California Environmental Protection Agency

Air Resources Board

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

SOP MV-AEROSOL-145
Version 5.2

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1 Introduction

1.1 This document describes the methodology used by the Aerosol Analysis and Method Evaluation Section (AAMES) of Emission Compliance, Automotive Regulations and Science Division (ECARS) 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 (Tier 3 Final Rule, dated June, 2014) 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, 300 series stainless steel tweezers 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 cleanliness of the weighing room in compliance with ISO 14644-1, analysts shall wear shoe covers, low-lint garments and powder-free gloves.

4 Instruments and Materials

4.1 Temperature and dew point controlled cleanroom that meets the Class Six specifications for cleanliness according to ISO 14644-1.

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 XP2U 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 ± 1 °C
Dew point:	9.5 ± 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 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.

5.3.3 Automated weighing: Verify the balance repeatability, using internal calibration weights, and performing 10 warm-up repetitions.

5.3.4 Manual weighing: Perform manual adjustment (zero and span) using the internal calibration weights and/or an external NIST traceable weight (nominal 100.0000 mg) 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 environmental 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 the 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 Po^{210} 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 (Po^{210}).

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 °C):

$$\log_{10}(P_{H_2O}) = 10.79574 \cdot \left(1 - \frac{273.16}{T_{sat}}\right) - 5.02800 \cdot \log_{10}\left(\frac{T_{sat}}{273.16}\right) + 1.50475 \cdot 10^{-4}\left(1 - 10^{-8.2969 \cdot \left(\frac{T_{sat}}{273.16} - 1\right)}\right)$$
$$+ 0.42873 \times 10^{-3} \cdot \left(10^{4.76955 \cdot \left(1 - \frac{273.16}{T_{sat}}\right)} - 1\right) - 0.2138602$$

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:

$$\rho_{air} = \frac{P_{abs} \cdot M_{mix}}{R \cdot T_{amb}}$$

$$M_{mix} = M_{air} \cdot (1 - \chi_{H_2O}) + M_{H_2O} \cdot \chi_{H_2O}$$

$$\chi_{H_2O} = \frac{P_{H_2O}}{P_{abs}}$$

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

P_{abs} - Absolute pressure in balance environment, kPa

M_{mix} - Molar mass of air in balance environment, g/mol

R - Molar gas constant; R = 8.314472 J/(mol·K)

T_{amb} - Absolute ambient temperature of balance environment, K

M_{air} = 28.96559 g/mol

M_{H_2O} = 18.01528 g/mol

χ_{H_2O} - Amount of water in an ideal gas, mol/mol

6.1.4 Calculate the buoyancy corrected mass of each filter:

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

m_{corr} - PM mass corrected for buoyancy

m_{uncorr} - PM mass uncorrected for buoyancy

ρ_{air} - Density of air in balance environment, kg/m^3

ρ_{weight} - Density of calibration weight used to span balance; e.g. 7950 kg/m^3

ρ_{media} - Density of PM filter; e.g. 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 or on the chain of custody form.

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 8µg, 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 is also 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 their mean exceeds ±2.5µg, the filter must be reweighed.

8 Maintenance

8.1 Clean room maintenance

Remedial and preventive maintenance services for the cleanroom are performed annually. The cleanroom certification tests are performed once a year to verify the compliance with US EPA 40 CFR, Part 1065, 2011 and ISO 14644-1 specifications.

8.2 Automated Filter Weighing System maintenance

The FWS is recalibrated and verified to meet the performance specifications annually.

8.3 Microbalance maintenance

8.3.1 The microbalance is calibrated and certified biannually by the manufacturer. Verify that the biannual 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 biannually.

9 Calibration Standard

One NIST traceable calibration weight of nominal 100.0000 mg is used to check the balance calibration and weighing performance.

10 References

10.1 US EPA 40 CFR Part 1065 Engine-Testing Procedures Final HD GHG FRM

10.2 ISO 14644-1 Cleanrooms and associated controlled environments – Part 1: Classification of air cleanliness

10.3 Operating Instructions manual for Automated Filter Weighing System – Measurement Technology Laboratories

10.4 Operating Instructions manual for XP/UMX microbalance, Metter Toledo AG Laboratory & Weighing Technologies

11 Revision History

11.1 Revised SOP language to reflect references to ISO 14644-1 and the final revision of 40 CFR Part 1065 dated 8/8/2011

11.2 Corrected a typing error; 10^{-4} was missing from the equation 6.1.2

11.3 Made minor editorial changes

11.4 Revised the SOP name from SOP No. MLD 145 to SOP MV - AEROSOL-145 due to Division and Branch name change