THE EVALUATION OF THREE DIESEL PARTICULATE MATTER SAMPLING AND ANALYSIS METHODS AT A HIGH SULPHIDE ORE MINING OPERATION

Submitted to

The Diesel Emissions Evaluation Program (DEEP) Technical Committee

Prepared by Michel Grenier, Program Manager, Underground Mine Environment Research Program, Sudbury Laboratory, CANMET, Natural Resources Canada.

OBJECTIVE

The aim of this project is to evaluate three methods for measuring the exposure of mine workers to diesel particulate matter in a high-sulphide ore mining operation. The three methods compared will
be: a size selective method based on inertial separation (impaction), an ashing method, which yields the mass of Respirable Combustible Dust (RCD), and an elemental carbon analysis method.

There are two main goals for the proposed work; first, to study the impact of the presence of respirable sulphides on the various sampling methods. It is suspected that this type of dust could significantly affect the RCD method. The second overall goal would then be to compare and evaluate alternatives to the RCD method under varying ratios of mineral to diesel dust.

**COLLABORATORS**

CANMET will be the main delivery group for this study, which will be conducted in close cooperation with the management, labour and safety and health representatives at Noranda Mining and Exploration's Brunswick Mining Division as well as the Noranda Technology Centre. Inco Ltd., the University of Minnesota and NIOSH will also be involved to supply analytical support and expertise.

**IMPACT**

This research should help demonstrate whether or not the mineral dust components interfere with the RCD assessment in this type of ore body. It should in turn lead to solutions and/or alternatives in the selection of a method to measure diesel particulate matter. The work will demonstrate the limitations and advantages associated with the three methods which will be compared. This work will also transfer information and technology on diesel particulate matter sampling and analysis at a local level as well as to the DEEP consortium. Finally, it will also help build the diesel particulate matter exposure database at the Brunswick mine.

**BACKGROUND INFORMATION**

The health effects of diesel exhaust contaminants have become a very important issue in the recent past. Because of this, there is unanimous support for monitoring and lowering the exposure of mine workers to diesel particulate matter. Whereas sampling and analysing for most chemical substances can be fairly straightforward, the nature of diesel particulate matter, its chemical complexity and interfering substances found in the workplace make it a very difficult component to sample and
analyse. In certain instances, it is believed that some chemical and physical properties of the mineral components of the respirable dust found in the workplace can interfere with the analytical process.

One such substance is suspected to be sulphide components in the ore being mined. When these compounds are ashed during the RCD analytical process, the sulphides may oxidise to sulphates thereby causing the overall mass of the dust deposit to increase. This phenomenon then either masks or overwhelms the mass loss expected from the ashing of RCDs. In any case, this would have a serious impact on the results obtained and would cause worker exposure to be underestimated. Preliminary analysis by CANMET of samples collected on silver membrane filters at the host mine show that pyrite seems to be transformed to iron sulphate which could be the cause of the observed mass increases when ashing for the RCD determination.

Massive sulphide ore bodies are common in Canada, hence the importance of assessing the impact of the mineralogy on our ability to measure diesel particulate matter. Routine analysis in British Columbia, Manitoba and Ontario as well as comprehensive RCD sampling exercises in New Brunswick (1) have shown that some samples experience significant mass increases. The exact processes, which cause these mass increases, are not fully understood and the potential solutions are not obvious.

This study will make use of mineralogy assessment technology in order to identify the mineral components that interfere with the analytical process used in the RCD method in a massive sulphide ore mine. Furthermore, the RCD method will be compared with the size selective method and the elemental carbon method for diesel particulate matter measurement.

**SAMPLING AND ANALYSIS METHODOLOGY**

**Respirable Combustible Dust (RCD) Sampling Method  Diesel Particulate Matter**

Respirable combustible dust samples are obtained using the approach described by the ACGIH (2). A pre-separator unit called a 10-mm cyclone made from a nylon material removes the non-respirable portion of the airborne dust cloud. When dust and air are passed through this cyclone at a flow rate of 1.7 L/min, the non-respirable portion is removed, while the smaller respirable dust particles are entrained by the air stream to the filter.
This method for measuring the exposure of personnel to diesel particulate was developed for use in mines and is based on the principle of ashing or burning of the combustible, carbon based components of the respirable dust cloud, which is collected on a silver membrane filter. This approach yields the mass of respirable combustible dust and will be referred to as the RCD method. Methods, which are comparable in approach, have been used elsewhere in metal mines with similar results and accuracy. The method was designed to be simple and compatible with existing sampling procedures used for sampling respirable dust and respirable silica in the mining workplace.

The main disadvantage of the method is that oxidation of the mineral portion of the dust sample is thought to interfere with the analysis, to the point where significant overall mass increases may result. Development of the method was steered by the Canadian ad hoc Diesel Committee and developed at CANMET in a two-phase approach described elsewhere (3,4,5,6). The RCD samples will be analyzed at the CANMET laboratory in Sudbury.

Size Selective Sampling Method  Diesel Particulate Matter

This sampling technique and the associated apparatus were developed as a result of a cooperative effort between the USBM (7,8) and the University of Minnesota’s Particle Technology Laboratory (9). The sampler was developed using size selective sampling techniques to specifically measure the mass concentration of diesel particulate in underground coal mines. For simplicity, this sampling technique is referred to as the size selective (SS) method.

With the SS approach, the samples are obtained using equipment similar to the RCD method as sampling pumps and cyclone pre-separators are used. The respirable portion of the dust then passes through a single-stage impactor. The impactor jets and flow rate were designed and chosen to provide a cut size of 0.8 micron. This means that particles greater than 0.8 micron in size (coal dust portion) would, for the most part, be removed from the air stream by impaction on the substrate which follows the jets. Particles less than 0.8 micron in diameter will continue on to be captured by a filter which follows the impactor portion of the sampler. Weighing that filter yields the estimated mass of diesel particulate matter.

Like the RCD method, the SS approach is fairly simple and inexpensive. Whereas interference due to oxidation of the mineral portion would not be a problem, the exposure value could be affected by any type of airborne dust small enough in size to reach the final filter. For this study, the sampling material (filters and impaction discs) will be supplied by the University of Minnesota. Samples will be sent back to Minnesota for analysis.
Elemental Carbon Sampling Method  Diesel Particulate Matter

Since elemental carbon constitutes a large fraction of the diesel particulate mass, it can be used as a measure of diesel particulate emissions. This method allows quantification of organic and elemental carbon at low levels (typically down to 5 \( \mu g \)) (10). The analysis for this work will be performed at CANMET's Sudbury facility.

Filter Preparation and Sampling

The sampling train used for collecting the field sample consists of a 37-mm cassette preceded by a 10-mm nylon cyclone through which air is drawn at a flow rate of 1.7 L/min using a self-regulated (constant flow), calibrated sampling pump. The filtration media of choice is a 37-mm diameter quartz filter (99.97% efficiency based on a 0.3 \( \mu m \) D.O.P. aerosol test). The filters are preconditioned by placing them in a muffle furnace for 2 hours at a temperature of 800\(^\circ\)C. This ensures that there is no carbon contaminant present in the filter matrix. The filters are then placed in three section 37-mm plastic cassettes. A second pre-treated quartz filter is used as a support pad. This is an alternative to the standard cellulose support pad and it is used in order to prevent any possible carbon contamination from the support pad. Other supports such as stainless steel or Teflon can be used. The cassette is then sealed and the sample number is written on the cassette. Weighing of filters is not normally required.

Prior to sampling, the cassette/cyclone assemblies are fitted to the calibrated sampling pumps using plastic tubing. The sampling trains are then worn by workers or installed at suitable locations for area sampling. After sampling, cassettes are sealed and brought back to the laboratory for analysis.

At the lab, the cassettes are opened along with field and analytical blanks. It is not necessary to condition or acclimatize the filters since they are not normally weighed and any moisture present will have no effect on the analysis. For analysis, a 1.5 cm\(^2\) rectangular portion of the filter is extracted using a metal punch. This allows three individual sample analyses for each sample. In this approach, a homogenous filter deposit is assumed.
Instrument and Method Description

In the thermal-optical method, speciation of organic, carbonate and elemental carbon is accomplished through temperature and atmosphere control. A helium-neon (He-Ne) laser and a photodiode receptor are used to correct for any pyrolytically generated elemental carbon or char, which is formed during the analysis of some materials.

Once the analysis has commenced the initial laser transmittance is calculated. Organic and carbonate carbon are evolved in an inert, oxygen-free helium (He) atmosphere as temperature is raised in four incremental steps to 900°C. Evolved carbon is catalytically oxidized to carbon dioxide (CO₂) in a bed of granular manganese dioxide (MnO₂) at a temperature of 870°C. CO₂ is reduced to methane (CH₄) in a nickel/firebrick methanator at a temperature of 500°C. The CH₄ is subsequently quantified by a flame ionization detector (FID). Any char which is produced will reduce the laser transmittance through the sample and will be corrected for during the next phase of the analysis.

The oven temperature is reduced to 525°C, an oxygen-helium mixture (2% O₂, He balance) is introduced into the sample oven, and temperature is again increased in four steps to 900°C. As oxygen enters the oven, pyrolytically generated elemental carbon is oxidized and a concurrent increase in filter transmittance occurs. The point at which the filter transmittance reaches its initial value is defined as the split between elemental carbon and organic carbon. Carbon evolved prior to the split is considered organic carbon this includes any carbonate that may be present. Carbon volatilized after the split is considered elemental. As in the previous step CO₂ is reduced to methane (CH₄) and the CH₄ is subsequently quantified by the flame ionization detector (FID).

After all the carbon has been evolved from the sample and quantified by the FID, an internal calibration is achieved through the injection of a known volume of methane into the sample oven.

Carbon Analyzer Program

The carbon analyzer program was written using Microsoft Visual Basic and Microsoft Visual C++ and directly controls the operation of the Carbon Analyzer. Using parameters preset in the program, temperature and atmosphere conditions are precisely controlled. Data is collected and
written to a raw data file for later quantification. Real-time plotting provides easy monitoring of sample analysis, showing temperatures, pressures, laser transmittance and FID output.

Sample Analysis Results

Quantification of the raw data file is undertaken by the OC-EC (Organic Carbon-Elemental Carbon) Calculation program which was written using Microsoft Visual Basic. (The program currently in use is a modified version of the program received with the instrument). Analytical results are displayed in g/cm² for organic, elemental and total carbon for each sample.

An output file is created which can then be imported into a spreadsheet program. The analytical results are then converted into a more user-friendly format. For example the results are converted from g/cm² to g or mg per filter and if the sampling volume amounts are provided then mg/m³ are also calculated. Finally, the tabulated results are printed.

The major advantages of this method are, firstly, the fact that it promotes a more direct approach to assessing diesel particulate concentration. Secondly, it allows quantification of organic and elemental carbon at low levels (typically down to 5µg). Thirdly, it is less prone to interference by other combustible or mineral sources, thus enabling quantification of diesel particulates where the other methods may not be adequate.

Variable-pressure Scanning Electron Microscopy

Variable-pressure scanning electron microscopy (VP-SEM) will be used to characterize the sample's mineralogy. With standard SEM, a conductive coat needs to be applied to samples. The fact that the samples are collected on silver membrane filters could make this coating process very difficult. VP-SEM does not require this coating process in order to provide a quantitative mineralogical assessment.

The VP-SEM study for this project will use RCD dust samples from the selected locations. Half of the samples will have been ashed as per the RCD analytical procedure while the other will not be processed. The amount of sample mass collected in standard sampling of airborne respirable dust should be sufficient for the VP-SEM studies. Additional samples will be collected from ore found
in the area. This will hopefully provide more insight as to the relative proportions of the various types of minerals found in the ore and in the air.

The VP-SEM study will measure the proportions of sulphate and sulphide minerals present in the dust before ashing and those present after ashing. This will help identify the contributors to any observed mass fluctuations, which could interfere with the RCD sampling and analytical process. These samples will be analysed at CANMET's Mining and Mineral Sciences Laboratories facilities in Ottawa. Because the analysis will be performed directly on a silver membrane filter substrate, the analysis is expected to yield sulphide and sulphate proportions (percentages) to an accuracy of better than 10%.

OVERVIEW OF STUDY

The study will take place in Bathurst, New Brunswick during a two-week survey at Noranda's Brunswick Mine. The Brunswick Mining Division is a zinc-lead mine, which is part of a massive sulphide ore body the exact composition of which varies with depth and exact location. Two main sites were selected to evaluate the effect of sulphide ores, the 1125-2 sub-level and the 725-2 sub-level areas. Schematic plan views of the selected sites are shown in Figures 1 and 2 located in the appendix. The 1125-2 sub-level north, will be the site where side by side sampling will be conducted during actual production and ore handling operations. The ore profile in the 1125-2 sub-level area consists of 35 % Pb/Zn sulphide and ore 65% pyrite/pyrrhotite. Although this area is not historically one where significant RCD sampling problems have been observed, the ore composition is such that sulphide to sulphate conversion should produce a measurable impact on the RCD method. The intent is to sample for diesel particulate matter using the three methods described earlier and to use VP-SEM and gravimetry to determine the amount of sulphates on post-ashed silver membrane filters used in the RCD method.

At this point it is anticipated that an LHD vehicle will be used to transport ore from the 253-2 stope to a dump site over a duty cycle of less than 15 minutes. The exact type of vehicle, the engine make and model and the type of after-treatment device to be used have yet to be determined. The details of sampling, including sampler numbers, locations and replications will be given in the next section.
The second site is the 725-2 sub-level area where production is no longer taking place. This area has been selected in order to compare the three sampling methods under conditions where diesel particulate matter (dpm) forms the bulk of the airborne dust mass. This will yield valuable data on its own but will also provide a baseline for comparing the ratio of dpm as measured by RCD to EC. This ratio obtained under conditions of high diesel particulate matter will be used to compare with one measured at the 1125-2 sub-level and deviations will be correlated to sulphate mass on post-ashed filters. The vehicle used in the 725-2 sub-level area will be an LHD vehicle of the same type as tested in the 1125-2 area. At a minimum the vehicle will be equipped with the same model of engine and after-treatment device.

For both series of tests, the vehicles will be sent to the maintenance area at the end of the night shift to be fitted with a new intake filter and to be checked for engine or hydraulic leaks.

**SURVEY DETAILS**

The need to obtain sound side by side data for the three methods under comparison, and the requirement to identify problematic mineral dust are the critical aspects of this study. In order to address these points, the targeted areas will be sampled in locations where a substantial amount of mineral dust is expected (at the face/muckpile and along the haulage route) as well as on the vehicle itself. Except for work that will be performed on the 725 sub-level, sampling will be done during actual production cycles.

Adjusting the distance between samplers and the ore handling operation can vary the ratio of mineral dust to diesel particulate matter. Distance from the ore handling operation would cause the mineral dust to sediment thereby increasing the soot to mineral dust ratio. For the particular conditions of anticipated total airflow volumes and drift cross-section (in the 1125-2 sub-level area) standard sedimentation theory and field studies indicate that sampling stations located within a 500 ft. distance from the dust source should produce a good range of diesel to mineral dust ratios (11, 12, 13).

Wherever sampling is conducted, a minimum of three samples will be collected for each method (EC, SS and RCD). Additionally, three more samples will be collected on silver membrane filters
for the determination of mineral oil mist concentration. The analysis will be performed by Inco Ltd. at their Copper Cliff, Ontario, Central Process Technology Laboratory. Three additional samples will be collected on silver membrane filters to allow for VP-SEM to be performed on non-ashed filters. This will permit the determination of the sulphide/sulphate ratio prior to ashing.

Fifteen samplers will therefore be used at each of the sampling stations described earlier. Tests at both study sites will be repeated five times (5 sampling shifts). These data will then be used to perform standard analysis of variance, which will allow the selected methods to be inter-compared to a 95% level of confidence. Furthermore, the sulphide/sulphate data will be analysed and correlated to the ratio of EC to RCD observed both in the production area and the 725-2 sub-level area.

The samplers will be deployed at the sampling locations shown in Figures 1 and 2. At the fixed stations, the samplers will be placed across the drift in an array, which is compact enough to sample the central area of the cross-section. In places where through traffic has to be allowed (STN-1 and STN-I at 1125-2 sub-level), a swinging gate consisting of a hinged 2-inch pipe to the bottom of which a piece of 4-inch mesh screen is attached will be used. This gate will be opened and closed by the technologist in charge of this particular sampling bench (samplers, gas monitors, continuous dust monitors and cascade impactors). The mobile stations (STN-V) at both test sites will be fitted with samplers arranged in low profile metal wire baskets which will be attached to the vehicle.

The targeted workplaces will be examined during a site visit, which will take place ahead of the study. Changes and enhancements in the logistics of deployment will be more fully developed at that time.

Variables such as vehicle type, engine make and model, after-treatment device and fuel may affect the characteristics of airborne dust. These data will be recorded for completeness and could become useful at a later date.

Other measurements in the areas selected will include cascade impactor samples which will allow the determination of the airborne dust size distribution, airborne dust concentration as a function of time and the airborne concentration of main exhaust gases. A sample of the fuel will be taken back for analysis and local ventilation data will also be recorded.
TECHNOLOGY TRANSFER

The DEEP consortium is committed to education and the transfer of technology for the benefit of the mining industry. This commitment implies contact/feedback with and the active involvement of mine personnel including Joint Occupational Safety and Health people, local labour representatives, fleet maintenance and ventilation personnel as well as any other mine employee who may benefit from exposure and participation to this project. At the end of the project time will be set aside for a debriefing session/seminar where a round-table discussion may be used to focus on the participants views of the study and the anticipated impact. This forum can also be used to bring any technical point, which may need to be explained or further clarified about the study.
REFERENCES


American Conference of Governmental Industrial Hygienists: Threshold Limit Values for Chemical Substances and Physical Agents Biological Exposure Indices; ACGIH, 1996.


APPENDIX
**WORK SCHEDULE**

**PROJECT TITLE:** Diesel particulate matter sampling and analysis methods evaluation.

**PROJECT LEADER:** Michel Grenier, CANMET

**CLIENT NAME:** DEEP Consortium

**CLIENT CONTACT:** Bill Howell, DEEP Secretariat

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SURVEY COSTS

CANMET COSTS

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* Sub-contractors and CANMET will submit separate official proposal documentation to CAMIRO

IN-KIND CONTRIBUTION
Brunswick Mining and Noranda Technology Centre labour and travel for support to the tests as described in this proposal.

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Michel G. Grenier

Education

1980. Honours B.Sc., Physics (cum laude), Laurentian University, Sudbury, Ontario.
1982. M.Sc. Physics (cum laude), Laurentian University, Sudbury, Ontario. Research thesis on the
affinity of radon decay products to airborne aerosols in uranium mines.

Professional Affiliations

- American Industrial Hygiene Association (AIHA)
- American Conference of Governmental Industrial Hygienists (ACGIH)
- Occupational Hygiene Association of Ontario (OHAO)
  Canadian Institute of Mining and Metallurgy - Sudbury Chapter

Professional Committees

- Technical advisor to the ONRSA's Workplace Environment Committee.
- Member of the Canadian ad hoc Diesel Committee.

Experience

1996 to pres.: Manager of the Underground Mine Environment program which includes
diesel research conducted at Bells Corners in the Ottawa region and ventilation, sampling and
analytical research conducted at the Sudbury Laboratory.

1991 to 1996: Project leader with the U/G Environment and Ventilation research group
with CANMET/Natural Resources Canada. In charge of mining research dealing with dust and gas
contaminant monitoring and control including silica and diesel exhaust contaminants. Is also
working on workplace sampling programs and strategies and on workplace safety issues.

1985 to 1991: Underground environmental scientist with CANMET/Energy, mines and
Resources Canada, involved in dust control and sampling research.

1981 to 1985: Physical scientist with CANMET/Energy, Mines and resources Canada,
involved in radioactive contaminants sampling, analysis and control in uranium mines.

Publications

35 refereed papers and published conference proceedings. Over 100 divisional reports of investigation
dealing with safety and health related to the underground workplace environment.
Mahendra Gangal

Education

1963. B.Sc., Agra University.
1974 Ph.D., Calgary University, computer applications in channel flow problems.

Specialty

Dynamometer testing for diesel engine emissions, evaluation of after-treatment devices and fuels.
Assessment of dieselized mine air quality and development of monitoring instrumentation
Standards development/regulations

Experience

Experience: 20 years Research and Development in the field of underground mine environment.

Emissions testing of diesel engines and determination of ventilation prescription according to CAN/CSA M424.2 standard for engine certification
Technical consultation with Provincial regulators with respect to regulations on the use of diesel machines in underground mines
Development of an advanced multi-gas monitoring system and other instrumentation specialized for dieselized underground mines
Assessment of exhaust treatment devices and bio-diesel fuel
Extensive experience in the design of advanced monitoring systems and air quality assessments at many u/g mines
Development of RCD/soot sampling and analysis methodology

Publications

Extensive publications in the field of diesel contaminant characterization and control as well as health topics related to the underground workplace environment. Amongst these:


Development of a Diesel Vehicle operator CO2 Exposure Meter, M. Gangal and D. Dainty, Proc. 6th
US Mine Ventilation Symposium, Published by Society for Mining, Metallurgy, and Exploration, Inc. (SME), 1993.
Ernest Don Dainty, M.A.Sc., P.Eng.

Education

1965. Instrumentation Design Course, MIT.

Affiliations

Association of Professional Engineers of Ontario.
Canadian Institute of Mining and Metallurgy.

Experience

1991 - date: Part-time (semi-retirement), CANMET, Natural Resources Canada.
1962 - 1991: Full-time, CANMET, Natural Resources Canada. Health and Safety Research and Development Studies as summarized by the following:
1968 - date: Established and supervised the CEAL Diesel Emissions Facility (relevant to all underground workings) for:
certification of diesel machinery;
researching diesel engine emissions and developing equipment to reduce emissions toxicity.
1987 - 1990 Coordinated an industry\govt. sulfide Dust Explosion Control Group.
1962 - 1970 Improved gaseous explosion control (coal mines) by:
researching gas explosion fundamentals related to mining electrical equipment and diesel machines; and
developing a standard for explosive gas instrumentation testing.

Productivity:

Chaired three CSA Technical Committees resulting in Canadian National Standards:

1990 "Diesel Machines in Non-Coal Mines", CAN\CSA M 424.2
1990 "Diesel Machines Brake Performance", CAN\CSA M 424.3
1988 "Diesel Machines in Coal Mines", CAN\CSA M 424.1

Recognition:
APEO Engineering Medal for outstanding research in diesel engine emissions and development of equipment to reduce emissions toxicity, 1990.

- **Canadian Award for Business Excellence**, gold medal in the Environment Category, for the diesel exhaust filtration technology, 1990.