Diesel Emissions Evaluation Program DEEP

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# **Diesel Particulate Matter Sampling Methods: Statistical Comparison**

### About the Study

Three sampling methods are used in mines to determine the concentration of diesel particulate matter (DPM): the respirable combustible dust method (RCD), the size selective method (SS), and the elemental carbon method (EC). This study used a statistical analysis to compare these methods and to determine the limitations and potential applications of each. Experimental data for the analysis was provided by three underground DPM sampling projects conducted in Canadian mines in 1996-1997 by various research teams.

The study was conducted by the University of Minnesota, Center for Diesel Research. The final report, submitted to DEEP in November 2000, is authored by Winthrop F. Watts.

### Background: DPM Sampling Methods

DPM has a mass median diameter of 0.2  $\mu$ m, with 90% of the particles less than 1.0  $\mu$ m in diameter. It is composed primarily of elemental carbon (EC), organic carbon (OC) in the form of adsorbed and condensed hydrocarbons, and sulfates. The proportion of OC to EC varies depending upon a number of factors, such as fuel, engine type, duty cycle, and more.

Three methods are routinely used in mines to quantify the DPM exposure.

### Respirable Combustible Dust (RCD)

In the RCD method, a personal sampling pump passes the gas through a cyclone, which acts as a respirable dust pre-classifier with a cut point of 4.0  $\mu$ m (i.e., removes large particles above 4  $\mu$ m in diameter). The respirable dust (RD) is collected from the sample after the cyclone using a silver membrane or pre-combusted, fiberglass filter. The RD is determined gravimetrically by weighing the filter before and after the sample is collected. The RCD is the amount of material removed (burned off)

from the silver membrane by controlled combustion.

RCD is composed of all combustible materials collected on a filter including EC, the soluble organic fraction of DPM, but also other combustible material such as drill oil mist or carbon-based material found in the ore dust. Thus, only a portion of RCD is attributable to diesel exhaust aerosol. Due to the presence of carbon in coal dust, this method is not suitable for coal mines.

### Size Selective Sampling (SS)

The SS method is based on a body of literature which suggests that the difference in the aerodynamic diameter of particles can be used to separate diesel aerosol from non-combustion aerosols in the collecting process; it is assumed that particles with diameters below  $0.8 \mu m$  originate from diesel emissions. This method was originally developed for coal mine use.

In the SS sampler, a personal sampling pump draws air through a cyclone followed by an inertial impactor with a 0.8 µm cut point. Large particles hit and adhere to an oiled aluminum surface in the impactor and are thus separated from the sample. DPM, which is mostly smaller than 0.8 µm, passes through without impacting and is collected on a polyvinyl chloride filter mounted in an MSA filter cassette (or a precombusted fiberglass filter if the EC method is to be used). The amount of DPM is determined gravimetrically (i.e. by weighing it) from the MSA filter. The SS method also allows measurement of the respirable dust from the material collected on the MSA filter and the aluminum substrate.

### Elemental Carbon (EC)

Various laboratory techniques have been developed for the analysis of carbon-based aerosols and the determination of their EC portion. A thermal-optical method NIOSH 5040 is most commonly used in North America. This method also allows determination of the OC content in the analyzed DPM samples. Total carbon (TC) is the sum of EC + OC.

Samples for EC are collected with or without an inertial pre-selector to remove particles larger than 0.8  $\mu$ m that may interfere with analysis. The simplest sampling train consists of a cyclone followed by a quartz fiber filter. Alternatively, the 0.8  $\mu$ m size selective impactor described above is also included in the sampling train to eliminate large, non-diesel particles such as coal dust.

Since EC is a product of combustion and is composed of inert graphitic carbon, it is a specific marker of diesel exhaust in many occupational settings where other combustion aerosols are not present. However, the OC portion of the collected sample is subject to interferences from other organic aerosols not directly connected with diesel exhaust, such as drill oil mist, hydraulic fluids, coal dust, cigarette smoke and other organic materials.

## Summary of results

The correlation of the RCD, SS, and TC measurements with the EC method (chosen as a reference because of it has the least interferences and biases) is shown in Figure 1 (a), (b), and (c), respectively.

From these correlations, the study determined specificity, sensitivity, and the limit of detection (LOD) for all methods in reference to the EC method, as shown in the following table.

Method	Specificity, µg/m³	Sensitivity	LOD, µg/m³
RCD	$25.5 \pm 3.6$	2.059	10.56
SS	$96.6 \pm 1.04$	1.68	24.3
TC	$14.76 \pm 0.487$	1.588	4.6
EC	-	-	2.0

The *specificity*, determined as the intercept of the correlation lines, is a measure of interferences that affect each method. For instance, in the case of RCD shown in Figure 1 (a), the intercept of  $25.5 \ \mu g/m^3$  means that the RCD method will give this reading when the EC method reads zero, due to interfering substances that affect the RCD measurement but not the EC method.

The slopes of the correlation lines define the *sensitivity* of the methods in relation to the EC method. The higher the sensitivity, the smaller the change in DPM concentration that can be measured by a method at a specified confidence level.



Figure 1. Correlation of RCD, SS, and TC methods with EC

The most sensitive and specific marker of diesel exhaust is EC, but TC provides the best estimate of total DPM exposure because EC accounts for only about 50% of the total exposure. However, unless care is taken to correct the OC measurement for non-diesel sources and other errors, the TC measurements will be inflated.