Title: National Air Pollution Surveillance (NAPS) Network Reference Method for the Measurement of PM2.5 Concentration in Ambient Air Using Filter Collection and Gravimetric Mass Determination		Copy No: ##
Method No.: 8.06/2.0/M	Effective Date: January 4, 2012	Location: ###

QSM Approval: _____

National Air Pollution Surveillance (NAPS) Network Reference Method for the Measurement of PM2.5 Concentration in Ambient Air Using Filter Collection and Gravimetric Mass Determination

1. Introduction and Scope

This method describes the operational requirements for sampling a known volume of ambient air over a 24 hour interval in order to collect a sample of fine particulate matter (PM) of aerodynamic diameter 2.5 µm and smaller (PM2.5) on a Teflon filter. Sampling frequency shall be as per NAPS program requirements (every 3 or 6 days, depending on site). The analytical procedures used for determination of particulate sample mass shall be the procedures described in the current revision of AAQS method 6.08/*.*/M, "Determination of the Weight of Particulate Matter Collected on Teflon Filters".

Installation and operating procedures specified by manufacturers of method-compliant samplers (see Section 6.1) must be followed, and are deemed to be part of this measurement method.

This method is fit for the intended use.

2. Principle of Method

All samplers that are compliant with this method feature two inlet system components which, due to their precise geometries, combine to mechanically separate ambient particulate matter into two size fractions, at a particle size cut-point of 2.5 µm. To achieve and maintain the required particle size cut-point throughout the sampling event, air velocity at the inlet sampling head must be precisely controlled at a constant value. A mechanical pump is used to draw air through the sampler, while a mass flow controller maintains air flow at the required velocity, which corresponds to an inlet flow rate of 16.7 litres per minute, at actual ambient conditions of temperature and pressure. At this flow rate, the sampling head is designed to pass particles having aerodynamic diameters no greater than 10 µm (PM10). Downstream of the sampling head, a cyclone (single channel PM2.5 samplers) or virtual impactor (dichotomous samplers) completes the separation of particulate matter into PM2.5 (fine) and $PM_{10\text{-}2.5}$ (coarse) fractions. The PM2.5 fraction is collected on a pre-weighed Teflon filter. Dichotomous samplers collect both the fine and coarse PM fractions on separate filters.

After the sampling period is completed, the exposed PM2.5 filter is removed from the sampler and returned to the laboratory, where it is weighed again. The mass collected on the filter is determined by weight difference, and the average PM2.5 mass concentration over the sampling interval (24 hours, midnight to midnight) is calculated as PM2.5 mass divided by actual sample volume (i.e. volume at actual conditions of temperature and pressure during sampling), in units of µg/m3.

3. Detection Limit and Range

The mass concentration detection limit associated with this fixed volume sampling method is dependent on the mass detection limit specified in the current revision of method 6.08/*.*/M. Mass concentration of PM2.5 is rounded to integer values in NAPS annual data summary reports, with a lower reporting limit of 1 $\mu g/m^3$.

The upper concentration limit for this method is at least 200µg/m3, as per U.S. EPA specification[1]. The actual range of this method is limited only by a sampler's ability to maintain its set point flow rate within software-defined limits throughout the 24-hour sampling event Samplers that are compliant with this method will automatically abort any sampling event where total mass loading and particle size distribution combine to create an excessive pressure drop across the filter, resulting in an out-of-specification flow rate profile.

4. Precision and Accuracy

Method 6.08/*.*/M addresses the precision and accuracy of the laboratory filter weighing process. Sampling event measurements of flow rate, ambient temperature, ambient pressure, and elapsed time are also relevant to the precision and accuracy of determined PM2.5 concentration values. Accurate measurement and precise control of flow rate are critical for achieving the correct particle size cut-point of 2.5 µm. Acceptable sampling event accuracy is achieved when the accuracy of sampler sensors for flow, temperature, and pressure remain within the control limits specified in the manufacturer's Operation Manual.

Notwithstanding adequate and documented control over the sampling and filter weighing processes, unrecognized physical loss of particulate matter during shipping and handling, and potential loss of semi-volatile PM2.5 components (e.g. inorganic nitrates) between the times of filter deposition and filter weighing represent sources of unquantifiable absolute error in the measurement of PM2.5 concentration. Particulate matter is a complex mixture of both inorganic and organic substances. The typical chemical composition of PM can vary significantly between different regions of Canada, and from season to season at a given location, due to general climate type (e.g. marine vs.

continental), as well as local and regional influences (both industrial and non-industrial). Since the absolute accuracy of measured PM2.5 concentrations can never be known, accuracy is therefore assessed relative to measurements obtained using a U.S. EPA-defined reference method.

Based on collocated sampler test data submitted by manufacturers of method-compliant samplers, a data quality objective of 10% coefficient of variation (CV) or better was established by the EPA for the operational precision of $PM_{2.5}$ monitoring data[1]. In the absence of any NAPS program requirement for collocated, identical samplers, NAPS data for PM2.5 mass concentration cannot be assessed for precision.

5. Interferences

This method is not subject to chemical interference. However, PM2.5 mass concentration could be underestimated due to post-sampling PM losses as described in the preceding section. To minimize potential volatile losses, the maximum intervals specified in Section 9, Table 1 for sample retrieval and filter weighing should not be exceeded. To minimize the potential for unrecognized, accidental PM losses, exposed sample filters must be returned to their assigned petri dishes and stored in the shipping container promptly after retrieval from the sampler.

Mass concentration could also be overestimated due to adsorption of excess moisture on the particulate sample during or following the sampling event. The potential for error due to this effect is eliminated by conditioning the sample filter in a controlled humidity laboratory environment prior to initial and final weighing, as described in method 6.08/*.*/M.

6. Sampler and Filter Specifications

6.1 EPA Specifications

The EPA has established detailed specifications for PM2.5 sampler design and performance, and designates any sampler meeting all specifications as a Manual Reference Method (referred to by manufacturers as Federal Reference Method, FRM). By definition, the PM2.5 mass concentration determined by the reference method is accepted as the true concentration. A manual sampler which does not meet all of the design specifications for designation as a reference method, yet meets defined performance specifications for data comparability against collocated FRM samplers, may receive EPA designation as a Manual Equivalent Method (also referred to as Federal Equivalent Method, FEM). **Samplers to which this method applies must have EPA designation as either FRM or FEM samplers**.

All specifications relating to the design, field testing, and performance for different designated classes of PM2.5 samplers and monitors are contained in Parts 50, 53, and 58 of Title 40 of the U.S. Code of Federal Regulations (current version available on the web at http://cfr.regstoday.com/40cfr.aspx). On a quarterly basis, the EPA publishes a listing of all air monitoring equipment which has met its specifications. This listing is available on the web at www.epa.gov/ttn/amtic.criteria.html

6.2 Particle Size Selective Inlet

The size selective inlet system shall consist of a PM10 sampling head and a Sharp Cut Cyclone (SCC) or virtual impactor (dichotomous samplers only). At a flow rate of 16.7 LPM, the inlet head will not pass particles with an equivalent aerodynamic diameter greater than 10 µm. The SCC or virtual impactor shall have the 50% cut point at 2.5 ± 0.2 µm equivalent aerodynamic diameter, and a performance curve equal to or better than the inlet specified for the EPA reference sampler. The inlet performance shall not be sensitive to wind direction.

6.3 Sample Flow Rate Control, Sampling Timer and Volumetric Measurement

The flow rate through the sample filter shall be monitored continuously, and controlled at a coefficient of variation of no greater than 2% during the sampling period, as calculated from the standard deviation of 5-minute average flow data logged automatically by the sampler, divided by the average flow rate for the sampling period, multiplied by 100.

The sampler timer should have an accuracy of ±1 minute or better over 24 hours. The time at the beginning and end of the sampling period shall be recorded. The time duration of sampling shall also be recorded.

The volume of sample air shall be calculated by using logged data for flow rate, ambient temperature and ambient pressure throughout the duration of the sampling event. The sample volume shall be reported at actual conditions of temperature and pressure.

6.4 Filter Temperature Control

The air temperature at a point 1.0 cm centrally downstream of the filter shall be monitored continuously during the sampling period, with averaged temperature data recorded automatically at 5-minute intervals. The temperature at the filter should not differ from that of ambient air by more than 5°C for more than 30 consecutive minutes during the sampling period. This control is normally achieved by forced ventilation with external air flowing through the filter chamber assembly at a sufficient rate.

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6.5 Ambient Temperature and Pressure Measurement

The ambient air temperature and pressure during the sampling period shall be monitored continuously, with averaged data logged automatically at 5-minute intervals. This data is used to calculate and automatically record the average ambient temperature and pressure for the sampling period.

6.6 Filter Media

The filter shall be circular with a diameter of 46.2 ± 0.25 mm. The material of the filter shall be polytetrafluoroethylene (PTFE Teflon) with a pore size of 2 µm. It shall have a collection efficiency of greater than 99.7% for particles with diameter of greater than 0.3µm at the operating flow rate of the sampler. The unexposed, weighed filter shall be fixed in a cassette at the laboratory. The cassette's support screen displays an etched serial number which is recorded at the laboratory and shall be recorded by the field operator on the Field Data Sheet.

7. Installation Requirements

The installation procedures detailed in the sampler's Operating Manual must be followed. Prior to installation, the following issues must be considered.

7.1 Sampler Location Criteria

The location of the sampler inlet should conform to the following criteria.

Height above ground	2 to 15 metres
Height of inlet above support structure	>2 metres
Distance from trees	the greater of 20 metres or 2X the height of the tree above the inlet
Distance from any air flow obstacles, (i.e. buildings)	> 2X the height of obstacle above the inlet

In some cases, ideal siting of samplers may not be possible for practical, logistical, or other reasons. Also, established NAPS sampling locations are generally intended to be "permanent", and locations which originally satisfied all siting criteria may no longer be ideal due to man-made or natural (e.g. tree growth) changes in the near surroundings over time. With respect to obstruction criteria, air flow should be unobstructed in at least three quadrants at the time of installation, including the direction of the predominant prevailing winds. Decisions on new sampler locations or sampler relocation must be taken by NAPS

operating agencies in consultation with Environment Canada's NAPS Operations Unit, particularly in cases where proposed new locations do not meet ideal siting criteria.

7.2 Collocation of PM Samplers

Space should be available at the selected sampling location for the installation of a collocated sampler for the purpose of audit sampling or possible future precision studies.

For collocated PM samplers with flows not greater than 16.7 lpm, the spacing between sampler inlets must be between 1 and 4 metres. For flow rates greater than 16.7 lpm, sample inlets must be separated by a distance of between 2 and 4 metres.

7.3 Local Source Influences

Minimum distance from local source influences such as furnace or incineration flues is dependent on the heights of the emission sources and the type of emission. Distance from roads is dependent on the height of the inlet and varies linearly from greater than 25 metres at inlet height of 2 metres to greater than 5 metres at inlet height of 15 metres.

7.4 Mechanical

The sampler must be securely supported so that it will not move due to vibration generated by the pumping system or wind. The tube connecting the size selective inlet to the filter compartment should be vertical without bends.

7.5 Electric Power Supply

The electrical line voltage to the instrument can influence its performance. Variations in pump speed can create feedback problems with the sampler's flow control system, and hence its ability to maintain flow constant within EPA specifications. Such instability would cause a sampling event to automatically abort. This problem is sometimes encountered at remote sites where power supply may be prone to frequency and voltage variations, including spikes.

7.6 Exterior Enclosures

The sampler's filter assembly must be located outside, so that the filter and ambient air temperature will remain within 5°C of one another. If the instrument has a control system which is separate from the filter assembly, it can be installed in a separate enclosure.

The purpose of this requirement is to minimize the potential loss of volatile species from the filter.

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8. Sampler Operating Parameters and Configuration

The sampler must be configured according to instructions in the manufacturer's Operating Manual. Sampler configuration and operating parameters must be recorded in a log book upon first operation of the sampler, and any time a parameter setting is changed (e.g. sampling frequency).

9. Operational Requirements

9.1 At the laboratory, an analyst shall: 1) coordinate shipment of weighed filters, Field Data Sheets, and other sampling supplies to sampling locations across the NAPS network on a timely basis; 2) maintain records of all filters used in the network; 3) receive samples and associated data sheets returned from the field and record date of receipt; 4) weigh filters as per the requirements of method 6.08/*.*/M; 5) transfer sampled filters to the appropriate laboratories for chemical analysis; and 6) archive Field Data Sheets and analyzed filters.

9.2 Field Data Sheets specifying required sampling event data from either single channel or dichotomous samplers are provided to field operators by Environment Canada.

9.3 One blank filter will be included with each shipment of filters. The blank shall remain in the petri dish and be returned to the laboratory with the same filter batch as received. The blank will be weighed in the normal manner. Should the weight of the blank filter deviate more than ±10µg, the cause shall be investigated.

9.4 A copy of the Field Data Sheet will accompany the return shipment of exposed filters from the field. The analyst will verify the IDs of the filter cassettes against the Data Sheet and record filter information into the filter data spreadsheet.

9.5 Table 1 summarizes the actions required of the analyst and field operator in order to obtain a valid sample, regardless of the model and make of sampler used.

Table 1

Operational Requirements

Action	Time Interval	Procedures	Documentation	Responsibility
Inspect filters for defects	Prior to conditioning	Current version of AAQS Method 6.08/*.*/M	Record batch ID number of any rejected filters	Lab analyst
Condition new filters	Minimum 24 hrs prior to weighing		Record conditioning temperature, relative humidity and duration	
Weigh filters, install in cassettes, place cassettes in covered petri dishes in shipping case	Within 60 days of sampling	Current version of AAQS Method 6.08/*.*/M	Record filter weights and cassette serial numbers in lab spreadsheet file	Lab analyst
Ship filter cases to attention of field operators and communicate same via e-mail	In a timely manner, as per established sampling schedules	Transport in cases designed for these filters	Record shipping date and intended recipient (i.e. addressee)	Lab analyst
Install filter cassette(s) in sampler and program sampling date(s) and sampling duration	Within 5 days prior to sampling for FRM samplers; within 30 days for sequential samplers.	Sampler Operation Manual	Record sampler ID, filter and cassette IDs and sampling date(s) on Field Data Sheet	Field operator
Post-sampling filter cassette retrieval	Retrieve within 5 days after the last sampling event.	Sampler Operation Manual	Record all required sampling event information on Field Data Sheet	Field operator

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Table 1

Operational Requirements

Action	Time Interval	Procedures	Documentation	Responsibility
Sampled filter storage after retrieval	Within minutes of retrieval from sampler	Place and cover sample filter cassette in same petri dish in which it was received		Field operator
Ship samples to attention of lab analyst and communicate same via e-mail	As soon as possible after last sample for current filter batch is retrieved	Transport in cases designed for these filters	Completed Field Data Sheet	Field operator
Remove sampled filters from cassettes and condition	Minimum 48 hours prior to weighing	Current version of AAQS Method 6.08/*.*/M	Record conditioning temperature, relative humidity and duration	Lab analyst
Weigh sampled filters	Within 60 days after sampling		Record mass of exposed filter	

10. Maintenance, Calibration and Audit Requirements

10.1 The maintenance requirements of the sampler as specified in the manufacturer's Operation Manual must be followed. Any sampler which is out of service pending repair must be clearly labeled as such.

10.2 The frequencies of activities included in Table 2 are NAPS minimum requirements. All of these activities must be documented by the field operator in a hardcopy log book, or as electronic records which are safeguarded against loss.

Table 2

Maintenance, Calibration, Audit Schedule

Action	Frequency	Procedures and Specifications	Documentation
Periodic maintenance of sampler components and pump	As per sampler Operation Manual	As per sampler Operation Manual	Record in log book at station
Check if sampler is reporting an error status code for most recent sample	Each filter retrieval date	Clear error status to return sampler to operational status	Report error status code and identify corresponding sampling event in Comments section of Field Data Sheet
System Leak Check	Monthly	As per procedures in sampler Operation Manual	Record results on Field Data Sheet
Flow check	Quarterly		
Calibration of ambient temperature and pressure sensors and flow	1) At installation, 2) following repair, 3) when flow check result is not within ± 5% of design flow value		Record in log book or as secure, easily accessible electronic record
Clean inlet head and SCC or impactor	6 months maximum		Record in log book
External audit	24 months	EC or other	Audit report

10.3 External audits of sampler performance may be conducted by Environment Canada NAPS Operations staff or by operating agency staff other than the usual operator of the sampler. Audits procedures and performance specifications shall be those contained in the manufacturer's Operation Manual or procedures and specifications

established by the external auditor (EC NAPS, or agency's own auditors). Audits should address the following elements of sampler operation and performance:

10.3.1 Verify that sampler location meets siting criteria, that the sampler is properly installed and configured, and that operating parameters correspond to parameters recorded in the sampler log book.

10.3.2 Verify that the sampler's clock setting is accurate within 5 minutes of local standard time.

10.3.3 Inspect the sampling head and SCC or virtual impactor for verification of reasonable cleanliness, and inspect the log book for records of previous cleanings.

10.3.4 Verify that the sampler is leak-free to manufacturer's specification.

10.3.5 Verify that sampler flow rate is within ± 5% of design value using a certified, traceable flow standard device other than the flow device normally used by the field operator for routine flow checks.

10.3.6 Verify that the sampler's temperature sensors and ambient pressure sensor are reading within audit specifications for accuracy using certified, traceable transfer standard devices.

10.3.7 Verify that Field Data Sheets are completed correctly.

10.3.8 Verify that filters are being handled and stored correctly by the field operator.

11. Quality Assurance and Quality Control (QA/QC)

Quality Assurance or Quality Control provisions described in the current version of the *National Air Pollution Surveillance Network Quality Assurance and Quality Control Guidelines*[2] which are applicable to non-continuous sampling shall also apply to this method.

12. References

1. United States Code of Federal Regulations, Title 40, Part 50, Appendix L, *Federal Register*, vol. 62. No. 138, page 57, July18, 1997.

2. National Air Pollution Surveillance Network Quality Assurance and Quality Control Guidelines, Report Series No. AAQD 2004-1, Analysis and Air Quality Section, Environment Canada, 2004.

13. Revision History

Nov. 2005: Author: James Mar; New document

March 2006: Lead Reviewer: James Mar

A: Section 4, revised
B: Section 10.2.3, official time changed to standard time.

January 2008: Lead Reviewer: Ron Halman

references to "manual" or "manufacturer's manual" changed to "operation manual" or "manufacturer's operation manual".

"station log book" and "site log book" shortened to "log book" (as NAPS agencies may maintain multiple hardcopy and/or electronic log books).

Specified use of current revision of AAQ Method 6.08/*.*/M for determination of filter mass and PM concentration, and deleted Appendix I, which was an outdated revision of this method.

Former Appendix II now Appendix I.

Dec. 2009: Lead Reviewer: Ron Halman

Sections 6.3, 6.4, and 6.5 corrected by stating that flow rate, filter temperature, ambient temperature, and ambient pressure are all measured continuously, with averaged data recorded automatically at 5-minute intervals, as per EPA method requirements.

Described calculation of flow rate coefficient of variation in Section 6.3, and corrected CoV specification to 2%, as per sampler operation manuals.

Jan. 2012 Lead Reviewer: Ron Halman

General revision to improve clarity and organization. Material changes to content are described below.

Section 1 - deleted reference to Appendix I (Appendix removed, with relevant information moved to Section 6, Sampler and Filter Specifications.)

Section 2 – deleted reference to pressure regulator and valve system for controlling flow (obsolete), and corrected statement re calculating PM2.5 concentration at both actual and standard conditions of temperature and pressure. Concentration is calculated at actual conditions only.

Section 3 – replaced statement of 2 $\mu g/m^3$ detection limit with reference to companion method 6.08, where current mass detection limit is specified. Added statement that NAPS annual data summary reporting limit is 1 $\mu g/m^3$.

Section 4 – Referenced method 6.08/*.* re precision and accuracy of PM mass determination in laboratory, and listed instrument parameters relevant to control of sampling event accuracy. Clarified that operational precision of 10% for PM2.5 data is an EPA data quality objective, is based on performance data provided to EPA by instrument manufacturers, and is not applicable to NAPS data (i.e. this method).

Section 5 – Method is not subject to any true interference, but described circumstances that could lead to either underestimation or overestimation of mass concentration, and described procedures to mitigate against either possibility.

Section 6 – Re-titled section and re-arranged order of existing sub-sections. Added new sub-section (6.1) stating method requirement for EPA designation of sampler make and model as either FRM or FEM (replaces former Appendix I, obsolete listing of method-compliant samplers).

Section 7 – Replaced "must" with "should" re conformance to sampler siting criteria (7.1), added paragraph explaining that other considerations may take precedence over conformance to all siting criteria, and stated existing practice of consultation between NAPS agencies and EC re decisions on suitability of proposed sampling locations. Possible option of installing filter compartment inside a heated enclosure due to extreme cold was removed from 7.6 (i.e. no longer permissible under any circumstances).

Section 8 – Deleted former Table 1 (redundant information)

Section 9 – Expanded detail on operational requirements, including detail in Table 1. Specified (9.2) that Field Data Sheets for either single channel PM2.5 or dichotomous samplers are provided to field operators by EC. Deleted redundant reference to method 6.08/*.*/M (former 9.5).

Section 10 – Added statement re labeling of out of service samplers to 10.1. Added check for error status codes at each filter retrieval to Table 2, as well as new requirement for quarterly flow check. Replaced requirement for calibration at maximum 6 month intervals with calibration upon installation, following repair,

and as required, based on flow check result. Added requirement for leak check as part of audit (10.3.4)

Section 11 – Deleted former Table 4 (redundant information), former 11.2 (incorrect information), and sample Field Data Sheet (out of date). Field Data Sheet templates are now controlled as Quality System forms.

Section 12 – deleted non-specific reference to sampler Operation Manuals.

Lead Reviewer: Ron Halman **Date**

Title: Supervisor, NAPS/QA Laboratory

Approved by: Luc White **Date**

Title: Manager, NAPS Operations