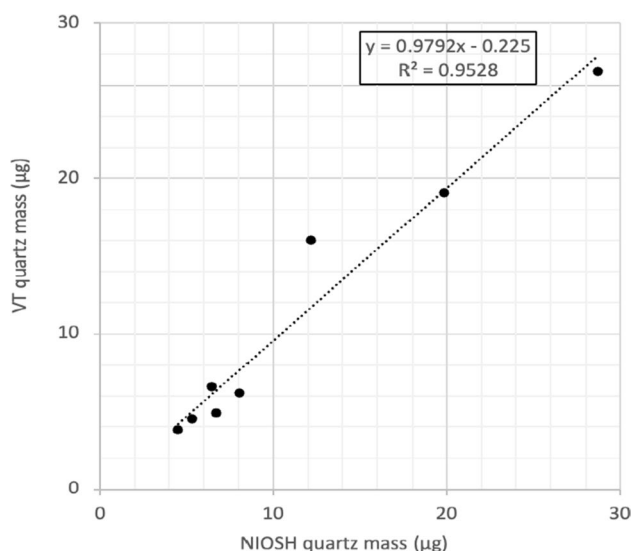


Fig. 5 VT and NIOSH estimates of quartz mass by the direct-on-filter FTIR method

by Reed et al. [27] too. While respirable dust concentrations could not be determined definitively in that study (i.e., due to the aforementioned problem with measuring filter sample masses), the estimates reported by Reed et al. do indicate dust concentrations were typically higher in the p-right, p-left, and return locations than in the exhaust location—which means the p-right, p-left, and return filter samples likely had greater masses, such that a > LOD FTIR result was also more likely.

Moreover, for the single sample (of 23) where the NIOSH and VT results did not agree (i.e., the vest sample from event 3), the Reed et al. [27] data indicates a relatively high concentration during the corresponding dust box cleanout event (i.e., a time-weighted average of about 1.240 mg/m^3 can be computed) relative to that in events 1, 2 and 4 (i.e., computed averages of about 0.564 , 0.391 and 0.462 mg/m^3 , respectively). Though the sample mass is expected to be fairly low (i.e., since the cleanout events and corresponding sampling times were fairly short), it was sufficient for the VT lab to obtain an FTIR result > LOD. (Incidentally, this sample also showed significant silica content by SEM–EDX.) It is unclear why the NIOSH FTIR result for this sample was < LOD, but might be due to factors such as a slightly off-center scan of the sample filter. Since the dust deposition pattern on filters collected in three-piece cassettes is center-heavy [57], an off-center scan will yield a low result—and, in the case of this sample, it could have caused the < LOD result.

3.3 Research Implications

The current work has several implications for future research. First, it shows that preserved samples can be

used to analyze respirable dust characteristics—and, depending on the level of information available about the original sampling design, results might be interpreted to assess variability with specific parameters. As demonstrated here, if the samples originate from a well-documented study, it could be possible to assess spatial (location to location) or temporal (event to event) effects, or the effects of certain dust controls (wet versus dry roof bolter dust collection system). However, there may also be opportunities to analyze samples with other origins (e.g., compliance dust sampling) to shed light on a number of other questions (e.g., how do dust characteristics vary between specific occupations? Between different mines? Between different regulatory eras?).

In any case, it is important to note that the most valuable datasets for evaluating respirable dust exposure hazards are likely to be those which include both concentration and characterization results. In essence, this is a problem of both quantity and quality. For the sample set investigated here, which was originally collected by Reed et al. [27], some understanding of respirable dust concentration was gained by the original study and this follow-up investigation added constituent and size distributions. The combined results indicate that use of the wet dust collection system on the roof bolter not only reduced the respirable dust concentration (mg/m^3) to which the operator was exposed during box cleanout, but it also reduced the proportion of silica + silicates in the respirable dust—and may have reduced the proportion of very fine particles in the dust too. In this case, it is appropriate to conclude that based on the available data, the wet dust collection system is expected to reduce the overall hazard of respirable dust exposure for the operator. However, conclusions may be more nuanced in cases where the control (or other study parameter) appears to yield competing effects on dust concentration versus characteristics. Thus, it seems prudent for future studies of respirable dust—whether in coal mines or other occupational or environmental settings—to include both concentration and characterization.

The current work additionally offers some key insights related to dust characterization methods, particularly with regard to particle-level analysis by SEM–EDX. While analysis of paired samples from two independent labs yielded generally similar results with respect to primary dust constituents, it is obvious that standardized methods are needed to enable robust comparisons between results obtained in different labs. Specifically, standardization should be pursued for sample handling, particle recovery, and redeposition procedures; particle analysis and data collection routines; analytical instrumentation capabilities and settings; particle classification criteria; and nomenclature.

Finally, it is worth mentioning that the direct-on-filter FTIR analysis for quartz mass has been proposed for end-of-shift silica monitoring [4, 29, 46, 47, 49]. The results presented here lend some evidence in support of this concept; specifically, quartz measurements were in agreement across two labs—even considering significant sample storage times, transport, and handling. That said, more effort is needed to investigate the accuracy of the method in the field and to train would-be analysts.

4 Conclusions

To investigate respirable dust characteristics in the vicinity of an active roof bolter, preserved samples from a prior NIOSH study (i.e., Reed et al. [27]) were analyzed by SEM–EDX. Results demonstrated that dust constituents and particle sizes varied around the operating roof bolter, likely due to local air circulation patterns and the influence of particular components of the machine’s dust collection system (e.g., dust around the system pre-cleaner was relatively coarse, whereas it was relatively fine at the system exhaust). Event-to-event differences also suggested that specific sources of dust in the vicinity of the roof bolter were variable, perhaps due to changes in the roof geology or relative contribution of coal or rock dust product in the intake air entering the bolter area. Moreover, dust characteristics were examined in a subset of the preserved NIOSH samples that represented the roof bolter operator’s potential exposure during cleanout of the dust collection system. When the system was equipped with a novel wet dust collection box—as opposed to a traditional dry box—results indicated the operator’s vest samples consisted of dust with less silicate + silica content (i.e., the constituents expected to be primarily sourced from the bolter drilling into to roof rock strata). The dry dust box vest samples silicate + silica content ranged from 40 to 44%, while the wet dust box vest sample silicate + silica content ranged from 7 to 25%—a reduction of 41–82%. This is further evidence (i.e., beyond that reported by Reed et al. [27, 28]) that the wet dust box can reduce hazardous respirable silica dust for the roof bolter operator.

The current study additionally allowed for inter-laboratory comparisons. When two labs independently applied a standardized method to determine quartz mass in the available respirable dust samples using direct-on-filter FTIR analysis, results were in very good agreement. This demonstrates method reproducibility. On the other hand, when two labs independently applied their own sample preparation and SEM–EDX analysis methods to evaluate dust constituents, more variability was observed. Importantly, the results presented here should not be used to imply a determination of performance by either of the participating labs. Rather they highlight the need for standardization of such particle-level analysis to enable comparison across labs.

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Data Availability Data related to this work is available from the corresponding author.

Declarations

Disclaimer The views, opinions, and recommendations expressed herein are solely those of the authors and do not imply any endorsement by the funding source or research partners. Mention of any company name, product, or software does not constitute endorsement by NIOSH.

Conflict of Interest The authors declare no competing interests.

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