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Real-time measurements of respirable crystalline silica, kaolinite, coal, and calcite

William Arnott
Atmospheric Science Program, Department of Physics, University of Nevada, Reno

Charles Kocsis
Mining Engineering Department, University of Utah, Salt Lake City

Xiaoliang Wang
Desert Research Institute, Reno, Nevada

Bankole Osho & Pedro Nascimento
Department of Mining and Metallurgical Engineering, University of Nevada, Reno

Samuel Taylor, Bjoern Bingham, Chauntelle Murphy & Michael Sandink
Atmospheric Science Program, Department of Physics, University of Nevada, Reno

ABSTRACT: Real-time measurements of coal mine speciated dust mass concentration (SDMC) are needed for health and ventilation applications. The current method requires capturing aerosol on filters by vacuum pump and Fourier Transform Infrared Spectroscopic (FTIR) analysis, providing end-of-work-shift measurements. We are developing a photoacoustic spectrometer (PAS) equipped with a tunable quantum cascade laser (QCL) to measure SDMC in real-time. The QCL is sequentially tuned to four unique wavelengths for each compound to quantify the SDMC of respirable crystalline silica (RCS), kaolinite, coal, and calcite. Each dust type also absorbs a little laser light at the other three wavelengths. A matrix of measurements developed quantifies the relationship between SDMC and aerosol light absorption at the four wavelengths. The inverse of this matrix provides SDMC from PAS measurements for a mixture containing all four dust types. Individual and mixed dust measurements demonstrate the current state of the art in PAS sampling of SDMC.

1 INTRODUCTION

Respirable (Vincent, 2005) coal dust is produced when coal is extracted from the seam by drilling, blasting, cutting, loading and transporting operations. In underground mines it consists of aerosol with diameters (0.01 - 20 μm) of coal, silica, kaolinite, and other minerals which can be inhaled and deposited into the lungs, to a worker's alveoli where oxygen enters the blood stream. This is a serious health concern because in this region of the lungs, the human body has few means of removing hazardous particles. As a result, respirable mine dust, which includes coal as well as carbonate (i.e. rock dust (Pokhrel, Keles, Jaramillo, Agioutanti, & Sarver, 2021)) and non-carbonate material (i.e. RCS) can generate serious health hazards if these elevated dust concentrations are not adequately controlled (Kachuri et al., 2014).

The long-term exposure of coal miners to high levels of respirable coal dust can cause pneumoconiosis, emphysema, and other occupational lung diseases such as the chronic obstructive pulmonary disease, which is collectively known as black lung disease. Overexposure to respirable silica dust can lead to silicosis. Short-term, but elevated respirable silica dust

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concentrations are considerably more hazardous than the same level of cumulative exposure at lower concentrations (e.g. recommend10-hour average exposure to RCS of < 0.05 mg m^{-3}) (Howard, 2011). Consequently, it is important to design, manufacture, and commercialize a new personal real-time monitoring instrument, which has the ability to measure and monitor both respirable coal dust and respirable silica dust concentrations in the production workings and throughout the mine.

Our goal is to develop, fabricate and test a new real-time personal coal and silica dust monitoring instrument, based on photoacoustic absorption spectroscopy, as opposed to time averaged filter-based design principles such as end-of-shift FTIR analysis (Pampena, Cauda, Chubb, & Meadows, 2020) or quantum cascade laser filter analysis (Wei, Kulkarni, Ashley, & Zheng, 2017) or aerosol light scattering that is not sensitive to aerosol composition (Zhang, Nie, Liang, Chen, & Peng, 2021). The instrument will have the ability to continuously measure respirable coal dust and respirable silica dust concentrations in the production areas and throughout the mine with no user intervention and minimal equipment maintenance needs. As opposed to the current filter-based technology, the new instrument is based on photoacoustic principles; the effects of the absorbed laser light by the carbon and silica particles are measured with an acoustic detection system. Details are in the following section. The photoacoustic measurement theory has been developed (Taylor, Nascimento, Arnott, & Kocsis, 2022) and experimental demonstration in the laboratory has occurred for dust containing coal, RCS, and kaolinite (Nascimento et al., 2022).

This manuscript extends our previous work to include calcite dust (i.e. rock dust), and to give the theory and results for measurements of the SDMC that are needed for autonomous instrument operation.

2 METHODS

Laboratory measurements of SDMC are obtained using our Marple-like (Marple & Rubow, 1983) dust resuspension chamber shown schematically in Figure 1 (Nascimento et al., 2022).

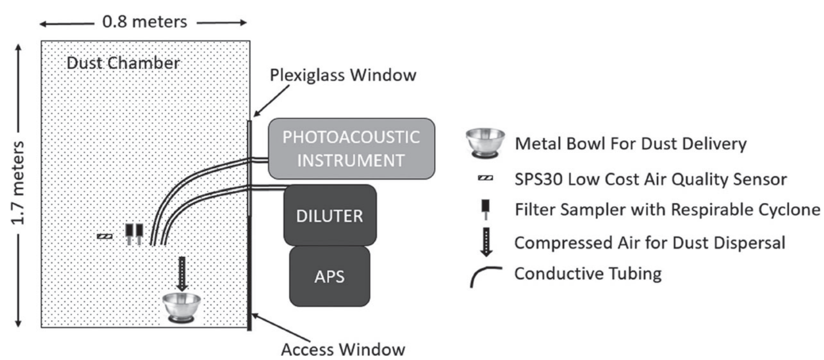

Figure 1. Schematic of the aerosol resuspension chamber used for measuring SDMC and evaluating and calibrating the photoacoustic instrument. The chamber is 2 meters long and the plexiglass window is centered. The current PA instrument is on a 2′ x 4′ optical table and the instrument enclosure fits in a 19" equipment rack.

Dust is dispensed from a container, measured at ¼ teaspoon, and placed in the metal bowl. A five second duration burst of compressed air suspends the dust. The dust mixes to a uniform state in about six minutes, its concentration diminishing slowly over about a three-hour interval. The inlets for each instrument are close together and are approximately 20 cm above the chamber floor.

The photoacoustic instrument measures aerosol light absorption by means of an acoustic detection system. A schematic of the instrument is shown in Figure 2, taken from (Nascimento et al., 2022) where details may be found. Briefly, sample air enters the inlet and is drawn through the instrument with a vacuum pump. The aerosols interact with laser light in the resonator section,

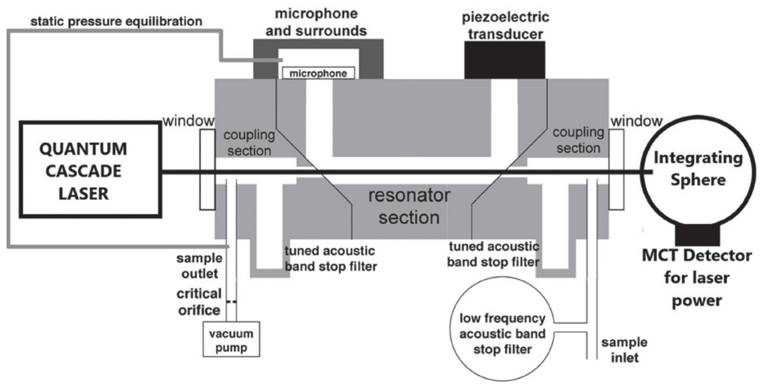

Figure 2. Schematic of the photoacoustic instrument.

creating the sound that is measured to quantify light absorption. Aerosol heats during light absorption and transfers heat to the surrounding air, thereby creating a very small pressure wave, sound, at the resonant frequency of the chamber since the laser power is modulated at this frequency (Arnott, Moosmuller, Rogers, Jin, & Bruch, 1999), then detected by a microphone. The instrument gains spectroscopic capability by use of a tunable quantum cascade laser (QCL), as the wavelength can be changed quickly. Laser power is measured with a mercury cadmium telluride (MCT) detector salvaged from a Fourier Transform Infrared Spectrometer (FTIR). The piezoelectric transducer is used to measure the resonance frequency, quality factor, and microphone calibration at resonance. Measurements are reported for both 1 and 16 second integration time. Noise equivalent light absorption measurements are obtained by integrating the microphone signal away from resonance (Lewis, Arnott, Moosmuller, & Wold, 2008).

The Aerodynamic Particle Sizer Spectrometer (APS, model 3321, TSI Incorporated, Shoreview, MN, USA) instrument optically detects the time of flight of accelerated aerosol to obtain aerodynamic diameter for individual aerosol. A 20:1 dilution ratio for sample air versus clean air was achieved using the TSI Aerosol Diluter model 3302A. The mass distribution for laboratory generated RCS is shown in Figure 3. Distributions for other dust types were similar. Most of the aerosol mass is below 4 μm, especially after the middle of the experiment, as the larger particles settle. The mass distributions are integrated to obtain real-time respirable dust mass concentrations. Because the user must input the dust density for mass concentration measurements, the APS is best suited for experiments involving homogeneous dust. Average mass distribution near coal mining fronts have peak diameters of 3-6 μm, and

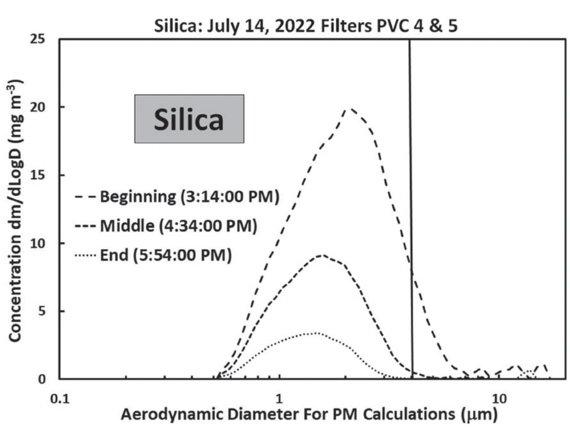

Figure 3. Mass distribution measurements from the APS for silica. Curves are for the indicated times. The vertical line is at diameter of 4 μm.

have more coarse mode aerosol than our laboratory aerosol, indicating a need to test our instruments in mines (Skubacz, Wojtecki, & Urban, 2016).

The SPS30 is a low cost air quality sensor manufactured by Sensirion (Sensirion, 2020). It is an optical particle counter that reports aerosol number concentrations for 4 size ranges in addition to PM1, PM2.5, PM4, and PM10. The recommended mass concentration range of PM2.5 is up to 1 mg m^{-3}. We have found that SPS30 PM4 values correlate well with gravimetric mass measurements, even with concentrations up to around 50 mg m^{-3}. Laboratory evaluation showed that the SPS30 over (under) reported PM2.5 for woodsmoke (Arizona road dust) compared with filter measurements, and that the SPS30 operated properly after prolonged sampling at concentrations as high as 33 mg m^{-3} (Tryner, Mehaffy, Miller-Lionberg, & Volckens, 2020).

Gravimetric and FTIR sampling were performed using PVC filters to obtain total respirable mass and silica concentrations using the NIOSH FAST technique. SKC aluminum cyclones (Zefon International, Catalog NO. ZA0060) selected respirable dust that was deposited through use of personal sampling cassettes (Zefon International, REF 745PVC-CF-FTIR) on 37 mm PVC filters (Zefon International, REF FSP37R). A vacuum pump external to the aerosol chamber was connected to the filter samplers with tubing, and operated nominally at 2.5 LPM as measured with a flow meter. The cyclone sampler operates at 2.5 LPM to obtain respirable dust. The exposed area of the filter was 6.92 cm^2. A custom filter holder was developed for FTIR measurements.

3 RESULTS

As previously mentioned, laboratory dust simulants may have different composition and aerosol size than dust found in mining environments. Many grades and type of coal are found in nature, though all absorb light throughout the 11-13 μm measurement range. Pre-dispersed silica dust can coagulate into larger, spherical clumps, perhaps due to static generated by the plastic storage container. Our research has identified key wavelengths to use to measure absorption by four coal-mine -relevant dust species. We have compared the SPS30 and APS measurements of total dust mass concentration with gravimetric measurements, and have developed a method to calculate SDMC from multispectral photoacoustic aerosol light absorption measurements.

FTIR analysis of individual filters collected during dust sampling procedures confirms the use of unique wavelengths for each components' absorption spectrum to obtain SDMC. The PAS can also be used to find the absorption spectrum of any sampled air; however, FTIR analysis is used as the industry's standard method to compare the accuracy of the PAS, as

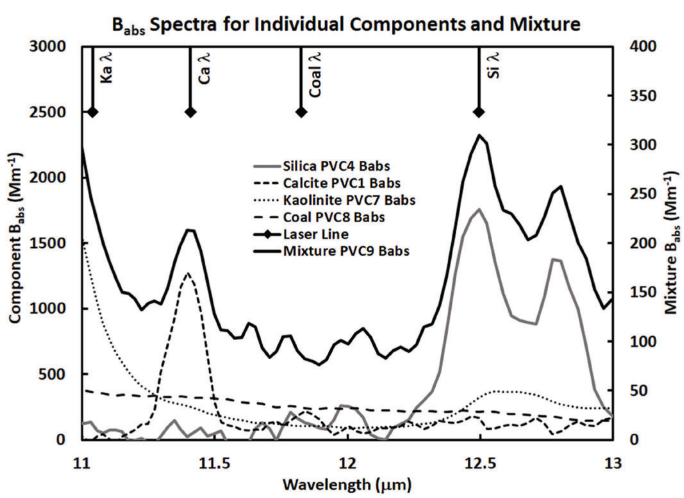

Figure 4. FTIR analysis of dust mixture and individual dusts, demonstrating the absorption spectra for each component, and how they come together to form the absorption spectra for the mixture. Also illustrated are laser lines for the wavelengths best suited for quantifying dust constituents.

well to provide supporting evidence for the wavelengths chosen to quantify SDMC. Figure 4 shows FTIR absorption spectra obtained as described in (Nascimento et al., 2022) of the individual dust types (kaolinite, calcite, coal and silica) along with the dust mixture of all four dust types in equal parts. This figure nicely illustrates the dust mixture's absorption as a combination of its constituents, represented by "Mixture PVC9 Babs."

Figure 4 also illustrates the wavelengths at which kaolinite, calcite, coal and silica absorb strongly, with the least amount of interference from water vapor and other minerals, and were carefully chosen to represent each mineral. Each dust type also absorbs a little laser light at the other three wavelengths. The wavelengths that will most effectively represent and quantify each mineral in question are 11.040 μm for kaolinite, 11.408 μm for calcite, 11.826 μm for coal, and 12.495 μm for silica. These wavelengths will be used in the further development of the PAS control software to automatically obtain concentrations of each mineral. The process of obtaining SDMC through analysis is elaborated on in Section 4. Strong light absorption by water vapor at 12.53 μm, for example, can be used along with relative humidity measurements to periodically evaluate the instrument calibration (Nascimento et al., 2022; Taylor et al., 2022).

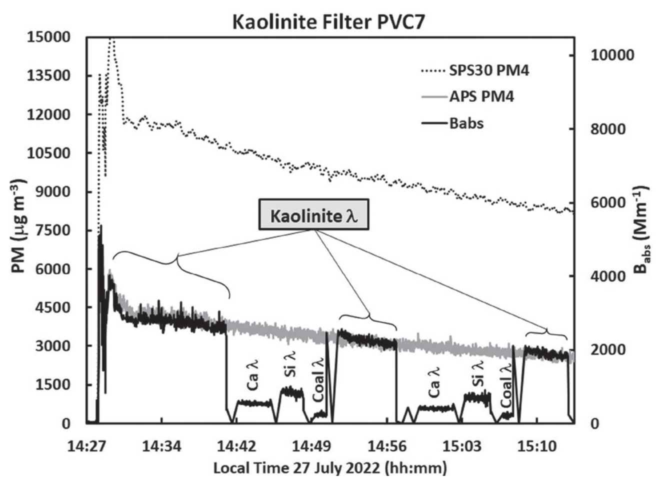

Figure 5. Photoacoustic measurements of kaolinite alongside PM4 concentrations measured by the APS and SPS30, demonstrating the absorption of laser light by kaolinite at the designated wavelength, 11.04 μm, compared to absorption at the other minerals' designated wavelengths.

Measurements of aerosolized kaolinite in the dust chamber are shown in Figure 5 to further demonstrate the absorption theory for SDMC. We can see how kaolinite has the greatest absorption at the 'Kaolinite λ'=11.04 μm, the wavelength chosen to quantify it. We can also see how kaolinite absorbs at the other wavelengths designated for calcite, silica and coal. The behavior of the dust as it is aerosolized can also be observed in this figure, from 14:27 for about 60 seconds. Once the dust is aerosolized, there is a large spike in absorption measurements from the PAS as well as PM4 measurements from the other instruments. Immediately afterwards, there is a sharp dip, representative of the dust plume as it is initially lifted, and then rises out of reach of the sensors and vacuum pumps in the chamber. For this reason, to more accurately represent mine conditions, data analysis and filter sampling doesn't commence until about six minutes after aerosolization. Data analysis for determining SDMC can be normalized using either data from the SPS30 or the APS. The APS is the standard for quantifying particulate matter concentrations however, our findings suggest the SPS30 might be a more suitable instrument for this purpose.

Gravimetric and optically determined aerosol mass concentrations by the SPS30 and APS are shown in Figure 6 for all four dust types. APS data like that shown in Figure 3 are integrated over diameter until 4 μm, and the SPS30 directly reports PM4. "PM4" is used to represent respirable gravimetric aerosol mass concentration. Figure 3 shows that most of the

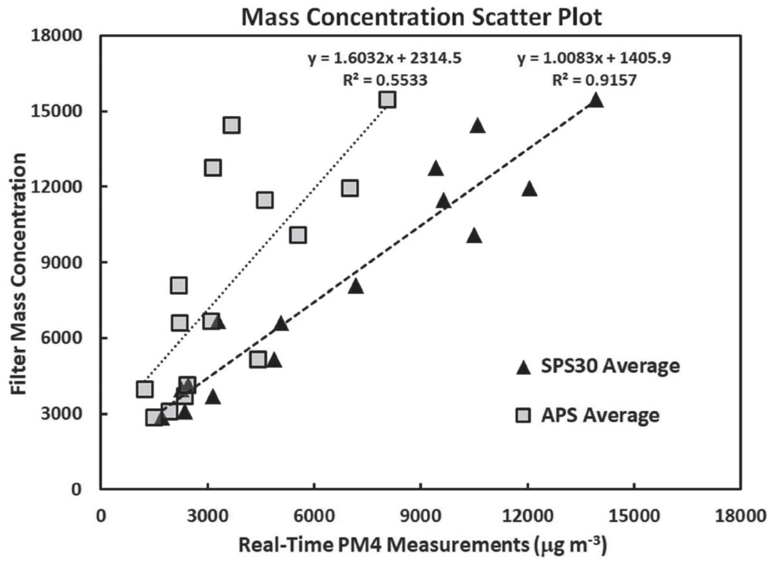

Figure 6. Scatter plot of filter mass concentration graphed against PM4 measurements for both the SPS30 and APS for all four dust types measured separately. The SPS30 has a much better fit to the gravimetric mass concentration, with an R-squared value of 0.92.

aerosol mass diameter is below 4 μm for our laboratory generated aerosol. The SPS30 data most closely resembles the filter mass, with a coefficient of determination of around 0.92, compared to the APS data, with a coefficient of determination of 0.55. Both however also have a large offset. These findings support the use of the SPS30 PM data for use in the matrix inversion calculations.

4 SPECIATED DUST MASS FROM PHOTOACOUSTIC MEASUREMENTS

There are two main equations that allow for the PAS to measure aerosol concentrations from light absorption. The first is an expression for aerosol light absorption, B_{abs}. It is given by

$$B_{abs} = \frac{P_m A_{res}}{P_L(\gamma - 1)} \frac{\pi^2 f_0}{Q} \tag{1}$$

where P_m is the acoustic pressure measured with the microphone, P_L is the laser power used, about 5 mW in our case, A_{res} is the cross-sectional area of the resonator, γ=1.4 is the is the ratio between isobaric and isochoric dry air specific heats, f_0 is the resonance frequency of the chamber, about 1500Hz, and Q is the quality factor, which is about 60. B_{abs} has units of Mm^{-1}, or inverse megameters. All of these values are either controlled by experimental parameters or measured with the PAS. The other equation vital to the success of our method is given by

$$B_{abs} = MAE \times PM \tag{2}$$

where PM has units of [$\mu g\ m^{-3}$] and MAE, or mass absorption efficiency, has units of [$m^2 g^{-1}$]. In determining the SDMC, which here are silica, kaolinite, black coal and as a new addition calcite, we must turn PM and B_{abs} into vectors, with dimensions defined by the number of species, which would subsequently turn MAE into a square matrix also with dimensions defined by species count. For this case, there are four species present, corresponding to a 4x4 MAE matrix, and 4x1 PM and B_{abs} vectors. The MAE matrix is separated into both species and wavelength used to measure a species to be able to quantify contributions from other species at wavelengths where a given species was strongly absorbing. For clarity, below is a more detailed look at Equation 2:

$$\begin{pmatrix} MAE_{si,\lambda 1} & MAE_{ka,\lambda 1} & MAE_{co,\lambda 1} & MAE_{ca,\lambda 1} \\ MAE_{si,\lambda 2} & MAE_{ka,\lambda 2} & MAE_{co,\lambda 2} & MAE_{ca,\lambda 2} \\ MAE_{si,\lambda 3} & MAE_{ka,\lambda 3} & MAE_{co,\lambda 3} & MAE_{ca,\lambda 3} \\ MAE_{si,\lambda 4} & MAE_{ka,\lambda 4} & MAE_{co,\lambda 4} & MAE_{ca,\lambda 4} \end{pmatrix} \begin{pmatrix} PM_{si} \\ PM_{ka} \\ PM_{co} \\ PM_{ca} \end{pmatrix} = \begin{pmatrix} B_{abs_{si}} \\ B_{abs_{ka}} \\ B_{abs_{co}} \\ B_{abs_{ca}} \end{pmatrix} \tag{3}$$

Of course, the end goal here is measuring PM for different species. So, multiplying the left of both equations with the inverse of the MAE matrix gives $MAE^{-1} \times B_{abs} = PM$. The inversion of the matrix is done after all elements are obtained.

To inspect the relationship between PM and B_{abs}, values of SPS30 PM4, SPS30 PM2.5, and APS PM4 were plotted with B_{abs} measurements as time series on separate axes for a given species at a given wavelength. To obtain MAE matrix elements, PM values were plotted against B_{abs} value as a scatter plot, and a linear regression was performed. For four dust species, each measured at four separate wavelengths, there are 16 total charts like this for each PM type. The slopes of the regressions for PM2.5 were used to obtain MAE matrix elements. The completed inverse MAE matrix, along with the example of all species combined together, is included as Equation 4.

$$\begin{pmatrix} PM_{si} = 518.8\frac{\mu g}{m^3} \\ PM_{ka} = 529.7\frac{\mu g}{m^3} \\ PM_{co} = 49.8\frac{\mu g}{m^3} \\ PM_{ca} = 119.8\frac{\mu g}{m^3} \end{pmatrix} = \begin{pmatrix} 2.0343 & -0.5131 & -2.0984 & 0.0 \\ 0.1270 & 2.7985 & -3.4895 & 0.0 \\ -0.4853 & -0.3894 & 7.2397 & 0.0 \\ 0.0495 & -0.2760 & -1.4918 & 1.4782 \end{pmatrix} \begin{pmatrix} B_{abs_{si}} = 356.61 Mm^{-1} \\ B_{abs_{ka}} = 227.01 Mm^{-1} \\ B_{abs_{co}} = 43.00 Mm^{-1} \\ B_{abs_{ca}} = 154.90 Mm^{-1} \end{pmatrix} \tag{4}$$

The inverse MAE matrix, Equation 4, has units of [$g\ m^{-2}$]. This matrix uses SPS30 PM2.5 values instead of the APS PM4 because one: SPS30 has proven to function with high-fidelity compared to the APS, as shown in Figure 6; and two: PM2.5 appears to have more accuracy in its prediction than does PM4. This is due to a more steady and consistent flow of PM2.5 for the PAS. These B_{abs} entries were calculated by fitting the PM2.5 time series for the filter sample of the dust mixture, containing equal parts by volume of coal, kaolinite, calcite and silica, to a second-order polynomial, applying this curve fit to B_{abs} to renormalize B_{abs} such that there is no drop in PM for a given time interval (30 minutes in this case), mimicking measuring all four species at once with four different lasers, instead of in sequence. It will be necessary to perform the measurements sequentially at each wavelength as quickly as possible (1-16 seconds per measurement) for a fully automated PAS, and to include a SPS30 to normalize the PM measurements.

5 CONCLUSION

We have developed a method for obtaining speciated dust mass concentration (SDMC) through mass absorption efficiency matrix inversion, found unique wavelengths at which to quantify absorption spectra of kaolinite, calcite, coal and silica dusts and provided evidence to support the use of SPS30 measurements in lieu of APS data. This was achieved with the use of the real-time personal coal and silica dust monitoring instrument, based on photoacoustic absorption spectroscopy. The procedure for obtaining SDMC was developed through use of measurements with the laser manually set to the four wavelengths used for each dust type.

The current instrument is mounted on a 2′ x 4′ optical table and the instrument enclosure fits in a 19” equipment rack. Future development of the photoacoustic spectrometer involves refinement and further testing in both laboratory and mine settings. The instrument will have the ability to continuously measure respirable coal and silica dust concentrations in the production areas and throughout the mine with no user intervention and minimal equipment maintenance needs. Areas in need of further development include: automatic calibration with relative humidity, measurement of speciated dust movement in-flight with rapid (1-16 second) measurements at the four laser wavelength, reducing the size of the acoustical resonator, replacing the liquid-nitrogen-cooled integrating sphere laser power detector with a smaller device—all to reduce instrument size and to improve commercial viability.

The relevance and importance of this research cannot be stressed enough—the health and safety of the men and women procuring natural resources, the very backbone of the developing and developed world, depends on our ability to monitor the hazards they are exposed to during a normal work day. Without the health and safety of our miners, the industry risks productivity and financial sustainability, not to mention loss of life.

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The Mine Ventilation Symposium series has always been a premier forum for ventilation experts, practitioners, educators, students, regulators, and suppliers from around the world to exchange knowledge, ideas, and opinions. **Underground Ventilation** features sixty-seven selected technical papers in a wide range of ventilation topics including: auxiliary and primary systems, mine fans, case studies, computational fluid dynamics applications, diesel particulate control, electric machinery, mine cooling and refrigeration, mine dust monitoring and control, mine fires and explosion prevention, mine gases, mine heat, mine ventilation and automation, occupational health and safety, renewable/alternative energy, monitoring and measurement, network analysis and optimization, and planning and design.

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