STANDARD OPERATING PROCEDURES

Determining Equivalent Black Carbon from PTFE® Filters with a Smoke Stain Reflectometer

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1.0 SCOPE AND APPLICATION

When the post-weighing of PTFE® filters is completed and validated, and they have returned from FTIR and HIPS analysis, the equivalent black carbon (EBC) content of the collected particulate matter is estimated using a Smoke Stain Reflectometer (SSR). These measurements are performed before specific analysis for other chemical components as some methods used are destructive to the filters. This SOP describes the steps taken to ensure quality EBC concentrations are determined from sampled PTFE® filters.

REVISION HISTORY			
Revision No.	Change Description	Date	Authorization
2.0	General reorganization and clarification	August 12, 2018	Crystal Weagle

2.0 SUMMARY OF METHOD

The SSR is stored, and all measurements taken, in a HEPA-filtered hood. Surface reflectance (R) as measured by the SSR is used to determine EBC, a surrogate for BC content. The reflectance is reported on a grey-scale from 100 to 0, with 100 representing pure white (no BC collected) and 0 representing pure black. Reflectance measurements are taken in triplicate (non-consecutively) of each sampled PTFE® filter and averaged. The natural logarithm of the ratio of the average reflectance to the initial reflectance of the filter (100) is multiplied by a conversion factor to obtain an EBC in μg. The conversion factor is the ratio of the filter surface area (cm^2) to the mass absorption cross-section ($cm^2\ \mu g^{-1}$), multiplied by 1.5 to account for the thickness of the PTFE filters. This conversion is summarized by the equation below:

$$[EBC] = \frac{-A}{qv} ln\left(\frac{R}{R_O}\right)$$

where A is the filter surface area (3.1 cm^2), *v* is the volume of air sampled (m^3) , R is the mean measured reflectance after sampling, R_o is the reflectance prior to sampling, and *q* is the product of the reflectivity path (0.5) and the mass-specific absorption cross section (σ_{SSR}, $cm^2\ \mu g^{-1}$). The σ_{SSR} used is 0.1 $cm^2\ \mu g^{-1}$.

3.0 CONTAMINATION CONTROL

To minimize potential contamination of filters during handling, all SSR measurements are taken in a HEPA-filtered hood. Clean nitrile gloves are worn when working with filters, and filters are only handled PTFE®-coated tweezers that have been wiped clean with methanol. The

circular mask of the SSR is also wiped clean with a methanol soaked Kimwipe®. Between measurements, filters are returned to their petri dishes and petri dishes are closed until the next measurement.

4.0 SAMPLE STORAGE AND RECORDKEEPING

Following FTIR analysis, filters are stored at room temperature in closed petri dishes, inside a sealed plastic bag. The filter IDs are recorded in site-specific spreadsheets and the triplicate SSR values are recorded during the measurement session. Approximately every month, the spreadsheets for each site is copied and pasted into the archived folder under the correct year and month, so that there will be records of previous versions of the SSR spreadsheets in case they are lost or need to be consulted. Once the measurement session is complete, filters are secured in their petri dishes and stored inside a sealed plastic bag.

5.0 EQUIPMENT

- Smoke Stain Reflectometer (SSR)
- SSR calibration plate
- HEPA-filtered hood
- PTFE®-coated tweezers
- Sampled 25mm PTFE® membrane filters (PT25DMCAN-PF03A, 3µm pore size with FEP support ring, stored inside petri dishes)
- Methanol

6.0 ANALYSIS WITH SMOKE STAIN REFLECTOMETER

The SSR should be in a HEPA-filtered hood for the calibration procedure, and for all measurements taken. To turn on the SSR use the main power switch located at the back of the device. When the SSR is initially powered on, the display will read “100” with no decimal place. The device is ready to use once the display begins showing numbers with a single decimal place. There are three main components of the SSR needed for each measurement session: the optical unit, the circular mask, and the calibration plate.

6.1 Calibration of SSR Optical Unit

The SSR must be calibrated prior to every measurement session following the procedure outlined below:

- Attach the circular mask to the bottom of the optical unit and place the hole at the center of the circular mask in the middle of the white portion of the calibration plate. The readout should be close to 100.0.
- Leave the SSR on and centered over the white disk of the calibration plate for 15 minutes to allow it to stabilize prior to calibration.
- After the stabilization period, press the "CAL" button, the system will display 100.0. If after pressing "CAL" button the readout does not stabilize at 100.0, press "CAL" again.
- Once the SSR stabilizes at 100.0 on the white panel of the calibration plate, move the optical unit over to the grey plate of the calibration unit. Once on the grey calibration plate, the SSR readout is required to be 36 ± 1.5. If the SSR does not give the required readout, repeat the calibration procedure on the white calibration plate. If after three attempts to calibrate the SSR the required readouts are not obtained, report the issue to a SPARTAN manager as the bulb in the optical unit likely needs to be replaced.

6.2 Reflectance Measurements of PTFE® Filters

Reflectance measurements of sampled filters are only to be taken after the SSR has been fully calibrated following the procedure outlined in section 6.1. All readings of sampled filters must be taken over the white calibration disk.

- Place a sampled filter at the center of the circular mask and slide the optical unit onto the circular mask until secure. If the filter is not centered the SSR will not display a consistent reading.
- Allow the SSR reading to stabilize and record the measurement in the site-specific spreadsheet for the corresponding filter ID. Repeat the reflectance measurement three times (non-consecutively) for each filter until all filters in the batch are completed.
- The standard deviation of the three measurements for each filter must not be higher than 1.5, or else the triplicate measurements must be repeated. If this criterion is not able to be met, the SSR is recalibrated and the measurements retaken.

7.0 DATA VALIDATION

7.1 Level 1 Data Validation

Level 1 data validation occurs immediately following measurement with the SSR.

- If the mean reflectance of a filter is > 90 or < 20 the determined EBC is flagged as invalid as it is outside the linear range for conversion of R to μg. The filter is flagged as "Too high" or "Too low" in the spreadsheets.

- The determined EBC mass must be less than the collected $PM_{2.5}$ mass, else the reflectance readings are repeated. If the after repeating the measurement the EBC remains higher than the $PM_{2.5}$ mass, the determined EBC concentration is flagged as invalid.

7.2 Level 2 Data Validation

When the flow rates and corresponding volumes for the sampled filters have been validated as described in SPARTAN SOP Gravimetric Analysis Revision 3.0, the determined EBC mass (μg) is converted to mass concentration in $\mu g\ m^{-3}$. The mass concentration determined for a given filter should be consistent with those around it, except for known cases of an event (e.g. local burning activity) that is expected to lead to an exceptional EBC concentration.