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Improvement of size-selective sampling of diesel aerosols in underground mines

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ABSTRACT: The quantification of exposures to diesel particulate matter (DPM) could be adversely affected by the presence of micron-sized aerosols in DPM samples containing organic and elemental carbon from other than combustion sources. Therefore, size classification plays an important role in the collection of representative DPM samples. The results of this evaluation were used to assess the potential for an improvement in the size separation of diesel and micron size aerosols by using lower cutoff-size impactors in DPM sampling cassettes. Three impactors with the cut-off diameters of 258, 466, and 716 nm were characterized in the laboratory using polystyrene divinylbenzene beads, and consequently the impactors were evaluated in the outby area of an underground coal mine incorporating diesel-powered equipment. Scanning electron microscope analysis of the field samples showed that reduction of the cut-off diameters of the impactors resulted in lower contamination of the DPM samples with micron-sized particulates, in this case primarily mineral dust. The total mass concentrations of organic and elemental carbon in the samples were found to be inversely affected by the cut-off size of the orifices, indicating some potential losses of DPM in the impactors with smaller cut-offs.

1 INTRODUCTION

Due to widespread use of various types of diesel-powered vehicles in hard to cost-effectively ventilate confined spaces, underground miners around the world are exposed to relatively high concentrations of submicron aerosols and gases (Cohen et al. 2002, Pronk et al. 2009, Peters et al. 2017, Susanto et al. 2018, MSHA 2022). Long-term exposure to aerosols and gases emitted by "traditional" (Ruehl et al. 2015) diesel engines was found to have adverse effects on pulmonary (Kim et al. 2018, Du et al. 2020), vascular (Mills et al. 2005, Nejad et al. 2013), and other systems (Faherty et al. 2021). The International Agency on Research on Cancer (IARC) recognizes the risks of lung cancer and categorized diesel engine exhaust as a carcinogen to humans (group 1) (IARC 2012). Rapid development of engine, aftertreatment, fuel, and lubrication oil technologies over the past couple of decades resulted in "clean" engines that produce substantially lower particulate and gaseous emissions (Fiebig et al. 2014, Ruehl et al. 2015, Dallman and Menon 2016). The implementation of various control strategies and technologies such as replacement of older with advanced engines (Ruehl et al. 2015, Bugarski et al. 2020a), retrofitting older engines with exhaust aftertreatment technologies (Bugarski et al. 2009, Bugarski et al. 2020b), the use of alternative fuels (Bugarski et al. 2014, Bugarski et al. 2016), and replacement of diesel-powered vehicles with battery-powered (Halim et al. 2022) electric vehicles have resulted in a gradual reduction in the industry-wide average exposures of underground metal and nonmetal miners to elemental carbon (EC) and total carbon (TC) concentrations (Bugarski and Potts 2018, Bugarski

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et al. 2022, Gren et al. 2022). In addition, the exposure to exhaust from contemporary "clean" diesel engines equipped with advanced exhaust aftertreatment systems were shown to have less adverse health effects (McDonald et al. 2015). However, some occupations across the industry are still exposed to relatively high concentrations of "traditional" diesel aerosols (Bugarski and Potts 2018, Gren et al. 2022, MSHA 2022).

Monitoring ambient concentrations and personal exposures to diesel particulate matter (DPM) is complicated by the fact that DPM describes very complex ever-changing products of incomplete combustion of petroleum diesel and alternative fuels (Bugarski et al. 2014, Bugarski et al. 2016). This is further complicated in many situations by the use of multiple diesel engines of various generations and designs (Bugarski et al. 2020b) operated over a wide spectrum of conditions at various locations in underground mines (Rubeli et al. 2004, Saarikoski et al. 2018, Saarikoski et al. 2019, Bugarski et al. 2020c, Bugarski et al. 2022). In addition, sampling, analysis, and measurement of DPM is even further complicated by other factors including small particle size, the dynamic nature of the formation and transformation of DPM, and the interaction with other aerosols and environment (Bugarski et al. 2012, Giechaskiel et al. 2014).

The currently used methods for compliance monitoring of exposures of underground miners to diesel aerosols at permissible exposure levels (PELs) of 160 $\mu g_{TC}/m^3$ (71 Fed. Reg. 33387 2006, OHSA 2017) and 100 $\mu g_{EC}/m^3$ (AIOH 2013) are grounded in the use of the National Institute for Occupational Safety and Health (NIOSH) Method 5040 (Birch 2002, NIOSH 2003, NIOSH 2016). These offline methods are founded on: (1) collecting representative samples of diesel aerosols on quartz fiber filters (QFFs) using size-selective sampling, and (2) subsequent carbon analysis of those filters using a thermo-optical transmittance (TOT) method (NIOSH 2003).

The sampling apparatus currently used for compliance sampling incorporates two consecutive pre-selectors (MSHA 2020, SKC 2022). This apparatus was designed using parameters established during development of the personal diesel aerosol sampler by the U.S. Bureau of Mines (Cantrell and Rubow 1991, McCartney and Cantrell 1992): (1) a respirable cyclone operated at 1.7 lpm with a cut-off size of approximately 4.0 μm, which is used to eliminate the majority of coarse, mostly mechanically generated dust from respirable dust, and (2) a four-orifice sharp-cut impactor with a cut-off size of approximately 0.8 μm when operated at 1.7 lpm (McCartney and Cantrell 1992, Noll et al. 2005) incorporated with the cassette is used to eliminate the remaining dust from diesel aerosols. The design criteria for the impactors (Huang 2005) used with the personal diesel aerosol sampler (PDAS) (McCartney and Cantrell 1992) and consequently in the SKC Inc. DPM cassette (Noll et al. 2005) were established using the results of the size-selective aerosol measurements in four underground coal mines in the United States in the late 1980s (Cantrell and Rubow 1991). Those measurements showed that contemporary diesel engines contributed aerosols with a mass median diameter larger than 150 nm. The sharp-cut impactor with cut-off size of 0.8 ± 0.1 μm was found to provide acceptable separation of coal dust and diesel aerosols. The average loss of diesel aerosols was estimated to be 0.2% to 8.4%, and contamination with coarse aerosols was estimated to be 0.1% to 3.6% (Cantrell and Rubow 1991). It is important to note that the concentrations of diesel aerosol in various areas in those mines where diesel vehicles were used were relatively high (substantially above current PEL) and contributed a substantial fraction of respirable aerosol mass (Cantrell et al. 1992).

The DPM collected on QFFs is analyzed for EC and organic carbon (OC) using TOT analysis (NIOSH 2003). The results are used to determine total carbon (TC=EC+OC). The TOT analysis was chosen over the traditionally used gravimetric analysis for its sensitivity and selectivity for monitoring DPM at the Mine Safety and Health Administration (MSHA) targeted PEL levels (Ramachandran and Watts 2003). The method was originally developed for EC analysis and later adopted for TC analysis (MSHA 2016). EC was proposed as a more suitable surrogate than TC for DPM monitoring (Birch and Noll 2004) because EC is: (1) selective of combustion byproducts of diesel fuels, (2) not present in other submicron aerosols in underground mines such as cigarette smoke, wood smoke, and oil mist, and is (3) present in small quantities in coal and other carbonaceous dust. However, since TC makes up the majority of "traditional" DPM and includes important carcinogenic organic compounds, it was selected by the MSHA as a surrogate for monitoring exposures of underground miners to DPM (71 Fed. Reg. 33387 2006). The appropriateness of the methodology of using TC as a surrogate for assessing exposures of underground miners to DPM have been challenged previously (Cohen et al. 2002, Noll et al. 2015). The deficiencies in the precision and accuracy of this methodology were primarily attributed to a number of issues including: (1) potential presence of OC interferences in an underground mining environment (Cohen et al. 2002, Birch and Noll 2004, Noll et al. 2015), (2) adsorption of gas phase (non-DPM) OC on QFFs (Noll and Birch 2008, Bosch 2015), (3) contamination of QFFs in SKC DPM cassettes (Noll et al. 2019), and (4) losses of DPM to coarse aerosols (Gaillard et al. 2019). At present, MSHA does not require monitoring exposure to DPM in underground coal mines in the United States. Monitoring DPM exposures of underground metal, nonmetal, and coal mines in Australian states and Germany is performed using EC as a surrogate (AIOH 2013).

Since the presence of micron-sized particles in the DPM samples can interfere with TOT analysis, the effective removal of micron-sized aerosols using size-selective sampling is critical to monitoring DPM exposures in underground mining environments. The presence of coal dust that contains non-trivial amounts of OC (Yang and Yu 2002) and to some extent EC (Birch and Noll, 2004) can cause overestimation of OC and EC and errors in determination of the EC/OC split. If present in the DPM samples, alkali, alkaline-earth, and transition metal salts could interfere with TOT analysis by changing EC oxidation temperature and causing more charring (Wang et al. 2010). Carbonates present in rock dusting material could also interfere with the DPM measurement (Cavalli et al. 2010).

The evolution of diesel technology resulted in lower emission rates, decrease in size, and changes in chemical composition of emitted diesel aerosols. Measurement of size distributions of diesel aerosols in underground mines (Skubacz et al. 2017, Saarikoski et al. 2019, Salo et al. 2021, Bugarski et al. 2022) shows that the size distributions of diesel aerosols in many contemporary underground mines, particularly in the mines with a high penetration of advanced diesel technology, are characterized with smaller diameters from those used when designing the PDAS (McCartney and Cantrell 1992) and DPM cassette (SKC). In the late 1990s and early 2000s when EC and TC were selected as surrogates for monitoring DPM exposures of underground metal and nonmetal mines in the United States (71 Fed. Reg. 33387 2006, MSHA 2016), the contemporary diesel engines emitted DPM that was primarily made of EC and OC (Lowenthal et al. 1994, McDonald et al. 2003). Although EC and especially OC still contribute substantially to total particulate matter mass, the dominant constituents of particulate mass emitted by advanced diesel engines equipped with diesel particulate filter (DPF) systems are noncarbonaceous compounds such as nitrites, sulfates, and ammonium (Herner et al. 2011, Khalek et al. 2011, Khalek et al. 2015, Ruehl et al. 2015).

The changes in the DPM concentrations and physical properties necessitates improvements in the precision and accuracy of the methodology used for monitoring DPM using TC as a surrogate, at levels substantially below current MSHA PEL of 160 $\mu g_{TC}/m^3$. Although previously proposed interventions (Noll et al. 2005, Noll et al. 2015, Noll et al. 2019) resulted in measurable improvements, additional work is needed to minimize collection of micron-sized particles that can interfere with TOT analysis. This evaluation was conducted to evaluate the potential for an improvement in sampling methodology for the collection of diesel aerosols by reducing and eventually optimizing cut-off size for the impactor in the DPM sampling cassette.

2 METHODOLOGY

Three impactors with different sized orifices were evaluated. The reference impactor (I-061) was the one in the commercially available DPM cassettes (SKC 225-317). The additional two impactors were constructed by modifying impactor assemblies in the existing DPM cassettes to develop the sharp-cut impactors with lower cut-off sizes. In the modified assemblies, the original 0.61-mm synthetic sapphire orifice inserts (SSOIs) were replaced with 0.52-mm SSOIs (O'Keefe Controls Co., Monroe, CT, SAP-44-SA) to obtain I-052 and with 0.44-mm SSOIs (O'Keefe Controls Co., Monroe, CT, SAP-44-SA) to obtain I-044. For the sake of simplicity, the distance between nozzle and impaction plate was kept identical to the one in the existing DPM cassette.

The penetration efficiencies of the impactors were determined using polystyrene divinylbenzene (PS/DVB) beads with nominal diameters of 200, 300, 400, 500, 600, 700, 800, and 900 nm in aqueous suspension (Thermo Fisher Scientific, Fremont, California, Nanosphere™ Polymer Microspheres). The PS/DVB beads were aerosolized using a 3-Jet Collison nebulizer (CH

Technologies, Westwood, New Jersey). The normalized cumulative size distributions of aerosolized beads measured with the scanning mobility particle sizer (SMPS) with the electrostatic classifier (TSI, Shoreview, MN, Models 3080L) and ultrafine condensation particle counter (TSI, Shoreview, MN, Models 3776), and Aerodynamic Particle Sizer® (APS™) (TSI, Shoreview, MN, Model 3321) are shown in Figure 1.

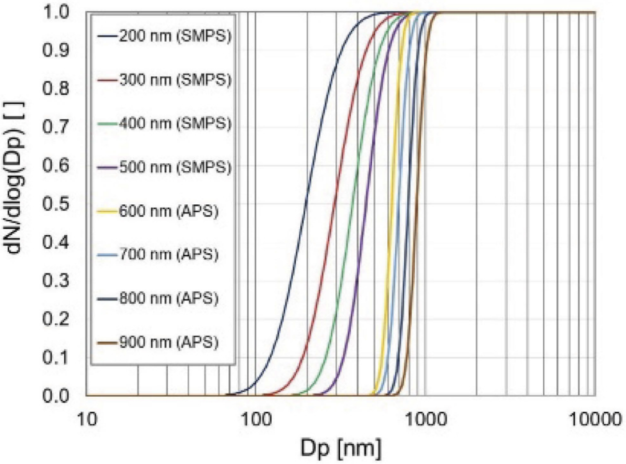
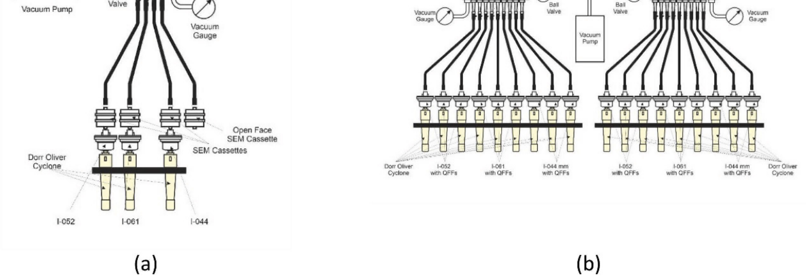


Figure 1. Normalized cumulative size distributions of aerosolized beads measured with SMPS and APS.

The impactors were field evaluated in the outby area of the underground coal mine during two shifts (S2 and S3) to assess: (a) performance of the I-061, I-052, and I-044 impactors with respect to penetration of micron-sized dust, and (b) differences in losses in diesel aerosols between those three impactors. This evaluation was executed as a part of the study described by Bugarski et al. (2022). The mine used "traditional" diesel engines in all categories of vehicles, retrofit type DPFs on the nonpermissible equipment, and filtration systems with disposable filter elements on permissible equipment. The aerosol sampling and measurements were performed at the continuous miner section. The size distribution of aerosols measured with the fast mobility particle sizer (FMPS, TSI Model 3091) during S2 and S3 were found to be bimodal with the agglomeration mode electrical mobility CMDs between 52 and 86 nm (Bugarski et al. 2022). By introducing the assumption that the density of diesel aerosols in the mine correlate with equations introduced by Maricq and Xu (2004) and Liu et al. (2009), the mass median diameters of those aerosols could be estimated to be between 123 and 172 nm.



(a)

(b)

Figure 2. Sampling apparatuses: (a) used to collect DPM for SEM analysis, and (b) used to collect DPM for carbon analysis.

The issue of penetration of micron-sized aerosols through the I-061, I-052, and I-044 impactors was investigated qualitatively on the results of scanning electron microscopy (SEM) analysis performed on the samples collected with the sampling apparatus shown in Figure 2a. Four different kinds of samples were collected concurrently using: (a) open-face 5-piece cassette, (b) 5-piece cassette preceded by cyclone and I-061, (c) 5-piece cassette preceded by cyclone and I-052, and (d) 5-piece cassette preceded by I-044. The samples were collected on the 25-mm, 0.2-µm track-etched polycarbonate membrane filters (Millipore Sigma, Whatman® Nuclepore™, Darstadt, Germany). The samples collected at a 600-second sampling time were used for analysis.

Twenty-one sections of each of the filters, one in the center and four groups of five radially distributed around the center at 90° spacing were analyzed by scanning electron microscopy (SEM) (Model S-4800 by Hitachi, Tokyo, Japan) using 10K magnification and energy dispersive x-ray spectroscopy using 20 to 70K magnification (by Bruker Quantax, Madison, Wisconsin). The particle imaging was performed at 20 kV, and elemental analysis was performed using 20-kV incident beam energy.

The issue of the differences in diesel aerosol (EC and OC) losses in the three evaluated impactors was studied using the results of the carbon analysis on sets of at least four filter samples of the same kind collected concurrently using the custom sampling apparatus shown in Figure 2b. The respirable cyclones (Zefon International, Ocala, FL, Zefon Nylon Dorr-Oliver Cyclone) were used ahead of each of the impactors. The DPM samples were collected on the primary and secondary quartz fiber filters (QFFs) (Millipore Sigma, Darmstadt, Germany, AQFA03700) housed inside of the DPM cassettes. To minimize OC contamination, the QFFs were removed from the impactors and preheated for two hours at a temperature of 800°C (Noll et al. 2019). The nominal sampling flow rates of 2.0 lpm were maintained using critical flow orifices installed in two common manifolds. A single vacuum pump (Oerlikon Leybold Vacuum, Cologne, Germany, Segovac SV25B) was used to draw all samples. All DPM samples were analyzed at the NIOSH Pittsburgh Mining Research Division using the NIOSH Method 5040 with modified temperature protocol. The analysis was performed using an OC/EC Aerosol Analyzer (Sunset Laboratory Inc., Portland, OR).

3 RESULTS

The penetration curves for the assessed I-061, I-052, and I-044 impactors are shown in Figure 3. The cut-off diameters (D_{50}) for those impactors were estimated to be 716, 466, and 258 nm, respectively. The geometric standard deviations (GSDs) were calculated (Marple et al. 2001) as 1.359, 1.582, and 2.151. The D_{50} and GSD for I-061 are similar to those assessed for the impactors in SKC DPM cassettes by Olson (2001) (746-770 nm and 1.391-1.420).

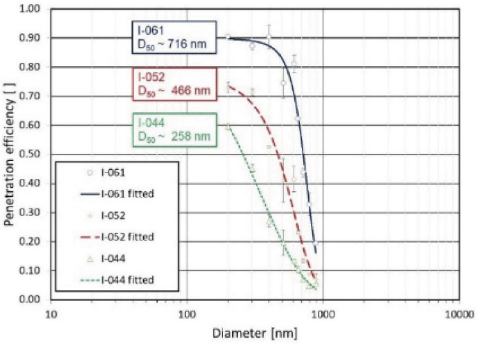
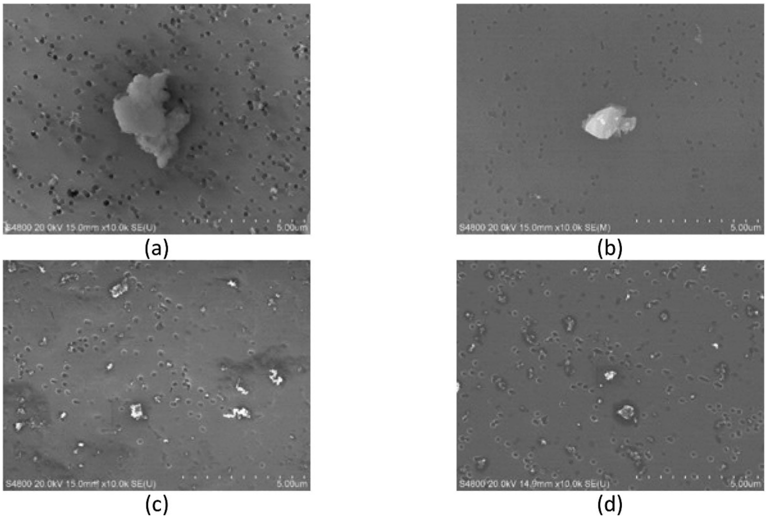


Figure 3. Penetration curves for the impactors.

Twenty-one images of each of the filters were examined for the presence of micron-sized particles. A total of twenty, eleven, seven, and three micron-sized particles were identified on the open face, I-061, I-052, and I-044, respectively. The SEM images of the filters collected as open face and with I-061, I-052, and I-044 impactors preceded with respirable cyclones are shown in Figure 4. Elemental analysis of the selected micron-sized particles found at the same locations showed that those particles were made primarily of the elements found in explosion suppression rock dust (Man and Teacoach 2009) that likely were aerosolized by movement of the diesel-powered equipment.

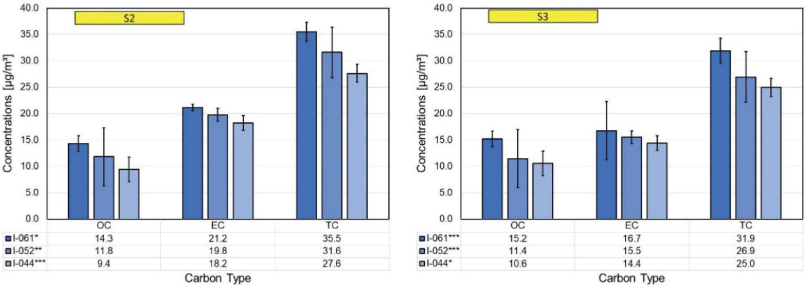
The results of carbon analysis performed on the samples collected with I-061, I-052, and I-044 impactors are shown in Figure 5. The mean concentrations of OC, EC, and TC were calculated using results for at least 4 samples of the same kind. Certain samples (Figure 5) were deemed outliers due to potential contamination during the manufacturing process and excluded from the calculations. The error bars represent one standard deviation of means for the specific sets of samples. The OC, EC, and TC concentrations between the impactors were compared using analysis of variance (ANOVA, OriginPro, OriginLab Corporation, Northampton, MA). All OC, EC, and TC concentrations were significantly different ($p<0.05$) between the impactors except OC concentrations in S2.



(a) (b) (c) (d)

Figure 4. SEM images for the samples collected: (a) without pre-selectors, (b) with cyclone/I-061, (c) with cyclone/I-052, and (d) with cyclone/I-044.

In the case of samples collected during S2, the OC, EC, and TC concentrations were 17.5%, 6.6%, and 11.0% lower for the samples collected with I-052 rather than I-061, and 34.3%, 14.1%, and 22.2% lower for the samples collected with I-044 rather than I-061. In the case of samples collected during S3, the OC, EC, and TC concentrations were 24.5%, 7.4%, and 15.6% lower for the samples collected with I-052 rather than I-061 and 30.4%, 13.9%, and 21.7% for the samples collected with I-044 rather than I-061.



(a) (b)

Figure 5. OC, EC, and TC concentrations measured during: (a) S2, and (b) S3 (Number of samples: * - four, ** - five, *** - six).

4 DISCUSSION

Reliable monitoring of exposures to TC, particularly the OC fraction of it, at the levels substantially below the current MSHA PEL of 160 $\mu g_{TC}/m^3$ is challenging due to earlier identified sampling and analytical issues. One possible improvement involves minimizing penetration of the micron-sized non-diesel aerosols that could potentially adversely affect the results of the TOT analysis (Yang and Yu 2002, Birch and Noll, 2004, Wang et al., 2010, Cavalli et al. 2010). We examined the potential of the SKC DPM cassettes with the modified impaction assemblies to eliminate micron-sized aerosols more effectively from DPM samples.

The results of SEM analysis performed on the limited set of samples collected in a contemporary coal mine showed that a substantial number of micron-sized aerosols present in the mine atmosphere penetrated the impactor currently used in the SKC DPM cassette (I-061, D_{50} = 716 nm). The I-052 (D_{50} = 466 nm) and particularly the I-044 (D_{50} = 258 nm) impactors were much more effective in preventing micron-sized aerosols from reaching the sampling filters.

The QFFs collected with I-061, I-052, and I-044 contained comparable amounts of EC and OC. The observed differences in EC and OC were at the level of limit of quantification (LOQ) for EC and OC of the NIOSH Method 5040. The losses in EC for I-052, 6.6 and 7.4% for S2 and S3, respectively, were found to be statistically significant, but still relatively minor. The losses in EC for I-044, 14.1 and 13.9% for S2 and S3, respectively, were somewhat more pronounced. The losses in OC for I-052, 17.5 and 24.5% for S2 and S3, respectively, and for I-044, 34.3 and 30.4% for S2 and S3, respectively, were found to be more substantial than those observed for EC. It is important to note that the uncertainty of OC measurements was substantially higher for the majority of samples than that of EC measurements.

This limited set of data indicate that due to relatively large losses in TC, the evaluated I-044 might not be suitable for DPMs sampling in the mines with a large presence of equipment powered by "traditional" diesel engines. Optimization of the preselector for specific applications would be necessary. Those efforts would require good understanding of the physical properties, particularly size distribution of diesel aerosols at the specific site. Unfortunately, this kind of data is currently available for a very few sites.

Additionally, the design of the impactor needs to be optimized. In the case of the I-044 impactor, the relatively large GSD of 2.151 indicates that the design, in particular sharpness of the impactor, can be potentially further improved by reducing the distance between nozzle and impaction plate.

5 CONCLUSION

The changes in concentrations and physical properties of diesel aerosols in contemporary underground mines necessitates improvements in precision and accuracy of the methodology used for sampling and analysis of DPM. This is particularly important if TC is used as a surrogate for monitoring exposure to DPM. The results of this evaluation showed the potential for improvements in the currently used size-selective sampling of diesel aerosols in underground mines. The SKC DPM cassettes with the modified impaction assemblies with lower cut-off sizes were shown to effectively eliminate micron-sized aerosols from DPM samples. The cut-off size of the impactor could be further optimized to minimize penetration of micro-sized aerosols and losses in EC or OC.

It is important to note that this evaluation was limited in scope and just proved the concept. Measurements were done over relatively limited period(s) of time in a single section of an underground coal mine that is using equipment powered by "traditional" diesel engines. Additional studies are needed to develop, optimize, and evaluate impactors for the specific applications, particularly in the underground mines with the substantial use of "clean" diesel technologies.

6 DISCLAIMER

The findings and conclusions in this manuscript are those of the authors and do not necessarily represent the official position of the National Institute for Occupational Safety and Health

(NIOSH), Centers for Disease Control and Prevention (CDC). Mention of company names or products does not constitute endorsement by NIOSH or CDC.

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