California Environmental Protection Agency

Air Resources Board

PROCEDURE FOR THE DETERMINATION OF PARTICULATE MATTER (PM) MASS COLLECTED ON FILTERS

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1 Introduction

1.1 This document describes the methodology used by the Aerosol Analysis and Method Evaluation Section (AAMES) of Monitoring and Laboratory Division (MLD) to determine the mass of particulate matter (PM) collected on filters from mobile source exhaust emissions.

1.2 This method meets requirements specified in the 40 CFR Part 1065 Draft, dated 7/11/2008, for the weighing of PM sample filters from heavy-duty diesel fueled vehicles.

2 Summary of Procedure

2.1 The weighing procedure is carried out in a temperature and humidity controlled clean room automatically and/or manually on a microbalance capable of measuring 0.1 microgram.

2.2 The sampling filters are weighed before (e.g. pre-test) and after (e.g. post-test) the vehicle emission testing. It is required to stabilize the pre-test and post-test filters in a temperature and humidity controlled environment prior to weighing. Both pre-test and post-test PM weights are buoyancy corrected.

2.3 The difference of the buoyancy corrected pre-test and post-test filter weights is reported as PM mass.

3 Measurement Interferences and Limitations

3.1 To prevent body moisture or oils from contacting the filters, both non-serrated and antistatic metal or nonmetal forceps are used to handle the filters at all times.

3.2 An electrostatic charge may interfere with the performance of the microbalance. To prevent a buildup of static charge on the filters, static neutralizers such as Po^{210} are used to neutralize charge prior to each weighing.

3.3 To maintain Class 1000 cleanliness, analysts shall wear shoe covers, low-linting garments and powder-free gloves.

4 Instruments and Materials

4.1 Temperature and dew point controlled room that meets the Class 1000 Standard for cleanliness.

4.2 Teflon filters – typically 47 mm Teflon membrane filters, 2µm pore size, with a support ring - Whatman, Pall Life Science or equivalent.

4.3 Disposable sterile plastic Petri dishes with covers, Falcon or equivalent
4.4 Plastic Petri slides, Millipore or equivalent
4.5 300 series stainless steel tweezers
4.6 Powder free gloves
4.7 Barcode reader

4.8 Weighing System

4.8.1 MTL Automated Filter Weighing System (FWS)

- Install on a vibration-isolation platform to isolate it from external vibration
- Robotic auto handler
- Seven vertical silos for loading and unloading filters
- Two garage stations for housing reference filters and laboratory standards
- Vaisala HUMICAP Humidity and Temperature Transmitter Series HMT 330 or equivalent
- Vaisala BAROCAP digital Barometer PTB 330 or equivalent
- Filter carriers
- NIST traceable calibration weight
- Mettler Toledo XP2U microbalance or equivalent - must meet the 40 CFR part 1065 balance performance specifications
- Electrostatic neutralizer bars (Po^{210}) built into weighing chamber
- Windows-based personal computer - FWS is controlled by MTL data acquisition system

4.8.2 Microbalance for manual weighing

- Must meet the 40 CFR part 1065 balance performance specifications, Mettler Toledo microbalance UMX2 or equivalent
- Install on a vibration-isolation platform to isolate it from external vibration
- NIST traceable calibration weights
- Electrostatic neutralizer bars (Po^{210})
- Personal computer
- Digital barometer - Druck Barom DPI 142 or equivalent
- Wrist grounding strap

5 Procedure

5.1 Clean room environmental conditions verification

5.1.1 Verify that temperature and dew point of the clean room are within the limits:

Temperature:	22 $\pm$ 1 °C
Dew point:	9.5 $\pm$ 1 °C

5.1.2 Verify that temperature and humidity of the clean room are within the specification of 5.1.1 for at least the preceding 60 min before stabilizing filters.

5.1.3 A minimum of 30 minutes filter stabilization time is required (Section 5.3)

5.2 Filter media stabilization

5.2.1 To stabilize PM filters, including reference filters, pre- and post-test filters, place them individually in Petri dishes that are partially open to the PM-stabilization environment for a minimum of 30 minutes.

5.2.3 If the PM mass loading of post-test filters is expected to be higher then 400μg assuming 38 mm diameter filter stain area, equilibrate the filters to the stabilization environment for at least 60 minutes before weighing.

5.2.4 Once stabilization of filters begins, they must be weighed within 80 hours.

5.3 Microbalance preparation

5.3.1 Exercise the balance, if necessary, several times with a NIST traceable weight or a reference filter without recording the values.

5.3.2 The NIST traceable calibration weights must not be greater than ten times the mass of an unused PM sample medium.

5.3.2 Automated weighing: Start with a balance internal calibration procedure, using the built-in calibration weight.

5.3.4 Manual weighing: Perform manual adjustment (zero and span) using the internal calibration weight and/or an external NIST traceable weight (nominal 100.0000 mg) by following the microbalance operation instruction manual.

5.4 Microbalance performance verification

Before weighing any filters, verify balance performance by weighing a NIST traceable calibration weight (~100.0000 mg). The actual weight must not exceed ± 10μg of its nominal or certified value. Repeat balance performance check if the difference between the measurement and certified value exceeds 10μg. Failing to meet this requirement three times consecutively, the balance may require a re-calibration or a remedy service.

5.5 Filter weighing session

A typical weighing session includes the following steps:

5.5.1 Weigh three reference filters (section 5.5) at the beginning of each weighing session. For each filter, the buoyancy correction (Section 6) is calculated by using the recorded environmental data closest to each filter weight measurement time. The environmental data include dew point, ambient temperature, and atmospheric pressure. The environment conditions of the clean weighing room are constantly monitored and recorded every minute.

5.5.2 Weigh filters (section 5.5), including pre-test and post-test filters, and correct for its buoyancy (Section 6). A replicate sample filter must be weighed for every ten post-test sample filters, and at the end of a set of post-test filters, as a Quality Control (QC) check. If the difference of the buoyancy corrected net masses for the replicate measurements exceeds ± 10μg, all ten samples must be reweighed.

5.5.3 Weigh the three reference filters (section 5.5) again at the end of each weighing session and correct for buoyancy (Section 6).

5.5.4 The change of buoyancy corrected net mass of all reference filters during each weighing session must not exceed ± 10μg or all filters in the process of equilibration are discarded and all data collected with respect to the failed reference filters is considered void. The reference filters must be replaced.

5.5.5 Each weighing session shall not exceed eight hours for manual weighing and eighty hours for automated weighing.

5.6 Filter weighing procedure

5.6.1 Automated weighing

a. Visually inspect filters (section 7.3).
b. Verify that the filter's stabilization time meets the requirements, according to section 5.2.
c. Check the environmental conditions status on the Automated Filter Weighing Screen; proceed if it indicates "In Tolerance".
d. Assign each filter to a filter carrier by scanning filter ID (barcode) on a cover of Petri dish and the filter carrier number on a side of the carrier.
e. Transfer the filters into metal carriers using a stainless steel tweezers.
f. Install the carriers with filters into the silos, placing them one on a top of another.
g. Choose the weighing procedure (or configure one) from the Automated Filter Weighing Screen.
h. Typically a direct read with zero drift correction method (when the automatic zero point correction function is off) and a triplicate weighing are used.
i. Before beginning any procedure, make sure that the auto handler is in its home

position.

j. Click “Begin Procedure” to begin the weighing process.

k. The static electricity is neutralized by the use of Po²¹º strips built into the weighing chamber.

l. When the weighing is complete, remove the filter carriers from the silos and transfer the filters back to the assigned Petri dishes.

m. All measurements and data related to the weighing procedure are automatically recorded by MTL’s software and exported into Laboratory Information Management System (LIMS).

5.6.2 Manual weighing

a. Visually inspect filter (section 7.3).

b. Verify that the filter’s stabilization time meets the requirements, according to section 5.2.

c. Scan the filter ID (barcode) and record in the Filter Weighing Access program.

d. Neutralize the filter by using static neutralizer bars (Po²¹º).

e. Place the filter on the balance weighing pan and close the door.

f. Take the weight reading after the microbalance is stabilized (i.e. stability indicator disappears from the screen).

g. Use the Filter Weighing Access program to record the uncorrected filter weight, barometric pressure, temperature, dew point, buoyancy corrected filter weight, filter net mass (for post-test sample), operator’s name, and time stamp.

h. Remove the filter from the microbalance and store it in the Petri dish.

6 Buoyancy Correction and PM Mass Calculation

6.1 Manual Weighing

6.1.1 Calculate the buoyancy corrected mass of each filter using the formula in 6.1.2, 6.1.3 and 6.1.4.

6.1.2 Calculate the vapor pressure of water for a given saturation temperature condition (T_{sat}) and the humidity measurements made at ambient temperatures from (0 to 100 ℃):

$$\log_{10}(\rho_{H_2O}) = 10.79574\left(1-\frac{273.16}{T_{sat}}\right) - 5.02800\log_{10}\left(\frac{T_{sat}}{273.16}\right) + 1.50475\left(1-10^{-8.2969\left(\frac{T_{sat}}{273.16}-1\right)}\right) + 0.42873\times10^{-3}\cdot\left(10^{4.76955\left(1-\frac{273.16}{T_{sat}}\right)}-1\right) - 0.2138602$$

P_{H_2O} = vapor pressure of water at saturation temperature condition, kPa,

T_{sat} = saturation temperature of water at measured conditions, K

6.1.3 Calculate the air density using the ideal gas relationship and molecular weights of standard air and water:

$$\rho_{air} = \frac{p_{abs} \cdot M_{mix}}{R \cdot T_{amb}}$$
$$M_{mix} = M_{air} \cdot \left(1 - \chi_{H_2O}\right) + M_{H_2O} \cdot \chi_{H_2O}$$
$$\chi_{H_2O} = \frac{P_{H_2O}}{P_{abs}}$$

ρ_{air} = air density in balance environment, kg/m^3

P_{abs} = absolute pressure in balance environment, kPa

$R = 8.314472$ J/(mol · K)

T_{amb} = absolute ambient temperature of balance environment, °C

M_{mix} = molar mass of air in balance environment, g/mol

M_{air} = 28.96559, g/mol

M_{H_2O} = 18.01528, g/mol

χ_{H_2O} = amount of water in an ideal gas

P_{H_2O} = water vapor pressure at the measured dew point, °C, $T_{sat} = T_{dewpoint}$

P_{H_2O} = 1.18489 kPa **@** 9.5 °C.

P_{abs} = wet static absolute pressure at the location of your dew point measurement, kPa

6.1.4 Calculate the buoyancy corrected mass of each filter:

$$m_{corr} = m_{uncorr} \times \frac{1 - \dfrac{\rho_{air}}{\rho_{weight}}}{1 - \dfrac{\rho_{air}}{\rho_{media}}}$$

m_{corr} = PM mass corrected for buoyancy

m_{uncorr} = PM mass uncorrected for buoyancy

ρ_{air} = density of air in balance environment

ρ_{weight} = density of calibration weight used to span balance; 7950kg/m^3

ρ_{media} = density of PM sample media, such as a filter; 920 kg/m^3

6.1.5 Calculate the net mass of each filter using the formula below:

Net Mass= buoyancy corrected post-test weight – buoyancy corrected pre-test weight

6.2 Automated Weighing

6.2.1 The buoyancy correction is performed on each balance reading using the formula in 6.1.2, 6.1.3 and 6.1.4 before any consideration is given to the number of repetitions or drift correction.

6.2.2 For direct read with zero drift correction weighing method, the weighing result is the buoyancy corrected balance reading minus the average of the two empty pan readings before and after it.

6.2.3 For triplicate weighing, the weighing result is the average of three corrected balance readings calculated in 6.2.2.

6.2.4 Calculate the net mass of each filter using the formula below (AutoZero off):

Net Mass = buoyancy and zero drift corrected post-test weight – buoyancy and zero drift corrected pre-test weight.

7. QC Requirements

7.1 Continuously measure and record the dew point and temperature of the clean room to determine if the environment remains within the tolerances specified in Section 5.1.

7.2 The annual calibration (within 370 days) of the microbalance must be valid at the time of use.

7.3 Filter’s integrity

7.3.1 Manually inspect filter before weighing. If there is any separation of ring, flashing, scratches, or pinhole(s) found on the filters before sample collection, the pre-test filter shall be discarded.

7.3.2 If filter’s irregularities are found after sample collection, a senior staff shall be consulted before the filter is discarded. Record all filter’s irregularities in the LIMS database.

7.3.3 Should the particulate matter on the filters contact the Petri dish, tweezers, microbalance or any other surface, the data with respect to that filter is void and the record is registered in the LIMS database.

7.4 Three reference filters will be used to monitor for ongoing gross particle contamination within the room during the weighing session. Only two reference filters' average change in mass will be used to validate PM weighing procedures. The reference filters shall be the same size and material as the sample filters.

7.5 A new reference filter shall be used to replace the reference filter if the change in buoyancy corrected mass relative to the initial mass is greater than 8 μg, using good engineering judgments.

7.6 The change of buoyancy corrected net mass of all reference filters during each weighing session must not exceed ± 10μg or all filters in the process of equilibration are discarded and all data collected with respect to the failed reference filters is considered void. The reference filters must be replaced.

7.7 A trip blank can also be incorporated in the vehicle testing as a quality control check. If the buoyancy corrected net mass of a trip blank exceeds +/- 6μg, the test engineer will be notified and the data will be flagged in LIMS database.

7.8 Automated weighing: If, within the triplicate weighing, the difference between individual weighing result and the triplicate average exceeds ±2.5μg, the filter must be reweighed.

8 Maintenance

8.1 Clean room maintenance

A list of the clean room maintenance and its schedule is attached in appendix A. It is recommended by the clean room contractor - Pacific Environmental Technologies, Inc.

8.2 Automated Filter Weighing System maintenance

The FWS maintenance is provided by MTL on annual basis.

8.3 Microbalance maintenance

8.3.1 The microbalance is calibrated and certified annually by the manufacturer. Verify that the annual calibration is valid.

8.3.2 No special daily maintenance is required for the balance. It is recommended that the balance remains in the power ON condition unless the balance will not be used for several weeks or longer.

8.3.5 Immediately clean all spilled material. Wipe it up only with lint-free paper cloth.

8.4 Po^{210} electrostatic neutralizer bars are replaced annually.

9 Calibration Standard

One NIST traceable calibration weight of nominal100.0000 mg is used to check the balance calibration and weighing performance.

10 References

10.1 40 CFR Part 1065 Draft 7/11/2008

10.2 Operating instructions manual for Metter Toledo AX and MX/UMX microbalance.

Appendix A

			Task	None	Daily	Weekly	Monthly	Quarterly	6 Mos	Annually	Comments
Architectural											
			Check door seals							X	
Mechanical											
	Airhandler										
		Supply fan		X							Direct drive motor
		Dx Coil		X							
		Condensate Drain	Check for water in trap				X				Line should be dripping constantly (may have to elevate the line)
		Cabinet/enclosure	Check joints for leaks or evidence of rain intrusion					X			
	Filters										
		FA prefilter	Replace						X		
		RA prefilters	Replace					X			
		OSA V-pleat filter	Replace							X	Replace at least annually
		HEPA filters	Check Δp							X	Adjust Iris dampers to bring up flow as required (2~3 years)
	Refrigerant System										
		Outdoor Coil	Check Cleanliness						X		
		Outdoor Coil	Water wash							X	Minimum 1 x annually
		Refrigerant Chg	Check Pressures						X		•
		Sight glass	Check for bubbling					X			
		Elec Connections	Check for tightness and visual heating						X	X	6 months first year, then annually
	Humidifier										
		Hose connections	Tighten						X		6 months first year, then annually
		Tank	Check for visual chronic leaks						X		
		Elec Connections	Check for tightness and visual heating problems						X	X	6 months first year, then annually
		Airflow switch	Check pneumatic hoses for tightness						X		Inside roof cabinet
		High limit humidity switch	Check for function						X		Adjust setting and check if HU shuts down
		DI water line	Check for leaks						X		Outside & below cabinet
	Ductheater										
		Elec Connections	Check for tightness and visual heating problems						X	X	6 months first year, then annually
Electrical											
	Lamps										
		Interior lamps									Replace as needed
	VFD	Elec Connections	Check for tightness and visual heating problems						X	X	6 months first year, then annually
Certification and Calibration											
	Vaisala controllers		Calibrate							X	or per ARB SOP
	Hot gas valve		Test							X	Test function only if performanc problems are evident
	Airflow		Certify							X	Measure duct flow with hot wire
	Particle count		Certify							X	or per ARB SOP
	Iris dampers		Adjust							X	Check only if air flow is not in balance with each HEPA filter
	Room Velocity		Certify							X	per ARB SOP