



# ***PM<sub>2.5</sub> Weighing Laboratory Training***

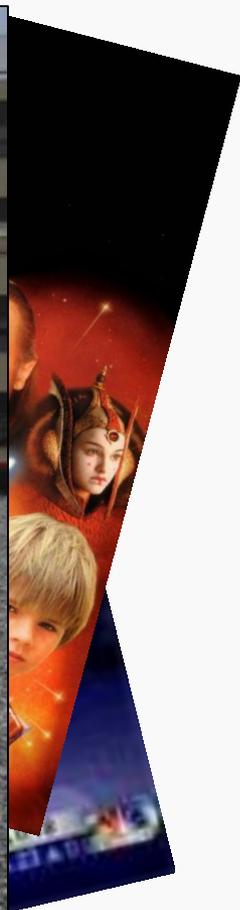
***Requirements, what it looks like, how to assess it  
and a little bit of fun...***



***Greg Noah***

***USEPA, OAQPS, Ambient Air Monitoring Group***

In 1999...



# Since 1999...



*We've been weighing  $PM_{2.5}$  filters for going on 15 years, why the attention now?*

## ***Recent Lab Audits Have Re-focused Our Attention***

*Regional questions*

*SLT questions*

*Headquarters questions*

*Need for national assessments (AQS)*

## ***Routine Assessments***

*Bias between FRM and FEM methods*

*Changes in  $PM_{2.5}$  PEP bias*



# Why Are We Seeing Issues Right Now?



## ***Changing of the Guard?***

*New leadership and new staff members – “You don’t know what you don’t know”*

## ***Better at Assessing Data?***

*Data analysts are finding better ways to pull and look at the data.*

## ***Lack of Expertise of Auditors (State and Feds)?***

*Is there a training need for people doing the work and auditing the program?*



# ***PM<sub>2.5</sub> Filter Weighing is Easy... and Complicated***



***Placing a filter on a microbalance is not difficult, knowing the requirements, guidance, and managing the program can be tough.***

## ***PM<sub>2.5</sub> Filter Weighing Requirements and Guidance:***

- *40 CFR Part 50, Appendix L, Section 8 (Method)*
- *40 CFR Part 50, Appendix L, Section 10 (Filters)*
- *Quality Assurance Guidance Document 2.12, Monitoring PM<sub>2.5</sub> in Ambient Air Using Designated Reference or Class I Equivalent Methods*
- *Quality Assurance Handbook for Air Pollution Systems, Volume II, May 2013*
- *PM<sub>2.5</sub> Performance Evaluation Program Standard Operating Procedures*

# Weighing Lab QA/QC Requirements



The QA Handbook summarizes the QA/QC needed for operation of a successful filter weighing laboratory

**Critical Criteria Table – Required by CFR, not meeting these will most likely result in an INVALID sample**

Laboratory Activities			
Post-sampling Weighing	all filters	≤10 days from sample end date if shipped at ambient temp, or ≤30 days if shipped below avg ambient (or 4° C or below for avg sampling temps < 4° C) from sample end date	1, 2 and 3) 40 CFR Part 50 App L Sec 8.3.6
Filter Visual Defect Check (unexposed)	all filters	Correct type & size and for pinholes, particles or imperfections	1, 2 and 3) 40 CFR Part 50, App L Sec 10.2
Filter Conditioning Environment			
Equilibration	all filters	24 hours minimum	1, 2 and 3) 40 CFR Part 50, App L Sec 8.2.5
Temp. Range	all filters	24-hr mean 20-23° C	1, 2 and 3) 40 CFR Part 50, App L Sec 8.2.1
Temp. Control	all filters	+ 2° C SD* over 24 hr	1, 2 and 3) 40 CFR Part 50, App L Sec 8.2.2
Humidity Range	all filters	24-hr mean 30% - 40% RH or ≤5% sampling RH but > 20%RH	1, 2 and 3) 40 CFR Part 50, App L Sec 8.2.3
Humidity Control	all filters	+ 5% SD* over 24 hr.	1, 2 and 3) 40 CFR Part 50, App L Sec 8.2.4
Pre/post Sampling RH Balance	all filters	difference in 24-hr means ≤ ± 5% RH located in filter conditioning environment	1, 2 and 3) 40 CFR Part 50, App L Sec 8.3.3 1, 2 and 3) 40 CFR Part 50, App L Sec 8.3.2

Web Link: <http://www.epa.gov/ttnamti1/files/ambient/pm25/qa/QA-Handbook-Vol-II.pdf> Appendix D, Page 15 of 48

# Noah's Notes on Critical Criteria Questions...



Criteria	Application	Requirement	Noah's Notes
Post-sampling Weighing	all filters	$\leq 10$ days from sample end date if shipped at ambient temp, or $\leq 30$ days if shipped below avg ambient (or $4^{\circ}$ C or below for avg sampling temps $< 4^{\circ}$ C ) from sample end date	The clock starts from the sample end date, period. Samples refrigerated until set out for equilibration. DO NOT FREEZE samples!
Filter Visual Defect Check (unexposed)	all filters	Correct type & size and for pinholes, particles or imperfections	Quick check in the field
Filter Conditioning Environment			
Equilibration	all filters	24 hours minimum	Filters should be in the climate controlled weighing lab
Temp. Range	all filters	24-hr mean $20-23^{\circ}$ C	24 hours immediately prior to weighing (5 min logging preferred)
Temp.Control	all filters	$\pm 2^{\circ}$ C SD* over 24 hr	We have chosen the SD to show control, a min/max could be acceptable
Humidity Range	all filters	24-hr mean 30% - 40% RH or $\leq 5\%$ sampling RH but $> 20\%$ RH	24 hours immediately prior to weighing (5 min logging preferred)
Humidity Control	all filters	$\pm 5\%$ SD* over 24 hr.	We have chosen the SD to show control, a min/max could be acceptable
Pre/post Sampling RH	all filters	difference in 24-hr means $\leq \pm 5\%$ RH	See range comments above
Balance	all filters	located in filter conditioning environment	No comment

# Weighing Lab QA/QC Requirements



## Operational Evaluations Table – Criteria developed to help assess/evaluate the system to ensure data quality, not mandated

Laboratory Activities			
<b>Filter Checks</b>			
Lot Blanks	9 filters per lot	less than 15 µg change between weighings	1, 2, 3) Recommendation and used to determine filter stability of the lot of filters received from EPA or vendor.
Exposure Lot Blanks	3 filters per lot	less than 15 µg change between weighings	1,2 and 3) Method 2.12 Sec. 7.7
Filter Integrity (exposed)	each filter	no visual defects	Used for preparing a subset of filters for equilibration
Filter Holding Times			1,2 and 3) Method 2.12 Sec. 7.10
<i>Pre-sampling</i>	<i>all filters</i>	<i>&lt; 30 days before sampling</i>	1,2 and 3) <a href="#">40 CFR Part 50, App.L, Sec 8.3.5</a>
<b>Lab QC Checks</b>			
<i>Field Filter Blank</i>	10% or 1 per weighing session	± 30 µg change between weighings	1) 40 CFR Part 50, App.L Sec 8.3.7.1 2 and 3) Method 2.12 Sec. 7.7
<i>Lab Filter Blank</i>	10% or 1 per weighing session	± 15 µg change between weighings	1) 40 CFR Part 50, App.L Sec 8.3.7.2 2 and 3) Method 2.12 Sec. 7.7
Balance Check (working standards)	beginning, 10th sample, end	≤ 3 ±µg	1,2 and 3) Method 2.12 Sec. 7.9
Duplicate Filter Weighing	1 per weighing session	± 15 µg change between weighings	1,2 and 3) Method 2.12 Sec 7.11
Microbalance Audit	1/yr	± 0.050 mg or manufacturers specs, whichever is tighter	1,2 and 3) Method 2.12 Sec. 10.2.6
<b>Verification/Calibration</b>			
Lab Temperature	1/6 months	± 2°C	1) Method 2.12 Table 3-2 2) Recommendation. Table 3-2 suggests every 3 mo. 3) Method 2.12 Table 3-2
Lab Humidity	1/6 months	± 2%	1) Method 2.12 Table 3-2 2) Recommendation Table 3-2 suggests every 3 mo. 3) Method 2.12 Table 3-2
<i>Microbalance Calibration</i>	<i>At installation and prior to each weighing session</i> 1/yr	Manufacturer's specification	1) 40 CFR Part 50, App.L, Sec 8.1 2) 40 CFR Part 50, App.L, Sec 8.1 and Method 2.12 Sec. 7.2 3) NA
<b>Calibration &amp; Check Standards -</b>			
Working Mass Stds. (compare to primary standards)	1/3 mo.	0.025 mg	1, 2 and 3) <a href="#">Method 2.12</a> Sec 4.3 and 7.3
Primary standards	1/yr	0.025 mg	

# Noah's Notes on Operational Evaluation Questions



<b>Criteria</b>	<b>Application</b>	<b>Requirement</b>	<b>Noah's Notes</b>
<i>Filter Checks</i>			
<i>Lot Blanks</i>	<i>9 filters per lot</i>	<i>less than 15 <math>\mu\text{g}</math> change between weighings</i>	<i>Verifies that 24 hours is enough time for equilibration, I highly recommend for new filters</i>
<i>Exposure Lot Blanks</i>	<i>3 filters per lot</i>	<i>less than 15 <math>\mu\text{g}</math> change between weighings</i>	<i>Verifies filters have equilibrated</i>
<i>Filter Holding Times</i>			
<i>Pre-sampling</i>	<i>all filters</i>	<i>&lt; 30 days before sampling</i>	<i>CFR criteria, reduces chance of contamination</i>
<i>Lab QC Checks</i>			
<i>Field Filter Blank</i>	<i>10% or 1 per weighing session</i>	<i><math>\pm 30 \mu\text{g}</math> change between weighings</i>	<i>Great tool for seeing field or sampling issues</i>
<i>Lab Filter Blank</i>	<i>10% or 1 per weighing session</i>	<i><math>\pm 15 \mu\text{g}</math> change between weighings</i>	<i>Great tool for isolating lab issues</i>
<i>Balance Check (working standards)</i>	<i>beginning, 10th sample, end</i>	<i><math>\leq 3 \pm \mu\text{g}</math></i>	<i>Shows balance precision over the course of the session</i>
<i>Duplicate Filter Weighing</i>	<i>1 per weighing session</i>	<i><math>\pm 15 \mu\text{g}</math> change between weighings</i>	<i>Demonstrates stability over the course of a weighing session</i>

# Noah's Notes on Operational Evaluation Questions



## Operational Criteria Continued...

<b>Criteria</b>	<b>Application</b>	<b>Requirement</b>	<b>Noah's Notes</b>
<i>Verification /Calibration</i>			
<i>Lab Temperature</i>	<i>1/6 months</i>	<i>± 2°C</i>	<i>I would verify more frequently, temp is critical and can invalidate data.</i>
<i>Lab Humidity</i>	<i>1/6 months</i>	<i>± 2%</i>	<i>Same as above</i>
<i>Microbalance Calibration</i>	<i>At installation and prior to each weighing session 1/yr</i>	<i>Manufacturer's specification</i>	<i>Verifies balance is functioning properly</i>
<i>Calibration &amp; Check Standards</i> -			
<i>Working Mass Standards and Primary Standards</i>	<i>1/3 mo. 1/yr</i>	<i>0.025 mg 0.025 mg</i>	<i>Ensures there is no shift in mass with the weights</i>



## ***Systematic Criteria Table – Specifications for the equipment needed in the laboratory to ensure precise measurements***

		Laboratory Activities	
<i>Microbalance Readability</i>	<i>at purchase</i>	<i>1 µg</i>	1, 2 and 3) 40 CFR Part 50, App.L Sec 8.1
Microbalance Repeatability	1/yr	1 µg	1) Method 2.12 Sec 4.3.6 2) Recommendation 3) Method 2.12 Sec 4.3.6
Primary Mass. Verification/Calibration Standards Recertifications	1/yr	0.025 mg	1, 2 and 3) Method 2.12 Sec 4.3.7 & Table 3-2

Web Link: <http://www.epa.gov/ttnamti1/files/ambient/pm25/qa/QA-Handbook-Vol-II.pdf> Appendix D, Page 18 of 48

# Noah's Notes on Systematic Criteria Questions



<b>Criteria</b>	<b>Application</b>	<b>Requirement</b>	<b>Noah's Notes</b>
<i>Microbalance Readability</i>	<i>at purchase</i>	<i>1 <math>\mu</math>g</i>	<i>Ensures MDL can be reached</i>
<i>Microbalance Repeatability</i>	<i>1/yr</i>	<i>1 <math>\mu</math>g</i>	<i>Ensures precision</i>
<i>Primary Mass. Verification/Cal Standard Recert</i>	<i>1/yr</i>	<i>0.025 mg</i>	<i>Requirement for Class 2 weights</i>

# *To Meet These Criteria, What Should My Lab Look Like?*



*Common sense elements that all weighing labs should incorporate*

*Clean and Well Maintained*

*Static Controlled*

*Good Procedures and QAPP*

*Consistent Weighing Procedure*

*Efficient Data Management System*

*Good Documentation*

*Routine Audits*

*Proper Resources*

# What Should My Lab Look Like?



## Clean and Well Maintained

### **Clean on an established schedule**

*Written in SOP, FOLLOWED!*

### **Minimize sources of contamination**

*Sticky mats, minimal fibrous material, gloves, lab coats*

### **Use anti-static solutions to clean**

*No brooms, semi-wet methods – damp cloth*

### **Clean everything!**

*Inside balance draft shield, forceps, ceiling*



# What Should My Lab Look Like?



## Static Controlled

### **Ground everything**

*Microbalances, stability trays, wear antistatic gloves/coat*

### **Minimize static sources**

*CRT monitors, cell phones, clothing*

### **Use static control devices**

*Polonium strips, ionizing bars*



# What Should My Lab Look Like?



## Good Procedures and QAPP

### **Know your SOP and QAPP**

*Keep them updated*

*Know why you do what you are doing*

*If something goes wrong have a good corrective action plan*

*Define action levels*

*Review the SOP and QAPP annually and submit modifications for EPA approval*



# What Should My Lab Look Like?



## Consistent Weighing Procedure

### **Use the same weighing analyst**

*Reduces bias, reduces confusion, same analyst should weigh filters pre- and post-*

### **Follow the SOP**

*Do not deviate from the procedure. Biggest way to get into trouble*

### **Develop a consistence process**

*In accordance with the SOP, develop a flow that works for your lab*



# What Should My Lab Look Like?



## Efficient Data Management System

### **Use a data management system**

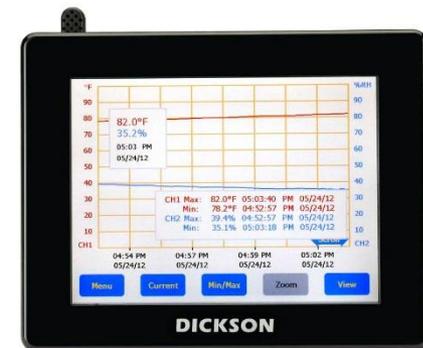
*Avoid Excel spreadsheets, hand-written bench sheets, loose documentation*

### **Integrate logging systems/balances**

*Integration of %RH and Temp statistics with lab system ensure conditions are recorded, integrating balances reduce entry errors*

### **Use conditional checks**

*Automate room condition criteria checks, calibration dates, QC checks -  
Make it obvious if there is a failure!*



# What Should My Lab Look Like?



## Good Documentation

### **Document criteria and QC check results**

*If you don't document you didn't do it or pass it, Period*

### **Ensure documentation is complete**

*Incomplete documentation is as bad as not documenting at all*

### **Records management**

*Follow your QAPP regarding records management. If you can't find it, you didn't do it*



*"The Ministry of Foreign Affairs plans to sort and digitise its stacks of paper archives stored haphazardly over the past 30 years"*

# What Should My Lab Look Like?



## Routine Audits

### ***Audit the lab, internally and externally***

*Identifies imprecision and shows bias, audit loggers, weights, balances, analysts, etc...*

### ***Use control charts***

*Control charts will identify trends **before** they become problems*

### ***Correct issues found in audits***

*Finding an issue and not correcting it is like not doing the audit at all, **FOLLOW UP!!***



# What Should My Lab Look Like?



## Proper Resources

### **Lab**

*Free of vibrations and drafts, well insulated, interior room, well lit*

### **Weighing Instrumentation**

*Meet accuracy requirements, keep balances stationary and level, integrate with data management software*

### **HVAC system**

*Large enough for expected temp and humidity ranges throughout the year, re-heat systems work well, HEPA filtered, service contract*

### **Staffing**

*Organizational skills are as important as technical skills in the weighing laboratory, not entry level position*

# Auditing Weighing Labs



***How do I know if my lab is operating well? AUDIT!!***

*The EPA Regions should audit the monitoring programs once every three years as a part of the Technical Systems Audit*

*PQAO(s) served by a lab (state, local or contract lab) should audit that lab each year as part of a Technical Systems Audit*

*If there are multiple analysts, each analyst could audit the other as a way to gain better consistency*

*Participate in weighing lab round robin audits.*

***Auditing remotely (desktop audit, data analysis) is in no means as effective as being there in person. Traveling to the labs must be a priority. DON'T AUDIT REMOTELY!***



## Audit Templates

### Two Choices Available from EPA

#### *Long form*

*Similar to traditional TSA checklists "process oriented"*

*The most detailed of the two*

*Long, taking more time to complete*

*Does not direct regarding data review*

#### *Short form*

*Focuses on validation criteria*

*Requires a better understanding of the lab procedures*

*Includes data retrieval and review in the form*

*Less emphasis on procedure*

# *Auditing Weighing Labs*



***Or...  
Create your  
own  
according to  
the method  
and your  
agency QAPP***



# Auditing Weighing Labs



## **Words of Caution...**

*When using a template, there is a temptation to look at the template and not the lab*



*The template is a guide, not a "catch all" security blanket*

*If you don't look at the QC data, you haven't looked at the lab*



# *In Summary...*



## ***We need to re-focus our attention to the weighing laboratories***

- ✓ *There is a lot of criteria, but learning it is not insurmountable*
- ✓ *The QA Handbook helps with learning the method and identifying the importance and use of the criteria*
- ✓ *There are common sense elements every lab should have*
- ✓ *Audit the lab routinely and in person – NO DESKTOP AUDIT!*
- ✓ *Two templates are readily available for use in auditing weighing laboratories*



# *Real World Audit*



*Let's take a look at a real lab*



*OAQPS Weighing Laboratory*

# *Real World Audit*



***What's good, what's not so good***

***This is the audience participation part...***

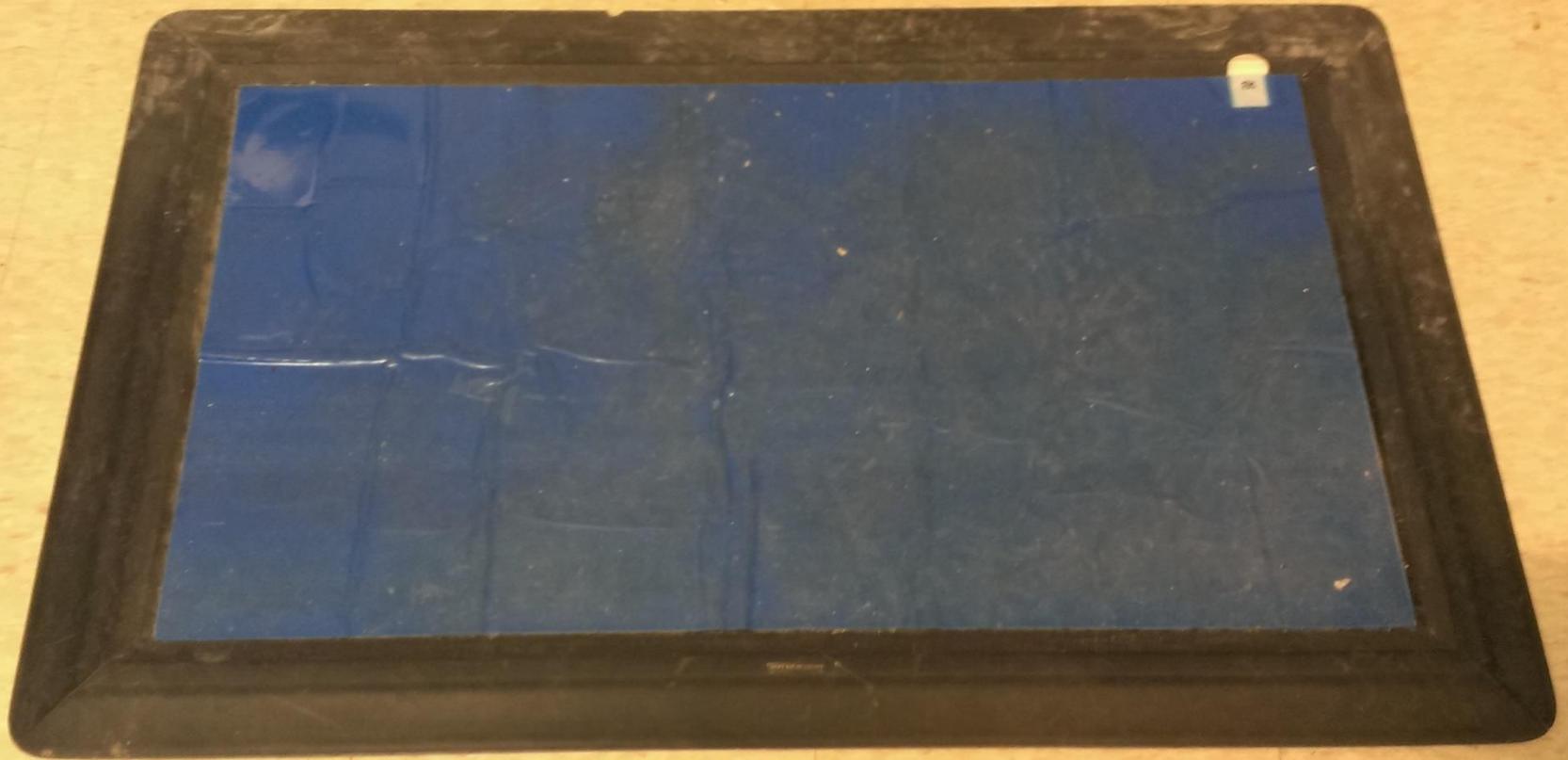
***You are the auditors***



# General Observations



# Housekeeping



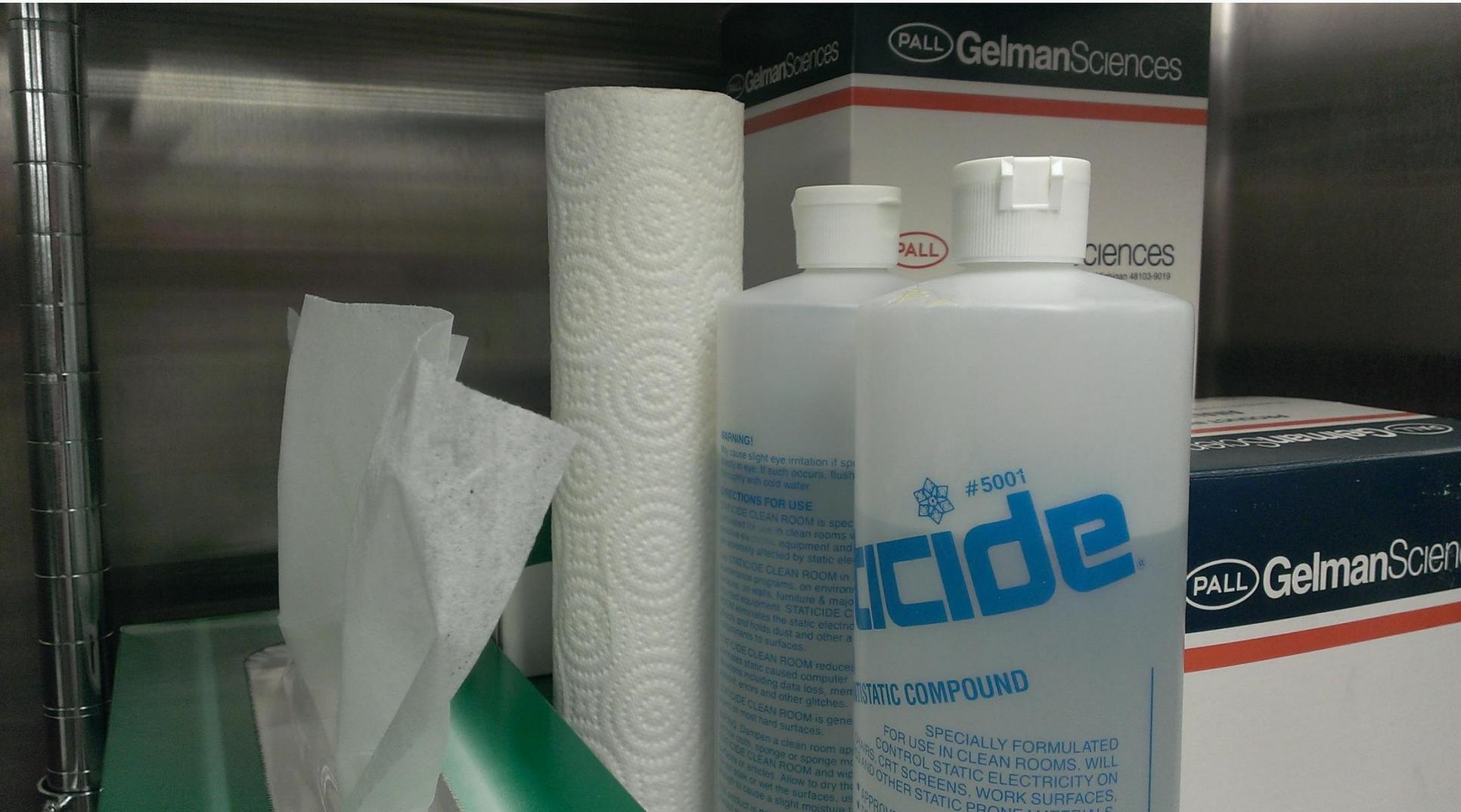
# Housekeeping



# Housekeeping



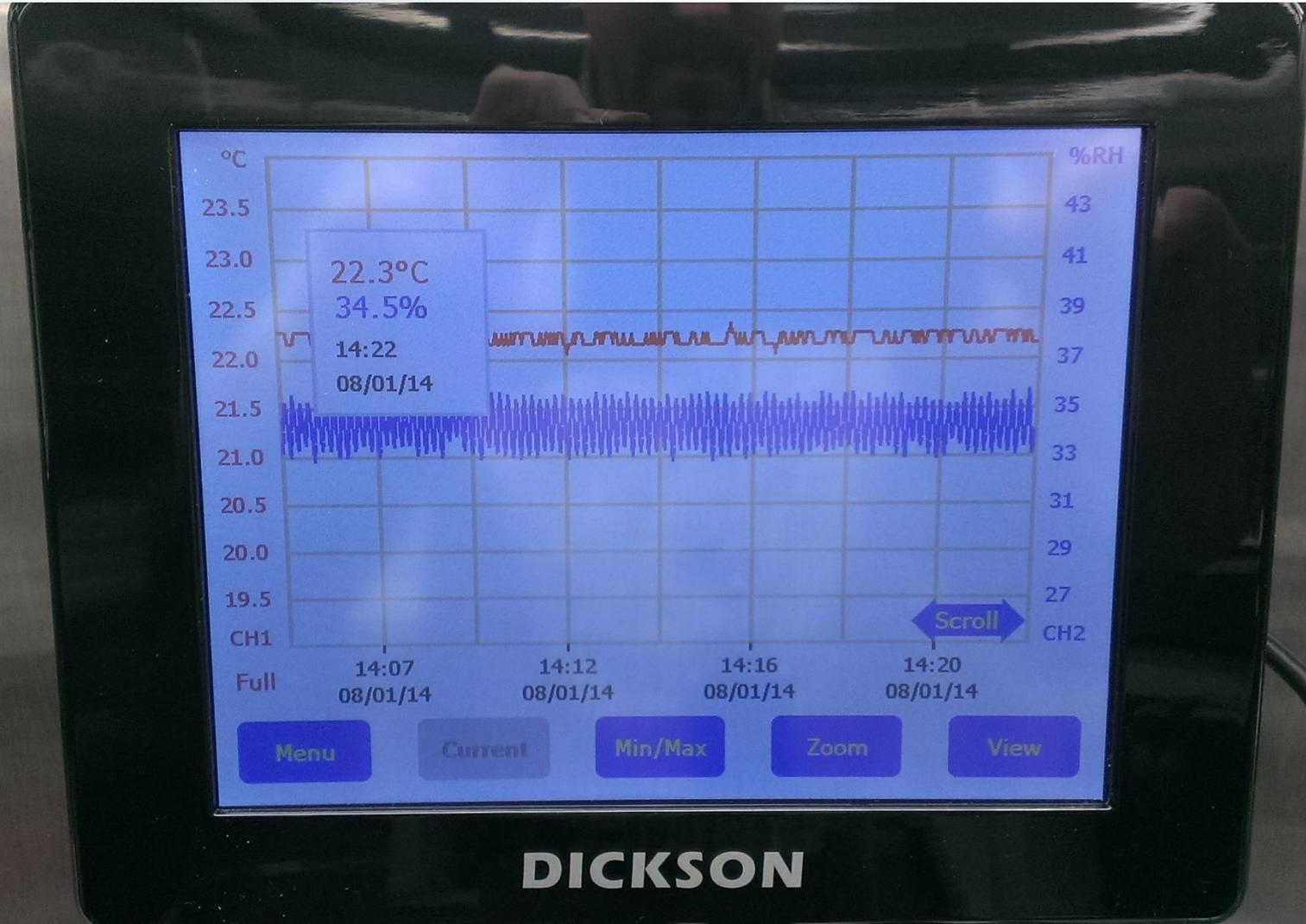
# Housekeeping



# Temperature and RH



# Temperature and RH



# Temperature and RH

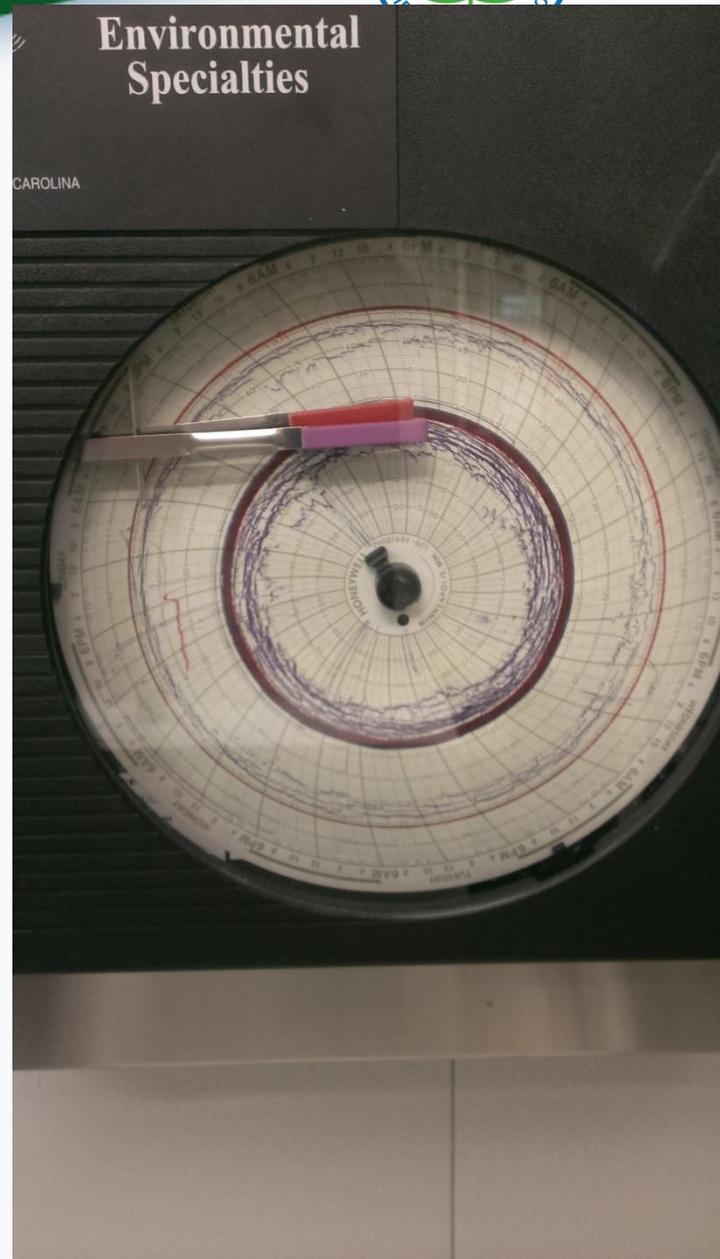


Graphing Complete

Table | Graph |

Date/Time	(FH625 )-Temp/°F	(FH625 )-Raw/None	Index
(1 sel) Minimum Value	72.1	350.0	
(1 sel) Average Value	72.1	350.0	
(1 sel) Maximum Value	72.1	350.0	
7/5/2014 5:18:32 PM	72.1	344.0	0
7/5/2014 5:20:56 PM	72.1	356.0	1
7/5/2014 5:23:20 PM	72.1	356.0	2
7/5/2014 5:25:44 PM	71.9	348.0	3
7/5/2014 5:28:08 PM	71.9	341.0	4
7/5/2014 5:30:32 PM	72.1	345.0	5
7/5/2014 5:32:56 PM	72.1	356.0	6
7/5/2014 5:35:20 PM	72.1	354.0	7
7/5/2014 5:37:44 PM	72.1	348.0	8

# Temperature and RH



# Temperature and RH



# Temperature and RH



# Static Control



**caution** Radioactive Material 500uCi Po210. Dangerous if solid material is ingested or inhaled. Do not touch strip under grid. Keep away from children. See instructions. Date manufactured: Model 2U500

NRD, LLC  
GRAND ISLAND, NY 14072

APR 2014



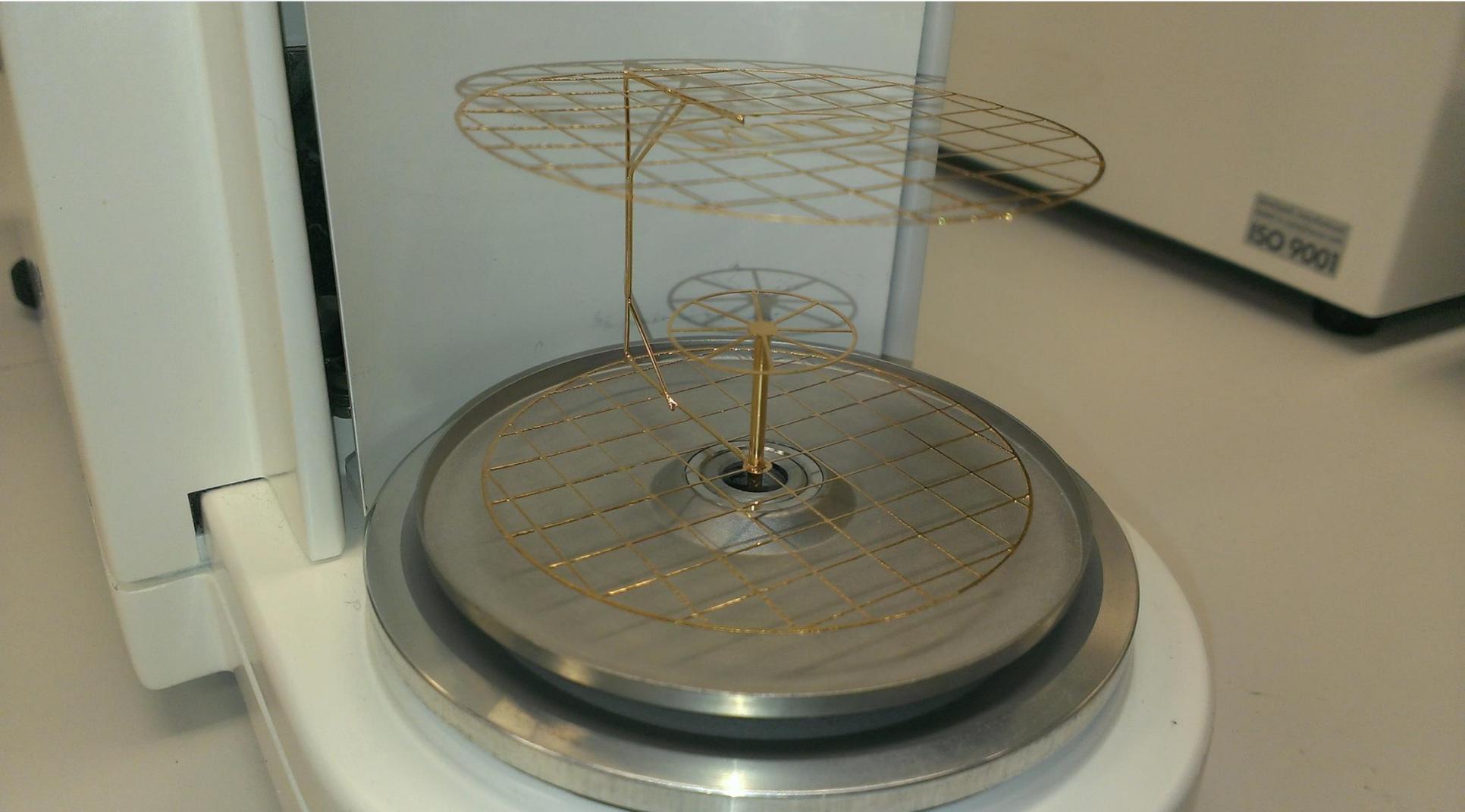
# Static Control



# Static Control



# Static Control



# Static Control



# Filter Weighing



# Filter Weighing



# Filter Weighing



# Filter Weighing



# Filter Weighing



NIST Calibration    Calibration/Repair:  
ID 80110585    **PRECISION**  
By GC    **WEIGHING INC.**  
1949 Evans Rd.  
Cary, NC 27513  
Cal Dt: 16 April    Ph. 919-678-0077  
Due: Apr 15    Fax 919-678-0075

U.S. EPA APPCD Calibration  
MetLab ID 03787  
Cal on 8/09/2011 by SAB  
PASS, See Report for Details

CF/    0    1    2    3    4    5    6    7    8

# *Filter Weighing*

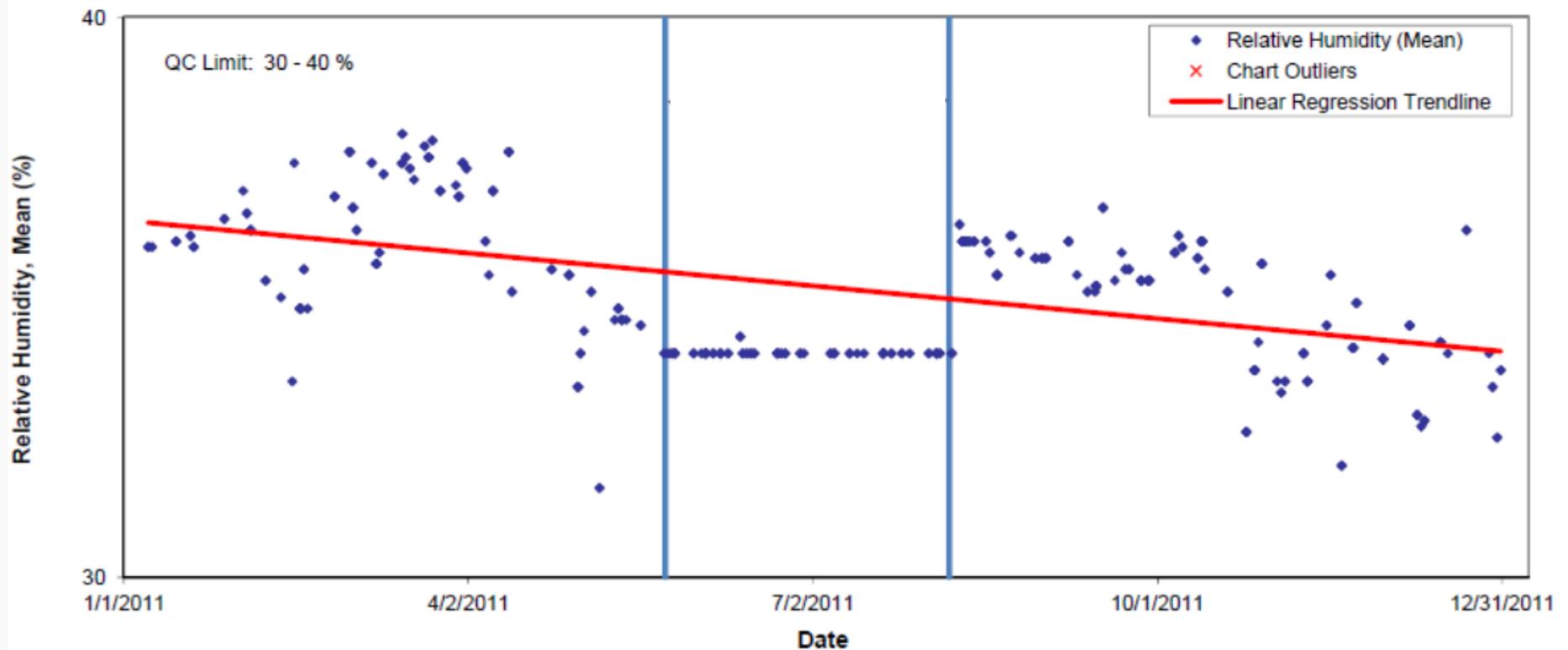


# Data Review



## %RH Control Chart

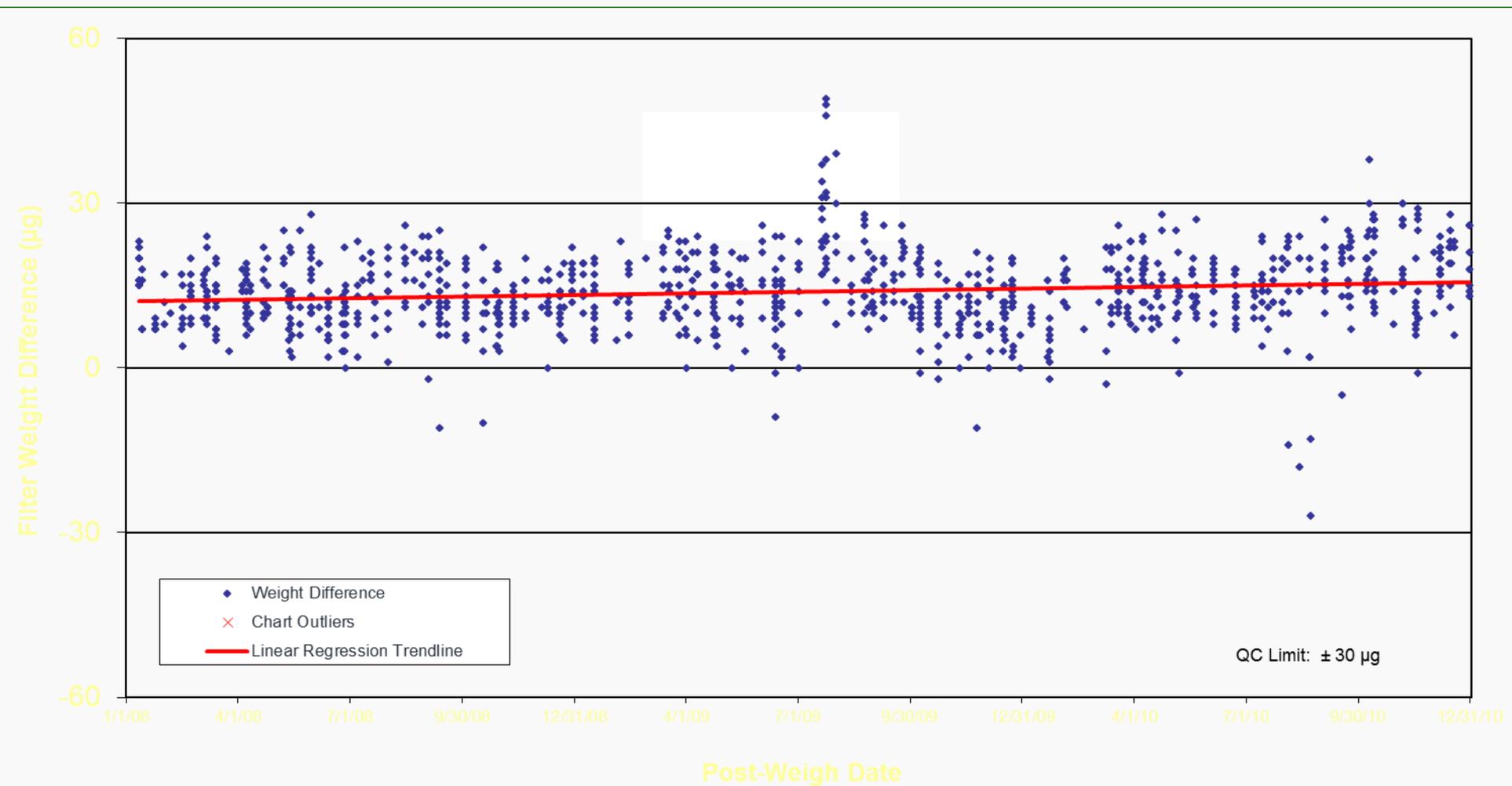
2011 Daily Relative Humidity (Mean) - Region 4



# Data Review



## Field Blank Control Chart

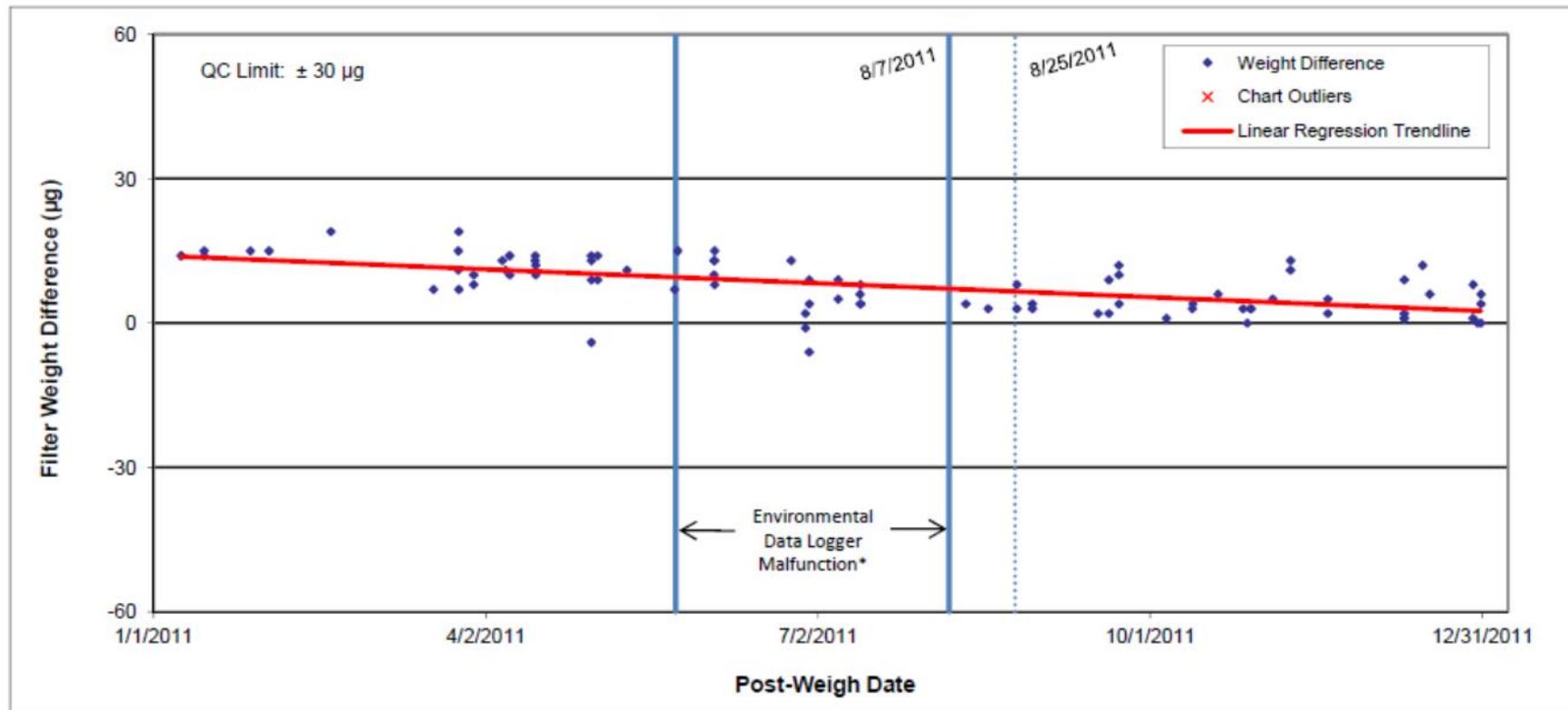


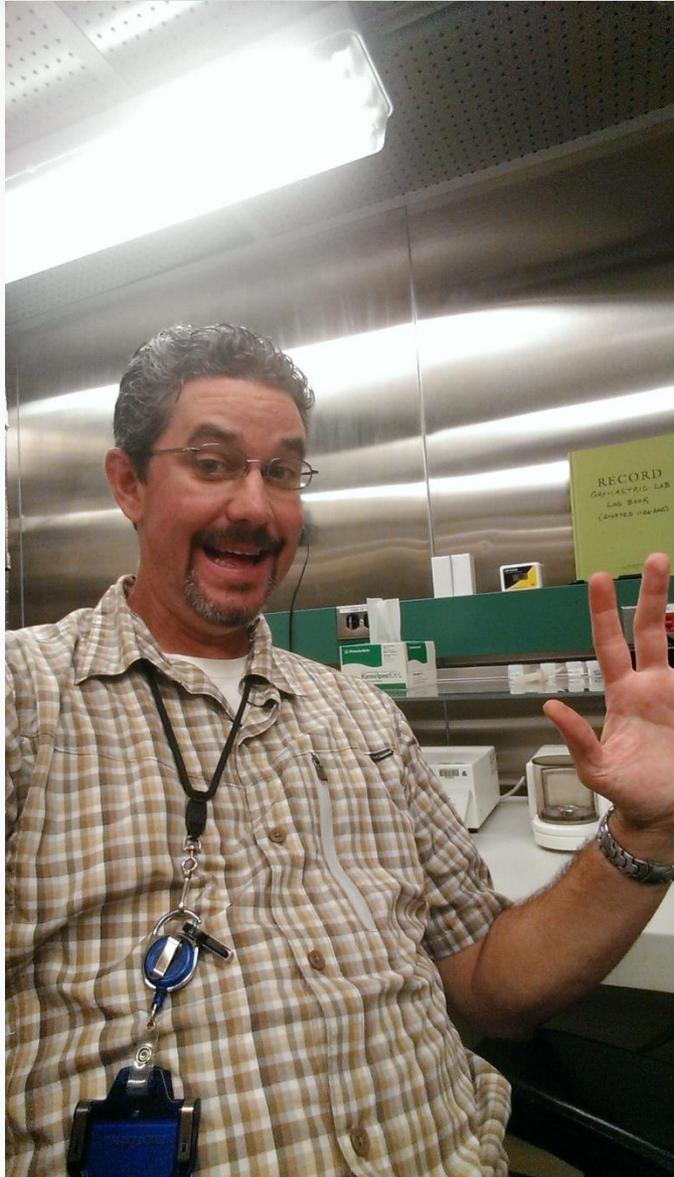
# Data Review



## Trip Blank Control Chart

2011 Trip Blanks - Region 4





# *Questions?*

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