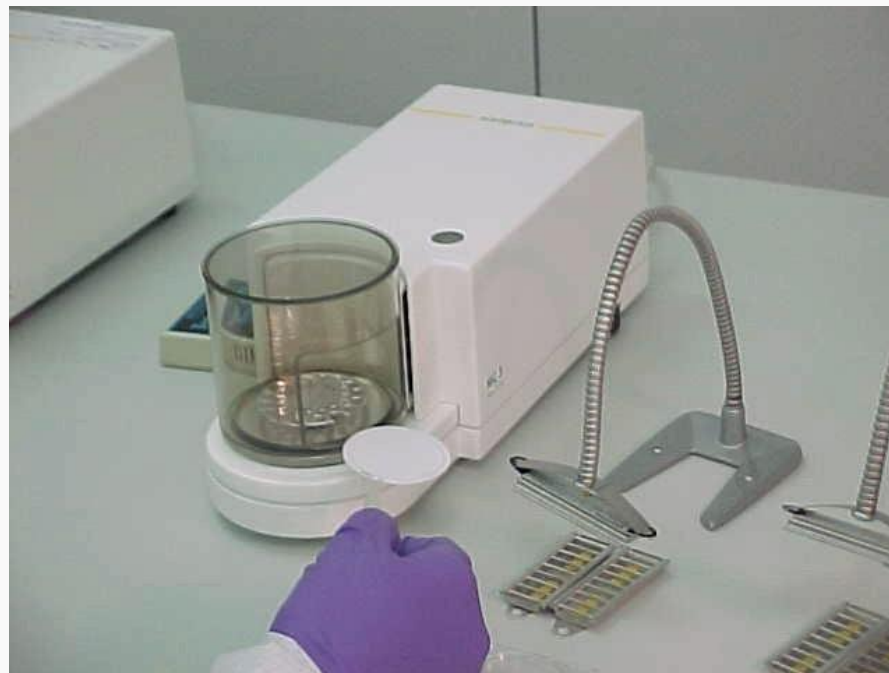


Welcome to the NAAMC!

PM_{2.5} Gravimetric Lab Training



Greg Noah, US EPA OAQPS
Stephanie McCarthy, US EPA Region 4

2016 National Ambient Air Monitoring Conference



NAAQS Designations

- **Primary Standards**
Protect Human **Health**
- **Secondary Standards**
Protect Human **Welfare** & **Vegetation**
- **Attainment**
Meets the standard
- **Non-Attainment**
Violates the standard

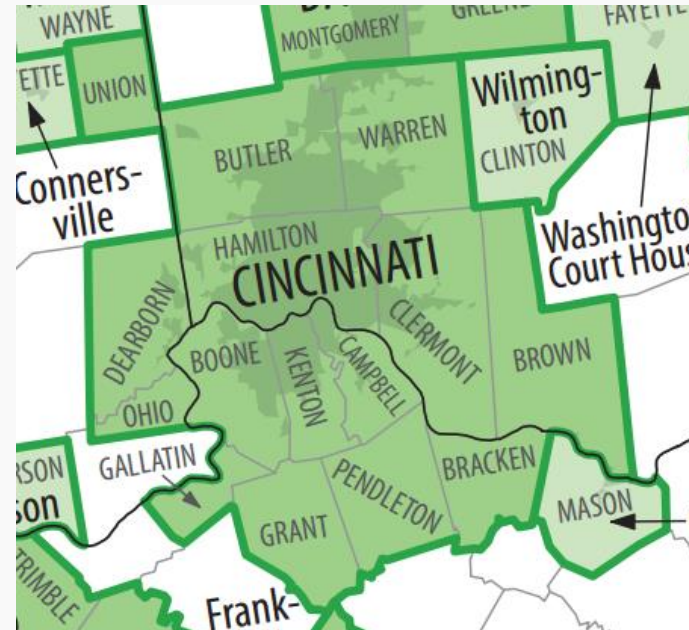
What happens to a county when it violates a NAAQS standard?

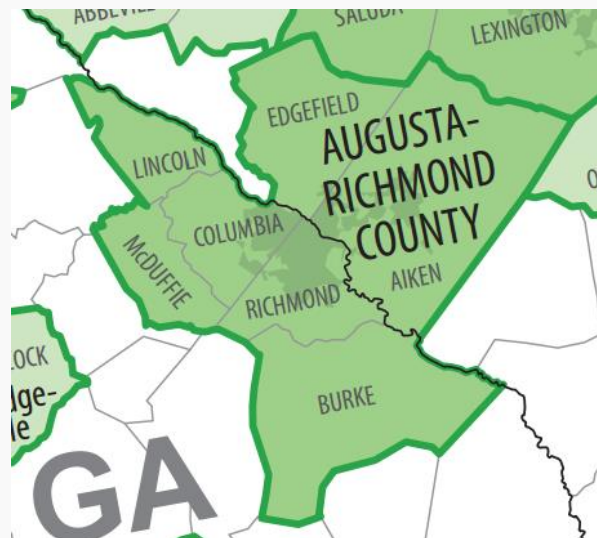
- Economic development slows
- Emission testing programs
- Loss of federal highway dollars

What's the Big Picture?



When a county violates a NAAQS, it will impact neighboring counties, and possibly neighboring states





MSA Boundaries

- Non-attainment restrictions will impact everyone within the designated non-attainment boundaries.
- As air monitoring agencies, you must be diligent & ensure the highest quality, **accurate** data possible, to promote fairness in the designations process, as well as protect the citizens of the States.



The Weighing Lab is the Keystone of Your Agency's $PM_{2.5}$ Network

“the final piece placed during construction and locks all the stones into position, allowing the arch to bear weight”





The Difference Between “Regulation” and “Guidance”

Regulations

- Are issued by various federal government departments and agencies to carry out the intent of legislation enacted by Congress
- A rule of order having the force of law
- **CFR**

Guidance

- Recommendations provide non-binding advice
- Not legally enforceable
- Federal guidance and technical reports are intended as basic guidelines
- **Method 2.12 & the QA Handbook**





8.0 *Filter Weighing.* See reference 2 in section 13.0 of this appendix, for additional, more detailed guidance.

8.1 *Analytical balance.* The analytical balance used to weigh filters must be suitable for weighing the type and size of filters specified, under section 6.0 of this appendix, and have a readability of $\pm 1 \mu\text{g}$. The balance shall be calibrated as specified by the manufacturer at installation and recalibrated immediately prior to each weighing session. See reference 2 in section 13.0 of this appendix for additional guidance.

8.2 *Filter conditioning.* All sample filters used shall be conditioned immediately before both the pre- and post-sampling weighings as specified below. See reference 2 in section 13.0 of this appendix for additional guidance.

8.2.1 *Mean temperature.* 20 - 25 °C.

8.2.2 *Temperature control.* $\pm 2 \text{ }^\circ\text{C}$ over 24 hours.

8.2.3 *Mean humidity.* Generally, 30-40 percent relative humidity; however, where it can be shown that the mean ambient relative humidity during sampling is less than 30 percent, conditioning is permissible at a mean relative humidity within ± 5 relative humidity percent of the mean ambient relative humidity during sampling, but not less than 20 percent.

8.2.4 *Humidity control.* ± 5 relative humidity percent over 24 hours.

8.2.5 *Conditioning time.* Not less than 24 hours.

8.3 *Weighing procedure.*

8.3.1 New filters should be placed in the conditioning environment immediately upon arrival and stored there until the pre-sampling weighing. See reference 2 in section 13.0 of this appendix for additional guidance.

8.3.2 The analytical balance shall be located in the same controlled environment in which the filters are conditioned. The filters shall be weighed immediately following the conditioning period without intermediate or transient exposure to other conditions or environments.

8.3.3 Filters must be conditioned at the same conditions (humidity within ± 5 relative humidity percent) before both the pre- and post-sampling weighings.

8.3.4 Both the pre- and post-sampling weighings should be carried out on the same analytical balance, using an effective technique to neutralize static charges on the filter, under reference 2 in section 13.0 of this appendix. If possible, both weighings should be carried out by the same analyst.

8.3.5 The pre-sampling (tare) weighing shall be within 30 days of the sampling period.

8.3.6 The post-sampling conditioning and weighing shall be completed within 240 hours (10 days) after the end of the sample period, unless the filter sample is maintained at temperatures below the average ambient temperature during sampling (or 4 °C or below for average sampling temperatures less than 4 °C) during the time between retrieval from the sampler and the start of the conditioning, in which case the period shall not exceed 30 days. Reference 2 in section 13.0 of this appendix has additional guidance on transport of cooled filters.

8.3.7 *Filter blanks.*

8.3.7.1 New field blank filters shall be weighed along with the pre-sampling (tare) weighing of each lot of $\text{PM}_{1.1}$ filters. These blank filters shall be transported to the sampling site, installed in the sampler, retrieved from the sampler without sampling, and reweighed as a quality control check.

8.3.7.2 New laboratory blank filters shall be weighed along with the pre-sampling (tare) weighing of each set of $\text{PM}_{1.1}$ filters. These laboratory blank filters should remain in the laboratory in protective containers during the field sampling and should be reweighed as a quality control check.

8.3.8 Additional guidance for proper filter weighing and related quality assurance activities is provided in reference 2 in section 13.0 of this appendix.

40 CFR Part 50, Appendix L, Section 8: Filter Weighing

The fine print seen
here is the **entire**
regulatory filter
weighing method!



40 CFR Part 50, Appendix L, Section 8.0

8.0 Filter Weighing. See reference 2 in section 13.0 of this appendix, for additional, more detailed guidance.

13.0 References

“2. Quality Assurance Guidance Document 2.12. Monitoring PM_{2.5} in Ambient Air Using Designated Reference or Class I Equivalent Methods. U.S. EPA, National Exposure Research Laboratory. Research Triangle Park, NC, November 1988 or later edition. Currently available at: <http://www.epa.gov/ttn/amtic/pmqaINF.html>.”





This morning's SOP for understanding weighing filters...

- Follow the logical flow of the procedure
- Focus on the 40 CFR Part 50, Appendix L requirements
- Add detail and fill in gaps using QAGD 2.12





Overview and Purpose of QAGD 2.12

*“This document reviews those formal requirements and provides clarification and supplemental information **in greater detail** than can be provided in the formal regulatory requirements.”*



Weighing Laboratory Preparation and Equipment





Prerequisites: Laboratory Personnel Qualifications

- All laboratory personnel should be **familiar** with **clean room environmental laboratory procedures & techniques**
- Those who operate the microbalance need to be very **conscientious** and **attentive to details** in order to report complete & high-quality PM_{2.5} data





Prerequisites: Training

Each individual should receive training appropriate to his or her duties in the PM_{2.5} monitoring program. Training should include:



Instructions on how to use all laboratory equipment and handle filters



Instruction on the agency's data management & recordkeeping systems



Overview of the field portion of the PM_{2.5} program



The NAAQS, the regulations, and Method 2.12!



Weighing Room

Climate-controlled room

- **Must** be capable of meeting 40 CFR 50, Appendix L, Section 8.2 requirements
- Determining compliance discussed later!





So what are the CFR requirements for climate control?

8.2.1 Mean temperature. 20 – 23° C.

8.2.2 Temperature control. $\pm 2^{\circ}$ C over 24 hours.

8.2.3 Mean humidity. Generally, 30-40 percent relative humidity; however, where it can be shown that the mean ambient relative humidity during sampling is less than 30 percent, conditioning is permissible at a mean relative humidity within ± 5 relative humidity percent of the mean ambient relative humidity during sampling, but not less than 20 percent.

8.2.4 Humidity control. ± 5 relative humidity percent over 24 hours.





Why does climate control matter?



Temperature control can affect humidity and balance operation



Humidity control can affect water vapor content on the filters



Reduces the effects of static on the filter weighing process



Provides consistent ranges for all weighing laboratories to enable data comparability



Weighing Room



Semi-clean room

- Cleaning regimes
 - Daily
 - Monthly
 - Yearly
- Positive pressure
- HEPA filters
- Limit activities to PM_{2.5}, if possible



8.1 Analytical balance. The analytical balance used to weigh filters must be suitable for weighing the type and size of filters specified, under section 6.0 of this appendix, and have a readability of $\pm 1 \mu\text{g}$. The balance shall be calibrated as specified by the manufacturer at installation and recalibrated immediately prior to each weighing session. See reference 2 in section 13.0 of this appendix for additional guidance.



Microbalance

Because of the greater sensitivity needed for measuring microgram-range weights or weight differences, microbalances are vulnerable to **relatively small changes** in physical environmental conditions, such as:

- ✓ **Vibration**
- ✓ **Electrostatic Charge Buildup**
- ✓ **Temperature**
- ✓ **Relative Humidity**





How do these environmental conditions impact the balance?

- **Vibration**
 - Instability in balance will cause faulty readings
- **Electrostatic Charge Buildup**
 - Causes instability
 - Static can slightly “levitate” a filter, causing an inaccurate weigh
- **Temperature**
 - Impacts volatiles (filter weight)
- **Relative Humidity**
 - Impacts water vapor (filter weight)





Microbalance Set-Up Guidelines



- Stationary
- Level
- Grounded
- Located away from drafts
- Located away from heating/cooling sources



Microbalance Set-Up Guidelines



Logging Systems

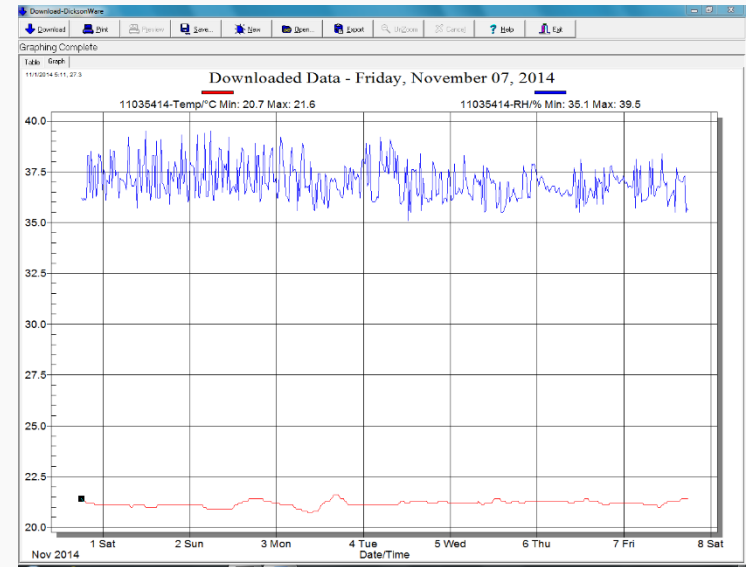


- RH and temperature conditions should be electronically measured and recorded on a **continuous** basis during filter conditioning
- **NIST-traceable** and recertified **annually** by vendor (i.e., every 365 days)
- **5-minute** values recorded (**minimum**)
- **Raw data**, as opposed to rolling averages
- Define programming in QAPP/SOP



Logging Systems

- Software packages available with many sensors
- Results displayed in tables, time-series graphs, or a combination of the two
- If no software, analyst will need to **manually** determine required statistics





Data Management Systems

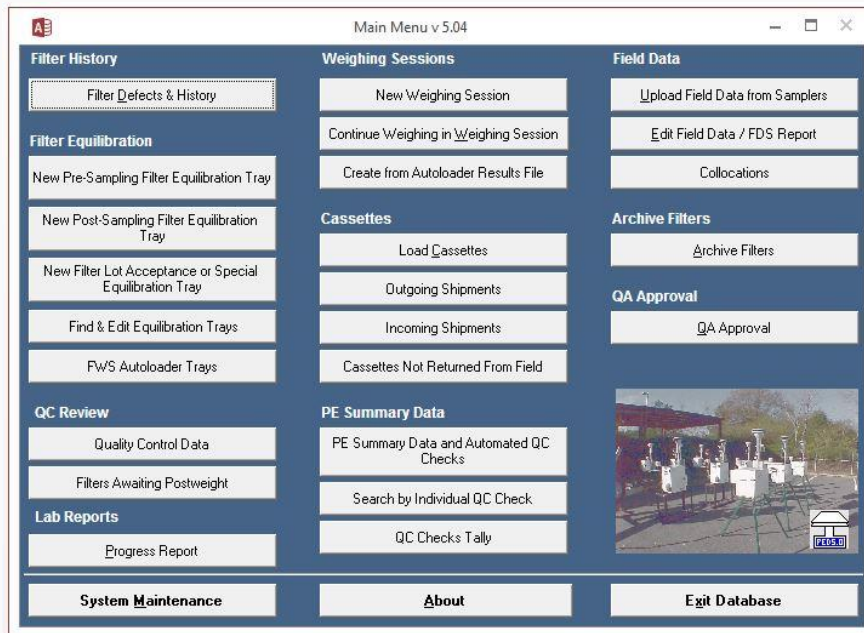
A screenshot of the "Weighing Session" software interface. The window title is "Weighing Session -". It contains several sections: "Equilibration Conditions" with fields for Average, Std. Dev, Maximum, and Minimum; "Autoloader (if applicable)" with fields for Tray Load File and Weighing Results File; "Remember Last Weigh Type" and "Fill Filter ID List from Tray"; "Weigh Type No." and "Weight (mg)" fields with an "Add to List" button; and three tables: "Filter Weighing Types", "Mass Reference Standards", and "Standard Weighing Types". The "Standard Weighing Types" table has 3 rows. At the bottom, there are buttons for "Add New Weighing Session" and "Exit Form", and a status bar showing "Record: 1 of 1" and "Filtered Search".

ID	Type	Wt (mg)	Time	Check	WS Flag
1	Routine QC				
2	Standard Verification				
3	Primary Standard				

The most efficient method of recording, storing, & manipulating PM_{2.5} lab data is to use an **electronic** data management system



Data Management Systems



- Commercially available
- “In-house” acceptable, but should be designed by someone fluent in the Appendix L method
- Should provide QC check results in a format that is easily reviewed **during** the weighing session to immediately assess data quality



STATIC ELECTRICITY

"Yeah, really funny... rub me on the carpet and then put me in the shipping box... You will pay for this!"



How do I know if I have a static problem?

Noisy readout

“Bouncing” around zero, balance never returns to zero

Drift

Slow consistent drift to the positive or negative

Sudden readout shifts

Wild swings after the balance seems to be stable





Static Control

Options Include:

- ✓ Polonium Strips
- ✓ Ionizer bars
- ✓ Ionizer fans
- ✓ Deionizing solutions
- ✓ Grounding



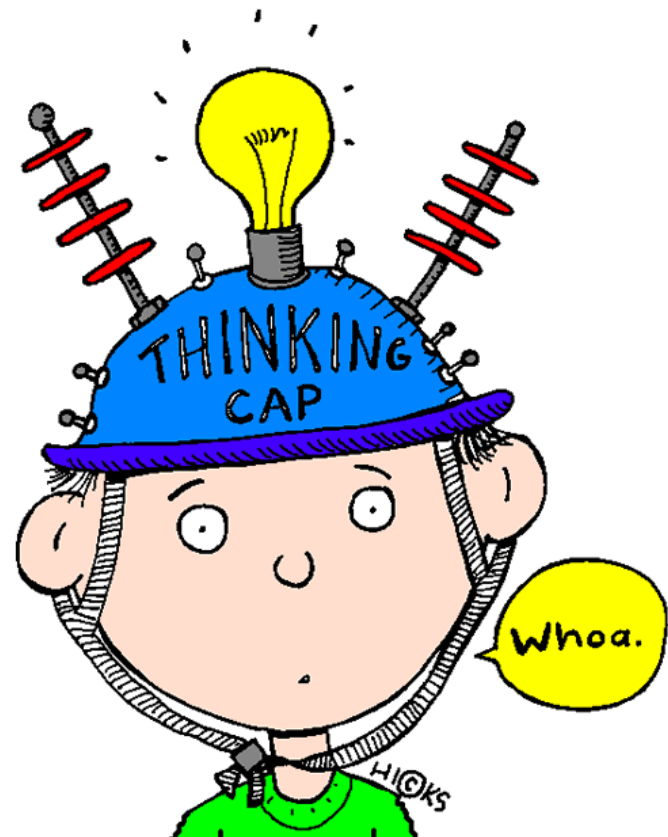


Other Considerations

Do not assume that grounding eliminates all electrostatic buildup because the electrical ground may not be perfect

Dryer environments may increase static charge in the weighing laboratory

Remove sources of static from the lab





Mass Reference Standards

Two separate sets of mass reference standards are needed

- Clearly label the weight sets
- Should be Class 0, 1, or 2 (Tolerance $\leq 25\mu\text{g}$)
- Primary set should ideally be of higher accuracy than working set





Primary Mass Reference Standards

Certification Procedures



Primary weight standards should be certified at least annually (~365 days) by an accredited metrology lab



Best laboratory practice, and improves the defensibility of the subsequent data sets produced



Review results of certification – examine the certificates closely!



Weight standards should not be used if certification has expired



Working Mass Reference Standards

Certification and/or Verification Procedures



When procured, working standards should be accompanied by a **certificate** of NIST-traceability – which documents the **certified mass**



Can be recertified by an accredited metrology lab on an annual basis -- as a best laboratory practice



In-house verification is needed on a quarterly basis (minimum)



Weight standards should not be used if certification/verification has expired



- **Nominal Weight**
 - Target/Approximate Mass
- **Conventional Mass**
 - Nominal Weight + Correction Factor
 - “Certified” Weight
 - Use this value!

- **Tolerance**
 - Maximum permissible error
 - Sum of correction factor + uncertainty
 - Smaller number, higher accuracy





Working Mass Standards

Verified against the primary standards every 90 days (quarterly) to check for mass shifts associated with handling or contamination



Repeated use of the working weight set can cause mass loss



Verification against the primary weight standards in essence “audits” the working weight set -- and ensures there is no shift in the mass weight



Document the verification checks in a logbook and/or on a standardized form



Verification of Working Mass Standards



- Verification does **not** provide a new mass weight!
- It's a QC check only, not an adjustment (*calibration*)!



Weighing Prep and Quality Control Time to put on the lab coat and gloves and get down to business...



Method 2.12, Section 10.2



Initial Weigh: 145.531 mg

Final Weigh: 145.574 mg

Difference = 0.043 mg

Wear gloves!



Filter Integrity Check

All filters should be visually inspected for defects before the initial weighing

Pinhole – A small hole appears as either:

- A distinct and obvious bright point of light when examined over a light table or screen
- A dark spot when viewed over a dark surface

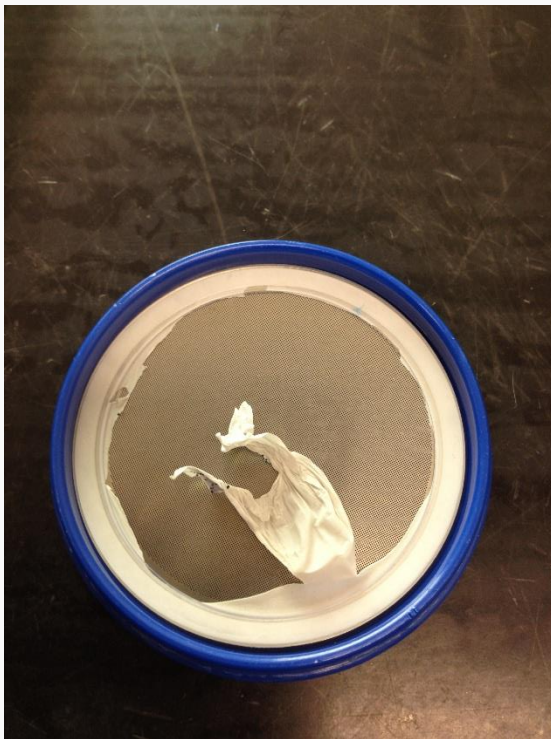
Discoloration

- Any obvious discoloration that might be evidence of contamination





Examples of Filter Damage





Filter Defects

Filter defects are expected periodically, so...

- Order a few more than you need at the beginning of the year to account for defective filters.
- If more than 10% are defective, report the issue to the EPA Regional Office; more filters can be sent to make up for the shortfall.
- Document which and how many filters fail inspection.





Filter Holding Time

8.3.6 The post-sampling conditioning and weighing shall be completed within 240 hours (10 days) after the end of the sample period, unless the filter sample is maintained at temperatures below the average ambient temperature during sampling (or 4° C or below for average sampling temperatures less than 4° C) during the time between retrieval from the sampler and the start of the conditioning, in which case the period shall not exceed 30 days. Reference 2 in section 13.0 of this appendix has additional guidance on transport of cooled filters.

**All filters must be received and maintained below 25° C
(40 CFR Part 50, Appendix L, 10.13)**



End of Sampling



Transported and Stored **Above**
Ambient Sampling
conditions



Post
Equilibration



Post-
Weighing

10 Days to Weigh Filters



End of Sampling



Transported and Stored **Below**
Ambient Sampling
conditions



Post
Equilibration



Post-
Weighing

30 Days to Weigh Filters



Determining Filter Holding Time

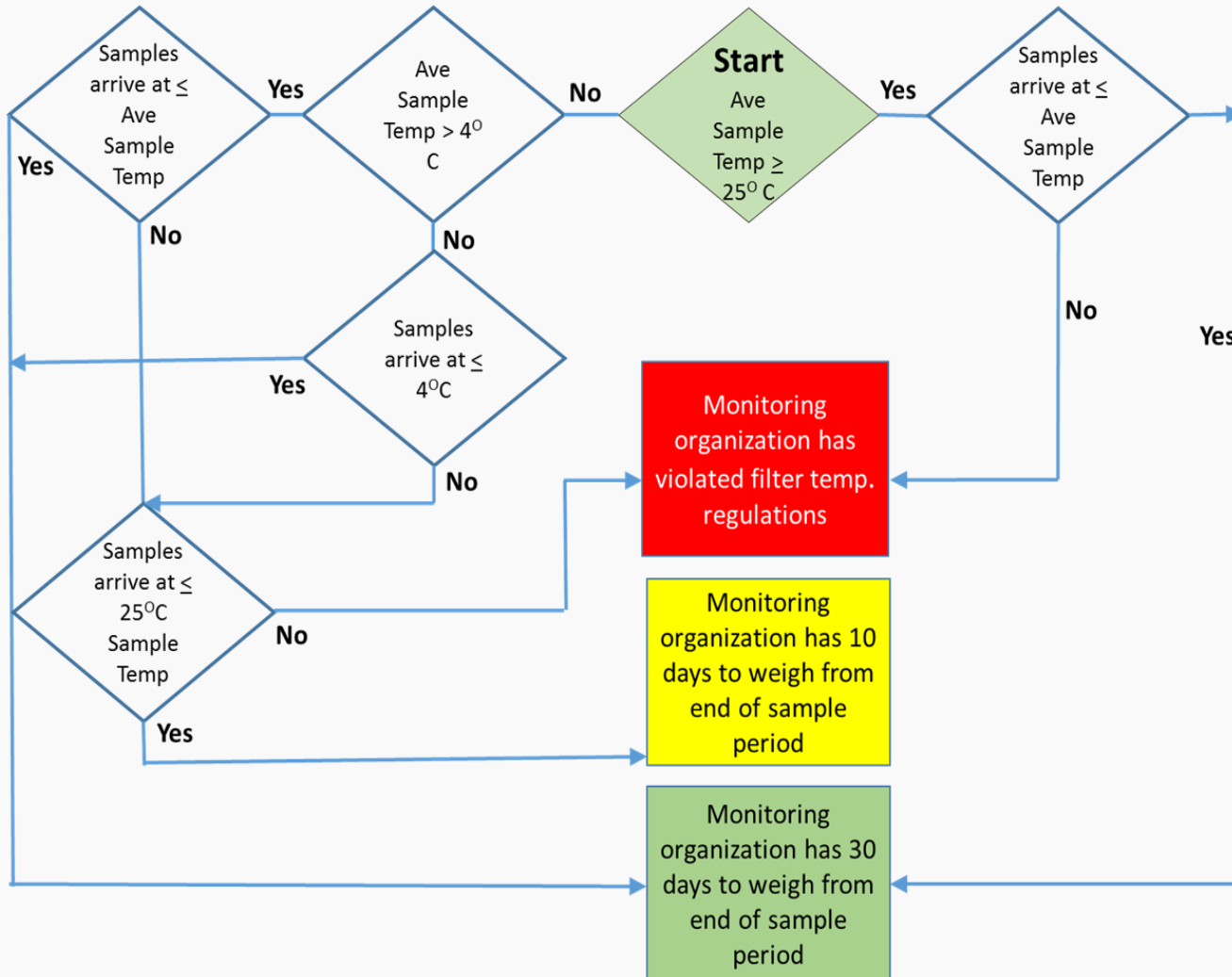
When samples are received at the lab, **verify** the temperature!



- Does the cooler contain ice substitutes?
 - Are they frozen or thawed?
- Is there a min-max thermometer?
 - If so, document the max temp
- Use an IR gun to obtain the current shipment temperature
- Document this value & proceed to the next step...



Determining Filter Holding Time: A Handy Tool





Filter Conditioning **Prior to Sampling – How Long?**

**Filters are always
equilibrated for at
least 24 hours prior
to weighing.**

however...

The lot stability test may indicate that a longer time is required.



Section 8.2 Filter Conditioning



8.2 Filter conditioning. All sample filters used shall be conditioned immediately before both the pre- and post-sampling weighings as specified below. See reference 2 in section 13.0 of this appendix for additional guidance.



Minimizes effects of humidity on the filters during a weighing session



Minimizes effects of humidity across weighing sessions (pre to post)



Establishes a consistent climate for labs to follow nationally so data can be comparable

**All filters must be equilibrated before pre and post weighing.
Conditioning time is 24 hours, minimum.**



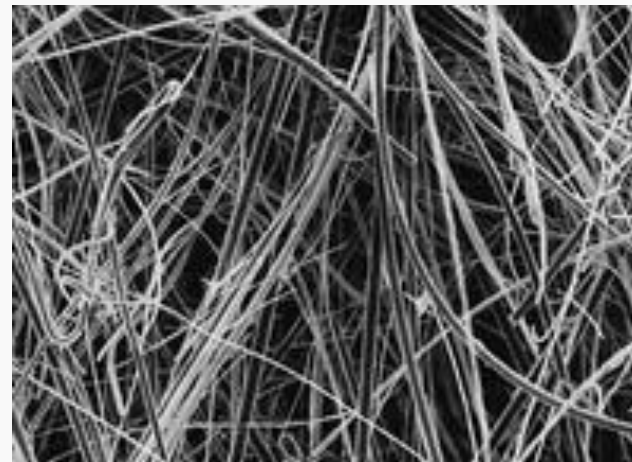
Filter Conditioning

Just a note...

It is the analyst's responsibility to guard against contamination in the lab.



If possible, avoid working with glass or quartz fiber filters in the same area as Teflon[®] filters. These are fibrous materials and can be a source of contamination.





Filter Conditioning After Sampling

Before conditioning exposed filters, determine the filter holding time. (Section 10.7)

If filters can or must be weighed promptly, begin the post-sample weighing activities (Section 10.7) and conditioning.

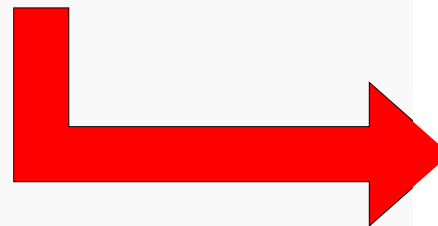




Filter Conditioning After Sampling

If filters cannot be weighed immediately...

Place the filters
in cold storage
(1 – 4 °C) until
weighing
activities occur.



DO NOT FREEZE!



Filter Conditioning After Sampling

Document when the filters were set out for equilibration to set a “**start date**”



Reminder:

Every step must be **documented** as proof that the requirements have been met.

**Otherwise,
it did not happen.**



Filter Conditioning After Sampling

Filters should be left in the weighing room to equilibrate for no more than 72 hours (minimally 24 hours).

Equilibration for extended periods of time can result in the loss of remaining volatiles from the filters.



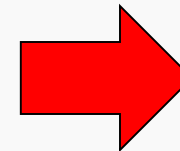


Filter Conditioning After Sampling

If a situation arises where the filters cannot be weighed within 72 hours (HVAC failure, illness)...

Return the filters to cold storage and document the reason and times that the filters were returned.

Be mindful of holding times of samples (10 or 30 days)





Filter Conditioning

Compliance and Specifications

According to 40 CFR Part 50, Appendix L:

Temperature

- ✓ *Mean temperature must be between 20 – 23 °C over 24 hours*
- ✓ *Control of not more than ± 2 °C over 24 hours*

Relative Humidity

- ✓ *Mean RH must be held between 30 - 40% over 24 hours*
- ✓ *Control of not more than ± 5 % over 24 hours*
- ✓ *Pre- and post- RH must be within ± 5 %*



Important clarifications...

1. Temp and RH means are calculated from the 24 hours immediately prior to weighing, **not** midnight to midnight.
2. The control criteria ($\pm 2\text{ }^{\circ}\text{C}$ and $\pm 5\%$ RH) do not mean you can add to the specified ranges. *The ranges are **NOT** 25 to 45% RH or 18 to 25 $^{\circ}$ C for $\text{PM}_{2.5}$.*
3. EPA recommends using the standard deviation for demonstrating control.





Example of Temperature Graph

Temperature

Return Excluded Data Hidden Update On Export Data

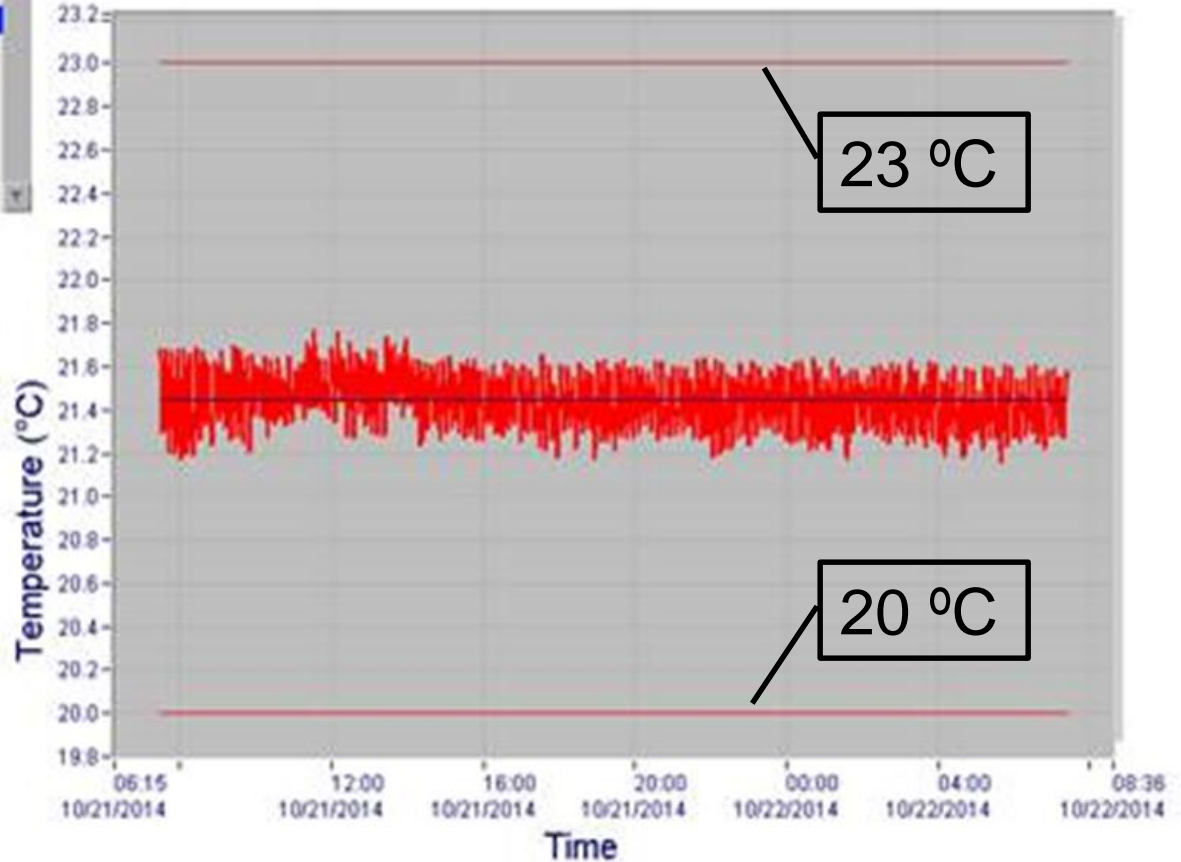
- Primary Location - 200 Fair Oaks Lab
 - Relative Humidity (Vaisala HMT333)
 - Temperature (Vaisala HMT333)**
- All Locations
- All Equipment

Current From 21 OCT 2014
Past To 21 OCT 2014

21 OCT 2014 - 0727 to 22 OCT 2014 - 0724

Mean: 21.44
Max: 21.77
Min: 21.16
Std Dev.: 0.13
Current Reading:
Integrated: 21.48
Immediate: 21.27

- Mean Temperature (°C)
- Temperature (°C)
- Desired 21.50
- High Limit 23.00
- Low Limit 20.00





Example of Relative Humidity Graph

Relative Humidity

Return Excluded Data Hidden Update On Export Data

Primary Location - 200 Fair Oaks Lab

- Relative Humidity (Vaisala HMT333)
- Temperature (Vaisala HMT333)
- All Locations
- All Equipment

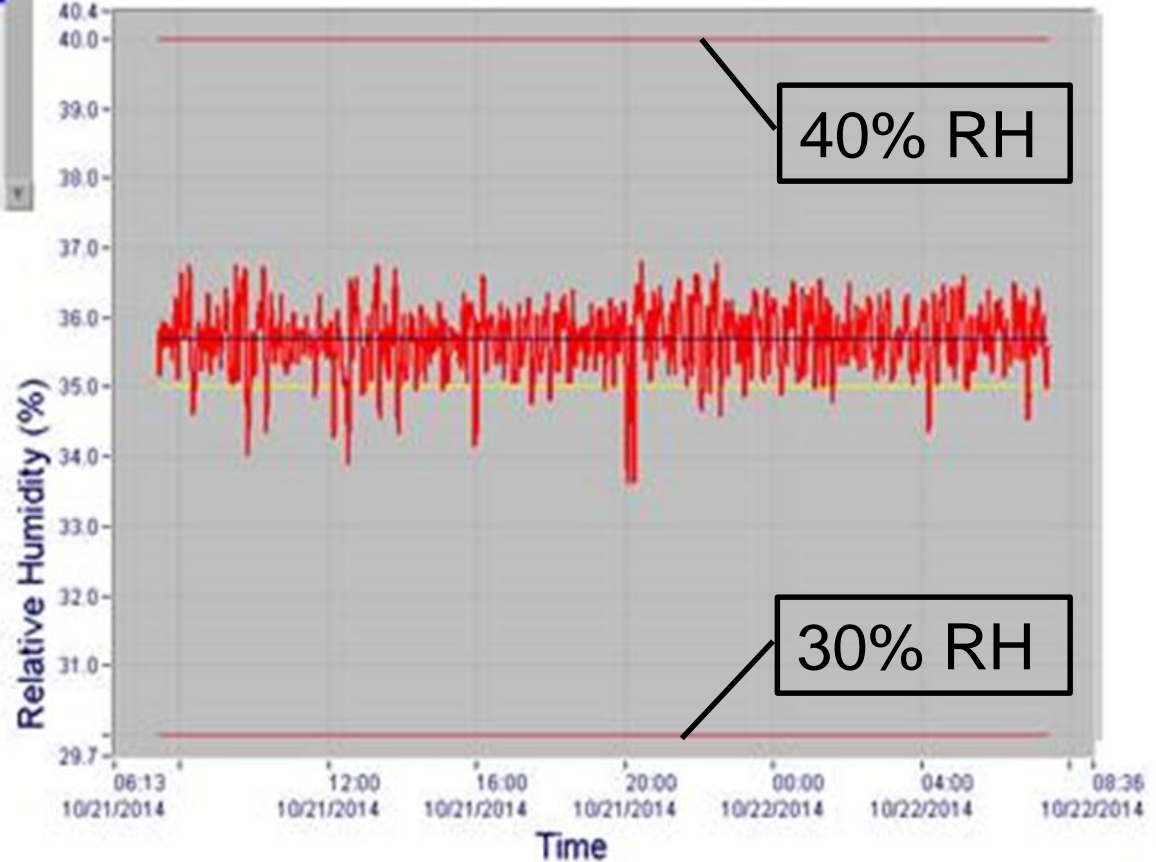
Current From 21 OCT 2014
Past To 21 OCT 2014

21 OCT 2014 - 0725 to 22 OCT 2014 - 0724

Mean: 35.70
Max: 36.79
Min: 33.63
Std Dev: 0.47

Current Reading:
Integrated: 35.58
Immediate: 35.43

- Mean Relative Humidity (%)
- Relative Humidity (%)
- Desired 35.00
- High Limit 40.00
- Low Limit 30.00





How is Lab Control Demonstrated? Two Common Accepted Methods

First Method- Preferred

Calculate a standard deviation (SD) for both the temperature and RH 24-hour period.

$$AS = \sqrt{\frac{n \cdot \sum_{i=1}^n |d_i|^2 - \left(\sum_{i=1}^n |d_i|\right)^2}{n(n-1)}}$$

Don't sweat the formula...
Use an electronic spreadsheet!

For example: If the calculated temperature SD is 1.1, the room passes the control criteria of $\pm 2^\circ \text{C}$.

This is a desirable method to show control because short-term variations, or spikes, in the laboratory data may not affect the SD enough to prohibit the weighing session from taking place.



How is Lab Control Demonstrated?

Two Common Accepted Methods

Second Method

All temperature measurements (min and max) must be within ± 2 °C of the 24-hour temperature mean and all relative humidity measurements must be within $\pm 5\%$ RH of the RH mean.

For example: If the RH mean is 36% RH, then to demonstrate control, all individual measurements must fall within 31% RH and 41% RH.

This is the most conservative way to show control. *If there are any temporary excursions outside of the control limits (± 2 °C from the mean or $\pm 5\%$ RH from the mean) in the weighing room conditions, then the analyst may not weigh filters.*

Section 8.3 Weighing Procedure



8.3.3 Filters must be conditioned at the same conditions (humidity within ± 5 relative humidity percent) before both the pre- and post-sampling weighings.



Pre and post weighing session prior 24-hour means must be within $\pm 5\%$ RH of each other to limit the affects of water vapor between sessions



Example: 33% RH (pre) and 36% RH (post) yields a difference of 3% = Pass



Example: 33% RH (pre) and 39% RH (post) yields a difference of 6% = Fail



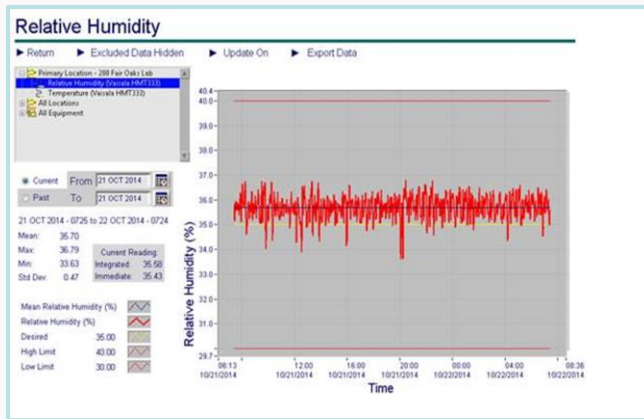
Weighing within prescribed ranges allows national comparability



More About the $\pm 5\%$ RH Criteria

For example:

Pre-weigh RH mean of
33% RH



The post-weigh session must fall within a range of
30% to 38% RH

33% - 5% equals 28%

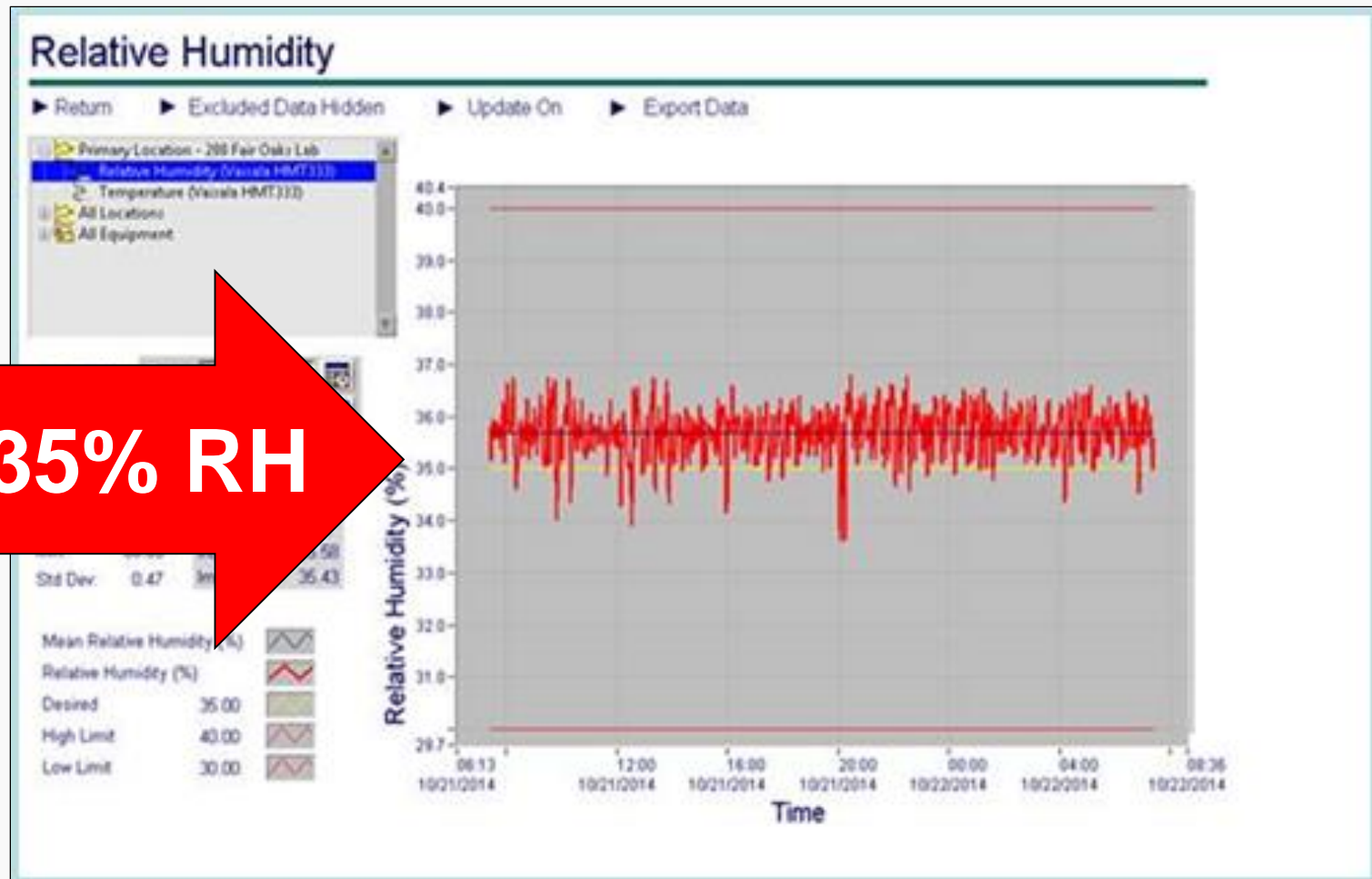
But, **28% is outside of the required RH weighing range!**

Thus 30% is the low end.

Method 2.12, Section 10.4.1.3



Keep the target set-point at or near **35% RH** to provide the widest range for meeting this criterion.





Types of QC Blanks



Required

- **Field Blanks**
- **Lab Blanks**

Recommended

- **Lot Blanks**
- **Trip Blanks**



Lot Blank

Unsampled filter from the **lot** that is used to determine filter weight stability over long periods of time due to the **volatilization** of material from the filter or to the **absorption** of gaseous material into the filter from the atmosphere



Determines the period of time the entire filter lot should be conditioned before it can be used for routine sampling



Laboratory Blanks (LB)

Laboratory blanks are conditioned, unsampled filters used to determine any weight change between pre- and post-sampling weighings due to **contamination in the microbalance environment**



Acceptance criterion is $\leq 15\mu\text{g}$

Weigh enough laboratory blanks during a pre-sampling weigh session to provide at least one **single-use** laboratory blank during each subsequent post-sampling weighing session

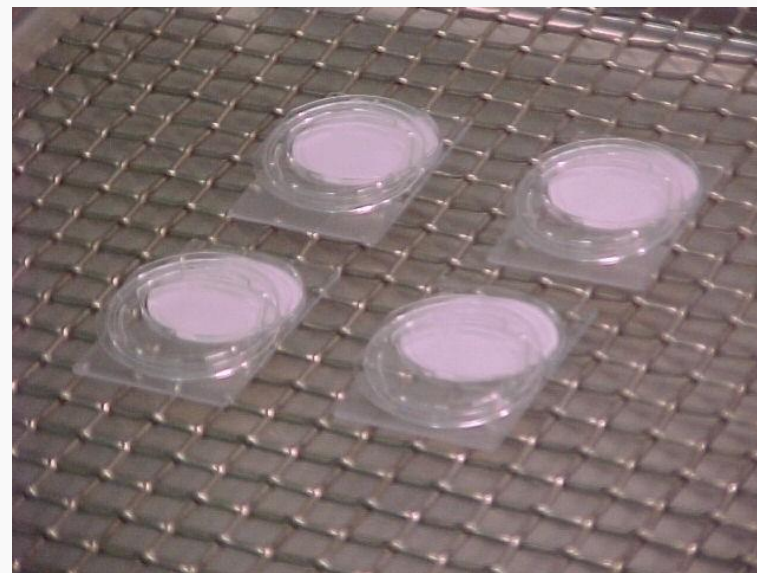


10% of batch, or at least 1 per weigh session



Laboratory Blanks (LB)

- The blanks **follow** the filters in the batch in both pre- and post-weigh sessions
- When routine filters are in the field, the lab blanks are **covered**





Field Blanks (FB)

Conditioned, unsampled filters used to determine whether contamination occurs **during sampling**



Acceptance criterion is $\leq 30 \mu\text{g}$



If exceeded, check results of lab blanks first to help isolate where the problem may be located



The sampler may need to be cleaned. Communicate and report the findings!



Field Blanks (FB)

FBs should be transported to the sampling site, momentarily installed in the sampler, removed, and stored in their protective containers inside the sampler's case at the sampling site, until the exposed filters are retrieved for post-sampling weighing





Trip Blanks

- Recommended best practice
- Treated the same as a FB, except **never placed in the sampler**
- Acceptance criterion is $\leq 15\mu\text{g}$
- Should be implemented at ~5% sampling frequency
- Compare to LB and FB results
- Isolates the source of contamination





Weighing Procedure





Before beginning EACH weighing session

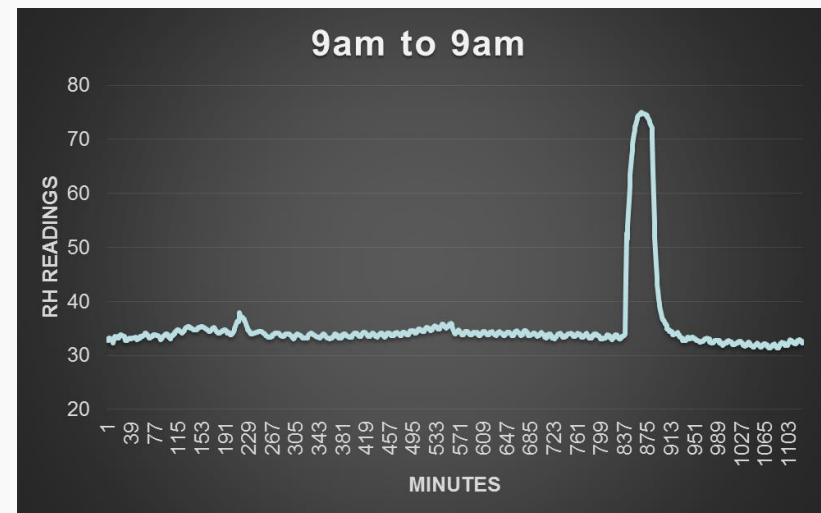
Record the prior 24-hour room means and demonstration of control in database or on bench sheets.

This should be done by the analyst!

If means or control are out of tolerance



DO NOT WEIGH



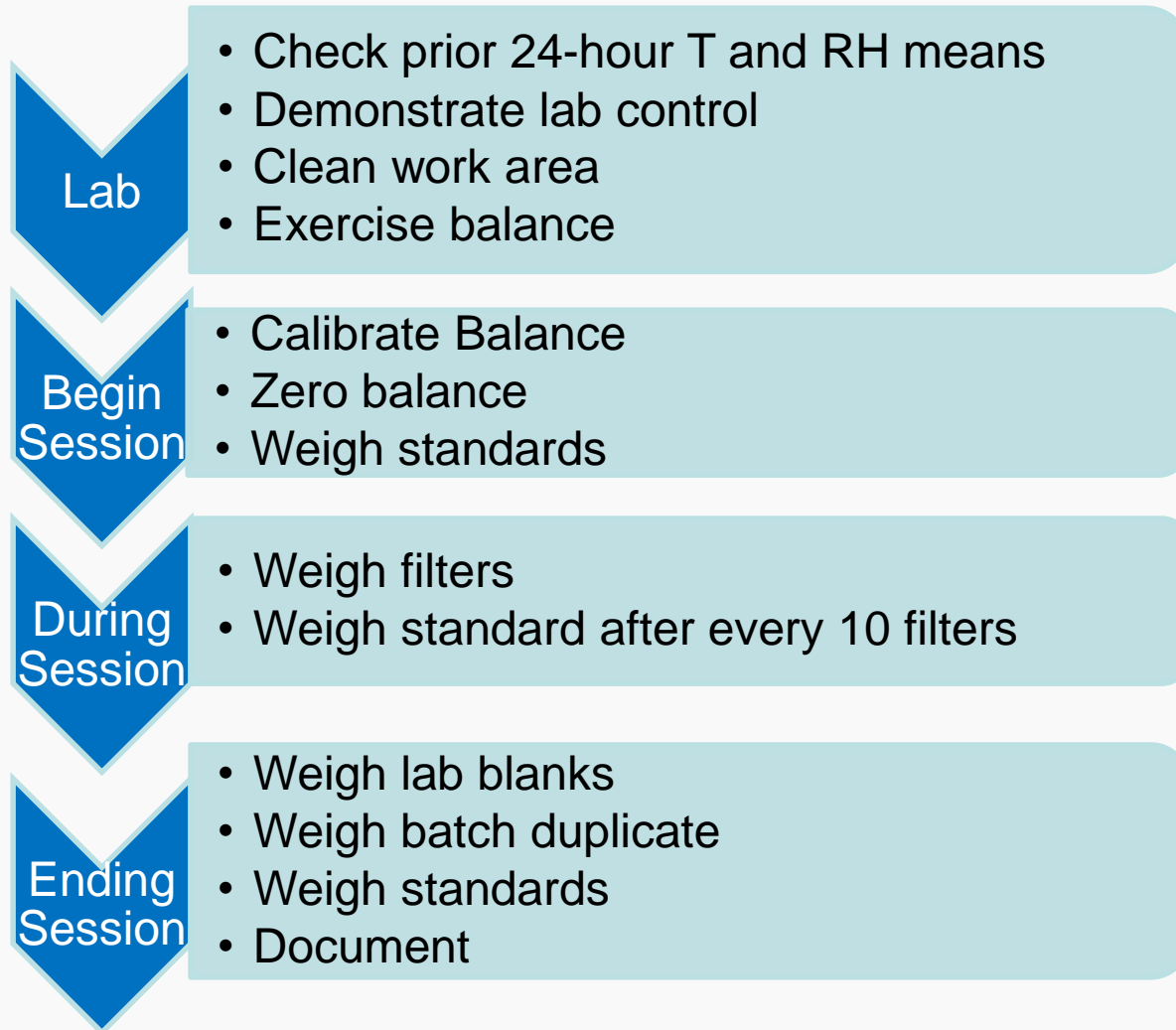


- Method 2.12 distinguishes the conditioning period from the weighing session in the text for convenience of discussion only.
- It is expected that **during** a weighing session the laboratory temperature and humidity conditions **are maintained** within the required specifications of 40 CFR Part 50, Appendix L, Section 8.



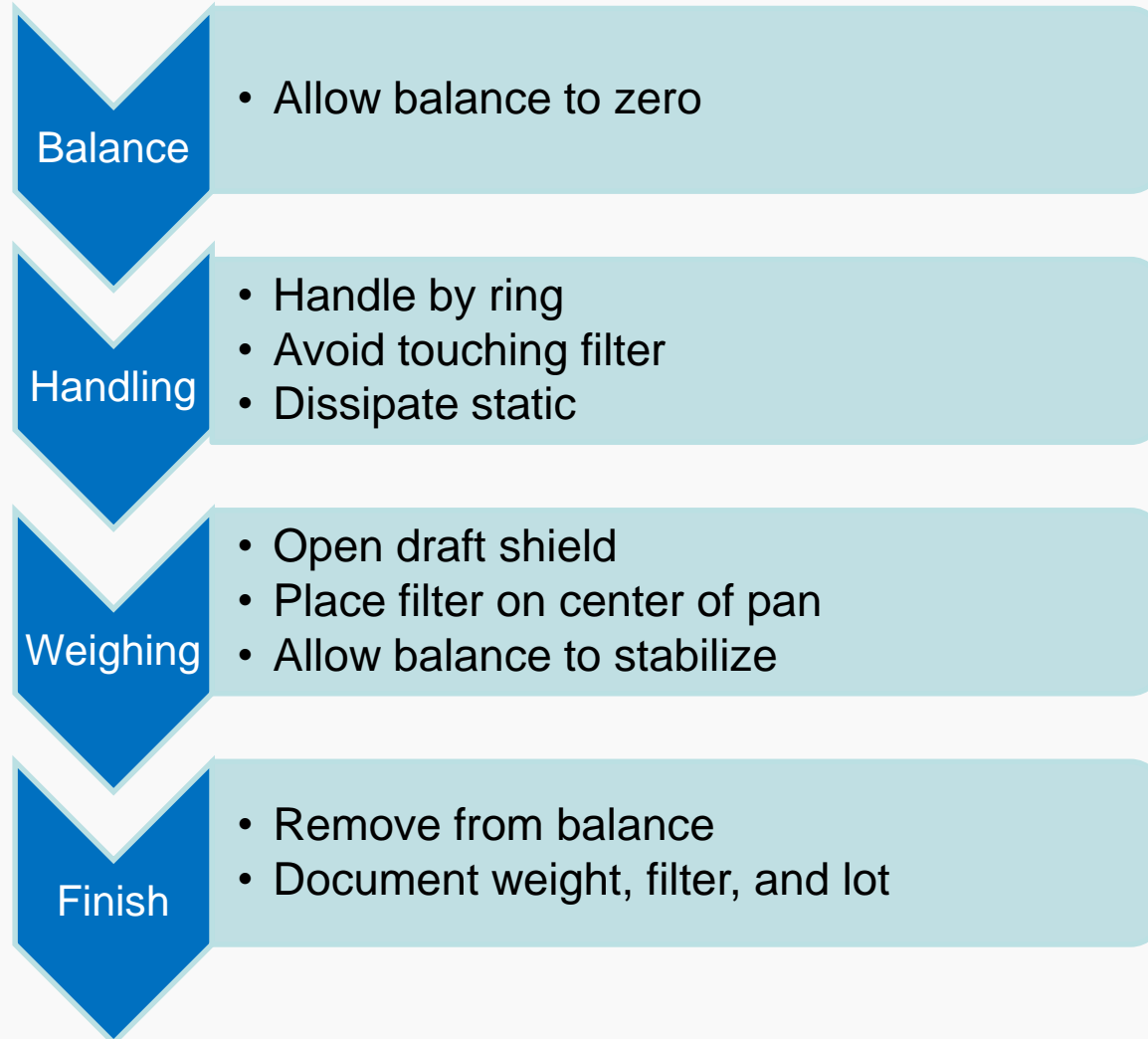


Weighing Summary for Session





Weighing Summary for Individual Filter



Method 2.12, Section 10.6



The QC or laboratory supervisor should **certify** on the laboratory data forms (or in the DMS) the **acceptability** of filter weighings and **QC checks** and the **completeness** of the data.

The QC or laboratory supervisor should **sign** or **initial** data package to validate the data.





Routine QA/QC Procedures



- **Internal QC**
 - Section 10.10
- **Performance Assessment**
 - Section 11
- **Data Audits**
 - Section 12
 - Control Charts
 - Verification of calculations



What is the difference between QA & QC?



QA \neq QC



Routine QA Activities

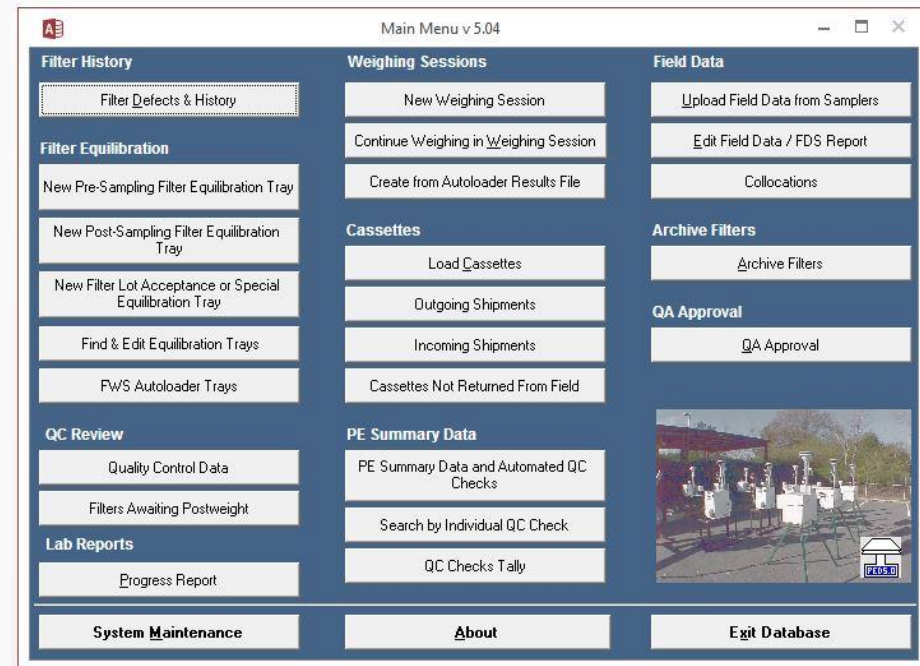


- QMPs, QAPPs, SOPs
- Certification of Standards
- Audits
 - Performance, systems, data
 - Internal & external
- Documentation



Routine QC Activities

- Calibrations
- Verifications
 - Balance checks
 - Quarterly weight checks
 - Replicate weighs
- Blanks
- Control Charts
- Documentation





QAPPs and SOPs are like a contract...

An agency is held accountable to the procedures they formalize in their QAPPs & SOPs





Develop and adhere to your SOP!

- **Know** why you are doing what you are doing
 - Understand the method
 - Ask questions!
- **Personalize** your SOP so it is reflective of true agency practices, rather than the “ideal”
- Strictly **follow** all procedures concerning weighing, labeling, and transporting filters to reduce the risk of measurement error
 - Be consistent!



Internal Quality Control

Record PM_{2.5} weighing lab data in laboratory database or laboratory logbook.

Custom laboratory database is preferred to organize, store and analyze PM_{2.5} specific lab and QC data

DATABASE





Internal Quality Control

Backup data, both electronic and hard copies, to a secure off-site location





Laboratory Recordkeeping

Types of Records

- Logbooks
- Standardized Forms
- Spreadsheets
- Databases
- Chain-of-custody forms
- Others?

Daily Weigh Room Report For PM_{2.5}

Weigh Room No.:
 Report Date:
 Date Range of Report:
 From: To:
Time Date Time Date

	Temperature (°C)	Relative Humidity (%RH)
Average	20.8 PASS	33.6 PASS
Standard Deviation (n-1)	0.3 PASS	0.9 PASS
Actual Minimum	19.9 In Range	31.4 In Range
Acceptable Minimum (Avg Temp -2/Avg %RH -5)	18.8	28.6
Actual Maximum	21.3 In Range	35.7 In Range
Acceptable Maximum (Avg Temp +2/Avg %RH +5)	22.8	38.6

Pre/Post Batch: Stability:
 Weighing Analyst: Printed Initials: Written Initials:
Approvals:
 Peer: Printed Initials: Written Initials:
 Manager: Printed Initials: Written Initials:

Pre-Batch Humidities (List all pre-batches weighed within 60 days of this date for post-batch sessions)

	Date	Pre-Batch Average	%RH	Diff from Avg RH above	Pass/Fail
1				33.6	FAIL
2				33.6	FAIL
3				33.6	FAIL
4				33.6	FAIL
5				33.6	FAIL

Comments:



Questions to Ask Yourself...



- Are records **organized**?
- If asked to retrieve a record from several years ago, **could you easily find it**?
- Could someone from **outside** your agency easily find it?



More Questions...

- Do your records have **detail**?
 - Will you remember several years from now exactly what the issue was?
 - Will someone besides you be able to recall & *understand* what happened based upon the written information?
 - Be specific!
 - Can you **recreate** your data?





Documenting laboratory logbooks is a QA/QC best practice!



- Calibrations
- Maintenance
- Equipment malfunctions & repair
- Discrepancies
- Software upgrades
- Other significant events



If it's not documented, **it did not happen!**





Equipment Audits in the Lab

- Temperature sensor
 - $\pm 2^{\circ}\text{C}$
- RH sensor
 - $\pm 2\% \text{ RH}$
- Balance
 - Primary standards
 - Class 1 weights
- Calibration verification checks!
- Identifies imprecision & bias
- Needed every 6 months
 - Recommend more frequent checks



External Systems Audits

**40 CFR Part 58, Appendix A,
Section 2.5**

*Technical systems audits of each ambient air monitoring organization shall be conducted **at least every 3 years** by the appropriate EPA Regional Office and reported to the AQS...*

Includes weigh labs!





Internal Systems Audits

- One of the best practices an air agency can implement is to conduct **internal systems audits** on a routine basis!
 - Should **include weigh lab**, to encompass entire PM_{2.5} program
- Include in QAPP





Internal Systems Audits

- Use an EPA checklist
- Or, develop your own!
- Implement routine audit schedule
 - Annual, at minimum
- Document findings & corrective actions

PEP Laboratory Technical Systems Audit Form

Finding Level: 1=exemplary; 2=satisfactory; 3=needs small improvement; 4=unsatisfactory and needs significant attention; 5=shortfall or catastrophic condition that needs immediate attention

Section Question No.	Audit Question	Yes	No	N/A	Response or Comment	Finding Level
Section 1 Laboratory Management and Quality System Requirements						
1-1	Does the laboratory operate under a Quality Management Plan?					
1-2	Are the EPA organizational structure and responsibilities of oversight...					
1-3						
1-4						
1-5						
1-6						
1-7						
1-8						
1-9						
1-10						
1-11						
1-12						
1-13						
1-14						
1-15						
1-16						
1-17						
1-18						
1-19						

**PM_{2.5} Filter Weighing Laboratory
Evaluation Form for Validation Criteria**

Evaluator: _____ Date: _____

Signature: _____

References for Evaluation: 40 CFR Part 50, Appendix L; Quality Assurance Guidance Document 2.12 Monitoring PM_{2.5} in Ambient Air Using Designated Reference or Class I Equivalent Methods; Quality Assurance Guidance Document, Method Compendium, Laboratory Standard Operating Procedures for the PM_{2.5} Performance Evaluation Program

Critical Validation Elements <small>(to be reviewed during a data validation of no less than 5 data points)</small>				
Elements:	Yes	No	NA	Comments
Post Sampling Weighing				
Filters weighed within 30 days? (rec'd ≤ 4° C)				
Filters weighed within 10 days? (rec'd ≤ 25° C)				
Filter Conditioning				
Pre-Equilibration (> 24 hours and according to lot stability test)				
Post-Equilibration (> 24 hours and according to lot stability test)				
Weighing Chamber Climate Control				
Temperature Range (24-hr mean 20-23 °C)				
Temperature Control (± 2° C SD over 24 hr)				
Humidity Range (24-hr mean 30% - 40% RH or ≤ 5% sampling RH but > 20% RH)				
Humidity Control (± 5% SD* over 24 hr)				
Pre- and Post Sampling RH Difference (24-hr means ≤ 5% RH)				
Visual Defect Check (examples)				
Balance kept in "on" status and in weighing room				
Balance is grounded for static control				
Print and review temperature and humidity graphs for two prior weighing sessions				
Weighing room criteria session 1:	24 Hour Temp Mean: _____ °C	Temp SD _____	%RH mean _____ %	RH SD _____
Weighing room criteria session 2:	24 Hour Temp Mean: _____ °C	Temp SD _____	%RH mean _____ %	RH SD _____
Data logger functioning correctly; no repetitive measurements				
Critical Element Review Notes:				

PM_{2.5} Weighing Laboratory Evaluation Form



Internal Audit Benefits

- Illustrates areas where supplemental training may be beneficial
- Prevents data loss
- Improves overall data quality
- Enhances quality system
- Small issues won't become big issues!
- **Significantly reduces EPA findings during the regulatory TSA!**





Calculations, Validations, and Reporting of PM_{2.5} Monitoring Data

- Check your work
- Don't rely on your computer to flag everything for you
 - Know the method & requirements!
- Data audits





Verification of Calculations

- Review weigh session results
- Verify math
 - All checks pass?
 - Computations correct?
- Independent reviewer preferred

*“A commonly used guideline is to check **7%** of the manual calculations, provided that at least one example of each type of calculation is checked.”*



Verification of Manual Data Entry

Duplicate Keying

- Data entry by two different operators
- Eliminates keystroke mistakes
- More cost-effective for large data sets

Proofing

- Visual comparison of data entered by a single operator against the original forms
- Less up-front costs



Validation of Software

“Software used to process, manage, & report PM_{2.5} data used for compliance purposes must be validated to ensure it is free of incorrectly coded calculations and errors.”





Validation of Software



- Correctness of calculations
- Correct assignment of input & output values
- Correct computation of statistics
- Correct application of error flags