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Preliminary field study to evaluate two thermal correction methods for removing VOC interference from DPM sample analysis

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ABSTRACT: Diesel particulate matter (DPM) is the solid portion of diesel exhaust. In underground mines prone to high DPM concentrations for prolonged time periods, exposures can be hazardous. For monitoring, filter samples are often collected and subject to thermal-optical analysis to measure the two primary components of DPM: elemental (EC) and particulate organic carbon (POC). However, the presence of volatile organic carbon (VOC) can interfere. The standard approach to correcting for VOC is use of a "dynamic blank" filter, but errors have been reported. Alternative approaches have also been proposed, including pre-baking samples to drive off VOC before typical thermal-optical analysis, or addition of low temperature isotherm(s) to the beginning of the analysis routine for the same purpose. As a preliminary study, these simple thermal correction methods were compared to the standard correction using field samples collected in three locations (two in an underground mine, one at a highway truck stop). The pre-baking method (at 175°C) was found to overcorrect for VOC, resulting in underestimation of POC. Efficacy of the modified thermal-optical analysis (with addition of an initial isotherm at 175°C) appeared to be dependent on sample mass and/or composition.

1 INTRODUCTION

Chronic exposure to diesel exhaust has been linked to a range of health hazards (Sydbom et al., 2001). In underground mines, where there is potential for high-concentration or long-duration exposures, monitoring and regulation of diesel exhaust may be necessary. In US mines, regulatory limits are imposed on the solid portion of the exhaust, called diesel particulate matter (DPM). While DPM is heterogeneous, it is predominantly composed of submicron particulate organic carbon (POC) and elemental carbon (EC), the sum of which is referred to as total carbon (TC). TC is commonly used as a surrogate analyte for DPM (NIOSH, 2016; Birch and Cary, 1996; EPA, 2002).

In US metal/nonmetal mines, the personal exposure limit for DPM is operationally defined as 160 μg TC/m^3 measured as an 8-hour time weighted average (30 CFR 57.5060). For compliance monitoring, particulate samples are collected onto quartz fiber filters using a special size selector (i.e., impactor and/or cyclone) to remove mineral dust particles (i.e., which are predominantly supramicron). Samples are analyzed per the standard NIOSH Method 5040, which is a thermal-optical method. Briefly, the sample is heated in two phases following a specific thermal program in a controlled atmosphere. In the first phase, OC is evolved in an inert atmosphere by incrementally increasing the temperature through four isotherms (i.e., 310°C, 475°C, 615°C, and 850°C). In the second phase, EC is evolved in an oxidizing atmosphere additional isotherm (i.e., 550°C, 625°C, 700°C, 775°C, 850°C, and 870°C). The evolved carbon in each phase is converted to CO_2, reduced to CH_4, and measured using a flame ionization detector (FID) or a nondispersive infrared sensor (NDIR), such that OC and EC can be derived (Birch and Cary, 1996; NIOSH, 2017; Chai et al., 2012).

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A major challenge for the NIOSH 5040 and other such thermal-optical methods for OC-EC measurement is the interference of gas-phase (volatile) OC, called VOC. Diesel exhaust itself is a primary source of VOC, but there may be other sources (e.g., cigarette smoke; Birch and Cary, 1996; Chai et al., 2012; Kirchstetter et al., 2001; NIOSH, 2016; Subramanian et al., 2004; Noll and Birch, 2008; Zhu et al., 2012) in the sampling environment or even associated with the sampling media (e.g., filters, impactors, cassettes; see Guse and Sarver, 2019). Since VOC evolves in the first phase of analysis, it generally results in seemingly high POC quantities. To correct for most VOC interferences, a "dynamic blank" filter is commonly used (NIOSH, 2016; Watson and Chow, 2009). Basically, two filters are stacked in the sampling cassette: the primary (i.e., top) filter collects the targeted particulates (i.e., POC and EC) and VOC, while the secondary (i.e., bottom) filter collects only VOC. A simple subtraction of the secondary filter result from that of the primary filter serves as a way to analytically remove VOC from the sample mass.

While the dynamic blank correction is currently standard practice for personal DPM sampling in US mines, it is imperfect and problems can arise from factors such as insufficient sampling time, variable quartz filter characteristics, and contamination from materials in the sampling train (Guse and Sarver, 2019; Kirchstetter et al., 2001; NIOSH, 2016; Noll and Birch, 2008; Turpin and Huntzicker, 1994). Such problems can lead to under or over-correction of VOC.

A range of alternatives for addressing VOC sampling artifacts have been considered, including use of a denuder in the sampling train, or collection of additional samples with Teflon filters to physically separate VOC during sampling. However, these present practical challenges for personal exposure monitoring. On the other hand, thermal analytical corrections or modifications to the sample preparation procedure, could be easier. Moreover, as opposed to the dynamic blank correction, they may also have an added benefit of only requiring analysis of a single filter.

Thermal correction for VOC is indeed based on the notion that VOC, due to its inherent volatility, should significantly evolve at lower temperatures than POC. Practically, this might be accomplished during standard analysis (e.g., by modifying the thermal ramp of the NIOSH Method 5040), or during sample preparation (e.g., by "pre-baking" samples at relatively low temperature to remove VOC before 5040 analysis). The latter option, if efficacious, could be particularly useful in expediting analysis since it could potentially be used to prepare large batches of samples.

Development of thermal corrections to reliably separate VOC from POC in DPM samples is largely dependent on identifying an optimal temperature range and atmospheric conditions. To date, data directly related to the application discussed here (i.e., DPM in underground mine samples) is not available, though some insights can be gained from a recent study of vehicle exhaust in a highway tunnel (likely dominated by diesel and gasoline emissions). Using the IMPROVE standard method (substantially similar to NIOSH Method 5040 for the purpose of this comparison), Zhu et al. (2012) showed that 56 and 23% of the VOC in PM2.5 samples evolved at 140 and 280°C, respectively. For PM0.1, 41 and 27% of the VOC evolved at the same temperatures – which are well below the lowest temperature (310°C) in NIOSH Method 5040. However, the results from Zhu et al. did indicate some POC evolution at 140 and 280°C, meaning there can be overlap between the VOC and POC evolution.

The purpose of the preliminary study presented in this paper was to test two possible thermal modifications to the standard preparation or analysis of mine DPM samples by NIOSH Method 5040: 1) a low-temperature pre-baking procedure, or 2) addition of an initial low-temperature isotherm to the 5040 routine. Based on the Zhu et al. (2012) study and consultation with researchers at NIOSH's Pittsburgh Mining Research Division, who have worked extensively on analysis of laboratory-generated DPM, 175°C was chosen as the target temperature for both modifications.

2 EXPERIMENTAL DETAILS

2.1 *Sample collection*

Two sets of DPM samples were collected in an underground stone mine, in which DPM is considered the only significant source of OC or EC. Sample set 1 (14 replicates) was collected just outby active production activities involving a diesel loader and haul trucks, where DPM concentration was expected to be relatively high. Set 2 (6 replicates) was collected near the primary crusher, which is located in the main exhaust airway. While this location should include DPM sourced from all areas of the mine, the average concentration should be substantially lower than near the production face. Sample sets 1 and 2 were each collected over a period of 5 hours. For comparison to the DPM samples, another set (Set 3, 6 replicates) was collected at highway truck-stop off of Interstate Highway 81 in southwest Virginia. These samples were collected over a total of 13 hours, and were expected to have multiple sources of OC and EC (including DPM and other vehicle emitted and ambient particulates).

All samples were collected using a typical sampling train for DPM. Escort ELF personal sampling pumps (Zefon International, Ocala, FL) were used to pull air through two sequential size selectors, a 10-mm Dorr-Oliver nylon cyclone (cut size of ~4μm cut size at 1.7 L/min; Zefon International) and then a DPM impactor (cut size of ~0.8μm at 1.7 L/min; SKC Inc., Eighty-Four, PA). The particulates passing the impactor were captured on a 37-mm quartz fiber (primary) filter, which was backed by an identical secondary filter and then a cellulose support pad (all from Pall Corporation, Port Washington, NY). The filters were housed inside a generic styrene cassette (Zefon International). Prior to sampling, all quartz filters were prepared by baking at 800°C for two hours, and then the cassettes were immediately assembled. They were stored at 0°C until sampling, and again until analysis to minimize OC sorption from sampling materials. Figure 1 shows representative primary filters from each sample set.

2.2 *Sample preparation and analysis*

Two punches (approximately 1.5 cm^2) were taken from each of the primary and secondary filters in each sample set, yielding two primary-secondary filter pairs per sample. The first pair was pre-baked before OC-EC analysis, and the second pair was analyzed without any preparation (Figure 2). (A total of six blank pairs was also analyzed without pre-baking.) Pre-baking was done in a Precision Convection Oven (Thermo Scientific, Waltham, MA) at 175±5°C, but the atmosphere was not otherwise controlled. The samples were baked overnight (8 hours) and left in the oven until removed for analysis the next morning.

All samples (i.e., whether pre-baked or not) were analyzed using one of two OC-EC Aerosol Analyzers (Sunset Laboratory Inc., Tigard, OR). (It is noted that variability between the two analyzers was investigated and revealed no bias.) The analysis followed a modified version of

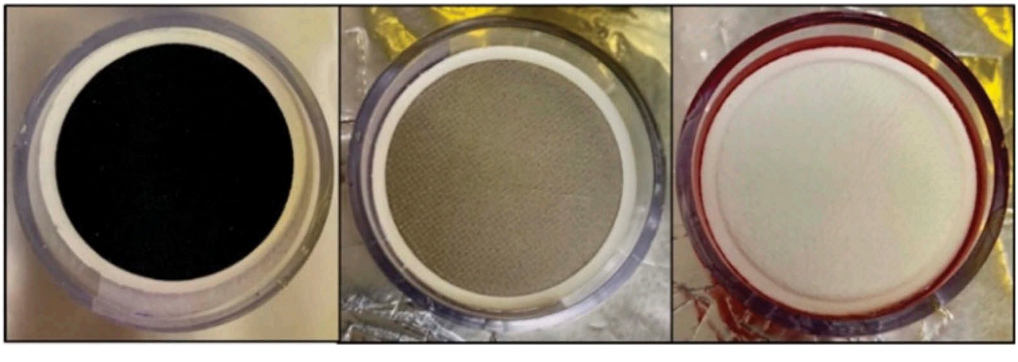

Figure 1. Representative primary filters from Sets 1-3 (left to right).

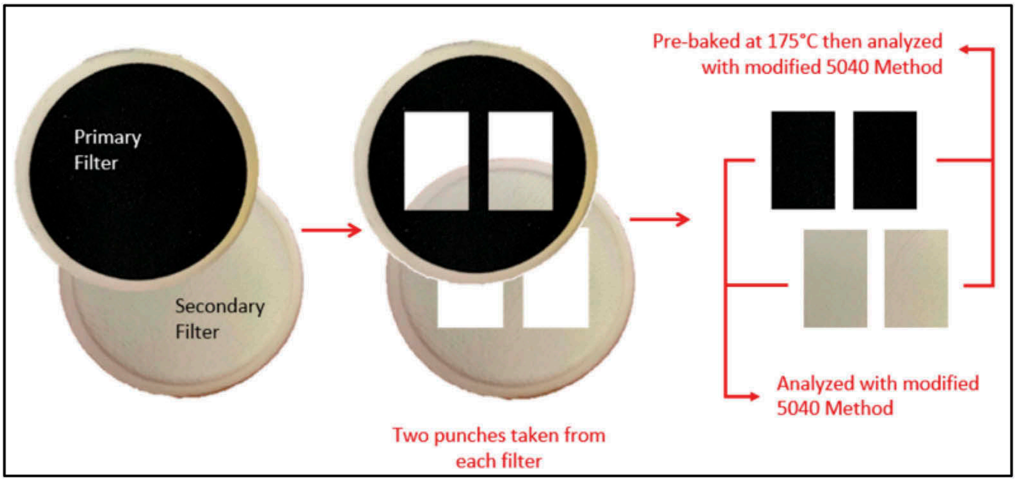

Figure 2. Flow chart for filter punching and preparation (pre-baked or not) prior to analysis with the modified 5040 method.

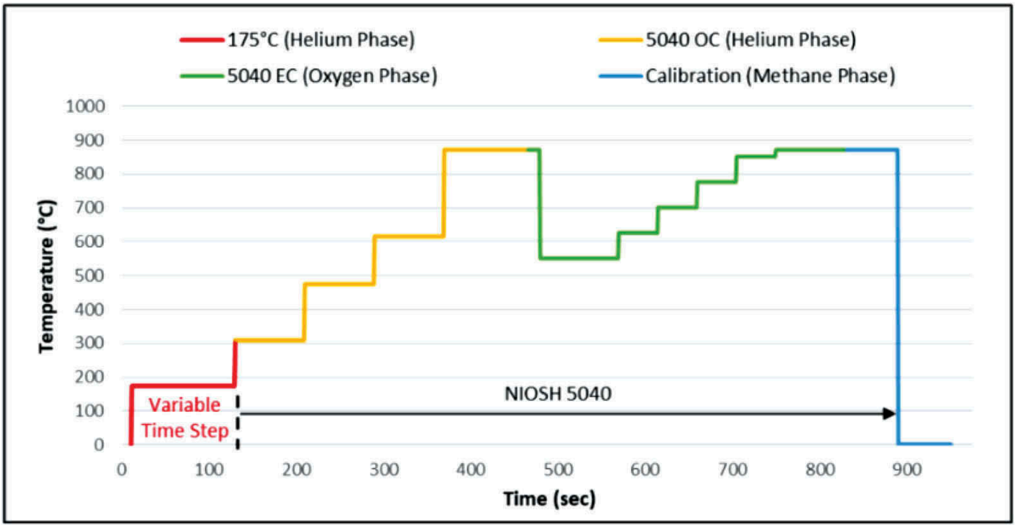

Figure 3. Modified NIOSH Method 5040 thermal profile, including an additional isotherm at 175°C.

the standard NIOSH Method 5040, in which the only change was inclusion of an additional initial isotherm at 175°C (Figure 3). This isotherm had a variable time step, meaning it was extended (up to 600 seconds) until the FID or NDIR indicated that carbon evolution was complete. It is assumed that any carbon evolved by the end of the 175°C isotherm would have evolved in the first isotherm (i.e., 310°C) of the standard Method 5040. Thus, the results from the sample pairs that were not pre-baked could be used for the standard dynamic blank VOC correction; and the primary filters from these pairs could also be used to test the modified correction that uses the additional isotherm at 175°C.

2.3 *Data analysis*

The OC-EC Analyzer outputs the evolved carbon mass (calibrated to peak area on a sample thermogram) for each isotherm or analysis phase of interest. For this study, the values of most import are the masses evolved during: the standard 5040 OC phase (i.e., in helium), the standard 5040 EC phase (i.e., in oxygen), and the additional isotherm at 175°C tested here. For each of the three methods of VOC correction considered here (i.e., "standard" dynamic blank, thermal modification using an additional "isotherm" at 175°C, and thermal modification using a sample "pre-bake" step), Table 1 shows the equations used to calculate each component of a sample (i.e., VOC, POC and TOC).

The average results of the six blank filter pairs were used to correct for VOC sourced from the sampling materials and sample preparation environment. This was done by subtracting the average contents of the blank samples from each field sample for both the primary and secondary filters; subtractions were made for each individual isotherm. While no blank filters were pre-baked, a comparative analysis between the baked and not pre-baked samples showed that only the first two isotherms (175 and 310°C) were affected by the baking process. Therefore, masses in these two isotherms were not blank-corrected in the pre-baked samples.

3 RESULTS AND DISCUSSION

Visual inspection of thermograms comparing the primary and secondary filters of blank, and not- baked and pre-baked DPM samples shows differences in each of VOC correction method considered here (Figure 4). For the blank filter pairs (Figure 4, left), the secondary filter consistently has more carbon than the primary. This is consistent with the findings of a previous study related to apparently negative OC results on low mass DPM samples, and suggests disproportional sorption of OC between the primary and secondary filter causing some error in the dynamic blank correction method (Guse and Sarver, 2019). The VOC on the secondary filter was observed to evolve under the third isotherm of the modified thermal ramp (i.e., 475° C) and is most likely sourced from the sampling material (i.e., cassette or support pad). Accordingly, this carbon peak is also present in the secondary filter thermograms of samples that were and were not pre-baked. Importantly the VOC observed on blanks in this study also demonstrates that not all VOC evolves at low temperatures.

Table 1. Summary of samples prepared and analyzed using each of the three methods of VOC correction. The equations used to calculate the VOC, POC and TC components are also shown for each method.

Sample pairs	Preparation	Component calculations for each VOC correction method			
		Standard		Modified	
Set : 14	none	standard dynamic blank	$VOC_{std} = OC_{\mathrm{sec}}$ (*Eq. 1*)	(1) additional isotherm	$VOC_{iso} = OC_{pri@175^{\circ}c}$ (*Eq. 4*)
Set 2: 6			$POC_{std} = OC_{pri} - OC_{\mathrm{sec}}$ (*Eq. 2*)		$POC_{iso} = OC_{pri} - OC_{pri@175^{\circ}c}$ (*Eq. 5*)
Set 3: 6			$TC_{std} = POC_{std} + EC$ (*Eq. 3*)		$TC_{iso} = POC_{iso} + EC$ (*Eq. 6*)
blank: 6					
Set 1: 12	pre-baked @175°C	N/A		(2) pre-bake	$POC_{backed} = OC_{backed\_pri}$ (*Eq. 7*)
Set 2: 6					$TC_{baked} = POC_{baked} + EC_{baked}$ (*Eq. 8*)
Set 3: 6					$^{*}VOC_{baked} = avg[OC_{pri}] - OC_{baked\_pri}$ (*Eq. 9*)

In Equations 1-9: OC_{pri} and OC_{sec} are the carbon masses evolved during the OC phase of analysis on the primary and secondary filters, respectively, of a given sample pair; EC is the mass evolved during the EC phase of analysis on the primary filter; and $OC_{\mathrm{pri@175°C}}$ is the mass evolved during the 175°C isotherm. Further, a subscript "baked" denotes the quantity is measured on a sample filter that was pre-baked at 175°C.

*Since VOC is assumed to be evolved during the pre-baking step, to estimate VOC on pre-baked samples in this study Equation 9 was used, where avg[OC_{pri}] is the average OC measured on un-baked replicate filters (i.e., from the same sample set).

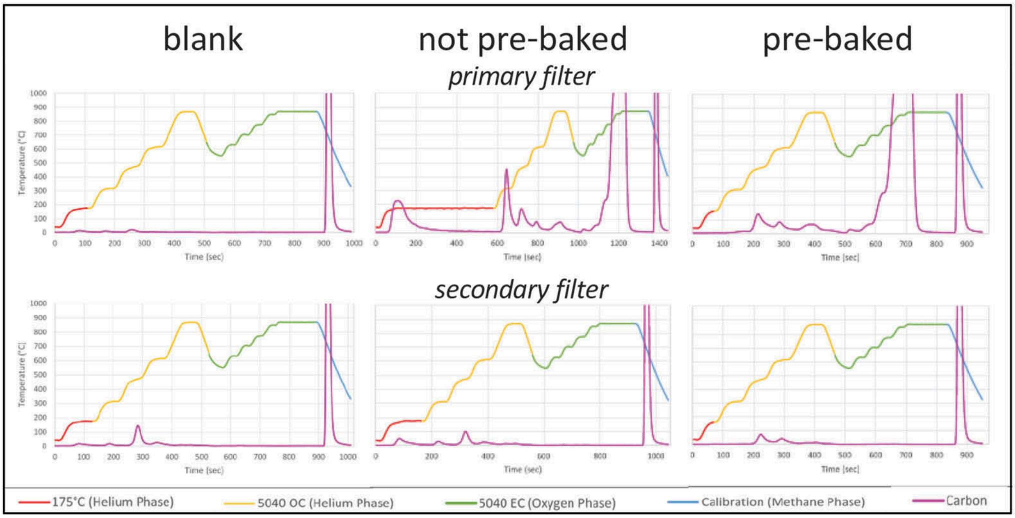

Figure 4. Representative thermograms from primary and secondary filter pairs. Blanks, which were not pre-baked per Table 1, are shown on the left. In the middle and right are replicate DPM samples for which the filters were or were not pre-baked.

The middle thermograms in Figure 4 are from a DPM sample in Set 1, where the filter punches were not pre-baked prior to OC-EC analysis. The carbon peak under the initial 175° C isotherm represents the amount of OC removed in the isotherm correction (i.e., VOC_{iso}). Compared to the total OC on the secondary filter (representative of the standard correction, VOC_{std}), the isotherm correction appears much larger, especially considering the subtraction of the blank sample secondary filter OC. This indicates that some POC evolves at 175°C along with VOC in these particular samples. Inspection of the secondary filter thermogram also indicates that, while some VOC evolves at 175°C, not all of it does; as mentioned above, the larger peak at 475°C attributed to VOC in the secondary filters from Set 1 is likely due to the sampling materials themselves. Nevertheless, the observations on the not pre-baked Set 1 filters suggest that there is not good separation between VOC and POC in these samples.

The right side of Figure 4 also shows results from same DPM sample in Set 1 where the filter punches were pre-baked. It is clear from the primary filter that pre-baking removed not only the carbon that would have evolved in the 175°C isotherm, but most of carbon that would have evolved in the second isotherm (310°C) as well. Comparing the secondary filter thermograms of all three samples in Figure 4 shows that pre-baking removes some of the VOC, but not all (e.g., 475°C isotherm and 610°C isotherm). Even though the oven was held between 170°C and 180°C, it appears that uncontrolled oxidizing atmosphere may have allowed for more OC evolution than intended. The outcome is that the pre-baking correction tested here removes some POC along with VOC, meaning it overcorrects compared to the standard correction method.

Figure 5 shows the results of POC, VOC, and EC for each correction method. Consistent EC results within each sample set indicate repeatability of the EC measurement, even following pre-baking of some filters; also, sample collection was not impacted by spatial variability. Each sampling location had different submicron particulate matter concentrations and/or sources. The truck stop had very little EC (average of 0.165μg/cm^2 on the sample filters). Both sets of mine samples shared sources of particulate matter, although air at the crusher is diluted and mixed – so lower EC concentration was expected there versus near production. Average EC on the crusher location samples was only 3.303μg/cm^2 versus 48.247μg/cm^2 on the production samples.

With respect to VOC, the performance of the isotherm correction (compared to the standard) varied by sample set (and presumably mass). The isotherm correction yielded a lower VOC than the standard for the truck stop samples. However, results for the mine samples

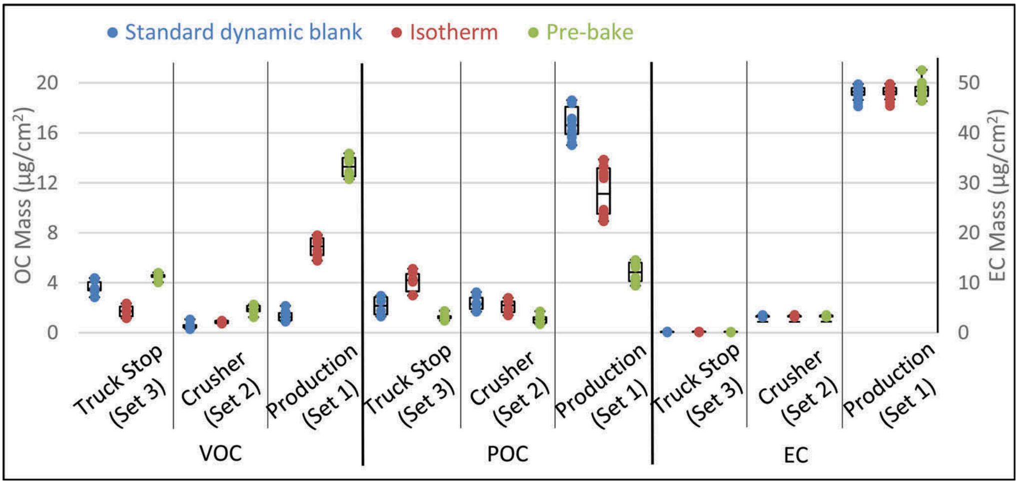

Figure 5. Box and whisker plot showing VOC, POC, and EC results for each of the three correction methods on all three sample sets. Results are shown as mass per sample filter area (µg/cm^2).

were different. The isotherm correction agreed with standard for the crusher samples, but was much higher than the standard for the production samples. Taken together, these results indicate that a universal temperature for a thermal-based correction may not be possible.

Figure 6 shows the average mass distribution by OC isotherm of primary and secondary filters from each sample set. A comparison of the mass distribution for a not pre-baked primary filter to the total mass for its secondary filter (representative of the standard dynamic blank correction) illustrates the variable performance of the isotherm correction method. For samples collected at the truck stop (Set 3), secondary filter mass is almost equal to the combined mass of isotherm 1 and 2 (i.e., 175°C and 310°C) of the primary filter. This demonstrates the under-correction of the isotherm method tested here. For the crusher samples (Set 2), the average secondary filter mass is about equal to the 175°C isotherm mass of the primary filter, which explains why the isotherm correction was in agreement with the standard

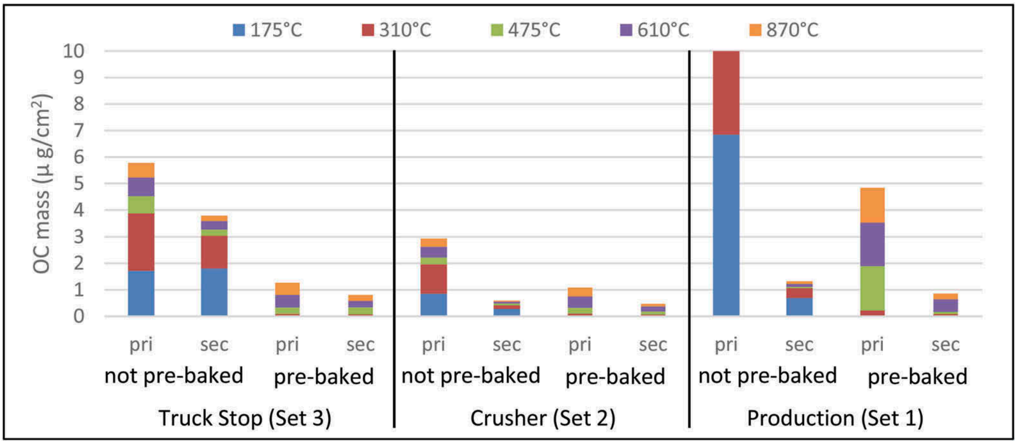

Figure 6. Average OC mass distribution by isotherm for the primary and secondary filters, both with and without pre-baking, from each sample set. Results are shown as mass per sample filter area (µg/cm^2). Note that the not pre-baked primary filter results from Set 1 had a total average OC mass of 18 µg/cm^2.

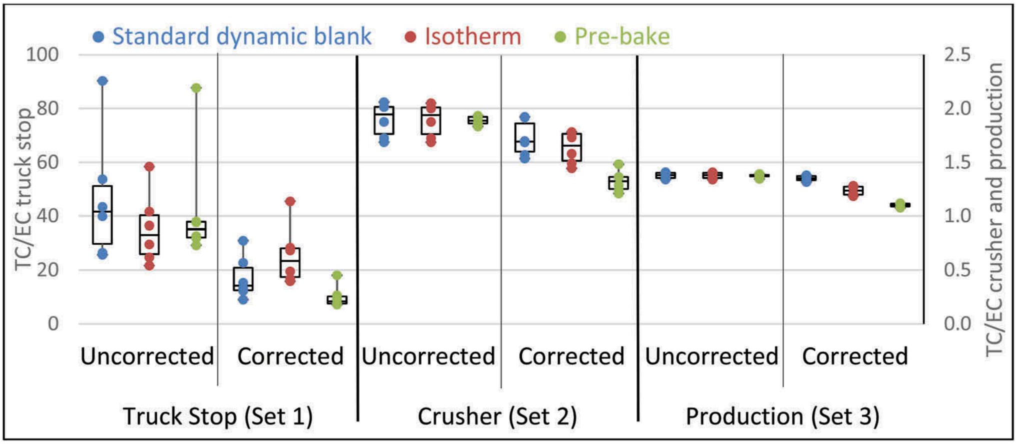

Figure 7. Box and whisker plot showing TC/EC ratio before and after each of the three VOC corrections for each sample set.

method. For the production samples, the secondary filter mass was only a fraction of the mass in isotherm 1 on the primary filter. This suggests that the additional isotherm at 175°C removed both POC with VOC on these samples.

The pre-baking correction consistently underestimated the POC at all locations (Figure 5), which is likely due to the oxidative oven atmosphere causing more evolution than intended. Upon examining the mass distribution of the pre-baked primary and secondary filters from each sample set (Figure 6), it appears that most of the mass from isotherms 1 and 2 (175°C and 310°C)were removed in the pre-baking process. This explains why the VOC determined with the pre-bake correction is relatively close to the standard for the truck stop samples.

The TC/EC ratios in each sampling location before and after VOC correction were also compared (Figure7). Ratios prior to corrections are similar, which again indicates that replicate samples were not affected by spatial variability and that the OCEC analyzers produced repeatable results. Compared to the mine samples, the samples collected at the truck stop had much higher TC/EC ratios, likely due to the different sources of particulate matter and VOC at this location. Carbon in these samples consisted of mostly OC, most of which was VOC. After the VOC correction, not only is there a reduction of the TC/EC ratio, but also a large reduction in error (i.e., shorter boxes and whiskers). Removing VOC from the calculation reduced variability, possibly indicating that VOC is more difficult to measure precisely than particulates. Less of a reduction in error is shown after the correction of mine samples, possibly due to proportionally less VOC in those samples.

While the crusher and production samples shared some sources of particulates and VOC (i.e., they were collected in the same mine), the production samples exhibited lower TC/EC ratio (i.e., corrected average $(TC/EC)_{std}$ was 1.35 versus 1.72 for the crusher samples). Other studies have shown that low-mass samples tend to have higher and more variable TC/EC ratios (Noll, 2007; Noll, 2015), perhaps due to premature EC evolution. This could be one explanation for the difference in TC/EC ratio observed here between the crusher and production samples. However, it may also be the case that specific aerosol sources (e.g., particular equipment) and factors (e.g., aerosol aging or disproportionate removal of EC as air moves through the mine) have significant effect on the TC/EC ratio.

4 CONCLUSIONS

While there is error associated with the standard dynamic blank method of correcting for VOC interference in diesel particulate matter samples, the current study could not prove that temperature-based correction is a suitable alternative. The accuracy of the dynamic blank correction is dependent on one major factor (i.e., equal sorption of VOC onto the primary and secondary filters). However, the accuracy of a temperature-based correction depends on many factors (i.e., separation temperature, POC and VOC sources, sample mass and composition). The separation temperature of 175°C examined here may produce comparable results at a certain location with specific sample composition, like it did for the crusher samples. However, 175°C was not effective at the other sampling locations. The behavior of VOC and POC seems to be source dependent and therefore a universal temperature for correction is likely not plausible. Studying locations with relatively consistent POC and VOC sources could lead to finding separation temperatures specific to those locations–or at least a better understanding of OC thermal evolution characteristics.

VOC causes interference in particulate matter sampling and is difficult to accurately measure. Monitoring in ambient environments where EC concentrations may be low, OC is an important component. But for occupational DPM monitoring in environments such as underground mining where EC concentration is consistently high, that might not be the case. OC is a complex component making it difficult to measure with confidence. EC on the other hand is a more robust and reliable component making it easier to measure. As suggested by others, EC alone maybe a more favorable surrogate for DPM measurement in underground mining.

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