Evaluation of the SKC® DPM cassette for monitoring diesel particulate matter in coal mines†

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In a previous study, the efficacy of commercial and prototype impactors for sampling diesel particulate matter (DPM) in coal mines was investigated. Laboratory and field samples were collected on quartz-fiber filters and analyzed for organic and elemental carbon. Coal dust contributed a minimal amount of elemental carbon when commercial cascade impactors and prototype impactors, designed by the University of Minnesota (UMN) and the US Bureau of Mines (BOM), were used to collect submicrometer dust fractions. Other impactors were not as effective at excluding coal dust. The impactors evaluated in that study were either not commercially available or were multi-stage, expensive, and difficult to use for personal measurements. A commercial version of the BOM impactor, called the DPM Cassette, was recently introduced by SKC®. Tests were conducted to evaluate the performance of the DPM Cassette for measuring diesel-source elemental carbon in the presence of coal dust. Bituminous coals from three mines in two different coal provinces were examined. The dust particle diameters were small and the coal dust contained a high percentage of carbon, thereby giving a worst-case condition for non-anthracite coal mines. Results for the DPM Cassette were essentially identical to those obtained by the BOM impactors in a previous study. At a respirable coal dust concentration of 5.46 mg m^{-3}, which is 3.8 times the regulatory limit, the DPM Cassette collected only 34 μg m^{-3} of coal-source elemental carbon.

Introduction

Diesel exhaust has been linked to acute health effects [1–3] and is considered a potential human carcinogen or similar designation by several organizations.[4–6] Underground miners are exposed to the highest levels of diesel exhaust in the United States. The Mine Safety and Health Administration (MSHA) has promulgated rules to reduce the levels of diesel particulate matter (DPM) in underground coal and metal/non-metal mines.[7–9]

In metal/non-metal mines, the MSHA rule regulates the limit of personal exposure to DPM. DPM is measured by collecting air samples on quartz fiber filters and analyzing the filters for elemental carbon (EC) or total carbon (TC) by National Institute for Occupational Safety and Health (NIOSH) Method 5040.[7,8,10] To avoid overloading with mineral dust and any potential interference of graphitic ore, a single-stage impactor having a cut point (diameter of particle collected with 50% collection efficacy) of 0.8 μm at 1.7 L min^{-1} is used to minimize (eliminate) ore dust collection.[11–13] In underground coal mines, the MSHA rule does not regulate an exposure limit for DPM. A standard based on EC or TC was not considered practical because of coal dust interference.

Previously, the Bureau of Mines (BOM) developed a size-selective sampler (impactor) for determination of DPM mass concentrations in underground coal mines.[11] The impactor cut point of 0.8 μm at 1.7 L min^{-1}, which is based on particle size distributions in underground coal mines, was chosen to provide optimal separation between diesel and coal particles. Most diesel particles have aerodynamic diameters less than 1 μm, while most coal dust particles have aerodynamic diameters greater than 1 μm. Thus, a size-selective sampler can be used to separate most of the DPM from coal dust. Based on particle mass, the BOM sampler was 90% effective in the exclusion of coal dust, so about 10% of the dust was collected by the sample filter.

In a previous study,[14] we examined the potential contribution of coal-source EC. Commercial and prototype impactors, including the BOM impactor, were evaluated in field and laboratory studies. A commercial, multi-stage cascade impactor (Marple Series 290, Andersen Instruments, Inc., Smyrna, GA) having a cut point of 0.93 μm effectively excluded coal dust. In various locations of five non-dieselized coal mines, the EC levels were negligible. Results obtained in laboratory tests of the BOM impactors (cut point = 0.8 μm at 1.7 L min^{-1}) were similar to those obtained with the commercial impactor. When the respirable coal dust concentration was 5 mg m^{-3} (total dust = 12 mg m^{-3}), the EC concentration was 31 μg m^{-3}. Other size-selective samplers did not exclude coal dust as efficiently.

The previous study showed that DPM-EC could potentially be measured in the presence of coal dust by using a size selective sampler. However, the impactors evaluated in that study were either not commercially available or were multi-stage, expensive, and difficult to use for personal measurements. SKC®, Inc. (863 Valley View Road, Eighty-Four PA 15330 USA) recently introduced a single stage size selective sampler, called the DPM Cassette, based on the original BOM

† Disclaimer: mention of a company name or product does not constitute endorsement by the Centers for Disease Control and Prevention.

impactor design. NIOSH and MSHA have tested the performance of the DPM Cassette for sampling DPM in underground metal/non-metal mines. In this environment, the device effectively collected DPM and excluded ore dust.[15,16] Based on these and other results, MSHA uses a submicron impactor (*e.g.* the DPM Cassette) for DPM compliance sampling in metal/nonmetal mines.

In the study reported in this paper, we evaluated the performance of the DPM Cassette for monitoring DPM (as EC) in coal mines. We anticipated performance similar to the BOM impactors because the DPM Cassette design is based on this design. For our investigations, we first looked at characteristics of coal dusts in mines across the country to determine what coal types present the worst-case scenario relative to interference. After reviewing the available information, we selected a small diameter dust with high carbon content. Various quantities of coal dust and DPM were introduced into a laboratory chamber to produce a variety of dust and DPM concentrations. The collected samples were analyzed by NIOSH Method 5040 to determine whether coal dust poses a significant interference in the measurement of diesel-source EC and total carbon (TC). Following this initial evaluation, two other coals were also examined.

Methods

Coal dust selection

As mentioned above, properties of coal dusts from across the United States were first examined to determine what types would present the worst-case scenario. A coal dust with the finest particle size distribution and high carbon content would most likely pose the worst interference problem. Anthracite coals were not considered in this study because they are a very small portion of mining production in the United States and anthracite is not representative of other types of coal.[14]

We examined the mass median diameter (MMD) and the geometric standard deviation (GSD) of different coal dusts in mines across the country to determine what particle size distribution would include the highest fraction of fine particles. The MMD is the particle diameter at which 50% (by mass) of the particles are less than that value. Finer coal dusts have a smaller MMD. The GSD is a measure of the broadness of the size distribution. The larger the GSD is, the broader the distribution.

To describe the geographical location of a coal mine, a province location is sometimes given for that mine. For example, the eastern portion of the United States is considered the Eastern Province. The other provinces include: the middle or Interior Province, the mid-west or Rocky Mountain Province, the northern mid-west or Northern Great Plains Province, the Gulf Province (near the Gulf of Mexico), and the west or the Pacific Coast Province.

A literature review and analysis of information from a database on mines provided some MMDs and GSDs for mines located in various provinces. Rubow *et al.* reported the MMD of coal dust in two mines as 7.2 μm.[17] Organiscak and Page measured the particle size distribution for three bituminous coal mines from different provinces.[18] Two mines were located in the Eastern Province, and the other mine was located in the Rocky Mountain Province. The dust from one of the mines in the Eastern Province was 61% fixed carbon and had a MMD of 7.5 μm. Less than 10% of the particle mass was under 1 μm in aerodynamic diameter. Coal dust from the other mine in the Eastern Province was 65% fixed carbon and had a MMD of 16.2 μm, with less than 5% under 1 μm. The dust from the mine in the Rocky Mountain Province was 48% fixed carbon. The MMD was 27.8 μm, and less than 5% of the dust was under 1 μm.

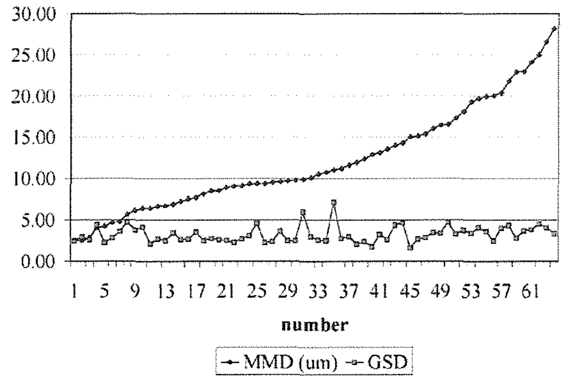

Fig. 1 The mass median diameter (MMD) and geometric standard deviation (GSD) for coal dusts at different locations in eleven mines. Data are from a NIOSH database.

Fig. 1 shows the MMDs and GSDs of coal dusts collected over several days in eleven mines across the country. Data are from a NIOSH Pittsburgh Research Laboratory database. Taking all the information into account, we selected the Keystone Mineral Black 325BA (Keystone Fillier and Mfg. Co., Muncy, PA) bituminous coal dust, which has a MMD of about 4.5 μm and a GSD of 3.3. By volume, about 10% of the particles have aerodynamic diameters less than 1 μm, and the coal dust is about 75% fixed carbon (information from specification sheet). The Keystone Black is a ground coal dust from a mine in the Eastern Province. It represents a worse case interference wise because it contains a high percentage of carbon (for a non-anthracite coal) and has a particle size distribution on the low end of the distribution range for coal mines across the United States. The smaller particle size presents a greater separation challenge to the DPM Cassettes, and the dust that does penetrate has a high, nonvolatile (fixed) carbon content.

Laboratory chamber experiments

Various concentrations (Table 1) of Keystone Black coal dust and DPM were sampled with DPM Cassettes and analyzed to determine how much EC is contributed by coal dust. An aerosol chamber,[15,19] capable of dispersing DPM and coal dust uniformly throughout its volume with a spatial variation of less than 5%, was used for these evaluations. For each target coal dust level, the following procedure was followed. As shown in Table 2, twelve DPM Cassettes and three respirable dust gravimetric samplers were placed inside the chamber. Coal dust was introduced to the chamber by a fluidized bed aerosol generator. A tapered element oscillating microbalance (TEOM 1400A Mass Measurement Systems, Rupprecht and Patashnick Co., Inc., Albany, NY), which measures the ambient particulate mass in real time, was used to monitor the coal dust concentration. Critical orifices were used to control the

Table 1 Targeted respirable dust and DPM-TC concentrations for each experiment

Experiment	Mine	Respirable dust/(mg m^{-3}	DPM-TC/μg m^{-3}
1	X	0.2	50, 400, 800
2	X	2	50, 200, 400, 800
3	X	5	50, 200, 400, 800
4	X	0.6	400
5	X	1.2	500
6	Y	2.9	100, 200, 400
7	Z	1.5	200
8	Z	1.3	200

Table 2 Steps used to collect coal dust, DPM, and mixed coal-DPM samples

Samplers	Set A: 3-respirable gravimetric		Set B: 3-SKC DPM Cassettes		Set C: 3-SKC DPM Cassettes		Set D: 3-SKC DPM Cassettes		Set E: 3-SKC DPM Cassettes		Set F: 3-SKC DPM Cassettes		Set H: 3-SKC DPM Cassettes		Set I: 3-SKC DPM Cassettes	
Status of pumps for sampler	on	off	on	off	on	off	on	off	on	off	on	off	on	off	on	off
Experimental steps																
Step 1: insert sets A through D in chamber																
Step 2: coal dust in chamber	x		x		x		x									
Step 3: collect about 0.2 mg[a] of respirable coal dust on filters		x		x		x		x								
Step 4: clear chamber, take out Sets A and B, insert sets E through I																
Step 5: DPM in chamber					x		x		x		x		x		x	
Step 6: collect about 200 µg[b] of DPM on filter						x		x	x		x		x		x	
Step 7: collect about 490 µg[c] of DPM on filter						x		x		x		x	x		x	
Step 8: collect about 975 µg[d] of DPM on filter						x		x		x		x		x		x
Step 9: clear and empty chamber																

[a] 0.2 mg of respirable coal dust (TEOM) is equivalent to collecting about 0.2 mg m^{-3} respirable coal dust for 8 h. [b] 200 µg of DPM (TEOM) is equivalent to collecting 167–200 µg m^{-3} DPM-TC for 8 h depending on TC content (usually 80–100%) in the DPM. [c] 490 µg of DPM (TEOM) is equivalent to collecting 408–510 µg m^{-3} DPM-TC for 8 h depending on TC content (usually 80–100%) in the DPM. [d] 975 µg of DPM (TEOM) is equivalent to collecting 812–1015 µg m^{-3} TC-DPM for 8 h depending on TC content (usually 80–100%) in the DPM.

flow rate through each of the samplers to 2.0 L min^{-1}. Respirable coal dust was sampled until the target equivalent 8-h time weighted average (8-h TWA) concentration was obtained using an estimation from the TEOM real time data. The 8-h TWA mass concentrations for coal and DPM samples were calculated using the following equation:

$$\mathrm{TWA}\ (\mu\mathrm{g\ m}^{-3}) \equiv \frac{m \times 1000}{q \times 480\,\mathrm{min}}$$

where m is the collected dust mass in µg, q is the sample flow rate in L min^{-1}, and 1000 is the conversion constant (1000 L per m^3 air).

After collection of the coal dust samples, the dust was cleared from the chamber. Three (forming a triplicate sample) of the twelve DPM Cassettes and all three respirable gravimetric samplers were then removed. Nine DPM Cassettes that sampled coal dust only remained in the chamber and were later used to collect DPM.

Nine unused DPM Cassettes were added to the chamber, and diesel exhaust from a Kubota engine with 80% load was introduced. 80% load was used since the DPM created by the engine with this load simulated the EC/TC ratios most common in underground metal/nonmetal mines.[8] The pumps connected to three of the DPM Cassettes that had sampled coal dust only and three unused DPM Cassettes were started. DPM was sampled until the DPM mass (monitored by the TEOM) on the filter corresponded to a DPM-TC concentration (8-h TWA) of about 50 µg m^{-3}. At this point, the pumps for these six DPM Cassettes were shut off. Pumps connected to three of the six remaining DPM Cassettes that had sampled only coal dust (but had not been turned on yet) and three unused DPM Cassettes were then turned on. The six DPM Cassettes collected DPM until the mass of DPM collected corresponded to a DPM-TC concentration (8-h TWA) of approximately 400 µg m^{-3}. The final six DPM cassettes (three exposed to coal and three unused) were used to collect DPM-TC concentrations (8-h TWA) of approximately 800 µg m^{-3}.

All DPM Cassettes were analyzed for TC and EC *via* NIOSH Method 5040. The quartz sample filters in the DPM Cassettes exposed to only coal dust were analyzed to determine the amount of coal dust TC and EC that had passed through the impactor to the filter. The DPM Cassettes that collected only DPM provided the TC and EC contributed by diesel exhaust, while those that collected both coal dust and DPM provided the TC and EC contributed jointly by coal and diesel. The difference between the carbon found with the DPM Cassettes that collected only DPM and those that collected both DPM and coal provided a measure of the coal-source TC and EC. The respirable dust samplers were used to determine (gravimetrically) the total respirable dust concentration.

This procedure of loading several DPM cassettes with coal dust and then using the loaded cassettes to sample different concentrations of DPM was repeated for respirable coal and DPM-TC concentrations shown in Table 1. Some samples were collected at a flow rate of 1.7 L min^{-1}, which is the flow rate at which the impactors were designed to operate. The two different flow rates (*i.e.*, 1.7 or 2 L min^{-1}) gave no significant differences in the impactor cut points (0.7 µm at 2 L min^{-1}, 0.8 µm at 1.7 L min^{-1}) or carbon results.

Through the experiments described above, we determined the amount of coal-source carbon collected by the DPM Cassette at different concentrations of respirable coal dust. The set of respirable coal dust concentrations generated provided a range around the current regulatory level for respirable coal dust (8-h TWA = 1.45 mg m^{-3}, or 2 mg m^{-3} as the MRE equivalent). (Note: an MRE equivalent is used by MSHA. It is calculated as 1.38 times the actual dust concentration found with a Dorr Oliver cyclone operated at 2 L min^{-1}. The calculated value is an estimate of the respirable concentration that would be found by a horizontal elutriator.) The DPM concentrations provided a range around the current DPM-TC regulatory level of 400 µg m^{-3} (8-h TWA).

To provide an indication of the potential interference posed by coals from different regions, two other coals, one from Mine Y and one from Mine Z, also were examined. To simulate a certain size distribution and to obtain a variety of coal dust concentrations, the bulk ores were collected at the mines and crushed in a laboratory apparatus at the Pennsylvania State University (see Table 1). Mine Z is in the Eastern Province, while Mine Y is in the Rocky Mountain Province. Coal, DPM and mixed coal-DPM samples were again collected in the laboratory chamber as described above.

The size distribution and carbon content of Mine Y coal dust were not known. The carbon content of Mine Z was also unknown. To determine the carbon content (TC and EC) of each dust, we collected respirable dust samples on quartz fiber filters along with respirable gravimetric samples on PVC filters when just coal dust was in the chamber. Carbon analyses (method 5040) were done on the quartz filters that collected the respirable dust. By knowing the respirable dust mass and the amount of carbon in that mass, the carbon content (TC and EC) of each coal dust was calculated. A Marple impactor also was operated in the dust chamber to determine the particle size distribution for Mine Y.

Carbon analysis

The carbon content of the sampled aerosols was determined by NIOSH Method 5040,[20] which employs a Sunset Laboratory OC-EC Carbon Analyzer (Sunset Laboratory, Inc., Forest Grove, OR), at the Pittsburgh Research Laboratory. The reported carbon concentrations are the average of the triplicate samples receiving simultaneous exposures.

Gravimetric measurement

Gravimetric samples, along with control filters, were desiccated and then equilibrated in a controlled environment (72 °F and 50% relative humidity) before weighing. Balance precision was better than 5 μg.

The airborne concentration of coal dust and DPM aerosol was calculated by dividing the mass gained by the filter (representing the collected mass) by the total volume of air sampled by the pump. The reported gravimetric results are the average of triplicate measurements.

Results and discussion

In a previous study, the DPM Cassette was shown to be very efficient at DPM collection.[15] In this work, we focused on the DPM Cassette's ability to exclude coal dust. The carbon in most coals is mainly organic carbon (OC), with the remainder being EC.[14] (During the analysis process, some of the OC in coal dust forms EC, which is a process commonly referred to as charring. Correction for char is accomplished in the NIOSH Method 5040 by monitoring laser transmission through the filter.) Thus, coal dust interference is expected to be greater with TC (TC = OC + EC) determinations than with EC.

Table 3 shows the EC and TC concentrations found by the DPM Cassettes at different concentrations of Mine X respirable dust. A significant amount of TC was found on the DPM Cassette filters, even for dust concentrations that were half the regulated level. Because adsorbed OC vapor can collect on the sample filter and positively bias the particulate TC result, the DPM Cassette actually contains two filters that are stacked. In theory, the second filter is also exposed to the OC vapor but

Table 3 EC from coal dust X on DPM cassettes

Respirable dust concentration/mg m^{-3}	Coal-EC collected on SKC DPM cassette/μg m^{-3}	Coal-TC collected on SKC DPM cassette/μg m^{-3}
0.21	4	17
0.6	17	65
1.27	22	115
2.45	22	83
5.46	34	125

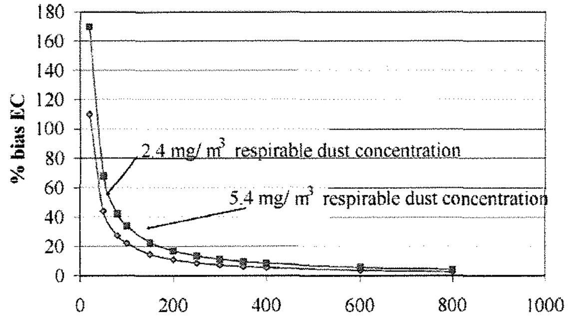

Fig. 2 Expected bias (%) in DPM-EC results due to submicrometer, coal-source EC. DPM Cassettes were used to sample respirable coal dust (Keystone Black) at two concentrations: 2.4 mg m^{-3} and 5.4 mg m^{-3}. The amount of EC collected on the DPM Cassette when exposed to each of the two dust concentrations was divided by a range of DPM-EC concentrations to estimate the error (%) that would be expected if the DPM Cassette is used to monitor DPM-EC in mines with high coal dust levels.

not the particulate carbon and can be used to correct the first filter for non-particulate OC. Thus, the TC results were corrected by subtracting the TC, which occurs as OC, found on the second quartz filter from the first filter TC value.

In contrast to TC, only about 34 μg m^{-3} (8-h TWA) of EC was found at a high coal dust concentration (8-h TWA = 5.46 mg m^{-3}, 3.8 times the regulated limit). These results are quite similar to those found with the BOM impactors in laboratory tests.[14] This level (34 μg m^{-3}) would only be a significant interference when measuring very low DPM-EC concentrations.

We can estimate the percent bias in the DPM-EC measurement at a given concentration (DPM-EC) through the following equation:

$$\text{Estimated bias (\%) for DPM EC} = \frac{\text{Coal EC}}{\text{DPM EC}} \times 100$$

where Coal EC is the coal-source EC concentration measured when the DPM Cassette was exposed only to coal dust at a certain concentration and DPM-EC is the DPM-EC concentration at which we want to estimate the percent bias. For example, if the amount of coal-source EC (22 μg m^{-3}) found after exposing the DPM Cassette to 2.4 mg m^{-3} of Keystone Black respirable coal dust is divided by a range of DPM-EC concentrations (20–800 μg m^{-3}) (Fig. 2), biases in the DPM-EC due to coal-source EC can be calculated.

As shown in Fig. 2, when measuring DPM-EC in the presence of respirable coal dust at concentrations over the regulatory limit for the worst case dust (Keystone Black) the bias does not become greater than 25% until DPM-EC concentrations are below 80 μg m^{-3}.

A similar observation was made when we looked at mixed coal-DPM samples. As described earlier, all DPM Cassettes were exposed to the same amount of DPM, but one set was also exposed to coal dust. The difference between the two sets should provide the amount of EC contributed by coal dust. For the mixed samples, the bias in the DPM-EC concentration was calculated as follows:

$$\text{Bias (\%)}EC_{DPM} = \frac{EC_{DPM/coal} - EC_{DPM}}{EC_{DPM}} \times 100$$

where $EC_{DPM/coal}$ is the average EC concentration for three DPM Cassettes exposed to both DPM and coal dust, and EC_{DPM} is the average of three DPM Cassettes exposed to DPM only.

As can be seen in Fig. 3, if the Keystone Black coal dust concentration was 5.46 mg m^{-3} (8-h TWA) or below, the bias introduced by the coal dust was less than 20% when EC_{DPM}

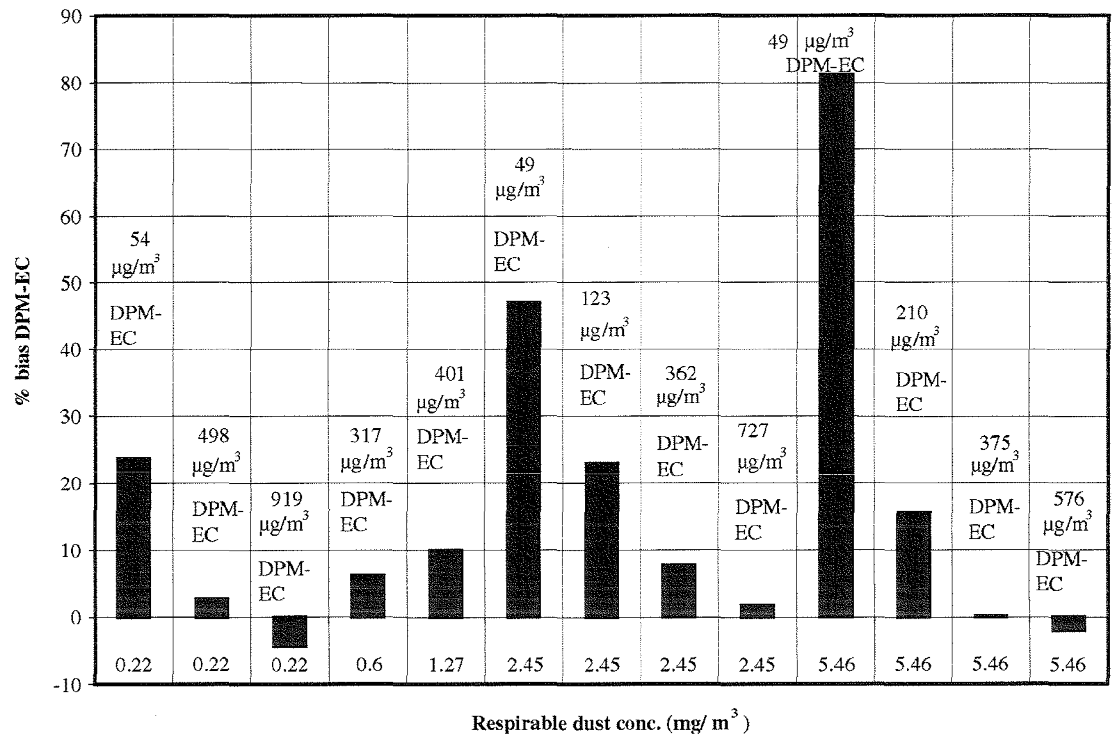

Fig. 3 Percent bias in the DPM-EC results for mixed coal-DPM samples collected with DPM Cassettes. Data were collected over a range of DPM and coal concentrations (see text for details). Coal dust was Keystone Black.

concentrations were greater than 200 μg m^{-3} (8-h TWA). When EC_{DPM} concentrations were above 100 μg m^{-3} (8-h TWA) in the presence of a respirable coal dust (Keystone Black) level of 2.45 mg m^{-3}, about 1.6 times the regulatory limit, the bias was less than 25%. At this dust level, the Keystone Black coal dust was a significant contributor to the $EC_{DPM/coal}$ concentration when the EC_{DPM} concentration was about 50 μg m^{-3} (8h-TWA). Thus, in the absence of an independent measure of the DPM-EC, measurement of such a mixture (50 μg m^{-3} DPM-EC and 2.45 mg m^{-3} respirable Keystone coal dust) would falsely attribute the EC from coal as DPM-EC.

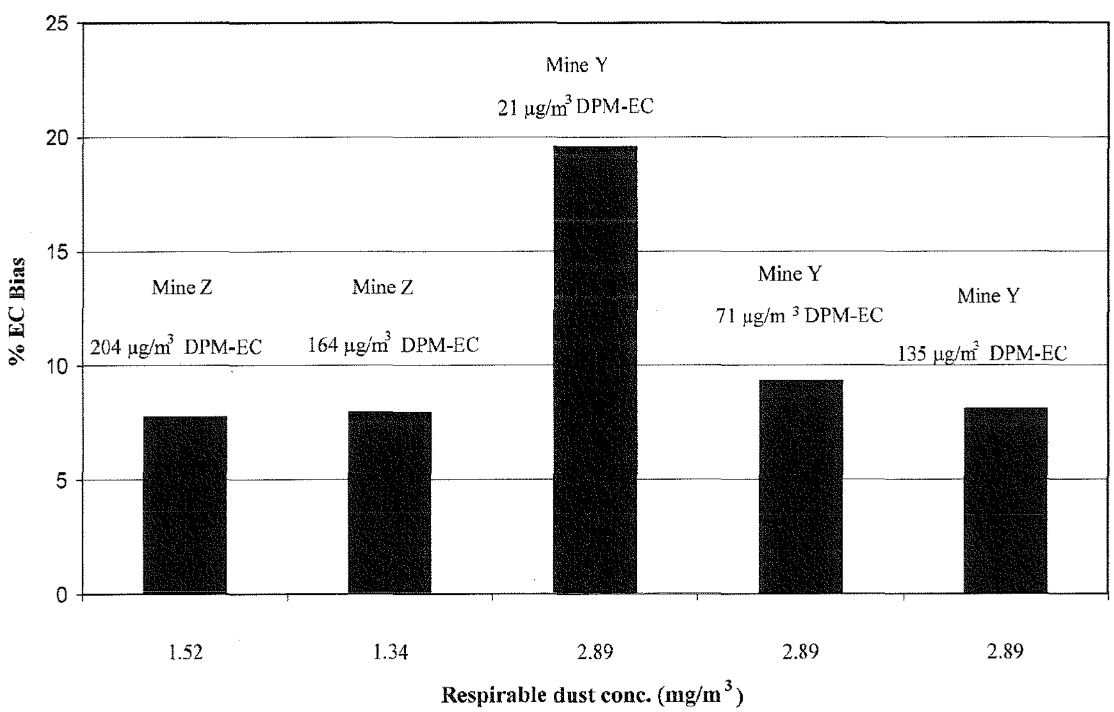

Fig. 4 Percent bias in the DPM-EC results for mixed coal-DPM samples collected with the DPM Cassettes. Coal dusts were from Mines Y and Z (see text for details).

The Keystone Black coal dust is abnormally fine and contains a high percentage of carbon (MMD 4.5 μm and fixed carbon of 75%) and thus represents an industry worst case. Other dusts with lower carbon contents (and larger particles) are expected to pose less interference. When looking at two different dusts from Mines Y and Z, we saw only a very small EC contribution. Mine Y dust size was small (MMD around 4 μm), but it had lower carbon content (44% TC, 1% EC) than Keystone Black dust. Mine Z had a MMD of about 5 μm and TC and EC contents of about 70% and 15–20%, respectively.

Bias in the DPM-EC results for the DPM Cassettes that collected DPM plus Mine Y and Mine Z dusts are shown in Fig. 4. Neither coal dust from the two mines contributed significantly to the EC results at the concentrations of DPM and coal dust tested (at and above the regulatory limit). Dust from Mine Y did not contribute even at high dust concentrations (2 times the regulatory limit) and very low DPM concentrations. With a Mine Y respirable coal dust concentration of 2.89 mg m^{-3}, the DPM-EC bias was less than 20% for a DPM-EC concentration of 21 μg m^{-3}. At the same coal dust level, the bias was less than 10% for DPM-EC concentrations of 71 μg m^{-3} and 135 μg m^{-3}. With an average concentration of coal dust from Mine Z of about 1.40 mg m^{-3} (8-h TWA) (close to the regulatory limit) and DPM-EC levels near 200 μg m^{-3} (8-h TWA), no significant contribution of EC from Mine Z coal dust was observed.

Conclusion

When monitoring DPM in the presence of coal dust, the DPM Cassette behaved similarly to commercial and UMN/BOM prototype impactors evaluated previously. In laboratory tests, it prevented most of the coal dust from collecting on the filter, even at high dust concentrations. The EC contributed by coal dust was minor except when relatively low DPM concentrations were sampled in the presence of a relatively high concentration of a very fine coal dust having high EC content. This is not representative of most coal seams. Limited testing with more representative coal dusts indicates that lower concentrations of DPM could be more accurately measured at coal dust concentrations near the regulatory limit. More laboratory and field data are needed to definitively determine a minimum DPM-EC concentration that can be measured without significant interference of coal dust.

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