Title: Evaluation of diesel particulate matter sampling techniques

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ABSTRACT

The study evaluated diesel particulate matter (DPM) sampling methods used in the South African mining industry. The three-piece cassette *respirable*, *open face* and *stopper* sampling methods were compared with the SKC DPM cassette method to find a comparable DPM sampling method for the non-coal mining industry. Controlled surface and underground static (i.e. area) and personal sampling studies were conducted. Triplicate analysis was carried out on each sampled filter using the NIOSH 5040 method to obtain elemental carbon, organic carbon and total carbon values. The results of the three-piece cassette sampling methods compared well with the SKC method and it was concluded that any of the three methods could be used to determine the DPM exposure of mineworkers in terms of the elemental carbon marker. In terms of standardising the DPM sampling methods for non-coal mining industries, the respirable method had certain advantages.

[143 words]

Key words: diesel particulate matter, NIOSH 5040, SKC cassette, DPM sampling method

INTRODUCTION

Diesel particulate matter (DPM) is an airborne pollutant generated by diesel-powered engines that causes respiratory diseases and that has been linked to lung cancer. Diesel engine exhaust is a highly complex and variable mixture of toxic chemicals that mainly consists of carbon dioxide, carbon monoxide, nitrogen oxides, hydrocarbons (including polycyclic aromatic hydrocarbons [PAHs]), sulphur oxides (depending on the fuel's sulphur content) and solid materials (fine particles). Particulate matter (PM) is the visible emission in diesel exhaust that consists of liquid droplets and carbon particles or "soot"[1,2,3]. The carbon particles also adsorb organic compounds, sulphates, metals and other trace elements[1]. The organic fraction of diesel exhaust mainly consists of unburned fuel and oil. Compounds such as aldehydes and PAH are also contained in the organic fraction, which is of particular concern as many PAHs are known carcinogens[1,3].

The health concern for DPM exposure lies in that DPM is a sub-micron aerosol (mass median diameter of 0.2 µm) and 90% of the particles are smaller than 1 µm.

They are therefore inhaled into the deepest part of the lungs where oxygen enters the bloodstream and are then absorbed in the body and cause damage[2,4,5,6]. Elemental carbon (EC) particles increase the long-term retention of adsorbed genotoxins and other chemical toxins because the particles have a high affinity for them.

With the acknowledged health risk associated with exposure to DPM an occupational exposure limit (OEL) for DPM exposure in South Africa is to be established soon. However, at present there is no clear standardised sampling method for this particulate.

There are various methods used for sampling DPM in the South African mining industry. The most commonly used methods are the SKC DPM cassette and a three-piece cassette loaded with a 37 mm heat-treated quartz filter and support pad, with three different sampling configurations.

The various three-piece cassette methods have not been researched in terms of sampling validity and it is not known if results obtained from any of these methods are representative and/or accurate in terms of DPM exposures in South African mines.

The objective of this study was to evaluate the different DPM sampling techniques used in the South African mining industry and compare them with the SKC DPM method. The SKC DPM method is regarded as the industry standard reference method and was used in the study as a control.

METHODOLOGY

The selection of the four sampling methods was based on information gained from a DPM questionnaire sent to 32 mines in the South African mining industry.

SKC DPM cassette method

An SKC DPM cassette and a GS-1 single-inlet cyclone were held together with a filter cassette/cyclone holder accessory for this configuration (see Figure 1). The sampling head was attached to a Sensidyne Gillian air sampling pump (GilAir-3) by means of flexible tubing or a u-bend, depending on the sampling requirement, and the sampling pump was calibrated at a flow rate of 1.7 l/min for sampling.

NIOSH research to validate suitability and uniformity of DPM deposition on the filter with the SKC DPM cassette method, made this sampling method a useful control in the current study[8,10].

Figure 1: SKC DPM sampling method

Three-piece cassette methods

In the following three methods the sampling head consisted of a three-piece clear styrene cassette loaded with a 37 mm heat-treated quartz filter, a stainless steel ring which provided a filter deposition area of 8.04 cm^2 and a support pad. The sampling head was attached to a GilAir-3 with flexible tubing. The differences between the three-piece cassette sampling methods are described below.

Respirable method (i.e. configuration)

The bottom part of the three-piece cassette was removed and a non-corrosive size-selective cyclone was attached in its place. To prevent air leakages between the cyclone and the filter cassette, insulation tape was used to seal off the connection (see Figure 2). The sampling pump was calibrated at 2.2 l/min, matching the specification from the cyclone supplier.

Figure 2: Respirable DPM sampling method

Open face method

For this method the bottom or inlet part of the three-piece cassette was also removed, leaving the cassette “face” open for sampling directly onto the filter (see Figure 3). Sampling was conducted at 1.7 l/min for this method, based on information obtained from the users within the mining industry.

Figure 3: Open face DPM sampling method

Stopper method

The three-piece cassette is protected from the environment with plastic stoppers (i.e. plugs) at the inlet and outlet of each cassette. The inlet stopper was removed with the *stopper* method, revealing only the inlet opening through which sampling took place (see Figure 4). The sampling pump was calibrated at 1.7 l/min, based on information obtained from users within the mining industry.

Figure 4: Stopper DPM sampling method

Sampling studies

Three different studies were conducted in which each of the above-mentioned sampling methods or configurations were used.

Controlled study

The controlled study was performed on surface in a diesel locomotive service shed. A locomotive with a 12-cylinder CAT diesel engine was pulled into the shed, started and performed under load (approximately 1 800 rpm) for an hour while sampling was conducted. All the doors and possible entrances to the shed were closed off to minimise ventilation during sampling and to keep the environmental conditions constant.

Ten sampling pumps, consisting of four SKC DPM sampling trains and two of each of the three-piece cassette sampling trains, were assembled in a wooden box. The wooden box with the pumps was lifted to approximately 10 metres above the locomotive towards the middle of the shed with a crane, where it remained for the duration of the sampling period. The study was repeated three times on three consecutive days.

Field study

A study under actual underground mining conditions was conducted to confirm the results obtained during the controlled study on surface. Static and personal sampling were repeated for four day shifts over four consecutive days.

Static sampling (i.e. area sampling)

Area sampling was conducted in an underground trackless section of a platinum mine at a tipping area. Each one of the four different sampling methods was assembled and placed as static samplers at the tipping area for the duration of the shift which lasted approximately eight hours. Activities that occurred within the area and environmental conditions during sampling were recorded.

Personal sampling

Personal sampling was conducted in the same section where the static sampling took place. Four workers that worked in this area were each supplied with a sampling train with one of the DPM sampling methods. The occupations sampled were an utility vehicle (UV) operator, a load haul dump (LHD) operator, a drill rig operator and a rock breaker operator (tip attendant).

Analytical procedures

Triplicate analysis was conducted on each sampled filter according to NIOSH 5040 method. The EC, OC and TC values were determined and converted to milligram (mg) by taking the filter deposition area into account.

Statistical analysis

The DPM results in milligrams (mg) were statistically analysed. In order to test whether the different types of filters showed differences in variability, as measured with the statistical measures of standard deviation or variance, Levene's test was used. Levene’s test is applicable to a relatively small number of samples and when no assumption about its underlying distribution can be made[9].

The percentage relative standard deviation (RSD%) was calculated on the basis of the standard deviation of the triplicate results per filter divided by the average of the three results.

RESULTS

The distribution of EC, OC and TC values for the controlled study are given in Figure 5. Under the controlled conditions on surface there were no statistically significant differences between the EC values for the *respirable*, *open face* and *stopper* methods when compared to the EC values from the SKC method ($p > 0.05$). However, the variance among the OC and TC values for the three methods showed significant differences when compared to the SKC method.

The average EC values (mg DPM) for the *open face* and *stopper* methods compared very well with the EC values of the SKC method in that their average values were within the NIOSH acceptance criterion range of ± 25% of the average SKC value. However, the average value for the *respirable* method was 35% higher than the SKC average. The difference in the *respirable* method could be related to the performance of the cyclone and/or the higher flow rate that was used compared to the other sampling methods.

The average relative standard deviation (RSD) for the EC values of the SKC method was 5.1%, which is similar to what was found by NIOSH. The RSDs for the EC values of the other methods were: *respirable* (8.1%), *open face* (5.5%) and *stopper* (10.1%). The average RSDs for the OC and TC values were higher than 5% for the *open face* and *stopper* methods and were below 5% for the *respirable* method. These values are summarised in Table 1.

Table 1: Summary of measurements for the controlled study (surface)

	Summary measure	SKC	Respirable	Open face	Stopper
Organic Carbon	Average (mg)	0.362	0.677	0.523	0.436
	Standard deviation	0.371	0.619	0.646	0.398
	Average RSD%	2.9	2.3	14.4	8.2
Elemental Carbon	Average (mg)	0.131	0.179	0.132	0.148
	Standard deviation	0.172	0.199	0.169	0.182
	Average RSD%	5.1	8.1	5.5	10.1
Total Carbon	Average (mg)	0.494	0.856	0.655	0.584
	Standard deviation	0.540	0.810	0.812	0.568
	Average RSD%	2.2	1.2	10.8	6.0

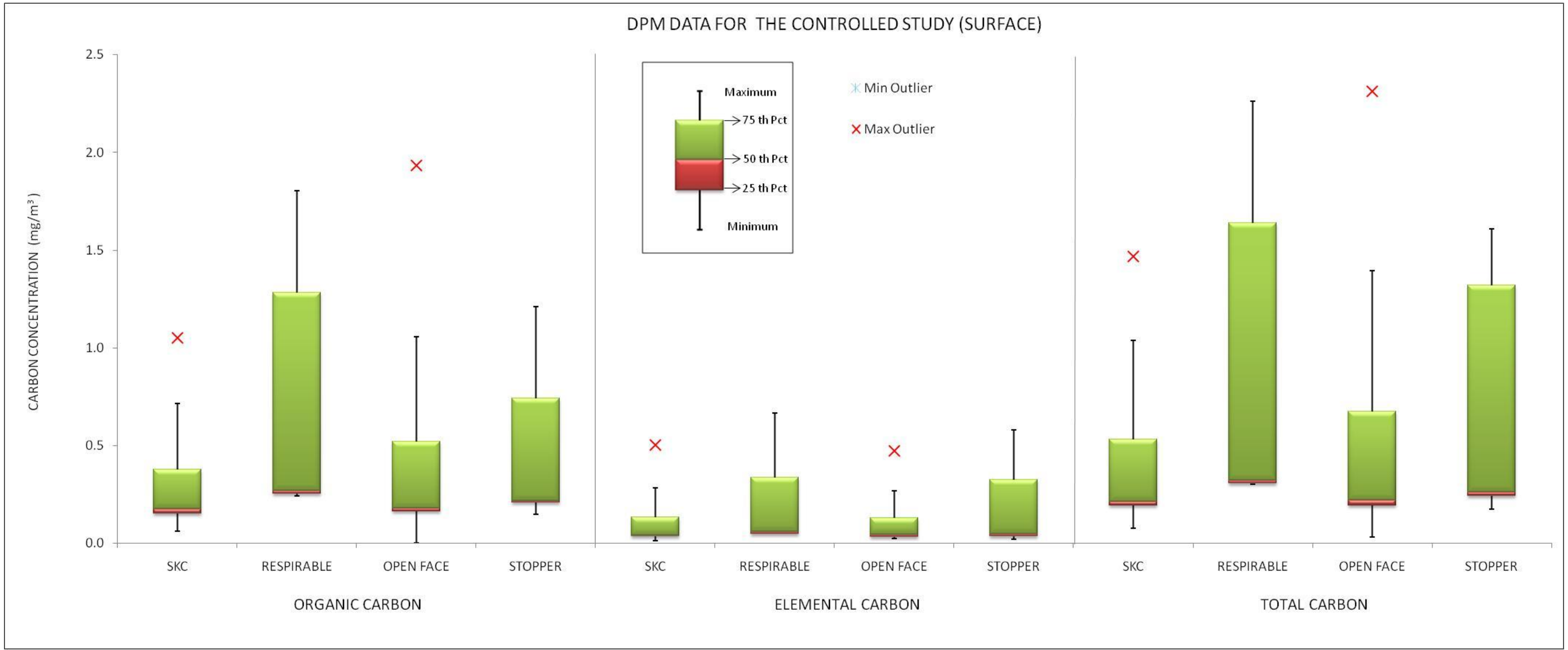

Figure 5: DPM data for the controlled study on surface showing the OC, EC and TC values

The distribution of the results from the underground static (i.e. area) sampling is shown in Figure 6.

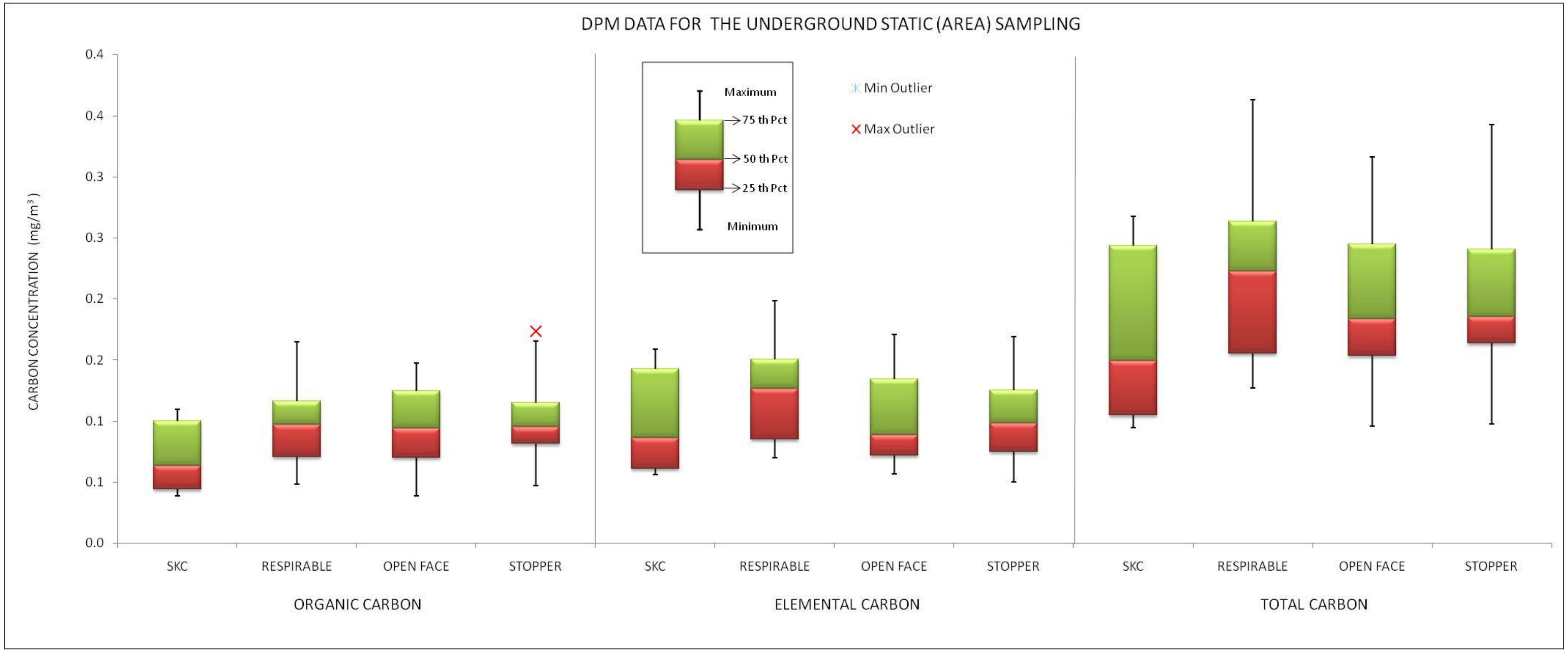

Figure 6: DPM data for the underground static (i.e. area) sampling showing the OC, EC and TC values

The results from the underground, static (i.e. area) DPM measurements showed that the average EC values for the *open face* and *stopper* methods were within the NIOSH criterion range of 25%. The average EC values from the *respirable* method were just outside of this range (+28%). The RSD for the EC values for each method was: SKC 4.1%, *respirable* 8.7%, *open face* 5.6% and *stopper* 2.9%. Similar to the controlled study on surface, the RSDs for OC and TC values were higher than 5% for the three-piece cassette methods, apart from the SKC method. The values are summarised in Table 2.

Table 2: Summary of measurements for the underground (static) DPM sample

	Summary measure	**SKC**	**Respirable**	**Open face**	**Stopper**
Organic Carbon	Average (mg)	0.070	0.100	0.095	0.102
	Standard deviation	0.030	0.038	0.039	0.033
	Average RSD%	2.6	10.0	10.2	17.1
Elemental Carbon	Average (mg)	0.099	0.127	0.105	0.103
	Standard deviation	0.044	0.044	0.044	0.043
	Average RSD%	4.1	8.7	5.6	2.9
Total Carbon	Average (mg)	0.169	0.227	0.200	0.204
	Standard deviation	0.073	0.082	0.080	0.073
	Average RSD%	3.4	8.0	7.0	9.7

Levene's test revealed no statistically significant differences among the variances associated with the *respirable*, *open face* or *stopper* methods when compared to the SKC method. All the p-values for the EC, OC and TC values were greater than 0.05 (average $p = 0.67$).

There was an initial concern that variations in measurements on the same filter (i.e. differences in triplicate measures) could influence the results of the Levene test. For this reason the values obtained from the triplicate measures were averaged per filter, and the Levene test was carried out again on these "averaged" values. The variance obtained from the "averaged" values differed slightly from that calculated on all data values. However, the significance patterns did not change.

The distribution of the underground personal sampling results is illustrated in Figure 7.

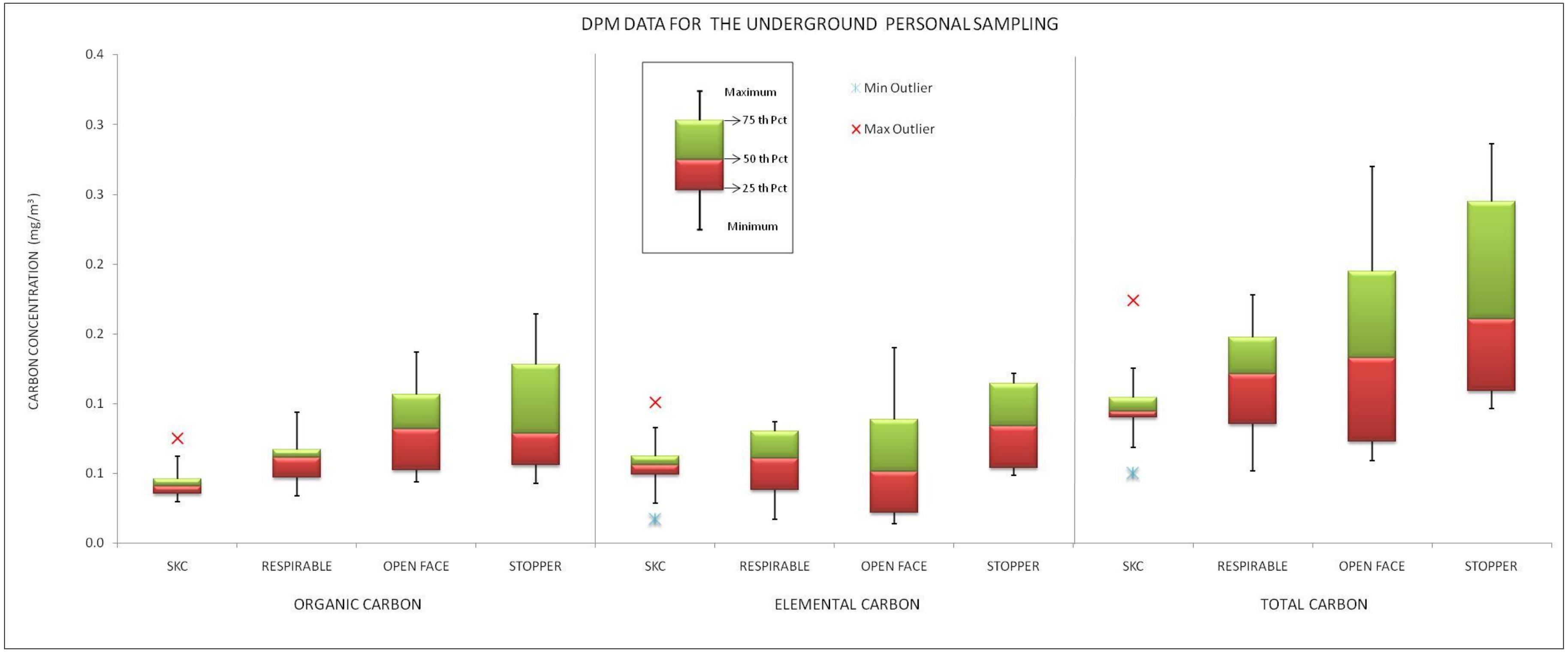

Figure 7: DPM data for the underground personal sampling showing the OC, EC and TC values

For the underground personal sampling the average EC values (mg DPM) of the *respirable* and *open face* methods were within 25% of the average SKC value. The average EC value for the *stopper* method was 54% higher than the SKC method.

The average RSDs for the EC values for all the sampling methods were below 5%. As with the controlled study on surface and the underground static sampling, the average RSDs for the OC and TC values were in general, higher than 5%. These values are summarised in Table 3.

Table 3: Summary of measurements for the underground personal DPM sample

	Summary measure	**SKC**	**Respirable**	**Open face**	**Stopper**
Organic Carbon	Average (mg)	0.046	0.060	0.083	0.091
	Standard deviation	0.015	0.019	0.034	0.043
	Average RSD%	6.8	7.8	10.5	19.8
Elemental Carbon	Average (mg)	0.055	0.056	0.062	0.084
	Standard deviation	0.029	0.028	0.049	0.033
	Average RSD%	4.0	3.6	4.1	3.1
Total Carbon	Average (mg)	0.101	0.116	0.145	0.175
	Standard deviation	0.043	0.045	0.082	0.074
	Average RSD%	3.5	4.9	7.4	10.4

The Levene test showed that there were no statistically significant differences between the variances of the TC, OC or EC values of the *respirable* method when compared to the SKC method (p-values were greater than 0.4).

When the variances of the TC, OC and EC values of the *open face* and *stopper* methods were compared to those of the SKC method, statistically significant differences could be found ($p < 0.05$).

Given that the EC value is the marker for DPM exposure measurements, it was interesting to note that the *respirable* and *stopper* methods showed no significant differences in variances when compared to the SKC method for underground personal sampling. This finding confirmed the result obtained in the controlled study on surface. The *open face* method showed a statistically significant difference in variance compared to the SKC method, but this difference was no longer significant when tested on the "averaged" data.

DISCUSSION

The three-piece cassette sampling methods compared very well to the SKC method. Under the controlled surface and the underground static conditions it was found that there were no statistically significant differences between the variances of the EC values associated with the three methods and the SKC method. For the personal sampling, no statistically significant differences could be found among the variances of the EC values for the *respirable* and *stopper* methods (at the 95% confidence level). For the *open face* method, a statistically significant difference in variance was found when compared to the SKC method, but this difference was not significant when the test was repeated on the "averaged" values.

OC and TC values do not originate solely from the diesel exhaust and are in many cases subject to interference from other sources of organic carbon[1,4,5]. As expected, there was more variance in the OC and TC values of the three methods when compared to the SKC method and the p-values did not show a consistent significance pattern.

The results obtained from this study indicate that any of the three methods can be used to determine the DPM exposure of mine workers in terms of the marker, EC. There are, however, a number of practical considerations to keep in mind:

- The *open face* sampling method lends itself to tampering, which could have a negative impact on the DPM exposure result;
- The *open face* and *stopper* sampling methods allow for the deposition of particles that are larger than ten micron; and
- The deposition of DPM on the filter is subject to the performance of the size-selective cyclone when using the *respirable* sampling method.

CONCLUSIONS AND RECOMMENDATIONS

The study showed that any of the three-piece cassette methods can be used to determine the DPM exposure of mine workers in terms of the EC marker, with values that should compare very well if the SKC method is used under the same conditions.

In order to standardise the DPM sampling methods for non-coal mining industries, it is recommended that the *respirable* method be used for DPM exposure measurements. The *respirable* method allows for the sampling of the respirable fraction of airborne dust of which DPM forms a part and the filter is protected against tampering. This method can be used on surface or underground, for personal or static sampling and should give comparable results to the SKC method.

ACKNOWLEDGEMENTS

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LESSONS LEARNED

1. The EC values from the three-piece cassette DPM methods show no significant differences when compared to the SKC cassette method.
2. The *respirable* method is recommended as an alternative method for use in the non-coal industry.

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