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Effects of temperature parameters on thermal-optical analysis of organic and elemental carbon in aerosol

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Abstract Thermal–optical analysis (TOA) is a popular method to determine aerosol elemental carbon (EC) and organic carbon (OC) collected on quartz fiber filter. However, temperature protocol adopted in TOA has great effects on OC and EC results. The purpose of this study is to investigate and quantify the effects of maximum temperature (T_{max}) and residence time (RT) for each step in helium stage on ECOC measurements. Fourteen typical source samples and 20 ambient samples were collected and six temperature programs were designed for this study. It was found that EC value decreases regularly as T_{max} ascends, i.e., EC results from T_{max} of 650°C, 750°C and 850°C are 0.89 ± 0.06, 0.76 ± 0.10, 0.62 ± 0.13 times EC value from T_{max} of 550°C, respectively, and the magnitude of EC drop (EC_d, percent) is significantly correlated with OC abundance in total carbon ($R_{OC/TC}$), expressed as $EC_d = 66.8R_{OC/TC} - 14.4$ (r = 0.87); pyrolized OC (POC) values are also sensitive to T_{max}, but there are various trends for samples with different OC constituents. On average of the samples studied here, prolonged RT reduces EC values by only 3%, almost negligible compared to the effect of T_{max}, and reduces POC by 9%, much less than that by previous report.

Keywords Thermal–optical analysis · Temperature · Elemental carbon · Organic carbon

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Introduction

Thermal–optical analysis (TOA) is a popular and commercially available technique in determination of ambient elemental carbon (EC) and organic carbon (OC) collected on quartz-fiber filters (QFFs). The basic premise of this method is that OC is volatilized from a sample when heated in the absence of oxygen while oxygen is required for EC evolution (Chow et al. 1993, 2001; Birch and Cary 1996). During TOA operation, a punch from a filter sample is inserted into the carbon analyzer and heated gradually to a special temperature (typically 550°C or 850°C) in a

pure helium atmosphere, resulting in volatilization of OC in the sample. Then temperature drops and the punch is reheated stepwise to near 900°C in oxygen-containing atmosphere (usually 2% oxygen and 98% helium) to burn out all carbon remained. The carbon evolved in each temperature step is converted to methane and measured by a flame ionization detector (FID). At the end of analysis, a known volume of pure methane is analyzed as calibration standard to quantify the carbon peaks. Some OC is pyrolytically converted to EC (char) when heated in inert atmosphere, and makes the filter darker. For charring correction, a He–Ne laser beam is introduced to monitor the sample transmittance (or reflectance) throughout the heating process. The time when the laser signal returns to its initial value (before heating) is called the split point of EC/OC, and char is considered to split from native EC in the sample at this critical point (Chow et al. 1993, 2001; Birch and Cary 1996).

There are two typical instruments for thermal/optical analysis of EC and OC, which adopt different thermal protocols (i.e., IMPROVE and NIOSH; Chow et al. 1993; Birch and Cary 1996). Intercomparisons showed that high variability existed for EC and OC results from these two methods, although there was a good agreement for total carbon (TC, sum of EC and OC; Chow et al. 2001; Schauer et al. 2003). Maximum temperature (T_{max}) and residence time (RT) of each temperature plateau in He atmosphere are the most important parameters for the great differences of EC and OC values (Conny et al. 2003). T_{max} is 550°C in IMPROVE method while 850°C or higher in NIOSH method, and there is disagreement about the carbon evolved after T_{max} goes beyond 550°C (Chow et al. 2001, 2004; Yu et al. 2002; Schauer et al. 2003). Unfortunately, there is still no EC standard material that represents EC in ambient samples to help solve this argument. Furthermore, charring of some OC during thermal evolution makes the split of EC/OC more complicated, because char always bears different absorptivity from native EC on the filter (Yu et al. 2002; Huntzicker et al. 1982). It has been found that char formation during TOA is related with RT and organic constituents (Yang and Yu 2002; Yu et al. 2002).

The purpose of this study is to perform further investigation of the effects of T_{max} and RT on ECOC measurements during TOA. Totally 34 PM2.5 samples (particles with an aerodynamic diameter of 2.5 μm or less) were collected for this study, which covered a great variation of OC composition and EC/TC ratios. Six temperature programs with different T_{max} and RT settings for TOA were designed to compare the ECOC values of these samples. This investigation will provide further information for understanding thermal/optical ECOC results.

Experimental section

Sample collection

The samples used for ECOC analyses were collected on 20 × 25 cm QFFs (Whatman) using Hi-Vol sampler (Andersen Instruments Inc., Smyrna, GA, USA). This sampler has an impactor inlet of 2.5 μm cut size at the flow rate of 1.13 m^3/min. All QFFs were baked at 550°C for 5 h prior to sampling and kept in prebaked aluminum foil envelopes. As listed in Table 1, there are 14 source emission samples and 20 ambient samples used in this study. The source samples include some important primary contributors to ambient particle concentrations, such as wood burning, vehicle exhaust, domestic coal combustion, cooking, etc. (Zheng et al. 2005; Schauer et al. 1996), and cover a wide range of carbonaceous characteristics. Samples S1–11 were taken in a special room designed for smoke sampling (Chen et al. 2005), while S12–14 were collected downward the emission flues. The ambient samples were collected in the urban areas of Shanghai (S15–28) and Guangzhou (S29–34), China. The weights of particles collected on the QFFs were obtained by weighing the filters before and after sampling under the same conditions (25°C, 50% RH, 24 h). All samples were stored in a refrigerator (−40°C) until ECOC measurements.

ECOC analysis

EC and OC fractions of all samples were determined using a thermal-optical carbon analyzer

Table 1 Samples used for this study

Source samples		Ambient samples[a]	
S1	Anthracite coal burning[b]	S15	SH-JD-20051012
S2	Raw bituminous smoke[b]	S16	SH-JD-20051019
S3	Bituminous briquette smoke[b]	S17	SH-JD-20051022
S4	Wood burning[b]	S18	SH-JD-20051026
S5	Plastic burning[b]	S19	SH-JD-20051030
S6	Tyre burning[b]	S20	SH-JD-20051102
S7	Paper combustion[b]	S21	SH-JD-20060117
S8	Rice straw burning[b]	S22	SH-JD-20060408
S9	Charcoal burning[b]	S23	SH-JD-20060726
S10	Cigarette smoke[c]	S24	SH-JD-20060805
S11	Idle diesel vehicle exhaust[d]	S25	SH-YC-20051012
S12	Road vehicle exhaust[e]	S26	SH-YC-20051015
S13	Charbroiling exhaust[f]	S27	SH-YC-20060217
S14	Cooking exhaust[g]	S28	SH-YC-20060423
		S29	GZ-TH-20070110
		S30	GZ-TH-20070111
		S31	GZ-TH-20070113
		S32	GZ-TH-20070114
		S33	GZ-TH-20070115
		S34	GZ-TH-20070117

[a]Twenty-four-hour average samples, collected in Jiading district of Shanghai (SH-JD), Yangchang district of Shanghai (SH-YC) and Tianhe district of Guangzhou (GZ-TH)

[b]Burn carbonaceous materials in our closed sampling room

[c]Smoke cigarette in our closed sampling room

[d]Duct the tailpipe exhaust of an idle diesel vehicle (Eveco, manufactured in Nanjing, China) into our closed sampling room

[e]Roadside of Guangyuan expressway, Guangzhou, with a traffic volume of about 8,000 per hour and diesel vehicles accounting for 20%

[f]Downwind a street barbecue

[g]Downwind the exhaust window of a restaurant

(Sunset Laboratory Inc., Forest Grove, OR, USA). This instrument has a temperature- and atmosphere-controlled oven and a transmittance-monitoring laser at the wavelength of 680 nm to generate an operational EC/OC split (Birch and Cary 1996). Six temperature programs were performed in this study (Table 2), which differed with each other in T_{max} and/or RT in pure He atmosphere. Four programs (T-550, T-650, T-750, and T-850, refer to different T_{max} settings of 550°C, 650°C, 750°C, and 850°C, respectively) were designed to study the effects of T_{max} on ECOC results and pyrolyzed OC (POC), among which T-550 acted as a base program with step temperatures in He stage the same as IMPROVE protocol but step durations fixed to 150 s (Chow et al. 1993). The other two programs with different residence times of temperature steps (RT-short, RT-long) were used to investigate the impacts of temperature steps' duration on ECOC measurement and charring. These two programs are similar to NIOSH (Birch 1998) and UST-3 method (Yu et al. 2002), respectively.

Quality control

Duplicate punches (1.5 cm^2) from each QFF sample were cut for ECOC analysis by the six temperature programs. Before ECOC measurement, the carbon analyzer was calibrated carefully using a blank QFF punch and a sucrose standard solution (5.0 μgC/μl, spiked 10 μl onto prebaked clean QFF using syringe). The helium used was of ultrahigh purity and passes through an oxygen- and moisture-trap before entering the analyzer.

A serial concentration (from 0.1 μgC/μl to 25.0 μgC/μl) of standard solutions of ethylene diamine tetraacetic acid (EDTA) was prepared to determine the instrumental constant (related to the actual amount of methane injected as internal

Table 2 Temperature programs used in this study

Atmosphere	T-550		T-650		T-750		T-850		RT-short		RT-long	
	RT/s	T/°C	RT/s	T/°C	RT/s	T/°C	RT/s	T/°C	RT/s	T/°C	RT/s	T/°C
He	150	120	150	120	150	120	150	120	60	250	150	250
He	150	250	150	250	150	250	150	250	60	500	150	500
He	150	450	150	450	150	450	150	450	60	650	150	650
He	150	550	150	550	150	550	150	550	90	850	150	850
He			150	650	150	650	150	650				
He					150	750	150	750				
He							150	850				

Temperature settings for O_2-containing stage are: 45 s, 550°C; 60 s, 650°C; 60 s, 750°C; 80 s, 850°C; 40 s, 870°C

standard and the conversion ratio of methanator) and linear range of quantification. The instrumental detection limit (IDL) was tested by eight times of low concentration (0.05 μgC/μl) of EDTA solution, and a value of 0.11 μgC/cm^2 (for 99% confidence) was calculated, somewhat lower than the reported value by Birch and Cary (1996) (0.15 μgC/cm^2). Method blank was tested every 10 samples and subtracted from the results.

Results and discussion

Carbonaceous characteristics of the samples

As shown in Fig. 1, the 34 PM2.5 samples tested in this study cover a large variation of carbonaceous characteristics, such as EC to TC ratios (0.01–0.82) and TC to PM2.5 ratios (0.12–0.84), significantly greater than the range of typical ambient samples. For example, ratios of EC/TC and TC/PM2.5 for Shanghai samples (S15–S28) vary within 0.06–0.29 and 0.22–0.52, respectively, and for Guangzhou samples (S29–S34), within 0.15–0.22 and 0.37–0.47, respectively. In addition, data of other cities have also been reported by previous studies. For example, ratios of EC/TC and TC/PM2.5 varied within the ranges of 0.15–0.29 and 0.26–0.37, respectively, in southeastern United States (5-year average during 1998–2003; Edgerton et al. 2005), and within the ranges of 0.08–0.16 and 0.16–0.42, respectively, in Beijing, China (monthly average in 2000; Zheng et al. 2005). Recent data for Hong Kong, China (Duan et al. 2007) and Amsterdam,

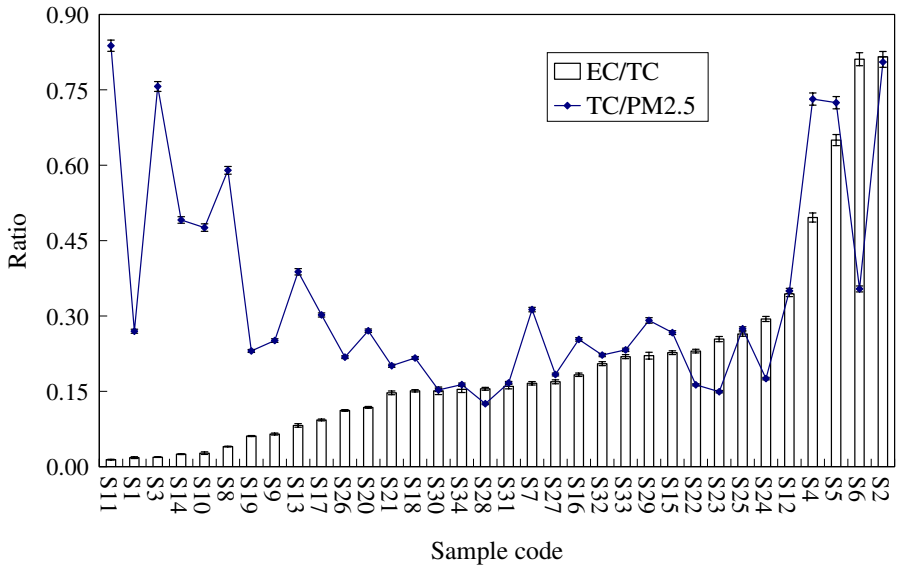

Fig. 1 The ratios of EC/TC and TC/PM2.5 of the 34 samples used in this study. The carbon values shown here were measured by the temperature program RT-short, which is similar to that in NIOSH standard method

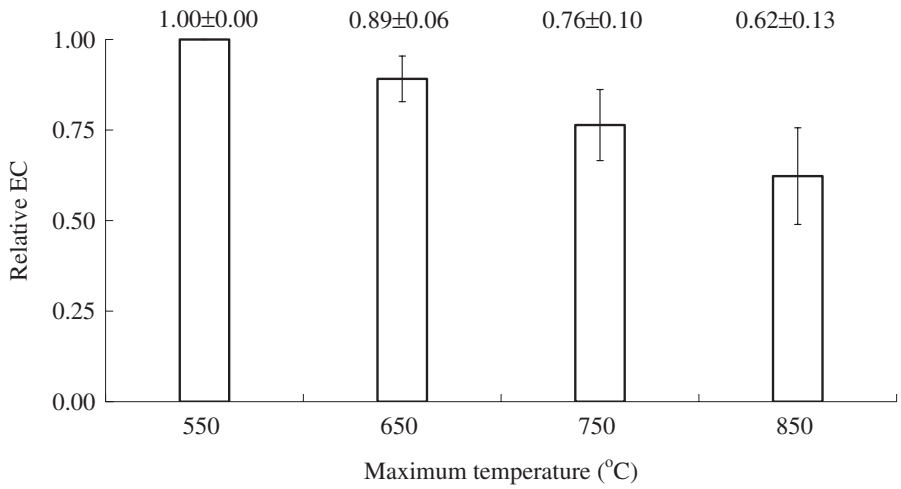

Fig. 2 The variation trends of EC results versus the maximum temperature (T_{max}) in helium atmosphere

The Netherlands (Viana et al. 2007) were both within the scopes mentioned above.

These samples also cover a wide range of OC compositions, because they came from different source emissions and locations (Table 1). It is well known that source samples are characterized by different OC constituents, which are the foundations of source apportionment using organic compounds as tracers (Schauer et al. 1996; Schauer and Cass 2000). For example, levoglucosan and resin acids are the most abundant compounds in wood burning smoke and can be used as tracers for this source (Fine et al. 2002; Simoneit et al. 1999). In addition, polar organic compounds have been demonstrated to tend to char compared to nonpolar and thermally stable compounds in thermal method (Yu et al. 2002). Therefore, the samples in this study are helpful to observe the charring of various OC constituents under different temperature programs.

Impacts of T_{max} on EC and OC results

T_{max} in Helium stage has great effects on the ECOC results, and the general trend is that the higher T_{max} used, the lower EC value measured. Figure 2 shows the averaged results of all 34 samples in this study. EC values from temperature programs with T_{max} of 650°C, 750°C, and 850°C (EC_{650}, EC_{750}, and EC_{850}) are only 0.89 ±

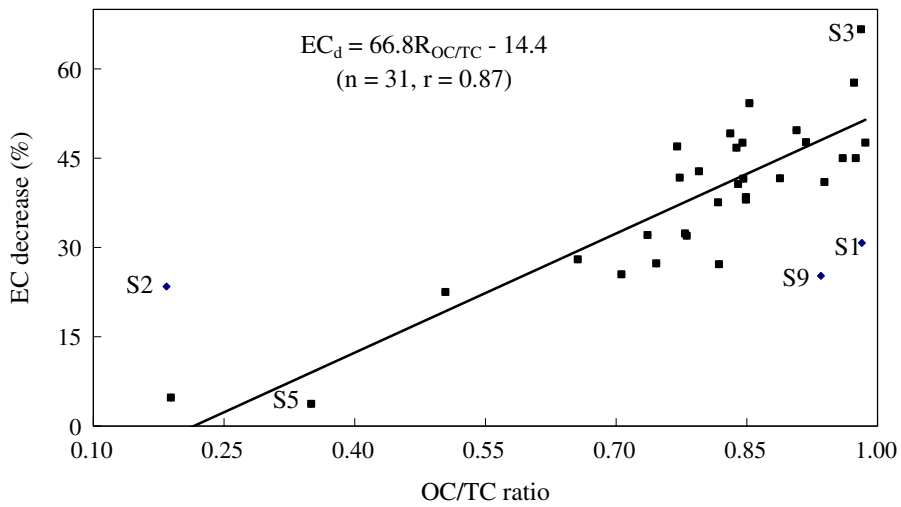

Fig. 3 Significant correlationship between T_{max} effects and OC/TC ratios in samples

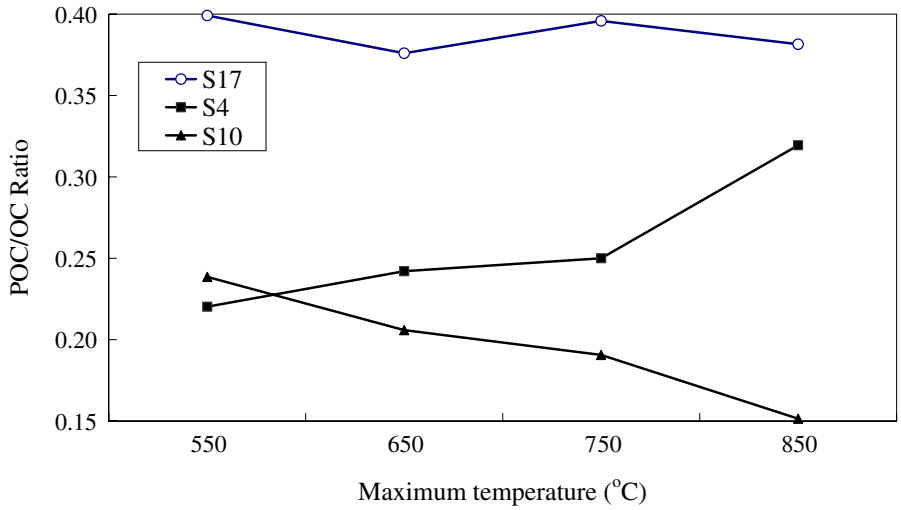

Fig. 4 Three models of T_{max} effects on POC/OC ratios represented by S4, S10 and S17, respectively

0.06, 0.76 ± 0.10, and 0.62 ± 0.13 times EC_{550}, respectively, which means that EC value will decrease by about 40% when T_{max} goes up from 550°C to 850°C. Chow et al. (2001) have reported similar results when comparing IMPROVE (T_{max} is 550°C) and NIOSH (T_{max} is 850°C). It was observed that some carbon evolved from the filter surface when temperature went beyond 550°C. Chow et al. (2001) considers this carbon fraction as OC, which is oxidized by the oxygen supplied by mineral oxides in particle mixture under high temperature. However, Schauer et al. (2003) and Yu et al. (2002) argue that 550°C may be too low to evolve all OC in nonoxidizing atmosphere. In fact, there is no distinct split between EC and some OC from both thermal and optical characteristics. Yu et al. (2002) showed that some OC needs very high temperature and long time for evolution in inert atmosphere, and some organic compounds with weak light-absorptivity disturb the differentiation of OC and EC. Essentially, there is no distinct split between EC and some OC from both thermal and optical characteristics (Andreae and Gelencsér 2006), and furthermore, there is still no EC standard material that represents EC in ambient samples.

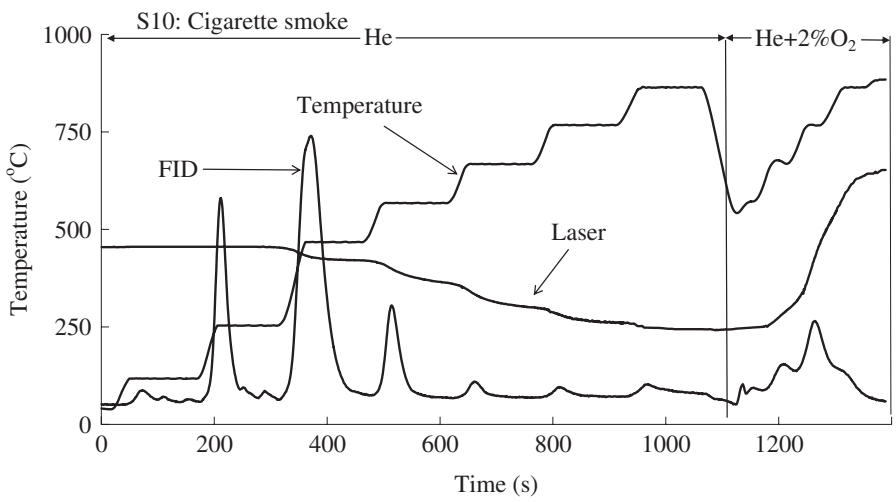

Fig. 5 Case of inconsistency between laser implication and reported POC. Laser and FID signals are in relative units

The effects of T_{max} on EC results were found to be related to organic constituents and OC abundance in TC ($R_{OC/TC}$) on the filter sample. For example, EC value of S3 (bituminous coal combustion) decreases by 67.2% when T_{max} changes from 550°C to 850°C, whereas EC value of S5 (plastic burning) decreases by only 3.7%. Figure 3 plots the relationship between $R_{OC/TC}$ and the percentage of EC decrease (EC_d) caused by T_{max} elevating from 550°C to 850°C, expressed as EC_d (%) = $66.8R_{OC/TC} - 14.4$ ($n = 31$, $r = 0.87$). Three samples (S1, S2, S9) were excluded from above equation. Deviations of S1 (anthracite coal combustion) and S9 (charcoal burning) originate from the premature emergence of OC/EC split during He stage. However, there's no enough explanation for the deviation of S2 (bituminous coal burning), which has the highest EC/TC ratio among all samples studied (Fig. 1).

Impact of temperature on pyrolyzed OC values

Charring of OC is hard to avoid in thermal method and is considered a big challenge to ECOC measurement (Chow et al. 2004; Schmid et al. 2001; Japar et al. 1984). The amount of POC is defined in the present paper as the amount of OC that evolves in the second stage (O_2-containing), and POC ratio is calculated as the ratio of POC to the total OC. Of all the samples tested in this study, POC ratios range from 0.07 to 0.58 (by RT-short program) with an average of 0.35, suggesting that samples vary from each other in organic constituents contained (Yu et al. 2002).

T_{max} setting of thermal program in helium stage significantly affects POC formation (charring). As shown in Fig. 4, there are three variation trends of POC ratios when T_{max} increases and each variation is represented by a typical sample. First, as exhibited by S10 (cigarette smoke), higher T_{max} leads to lower POC ratios, with 13 samples falling into this category (S1, S5, S7, S9, S10, S13, S14, S18, S19, S23, S30, S31, S34). This may be attributed to increased OC desorption at elevated T_{max} before going into the second stage (O_2-containing stage) and resultingly left less carbon calculated as POC. Second, as exhibited by S4 (wood smoke), three samples (S2, S4, S15) display an opposite tendency that higher T_{max} program generates lower POC results, implying that these samples contain more fraction of polar organic compounds than the others (Yu et al. 2002). Cadle and Mulawa (1990) also observed that significant charring still occurs even if wood smoke aerosol samples are heated to 950°C. Third, as exhibited by S17 (ambient aerosol at JD, Shanghai), all the others show irregular POC responses to T_{max}

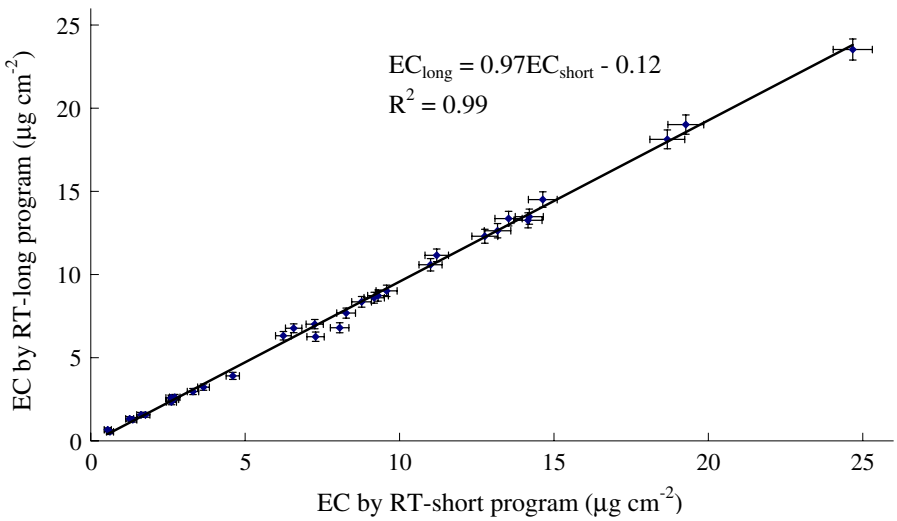

Fig. 6 Effects of RT on EC

change. It is difficult to explain such phenomenon clearly, because the optical behavior of POC and even native EC in the sample may vary with temperatures (Conny et al. 2003).

It's worth noting that the trend of POC variation caused by T_{max} may be contradictory to observed laser trace. Figure 5 is a thermogram of S10 (cigarette smoke). Laser intensity keeps falling when T_{max} rises from 550°C stepwise to 850°C, implying that more char forms with T_{max} elevating. However, this implication is obviously contrary to the first trend mentioned above, in which S10 generates less char when T_{max} elevates. Such disagreement can be attributed to two simultaneous processes present in one analysis. On the one hand, gradually ascending temperature gives OC increasing chances to pyrolize (producing more char; Cadle and Mulawa 1990), and on the other hand, leaves decreased organic carbon to be oxidized before split point (reducing char measured). If the second process dominates, the disagreement shown in Fig. 5 arises.

Impacts of residence time on EC and POC results

The residence time (RT) of each temperature step in helium stage has been observed to affect EC and POC results. As shown in Fig. 6, less EC is measured when prolonging RT. The relationship can be expressed as $EC_{long} = 0.97EC_{short} - 0.12$ ($n = 34$), signaling 3% decrease of EC is effected in general when RT is prolonged in this study. It was noticed that FID signals by RT-short program often failed to return to the baseline at the end of He stage because of insufficient temperature maintenance, and led to incomplete OC desorption during He stage. The remaining OC would evolve after the addition of O_2–He mixture and contributed to the overestimation of EC by RT-short. Moreover, by comparison of the effects between T_{max} and RT, it is believed that RT has less effects on EC values than T_{max} does, suggesting T_{max} is more active than RT in TOA.

As for the RT effect on charring, it is observed that most source samples (12 of 14 samples) and all ambient samples have decreased POC results when RT is prolonged. For example, POC values by RT-long are 14% and 8% lower than those by RT-short, respectively for S10 (cigarette smoke) and S30 (ambient sample from Guangzhou). S2 (bituminous coal burning) and S6 (rubber smoke) are two exceptions—RT-long program generates more POC than RT-short program. This situation may be caused by their significantly higher EC/TC ratios (larger than 0.80) than other samples (less than 0.65). In fact, EC/TC ratios in typical ambient aerosol samples are always less than 0.5 (Edgerton et al. 2005; Zheng et al. 2005; Viana et al. 2006). On average, POC by RT-long accounts for 0.91 times that by RT-short, signifying a decrease of 9%, much less than 43% reported by Yu et al. (2002). The use of water soluble organic compounds rather than actual ambient samples in their work should account for the disagreement.

Conclusions

Totally 34 samples and six temperature programs were studied to investigate the effects of T_{max} and RT on ECOC measurement by TOA.

(i) EC values are sensitive to T_{max}, which can be expressed as: $EC_{650} = (0.89 \pm 0.06)\ EC_{550}$, $EC_{750} = (0.76 \pm 0.10)\ EC_{550}$, $EC_{850} = (0.62 \pm 0.13)\ EC_{550}$. The sensitivity is significantly correlated with OC abundance in TC, demonstrated by $EC_d\ (\%) = 66.8R_{OC/TC} - 14.4$ ($r = 0.87$).
(ii) POC values are sensitive to T_{max}, and there are three different trends for various samples, attributable to organic constituents in the samples of different sources.
(iii) Long RT program generally results in lower EC results. However, the effect of RT on EC is by far weaker than that of T_{max}.
(iv) Prolonged RT reduces char formation by 9% averagely, much lower than that described in previous study.

The results of this study will not only enrich the knowledge of TOA methods, but also contribute to the worldwide standardization of thermal/optical ECOC determination.

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