

# Evaluation of a Portable Photometer for Estimating Diesel Particulate Matter Concentrations in an Underground Limestone Mine

WINTHROP F. WATTS\*, DAVID D. GLADIS, MATTHEW F. SCHUMACHER, ADAM C. RAGATZ and DAVID B. KITTELSON

*Department of Mechanical Engineering, Center for Diesel Research, University of Minnesota, 111 Church Street Southeast, Minneapolis, MN 55455, USA*

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**A low cost, battery-operated, portable, real-time aerosol analyzer is not available for monitoring diesel particulate matter (DPM) concentrations in underground mines. This study summarizes a field evaluation conducted at an underground limestone mine to evaluate the potential of the TSI AM 510 portable photometer (equipped with a Dorr-Oliver cyclone and 1.0- $\mu\text{m}$  impactor) to qualitatively track time-weighted average mass and elemental, organic, and total carbon (TC) measurements associated with diesel emissions. The calibration factor corrected correlation coefficient ( $R^2$ ) between the underground TC and photometer measurements was 0.93. The main issues holding back the use of a photometer for real-time estimation of DPM in an underground mine are the removal of non-DPM-associated particulate matter from the aerosol stream using devices, such as a cyclone and/or impactor and calibration of the photometer to mine-specific aerosol.**

*Keywords:* diesel exhaust; diesel particulate matter; direct-reading instruments; photometer; real-time measurement

## INTRODUCTION

Diesel exhaust is a complex mixture of gases that includes nitric oxide (NO), nitrogen dioxide (NO<sub>2</sub>), carbon monoxide, carbon dioxide, sulfur dioxide, volatile hydrocarbons, and diesel particulate matter (DPM). Exposure to DPM is primarily a concern because of its potential carcinogenicity. In 1988, the National Institute for Occupational Safety and Health (NIOSH) recommended that whole diesel exhaust be regarded as a 'potential occupational carcinogen' and stated that reductions in workplace exposure would reduce cancer risks (NIOSH, 1988). In 1989, the International Agency for Research on Cancer (IARC) declared 'Diesel engine exhaust is probably carcinogenic to humans' (IARC, 1989).

The US Mine Safety and Health Administration (MSHA) has established 160  $\mu\text{g m}^{-3}$  of total carbon (TC) as the time-weighted-averaged, full-shift, permissible exposure limit to DPM in metal and non-metal mines. TC is the sum of elemental carbon (EC) and organic carbon (OC) as determined by the NIOSH method 5040 (NIOSH, 2003). Details on the method of enforcement and error factors used in the calculations are given elsewhere (MSHA, 2008).

The physical and chemical characteristics of DPM are important considerations when measuring DPM in the presence of other aerosols. A typical DPM size distribution is shown in Fig. 1 (Whitby and Cantrell, 1976; Kittelson, 1998). Figure 1 shows the relationships between the nucleation, accumulation, and coarse modes for three weighted size distributions (number, surface area, and mass). The curves have a log-normal trimodal form and the concentration in any size range is proportional to the area under the corresponding curve in that range.

\*Author to whom correspondence should be addressed.  
Tel: +1-612-625-2045; fax: +1-612-624-1578;  
e-mail: watts012@umn.edu

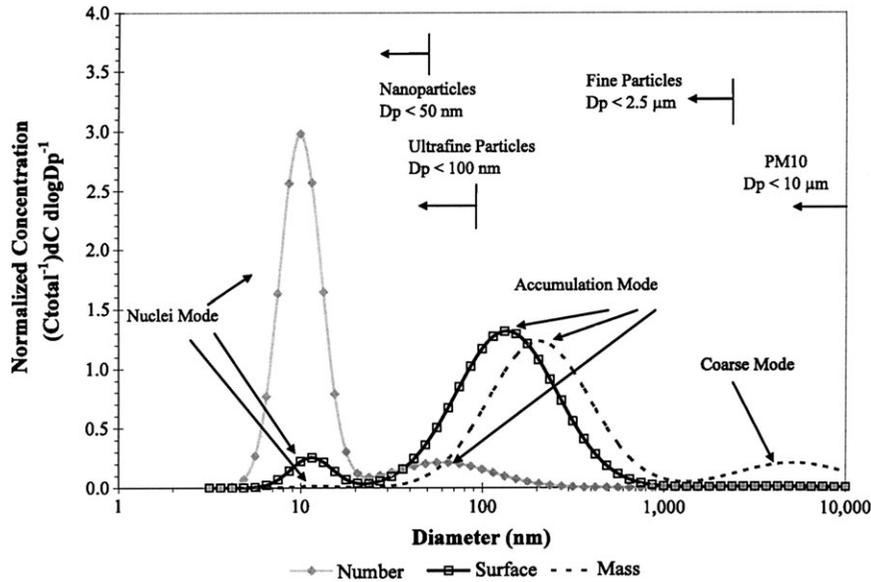


Fig. 1. Typical diesel number, surface area, and mass-weighted size distributions (Source Whitby and Cantrell, 1976; Kittelson, 1998).

The coarse mode consists of particles  $>1000$  nm and contains 5–20% of the mass. Coarse mode particles are formed by re-entrainment of DPM, which was deposited on cylinder and exhaust system surfaces. Most of the DPM mass is found in the accumulation mode (roughly 50–500 nm) and is composed of carbonaceous agglomerates and adsorbed materials.

The nucleation mode (3–50 nm) typically contains  $<10\%$  of the particle mass but  $>90\%$  of the particle number. Nucleation mode particles are usually composed of nearly all volatile material (Kittelson *et al.*, 2005, 2006). Hydrocarbons are the main constituents of these particles for engines running under normal conditions with ultra-low sulfur ( $<15$  p.p.m. S) fuels (Ziemann *et al.*, 2002; Sakurai *et al.*, 2003a,b).

The importance of the nucleation, accumulation, and coarse size modes becomes more obvious when aerosol sampling in an underground mine where mine dust is present. Previous studies conducted in dieselized and non-dieselized mines have shown that the distinctive nature of the DPM size distribution can be used to separate DPM from other mine dusts. In gassy areas of underground coal mines, diesel-powered vehicles are equipped with water scrubbers that cool the exhaust and arrest flames and sparks to prevent the ignition of coal dust or methane gas. As much as 20% of the DPM mass found in the coarse mode is removed by the water scrubber (Mogan, *et al.*, 1986), thus tailoring the resulting exhaust

aerosol into two size fractions;  $<0.8$ - $\mu\text{m}$  diesel fraction and the  $>0.8$ - $\mu\text{m}$  dust fraction. In metal and nonmetal mines,  $\sim 20\%$  of the diesel aerosol contributes to the  $>0.8$ - $\mu\text{m}$  fraction (Cantrell and Rubow, 1990). This division of DPM aerosol from mechanically generated mine dusts led to the size-selective sampling method for DPM that is described in detail elsewhere (Cantrell and Rubow, 1990; Rubow *et al.*, 1990a,b; McCartney and Cantrell, 1992; Cantrell *et al.*, 1993). The method is based upon using inertial impaction for size classification and gravimetric analysis. This method of pre-classification is also useful to prepare an aerosol for other methods of aerosol measurement, such as the EC, OC, TC NIOSH 5040 method, or instruments that measure aerosol concentration in real time. An underground mine statistical comparison between the size-selective and the EC–OC methods is published elsewhere (Ramachandran and Watts, 2003).

In a previous study (Bagley *et al.*, 2001; Watts, 2004) conducted in a Louisiana salt mine, a portable diffusion charger (DC), a portable photoelectric aerosol sensor (PAS), and a condensation particle counter (CPC) were used for monitoring diesel aerosol concentrations in the mine. The goal of the study was to determine if the use of modern low-emission diesel engine technology introduced any new potential health concerns into the underground mine environment. The study demonstrated the usefulness of real-time instruments to track diesel aerosol as

a function of diesel activity and to evaluate low-emission engine technology. However, use of the instruments was complicated by the need to dilute the sample aerosol because of the high-dust environment.

The objective of this study was to select and evaluate a portable battery-powered aerosol monitor for real-time measurement of DPM in an underground non-coal mine. A laboratory study was conducted in which a suite of portable, battery-powered, and non-portable aerosol instruments sampled diluted diesel aerosol. The portable instrument yielding the highest correlation with the EC, OC, and TC size-selective method used by MSHA was selected for evaluation underground. For the underground evaluation, the size-selective inlet developed by the US Bureau of Mines was used to eliminate aerosol  $>1.0 \mu\text{m}$  in size.

## METHODS

### *Evaluation of laboratory-generated diesel aerosol and instrument selection*

Rack-mounted and portable aerosol instruments were evaluated in the laboratory using exhaust from a model year 2005, Deere 4045H, 4-cylinder, 4.5-l, 129-kW (at 2400 r.p.m.) diesel engine. This engine is turbocharged, aftercooled with common rail fuel injection, and has US Environmental Protection Agency tier 2 approval for off-highway applications. Exhaust gas recirculation was not used and tests were conducted with and without a diesel oxidation catalyst (DOC) in the exhaust even though some underground mines do not use catalyzed aftertreatment devices because of concern for  $\text{NO}_2$  production. However, DOCs are known to reduce the organic portion of the DPM, which can impact instrument response (Kittelson *et al.*, 2005) so the evaluation included tests with and without the DOC in the exhaust stream.

Tests were conducted at steady-state conditions at four engine speed and load conditions that were selected to challenge the instruments to diesel aerosols having different size characteristics as shown in Fig. 2. Size distributions were determined using a TSI 3080 Scanning Mobility Particle Sizer (SMPS). Measurements were made using a two-stage ejector dilutor system and sampling methods similar to those described in detail elsewhere (Abdul-Khalek *et al.*, 1999; Kittelson *et al.*, 2002, 2006).

Figure 2 shows the weighted average size distributions for the four test conditions. In this case, the engine was fueled with ultra-low sulfur diesel fuel (ULSD) containing 6 p.p.m. S. No DOC was in the

exhaust and standard deviations are shown. The weighted average was calculated as the sum of the measurements divided by their variances of the mean, divided by the sum of the reciprocals of the variances of the mean. Evaluations were also conducted with B50 fuel, which is a 50% blend of soy methyl ester biodiesel and ULSD. The limestone mine that volunteered to host the underground portion of this study was using B50 fuel at the time the study took place and was planning to transition to a B99 renewable fuel.

During the evaluation of the portable instruments, a TSI 3090 Engine Exhaust Particle Sizer (EEPS) was also used to determine the aerosol number size distribution. In addition, a suite of rack-mounted and portable battery-operated aerosol instruments were used. The rack-mounted instruments were used to characterize the number, surface area, and volume characteristics of the diesel test aerosol. These instruments included:

- Portable and desktop CPCs (TSI 3007, 3025A) that determine the total particle number concentration.
- Portable and desktop DCs and PASs (EcoChem Analytics and Matter Engineering) that determine active surface area concentration.
- A nanoparticle surface area monitor (TSI 3550 NSAM) that monitors either the alveolar (setting used in this study) or the thoracic lung-deposited surface area, an electrical aerosol detector (TSI 3070A EAD) that determines particle length concentration.
- Portable photometers (TSI DustTrak, TSI AM 510) that estimate the mass concentration.

A four position cassette filter holder (Kittelson and Watts, 2008) was used to collect 37-mm filter samples for gravimetric or EC–OC analysis. Spacers were inserted into the cassettes to ensure even DPM distribution for EC analysis, and backup high-purity quartz filters were used to correct for EC–OC artifacts. Flow through the filters was measured and recorded using TSI 41221 ( $0.01\text{--}20 \text{ l min}^{-1}$ ) flow meters. Flow through the filters was controlled by a manifold holding 12 critical orifices. The flow rates were 4, 8, or  $12 \text{ l min}^{-1}$ , and it was possible to collect four samples at one of three flow rates during each test period. The flow rate used for sampling was determined by the expected filter loading time. A Cahn microbalance was used to weigh the gravimetric samples; further details, including photographs of the sampling manifold, are available elsewhere (Kittelson and Watts, 2008). The

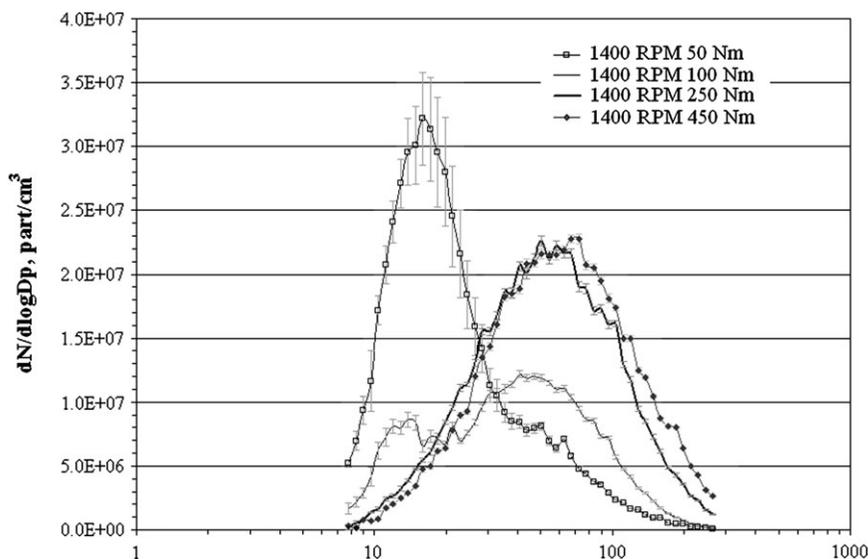


Fig. 2. Weighted average SMPS size distribution for the four test condition with standard deviation for ULSD fuel and no DOC.

gravimetric procedure described below in the mine study section was used.

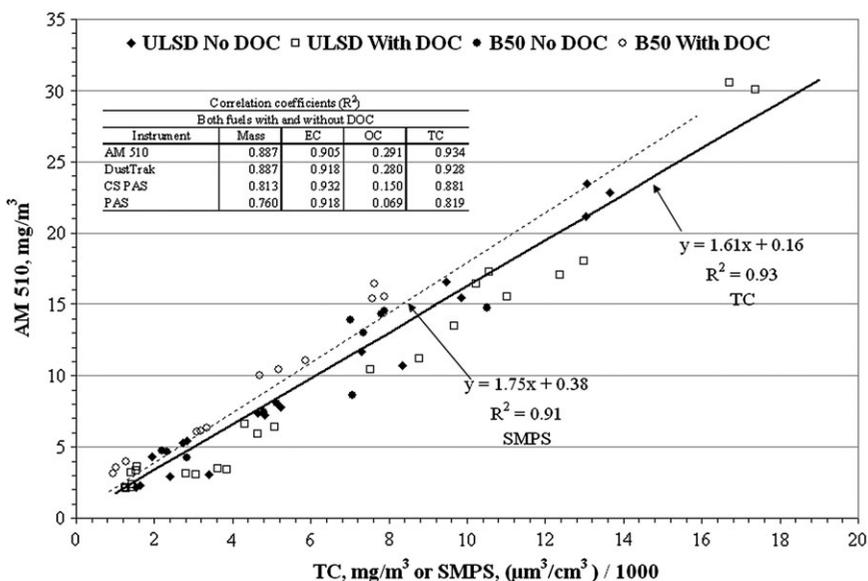
The first-stage dilution ratio was determined by measuring the raw exhaust NO concentration using a Rosemount 880A NO<sub>x</sub> analyzer. The diluted NO concentration was measured using a CAI 600 series NO<sub>x</sub> analyzer. Heated sampling lines were used with both analyzers. The second-stage dilution ratio was monitored using a separate high sensitivity EcoPhysics NO<sub>x</sub> analyzer. If additional dilution was required for the 3025A CPC, which has an upper limit of 100 000 part cm<sup>-3</sup>, a leaky filter dilutor was used. The leaky filter dilution ratio was determined using 100 p.p.m. dioctyl sebacate aerosol with and without the leaky filter in-line. A mixing orifice was placed after the leaky filter to ensure proper mixing before the sample entered the 3025A CPC.

Instrument response was characterized with and without a catalytic stripper (CS) that removed volatile material that forms nucleation mode particles. Large nucleation modes are known to adversely impact the response of the PAS (Kittelson *et al.*, 2005). Additional details of the sampling methods, instrumentation including the CS, quality assurance procedures, and data acquisition hardware and software are found elsewhere (Kittelson and Watts, 2008).

Figure 3 summarizes the results from the laboratory instrument comparison for ULSD and B50 fuels with and without the DOC. Shown in the figure is a matrix of correlation coefficients ( $R^2$ ) for the top performing, battery-powered, portable instruments (AM 510,

DustTrak, PAS with and without a CS) and mass, EC, OC, and TC. The order in the table is determined by the TC  $R^2$  value—highest to lowest. The two photometers correlated best with mass and TC while the CS PAS and PAS correlated better with EC. None of the instruments correlated well with OC, which is consistent with previous findings that show the nucleation mode composed primarily of volatile material can cause problems for photometers and the PAS.

Figure 3 also shows the TC concentration plotted against the AM 510 concentration for the two fuels with and without a DOC in the exhaust. The points are coded to delineate the four test conditions ULSD no DOC, ULSD with DOC, B50 no DOC, and B50 with DOC. The  $R^2$  value for the line that these points represent is 0.934, the highest value shown in the inserted table. The same AM 510 values are plotted against the corresponding SMPS volume concentrations and the resulting regression line is shown with an  $R^2$  of 0.91. The volume concentration, which can be determined in near-real time using the SMPS or in real time using the EEPS, is frequently used as a surrogate for gravimetric mass concentration. Additional data from the instrument comparison including means, standard deviation, and size distributions are available in the contract final report (Kittelson and Watts, 2008). The AM 510 photometer was selected for evaluation in an underground limestone mine because of these results and because the AM 510 is the smallest and least expensive of the battery-powered portable instruments.



**Fig. 3.** TC concentration plotted against the AM 510 concentration for ULSD and B50 fuels with and without a DOC in the exhaust, AM 510 concentration plotted against the SMPS volume concentration for the same test conditions, and a summary instrument correlation matrix.

### Underground mine evaluation

The underground limestone mine is a room and pillar mine with three levels located in Kentucky. The mine operates two 10-h shifts per day (production and maintenance), 5 days a week, and a single 8-h shift on Saturday. The underground mine is accessed by decline ramps. Limestone is drilled and blasted and the shot rock is loaded onto haul trucks by front-end loaders and transported to the underground primary and secondary crushing plants where it is crushed and sized. The material is then conveyed to the surface crushing and screening plants for further processing and stockpiling. Other diesel equipment included roof bolters, scalers, oilers, and water trucks. Diesel pickup trucks were the main source of personnel transportation and were used throughout the mine. The mine was using B50 fuel containing 3 p.p.m. sulfur at the time of the study.

The objective of the study was to evaluate the ability of the TSI AM 510 photometer to estimate DPM concentrations. Ten AM 510 photometers were equipped with Dorr-Oliver cyclones and US Bureau of Mines impactors. The photometers were modified by TSI to operate at a flow rate of  $1.7 \text{ l min}^{-1}$ , which yielded an effective 50% cut for the impactor between 0.9 and  $1.0 \mu\text{m}$ . Combination of the time-weighted average (TWA) mass concentration obtained from the AM 510 with the TWA mass concentration obtained from the impactor substrate ( $>1.0 \mu\text{m}$  mass)

provides an estimate of the TWA respirable dust concentration. A CS was not used underground because it is neither portable nor battery powered.

Sample locations were selected after visiting the mine and consulting with mine personnel. The locations were selected to provide a wide range of aerosol concentrations with varying mixtures of diesel aerosol and limestone dust. The locations included the crusher operator's air-conditioned control room on Level 3, the maintenance area on Level 2, the main ramp from Level 2 to Level 3, and a haul truck operating on Level 3. The baskets on the haul truck were located outside the operator's cab near the entry ladder and were exposed to the highest concentrations of limestone dust. The crusher operator's control room had the lowest total concentrations.

At each of the three sampling locations, two baskets of instruments were used. The baskets contained three photometers as described previously, one gravimetric sampler, and three EC-OC samplers. The 10th photometer was placed in a basket either at the ramp or at the maintenance location. The gravimetric sample was collected using the size-selective method developed by the US Bureau of Mines (McCartney and Cantrell, 1992). In that method, a Dorr-Oliver cyclone, US Bureau of Mines impactor, and a Mine Safety Appliance (MSA) filter cassette (model 457193) are held in a MSA model 456243 personal respirable dust sampler holder. An MSA Escort<sup>®</sup> Electronic Laminar Flow (ELF) pump

calibrated for  $1.7 \text{ l min}^{-1}$  provided sample flow. The MSA filters were preweighed and postweighed using a Cahn microbalance located in a temperature- and humidity-controlled room after a 4-h equilibration period. Both laboratory and field blank filters were included as gravimetric controls. The EC-OC samples were collected using Dorr-Oliver cyclones and SKC cassettes containing the  $1.0\text{-}\mu\text{m}$  built-in impactor (catalog No. 225-317). Each SKC cassette contains a backup high-purity quartz filter and one of every three backup filters was analyzed to determine an EC-OC artifact correction. EC-OC analysis was conducted by the NIOSH Pittsburgh Research Laboratory using NIOSH method 5040. An MSA ELF pump calibrated for  $1.7 \text{ l min}^{-1}$  provided sample flow. Figure 4 shows the sampling array.

During the first week, the sampling locations included crusher operator control room, main ramp, and haul truck. During the second week, the maintenance bay sampling site was used rather than the haul truck because of the high-dust concentrations encountered on the haul truck and the desire not to overload the impactors with limestone dust. The main ramp samples were collected at a height of 1.5 m beside the ramp. The maintenance bay samplers were located on a parts cabinet located near a rib at a height of  $\sim 1.5$  m. Only the main ramp and crusher operator room locations were sampled on Saturday. Sampling lasted  $\sim 6$  h each weekday and 3 h on Saturday. The duration of sampling was established by estimating the amount of collected mass and working with the miners' schedule to ensure work completion by the end of the work shift.

Photometer and MSA ELF pump flow calibrations were done underground either at the beginning or at the end of each day using a bubble meter (Sensidyne

Gilibrator) that was factory calibrated prior to the beginning of the study. All instruments were calibrated to  $1.7 \text{ l min}^{-1}$  with the cyclone and impactor in-line and contained in a calibration jar. The AM 510 was also zeroed each day. Table 1 summarizes the samples that were collected at each location with a description of data resulting from each sample.

## RESULTS AND DISCUSSION

Tables 2–5 summarize the results for the gravimetric, EC, OC, TC, and AM 510 samples collected at the mine. In the tables, the number of samples refers to the number of TWA samples used to calculate the mean and standard deviation. For the AM 510 and EC-OC samples, where multiple samples were collected at each location, the samples were averaged to provide a single TWA concentration for each location on each day.

Table 5 shows average ratios obtained from the AM 510, MSA, and EC-OC samples. These values could be used to establish alternative calibration factors for the AM 510 under the assumption that the gravimetric or EC-OC samples act as reference samples. The AM 510 is factory calibrated to the respirable fraction of standard International Standards Organization 12103-1, A1 Test Dust (Arizona test dust  $1\text{--}10 \mu\text{m}$  in size). The calibration factor established at the factory is 1.0. A new calibration is established by dividing the average obtained from the reference sampler by the AM 510  $<1.0\text{-}\mu\text{m}$  average. To illustrate the calculation, TC is used as the reference sample since the current MSHA standard is based on TC. The ratio 0.219 is obtained by dividing  $0.0405 \text{ mg m}^{-3}$  (crusher TC) from Table 4 by  $0.185 \text{ mg m}^{-3}$  (crusher AM 510  $<1.0 \mu\text{m}$ ) from



Fig. 4. Sampling instrumentation from left to right: gravimetric sampler, EC-OC sampler, AM 510, EC-OC sampler, two AM 510s, and an EC-OC sampler.

Table 1. Summary of samples collected during the mine study

Sampler	Respirable dust, mg m <sup>-3</sup>			EC (mg m <sup>-3</sup> ), <1.0 µm	OC (mg m <sup>-3</sup> ), <1.0 µm	TC (mg m <sup>-3</sup> ), <1.0 µm
	<1.0 µm	>1.0 µm	Total			
Size-selective MSA	Yes <sup>a</sup>	Yes <sup>b</sup>	Yes			
SKC DPM				Yes <sup>c</sup>	Yes <sup>c</sup>	Yes
AM 510	Yes <sup>d</sup>	Yes <sup>b</sup>	Yes			

<sup>a</sup>Determined by gravimetric analysis of MSA filter.

<sup>b</sup>Determined by gravimetric analysis of impactor substrate.

<sup>c</sup>Determined by gravimetric analysis of impactor substrate.

<sup>d</sup>Determined by photometer.

Table 2. MSA gravimetric samples collected at each mine sampling location

Location	Number of samples	<1.0 µm, mg m <sup>-3</sup>		>1.0 µm, mg m <sup>-3</sup>		Response concentration, mg m <sup>-3</sup>	
		Average	Standard	Average	Standard	Average	Standard
Crusher	10	0.113	0.095	0.116	0.081	0.229	0.092
Vehicle	5	0.381	0.081	0.846	0.294	1.228	0.234
Ramp	10	0.436	0.167	1.099	0.403	1.534	0.441
Maintenance	4	0.278	0.086	0.411	0.205	0.690	0.161

Table 3. AM 510 photometer samples collected at each mine sampling location

Location	Number of samples	<1.0 µm, mg m <sup>-3</sup>		>1.0 µm, mg m <sup>-3</sup>		Response concentration, mg m <sup>-3</sup>	
		Average	Standard	Average	Standard	Average	Standard
Crusher	30	0.185	0.075	0.132	0.118	0.316	0.161
Vehicle	15	1.014	0.181	0.851	0.281	1.866	0.366
Ramp	37	1.120	0.173	1.057	0.343	2.177	0.437
Maintenance	15	0.645	0.065	0.375	0.159	1.020	0.192

Table 4. EC–OC &lt;1.0 µm filter samples collected at each mine sampling location

Location	Number of samples	EC µg m <sup>-3</sup>		OC µg m <sup>-3</sup>		TC µg m <sup>-3</sup>		TC <sup>a</sup> % >TLV	% EC
		Average	Standard	Average	Standard	Average	Standard		
Crusher	30	21	9	19	29	40	33	0.0	52.6
Vehicle	15	132	51	81	36	212	74	66.7	62.0
Ramp	30	134	33	105	36	239	60	90.0	55.9
Maintenance	12	45	28	39	71	83	79	8.3	53.6

<sup>a</sup>Threshold limit value for TC established by the MSHA is 160 µg m<sup>-3</sup>.

Table 3. The ratios for the crusher, ramp, and haul truck locations are relatively close together ranging from 0.209 to 0.219 while the maintenance area location is lower at 0.129. This suggests that a different aerosol mixture is present in the maintenance bay, which is consistent with the welding activity that takes place in that area. It is important to recognize that presence of welding fumes, drill oil mist, or other aerosol in specific areas of the mine can impact instrument performance and calibration. At this mine, the main sources of aerosol were mining

activity generating limestone dust and diesel vehicles generating DPM. In areas where drill oil mist or welding fumes are present, separate calibration factors should be established.

Included in Table 5 are the ratios obtained from the average mass concentrations obtained from the >1.0-µm impactors used with the MSA and AM 510 samplers. The impactors performed well regardless of application with average ratio of 1.0 ± 0.09. Since impactor performance is mainly a function of sample flow, the agreement suggests that pump

flow rates changed relatively little during the sampling periods and the impactors were not overloaded.

Figure 5 summarizes the relationship between the AM 510  $<1.0\text{-}\mu\text{m}$  samples and TC obtained by multiplying the individual daily average AM 510 results by the average calibration factor (0.214) obtained from the crusher, ramp, and vehicle samples shown in Table 5 and excluding the maintenance area samples. The correlation coefficient obtained after the correction and exclusion of the maintenance area samples is 0.93. Had the maintenance area samples been included in the figure and the same calibration factor applied, the correlation coefficient would have been 0.88.

### CONCLUSIONS

The objective of this study was to select and evaluate a portable aerosol monitor for real-time measurement of DPM in an underground non-coal mine. A labora-

tory study was conducted to evaluate a suite of portable aerosol instruments sampling diluted diesel aerosol. The TSI AM 510 photometer performed best in this evaluation and was selected for evaluation in an underground limestone mine. TWA photometer response was compared to the EC, OC, and TC method developed by NIOSH and used by MSHA to determine compliance in non-coal mines and the size-selective method developed by the U.S. Bureau of Mines.

In field tests conducted at an underground limestone mine, the TSI AM 510 portable photometer (equipped with a Dorr-Oliver cyclone and  $1.0\text{-}\mu\text{m}$  impactor) qualitatively tracked TWA mass and EC-OC measurements. The correlation coefficient ( $R^2$ ) between the TC and the adjusted photometer measurements was 0.93. The main issues holding back the use of a photometer for real-time estimation of DPM are the removal of non-DPM-associated particulate matter associated with mining activity from the aerosol stream using devices, such as a cyclone

Table 5. Ratios of reference samples to AM 510 photometer samples

Location	Respirable dust ratios		$<1.0\ \mu\text{m}$	$<1.0\ \mu\text{m}$
	MSA/AM 510	RD $>1.0\ \mu\text{m}$ impactor MSA/AM 510	TC/AM 510	EC/AM 510
Crusher	0.724	0.878	0.219	0.115
Vehicle	0.658	0.994	0.209	0.130
Ramp	0.705	1.039	0.214	0.119
Maintenance	0.676	1.098	0.129	0.069

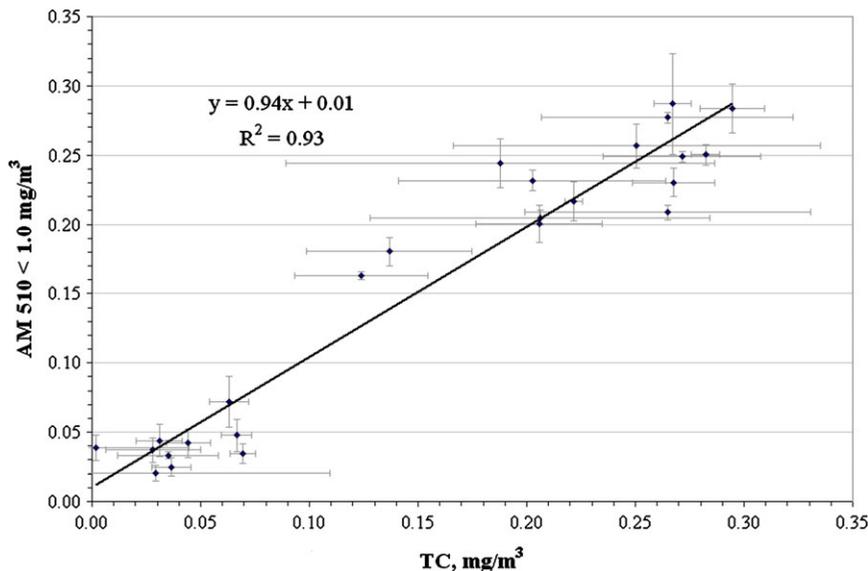


Fig. 5. Relationship between TC and AM 510  $<1.0\ \mu\text{m}$  samples adjusted for the average calibration factor obtained from the crusher, truck and ramp samples and excluding the maintenance area samples.

and/or impactor and calibration of the photometer to mine-specific aerosol. The photometer calibration for DPM must be re-evaluated when other aerosols like drill oil mist or welding fume are present in the sampling area.

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