Title: Determination of the Weight of Particulate Matter Collected on Teflon® Filters		Copy No: ##
Method No.: 6.08/1.6/M	Effective Date: September 13, 2013	Location: ###

QSM Approval: _____

Determination of the Weight of Particulate Matter Collected on Teflon® Membrane Filters

1. INTRODUCTION and SCOPE

1.1. This method is applicable to the gravimetric determination of particulate matter (PM) deposited on Teflon® membrane filters (47-mm or 37-mm diameter, 2 µm pore size, O-ring mounted).

1.2. This methodology is recommended for use only by or under the supervision of analysts experienced in the use of microbalances.

2. SUMMARY of METHOD

2.1. The principle of the method is based on the gravimetric determination of the weight of a specific filter before and after a known volume of air has been drawn through it. The weight gained by the filter after exposure is taken as the weight of the PM collected.

2.2. A microbalance (Mettler MX5 or comparable) calibrated annually using standards traceable to the National Institute of Standards and Testing (NIST) is used for the determination.

2.3. Particulate concentrations are reported in µg/filter and µg/m^3, based on the air flow, sampling volume and time of sampling, (see Form 6.08M/F1_ver*.*).

2.4. Filters can be weighed both manually and using the Automated Filter Weighing System. (SOP 6.21/*.*/S).

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3. SAMPLE REQUIREMENT

3.1. Environmental Conditions

3.1.1. The laboratory temperature is controlled at 23°C with a variability of not more than ± 3°C over 24 hours (20°C to 26°C).

3.1.2. The laboratory humidity is controlled within the range of 42% Relative Humidity (R.H.) with a variability of not more than ± 5% over 24 hours (37% to 47%). Deviation from this range is addressed in section 7.3.4.

3.1.3. The room temperature and humidity are recorded in the filter data file at the beginning of each weighing session using a NIST traceable thermometer and hygrometer. The temperature and humidity conditions are also continuously monitored by a hygrothermograph located in the balance room to follow trends over 24 hours. All records must be retained for a minimum of 12 months.

3.1.4. There are a minimum of 6 air changes per hour in the balance room and HEPA filters are used to filter the incoming air.

3.1.5. All working surfaces, shelves, table and floor must be clean and dust free. Neither aerosol cleaning agents nor vacuum cleaners should be used in this facility. Floor should be damp mopped only.

3.1.6. Lab coats must be worn by all persons in the laboratory to minimize dust contamination.

4. EQUIPMENT

4.1. The balances used are Mettler model MX5. The balance readability is 1 µg with a weighing capacity of 5 grams.

4.2. Balances are located on stone table to minimize vibrations (avoid leaning on the table and take care not to bump it with the armrest of the chair).

4.3. Prior to weighing, static charge on the filter is eliminated by passing it through a Haug ionization system (consisting of a U-shaped ionizer and high-voltage power pack) and a beta emitting radioactive electrostatic eliminator bar. Any remaining electrostatic charge on the filter is neutralized by the presence of a beta emitting radioactive electrostatic eliminator bar within the balance chamber. The

radioactive electrostatic eliminator is replaced with a new one 12 months after the manufacture date.

5. STANDARDS

5.1. Calibration Standards

5.1.1. The balance has 2 fully motorized internal 2.5 g weights used for self-calibration.

5.1.2. A 100 mg analytical weight is measured everyday that the balance is used.

5.2. Quality Assurance Standards

5.2.1. Three Teflon® membrane filters are measured everyday that the balance is used for Quality Assurance (QA).

6. CALIBRATION

6.1. Self-Calibration Procedure

6.1.1. The purpose of the self-calibration procedure (adjustment and linearization) is to compensate for the change in performance of the mechanical parts of the balance as the temperature and relative humidity change in the balance room. The balance automatically performs a self-calibration procedure using two fully motorized internal 2.5 g weights when a change in the ambient conditions makes this necessary. The balance will indicate if a calibration is necessary by displaying "Autocalin" and will self-calibrate after a few minutes if the balance is not in use. Weighing of filters can continue uninterrupted with the "Autocalin" indicator on. Do not lean on the table or disturb the balance while the self-calibration procedure is in process.

6.1.2. If necessary, to trigger the self-calibration, perform the following steps:

6.1.2.1. Press the "Menu" key until the display reads "Cal Int".

6.1.2.2. Press "Set"/"Re-zero"/"ON-OFF" key.

6.1.2.3. At the completion of the self calibration, "CalEnd" is displayed briefly. The balance then returns to the weighing mode.

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6.2. Standard Weight Verification

6.2.1. This procedure must be performed once a day for every day that the balance is used.

The following steps are for manual weighing. For automated weighing follow the steps indicated in SOP 6.21/*.*/S.

6.2.1.1. Ensure that the door to the balance chamber is closed.

6.2.1.2. Zero the balance if it does not already read 0 µg.

6.2.1.3. Carefully remove the 100 mg standard weight from its protective case using the Teflon® coated tweezers and place it in the centre of the balance pan.

6.2.1.4. Wait approximately one minute for the elimination of static electricity and for the balance to stabilize then record the mass in the QA spreadsheet.

6.2.2. The 100 mg weight should fall within ± 5 µg of expected value. If it does not fall within this range contact the Laboratory Supervisor.

6.3. Quality Assurance Filters

6.3.1. This procedure must be performed once a day for every day that the balance is used.

The following steps are for manual weighing. For automated weighing follow the steps indicated in SOP 6.21/*.*/S.

6.3.1.1. Ensure that the door to the balance chamber is closed.

6.3.1.2. Zero the balance if it does not already read 0 µg.

6.3.1.3. Carefully remove the first Teflon® membrane filter from its Petrie dish using the tweezers and place it in the centre of the balance pan.

6.3.1.4. Wait approximately one minute for the elimination of static electricity and for the balance to stabilize then record the mass in the QA spreadsheet.

6.3.1.5. Repeat steps 6.3.1.1. to 6.3.1.4. for the other Teflon® membrane filters.

6.3.2. All three QA Teflon® membrane filters should fall within ± 10 µg of previous measurements. If they do not fall within this range contact the Laboratory Supervisor.

6.3.3. The three QA Teflon® membrane filters are changed every six months and the date recorded.

7. PROCEDURE FOR DETERMINING FILTER MASS

7.1. Filter Handling Procedures

7.1.1. To avoid contamination, filters must be handled with non-serrated tweezers. Filters must be held only at the edge by the sealing ring. Individual filters should be placed in the container dish and then transferred directly to the weighing pan of the balance. The filter should never be placed on the working surfaces of the laboratory. Once weighed, the filter should be returned to the filter holder, closed and stored.

7.2. Micro Balance Stabilization and Calibration Verification

7.2.1. The micro balance is to remain powered up at all times. In the event of power failure, the balance should be permitted to warm up for a minimum of two hours.

7.2.2. The precision and accuracy of the micro balance and the weighing system is verified daily prior to a weighing session using both the standard weight and QA filters (see Sections 6.2. and 6.3.).

7.3. Conditioning of Unexposed Filters

7.3.1. Remove the filters from the packaging.

7.3.2. Visually inspect each filter prior to weighing. A filter will be rejected for use in the program if it contains any of the following defects:

- Pinholes
- Separation of the support ring from the Teflon membrane
- Chaff or flashing
- Loose material
- Discoloration
- Filter non-uniformity
- Any other observed defect which might degrade filter performance

7.3.3. Reject all defective filters.

7.3.4. Place filters on the shelves in a vented cabinet in the weighing room and in contact with ambient air. Filters must be protected from deposition of dust from ambient air movements. Condition filters for at least 24 hours to allow their weights to stabilize before being weighed. Record RH and temperature (see section 3.1). If spikes of temperature or RH occur during the conditioning period, the laboratory supervisor is informed and data is evaluated to determine if the conditioning period has been compromised and needs to be repeated. The evaluation of temperature and RH data should include blank performance in relation to the spike.

7.3.5. Before a new lot number of filters is put into service the stability of the filters must be established by weighing a set of 3 filters over a 3 day period. Filters which change more than 20 µg in mass may be leaching out impurities. Reweigh the filters daily until the weight has stabilized. All filters from this lot number will then require that amount of time to condition before determination of the filter weight then record the mass in the QA spreadsheet.

7.4. Weight Determination of Unexposed Filters

The following steps are for manual weighing. For automated weighing follow the steps indicated in SOP 6.21/*.*/S.

7.4.1. After the micro balance is stabilized, place the filter on the weighing pan of the balance. Close the balance pan chamber door and wait approximately one minute for the reading to stabilize. Observe the reading of the balance and enter it in the corresponding database file as the initial weight ($m_{initial}$), with the identification number of the filter.

7.4.2. Remove the filter from the weigh pan; return it immediately to the container and cover. Verify that the identification code on the cover corresponds correctly to the identification code entered in the database file.

7.4.3. Unexposed filters are then placed in their appropriate cassettes in petri dishes within their shipping boxes and shipped to the station operators. There are specifically labelled cassettes to be used for each station.

7.5. Reception of Exposed Filters and Reviewing of Field Data Sheets

7.5.1. Routine sample collection is a field activity, and the details of field activities are beyond the scope of this method.

7.5.2. Upon reception of the shipping box of exposed filters, open each box and remove the field data sheets and filters checking that the serial numbers on the sheets match those on the filters cassettes. If the field data sheet and/or filters are missing, notify the laboratory supervisor and begin the process of contacting the station operators in order to obtain missing information and/or filters.

7.5.3. Review the comments made by the field operator and take action accordingly. If an equipment or hardware malfunction is reported, inform the supervisor and NAPS Operations.

7.5.4. The Field Data sheets are reviewed and information entered into their corresponding Excel® spreadsheet. If correction or explanation of information on the sheet is required, the supervisor shall be consulted. Corrections shall be made in pen and initialled by the person making the

correction/comment. The sheet is dated, signed then scanned to a PDF file and saved for future reference.

7.5.5. Verify that the sampling duration is approximately 24 hours (1440 minutes).
Samplers must have operated for at least 23 hours (1380 minutes) but not more than 25 hours (1500 minutes). Filters with elapsed times greater than 25 hours may have sampled on successive days and are invalid. Samples taken for less than 23 hours up to 18 hours (1080 minutes) are still considered valid at this point, and the filter is passed on for chemical analysis with an appropriate comment added to the Excel® spreadsheet. Samples with elapsed times of less than 18 hours are invalid.

Sampling Duration (minutes)

<1080	Invalid with comments
1080-1379	Valid with comments
1380-1500	Valid
>1500	Invalid with comments

7.5.6. Verify that the sampling average flow rate was approximately 16.37 lpm or 10.00 lpm for the Partisol or SASS Speciation samplers respectively. If the sampler flow rate decreased because of heavy particulate loading on the filter, the sample should not be invalidated because the heavy loading may indicate an episodic situation that deserves study.

Sampling Flow Rate for Partisol Samplers (16.37 lpm)

<13.34	Invalid with comments
13.34-15.84	Valid with comments
15.85-17.50	Valid
17.51-20.00	Valid with comments
>20.00	Invalid with comments

Sampling Flow Rate for SASS Speciation Samplers (10.00 lpm)

<8.00	Invalid with comments
8.00-9.50	Valid with comments
9.51-10.50	Valid
10.51-12.00	Valid with comments
>12.00	Invalid with comments

7.5.7. Visually inspect each filter for the following:

- Pinholes
- Separation of the support ring from the Teflon membrane
- Chaff or flashing
- Loose material
- Discoloration of particulate loading
- Particulate non-uniformity

7.5.8. A considerable investment is associated with each loaded filter, and it should not be automatically invalidated for the observed flaws mentioned above. Consult with the laboratory supervisor then record an appropriate comment for the affected filter.

7.5.9. If the samples are not valid, they are generally removed from the normal system at this point and placed in the Archive for at least 6 months before disposal.

7.5.10. Valid Filters are conditioned following procedures outlined in Section 7.3.4

7.6. Weight Determination of Exposed Filters

The following steps are for manual weighing. For automated weighing follow the steps indicated in SOP 6.21/*.*/S.

7.6.1. Before placing the filter onto the balance for weighing the filter must be visually inspected for defects. The inspection will look for any defects in the filter, such as tears, holes, distortion, discoloration, integrity of the support ring and any other abnormalities.

7.6.2. Defective filters are noted in the data file and rejected. The identification and all particulars of the rejected filters are entered into the data file as invalid samples.

7.6.3. After the micro balance is stabilized, remove the filter from the container dish and place it on the weighing pan of the balance. Close the balance pan chamber door and wait approximately one minute for the reading to stabilize. Observe the reading of the balance and enter it in the corresponding database file as the final weight (m_{final}), with the identification number of the filter.

7.6.4. Remove the filter from the weigh pan and return it immediately to the container with the particulate laden side facing up and cover. Verify that the identification code on the cover corresponds correctly to the identification code entered in the station file.

7.6.5. Store the valid filters in a designated box in the weighing laboratory. Each box is unique to the requested analysis for the filters. Invalid filters are stored in their corresponding box. *Untreated filters can be stored indefinitely.*

7.6.6. Exposed filters are weighed within 30 days of reception.

8. REPORTING of DATA

8.1. Once a box of exposed filters (section 7.6.5) is full, prepare the internal report using the template available in the Gravimetric Lab sub-directory.

8.2. Print out the sample tracking sheet using the template available in the Gravimetric Lab sub-directory

8.3. After the internal report is completed, print, fill-out and sign the Data Validation Checklist. The approved reports shall be password-protected and the hardcopy of the signed Data Validation Checklist shall be stored for at least one year.

8.4. Transfer to samples, tracking sheet and Data Validation Checklist to the lab supervisor for review and approval.

8.5. After approval samples are transferred to the appropriate laboratory for chemical analysis along with the completed tracking sheet.

8.6. Validate the EXCEL templates containing formulas and/or macros at least once a year or after major changes of the acquisition method (SOP 2.11/*.*/S). The validated templates should be password- protected (Read Only).

9. DETECTION LIMIT

9.1. The detection limit of the method is dictated by the precision of the analytical balance used in the weighing process. The detection limit of this method is at the level of 10 µg.

9.2. The detection limit for the weighing procedure is calculated from the standard deviation of the blank media weights.

9.3. The blank media used are the QA Teflon® membrane filters.

9.4. Method detection limits (MDLs) are established from at least 10 replicate analyses of 3 blank Teflon membrane filters measured over successive days. Detection limit is calculated as three times the standard deviation of the weight of replicate analyses for a 99% confidence level.

9.5. For the record of calculated and reported MDLs refer to the QA binder (Room 131).

9.6. MDLs should be checked at least once a year, and/or after any major modification of the instrument and/or method.

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10. QUALITY CONTROL

10.1. Quality control consists of daily measurements of the 100mg standard weight (see section 6.2) along with the measurement of 3 blank Teflon® membrane filters (see section 6.3).

10.2. Further quality control consists of re-weighing at least 10% of the filters in the weighing session.

10.2.1. The difference between the first and the second weight should be within 10 µg for unexposed filters and 25 µg for exposed filters.

10.2.2. If this limit is exceeded, the cause for the discrepancy shall be determined. When the cause of the error is corrected, all filters weighed during that session must be re-weighed.

10.3.3. The re-weigh data is entered in the Gravimetric Analysis spreadsheet.

11. ESTIMATION of MEASUREMENT UNCERTAINTY

11.1. Potential sources of uncertainty of this method are:

11.1.1. Material accumulated on the filter. This can be water moisture, dust and artefacts due to chemical reactions of the material on the filter and ambient air.

11.1.2. Static electricity can also significantly alter the accuracy of the filter weight. Steps in this procedure are designed to minimize these effects.

11.1.3. Variations in temperature and humidity (Sections 3.1.1. and 3.1.2.) within the acceptance range for storage conditions and sample pre-treatments can also be a source of uncertainty.

11.1.4. Variation in weight stability of the manufactured Teflon® membrane filters over a period of time (Section 7.3.5).

11.1.5. Analyst uncertainty.

11.1.6. Balance uncertainty. The balance is calibrated annually by an ISO/IEC 17025 accredited service provider. The uncertainty on the balance is provided on the certificate.Balance uncertainty is calculated by a qualified service technician during the annual balance. If for any reason balance uncertainty is needed to be calculated between visits of the service technician, it is performed in-house using experimental data.

11.2. Method uncertainty is based on the Type A approach recommended by the Canadian Association for Laboratory Accreditation Inc. (CALA). Refer to SOP 2.10/*.*/S for detailed estimation approach.

11.3. For this method, Reported Uncertainty (95% CL) is approximately ±10 µg.

11.4. The uncertainty should be estimated at least once a year, and/or after any major modification of the method. The most recent estimated values should be reported to the client.

11.5. For the record of calculated and reported Uncertainties refer to the QA binder (Room 131).

12. MAINTENANCE

12.1. The micro balance is calibrated every year by a qualified service technician. It will be noted in the balance log book when the balance was serviced and/or calibrated. The applicable certificates/paperwork will be maintained on file.

This method is fit for the intended use.

13. APPLICABLE METHODS and SOPS

2.10/*.*/S, “Estimation of Uncertainty in Chemical Analysis"
6.1/*.*/S, "Sample Management"
6.21/*.*/S “Operation of the MTL AH225 Automated Filter Weighing System”
8.06/*.*/M “National Air Pollution Surveillance Network (NAPS) Reference Method for the Measurement of PM2.5 Concentration in Ambient Air Using Filter Collection and Gravimetric Mass Determination”

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14. REVISIONS

Feb 2006: Section 11.1.4 Add new section on variation in weight stability of manufactured filters.
Section 11.1.5 Add new section on analyst uncertainty.
Section 11.1.6 Add new section on balance uncertainty.
Section 11.2 Relocate references regarding balance uncertainty to section 11.1.6. and include both balance and method uncertainty values.
Revisions Section. Add a new section called Sept 1999, and reference original document name and author.
Revisions Section. Add to October 2003 section, "Major revision to format of the method by David Mathieu"
Section 11.1.5 Add new section on analyst uncertainty.

Dec 2009: Section 2.2 "or comparable".
Section 2.2, 4.1 and 15.1 change balance type from MT5 to MX5.
Section 3.1.2 and 7.3.4 Change humidity from 45% to 42%.
Section 7.1.1 Change "proper" to "non-serrated".
Section 7.6.5 add sentence "Untreated filters can be stored indefinitely".
Section 11.2 Change CAEAL to CALA
Section 11.3 add the word "approximately"
Section 11.4 New section added indicating annual estimation of uncertainty performed
Section 15.3 Reference to EPA document added.

Aug 2011: Lead Reviewer, David Mathieu
Section 1.1 Delete "and other environmental samples ", add 37mm diameter filter size, and remove 47mm reference in subsequent sections 5.2.1, 6.3.1.3, 6.3.1.5,
Section 2.3 change "µg" "µg/filter"
Section 2.4 Add reference to the automatic filter weighing system.
Section 3.1.2 Reword last sentence to make it more clear
Sections 4.1 and 4.2 Re-word description from one to two balances
Section 4.3 Include reference to radioactive static eliminator use prior to placing filter in balance chamber
Sections 6.2.1, 6.3.1, 7.4 and 7.6 Add paragraph indicating the SOP to follow when performing automatic weighing using the Automated Filter Weighing System.
Section 7.3.4 Include information on evaluation of data if conditioning limits not respected.
Section 7.5.2 Change flow and time limits for invalidating filters to 5%
Section 7.5.3 and 7.5.4 are removed and a reference to filter conditioning is now section 7.5.3
Remove reference to Section 8.3 Delete this section as the LIMS is no longer used

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Section 9.1 Change "in the range of" to "at the level of" for detection limit
Section 11.1.6 Delete reference to SOP 2.9/*.*/S.
Section 13 Add SOP 6.21/*.*/S "Operation of the MTL AH225 Automated Filter Weighing System" and delete all SOPs' referencing the LIMS and delete reference to 2.09/*.*/S, "Determining Uncertainty of Gravimetric Measurement Devices"
Deleted revisions history from Sept. 1999 to Oct. 2005

Sept 2013: Add "Membrane" to description of Teflon® Filters
Section 2.3. Refer to form not "appendix A"
Section 6.2.14 and 6.3.1.4 Refer to QA spreadsheet not "appendix B and C"
Section 7.3.2 Add more detail to filter inspection
Section 7.4.3 Add reference to specific cassettes used for each station
Section 7.3.5 Add reference to recording new filter lot mass verification
Section 7.5. Rewrite section to include filter/data review, validation and scanning
Section 7.6.5 Add information on storing samples in appropriate boxes
Section 8 Rename from "Recording of data" to "Reporting of Data", and now include report preparation, data approval and sample transfer for analysis information
Section 9.1. Change accuracy to precision
Section 9.4, 9.5, 9.6 Clarify detection limit paragraph including frequency and location of documents
Section 10.1. Add references to Sections 6.2 and 6.3 here, delete Section 10.2. Re-numbering subsequent sections accordingly

15. REFERENCES

15.1. Mettler MX5 operating manual
15.2. EPA Quality Assurance Document, Method Compendium, PM 2.5 Mass Weighing Laboratory Standard Operating Procedures, PEPL-2.01-13.01, Oct 1998
15.3. US EPA, Compendium Method IO-3.1, Selection, Preparation and Extraction of Filter Material, June 1999
15.4. US EPA QA Handbook Volume II, Appendix D – Measurement Quality Objectives and Validation Templates
15.5. State of Oregon Department of Environmental Quality, Gravimetric Analysis of Particulate Collected with R&P Partisol samplers and MetOne SASS Samplers. DEQ03-LAB-0027-SOP.
15.6. California EPA, Standard Operating Procedure for the Determination of PM 2.5 Mass in Ambient Air by Gravimetric Analysis SOP MLD.

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Lead Reviewer: David Mathieu
Title: Supervisor, Particulate Characterization Unit

Approved by: Ewa Dabek
Title: Head, Particulate Characterization Unit