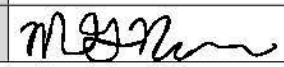
State of California
California Environmental Protection Agency
Air Resources Board
MLD/QMS-066 (NEW 4/14)

QUALITY MANAGEMENT DOCUMENT ADDENDUM

(District completes Sections 1 through 6 -- please type)

Section 1. ARB Document
☐ Quality Management Plan (QMP)
☐ Quality Assurance Project Plan (QAPP)
☒ Standard Operating Procedure (SOP)

Section 2. District Information		
District Name:	CARB – Monitoring and Laboratory Division – Northern Laboratory Branch	
District Address:	1927 13th St, Sacramento, CA 95811	
District Contact Name/Phone Number:	Maria Navarro	279-208-7904
District Signature/Date:		7/18/24

Section 3. Document Title *(specify exact title, revision #, and date of ARB Document(s) that your District proposes to modify)*	**Date**
Standard Operating Procedure for Determination of PM10 Mass by Gravimetric Analysis, MLD 016 Revision 7.1	9/27/2022

Section 4. Proposed Deviation(s)
(specify exact section(s), page number(s) and language in existing ARB document that your District proposes to modify and then specify proposed modification (including any spreadsheets or forms).

See next page.

Current Section 9.3.2

The following outlines a recommended analytical sequence procedure for both pre- and post- weighing sessions:

- Control (4 g)
- Low Standard check (3 g)
- High Standard check (5 g)
- Samples 1 through 9
- Replicate of sample 1
- Low Standard check (3 g)
- Samples 10 through 18
- Replicate of sample 10
- Low Standard check (3 g)
- High Standard check (3 g)

Update to:

The following outlines a recommended analytical sequence procedure for both pre- and post- weighing sessions:

- Control (4 g)
- Low Standard check (3 g)
- High Standard check (5 g)
- Samples 1 through 9
- Replicate of sample 1
- Low Standard check (3 g)
- Samples 10 through 18
- Replicate of sample 10
- Low Standard check (3 g)
- High Standard check (5 g)

Current Section 9.8

9.8 Required Records for Weighing Session

The required records for every weighing session are a Daily Conditions graph, the LIMS Transfer Results report, and the actual samples weighed with the COCs.

9.8.1 Each sample weighed is contained in a labeled manila folder, and includes the filter, the matching COC specific for each filter, and the glassine envelope. Post-weighed samples also contain a sampler record, such as a Dickson Chart.

9.8.2 Review the weighing session records and, if correct, then transfer the weighing records and the actual samples to the peer reviewer for their review. The analyst must initial and date all weighing records reviewed. For pre-weights, LIMS electronically includes the analyst and date on the COCs. For post-weights, the analyst must handwrite the initials and date on the COCs.

9.8.3 The peer reviewer must verify the Daily Conditions Graph is complete, each COC matches the actual filter exactly, and each COC matches the weights shown in the LIMS Transfer Results report, including filter conditioning start and the dated initials of the analyst for each sample weight, all QC, replicate weight, and filter blank calculated difference. Peer reviewer must date and initial the Daily Conditions Graph showing filter conditioning start and 24-hour conditioning averages and SD, and the LIMS Transfer Results report. Peer review must be completed before pre-weighed filters are used to collect air samples.

9.8.3.1 Any changes made to the data after it has been transferred to LIMS must be documented, initialed, and dated on the LIMS transfer report and corresponding COC.

9.8.4 Once peer review of a LIMSLink data transfer report is complete file the reviewed report in the appropriate binders.

Update to:

9.8 Peer Review Package for Weighing Session and Archive

The peer review package for Weighing Session includes the following documents: Peer Review Checklist, Daily Conditions graph, LIMS Transfer Results report, and labeled manila folders with COC, filter, and glassine. Post-weighed samples include a sampler Dickson Chart that's stapled to the COC.

9.8.1. After weighing is complete, prepare the peer review package. For pre-weights, prepare peer review package with Pre-weight Peer Review Checklist. For post-weights, prepare peer review package with Post-weight Peer Review Checklist. Examples of both Peer Review Checklists are included in Appendix A.

9.8.2. Analyst must review the peer review package and make sure data is complete and all documents are initialed and dated including the Daily Conditions graph.

9.8.3. The peer reviewer must use the peer review checklist to verify data is complete and that analyst initialed and dated all documents. In addition, the peer reviewer must sign the Peer Review Checklist, and initial and date both the Daily Conditions graph and the LIMS Transfer Results report.

9.8.3.1. If any discrepancies, notify analyst immediately.

9.8.3.2. Any changes made to the data after it has been transferred to LIMS must be documented, initialed, and dated on the LIMS transfer report and corresponding COC.

9.8.4. When peer review is complete, return Peer Review Package to analyst.

9.8.5. Review the data package and confirm the peer reviewer signed the Peer Review Checklist, and initialed and dated both the Daily Conditions Graph and LIMS Transfer Results report.

9.8.6. Archive the Peer Review Checklist, Daily Conditions graph, and LIMS Transfer Results Report in the appropriate binders. Refer to 11.5 for record retention requirements.

9.8.7. PM10 filters are archived as received in manila folders to protect samples from damage. Organize the manila folders by site then sampling date and place in the designated banker box. Refer to 11.5 for record retention requirements.

Appendix A

Update to add PM Peer Review Checklists.

See next page.

Update to:

6. EXAMPLE OF PRE-WEIGHTS PM10 PEER REVIEW CHECKLIST

Pre-weights -- Peer Review Checklist for PM10 Hi-Vol
Determination of PM10 Mass in Ambient Air by Gravimetric Analysis

LIMS QC Batch ID: _____ Date: _____ Batch ____ of ____
For this Batch, the content of the COCs generated by LIMS were verified: _____

1. Controlled-environment Room Conditions printout shows, at a minimum:

a.	**Graphical summary** of conditions for the period at least 24 hours prior to the START of the pre-weighing session	☐ Yes	☐ No
b.	**Temperature (Temp):** recorded on Conditions graph are calculated 24-hour average (Ave) within 15 – 30ºC and calculated standard deviation (SD) within ± 3 °C	☐ Yes	☐ No
c.	**Percent Relative Humidity (% RH):** recorded on Conditions graph are calculated 24-hour average (Ave) % RH within 30 – 40% RH and calculated standard deviation (SD) within ± 5% RH	☐ Yes	☐ No
d.	**START and STOP conditions** recorded by Analyst on Room Conditions graph meet above criterion	☐ Yes	☐ No

2. "PM10 Hi-Vol Transfer Results" printout:

a.	Analyst initialed and dated the LIMS Transfer Results printout	☐ Yes	☐ No
b.	PM10 Daily Balance Control result of 4 g (1 g and 3 g weights combined, i.e., 4 g) is within ± 0.0005 g *This control appears on Row #1 of Batch 1 for the day:* _____	☐ Yes	☐ No
c.	Standard Checks (3 g & 5 g) bracket samples at beginning, alternating every nine samples, and at the end	☐ Yes	☐ No
d.	All Standard Checks are within ± 0.0005 g	☐ Yes	☐ No
e.	All PM10 Pre-weight Replicates are within ± 0.0028 g	☐ Yes	☐ No

3. Each pre-weighed filter in this batch:

a.	A manila folder along with a glassine and a COC	☐ Yes	☐ No
b.	Label applied to manila folder tab shows LIMS ID (example: S200123456) matching the LIMS ID printed on the COC	☐ Yes	☐ No
c.	Filter Number (example: 9876543) printed on the COC matches filter number stamped on the actual filter in the folder	☐ Yes	☐ No

4. Peer Reviewer:

a.	Peer Reviewer confirms numeric sequence on PM10 Hi-Vol Transfer Results is complete – no missing Row numbers	☐ Yes	☐ No
b.	Peer Reviewer initialed and dated the "Reviewed by" line of the Transfer Results	☐ Yes	☐ No
c.	Peer Reviewer initialed and dated the Room Conditions printout	☐ Yes	☐ No
d.	Any discrepancies- notify PM10 analyst immediately	☐ Yes	☐ No

Peer Reviewer _____ Date: _____

7. EXAMPLE OF POST-WEIGHTS PM10 PEER REVIEW CHECKLIST

Post-weights -- Peer Review Checklist for PM10 Hi-Vol
Determination of PM10 Mass in Ambient Air by Gravimetric Analysis

LIMS QC Batch ID: _____ Date:_____ Batch ____ of ____

1. Controlled-environment Room Conditions printout shows, at a minimum:

a.	**Graphical summary** of room conditions shows period of at least 24 hours prior to the START of the post-weighing session	☐ Yes	☐ No
b.	**Temperature (Temp):** recorded on the Conditions graph are calculated 24-hour average (Ave) within 15 – 30°C and calculated standard deviation (SD) within ± 3°C	☐ Yes	☐ No
c.	**Percent Relative Humidity (% RH):** recorded on the Conditions graph are calculated 24-hour average (Ave) % RH within 30 – 40% RH and calculated standard deviation (SD) within ± 5% RH	☐ Yes	☐ No
d.	**START and STOP conditions** recorded by Analyst on Room Conditions graph meet above criterion	☐ Yes	☐ No

2. "PM10 Hi-Vol Post-Weight Transfer Results" printout:

a.	Analyst initialed and dated the Reported by" line on the PM10 Hi-Vol Post-Weight Transfer Results printout	☐ Yes	☐ No
b.	PM10 Daily Balance Control result of 4 g (1 g and 3 g weights combined, i.e., 4 g) is within ± 0.0005 g (LIMS ID = Calib Wt) *This control appears on Row #1 of Batch 1 for the day:*_____	☐ Yes	☐ No
c.	Standard Checks (3 g & 5 g) bracket samples at beginning, alternating every nine samples, and at the end	☐ Yes	☐ No
d.	All Standard Checks are within ± 0.0005 g	☐ Yes	☐ No
e.	All PM10 Post-weight Replicates are within ± 0.0050 g	☐ Yes	☐ No
f.	LIMS Status is "Completed" for each result shown on the PM10 Hi-Vol Post-Weight to LIMS printout	☐ Yes	☐ No
g.	Filter numbers and post-weights on the PM10 Hi-Vol LIMS printout match the filter numbers and post-weights indicated on the COCs	☐ Yes	☐ No

3. Peer Reviewer:

a.	Peer Reviewer confirms numeric sequence on the PM10 Hi-Vol Transfer Results is complete – no missing Row numbers	☐ Yes	☐ No
b.	Peer Reviewer initialed and dated the "Reviewed by" line of the Transfer Results	☐ Yes	☐ No
c.	Peer Reviewer initialed and dated the Graphical Summary of the Room Conditions printout	☐ Yes	☐ No
d.	Any discrepancies- notify PM10 analyst immediately	☐ Yes	☐ No

Peer Reviewer _____ Date: _____

Current Table of Contents

Update to:

Section 5. Justification for Deviation(s) *(provide explanation of why modification(s) to existing ARB document is necessary)*
Proposed modifications aim to correct a typo in Section 9.3.2, clarify contents of peer review package, and add example Peer Review checklists to Appendix A aligning with the current PM10 Peer Review Procedure.

Section 6. Attachment(s) ☐ *(specify attachment titles and number of pages, include modified spreadsheets or forms)*	**# of Pages**

Section 7. ARB Approval *(completed by ARB)*		
Name/Phone Number:	Manisha Singh, Ph.D.	279-208-7896
Title:	Chief, Quality Management Branch	
Signature/Date:	Singh	7/15/2024
Addendum Number	A49	

Completed form must be scanned/emailed or mailed to:

Quality Management Section
1927 13th Street, P.O. Box 2815
Sacramento, California 95811
qa@arb.ca.gov or the email of your PQAO liaison

CALIFORNIA
AIR RESOURCES BOARD

Standard Operating Procedure for Determination of PM_{10} Mass by Gravimetric Analysis

MLD016
Revision 7.1

Northern Laboratory Branch
Monitoring and Laboratory Division

Approval Signatures	Approval Date
Manisha Singh, Ph.D., Chief Quality Management Branch	9/23/2022
Michael Werst, Chief Northern Laboratory Branch	9/27/2022

Disclaimer: Mention of any trade name or commercial product in this standard operating procedure does not constitute endorsement or recommendation of this product by the California Air Resources Board. Specific brand names and instrument descriptions listed in the standard operating procedure are for equipment used by the California Air Resources Board's laboratory. Any functionally equivalent instrumentation is acceptable.

Table of Contents

List of Tables

Standard Operating Procedure
Determination of PM_{10} Mass by Gravimetric Analysis

1. Introduction

This method provides the laboratory procedures for gravimetric measurement of particulate matter with an aerodynamic diameter less than or equal to a nominal ten micrometers (PM_{10}) collected on quartz microfiber filters from exposure to ambient air over a 24-hour period, in accordance with Code of Federal Regulations (CFR) Title 40, Chapter 1, Subchapter C, Part 50, Appendix J (40 CFR, Part 50, Appendix J). Quality assurance requirements for these procedures are provided in "*Quality Assurance Handbook for Air Pollution Measurement Systems Volume II*," EPA-454/B-17-001, January 2017.

The sampling of PM_{10} follows the United States Environmental Protection Agency (U.S. EPA) monitoring schedule. The monitoring schedule is referenced in CFR Title 40, Chapter 1, Subchapter C, Part 58, Subpart B.

2. Summary of Method

PM_{10} mass is determined by gravimetric analysis in an environmental controlled room. The difference between the pre-weight of a filter and the post-weight of the same filter after sampling is used to quantify the mass. Gravimetric measurements of PM_{10} generated by this method are converted to aerometric concentrations for determining attainment and maintenance of Federal and State ambient air quality standards specified in § 50.6 of 40 CFR Part 50 and California Health Safety Code (Sections 425, 39606), respectively. The gravimetric measurement process is nondestructive, and the PM_{10} sample can be subjected to subsequent physical or chemical analyses following gravimetric analysis.

3. Acronyms and Definitions

Table 3. Acronyms and Definitions

Acronym or Term	Definition
°C	Degrees Celsius
AQSB	Air Quality Surveillance Branch
CARB	California Air Resources Board
ASTM	American Society for Testing and Materials
CFM	Cubic feet per meter
CFR	Code of Federal Regulations
COC	Chain of Custody: A hardcopy of the historical record of the sample filter media through the entire PM_{10} sampling process; the

Acronym or Term	Definition
	laboratory pre-sampling analysis of the filter, the field handling of the filter for PM_{10} sample collection, and the laboratory post-sampling analysis of the filter.
Daily conditions graph	A hardcopy graph of the controlled conditions of the Balance Room.
g	Grams
LIMS	Laboratory Information Management System: Database containing sample metadata, raw and reported concentration results, and quality control samples and results.
K	Kelvin
kPa	Kilopascal
LIMSLink	Software allowing review of raw sample data and quality control results, and transfer of data electronically retrieved from the analytical balance to Laboratory Information Management System (LIMS).
LTP	Local temperature and pressure
m^3	Cubic meter
m^3/min	Cubic meter per minute
MFC	Mass flow controller
mg	Milligram
MLD	Monitoring and Laboratory Division
mm Hg	Millimeters mercury
NAAQS	National Ambient Air Quality Standard
NIST	National Institute of Standards and Technology
NLB	Northern Laboratory Branch
PM_{10}	Particulate matter with an aerodynamic diameter less than or equal to a nominal ten micrometers
QC	Quality control
RH	Relative humidity
SCFM	Standard cubic feet per meter
SD	Standard deviation
SOP	Standard operating procedure
STP	Standard temperature and pressure
Temp	Temperature
U.S. EPA	United States Environmental Protection Agency
VFC	Volumetric flow controller
Weighing chamber	A clear glass enclosure placed over the balance to minimize the effects of air movement on balance readings.
Weighing rack	A removable stand placed on balance pan and tared to hold filters for weighing.
Weighing session	The period to complete weighing indicated on one LIMSLink worksheet.

4. Interferences

4.1 The moisture content of a filter affects its weight. Factors affecting the moisture content of filters are temperature (Temp) and relative humidity (RH). Filters must be equilibrated for moisture content in the same controlled conditioning environment used for weighing.

4.2 Powder-free and anti-static gloves must be worn when handling the filters, the balance, or the weights to prevent contamination from body moisture and oils.

4.3 Static electricity can build up on the quartz filters causing erroneous mass results or failure of the balance to stabilize. Antistatic devices, such as polonium antistatic strips, are kept in the balance weighing chamber to dissipate static charge. These devices must be replaced annually. In addition, use non-metallic, plastic forceps to reduce static build-up. Metal forceps, even if Teflon-tipped, may contribute to static build-up near the balance. Antistatic wet wipes are used to clean surfaces and minimize static build-up.

4.4 Airborne particulates from dust contamination can lead to inaccurate mass measurements. Dust contamination can be reduced by maintaining clean laboratory benchtops, tidy weighing areas, application of an adhesive entryway mat, and wearing a clean lab coat over regular clothing.

4.5 Retention of gaseous species on the quartz filters at any point can yield a variety of organic and inorganic artifacts, which could affect the total mass attributed to PM_{10}. Eliminate exposure of PM_{10} filters to the vapors of cleaning solvents and industrial-strength detergents. Work surfaces in the Balance Room should be dusted using antistatic wipes or disposable lab towels moistened with Nanopure water.

4.6 An anti-vibration table must be used to offset motion impacts to the balance during filter weighing. Use of the balance on an unstable surface will adversely affect mass measurements.

4.7 Air conditioning ductwork, printers, and frequently opened doorways may create undue air flow. This air flow can adversely affect mass measurements. Appropriate placement of the balance, use of a double door entryway, and not weighing when the fan is blowing can minimize airflow on the instrument.

5. Personnel Qualifications and Training

Prior to performing this method, new personnel must be trained by staff with detailed knowledge of this method. Personnel must be trained to understand the program's

requirements per any applicable State and federal regulations and guidance, and this Standard Operating Procedure (SOP). Personnel will also be trained on how to safely and properly operate the equipment needed to perform the method, the quality assurance components, and LIMS functionality pertaining to the program.

Personnel should provide an initial demonstration of capability prior to performing this method on real-world samples (i.e., data for record). Training will be documented and maintained by the laboratory supervisor.

6. Safety Requirements and Hazardous Waste

All personnel must follow the general health and safety requirements found in Northern Laboratory Branch's (NLB) Chemical Hygiene Plan.

Radioactive (alpha particle) Polonium-210 antistatic strips for static charge neutralization are used in the laboratory. While it is not a hazard to outside of the body, Polonium-210 is a radiation hazard if it enters the body through inhalation, digestion, or open skin wounds. Make sure to wear gloves specified in Section 7.15 to handle the antistatic strips. Upon receipt of new strips, return used strips to the manufacturer for proper disposal.

7. Equipment, Supplies, and Chemicals

7.1 Balance Room, a controlled environment. See Section 9.1.

7.2 Two Temp and RH data loggers and sensor probes.

7.2.1 All sensor probes installed must be certified as calibrated National Institute of Standards and Technology (NIST)-traceable annually by an outside source. If existing sensor probes cannot be re-certified for calibration, the existing sensors must be replaced with sensor probes that have been certified as calibrated NIST-traceable within the previous 12-months or less. Minimal performance specifications of sensor probes must allow connectivity and necessary communication with an environmental monitoring system, and have adequate quality to compare with CFR criteria.

7.3 Laboratory Information Management System (LIMS) and LIMSLink.

7.4 Analytical balance equipped with a balance pan, a removable filter weighing rack, and be enclosed in a weighing enclosure.

7.4.1 Balance must have a minimum resolution of 0.1 milligram (mg) and minimum precision of 0.5 mg and must have electronic data transfer interface with LIMSLink.

7.4.2 Filter weighing rack must be sized to fit on the balance pan and securely hold 8” x 10” filter media with the balance door fully closed.

7.5 Anti-vibration table (e.g., balance table made of granite or marble).

7.6 Two 1 gram (g) weights categorized by American Society for Testing and Materials (ASTM) as Class 1, non-corroding, and certified as NIST-traceable. Each weight has a certification document with serial number identification.

7.7 Two 3 g weights categorized by ASTM as Class 1, non-corroding, and certified as NIST-traceable. Each weight has a certification document with serial number identification.

7.8 Two 5 g weights categorized by ASTM as Class 1, non-corroding, and certified as NIST-traceable. Each weight has a certification document with serial number identification.

7.9 PM_{10} filters, 8” x 10” high purity quartz microfiber filters (typically supplied by U.S. EPA).

7.10 Light inspection table.

7.11 Antistatic strips for static charge neutralization typically containing radioactive (alpha particle) Polonium-210. Minimum of two strips must be placed inside the balance chamber to dissipate static charge. Strips must be replaced annually.

7.12 Antistatic brush; radioactive insert of brush must be replaced annually.

7.13 Non-metallic, plastic forceps.

7.14 Timer.

7.15 Antistatic and powder free gloves.

7.16 PM_{10} sample Chain-of-Custody (COC) forms.

7.17 Self-adhesive labels.

7.18 U.S. EPA-issued monitoring sampling schedule.

7.19 Mail out supplies to include:

Manila folders (8 ½” x 11”)
Glassine envelopes (8 ¼” x 10 ¼”)
Clasp envelopes (10” x 13”).

7.20 Adhesive entryway mat or sticky mat.

7.21 Disposable laboratory towels or wipes to dust surfaces.

7.22 Antistatic wet wipes to clean surfaces and minimize static build-up.

7.23 Nanopure water (ASTM Type I water, electrical resistivity >18 MΩ-cm at 25 degrees Celsius (°C).

8. Filter Handling and Inspection

8.1. Filter Handling

PM_{10} filters are constructed of compressed quartz microfibers and are subject to sheering and breakage. Sampled PM_{10} filters holding collected particulate matter may be very brittle. Analysts must handle all PM_{10} filters with care.

8.2. Pre-Sampled Filter Inspection

All new filters must be visually inspected to determine whether usable or unusable for sampling.

8.2.1 Clean the light inspection table and the surrounding inspection area with a disposable laboratory wipe moistened with Nanopure water. Use a dry wipe to make sure the area is clean and dry.

8.2.2 Place pre-sampled quartz microfiber filter flat on the light inspection table, inspecting both sides for any defects. Staff must carefully inspect filters using criteria set forth in Appendix A of this SOP.

8.2.3 Place usable filter into a manila folder, followed by a glassine envelope.

8.2.4 Usable filters must be conditioned in an environmentally controlled Balance Room for weighing. See Section 9.1

8.3. Post-Sampled Filter Inspection

All sampled filters must be visually inspected for validation. All samples, whether valid or invalid, are analyzed.

8.3.1 The post-sampled PM_{10} filter must be received in the lab inside a glassine envelope, folded in half lengthwise, with the sampled particulates contained inside the folded filter. Carefully remove the folded filter from the glassine envelope. Place the empty glassine

envelope on top of the open manila folder to create a post-sampled filter inspection area exclusive for each filter. Unfold the filter fully for inspection of the particulate collection side. Do not touch the collected particulate area.

If the PM_{10} filter is received outside of the glassine envelope, the sample is invalid.

8.3.2 Inspect post-sampled filters prior to conditioning. See Section 9.1 for conditioning. See Appendix A for inspection.

8.3.3 If invalidated, document the reason in the “Comments” section of the COC form.

8.3.4 The filter number stamped on each filter by the manufacturer is unique. In turn, the LIMS ID assigned to each filter number is also unique. The stamped filter number of each post-sampled filter received by the lab must match the filter number specified on the COC for the assigned LIMS ID. The LIMS ID on the manila folder holding the filter must match the COC. If any of these numbers do not match, the sample is invalid.

8.3.5 Return the inspected filter to its manila folder with the sampled side folded inward. Ink from the COC may transfer to filter, which invalidates the sample. The folded filter should only rest against the glassine envelope and the manila folder to prevent ink contamination.

8.3.6 Place the manila folders in an environmentally controlled Balance Room to equilibrate with the conditions. See Section 9.1. Note the Filter Conditioning Start on the COC to document equilibration is met. See Section 9.2.5.

9. Procedures for Weighing

Analysts must wear powder-free and antistatic gloves for all procedures for handling weights, forceps, balance pan, chamber doors, weighing rack, and all filter media.

All weighing must be performed with all glass enclosure doors closed. There must be two antistatic strips located inside the balance chamber to dissipate static charge.

9.1 Filter Conditioning in Controlled Environment

The Balance Room is a controlled environment for Temp and RH conditions. Sensor probes in the Balance Room electronically record conditions of Temp

and RH every 2 minutes. Every filter must be exposed without interruption to the controlled environment for at least 24-hours prior to and during all weighing.

9.2 Verification of Filter Conditioning and Weighing Conditions

9.2.1 Before starting the first weighing session of each date, download the Temp and RH readings of the controlled environment representing the 24-hour period of readings before weighing begins. Open the downloaded readings in a spreadsheet program.

9.2.2 Use spreadsheet cell formulas to separately calculate the 24-hour average and standard deviation (SD) for the Temp and RH parameters.

Table 9.2.2 Calculation of Average and SD - Daily

Parameter	Cell formula	Example
Average	=Average(cell start:cell stop)	=Average(F2:F722)
SD	=STDEV(cell start:cell stop)	=STDEV(F2:F722)

9.2.3 Verify that the calculated results for Temp and RH parameters meet the acceptance criteria before starting a weigh session. See Section 10.1.

9.2.4 The 24-hour conditions file is saved with a file name that indicates the conditioning date range. Example: 01012018 to 01022018.xls.

9.2.5 Print the Daily Conditions graph depicting the Temp and RH readings for the 24-hour conditioning period. The graph can be printed directly from the sensor probe website or using an email sent to the analyst by the website. On the print of the Daily Condition graph, handwrite the filter conditioning and weighing conditions for each weighing session as verification.

Table 9.2.5 Verification of Weighing Conditions – Daily

Verification record	Details to include
Filter Conditioning Period	24-hour Filter Conditioning period (00:00 to 00:00)
Filter Conditioning Start	Start date and time (mm/dd/yy 00:00) along with the starting parameters

	(Temp and RH) as indicated on downloaded readings file for the filter conditioning period
24-hour Conditioning Calculated Results	Averages +/- SD for Temp and RH
Weighing Conditions for each weigh session	Start and Stop times of each weighing session along with the observed current Temp and RH readings

9.3 Recommended Analysis Sequence

9.3.1 Weigh the Control as the first sample of the first weighing batch of the day, followed by the low and high standard checks. Next, consecutively weigh filters in groups of 9 or less filters, along with the group replicate. A group replicate must be assigned to the first sample of each sample group of 9 or less filters. Analyzed consecutively between each filter group is the alternatingly Low and High Standard Check. After the last replicate sample in the session, the last samples weighed must be the Low Standard Check (3 g) followed by the High Standard check (5 g).

9.3.2 The following outlines a recommended analytical sequence procedure for both pre- and post- weighing sessions:

- Control (4 g)
- Low Standard check (3 g)
- High Standard check (5 g)
- Samples 1 through 9
- Replicate of sample 1
- Low Standard check (3 g)
- Samples 10 through 18
- Replicate of sample 10
- Low Standard check (3 g)
- High Standard check (5 g)

9.4 Setup the LIMSLink Worksheet for Every Weighing Session

9.4.1 Populate a LIMSLink worksheet with the Analysis Sequence for filter samples to be weighed and all quality control (QC) measures.

9.4.1.1 For pre-weighing session, verify that each filter number appearing on the worksheet is an identical match of the number on the actual filter.

9.4.1.2 For post-weighing session, verify that each LIMS ID as well as filter number appearing on the worksheet is an identical match on the actual sample COC. Also verify the LIMS ID on the manila folder matches the COC and worksheet.

9.4.2 Enter the details for the Filter Conditioning Start into the LIMSLink worksheet header. See Section 9.2.5.

9.4.3 Proceed a weighing session by following the recommended analytical sequence. Review all data transferred to the LIMSLink worksheet for accuracy prior to transferring data to LIMS.

9.4.4 Generate LIMS Transfer Results report.

9.4.5 For pre-weighing session, print the COC for each filter using LIMS. Verify that each COC matches the transfer report exactly. Check the LIMS ID, filter number, pre-weight mass, conditioning parameters indicated on the Daily Conditions Graph, analysis date and analyst.

9.5 Verification of Balance Calibration – Daily

9.5.1 The record of the control weight is used as verification of the balance calibration. On every weighing date, the first sample weighed of the first weighing batch must be the control.

9.5.2 The control is created by placing the 1 g and 3 g weights together on the balance to yield the target weight of 4 g.

9.5.2.1 Tare the balance with the weighing rack on the balance pan before placing the control weights.

9.5.2.2 Wait until the mass reading has stabilized for 4 seconds on the balance and then record the value.

9.5.3 An acceptable control weight must be ± 0.0005 g of the target.

9.6 Weighing Standard Checks

9.6.1 Two standard checks (3 g, 5 g) are independently weighed by following the recommended analytical sequence.

9.6.1.1 Tare the balance with the weighing rack on the balance pan before placing the standard check weight.

9.6.1.2 Wait until the mass reading has stabilized for 4 seconds on the balance and then record the value.

9.6.2 An acceptable standard check weight must be ± 0.0005 g of the target.

9.7 Weighing a Quartz Microfiber Filter

9.7.1 Remove the filter weighing rack off the balance pan. Dust the rack with the antistatic brush.

9.7.2 Return the dusted filter weighing rack to the balance pan. Allow the balance to stabilize. Tare the balance.

9.7.3 Place a PM_{10} filter on the filter weighing rack of the tared balance. Confirm the filter is not touching the balance pan or balance wall. Confirm all balance doors are closed. Verify that the filter numbers on the filter and corresponding worksheet match while allowing the filter mass to stabilize on the balance.

9.7.4 Ensure mass is stabilized before submitting the filter weight reading on the balance to the LIMSLink worksheet. If the weight value on the balance does not fluctuate for 4 seconds, the mass is stable.

9.7.4.1 Quartz microfiber filters must have a mass between 3.7 g to 4.7 g. Any quartz fiber filter pre-weighing outside this acceptable range must be rejected and removed from the set of useable PM_{10} filters.

9.7.4.2 For post-weighing of filters, manually record the post-weight value, including each replicate, as well as Filter Conditioning Start onto the COC. Initial and date.

9.7.5 Place the weighed filter back to its respective manila folder along with the glassine enclosure.

9.8 Required Records for Weighing Session

The required records for every weighing session are a Daily Conditions graph, the LIMS Transfer Results report, and the actual samples weighed with the COCs.

9.8.1 Each sample weighed is contained in a labeled manila folder, and includes the filter, the matching COC specific for each filter, and the

glassine envelope. Post-weighed samples also contain a sampler record, such as a Dickson Chart.

9.8.2 Review the weighing session records and, if correct, then transfer the weighing records and the actual samples to the peer reviewer for their review. The analyst must initial and date all weighing records reviewed. For pre-weights, LIMS electronically includes the analyst and date on the COCs. For post-weights, the analyst must handwrite the initials and date on the COCs.

9.8.3 The peer reviewer must verify the Daily Conditions Graph is complete, each COC matches the actual filter exactly, and each COC matches the weights shown in the LIMS Transfer Results report, including filter conditioning start and the dated initials of the analyst for each sample weight, all QC, replicate weight, and filter blank calculated difference. Peer reviewer must date and initial the Daily Conditions Graph showing filter conditioning start and 24-hour conditioning averages and SD, and the LIMS Transfer Results report. Peer review must be completed before pre-weighed filters are used to collect air samples.

9.8.3.1 Any changes made to the data after it has been transferred to LIMS must be documented, initialed, and dated on the LIMS transfer report and corresponding COC.

9.8.4 Once peer review of a LIMSLink data transfer report is complete file the reviewed report in the appropriate binders.

10. Quality Control

QC criteria must be met for all PM_{10} Balance Room operations, filter conditioning, and weigh sessions.

10.1 Balance Room criteria for PM_{10} Filter Conditioning and Weighing is shown in Table 10.1.

Table 10.1 Balance Room Criteria for PM_{10} Filter Conditioning and Weighing

Criteria	Corrective Action
Temp: The Balance Room mean Temp must be within 15°C to 30°C for the previous 24 hours before any weighing occurs. The previous 24-hour SD must also not exceed 3°C.	No weighing shall occur until the room is within limits for a period of 24 hours. Determine if HVAC system needs repair or adjustment and schedule if needed. If the weighing

Criteria	Corrective Action
RH: The Balance Room mean RH must fall within 30% to 40% for the previous 24 hours before any weighing occurs. The SD of previous 24 hours must also not exceed 5% RH. California Air Resources Board (CARB) laboratory uses more stringent environmental conditions than those required by 40 CFR Part 50, Appendix J, to facilitate $PM_{2.5}$ program joint use of the balance room.	conditions remain unacceptable for an extended period, relocate weighing operations to the backup Balance Room. Notify the lab supervisor. Location and balance used for each weigh session is electronically documented in LIMS.

10.2 Temp and RH Sensor Probes

The Balance Room must have two Temp and RH sensor probes, one designated as primary and the other as secondary. All sensor probes installed must be certified as calibrated NIST-traceable annually by an outside source. If existing sensor probes cannot be re-certified for calibration, the existing sensors must be replaced with sensor probes that have been certified as calibrated NIST-traceable within the previous 12-months or less. Documentation and NIST-traceability of both sensors is kept with the equipment. These records are peer-reviewed, manager-approved, and archived in the Balance Room records.

10.2.1. Quarterly Verification of Temp and RH Sensor Probes

On the first weighing day of each calendar quarter, download the simultaneous readings from both the primary and secondary sensor probes. Randomly select a comparison range of ten (10) consecutive 2-minute reading intervals. In a spreadsheet program, tabulate the readings and calculate the separate averages of the Primary and Secondary sensor readings. Calculate the Average Differences and verify acceptability of the results.

Table 10.2.1 Quarterly Verification of Temp and RH Sensor Probes

Criteria	Corrective Action
The average Temp difference between the sensors must be within ± 2°C. The average RH difference must be within ± 2%.	No weighing shall occur if the sensors do not fall within the criteria. Re-perform the check. Contact the sensor probe manufacturer or replace the probes. Document in the Balance Room laboratory notebook.

10.3 Mass Reference Standards, hereafter referred to as weights, should be ASTM Class 1 category, non-corroding, and NIST-traceable. Documentation of NIST-traceability must be kept in Balance Room where the weights reside. Two sets of weights are needed. One set is designated the primary weights and one set is designated the workings weights. The weights used and certified for PM_{10} mass procedures have a target value of 1 g, 3 g, and 5g.

10.3.1 The primary weights are used to verify the working weights. The primary weights are annually recertified by an outside source. The outside certification source must provide a NIST certificate for each certified weight.

10.3.2 The working weights are used during each weighing session for quality control of the ambient sample masses obtained. The control verifies the calibration of the balance at the start of the weighing session. The standard checks provide continuous checks of the balance during the weigh session. Working weights remain in the Balance Room.

10.4. Standard Checks

10.4.1. The two standard checks (3 g and 5 g) are sequentially weighed after the Control at the beginning of each weigh session and after the final replicate at the end of each weigh session. In addition, the 3 g and 5 g standard checks are alternately weighed after each replicate sample to verify the calibration is maintained during each weigh session.

Table 10.4.1 Standard Checks Criteria

Criteria	Corrective Action
Standard check must be ± 0.0005 g from its certified mass.	If a standard check is greater than ± 0.0005 g from its certified mass, re-weigh the control, QC, and all samples weighed since the last valid standard check. If the standard check is still out of range, the weigh session is not valid. Restart the weigh session. If the standard check is again out of range, no weighing is performed. Alert management and contact the manufacturer to adjust or replace the faulty weight. Document the incident on the COC.

10.5 Analytical Balance

10.5.1 Verification of Analytical Balance Calibration - Daily

The balance calibration must be verified by a control once a day prior to any weighing sessions. The control combines the 1 g and 3 g standards for a total mass of 4 g.

Table 10.5.1 Verification of Analytical Balance Calibration - Daily

Criteria	Corrective Action
Control must be ± 0.0005 g from the sum of certified masses.	If the weights are out of range, repeat anti-static and dusting of the balance pan, tare the balance, and reweigh the weight. If rechecked weights are not valid, no weighing shall occur until the error is corrected. Alert management, reevaluate the balance level and/or stability, and verify that the antistatic strips are up-to-date. Contact the vendor to recalibrate the balance or replace the faulty weight if the problem continues.

10.5.2 Quarterly Verification of Analytical Balance Calibration

On the first weighing day of each calendar quarter, record into a lab notebook the results from weighing both the Working and Primary weights (1 g, 3 g, and 5 g) five times each. Transfer the results into a spreadsheet program and proceed to calculate the average of observed masses for each weight. Calculate the difference for each observed Working and Primary average with the expected mass for each weight.

Table 10.5.2 Quarterly Verification of Analytical Balance Calibration

Criteria	Corrective Action
The difference between the expected mass and the average of observed masses for each Primary and Working weight must be within ± 0.0005 g.	If the difference is out of range, reweigh the failed weight. If rechecked weight still fails, no weighing will occur until the error is corrected. Verify the balance was calibrated and certified by an outside source annually on or before the anniversary date. Reevaluate the balance level and/or stability and verify that the antistatic strips are up-to-date. Use another balance to verify the weight. Complete the daily calibration of the balance and the quarterly calibration verification again. Contact the manufacturer to recalibrate the balance or replace the faulty weight if the problem continues. Alert management.

10.5.3 Annual Calibration of Analytical Balance

Balance must be calibrated and certified by an outside source annually on or before the anniversary date.

Table 10.5.3 Annual Calibration of Analytical Balance

Criteria	Corrective Action
Balance must be calibrated and certified annually by an outside source. The calibration must be traceable to NIST.	If the balance is determined to be out of calibration, it must be recalibrated according to the manufacturer's instructions and recertified by an outside source. No weighing will occur.

10.6 Replicates

10.6.1 Replicates must be weighed with every set of filters. The replicate mass rate must be greater than or equal to ten percent (≥10 %). A replicate weighing of the first sample of a set must be weighed as the tenth sample position of a set. If a set comprises nine or less PM_{10} samples, a replicate of the first sample must still be weighed.

Table 10.6.1 Replicates

Criteria	Corrective Action
For pre-weights, the original and replicate mass values must be within ± 0.0028 g.	If outside criteria, check the balance and antistatic strips. Samples in the associated batch (i.e., the preceding set of nine (9) samples) must be reanalyzed to be valid. Check the Balance Room conditions. Verify the balance with control and standard weights. If the replicate is out again, re-weigh random samples to troubleshoot possible cause, or alert management.
For post-weights, the original and replicate mass values must be within ± 0.0050 g.	If outside criteria, check the balance and antistatic strips. Samples in the associated batch (i.e., the preceding set of nine (9) samples) must be reanalyzed to be valid. Check the Balance Room conditions. Verify the balance with control and standard weights. If the replicate is out again, re-weigh random samples to troubleshoot possible cause, or alert management.

10.7 Quartz microfiber filters must have a mass between 3.7 g to 4.7 g. Any quartz fiber filter pre-weighing outside this acceptable range must be rejected and removed from the set of useable PM_{10} filters.

10.8 Field Blanks

10.8.1. Field blanks are unexposed quartz filters that are placed on a PM_{10} sample with no air flow passing through the filter.

Table 10.8.1. Field Blanks

Criteria	Corrective Action
The difference between the pre- and post- weight should be within ± 5 mg.	Re-weigh the sample to verify the weight. Check QC, balance, and room conditions. Notify the site manager, site operator, and laboratory manager if the field blank concentration exceeds ± 5 mg. Document this difference on the COC. No actionable criteria for samples exceeding these limits.

11. Data Management

Data management involves PM_{10} samples logged into LIMS, transfer and review of PM_{10} pre- and post- weight data to LIMS, documentation of unusual occurrences and their resolutions, creation of data packages (monthly, amendments, and special projects) for peer review and management approval, storage of Balance Room conditions to the shared drive, and archiving of PM_{10} filters and their COCs. Program and maintenance logbooks are to be always kept with the instrumentation.

11.1 After PM_{10} samples are weighed and transferred to LIMS they must be peer reviewed to ensure accuracy. The peer reviewer must check the daily calibration and all QC to ensure it was performed within criteria, the PM_{10} pre-/ post- weight to LIMS to make sure all records show “complete” under LIMS status, and PM_{10} pre-/ post- weight summaries to certify all weighed mass values match those on the COC.

11.2 PM_{10} sample data packages undergo a multi-level data validation process. This process includes analyst review, peer review, and laboratory management review and approval in line with NLB’s Laboratory Quality Control Manual. Data packages created by analyst must consist of the following:

11.2.1. Documentation of the method and program name, description of standards (re-certification and expiration dates), identifiers of standards used (i.e., serial number), criteria limits, and comments for special projects.

11.2.2. Summary of the data generated for a sample date period. This includes blank data.

11.2.3. Summary of QC data, calibration checks, and any additional QC documentation connected to the data of interest.

11.2.4. Documentation of any unusual occurrences and how they were resolved. This includes lab action emails to site operators, analyst notes, as well as a record of involvement by management.

11.3. All PM_{10} mass exceedances of the National Ambient Air Quality Standard (NAAQS) should be reported to laboratory management. PM_{10} Mass data clients should be notified via email if a PM_{10} Mass result exceeds the standard. This information should also be documented in the data package.

11.4. A continuous digital data from the secondary Temp and RH sensor probes is used to document and store environmental conditions of the Balance Room.

11.5. PM_{10} filter samples, their respective COC forms, and any program related reports are archived for 5 years plus the current year according to our records retention policy. Samples and reports that exceed this time period are no longer required to be stored by the laboratory.

11.6. Laboratory management must approve destruction of any archival data older than five years plus the current year.

12. Calculations

12.1 PM_{10} Mass Calculations:

PM_{10} mass data reported to the Federal database (i.e., Air Quality System) must be reported at standard conditions in standard cubic feet per minute (SCFM). SCFM is the flowrate referenced to a Temp of 25°C and a pressure of 760 millimeters mercury (mm Hg). This is considered standard Temp and pressure (STP).

PM_{10} mass data is also reported in local conditions in actual cubic feet per minute (CFM). Local Temp and pressure (LTP) conditions are used to determine PM_{10} mass data in local conditions.

Volumetric flow controller (VFC) samplers calculate the average flow rate over the sampling period corrected to U.S. EPA reference conditions, indicated as Q_{std}. However, mass flow controller (MFC) samplers do not require Q_{std} calculations. When the sampler's flow indicator is calibrated in actual volumetric units, indicated as Q_a, Q_{std} is calculated as:

Equation 1:

$$Q_{std} = Q_a \left(\frac{P_{av}}{T_{av}}\right)\left(\frac{T_{std}}{P_{std}}\right)$$

Equation 2:

$$Q_a = \left[45.379\left(\frac{P_o}{P_{av}}\right) - 2.243\right] + [(T_{av} - 25)(0.059)]$$

Where,

Q_{std} = average flow rate at U.S. EPA reference standard conditions, expressed as standard cubic meters per minute (std m^3/min);

Q_a = average flow rate at ambient conditions, local m^3/min;

P_{av} = average barometric pressure during the sampling period or average barometric pressure for the sampling site, in kilopascal (kPa) or mm Hg;

T_{av} = average ambient Temp during the sampling period or seasonal average ambient Temp for the sampling site, in Kelvin (K);

T_{std} = standard Temp, defined as 298 K;

P_{std} = standard pressure, defined as 101.3 kPa (or 760 mm Hg);

$\frac{P_o}{P_{av}}$ = pressure ratio, $\left(1 - \frac{P_f}{P_{av}}\right)$;

P_f = differential pressure across filter (mm Hg);

Q_a equation is referenced as Volumetric Flow in CFM from Air Quality Surveillance Branch (AQSB) SOP 408 for local conditions.

Calculate the total volume of air in both standard and local conditions sampled as:

Equation 3:

$$V_{std} = Q_{std}(t)$$

Equation 4:

$$V_a = Q_a(t)$$

Where,

V_{std} = total air sampled in standard volume units, standard cubic meters (m^3);

V_a = total air sampled in ambient conditions volume units, local m^3;

t = sampling time, minutes.

Calculate the PM_{10} concentration in both standard and local conditions as:

Equation 5:

$$PM_{10\,std} = \left[\frac{(W_f - W_i)(10^6)}{V_{std}}\right]$$

Equation 6:

$$PM_{10\,a} = \left[\frac{(W_f - W_i)(10^6)}{V_a}\right]$$

Where,

$PM_{10\,std}$ = mass concentration of PM_{10}, micrograms per standard m^3;

$PM_{10\,a}$ = mass concentration of PM_{10}, micrograms per local m^3;

W_f = final weight of filter collecting PM_{10} particles, g;

W_i = initial weight of filter collecting PM_{10} particles, g.

12.2 Balance Room Environmental Conditions Calculations.

12.2.1. Average and SD of Temp and RH

Equation 7:

$$Average = \frac{1}{n}\sum_{i=1}^{n} a_i = \frac{1}{n}(a_1 + a_2 \ldots a_n)$$

Where,

n = sample size,

a_i = Individual observed values,

Equation 8:

$$Standard\ Deviation = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2}$$

Where,

n = sample size;

x_i = observed values;

$\bar{x}$ = mean value of observations.

12.3 Quarterly Calibration Verification Calculations:

12.3.1. Average Temp and RH Difference

The Temp and RH readings from both primary and secondary sensor probes are compared to each other 10 times using an interval of 2 minutes between each parameter. The formula to compare these sensors is:

Equation 9:

$$Average\ Difference = (\frac{1}{n}\sum_{i=1}^{n} SS_i) - (\frac{1}{n}\sum_{i=1}^{n} PS_i)$$

Where,

n = sample size;

SS_i = secondary sensor Temp or RH observed values;

PS_i = primary sensor Temp or RH observed values.

12.3.2. Average Analytical Weight Difference from Certified Mass

The average of five readings from each of primary and working weights (1 g, 3 g, and 5 g) is compared to its certified mass. The formula to compare these weights is:

Equation 10:

$$Average\ Difference\ from\ expected\ mass = (\frac{1}{n}\sum_{i=1}^{n} W_i) - certified\ mass\ value$$

Where,

n = sample size (5);

W = analytical weight observed values.

13. References

13.1 Code of Federal Regulations, Title 40, Chapter 1, Subchapter C, Part 50, Appendix J, "Reference Method for the Determination of Particulate Matter as PM_{10} in the Atmosphere."

13.2 Code of Federal Regulations, Title 40, Chapter 1, Subchapter C, Part 50, Appendix L, "Reference Method for the Determination of Fine Particulate Matter as $PM_{2.5}$ in the Atmosphere."

13.3 Code of Federal Regulations, Title 40, Chapter 1, Subchapter C, Part 50, § 50.6.

13.4 Code of Federal Regulations, Title 40, Chapter 1, Subchapter C, Part 58, Subpart B, "Monitoring Network."

13.5 California Health and Safety Code, Division 1, Part 1, Chapter 2, Article 9, § 425.

13.6 California Health and Safety Code, Division 26, Part 2, Chapter 3, § 39606.

13.7 Section 2.11, "QA Handbook Reference Method for the Determination of PM10 (High Volume PM10 Sampler) at Standard Temperature and Pressure." U.S. Environmental Protection Agency, January 1990. https://www.epa.gov/sites/default/files/2021-04/documents/m211_ocr.pdf

13.8 Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II. U.S. Environmental Protection Agency, January 2017. https://www.epa.gov/sites/default/files/2020-10/documents/final_handbook_document_1_17.pdf

13.9 Quality Assurance Handbook Volume II, Appendix D Measurement Quality Objectives and Validation Templates. Revision No. 1, U.S. Environmental

Protection Agency, March 2017. https://www3.epa.gov/ttn/amtic/files/ambient/pm25/qa/APP_D%20validation%20template%20version%2003_2017_for%20AMTIC%20Rev_1.pdf

13.10 Compendium Method IO-2.1, “Sampling of Ambient Air for Total Suspended Particulate Matter (SPM) and PM10 Using High Volume (HV) Sampler.” EPA/625/R-96/010a. Center for Environmental Research Information, Office of Research and Development, U.S. Environmental Protection Agency, June 1999. https://www.epa.gov/sites/default/files/2019-11/documents/mthd-2-1.pdf

13.11 Compendium Method IO-3.1, “Selection, Preparation, and Extraction of Filter Material.” EPA/625/R-96/010a. Center for Environmental Research Information of Office of Research and Development, U.S. Environmental Protection Agency, June 1999. https://www.epa.gov/sites/default/files/2015-07/documents/epa-io-3.1.pdf

13.12 U.S. EPA PM Sampling Schedule Calendar. https://www.epa.gov/amtic/sampling-schedule-calendar

13.13 CARB NLB Laboratory Quality Control Manual, Revision 5.0, December 7, 2021.

13.14 CARB, “Chemical Hygiene Plan for Northern Laboratory Branch 1927 13th Street, 1900 14th Street,” November 2021 or current.

13.15 Excellence Plus Analytical Balances Operating Instructions, XP Models – Part 1. https://www.mt.com/dam/P5/labtec/02_Analytical_Balances/03_XP/03_Documentations/03_Operating_Instructions/OI_XP_Analytical_Part_1_EN.pdf

13.16 CARB AQSB SOP 408, “Standard Operating Procedures for Tisch Environmental Model TE-6070V Size Selective Inlet PM10 Sampler with Volumetric Flow Controller”, Third Edition, March 2020.

13.17 CARB NLB MLD076 Rev. 1.0, Standard Operating Procedure for Preparation of Northern Laboratory Branch's Standard Operating Procedures, December 30, 2021.

13.18 CARB Quality Assurance Program Plan for Particulate Matter Pollutant Air Monitoring Program, February 2020.

14. Revision History

Table 14. Revision History

SOP/Addendum Identification	Approval Date	Description of Change
NSL016 Revision 0.0	May 1, 1986	New method.
MLD016 Revision 1.2 SOP for the Mass Analysis and Subsequent Extraction of SSI-Sampled PM10 from Exposed Quartz Microfiber Filters	September 15, 1992	Effective March 21, 1991. Combines methods NSL Revision 0.0 and MLD030 (approved April 1, 1990; SOP for the Extraction of SSI-Sampled PM10 from Exposed, Post-weighed, Quartz-Fiber Filters).
MLD016 Revision 5.0	January 18, 2002	Unknown.
MLD016 Revision 6.0	January 17, 2008	Effective November 13, 2007. Unknown.
Addendum A04 MLD016	August 12, 2015	Effective July 1, 2015, filter conditioning environment now reflects conditions more stringent than 40 CFR Part 50, Appendix J for PM10 mass analysis: - Temp range is 15°C to 30°C with control ± 3°C - RH range is 30% to 40% with control ± 5%RH. This alignment accommodates requirements for PM2.5 to use Balance Room as a back-up.
MLD016, Revision 7.0	April 30, 2018	1. February 19, 2012: Filter conditioning environment now reflects 40 CFR Part 50, Appendix J for PM10 mass analysis: - Temp range is 15°C to 30°C with control ± 3°C (from Temp at 21.3°C ± 3°C (18.3-24.3°C)); - RH range is 20% to 45% with control ± 5% RH (from and RH at 37% ± 5% RH (32-42 % RH)). 2. June 17, 2013: Removed subsequent extraction of PM10 samples and Total Suspended Particulates (TSP) sampling procedures.

SOP/Addendum Identification	Approval Date	Description of Change
		3. July 1, 2013: A continuous digital data recorder (Omega sensor) is used to document and store environmental conditions of the Balance Room. The environmental conditions of the room are stored on the Omega unit and backed up to the shared drive weekly. Data recorder manual is stored in the PM10 references binder which is kept in the Balance Room (from Honeywell recorder.) 4. July 1, 2013: One field blank per quarter for each site implemented per U.S. EPA Technical Systems Audit recommendations. 5. August 22, 2013: Per U.S. EPA recommendations, filters should not be used if they are more than three years old. A first in/ first out approach should be used by all inventory holders so that filters do not age past this point. 6. August 23, 2013: Mettler Toledo XP204 balances were purchased and installed in 2013. Comparison study has been done with existing Sartorius A200S, and approved by the Quality Management Branch (QMB). 7. November 20, 2013: A make-up sample must be collected prior to the next scheduled sampling day. Reference AQSB SOP 408 for more information. 8. May 18, 2014: Updated COC form including date and initials of chemist for post-weighing sample. 9. April 1, 2015: Date and time filter equilibration started in the controlled environmental Balance Room is written on the COC for every filter received. This is annotated on the samples received by the laboratory line. If for any reason samples are removed from environmentally conditioned roomed it must be documented on the COC along with the new equilibration start time. 10. April 6, 2015: Ecotech samplers use a modified COC because the sampler does not use a Dickson chart. It provides a calculated LTP volume and standard corrected volume. Site operators are now required to send in a printout of the Ecotech data file in place of the Dickson chart to verify sampling duration.
MLD016, Revision 7.1	September 27, 2022	Two sets of analytical weights, instead of three, are used as primary and working weights.

SOP/Addendum Identification	Approval Date	Description of Change
		Procedural change: LIMS ID is assigned to each filter before pre-weighing session. The accompanying COC is printed after pre-weighing session, where LIMS ID, filter number, pre-weight filter conditioning start date and time, pre-weight Temp and RH, and pre-weight mass for each filter are automatically recorded on the COC. Internal calibration is not required and removed from the procedure. Removed field make-up requirements. Updated SOP to align with MLD076 Revision 1.0 (SOP Preparation of NLB's SOPs).

APPENDIX A

1. REJECTION CRITERIA FOR PRE-SAMPLED PM10 FILTERS (UNUSABLE)

Observation	Definition
Pinhole	A small hole that can be identified by examining both the front and back of the filter. A filter with such a defect is considered a reject filter and is unusable.
Dense Spot	Viewed from the filter back, this appears as a dark area (approximately 1/8"- 1/4" in diameter) without sharply defined edges. Viewed from the front, an accumulation of filter fibers can be seen. Any filter which contains more than one dense spot or one spot larger than 1/4" shall be considered a reject.
Dark Spot (2 or more)	These spots are distinguished from the dense spots in that such dark spots resemble a fly speck. Any filter containing two or more such dark spots will be considered a reject.
Loose Fiber on Filter Back (Not Removeable)	This appears as if a rough object had been moved across the filter back and loosened the filter base. If the number of fibers is small and can be brushed off, the defective filter can be used. If the fibers are too large or too numerous to remove, the filter will be considered a reject.
Quartz Fiber (Detached)	When viewed from the back, this defect resembles a thin spot. The shape can be circular or oval. When rubbed, the quartz may become detached. No evidence of this defect can be seen from the front. If it becomes detached and creates a pinhole, the filter is considered a reject.
Coloration	Yellow, red, or other colored spots. A filter with such coloration is considered a reject.

2. ACCEPTABLE DEFECTS FOR PRE-SAMPLED PM10 FILTERS (USABLE)

Observation	Definition
Line	Occasionally a fine line is created by the manufacturing screen across the filter. A filter with such a defect is considered usable.
Dense Spot	Viewed from the filter back, this appears as a dark area (approximately 1/8"- 1/4" in diameter) without sharply defined edges. Viewed from the front, an accumulation of filter fibers can be seen. If there is only one dense spot per filter, and the area covered is less than 1/4" in diameter, the filter will be considered a defective filter.
Thin Spot	A small area viewed on the filter that appears to be weak. More light can be seen through this area than through the surrounding area. There can be several spots per filter. A filter with such a defect is considered defective.
Dark Spot (1 or less)	These spots are distinguished from the dense spots in that such dark spots resemble a fly speck. Their presence results in a defective filter. Any filter containing one dark spot is considered a defective filter.
Quartz Fiber (Not Detached)	When viewed from the back, this defect resembles a thin spot. The shape can be circular or oval. When rubbed, the quartz may become detached. No evidence of this defect can be seen from the front. If it becomes detached and creates a pinhole, the filter is a reject. Otherwise, it is considered defective.
Loose Fiber on Filter Back (Re-moveable)	This appears as if a rough object had been moved across the filter back and loosened the filter base. If the number of fibers is small and can be brushed off, the defective filter can be used.
Non-uniformity	Any obvious visible non-uniformity of the appearance of the filter when viewed over a light table and might indicate gradations in porosity across the face of the filter is considered a defect.
Other	A filter with any imperfection not described above, such as frayed edges or indentations or the results of other poor workmanship, may be considered defective.

3. VALIDATION CRITERIA FOR POST-SAMPLED PM10 FILTERS

Observation	Definition
Filter Contamination	Filters which are dropped or become contaminated by any foreign matter (e.g., dirt, finger marks, ink, liquids, etc.) are invalid.
Damaged or Torn Filters	Filters with tears or pinholes which occurred before or during sampling are invalid. Note: a filter that has a hole or is torn before or during sampling will show evidence of particulate matter collection on both sides of the filter caused by the lack of filter integrity. If a damaged or torn filter is received by the lab with all pieces included that does not show evidence of tears or pinholes prior to sampling, and is not invalidated by the site operator, the sample is valid.
Dickson Recorder Chart (Note: Ecotech samples do not contain a Dickson chart and are exempt from this validation criteria)	A complete 24-hour Dickson recorder chart, documenting the flow rate through the sampler for 24 hours, must be submitted to the laboratory with each filter sample. Filter samples without a complete Dickson recorder chart record are invalid. Note: In cases of inking problems where the trace is not complete, if the operator validates the sampler operated properly in the comments section of the report form, the sample will be considered valid.
Filter Leakage	Filters that show signs of air leakage due to a worn or improperly seated gasket (e.g., the increased fuzziness of the sample outline) are invalid. Contact the site operator to replace the gasket.
Date Samples are Received in the Laboratory	Samples should be weighed as soon as possible to minimize volatile particle mass loss. Because of this, U.S. samples received in the laboratory 30 days or more after they are sampled are invalid.
Incorrect Filter Number and/or LIMS ID	If the filter number does not match the COC's filter number, the sample is invalid. If the LIMS ID on the manila folder does not match the COC's LIMS ID, the sample is invalid.
Filters Not Received in Glassine Envelopes	If the filter is not received by the lab inside a glassine envelope, folded in half lengthwise, with the sampled particulates contained inside the folded filter, then the sample is invalid.

4. EXAMPLE OF LIMSLINK WORKSHEET

PM10 HiVol Post-Weight to LIMS		Temperature (C):	21.7		Start Balance Room Conditioning		Worksheet Created on 6/23/2022 10:19:06 AM			Temp in LIMS:	21.7	Temp/RH LIMS Status		Chemist: Betsy R
Adjust QCB Positions if Samples Skipped		Rel. Humidity (%):	32.8		06/22/2022 12:45		QC Batch Name: QCB220627001			RH in LIMS:	32.8	Completed		

	QCB Position	QC Name/ LIMS ID	Type	Filter # or QC LIMS ID	Pre-Wt (grams)	Post-Wt (grams)	Post-Wt Date	Post-Wt Time	Post-Wt Bal/Room	Repl/FBlk Diff (g)	Notify if QC Out	STP/LTP Mass (ug/m3)	Notify to Re-Weigh	LIMS Status	NO for QCB	New QCB Created	Ready for LIMS	Sent to LIMS	Mass in LIMS	Temp/RH Ready for LIMS	Temp/RH Sent to LIMS	Temp/RH in LIMS
1	n	ctl3	QC	S220627001		3	06/23/2022	13:07	XP204-13th					Completed		Y	N	Y	Y	N	Y	Y
2	2	ctl5	QC	S220627002		4.9999	06/23/2022	13:08	XP204-13th					Completed		Y	N	Y	Y	N	N	N
3	3	S210726162	Sample	2000994	4.4652	4.5058	06/23/2022	13:13	XP204-13th			24.9 / 26		Completed		Y	N	Y	Y	N	N	N
4	4	S211025074	Sample	2001349	4.4902	4.5185	06/23/2022	13:14	XP204-13th			17.2 / 17.9		Completed		Y	N	Y	Y	N	N	N
5	5	S211026003	Sample	2004111	4.4731	4.4798	06/23/2022	13:17	XP204-13th			4.1 / 4.1		Completed		Y	N	Y	Y	N	N	N
6	6	S211026004	Sample	2004112	4.4675	4.4782	06/23/2022	13:19	XP204-13th			6.4 / 6.6		Completed		Y	N	Y	Y	N	N	N
7	7	S211026005	Sample-FB	2004115	4.497	4.494	06/23/2022	13:22	XP204-13th	-0.003				Completed		Y	N	Y	Y	N	N	N
8	8	S211026006	Sample	2004116	4.5039	4.5144	06/23/2022	13:24	XP204-13th			6.2 / 6.5		Completed		Y	N	Y	Y	N	N	N
9	9	S211026027	Sample	2004143	4.4279	4.4464	06/23/2022	13:25	XP204-13th			11 / 11.4		Completed		Y	N	Y	Y	N	N	N
10	10	S220120027	Sample	2004101	4.4876	4.5069	06/23/2022	13:27	XP204-13th			11.9 / 11.6		Completed		Y	N	Y	Y	N	N	N
11	11	S220120028	Sample	2004102	4.5076	4.5206	06/23/2022	13:28	XP204-13th			8.1 / 7.9		Completed		Y	N	Y	Y	N	N	N
12	12	S210726162-REP	QC	S220627003		4.5056	06/23/2022	13:31	XP204-13th	0.0002				Completed		Y	N	Y	Y	N	N	N
13	13	ctl3	QC	S220627004		3	06/23/2022	13:32	XP204-13th					Completed		Y	N	Y	Y	N	N	N
14	14	S220120029	Sample	2004104	4.517	4.5389	06/23/2022	13:33	XP204-13th			13.4 / 13		Completed		Y	N	Y	Y	N	N	N
15	15	S220127057	Sample	2001388	4.4812	4.49	06/23/2022	13:35	XP204-13th			5.4 / 5.3		Completed		Y	N	Y	Y	N	N	N
16	16	S220120029-REP	QC	S220627005		4.539	06/23/2022	13:40	XP204-13th	0.0001				Completed		Y	N	Y	Y	N	N	N
17	17	ctl3	QC	S220627006		2.9999	06/23/2022	13:41	XP204-13th					Completed		Y	N	Y	Y	N	N	N
18	18	ctl5	QC	S220627007		5	06/23/2022	13:42	XP204-13th					Completed		Y	N	Y	Y	N	N	N

5. EXAMPLE OF CHAIN OF CUSTODY

California Air Resources Board
MFC Sampler

S210322135

Site Name:
AQS Site Number:
Agency:
Field Technician:
Phone or Email:

Filter ID: 9534197

County	Site	Agency
	1	1

Instrument Number

Sampling Conditions ☑

Local Condition Codes: (Enter Appropriate Code in the Box at Left)

☑ - No Unusual Conditions
A - High Winds
E - Forest Fire
F - Structural Fire
J - Construction Nearby
K - Farming Nearby
L - Highway Construction
N - Sanding/Salting Streets
P - Roofing Operations
Q - Prescribed Burn
X - Rain
Y - Snow
Z - Other (Explain in Field Comments)

SAMPLE COLLECTION DATA

	Date			Time		Elapsed Time Meter (Min.)
	Year	Month	Day	Hours	Min.	
Finish	21	09	26	00	00	64482.6
Start	21	09	25	00	00	63042.6
Sampling						1440.0

Indicated Flow Rate

02:00	08:00	14:00	20:00
37.1	37.0	37.7	37.8

Ta 21.6 °C Pa 756 mmHg

Date of Last Calibration: Year 21 Month 08 Day 24

Slope 1.00

Intercept 3.35

Average Std Flow (SCFM) 40.8

Average Ind. Flow Rate 37.4

Type of Sample: [✓] Regular [ ] Collocated [ ] Make up [ ] Field Blank

MUST BE COMPLETED BY SAMPLER OPERATOR:

☑ Inspection of sampler and filter indicates that sample collected is in compliance with quality control standards for sampling.
☑ Filter and Dickson recorder chart enclosed.
☐ Sample does not meet quality control standards for sampling and should be invalidated.

Make up sample scheduled for:

Reasons for invalidation:

☐ Filter Contaminated or Damaged
☐ Power Outage
☐ Other (Explain in Field Comments)
☐ High/Low Flowrate
☐ Missing All or Partial Data Output
☐ Erratic Flowrate
☐ Timer Problem

Field Comments:

SAMPLE TRACKING

Action	Transfer Method (Check One)		Name & Initials	Date/Time
	Carrier	Person		
Released by Field	✓		MB	9-29-21 08:30
Received by Lab			[illegible]	10-01-21 1330
Inspected by Lab			[illegible]	Cond. starts

FOR LABORATORY USE ONLY

Sample Conditions Upon Received: ☑ VALID ☐ INVALID

Balance Room Conditions

	Pre-Weight	Post-Weight
Condition Start	03/23/2021 08:05	10-11-21 820
Temperature (C)	21.1	
RH (%)	37.3	

	Pre-Weight	Post-Weight
Analyst	~~BBrose~~ TN	
Date	03/24/2021 09:51	
Weight	4.0836	
Duplicate Weight (If Applicable)		

Lab Comments: