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Analytical Instrument Performance Criteria

Occupational Monitoring of Particulate Diesel Exhaust by NIOSH Method 5040

Kevin Ashley, Column Editor

Reported by M. Eileen Birch

Exposure to particulate diesel exhaust and other fine-particle pollution is of concern because studies indicate a positive association between airborne levels and respiratory illness and mortality. In the United States alone, over a million workers (e.g., trucking, mining, railroad, agriculture) are occupationally exposed to diesel exhaust, which is a suspect human carcinogen.^(1,2) Exposures in the mining industry are of particular concern because air concentrations are much higher.^(3,4) In mines, diesel exhaust levels sometimes exceed 2 mg/m³, which is 1,000 times a typical environmental level. Assessing exposure and the associated health risks is challenging because diesel exhaust is a highly complex aerosol. Methods based on determination of the particulate mass have been applied in mines, but these lack selectivity and are inadequate for low-level measurements.

A carbon analysis method based on a thermal-optical technique⁽⁵⁾ was evaluated and published as Method 5040 in the NIOSH *Manual of Analytical Methods (NMAM)*. The method was initially published in 1996, and was later updated.⁽⁶⁾ Both organic and elemental carbon (OC and EC, respectively) are determined. Carbon is a logical analyte choice, because diesel particulate matter is mostly carbon, but there are other sources of OC. Elemental carbon is a selective marker of exposure in workplaces where diesel equipment is operated. For this reason, an EC marker was recommended.^(5,6) Elemental carbon is now accepted internationally as a surrogate measure of exposure to this industrial pollutant. *NMAM*

5040 has been used in numerous industrial hygiene surveys⁽⁷⁻¹⁴⁾ and in an epidemiological study of miners. The ongoing study is being conducted jointly by NIOSH and the National Cancer Institute. This report summarizes Method 5040, as well as interlaboratory comparison results and quality assurance data. Recently proposed exposure criteria are also discussed.

Thermal-Optical Analysis

NMAM 5040 is based on a thermal-optical analysis technique for organic and elemental carbon. The thermal-optical analyzer has been described previously.^(5,6) Design improvements have since been made, but the operation principle remains unchanged. The analyzer is equipped with a pulsed diode laser and photodetector that permit continuous monitoring of the sample filter transmittance. This optical feature corrects for the "char" formed during the analysis of some materials. Like EC initially present in the sample, char is a black material that strongly absorbs light, particularly in the red/infrared region. It is formed through pyrolysis of certain organic substances when heated to elevated temperatures in an inert (nonoxidizing) atmosphere. Both volatile products and char are formed in the process, which can begin at temperatures as low as 300°C.

In the thermal-optical analysis, a portion of a quartz-fiber filter sample is placed in the sample oven, which is then tightly sealed. The analysis proceeds in inert and oxidizing atmospheres. First, OC (and carbonate, if present) is removed in helium as the temperature is increased to a preset maximum (about 870°C in *NMAM* 5040). If charring oc-

curs, a decrease in filter transmittance results as the temperature is stepped to the maximum. After removal of OC, an oxygen-helium mix is introduced to effect combustion of the remaining material. An increase in filter transmittance is seen as light-absorbing carbon is removed. The split between the organic and elemental carbon is designated at the point where the filter transmittance reaches its initial baseline value. The carbon removed prior to the OC-EC split is considered organic, and that removed after the split is considered elemental. In both atmospheres, the evolved carbon is catalytically oxidized to carbon dioxide, which is then reduced to methane and quantified with a flame ionization detector (FID). The total carbon (TC) in the sample is the sum of OC and EC. If carbonate is present, the carbon in it is quantified as OC unless a carbonate-subtracted value is requested from the laboratory. Depending on the type of carbonate, a carbonate-subtracted value is obtained through acidification of the sample before analysis, or separate integration of the carbonate peak. Normally, the carbonate content of environmental samples is negligible, but limestone and trona mines contain high levels of calcium carbonate and sodium sesquicarbonate, respectively. In mines, the Mine Safety and Health Administration (MSHA) recommends use of a size-selective impactor to minimize collection of carbonates and other carbonaceous materials.⁽¹⁵⁾

Interlaboratory Comparisons

NMAM 5040

When the thermal-optical method was initially evaluated, an interlaboratory

comparison was not possible because the instrument was only available in one laboratory. Interlaboratory comparisons are especially important with this type of analysis because variable results have been obtained with different OC-EC methods, and there is currently no reference material available. After additional laboratories acquired thermal-optical instruments, a round robin comparison⁽¹⁶⁾ was conducted. Matched sets of filter samples containing different types of complex carbonaceous aerosols were distributed to 11 laboratories. Six of the 11 analyzed the samples according to *NMAM 5040*, while five used purely thermal (i.e., no char correction) methods. Good interlaboratory agreement was obtained between the six laboratories that used *NMAM 5040*. In the analysis of samples containing diesel particulate matter, the relative standard deviation (RSD) for the EC results ranged from six to nine percent. Only low EC fractions were found in wood and cigarette smokes, so these materials pose minimal interference in the analysis of diesel-source EC. In addition, only minor amounts of EC were found in two OC standards that char: about 1 percent and 0.1 percent for sucrose and the disodium salt of ethylenediaminetetraacetic acid (EDTA), respectively. Two aqueous solutions of OC standards were included in the comparison as a check on the validity of the char correction and accuracy of the TC results. Variability (RSD) in the TC results for the two standard solutions and five filter samples ranged from three to six percent.

NMAM 5040 and Thermal Methods

Different carbon methods have given consistent agreement for TC, but OC-EC results have been quite variable, with the degree of variability depending on the sample type. In general, there is greater disagreement between *NMAM 5040* and other methods when samples contain materials that char (e.g., wood and cigarette smokes), especially with methods that employ a lower maximum

temperature and do not correct for char. When a lower temperature (typically 550°C) is used, refractory organic materials and carbonates are removed to a lesser extent. This, as well as lack of char correction, can positively bias the EC results. In the round robin study⁽¹⁶⁾ discussed above, three laboratories employed four purely thermal methods that specified a maximum in nitrogen (not helium) of about 550°C. Unlike the 5040 results, these laboratories reported high EC contents for the two OC standards (about 52% for sucrose and 70% for EDTA). Similar findings (i.e., positive bias) for thermal methods with no char correction were obtained recently in another international round robin.⁽¹⁷⁾

In contrast to round robin results obtained previously,⁽¹⁶⁾ relatively good agreement was seen in a recent comparison⁽¹⁸⁾ between *NMAM 5040* and a thermal method (ZH 1/120.44) used in Germany. The comparison was limited to two laboratories. Method ZH 1/120.44 specifies a 550°C maximum in nitrogen. The other European laboratories that participated in the previous round robin⁽¹⁶⁾ used variations of this method. In the European labs, nitrogen is used as the inert gas, and carbon determination is based on coulometric titration of carbon dioxide. For the comparison,⁽¹⁸⁾ samples were obtained in a mine where diesel equipment was being operated, and they had a much higher EC content (about 50%) than did the round robin samples. No charring was noted in the thermograms (output signal of thermal-optical instrument), and only a minor amount of carbon was removed above 550°C. Although differences in the OC-EC results were again seen, they were small relative to those obtained in the round robin.⁽¹⁶⁾ The mean EC fractions (EC/TC) found with methods ZH 1/120.44 and *NMAM 5040* were 0.53 ($\sigma = 0.19$) and 0.46 ($\sigma = 0.15$), respectively, which is typical of diesel particulate emissions in mines. The relatively minor difference in the reported fractions was attributed to the different thermal programs employed.

Alternative Thermal-Optical Method

Another thermal-optical method, called the IMPROVE (Interagency Monitoring of Protected Visual Environments) method,⁽¹⁹⁾ was also included in the 5040 round robin.⁽¹⁶⁾ Relatively good agreement between *NMAM 5040* and the IMPROVE method was obtained, although the IMPROVE EC was consistently a bit higher. The carbon analyzers used for the two methods are based on similar measurement principles, but they differ with respect to design and operation. For example, the optical correction in *NMAM 5040* is based on filter transmittance, whereas that for the IMPROVE method is reflectance-based. Different components are used to monitor these signals. The instrument (Sunset Laboratory, Inc., Forest Grove, OR) used for *NMAM 5040* incorporates a pulsed diode laser (670 nm) and photodetector positioned on opposite sides of the filter. The instrument for the IMPROVE method uses a quartz rod and fiber optic to measure He-Ne laser light (632.8 nm, unmodulated) reflected from the filter surface. In addition to instrumental differences, *NMAM 5040* specifies a higher maximum temperature (850°C) in helium than the IMPROVE method (550°C). As discussed in the preceding section, a higher temperature is used to better remove carbonates and refractory OC components. In the 5040 analysis, the transmittance of some samples continues to decrease as the temperature is stepped to 850°C, which indicates charring is not complete at 550°C. More exhaustive charring results in a lower EC result because the correction for it is larger, and because volatile pyrolysis products (OC) can evolve to a greater extent. Although environmental samples contain very little carbonate, carbonate levels in some workplaces (e.g., mines, construction sites) can be relatively high. Collection of carbonate can be minimized (or eliminated) through use of an impactor.⁽⁶⁾

Two comparisons^(20,21) between the *NMAM 5040* and IMPROVE methods have been conducted recently. In one,⁽²⁰⁾

the *NMAM* 5040 EC was typically less than half the *IMPROVE* EC, but a Sunset Laboratory instrument was not used. Instead, samples were analyzed by running two different thermal programs on a prototype instrument (GreenTek, Douglasville, GA). Results of this comparison may not be representative of those obtained in a direct comparison because other factors (differences in instrument design, oxygen contamination) may be operative. Adjustment of the temperature program according to another method's specifications does not necessarily produce the same results as that method. For example, lower EC results were obtained⁽¹⁶⁾ with Sunset's instrument when a sucrose standard was analyzed according to the temperature program specified for a method used in Europe. No pyrolysis correction was made, and the total analysis time was shorter, yet the EC result was much lower (about 3 μg with Sunset's instrument, and 11 μg with a different instrument). When the 5040 thermal program was used on the prototype (GreenTek) instrument, the filter transmittance reportedly exceeded its initial value before the addition of oxygen.⁽²⁰⁾ These results contrast with those obtained by other laboratories, and such behavior was not seen in another recent comparison.⁽²¹⁾ In that comparison,⁽²¹⁾ samples were analyzed at Sunset Laboratory and Desert Research Institute (DRI), so it was a direct comparison of the two methods. Only two of 52 samples analyzed by Sunset Laboratory showed an increase in transmittance before the addition of oxygen, and the OC-EC split for these was near the point where oxygen was added. Both samples had low carbon loadings (OC = 4.0 $\mu\text{g}/\text{cm}^2$, EC = 0.6 $\mu\text{g}/\text{cm}^2$; OC = 3.3 $\mu\text{g}/\text{cm}^2$, EC = 0.5 $\mu\text{g}/\text{cm}^2$) and were likely wood-smoke-dominated. As pyrolysis was evident, the increase in filter transmittance was attributed to char removal. Comparable EC results would have been obtained if the char had not been removed until after oxygen was added, because char is part of the OC fraction. In the 5040 analysis, partial char loss is sometimes seen with organic

compounds that char, but its removal is seldom complete before the addition of oxygen.

Early EC Loss

In the 5040 analysis, oxidation of original EC (as opposed to char) can occur during the last temperature step in helium, but this is generally uncommon. If early loss occurs, it is important to ensure that oxygen contamination is not responsible. This check can be performed by analyzing a sucrose standard solution applied to a clean, unused filter punch. Although minor char loss is sometimes observed, the filter transmittance should not reach its initial value until after oxygen is introduced. If oxygen contamination can be ruled out, the early EC loss is likely due to oxidants in the sample. To date, EC loss in helium has only been observed with samples from a few mines.⁽⁶⁾ With these particular samples, the transmittance reached its initial value during the last temperature step in helium. Sample pyrolysis was not observed, so the increase was not caused by char loss. Analysis of sucrose standards revealed only a minor increase in transmittance in helium, and no increase was seen with samples from other mines. For these reasons, oxygen contamination was ruled out. On average, about 10 percent of the EC was removed at 850°C in helium,⁽²²⁾ but the carbon removed was still included in the EC result because the OC-EC split was assigned when the initial transmittance was reached (in helium). Reducing the maximum temperature to 750°C was recommended⁽⁶⁾ for these types of samples (i.e., no pyrolysis and early splits in helium) to prevent EC oxidation in helium, which was not seen at 750°C.⁽⁶⁾ A lower temperature may be required, depending on the type and amount of internal oxidant present. Although the analytical precision was not affected by the early splits, a reduced temperature was recommended in the interest of interlaboratory precision, which could be adversely affected. Interlaboratory testing has not been conducted to determine if this is indeed the case, and, if so, whether

use of a lower temperature improves precision. Within-laboratory precision was not adversely affected by the early split.

Occupational Exposure Criteria (United States)

In 1995, the American Conference of Governmental Industrial Hygienists (ACGIH[®]) proposed a threshold limit value (TLV[®]) for diesel exhaust (see Notice of Intended Changes list for 1995–1996). A TLV of 150 μg of submicrometer particulate matter per cubic meter air was proposed. Four years later, a value of 50 $\mu\text{g}/\text{m}^3$ was proposed.⁽²³⁾ Because EC is a demonstrated exposure marker for diesel particulate exhaust and can be accurately quantified at low levels,⁽²⁴⁾ the standard was recast in terms of an equivalent elemental carbon measurement⁽²⁴⁾ in 2001. A TLV[®] TWA (time-weighted average) of 20 μg EC per cubic m air was recommended.⁽²⁵⁾

Obviously, an EC standard would simplify interpretation of the analytical results, because the target analyte (EC) and exposure standard are the same. This eliminates the problem of extrapolation (EC to submicrometer mass), which is not straightforward, because the EC content of diesel particulate matter is variable. When EC concentrations are high and samples are relatively free of OC contaminants, the EC/TC ratios can be used to estimate this fraction, but these conditions are uncommon outside of mines. In general industry, EC levels are normally much lower. When EC levels are low, the EC/TC ratios are not reliable estimates of the EC fraction of diesel particulate matter because OC interferences skew the ratios low and increase variability.

The U.S. Mine Safety and Health Administration (MSHA) has proposed an exposure standard for diesel particulate exhaust in metal and nonmetal mines.⁽³⁾ MSHA standards consider health risks as well as technical and economic feasibility (based on available engineering controls and the cost of implementing them). An interim standard of 400 μg TC per cubic m air has been proposed. This

standard goes into effect 18 months after publication of the final rule and remains in effect until five years after the publication date. Five years after this date, a lower exposure standard of 150 μg TC per cubic m air will apply.

Quality Assurance

Unfortunately, a suitable reference material for organic and elemental carbon is not yet available. A new Certified Value (17.68% g/g) for the TC mass fraction of an urban dust standard (SRM 1649a, formerly SRM 1649) was recently reported by the National Institute of Standards and Technology (NIST), but only information values are provided for the EC content. A variety of methods were used to determine the EC/TC fractions. As expected from past studies, the reported fractions are quite variable. The EC/TC fractions found by 13 methods ranged from about 7 to 52 percent, with the data being distributed in three clusters. Method bias was not evaluated. The reported range is obviously too broad to use this material as an analytical standard. According to the NIST Certificate of Analysis, the reported EC contents may be useful for comparison with results obtained by similar methods, but this may not be the case for methods with optical corrections, because filter samples are not available. Depending on its placement, bulk dust on a filter can present different optical properties, and this may increase variability.

Quality assurance (QA) procedures followed by NIOSH and contract laboratories have included repeat analyses, analysis of OC standards, and analysis of media and field blanks. Results⁽²⁶⁾ for repeat analyses of over 200 filter samples at a contract laboratory (DataChem Laboratories, Inc., Salt Lake City, UT) gave a pooled RSD (95% confidence) of about four percent for EC and TC, and five percent for OC. The samples were respirable and total dust samples collected in mines, and some contained other carbonaceous matter such as limestone. These results are quite good considering the dusty sampling environment and particle size fractions collected. In the analysis of OC

standards, recovery is typically within six percent of the expected value.⁽²⁶⁾ For example, the mean recovery for over 400 sucrose standards analyzed at DataChem over a one-year period was 99.97 ± 6.07 percent. Nearly identical results ($100.33\% \pm 5.15\%$, $n = 462$) were obtained by Clayton Laboratory Services (Novi, MI). Different sets of field blanks (41 at DataChem and 129 at Clayton) analyzed by the two laboratories also gave comparable results.⁽²⁶⁾ Mean blank results (μg carbon per filter) were as follows: DataChem OC = 9.96 ± 7.05 , Clayton OC = 12.80 ± 5.76 ; DataChem EC = -0.54 ± 1.89 , Clayton EC = -0.08 ± 2.14 ; DataChem TC = 9.26 ± 7.30 , Clayton TC = 12.70 ± 6.18 .

In lieu of an OC-EC reference material, a limited confirmation of results by a second laboratory is advisable. Our laboratory (NIOSH, Cincinnati, OH) has routinely performed repeat analyses on samples previously analyzed at a contract laboratory. As an added check, subsets of representative field samples have also been analyzed by a third laboratory. In the analysis of 50 samples (analyzed at DataChem, NIOSH, and Clayton) from mines, the pooled RSD (95% confidence) was about six percent for TC, 10 percent for EC, and 12 percent for OC.⁽²⁶⁾ These results are consistent with those found in a previous round robin.⁽¹⁶⁾

The U.S. Environmental Protection Agency (EPA) recently conducted a special study⁽²⁷⁾ as part of the QA oversight for the PM_{2.5} Speciation Trends Network (STN). Samples collected as part of this network were removed from refrigerated storage eight months after they were analyzed at Research Triangle Institute (RTI, Research Triangle Park, NC). The samples were then shipped to the EPA New England Regional Laboratory (NERL, Lexington, MA) for reanalysis. Good interlaboratory agreement was reported.⁽²⁷⁾ The acceptance criteria for the archived samples were based on filter loading ($\mu\text{g}/\text{cm}^2$). The three acceptance criteria are as follows: a difference $\leq 1 \mu\text{g}/\text{cm}^2$ for loadings less than $5 \mu\text{g}/\text{cm}^2$, a relative percent difference (RPD) ≤ 20 percent for loadings

from 5–10 $\mu\text{g}/\text{cm}^2$, and an RPD ≤ 15 percent for loadings above 10 $\mu\text{g}/\text{cm}^2$. The OC, EC, and TC data had 85, 96, and 93 percent of the interlaboratory results, respectively, within the acceptance criteria. The lower percentages for OC and TC are thought to reflect contamination of lightly loaded samples from adsorbed vapors and handling. An OC blank correction was applied, but it was not representative of all samples. An alternative approach for blank correction is discussed in the following section.

OC Blank Correction

Quartz-fiber filters are commonly used to collect airborne particulate matter for subsequent determination of its carbon content, but adsorption of organic vapor can positively bias the particulate OC (and TC) results. The amount of vapor adsorbed by traditional filter blanks (media and field) is variable,⁽²⁶⁾ and it is not representative of the samples, because blank filter media collect vapor passively while samples collect it actively (during sampling). Passive blanks underestimate the amount of adsorbed vapor, which results in overestimation of the particulate OC (and TC) concentration.⁽²⁸⁾ A more representative correction for adsorbed organic vapor can be obtained through use of two quartz filters in tandem.⁽²⁸⁾ After sampling, the bottom filter of the stacked pair is used for blank correction. The vapor adsorbed on the bottom filter more closely represents that adsorbed on the sample because both are collected actively. For sets of eight air samples collected at 2 L/min for eight hours on four different days, the mean ($n = 32$) OC on the bottom quartz filters was $2.84 (\pm 0.50) \mu\text{g}/\text{cm}^2$, with the variability (RSD) ranging from six to 10 percent on a given day. The mean carbon loading on the top filter of the stacked-quartz pair was about $4 \mu\text{g}/\text{cm}^2$ on days 1–3, and about $23 \mu\text{g}/\text{cm}^2$ on day 4 (a diesel sample). Adsorbed OC constituted about 70 percent of the TC found on days 1–3, and 14 percent of that found on day 4 (diesel sample). The passive blank loading was much lower, with a mean OC

ranging from 0.25 to 0.74 $\mu\text{g}/\text{cm}^2$ over the four days. These results indicate that a backup quartz filter provides a better correction for the positive artifact caused by adsorption of organic vapor. Additional information is provided in a recent *NMAM* 5040 update.⁽²⁸⁾

Summary

NMAM 5040 is a particulate carbon method based on a thermal-optical analysis technique.⁽⁵⁾ The method was evaluated and published as a method for monitoring occupational exposures to particulate diesel exhaust, but it is applicable to particulate carbon aerosols in general, and has been routinely used in both occupational and environmental settings. Both organic and elemental carbon are determined, but EC is a more selective measure of workplace diesel exposure.^(5,6) In previous studies, good agreement between TC results obtained by different methods has been achieved, but the OC-EC results for different methods have been quite variable. Although a reference material is not currently available to test the accuracy of different methods, previous studies^(16,17) indicate that purely thermal methods are subject to positive bias from organic materials that char. Charring and inadequate removal of refractory OC components during the nonoxidative mode (typically 550°C in nitrogen) likely explain the positive bias of thermal methods, as well as the large variability across methods. These interferences may be negligible in some cases (e.g., samples from mines), but they present significant biases in others (e.g., urban air samples, samples containing wood or cigarette smokes). Good interlaboratory agreement was obtained in a round robin comparison between six laboratories that used *NMAM* 5040, which was not the case with purely thermal methods. Good agreement has also been seen in smaller-scale comparisons conducted for quality assurance purposes. Until a suitable reference material becomes available, such comparisons are recommended as part of a laboratory's QA procedures. At present, five

commercial laboratories (4 in the United States and 1 in Canada) perform the 5040 analysis, and over 40 instruments are in use globally for environmental and occupational monitoring.

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