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VOC INTERFERENCE IN DPM SAMPLE ANALYSIS: SAMPLING MATERIALS AS POSSIBLE VOC SOURCES

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ABSTRACT

Diesel particulate matter (DPM) is monitored in some occupational environments, including mines, by measuring its primary components: elemental (EC) and organic carbon (OC). While EC only occurs in the solid phase, OC can occur in both the solid and gas phases. The latter is termed volatile OC or VOC. Aside from diesel exhaust, there can be a variety of sources that contribute VOC to DPM samples, including the sampling materials themselves. Regardless of the source, VOC presents analytical interference for particulate OC measurements. To correct, a tandem-filter approach is often used whereby a primary filter should collect both OC phases, while a secondary filter beneath only collects the VOC; then, a simple difference between primary and secondary filter results is used to determine particulate OC. However, anecdotal observations of higher OC on the secondary filter indicate that, in some cases, VOC might preferentially sorb to the secondary filter. This leads to underestimation of the particulate OC, which in some cases can even appear negative. One possibility is that sampling material-sourced VOC could produce such an artifact. To explore, three existing datasets containing both blank filters and field and lab DPM samples were examined to look for instances and conditions surrounding higher secondary than primary filter OC. Then, a follow-up study was conducted to investigate the relative VOC contribution to blank filters by standard materials used for mine sampling. Testing was done under both hot and cold storage conditions.

INTRODUCTION

The exhaust from diesel engines is a complex and heterogeneous mixture of volatile and solid constituents. It is generally associated with a range of acute and chronic health effects (Ping, et al., 2017; NIOSH, 1988; Attfield, et al., 2011; IARC, 2012), and exposure control is increasingly of concern in occupational environments such as mines. Due to the complex nature of whole diesel exhaust, monitoring is typically focused on the solid portion of the exhaust, called diesel particulate matter (DPM). DPM is also complex, but is primarily made up of elemental (EC) and organic carbon (OC) (EPA, 2002), which can be used as analytical surrogates. Since the ratio of EC to OC can vary, these analytes are often summed to report total carbon (TC). In the US metal/nonmetal mines, DPM is regulated on the basis of a personal exposure limit to TC, which has been set at 160µg/m^3 for an 8-hr time-weighted average (30 CFR part 57). In US coal mines, where coal dust particles themselves may interfere with measurements of DPM-sourced carbon, personal exposures are not directly limited; instead, emissions are regulated at the tailpipe (30 CFR part 72D)

Most DPM analysis is done on collected filter samples, using thermal-optical methods such as the NIOSH 5040 Standard Method (2017). As explained in more detail below, these methods seek to separately measure OC and EC by carefully controlling their evolution with both temperature and atmosphere. While EC occurs only in the particulate phase, OC occurs in both particulate and gas phases. Gas-phase OC is commonly called volatile OC (VOC). VOC compounds may be part of the diesel exhaust itself, or may be present in the sampling atmosphere due to other sources (e.g., cigarette smoke) (Birch and Cary, 1996; Bugarski, et al., 2011; NIOSH, 2017). In either case, they will be sampled with DPM - unless a special setup is used (e.g., a denuder) in the sampling train, which is atypical. VOCs might also be contributed by sampling equipment or materials (Birch, 2003). Regardless of the source, since measurement of DPM inherently targets only particulate OC, VOCs must somehow be removed from the result.

A common way of dealing with this problem has been to use a tandem-filter (also called a dynamic-blank) approach (Birch, 2016). Basically, two filters are used in series: the idea is that primary filter collects the DPM and any VOC components, and the secondary filter only collects the VOCs. The difference between the primary and secondary filter OC is thus regarded as the *particulate* OC – i.e., the portion of OC that contributes to DPM. In the simplest conception of the tandem-filter correction, it is obvious that the primary filter OC must always be greater than the secondary filter OC. However, anecdotal observations have shown that occasionally this is not true, in which case the particulate OC result appears negative. As a practical consequence, TC may be underestimated.

A logical explanation for unexpectedly low or even negative particulate OC results might be higher VOC on the secondary filter than on the primary. This situation could potentially occur due to complex sorption/desorption mechanisms during sampling (Maimone, 2011). It might also occur if sampling materials themselves contribute VOC that is disparately sorbed to the secondary filter. The issue of pre-existing VOC from sampling materials is well known, and indeed it is recommended that filters be pre-cleaned (i.e., by baking) prior to sampling to minimize this VOC source (Birch, 2016). However, the issue of whether the primary and secondary filter are similarly affected by this source has not been addressed.

In order to investigate the circumstances surrounding observations of apparently negative particulate OC, a preliminary study was conducted using three existing 5040 datasets. Then, a follow-up study was conducted to explore the possibility that VOCs contributed by standard materials used for mine sampling (i.e., polystyrene cassettes, cellulose filter support pads, and DPM impactors used for particle size selection) can produce this artifact.

NIOSH 5040 STANDARD METHOD

The 5040 Method is an accepted analytical method for measuring EC in DPM samples. Where TC determination is needed (e.g., for regulatory compliance monitoring in US mines), the method is also used to measure OC, in which case the tandem-filter approach described above is recommended (NIOSH, 2017).

Samples are collected onto quartz fiber filters (primary and secondary), which have been pre-cleaned by baking at a high temperature (i.e., 800°C or more); filters can be purchased pre-baked and/or can be baked in-house. A size selector is recommended in the sampling train to remove oversized particles (e.g., mineral dust), and a special DPM impactor (DPMI) is available especially for this purpose from SKC Inc. (Eighty Four, PA). Following sampling, a small punch (usually 1.5cm^2) is taken from each filter and analyzed using a thermal optical analyzer such as the OCEC Laboratory Analyzer available from Sunset Laboratory Inc. (Tigard, OR) (Birch, 2003; NIOSH, 1999).

The 5040 Method specifies two controlled temperature ramps for the thermal-optical analysis. First, the sample is heated in helium (i.e., inert) atmosphere in order to evolve OC, which can thermally degrade in the absence of oxidizers. As the OC evolves, the resulting CO_2 is reduced to CH_4, which can be measured either by flame ionization detector (FID) or infrared spectroscopy (IR) to determine OC mass. Pyrolysis of OC is also possible, which effectively converts some OC to EC in the first stage of the 5040 Method analysis. Generation of this EC, which is commonly called char, is indicated by a decrease in laser transmittance through the sample filter.

In the second stage of the analysis, the sample is reheated in helium/oxygen atmosphere to oxidize off the EC. Again, CO_2 converted to CH_4 is monitored in the evolved gas. To distinguish the original sample EC from the OC-derived char, the laser transmittance is used. Assuming that the char is evolved first, the point where the transmittance returns to its original value is used to separate regions of char and EC oxidation on the 5040 thermogram (Birch, 2016; NIOSH, 1999; Peterson, et al.). An example thermogram is shown in Figure 1 to illustrate the various analysis regions and analytes of interest.

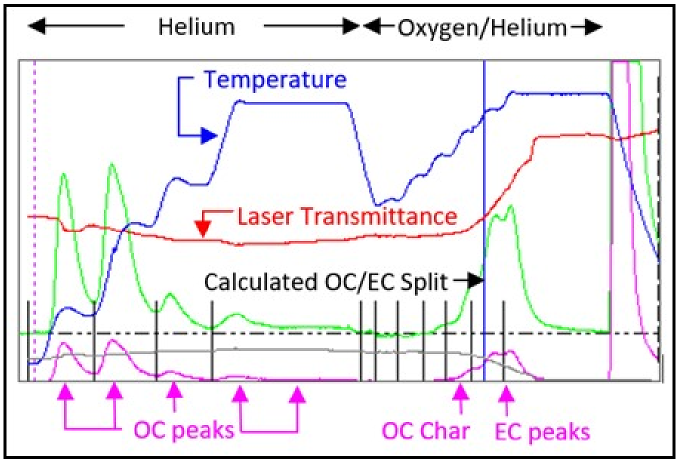

Figure 1. Example thermogram from a DPM sample.

PRELIMINARY STUDY TO EXPLORE APPARENTLY NEGATIVE OC RESULTS

To explore the occurrence and features of apparently negative particulate OC results, three existing datasets were studied (see below). Each set contained data from primary and secondary quartz fiber filter pairs that had been analyzed by the 5040 Method using an OCEC Analyzer (Sunset Laboratory Inc., Tigard, OR) operated at the NIOSH/OMSHR diesel research laboratory in Pittsburgh, PA. The analyzer exports the raw data into *.txt* files that are then run through Sunset's OCEC calculation software program. This program generates a thermogram from each sample and calculates total OC mass, total EC mass, as well as the OC and EC masses of the individual peaks in each region of analysis (e.g., Figure 1).

For each filter pair, the total OC values were compared (i.e., primary filter value – secondary filter value). In cases where the total OC on the secondary filter was greater than on the primary, yielding a negative particulate OC value, the sample was classified as "problematic." To gain insight about the problem source(s), the primary and secondary filter results were also compared with respect to OC values associated with all five individual peaks in the helium region of the 5040 Method (i.e., which occur at 250°C, 500°C, 650°C, and 850°C).

Description of Datasets

Dataset 1: Blank Filters. This dataset contains seven pairs of primary and secondary blank filters, which originated form a NIOSH investigation of pre-existing OC on the filters that come pre-loaded in the DPM sampling cassettes purchased form SKC. These special cassettes come pre-assembled with two (pre-baked) quartz filters, a cellulose support pad, and DPMI (i.e., which is housed inside the cassette). The cassettes used here had not yet reached their expiration date (which is stamped by SKC), but their age (i.e., time from assembly to testing) was unknown. Initially a single punch from each primary and secondary filter from each cassette was analyzed (as received from SKC). Then the remnant of the primary filter was re-baked at 820°C for two hours in-house (i.e., to drive off OC that had sorbed since it was originally prepared by SKC) and another punch was taken for immediate analysis. Thus, for each sample pair, there were three 5040 results: primary filter, secondary filter, and baked primary filter.

Dataset 2: Field DPM Samples and Blanks. This dataset contains nine pairs of primary and secondary filters, which originated from a field study of DPM in specific locations of an underground limestone mine (Gaillard, 2017). The samples were collected in the SKC DPM cassettes, from which the filters were removed and pre-baked in house several days to several weeks prior to sampling. No field blanks were collected using the SKC DPM cassettes, as the target analyte for the study was EC, but six blanks were prepared in generic polystyrene cassettes with cellulose support pads (both from Zefon International, Ocala, FL); again, the primary and secondary filters were pre-baked and loaded into the cassettes several days to weeks prior to sampling. (Notably, these blanks were actually prepared to complement other samples in the Gaillard study, which were collected in larger size ranges to capture both DPM and carbonate mineral dust in the mine. The DPM+dust samples are not reported here as carbonate dust can affect apparent OC in 5040 Method analysis if it is not specifically identified and corrected. However, carbonate dust should not have affected the DPM samples collected in this study with the SKC DPM cassettes, since the impactor effectively removed all dust from the sample.) From the nine DPM samples and the six blank studied here, a single punch from each primary and secondary filter was analyzed several months after sample collection.

Dataset 3: Lab DPM Samples. This dataset contains 14 pairs of primary and secondary filters from laboratory-generated DPM samples. These originated from a study of several continuous DPM monitors, which were being compared to the traditional DPM filter samples analyzed by the 5040 Method (Barrett, 2018). The filters were pre-baked in-house several days to weeks before sample collection, and were loaded into generic polystyrene cassettes with cellulose support pads (both purchased from Zefon). The samples were collected in the Marple chamber at the NIOSH/OMSHR diesel research laboratory. The chamber was fed with diesel exhaust from a small engine operating at low load. Because the target analyte for the Barrett study was EC, blanks were not collected. 5040 Method analysis was conducted several days to weeks following sample collection.

Results and Discussion

As summarized in Figure 2, a total of nine samples across all three datasets were found to be problematic, but eight of these were blanks – which should ideally exhibit no real difference between the primary and secondary filter OC. In the Dataset 1 blanks, total OC values on the primary and secondary blank filters were generally 3-4µg/cm^2, with three of seven samples having higher secondary OC. Upon re-baking the primary filters from this dataset, the total OC was reduced to less than 1µg/cm^2. In the Dataset 2 blanks, the primary filter OC was generally around 1µg/cm^2 and the secondary filter OC was around 2µg/cm^2.

Figure 3 shows the apparent particulate OC for all sample pairs as a function of total EC mass on the primary filter, which can serve as a sort of proxy for DPM sample mass. As expected, with more sample mass the difference between primary and secondary filter OC increases and should effectively represent the particulate OC. The only DPM sample that produced higher OC on the secondary filter than on the primary was a relatively low-mass sample (collected in a SKC DPM cassette, Dataset 2).

For the blanks, the largest differences between primary and secondary filter OC were a little over 2µg/cm^2, and differences were somewhat higher on blanks from Dataset 2 versus those from Dataset 1. Taken with the total OC values on the primary versus secondary filter (noted above), this may indicate that the SKC DPM cassette and the generic cassette assemblies contribute VOC to the filters differently. While secondary filters in the generic cassettes generally had higher VOC than primary filters, filters in the SKC DPM cassettes generally had higher VOC overall.

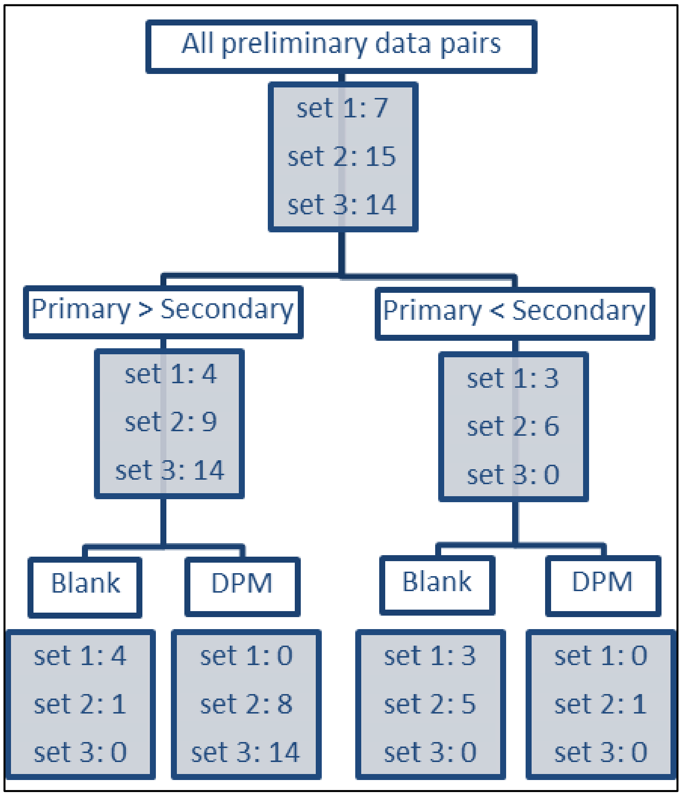

Figure 2. Breakdown of data set pair characteristics.

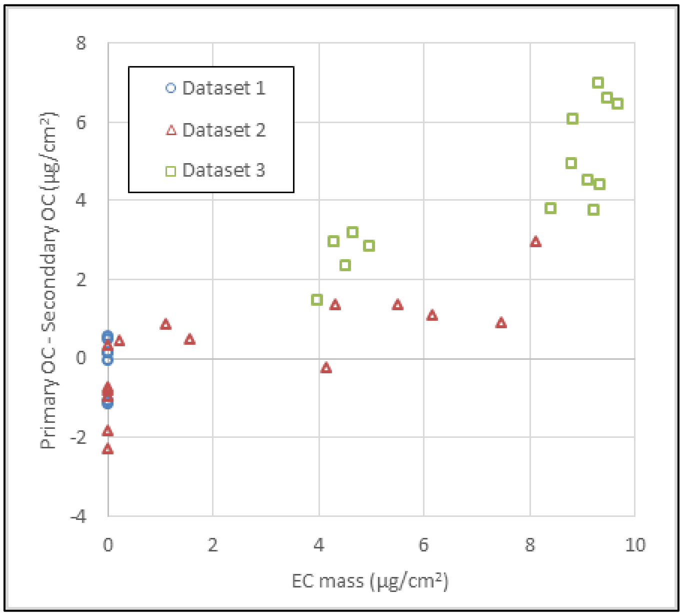

Figure 3. Primary OC – secondary OC versus total EC mass for the three datasets.

To better understand the OC evolution during 5040 Method analysis, a visual inspection was conducted by overlaying the primary and secondary filter thermograms. Figure 4 shows illustrative thermograms for a typical (i.e., non-problematic) DPM sample from Dataset 3, and for a blank sample from Dataset 2 where secondary filter OC was higher than primary. OC on the blank samples, which could have only been VOC, almost fully evolves in the first three peaks, whereas OC on samples with significant DPM mass continues evolving in fourth and possibly fifth peaks.

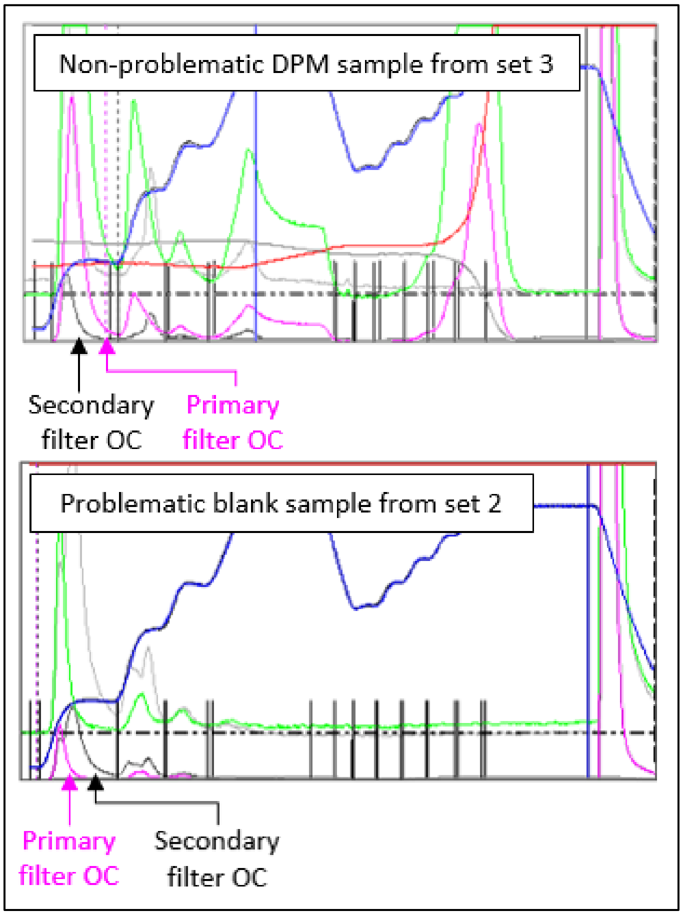

Figure 4. Overlay of the primary (pink OC line) and secondary (black OC line) filters thermograms for a non-problematic DPM sample from Dataset 3 and a problematic blank sample from Dataset 2.

Table 1 shows a summary of the OC values observed in each of the individual peaks on the 5040 Method thermograms for primary versus secondary filters in each dataset. For problematic samples, the secondary filter values were generally higher than the primary filter values for all peaks. However, even for blanks and low-mass samples with higher total OC on the primary filter (i.e., Datasets 1 and 2), the secondary OC values were found to be higher than the primary in some individual peaks.

FOLLOW UP STUDY TO INVESTIGATE SAMPLING MATERIAL AS VOC SOURCES

To gain more insight to if and how standard materials used for DPM sampling in mines may affect pre-existing VOC on primary and secondary filter pairs, a follow-up study was conducted.

Experimental Method

Five sampling materials conditions were tested: two brands of generic polystyrene cassettes (purchased from SKC and Zefon); typical cellulose support pads that can be used behind quartz filters during sampling (Zefon brand tested here in SKC polystyrene cassettes); and the SKC DPM cassettes, tested both with and without the impactor device installed (Figure 5). Each of the five material conditions was also tested under two temperature conditions: cold (i.e., refrigerated at 0-5°C) or heated (i.e., at 35-45°C). For all conditions, samples were prepared by pre-baking 37-mm diameter quartz fiber filters (Zefon, Ocala, FL) at 800°C for two hours, and then immediately placing two filters (i.e., primary and secondary) into their respective cassettes and temperature conditions. All conditions were tested in triplicate. Table 2 shows conditioning time (i.e., days until analysis) for each test. At that time, a single punch was taken from each filter and analyzed by the 5040 Method using an OCEC Analyzer at Sunset Laboratory's facility in Durham, NC.

Table 1. Summary of average OC mass (and standard deviation) in each individual peak for primary (p) and secondary (s) filters for the three datasets. Samples are split into groups by sample type (blank or DPM sample) and relative difference between primary and secondary filter results.

Dataset and group		Peaks				
		1	2	3	4	5
Dataset 1: Blank pri>sec (n=4)	p	1.906 (0.236)	0.698 (0.212)	0.455 (0.086)	0.635 (0.142)	0.060 (0.020)
	s	1.690 (0.062)	0.725 (0.206)	0.366 (0.094)	0.457 (0.130)	0.040 (0.019)
Dataset 1: Blank pri<sec (n=3)	p	1.621 (0.027)	0.573 (0.250)	0.351 (0.186)	0.414 (0.273)	0.022 (0.019)
	s	1.883 (0.254)	0.871 (0.344)	0.481 (0.088)	0.595 (0.046)	0.027 (0.006)
Dataset 2: Blank pri>sec (n=1)	p	0.480	0.431	0.187	0.168	0.005
	s	0.422	0.374	0.104	0.063	0.001
Dataset 2: Blank pri<sec (n=5)	p	0.603 (0.278)	0.2530 (0.080)	0.107 (0.017)	0.060 (0.016)	-0.001 (0.009)
	s	0.922 (0.617)	0.714 (0.306)	0.399 (0.454)	0.200 (0.097)	0.010 (0.009)
Dataset 2: Field samples pri>sec (n=8)	p	1.350 (0.513)	1.1676 (0.583)	0.362 (0.144)	0.478 (0.203)	0.000 (0.009)
	s	0.962 (0.250)	0.745 (0.272)	0.297 (0.072)	0.270 (0.034)	0.007 (0.008)
Dataset 2: Field samples pri<sec (n=1)	p	0.847	2.021	0.477	0.546	-0.003
	s	0.751	2.044	0.576	0.708	0.010
Dataset 3: Lab samples pri>sec (n=14)	p	4.404 (1.384)	1.073 (0.288)	0.370 (0.088)	1.990 (0.576)	0.040 (0.019)
	s	0.917 (0.304)	0.692 (0.544)	0.168 (0.084)	0.259 (0.164)	0.025 (0.037)

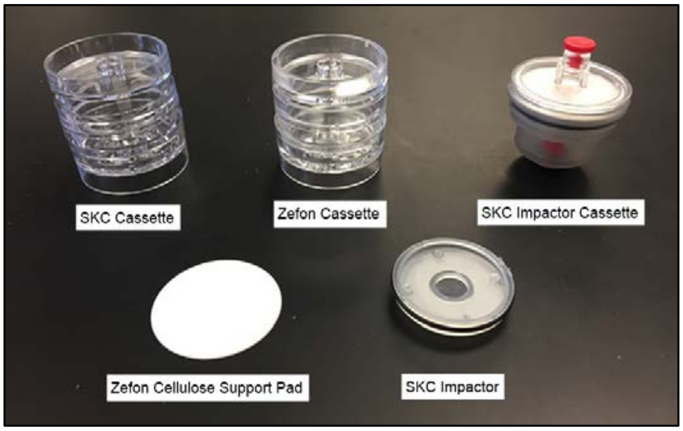

Figure 5. Five sampling material conditions tested to determine relative VOC contribution to primary and secondary DPM sampling filters.

After the punches were taken for analysis, the filters were placed into their original cassettes and stored together in varying temperature conditions (i.e., not controlled heating or refrigeration). After another period of conditioning, a second punch was taken from two of the filter pairs in three conditions (see Table 2) and analyzed again. One of the filter pairs from each of these conditions was run using a modified thermal ramp where the initial isotherm was lowered to 200°C and extended for 200s. The other filter pairs were run using a modified ramp where the initial isotherm was lowered to 175°C for 200 seconds. (i.e., from the standard 250°C for 60s). The goal analyzing with the modified methods was to determine if VOC would evolve readily at low temperatures. If so, this might shed some light on an alternative approach to dealing with VOC interferences altogether: a modified thermal program to better separate VOC from particulate OC on a single (i.e., primary) filter, rather than use of a tandem-filter for OC correction. The second analysis was conducted at the NIOSH/OMSHR diesel laboratory in Pittsburgh, PA.

Table 2. Matrix of test conditions and conditioning times (days) before 5040 Method analysis.

Materials	Cold	Hot
SKC cassette with cellulose support pad	1st analysis: 45 2nd analysis: 65	1st analysis: 45 2nd analysis:65
SKC cassette without cellulose support pad	1st analysis: 45 2nd analysis: 65	1st analysis: 45 2nd analysis: 65
Zefon cassette without support pad	1st analysis: 25	1st analysis: 25
SKC DPM cassette with impactor	1st analysis: 35 2nd analysis: 55	1st analysis: 35 2nd analysis: 55
SKC DPM cassette without impactor	1st analysis: 35	1st analysis: 35

Results and Discussion

Figure 6 shows a comparison of the average total OC observed on primary versus secondary filters in each test condition. As expected, hot conditioning and longer conditioning times seem to promote higher VOC contribution to the sample filters overall. Where differences between the secondary and primary filters were observed, the secondary tended to have higher OC than the primary and a few exceptions (i.e., after hot conditioning time in the SKC and Zefon polystyrene cassettes, and after a long cold conditioning time with the cellulose support pad, and after hot conditioning in SKC DPM cassettes with an impactor). For the support pads and the cold cassette materials, there may be some effect due to direct contact between the materials and the secondary filter surface.

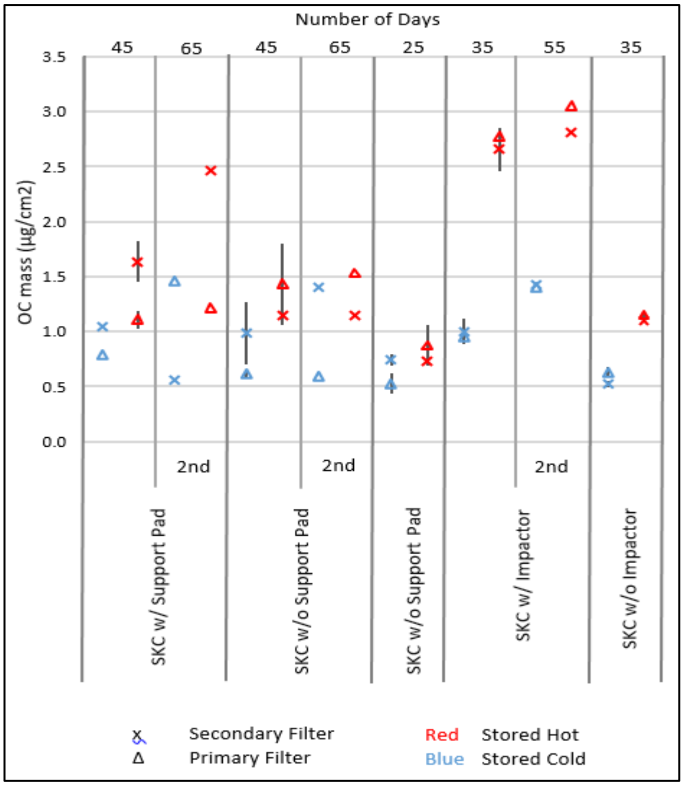

Figure 6. Average total OC observed on primary and secondary filters in each test condition (i.e., material and temperature). Where applicable, results from the second analysis is also shown. Error bars represent standard deviations where triplicate samples were analyzed. (Note that results from one sample from SKC with support pad stored in the cold condition was thrown out due to contamination.)

Regarding the most significant VOC contributions overall, the SKC impactors clearly had the greatest effect when stored in a heated environment. Unlike the cellulose pad, however, the VOC from the impactor appears to be sorbed more similarly by the primary and secondary filters. The filters in the hot conditioning with the impactor had 2.5x more OC than those in the cold condition.

Representative thermograms for primary and secondary filters for each condition are shown in Figures 7-11, including for samples analyzed during the second analysis (i.e., with modified initial isotherms). The majority of OC present in each case is located in the first and second peak areas, and these are also the areas that seem to be most affected when differences were observed between the primary and secondary filters.

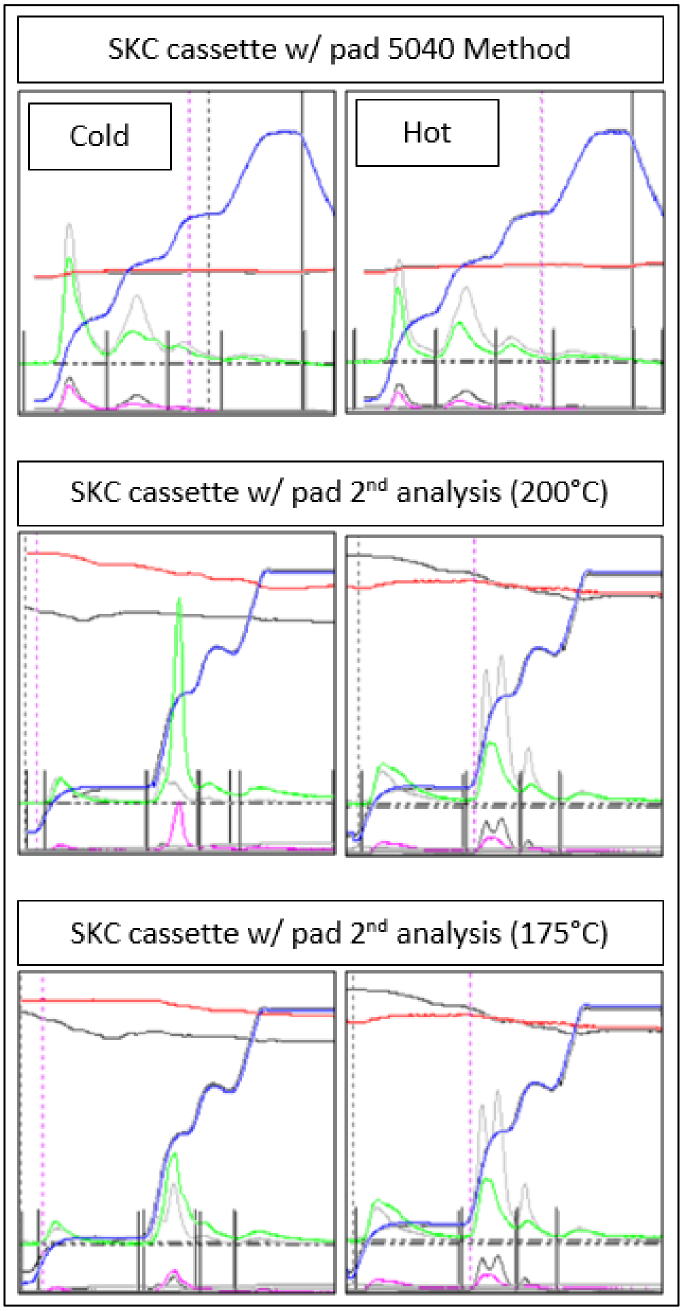

Figure 7. Representative thermograms (cropped to show only the helium region of interest) for the generic SKC cassette with support pad. The primary (pink) and secondary (black) filter results are overlaid for comparison.

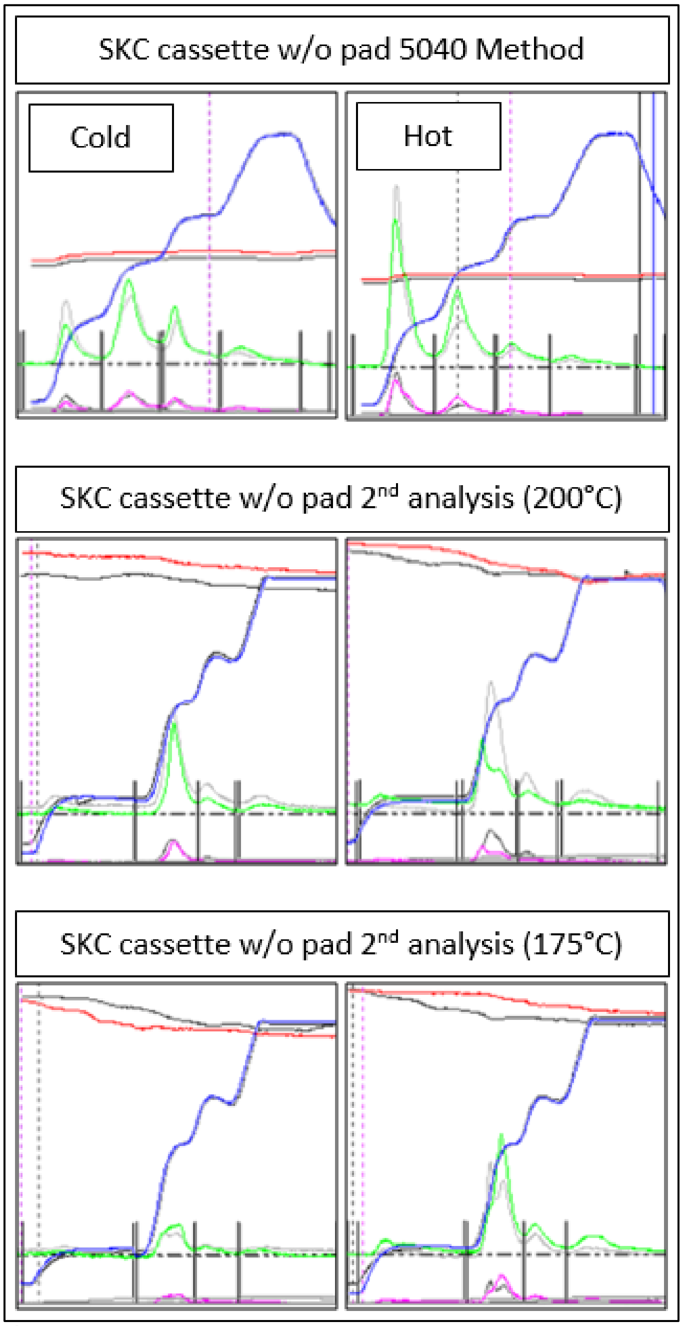

Figure 8. Representative thermograms (cropped to show only the helium region of interest) for the generic SKC cassette without support pad.

Comparing the hot versus cold storage conditions, the additional OC in the hot-conditioned samples is most often in the first peak. This is consistent with expectations since only the most volatile compound should be affected under the hot storage used here (i.e., which actually represents very mild heating compared to the temperature reached in the first peak of the 5040 Method). For the SKC cassettes with or without the cellulose pads, there was little to no OC evolution at 175°C. There was evolution at 200°C, but it was not near complete. For the SKC impactor samples, OC evolution was higher in the first peak of the modified analyses at both temperatures, though still not complete. Further work will be necessary to identify an optimal temperature to separate VOC sourced from diesel exhaust and other sources in the sampling atmosphere – on top of VOC sourced from the sampling materials themselves.

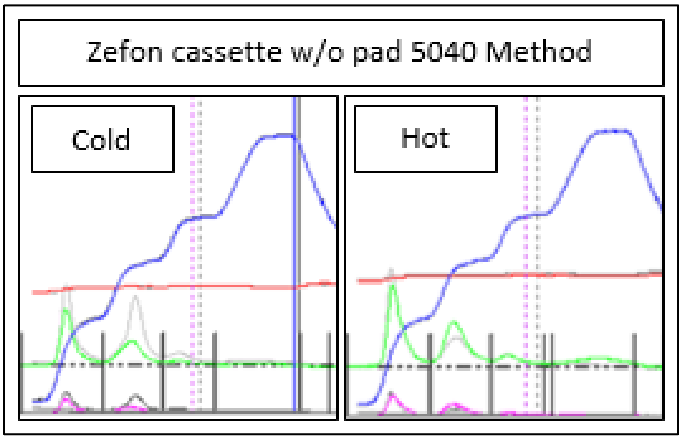

Figure 9. Representative thermograms (cropped to show only the helium region of interest) for the generic Zefon cassette without support pad.

CONCLUSIONS

From the pre-existing datasets examined here, instances of apparently negative particulate OC in tandem-filter corrected samples are generally limited to blanks. Such instances can occur when sampling materials add VOC to the filters prior to analysis. Results presented here indicate that polystyrene cassettes, cellulose support pads and the SKC DMP impactor may all contribute VOC, especially under conditions of heated or prolonged storage. In some cases, VOC sorption to the secondary filter might be preferential.

Particularly low-mass DPM samples could be significantly affected by sampling-material sourced VOC. In these cases, the particulate OC might not appear negative, but rather the interference and its related error might go undetected (i.e., perceived merely as a relatively low particulate OC result). For this reason, care should be taken to minimize the opportunity for sampling materials to contribute VOC.

ACKNOWLEDGEMENTS

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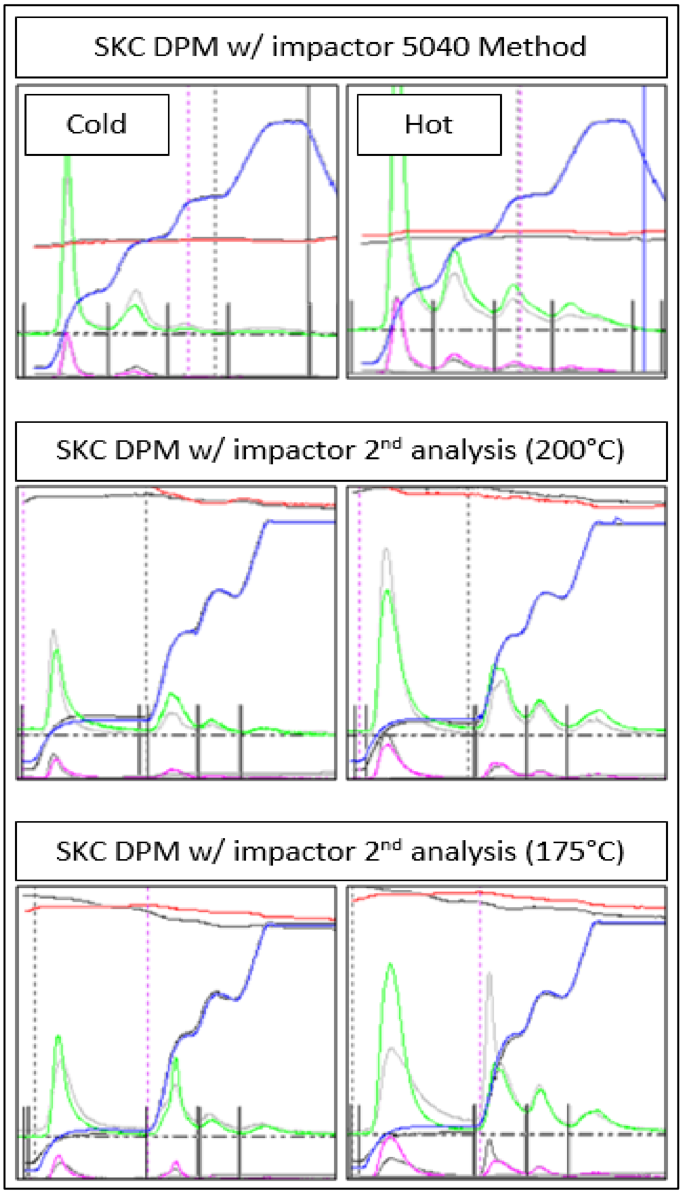

Figure 10. Representative thermograms (cropped to show only the helium region of interest) for the SKC DPM cassette with impactor.

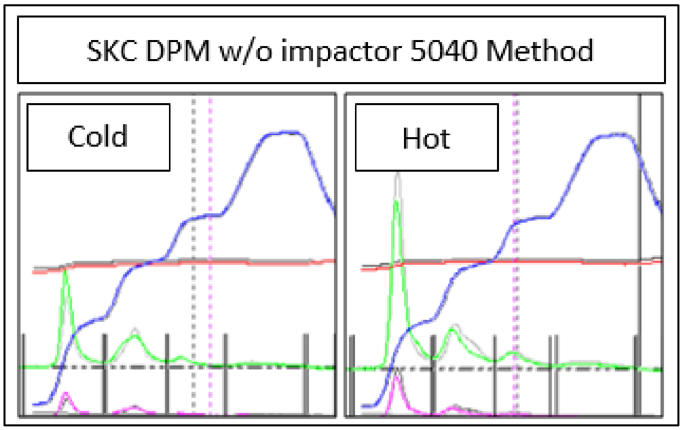

Figure 11. Representative thermograms (cropped to show only the helium region of interest) for the SKC DPM cassette without impactor. The primary (pink) and secondary (black) filter results are overlaid for comparison.