In Mine Comparison of Diesel Particulate Sampling Methods

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In an effort to quantify diesel particulate exposures in the mining industry, various organizations have developed diesel particulate sampling instruments. These instruments work on a variety of different principles. These principles include the use of size-selective sampling utilizing inertial impaction, the measurement of respirable combustible dust (RCD), and the measurement of elemental carbon. The RCD methods provide measurements of whole diesel particulate. The size-selective and elemental carbon methods provide a measurement of a fraction of the diesel particulate matter. The size-selective and RCD methods are limited by gravimetric analysis. The elemental carbon method is limited by filter loading. A series of tests were conducted to compare the measurements obtained with the various diesel particulate sampling instruments and analytical methods. The tests were conducted at two underground mining facilities, a zinc mine and a potash mine. Similar tests are scheduled to be conducted in coal and other dieselized metal mines. A portable test chamber containing 30 sample ports was utilized for these tests. The sample chamber inlet was equipped with two cyclones that matched the American Conference of Governmental Industrial Hygienists respirable dust criteria at air flows up to 30 L/min. The three different sampling devices were attached to the chamber in groups of ten for each test. The sample pumps utilized for the size-selective and the RCD sampling devices were calibrated and operated at 1.7 L/min. The flow rate of the sample pumps utilized with the elemental carbon filters was varied from 1.0 to 1.7 L/min depending on the test location to reduce overloading of the filter. This article provides a description of the test procedures and a preliminary summary of the results. For each type of measurement, the average for each sample type is compared and a coefficient of variation is calculated. Haney, R.A.; Fields, K.G.: In Mine Comparison of Diesel Particulate Sampling Methods. Appl. Occup. Environ. Hyg. 11(7):717–720; 1996.

In 1992 the Mine Safety and Health Administration (MSHA) issued an advance notice of proposed rule making on diesel particulate exposure.[1] As part of this notice, information was requested on diesel particulate sampling methodology.

Several sampling and analytical methods have been proposed to measure diesel particulate matter. These methods include the use of size sampling using inertial impaction devices,[2–5] the measurement of respirable combustible dust (RCD),[6,7] and the measurement of elemental carbon.[8]

Size-selective sampling was initially developed to measure diesel particulate levels in coal mines. The chemical analyses that were commonly used (i.e., benzene solubles and combustion) could not differentiate between coal dust and diesel particulate. Studies have shown that over 90 percent of diesel particulate is less than 0.8 μm in aerodynamic diameter and that less than 10 percent of respirable coal mine dust is less than 0.8 μm in diameter. The lower limit of use for both impactors and the RCD determination is limited by gravimetric capabilities.

The elemental carbon method was developed to provide a sensitive analytical technique. While the elemental carbon method of analysis can detect levels of 0.001 mg (1 μg) of particulate, the method may be limited by an upper limit, approximately 0.3 mg on a 37-mm filter.

A previous study[9] compared the results obtained from the two size-selective sampling devices. This study showed that there was no difference between the results obtained from these two impaction samplers. In this study measurements were obtained in several underground coal mines. Groups of five of each sampler were placed side by side in a mine entry. Complete sampling systems were operated during the same time period. However, because of the side-by-side sampling locations, spatial variability may not have been considered. Additionally, because of the number of samplers, only differences between samplers of greater than 0.25 mg/m^3 could be resolved.

In an effort to compare the three sampling methods (impaction, RCD, and elemental carbon), MSHA has conducted a series of tests in underground mines using diesel equipment. This article provides a description of the test procedures and a preliminary summary of the results. For each type of measurement, the average for each sample type is compared and a coefficient of variation is calculated. The following studies are part of an ongoing program being conducted by MSHA to compare diesel particulate sampling methods in underground mines. Future tests will be conducted in coal and metal dieselized mines. A correlation between these studies and future studies will be conducted before a final conclusion is reached.

Description of Mines

Sampling for this study was conducted in two underground metal and nonmetal mines. These mines included a single-level potash mine in New Mexico and a multilevel zinc mine in New York.

The potash facility was an underground mine in which a 6-ft (1.82-m) thick horizontal ore body is mined by a room-and-pillar mining method using continuous mining equipment. The mine floor and walls were dry. There were nine active working sections at the time of this study. Four of these working sections employed diesel-powered face haulage equipment, and the remaining five employed an electric con-

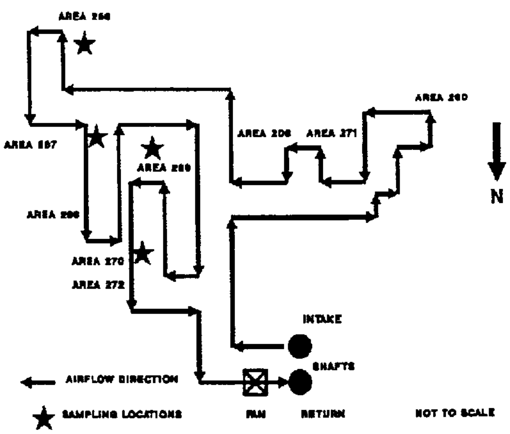

FIGURE 1. Mine schematic and sample locations.

tinuous haulage system. Figure 1 shows a schematic of the mine ventilation and the areas sampled.

Maintenance and supply equipment was also diesel powered. Engine horsepower ranged from 50 to 150 brake horsepower. The mine was reportedly using low sulfur, off-highway (red dye) diesel fuel.[10]

The zinc mine was developed by following ore concentrations. The actual mining process consists of fracturing the ore with explosives and loading the ore out of the headings with diesel-powered equipment. Figure 2 shows a schematic of a mine heading.

At the time of this survey, three headings were being developed and production was sporadic. The mine floor and walls were wet. Active production occurred at only two headings during the survey. Headings were approximately 19 ft high and 17 ft wide. The front-end loader operator would load an idling truck and then drive the truck to the surface while the front-end loader idled. After dumping the ore on the surface, the truck would be driven back to the heading and the cycle would be repeated. Front-end loader and haulage truck engines had horsepower ratings of 230 and 277, respectively. Vehicles were equipped with oxidation catalytic converters (OCCs). Mine personnel indicated that, with the exception of one truck, all vehicles were operated using low sulfur fuel. Low sulfur fuel is required to avoid damage to OCC devices.

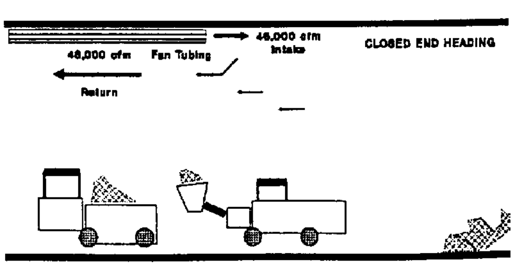

FIGURE 2. Schematic of heading.

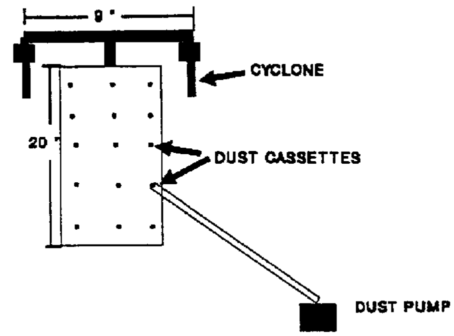

FIGURE 3. Sample chamber.

Instrumentation and Test Procedures

To conduct sampling that would account for spacial variability, a multiport sampling chamber was developed by MSHA. The sample chamber provides a common aerosol inlet to 30 sampling ports. The chamber inlet was equipped with two cyclones that would approximate the American Conference of Governmental Industrial Hygienists respirable dust criteria at an air flow of 25 L/min. Figure 3 shows a schematic of the 30-port sample chamber used to assess the diesel particulate sampling methodologies.

Tests conducted in a laboratory dust chamber have shown that the sample chamber provides a uniform distribution of dust (±1%) throughout the sampling chamber. Thirty ports provided for three groups with ten samplers in each group. This allows for differences of 0.1 mg/m^3 to be resolved between samplers. For each type of measurement, the average for each sample type was calculated. The standard deviation and coefficient of variation were also calculated.

Diesel particulate samples were collected by three different methods. These methods included RCD samplers, submicron impactors, and elemental carbon samplers. The flow through the RCDs and impactors was induced by pumps calibrated and operated at 1.7 L/min. The flow through the elemental carbon samplers was induced by pumps which were calibrated and operated at two flows: 1.0 and 1.7 L/min. Size-selective and RCD samples were analyzed gravimetrically, with preweighing and postweighing to ±0.001 mg. Elemental carbon samples were analyzed by the National Institute for Occupational Safety and Health method[8] at Sunset Laboratories, Forest Grove, Oregon.

The RCD samples were collected on 37-mm silver membrane filters with a 0.8-μm pore size. The silver membrane filters were preconditioned by baking in a muffle furnace at 400°C for 1 hour. After the dust samples were collected, the silver membrane filters were weighed and then again heated to a temperature of 400°C for a minimum of 1 hour. After baking, the silver membrane filters were reweighed. All weighings were to ±0.001 mg. The weight loss was considered diesel particulate. This method provides a measure of whole diesel particulate.

The impactors utilized were designed by MSHA. The impactor is a dichotomous sampler which permits both diesel particulate and respirable dust to be measured. The impactor is

TABLE 1. Results of Tests Comparing Various Diesel Particulate Measuring Methods

	Impactor Diesel Concentration (mg/m³)	RCD Diesel Concentration (mg/m³)	Elemental Carbon Concentration (mg/m³)	Elemental Carbon/Impactor Diesel Ratio (%)
Zinc mine, test 1				
Average	0.22	0.19	0.14	65.19
SD	0.02	0.06	0.01	
CV	6.85	30.01	5.04	
Samples	10	10	10	
Zinc mine, test 2				
Average	0.44	0.36	0.26	59.83
SD	0.03	0.03	0.01	
CV	6.68	9.35	3.92	
Samples	10	10	9	
Potash mine, test 3				
Average	0.43	0.43	0.15	34.79
SD	0.07	0.08	0.01	
CV	15.71	19.25	4.13	
Samples	10	8	10	
Potash mine, test 4				
Average	1.10	1.23	0.45	42.01
SD	0.16	0.31	0.05	
CV	14.97	24.95	10.21	
Samples	8	9	10	
Potash mine, test 5				
Average	1.21	1.48	0.42	35.23
SD	0.11	0.41	0.04	
CV	9.31	27.98	10.02	
Samples	9	9	10	

SD = standard deviation; CV = coefficient of variation.

an expanded version of the MSA respirable dust cassette. A 3/8-inch spacer was inserted between the MSA plastic cassette pieces to support the impaction plate. The MSA cassette's inlet is fitted with a machined brass insert which has a 1.0-mm diameter nozzle (No. 61 drill). Dust passes through this opening and is separated according to its aerodynamic size. At a flow rate of 1.7 L/min, dust particles greater than 0.9 μm (supermicron) impact on a greased stainless steel plate located 3.0 mm from the outlet of the nozzle. Dust particles less than 0.9 μm (submicron) do not impact on the plate and are collected on a 37-mm FWS-B polyvinylchloride filter. The lower limit of detection is limited by the gravimetric analysis.

Elemental carbon samples were collected on preconditioned quartz fiber filters. The silver membrane filters were preconditioned by baking in a muffle furnace at 400°C for 1 hour. After the samples were collected, they were sent to Sunset Laboratories for analysis. The elemental carbon method provides a measurement of a fraction of the diesel particulate. An estimate could be made of the whole diesel particulate by summing the elemental carbon and the organic carbon. However, due to sample handling and packaging, organic carbon values obtained from sample analysis were considered unreliable. Because the elemental carbon analysis was reportedly limited by loading, for tests 4 and 5 the induced air flow across the filters was reduced from 1.7 to 1.0 L/min to increase the allowable sampling time.

To account for different flow rates, samples were compared on a concentration basis. Concentrations of diesel particulate levels were calculated by the following formula:

$$\text{concentration (time-weighted average)} = \frac{\text{diesel mass (mg)} \times 1000\ (\text{L/m}^3)}{1.7\ \text{L/min} \times \text{time (min)}}$$

A total of five tests were conducted: two at the zinc mine and three at the potash mine. One of the diesel particulate sampling devices and a sampling pump were connected to each of the sample ports.

The sampling devices included combinations of 10 RCD samplers, 10 elemental carbon samplers, and 10 size-selective samplers. The chamber with the sampling devices was located in the haulage areas. Sample time for the chamber varied for each test to compare samples at different weights. Concentrations were calculated and comparisons were made utilizing actual sampling times.

Results and Discussion

Results of chamber sampling indicate that the average diesel particulate measurements from the impactor and the RCD measurements varied from 0.19 to 0.44 mg/m³ for tests conducted at the zinc mine (tests 1 and 2). For tests conducted at the potash mine (tests 3 through 5), the average diesel particulate concentrations varied from 0.43 to 1.48 mg/m³. Table 1 shows the diesel particulate measurement comparisons made using the sample chamber. The coefficients of variation for impactor measurements of diesel particulate ranged from 6 to 15 percent for the five tests. The coefficients of variation for RCD diesel particulate measurements ranged from 9 to 30 percent for the five tests. The coefficients of variation for elemental carbon diesel particulate measurements ranged from 4 to 10 percent for the five tests.

A two-sided *t*-test was used to make a statistical comparison for each test to determine if the average diesel particulate measurements made with the impactors and the RCD filters differed significantly. The comparison was made on the assumption that the variability between each of the impactors was unknown but approximately the same as the variability between the RCD filters. The level of significance of the test was chosen as 0.05. The degrees of freedom were determined by the total sample size (which varies from test to test because of voided samples) of each test minus 2. This comparison indicated that there was no significant difference between the average diesel particulate measurements made with the impactors and the RCD filters for tests 1, 3, 4, and 5.

The average ratio of elemental carbon diesel particulate concentrations to impactor diesel particulate concentrations was 60 and 65 percent, respectively, for the tests conducted in the zinc mine. The average ratio of elemental carbon diesel particulate concentrations to impactor diesel particulate concentrations was 35, 42, and 35 percent, respectively, for the tests conducted in the potash mine.

These results indicate a potential inconsistency in the ability to use the elemental carbon method to estimate the diesel particulate concentrations comparable to the other sample methods. Additionally, because the impactor is designed to collect only the aerosol less than 0.8 μm, it is unclear why there was agreement between the impactor and RCD determinations. Samples should also be processed so that organic and elemental carbon can be summed up for comparison with RCD and impactor measurements.

Samples collected in the potash mine at 1.0 and 1.7 L/min had similar elemental carbon to whole particulate (the total of organic and elemental carbon) ratios. As a result, it would appear that sampling at 1.0 L/min is a viable method to extend the sampling time for elemental carbon measurements.

As a result of these preliminary tests, MSHA will conduct additional studies to examine the inconsistencies between the elemental carbon fraction and the other diesel particulate measurements. These studies will be conducted in mines with a variety of types and usages of diesel equipment. Differences in diesel particulate generation due to variations in engine size, engine type, duty cycle, fuel, and the use of OCC devices will be compared using elemental carbon, RCD, and submicron impactor samplers.

Summary

1. There was no significant difference between the diesel particulate measurement with impactor and RCD samplers.
2. For average diesel particulate concentrations less than 0.50 mg/m^3, each of the sampling methods had standard deviations less than 0.10 mg/m^3.
3. The elemental carbon to whole diesel particulate ratio ranged from approximately 40 percent at a potash mine to approximately 62 percent at a zinc mine.
4. Sampling at 1.0 L/min appears to be a viable method to extend the sampling time for elemental carbon measurements.
5. Additional studies should be conducted to further evaluate the variability in elemental carbon to whole diesel particulate ratio.

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