https://doi.org/10.5194/egusphere-2026-1213
Preprint. Discussion started: 29 April 2026

Reducing uncertainties in elemental carbon quantification using solvent-extraction–based mass balance and temperature adjustment in thermal–optical protocols

Sumin Ok[1], JiYi Lee[2], Naeun Kim[1], Jinsang Jung[1*]

[1]Gas Metrolgy Group, Korea Research Institute of Standards and Science (KRISS), Daejeon 34113, Republic of Korea
[2]Department of Environmental Engineering, Ewha Womans University, Seoul 03760, Republic of Korea

Correspondence to: Jinsang Jung (jsjung@kriss.re.kr)

Abstract. This study constrains protocol-dependent uncertainty in elemental carbon (EC) quantification by thermal–optical analysis (TOA) using a novel solvent-extraction-based mass balance framework. To eliminate organic particulate matter interference, PM2.5 samples underwent sequential water and organic solvent extraction. A backup filter was strategically employed to account for EC redistribution during the extraction process, which was found to involve 37 ± 6 % of the total EC. The resulting solvent-extracted EC, corrected for redistribution, served as an operational reference largely independent of thermal charring artifacts. Comparative analysis revealed that EC determined by the IMPROVE protocol was consistently higher, whereas the default NIOSH protocol yielded systematically lower values than the reference. By reducing the maximum OC analysis temperature (OC4) in the NIOSH protocol to 650 °C, the EC values showed improved agreement with the reference (ratio = 1.08 ± 0.13). Furthermore, a logarithmic regression of the solvent-extracted EC to bulk EC ratio as a function of OC4 temperature identified a unity condition at 615 °C, defined here as the "KRISS temperature". This framework provides a robust, reproducible basis for OC4 temperature selection and enhances inter-protocol comparability by explicitly constraining protocol-dependent uncertainties.

1 Introduction

Particulate matter (PM) adversely affects human health and is a major climate forcer. Carbonaceous aerosols typically constitute 10–50 % of PM mass (Pio et al., 2001; Putaud et al., 2004; Yttri et al., 2007; Putaud et al., 2010; Karanasiou et al., 2011; Karanasiou et al., 2020). They are commonly grouped into organic aerosols (OA), elemental carbon (EC), and carbonate carbon (CC), each exerting distinct radiative impacts. EC absorbs solar radiation and can enhance snow/ice melt when deposited by lowering surface albedo (Jacobson, 2001; Sato et al., 2003; Jacobson, 2010; Bond et al., 2013; Doherty et al., 2013), whereas OA predominantly scatters light and generally cools the atmosphere (Kopp and Mauzerall, 2010). Robust quantification of carbonaceous components is therefore essential for air-quality assessment and climate-related applications.
EC is primarily emitted from incomplete combustion of fossil fuels and biomass, while OA includes both primary emissions and secondary formation through atmospheric oxidation of volatile organic compounds followed by condensation of low-

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volatility products (Malaguti et al., 2013). CC is mainly associated with mineral dust and is often negligible outside dust-influenced environments (Karanasiou et al., 2011; Wu et al., 2012). Because OA comprises a complex mixture of numerous compounds, it is commonly characterized operationally as organic carbon (OC), while EC is likewise operationally defined by the analytical method used.

Thermal–optical analysis (TOA) remains the most widely used approach for separating and quantifying OC and EC on quartz filters. However, OC/EC partitioning is sensitive to protocol-dependent temperature settings and optical charring corrections, which influence pyrolyzed organic carbon formation and the OC/EC split point. Among TOA protocols, IMPROVE and NIOSH are the most widely applied globally, and EUSAAR (EUSAAR2) was developed to address limitations of earlier protocols. A key distinction is the maximum temperature applied during the OC phase: up to 870 °C in NIOSH versus 580 °C in IMPROVE, with EUSAAR2 adopting an intermediate value (~650 °C). Such differences can cause substantial protocol-dependent biases; intercomparison studies have reported OC/EC ratio differences of up to several-fold between NIOSH and IMPROVE, with discrepancies attributed mainly to thermal effects and, to a lesser extent, optical correction methods (Chow et al., 2004; Bautista et al., 2015; Wu et al., 2016; Oh et al., 2018). These effects depend on aerosol composition, aging, biomass-burning influence, and metal oxides (Chow et al., 2001; Piazzalunga et al., 2011; Cheng et al., 2014; Wu et al., 2016). Recent work continues to highlight the need for improved uncertainty characterization and protocol optimization: an interlaboratory study quantified TOA reproducibility-related expanded uncertainties on the order of ~10–20 % for EC/OC/TC under controlled conditions, underscoring the importance of protocol- and laboratory-dependent effects (Sipkens et al., 2024).

Despite extensive intercomparisons of thermal–optical protocols, constraining EC remains challenging because EC is operationally defined and sensitive to protocol-specific temperature settings and charring correction. In this study, we address this limitation by independently constraining EC using a solvent-extraction-based mass balance approach prior to thermal–optical analysis. Sequential removal of water-soluble and water-insoluble organic material, combined with correction for EC redistribution using a backup filter, provides an operational reference EC that is largely independent of thermal artifacts. By systematically varying the OC analysis temperature in the IMPROVE and NIOSH protocols and applying regression-based analysis, we quantitatively evaluate temperature-dependent bias and define an operational OC4 temperature for improved EC comparability. This work focuses on protocol optimization rather than redefining EC chemically, consistent with the scope of Atmospheric Measurement Techniques.

2 Experimental Methods

2.1 Atmospheric aerosol sampling

$PM_{2.5}$ samples were collected at a suburban site in Daejeon, Korea (36°23′ N, 127°22′ E), on the rooftop of a chemistry building at the Korea Research Institute of Standards and Science (KRISS). Twenty-four-hour integrated samples were

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collected from 31 March to 13 April 2014 using a high-volume aerosol sampler (TE-6070XZ-2.5-HVS, Tisch Environment Inc., USA) operated at a flow rate of 67.8 m^3 h^{-1}. Aerosols were collected on pre-baked quartz fiber filters (20 × 25 cm, Pall Life Sciences, USA).

Quartz fiber filters were heated to 550 °C for 12 h to remove adsorbed impurities before sampling. Following sampling, filters were wrapped in aluminum foil and stored at −20 °C prior to analysis. A total of 14 $PM_{2.5}$ samples were collected, along with field blanks obtained before and after the sampling campaign to assess background contamination.

2.2 OC/EC analysis by thermal-optical method

Organic carbon (OC) and elemental carbon (EC) were quantified using a thermal–optical carbon analyzer (Sunset Laboratory Inc., Model RT-3140) with the thermal–optical transmittance (TOT) method for charring correction. A 1.5 cm^2 punch from each quartz filter was analyzed. External calibration was performed using known amounts of sucrose.

OC/EC analyses were conducted using both the IMPROVE-A and NIOSH 5040 temperature protocols. In order to investigate the influence of temperature settings on EC quantification, the maximum temperature applied during the OC analysis phase (OC4) was systematically modified. For the NIOSH protocol, the default OC4 temperature of 870 °C was decreased stepwise to 650 °C. All other protocol parameters were kept unchanged. The detailed temperature profiles are summarized in Table 1. OC and EC were quantified using the thermal–optical transmittance approach following established principles described by Birch and Cary (1996) and Chow and Watson (1998), and protocol-dependent effects were evaluated based on previous intercomparison studies (Chow et al., 2004; Wu et al., 2016).

Table 1: Temperature profile of thermal optical transmittance method.

Step	Carrier Gas	Oven temperature (°C)					
		He4-580 (IMPROVE)	He4-650	He4-700	He4-750	He4-800	He4-870 (NIOSH)
OC1	He	140	310	310	310	310	310
OC2	He	280	475	475	475	475	475
OC3	He	480	615	615	615	615	615
OC4	He	580	650	700	750	800	870
	He				Cool oven		
EC1	O_2/He	580	550	550	550	550	550
EC2	O_2/He	740	625	625	625	625	625
EC3	O_2/He	840	700	700	700	700	700
EC4	O_2/He		775	775	775	775	775
EC5	O_2/He		850	850	850	850	850

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2.3 Solvent extraction procedure (concise)

Sequential solvent extraction was applied to remove organic particulate matter from quartz filter samples prior to thermal–optical analysis. Filter punches (1.5 cm^2) were placed in a custom-designed filter holder to maintain mechanical stability during immersion. Samples were first immersed in ultrapure water to remove water-soluble organic components, followed by extraction with a mixed organic solvent (dichloromethane:methanol, 2:1, v/v) to remove water-insoluble organics. A brief immersion in dichloromethane was subsequently applied to remove residual polar compounds.

To account for potential redistribution of particulate material during extraction, a backup quartz filter was positioned downstream of the sample filter within the holder. After extraction, both sample and backup filters were purged with nitrogen and dried at 120 °C to ensure complete removal of residual solvents prior to OC/EC analysis. Further evaluation of solvent interference, EC redistribution, and the role of the backup filter is presented in Sect.3.

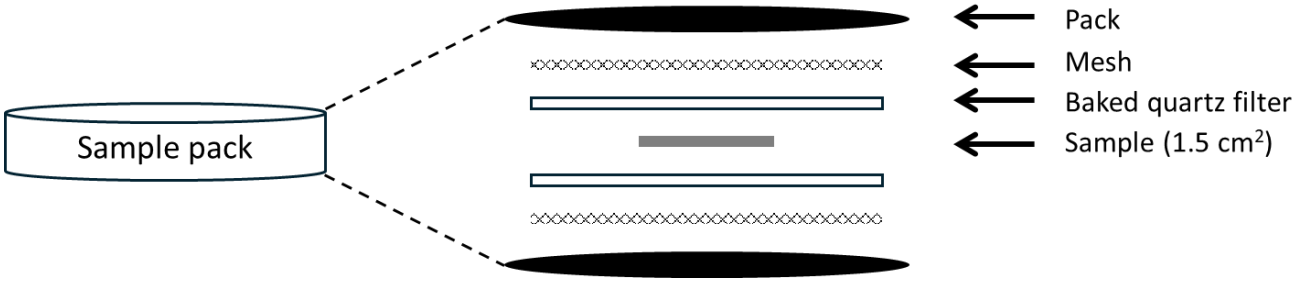

Figure 1: Schematic diagram of the filter holder used to correct EC redistribution during solvent extraction.

3 Results and Discussion

3.1 Establishment of an operational reference EC using solvent extraction

Thermal–optical EC quantification is inherently operational and sensitive to artifacts arising from incomplete removal of organic material and charring during OC analysis. To independently constrain EC, sequential solvent extraction was applied prior to analysis, and potential solvent-related interference was first evaluated using blank quartz filters.

Blank filter experiments showed that drying at temperatures ≤ 100 °C was insufficient to fully remove residual organic solvents, resulting in positive OC artifacts on the order of 0.4–0.9 μg cm^{-2} after pretreatment. Extending the drying time at these temperatures did not substantially reduce the residual OC, indicating incomplete solvent volatilization during subsequent thermal–optical analysis. By contrast, under optimized conditions combining sequential solvent extraction, nitrogen purging, and drying at 120 °C for 60 min, the difference in OC measured before and after pretreatment was reduced

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to approximately 0.1 μg cm^{-2}, which is within the analytical uncertainty of the thermal–optical measurement. These results confirm that solvent-related artifacts were effectively minimized.

Although elemental carbon (EC) is generally regarded as insoluble in water and organic solvents, substantial redistribution of EC was observed during the solvent extraction process. On average, 37.4 ± 6.4 % of total EC was recovered on a backup filter positioned downstream of the sample filter. Similar sensitivity of EC quantification to pretreatment has been reported in previous studies examining removal of water-soluble components prior to thermal–optical analysis (Chow et al., 2001; Piazzalunga et al., 2011).

This redistribution does not indicate chemical dissolution of EC, but rather suggests physical detachment of loosely bound carbonaceous material during immersion. Such material may include weakly aggregated soot, hydrophilic brown carbon, or charred organic material exhibiting EC-like optical properties, which are more susceptible to operational definitions and analytical conditions than refractory soot cores (Bond et al., 2013; Wu et al., 2016). This behavior is consistent with the well-recognized operational nature of EC determined by thermal–optical methods (Chow et al., 2004).

To ensure EC mass balance closure, EC measured on both the sample and backup filters was incorporated. Figure 2 shows the temporal distribution of EC recovered on each filter, demonstrating consistent EC recovery on the backup filter across samples. Incorporation of the backup filter therefore proved essential for correcting EC redistribution and for establishing a robust operational reference EC for subsequent protocol comparisons, directly addressing one of the major sources of uncertainty in thermal–optical EC measurements (Chow et al., 2004; Sipkens et al., 2024).

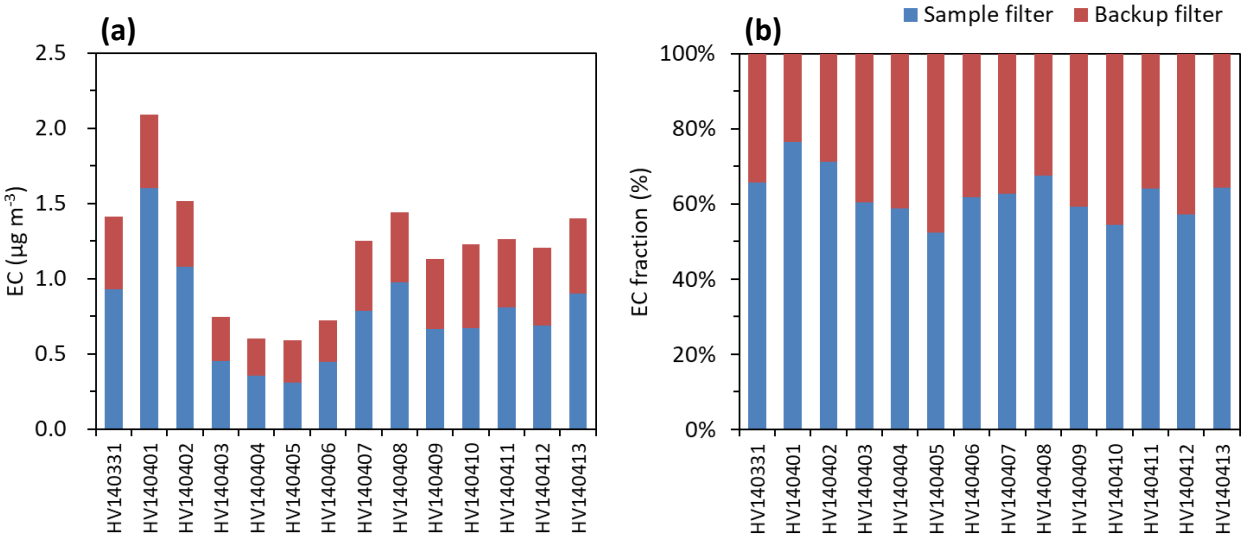

Figure 2: (a) Temporal variation of EC mass concentration on the sample and backup filters after the solvent extraction. Their relative contribution is also shown in (b).

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3.2 Effect of organic removal on thermal–optical EC evolution

Removal of organic particulate matter through solvent extraction resulted in pronounced changes in thermal–optical thermograms. Comparison of thermograms obtained under different extraction conditions (Fig. 3) shows that organic solvent extraction alone primarily reduced lower-temperature OC fractions (OC1–OC3), whereas ultrapure water extraction removed substantial portions of both low-temperature OC and part of the higher-temperature OC fraction.

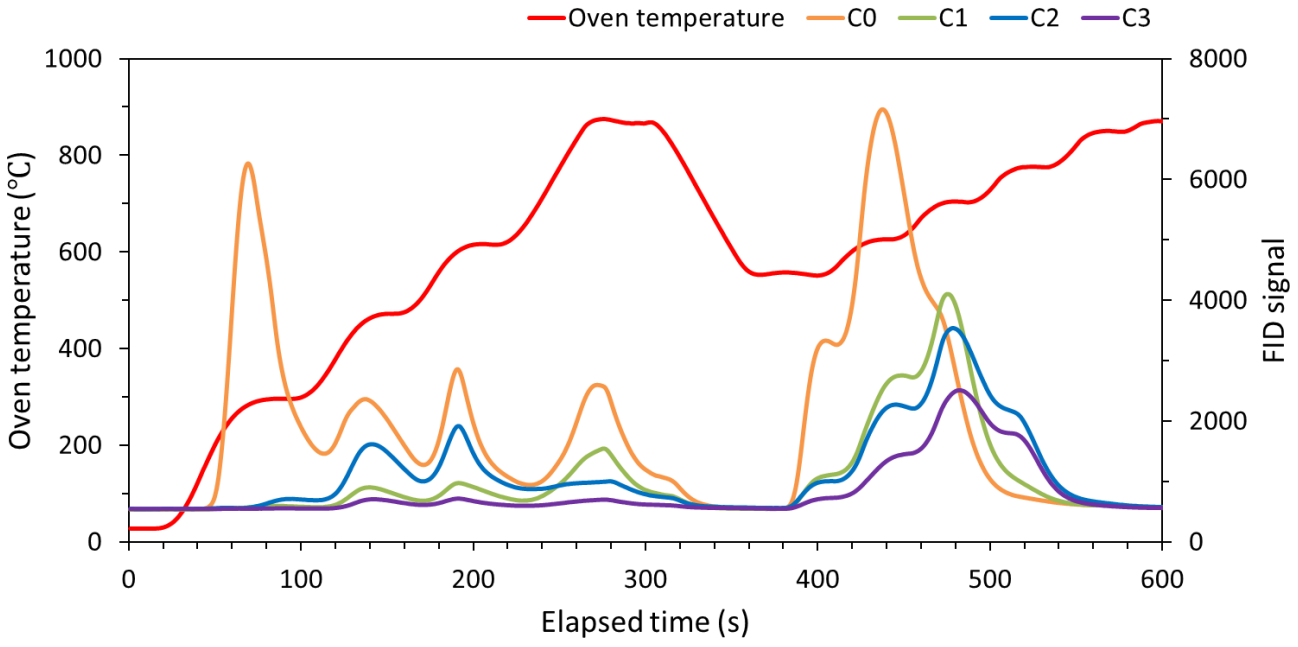

Figure 3: Thermal–optical thermograms of the field sample obtained under different extraction conditions. C0 represents the untreated sample; C1 and C2 correspond to solvent-only and water-only extraction, respectively; C3 represents sequential water extraction followed by solvent extraction.

When water and organic solvents were applied sequentially, FID signals were markedly reduced across all OC temperature segments, indicating the most effective removal of organic particulate matter. These results demonstrate that water-soluble and water-insoluble organic components contribute differently across OC temperature ranges and that neither extraction alone is sufficient for comprehensive OC removal.

In contrast, EC thermograms were broadly similar across extraction conditions, indicating that the refractory EC fraction was not fundamentally altered by the extraction procedures. However, compared with untreated samples, solvent-extracted samples exhibited EC evolution at systematically higher temperatures. This shift indicates reduced interference from thermally unstable organic carbon and charred material, which otherwise contribute to apparent EC at lower temperatures in untreated samples. Minor differences in EC evolution at high temperatures (> 700 °C) between water-only and solvent-only

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pretreatments further suggest that water extraction is more effective at removing certain thermally stable organic fractions that may overlap with EC.

The final pretreatment procedure applied in this study is summarized below.

- Cut the quartz filter into 1.5 cm^2 sections.
- Place the filter section into a dedicated filter holder.
- Immerse the filter in 50 mL ultrapure water for 30 min.
- Immerse the filter in 100 mL mixed organic solvent (DCM:MeOH = 2:1, v/v) for 30 min, repeated twice.
- Immerse the filter in 50 mL DCM for 1 min.
- Dry the sample and backup filters together at 120 °C for 60 min under a nitrogen atmosphere to remove residual solvents.
- Analyze the dried filters using thermal–optical carbon analysis.

3.3 Temperature-dependent bias in thermal–optical EC quantification

Using the operational reference EC derived from solvent-extracted samples, EC quantified from untreated samples using the IMPROVE and NIOSH 5040 protocols was systematically evaluated. EC concentrations measured using the IMPROVE protocol were consistently higher than the operational reference, whereas those obtained using the default NIOSH protocol were systematically lower, consistent with previous intercomparison studies (Chow et al., 2004; Wu et al., 2016). Despite these systematic offsets, EC concentrations from both protocols showed good correlations with solvent-extracted EC (R^2 = 0.97 for IMPROVE and R^2 = 0.98 for NIOSH), indicating that the discrepancies arise primarily from protocol-dependent effects rather than random variability.

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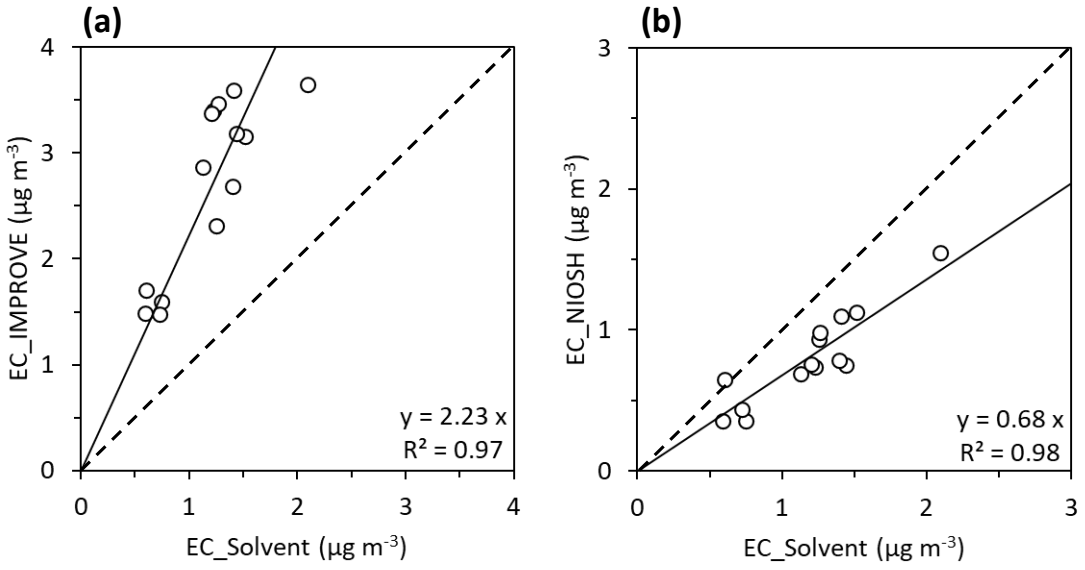

Figure 4: Scatter plots comparing elemental carbon (EC) mass concentrations obtained from untreated samples analyzed using (a) the IMPROVE and (b) the NIOSH protocols with operational reference EC derived from solvent-extracted samples. The solid lines indicate linear regressions.

Relative to the operational reference, EC concentrations obtained using the default NIOSH protocol were, on average, 0.68 ± 0.15 times lower, while those obtained using the IMPROVE protocol were 2.34 ± 0.38 times higher. These differences are largely attributable to thermal effects associated with the OC/EC split point and its sensitivity to the maximum OC analysis temperature (Chow et al., 2004; Cheng et al., 2014).

To further investigate this dependence, EC concentrations obtained after solvent extraction were compared with those analyzed using modified NIOSH protocols in which the maximum OC analysis temperature (OC4) was systematically varied (Table 2). As the OC4 temperature decreased from 870 °C to 650 °C, bulk EC concentrations increased and progressively converged toward the solvent-extracted EC. At an OC4 temperature of 650 °C, the ratio between solvent-extracted EC and bulk EC approached unity (1.08 ± 0.13), indicating the closest agreement among the tested temperature settings.

Table 2: Elemental carbon (EC) mass concentrations obtained using the IMPROVE protocol, modified NIOSH protocols, and solvent-extracted samples, together with ratios of modified NIOSH EC to solvent-extracted EC.

Temperature protocol	EC (µg m^{-3})	Solvent extracted EC/EC bulk ratio
Solvent extracted	1.19 ± 0.41 (0.59 - 2.10)	1
IMPROVE(default)	2.71 ± 0.83 (1.48 - 3.65)	0.38 ± 0.07
NIOSH650	1.12 ± 0.40 (0.53 - 1.94)	1.08 ± 0.13

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NIOSH700	0.98 ± 0.33 (0.48 - 1.51)	1.22 ± 0.16
NIOSH750	0.92 ± 0.34 (0.40 - 1.55)	1.31 ± 0.19
NIOSH800	0.83 ± 0.33 (0.38 - 1.65)	1.47 ± 0.23
NIOSH870(default)	0.80 ± 0.33 (0.35 - 1.55)	1.54 ± 0.30

3.4 Regression-based determination of the KRISS temperature

A quantitative relationship between the solvent-extracted EC to bulk EC ratio and the OC4 temperature enables an objective determination of an optimized OC analysis temperature. The ratio of solvent-extracted EC to bulk EC quantified using modified NIOSH protocols increased monotonically from 1.08 ± 0.13 at 650 °C to 1.54 ± 0.30 at 870 °C, indicating progressive underestimation of bulk EC with increasing OC4 temperature.

Logarithmic regression between the ratio and OC4 temperature yielded a unity condition at 615 °C (Fig. 5), corresponding to the temperature at which bulk EC quantified by thermal–optical analysis is, on average, equivalent to solvent-extracted EC corrected for redistribution. Based on this regression-based criterion, 615 °C is defined in this study as the KRISS temperature, representing an operationally optimized OC4 temperature for EC quantification under the investigated aerosol conditions.

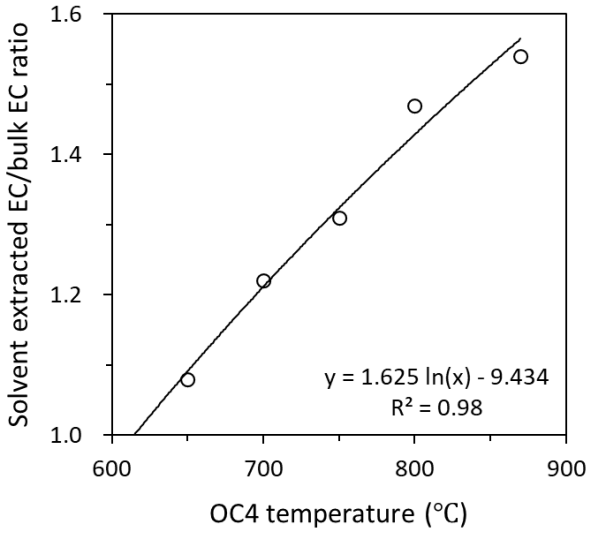

Figure 5: Dependence of the solvent-extracted EC to bulk EC ratio on the maximum OC analysis temperature (OC4) and the solid line shows a logarithmic regression fit (ln function). The regression identifies an OC4 temperature of approximately 615 °C at which the ratio equals unity, corresponding to the KRISS temperature defined in this study.

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Although the commonly applied OC4 temperature of 650 °C provides EC concentrations close to the operational reference, the regression analysis indicates that it slightly overestimates EC relative to the unity condition. The KRISS temperature therefore constitutes a quantitatively defined reference point derived from solvent-extraction-based constraints rather than protocol convention alone. This approach provides a reproducible framework for temperature adjustment and highlights the critical role of OC analysis temperature in controlling protocol-dependent uncertainty in EC quantification (Wu et al., 2016; Sipkens et al., 2024).

4 Conclusion

This study constrains protocol-dependent uncertainty in elemental carbon (EC) quantification by thermal–optical analysis using a solvent-extraction-based operational framework. Sequential water and organic solvent extraction, combined with a backup filter, enabled correction of EC redistribution during pretreatment (37.4 ± 6.4 % of total EC) and provided an operational reference EC that is largely independent of charring artifacts. Comparisons against this reference revealed systematic protocol-dependent biases, with EC quantified by the IMPROVE protocol consistently higher and EC quantified by the default NIOSH protocol systematically lower. Sensitivity analysis demonstrated that EC quantification is strongly controlled by the maximum OC analysis temperature (OC4), with 650 °C yielding the closest empirical agreement with the operational reference (1.08 ± 0.13).

Further quantitative constraint was obtained through regression analysis of the solvent-extracted EC to bulk EC ratio as a function of OC4 temperature. This analysis identified a unity condition at 615 °C, defined here as the KRISS temperature, representing an operationally optimized OC4 temperature derived from solvent-extraction-based EC mass balance rather than protocol convention alone. While not universally optimal, the proposed framework provides a reproducible basis for OC4 temperature selection and improves inter-protocol comparability in thermal–optical EC measurements by explicitly accounting for protocol-dependent uncertainty.

Data availability

The data sets used in this study are available from the corresponding author upon reasonable request.

Author contributions

Jung, J. and Ok, S. wrote the manuscript. Lee, J.Y. revised the manuscript. Ok, S. and Kim, N. collected the aerosol samples and performed the analyses.

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Competing interests

The authors declare that they have no conflict of interest.

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Acknowledgements

The authors acknowledge the Korea Research Institute of Standards and Science (KRISS) for providing the sampling site and the research infrastructure of the Division of Chemical and Material Metrology. We also thank the members of the Gas Metrology Group for their technical support and helpful discussions throughout the study.

Financial support

This work was supported by the Korea Research Institute of Standards and Science (KRISS) under the Basic R&D Project (Grant No. 26011043).

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