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UCD IMPROVE Technical Information #251A

Reference Weights

Interagency Monitoring of Protected Visual Environments
Air Quality Research Center
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DOCUMENT HISTORY

Revision	Release Date	Initials	Section/s Modified	Brief Description of Modifications
	05/04/2020	GRM	All	Updated wording & formatting.
	03/04/2021	TAK	All	Updated balance QC and general procedures to include weighing in the MTL AH500 Autohandler. Updated wording for filter acceptance.
	04/12/2021	GRM	All	Updated wording to accommodate for secondary AH500 unit & move to Drew Ave.
	01/31/2022	SRS	All	Previously anthologized version separated into individual TIs.
	03/23/2022	TAK	All	Updated “calibration” terminology.
	4/15/2022	TAK	6.6	Added Inter-comparison Tests
3.1	2/09/2024	TAK	All	Minor corrections. Updated QC criteria: +/-.003 to +/-.004 for test weights, added 50mg test weight, and removed 400mg test weight from daily use. Manual equipment and intercomparison filters identified more-clearly. Updated balance prevision. Updated intercomparison procedure.

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1. PURPOSE AND APPLICABILITY

The purpose of this technical information (TI) is to describe how the microbalances and ultrabalances in the sample handling laboratory are adjusted, plus how reference weights are used to monitor balance measurements.

2. SUMMARY OF THE METHOD

Microbalances and ultrabalances in the sample handling laboratory are adjusted before routine weighing can begin. They are then tested every four hours thereafter as necessary. Relative humidity and temperature values are recorded with every measurement for automated weighing, and every five minutes on a terminal server. Lab blanks and sampled "test" filters are weighed once a week. Beginning 11/13/2023, the reported balance precision was increased to 0.0001 mg.

3. CAUTIONS

Be careful when handling external test weights, lab blanks, and sampled reference filters. Dropping and/or damaging these can change their weight measurements significantly, creating a discontinuity in the data. Report any drops or filter tears while weighing the lab blanks and sampled reference filters, as they do not have replacements.

4. PERSONNEL QUALIFICATIONS

Trained laboratory technicians are the only personnel who perform calibrations and external test weights.

5. EQUIPMENT AND SUPPLIES

- Mettler Toledo XP6 Microbalance
  - Used for manual weighing when chambers are not used
- Mettler Toledo XPR6UD5 Microbalances
- MTL Autohandler Chambers
- Computers with IMPROVE Filter Processing application and MTL Filter Weighing Software
- Metal test weights (50 mg, 100 mg, 200 mg, 400 mg)
  - 400mg only used for weekly intercomparisons
- Forceps with ceramic tips
- 4 Laboratory blanks (labeled by their Filter Code, the MTL Autohandler Chamber's FWS database identifier)
  - Only used for weekly intercomparisons

- 3 Sampled reference filters (labeled E, F, and G)
  - Only used for weekly intercomparisons

6. PROCEDURAL STEPS

6.1 Manual Weighing Mettler XP6 Balance Calibration Information

Manual weighing for routine operations is only performed when the automated weighing systems are both inoperable. When the Mettler XP6 microbalance is adjusted, it generates a 3-point adjustment equation using two internal masses. The weights of these internal masses combined are in the range of 5-6 grams. The balance will then perform a 2-point test of the adjustment equation, showing temperature (˚C), nominal percentage (always 100.00000% accuracy), reported accuracy (must be between 99.99998 and 100.00002%), and difference (must be 0.00002% or less).

Balance adjustment history can be found locally on the balance by navigating through "Setting > System > Adjust/Test > Test History-Define > Adjustment History-Show."

There are a variety of ProFACT adjustments available on the Mettler XP6 balance, but the only one activated on the manual-weighing balance in the sample handling laboratory is ProFACT Auto Zero. This allows the balance to automatically zero for minute deviations away from zero.

Static may cause the balance not to zero. In this case, wait for the non-zero weight to stabilize, zero the balance, tare the balance, and then check the External Test Weights using the procedures described in section 6.3.

There are guides on the pan for centering the filters and standards; make sure to use them. When the balances are calibrated by a Mettler technician, the internal calibration masses are compared against Mettler's own traceable external calibration weights. Essentially, the yearly calibration is an adjustment of the balance electronics.

6.2 Manual Weighing Balance Calibration Procedure

1) Perform adjustments prior to test weights and routine weighing. Check that the balance is level by making sure the bubble is within the circle on the top of the balance. If the balance is out of alignment, level the balance by adjusting the two barrel-adjusters in the back. Also check to be sure that the balance reads zero before beginning.
2) Zero the balance by pressing **→0←**, and then tare the balance by pressing **→T←**.
3) To begin, press the **Internal Adjustment** button on the bottom left of the balance screen. This takes several moments.
4) When the internal adjustment has finished, press **OK** to exit.
5) Press the **Internal Test** button. The internal test takes several moments.

6) Note the nominal value, actual value, and difference, which will all be displayed on the screen. If the difference is greater than ± 0.00002%, repeat the internal calibration and test.

7) If three repeated internal adjustments do not meet the criteria, exercise the balance by weighing a 2-5 grams mass on the balance several times. Then, repeat the internal adjustment.

8) If the balance will not pass internal test tolerances after performing the steps above, contact a Mettler technician.

6.3 Manual Weighing Stainless Steel External Test Weights

External test weights are weighed after balance adjustments. Open the Improve Filter Processing program. Navigate through Admin > Balance quality control > Weight reference filter. In the pop-up box, enter 3-letter user initials. Confirm that "Pick type" is set to "Metal test weight". Under "Pick weight", select **100#**, where # identifies the test weight.

1) Start the balance by pressing **START SCALE (F5)** at the top. Wait several seconds and the program will display "Scale zeroed". Put weight in scale.

2) Keeping the wrist maintained in the C-shaped prongs of the ionizing zone, pass the 100 mg weight through the ionizing unit and place it on the balance to wait for a stable reading. Confirm that the head-unit display's characters are in black, that the number matches the application reading with a green background, and that it also matches the suggested weight in the text to the right. If all three numbers do not match, press **R** to refresh. When the application suggests the correct stable weight, press **A** to accept.

3) Confirm the entries for Initials, Time, and Recorded weight. Record relevant notes in the text box, then press **WEIGH ANOTHER (F8)**.

4) Remove the test weight.

5) Confirm that "Pick type" is set to "Metal test weight" and repeat steps 2-3 for the 50# test weight under "Pick weight".

6) Confirm the entries for Initials, Time, and Recorded weight. Record relevant notes in the text box, then press **DONE (F9)**.

7) A history of the most recent measurements is displayed on the lab app screen on the left. This lists the date and time of the measurement, along with the measured value, and balance source. Additional information can be found in Section 7: Data and Records Management.

8) If one external test weight weighs 0.003 mg or more outside of the expected average (Nominal mass), reweigh that mass. If one external test mass continues to weigh ± 0.003 mg or more different than the average, test it on another balance to see if it agrees.

9) Through these external tests, consistency between balances can be tracked. Each mass should weigh within 0.003 mg when compared between balances. If this test fails, reweigh that mass on each balance. If two balances agree that one external test weight is out of range, consider sending that weight back for recertification and replace it with a different weight. If one or more of the masses still disagree, this indicates straying balance equations. Note historical averages for each mass for each balance, and internally adjust the balance that shows the most inconsistency or drifting average. If repeated internal adjustment of one balance does not fix the problem, attempt internal adjustment on the other balance.

10) If repeated internal adjustments fail and the balance equations do not match, consider contacting a Mettler technician. If both balance equations disagree, most likely both metal test weights will not weigh consistently between balances. Monitoring of the 50 mg and 100 mg weights (which are in the range of PTFE filter weights) is most critical.

11) Each 50 mg and 100 mg mass is recertified yearly to compare balance equations to traceable standards.

6.4 Automated Weighing Balance Adjustment Procedure

Balance adjustments and the weighing of stainless-steel external test weights are completed through the MTL Filter Weighing Software (further referenced as FWS). All stainless-steel external test weights are stored in the MTL chamber's garage positions underneath the silos (the bottom two positions of every column where filters are loaded). There are currently 4 silos and 3 garage positions each, however the top garage spot of each silo has a cover plate and cannot be used. Balances are adjusted before any filters are weighed. Stainless steel external test weights are weighed immediately after that, and every four hours that the MTL chamber is in use. For reference, the first MTL Autohandler is referenced to as AH1 or Luna and the newer one is referenced to as AH2 or Sol. Both units are AH500E models.

ProFACT Auto Zero is disabled for the MTL Autohandler Chamber's balance. Balance drift is addressed by drift correction adjustments applied to reported measurements; balance drift, measured by difference between recorded zero measurements before and after the direct read of a filter or test weight, is subtracted from the direct read.

1) Open the MTL Filter Weighing Software. Navigate to Automated Filter Weighing and select an Operator from the dropdown.

2) In the upper right section of the next screen, select a Procedure.

    a. For routine weighing on a weekday evening, select **Routine Weighing**. A balance adjustment will be performed with external test weights weighed immediately, plus every four hours while the MTL chamber is in use.

    b. For routine weighing over the weekend, select **Weekend Weighing**. The X symbolizes the two different Autohandler Chamber names in the Filter

Weighing Software. A balance adjustment will be performed with external test weights weighed immediately, plus every four hours while the MTL chamber is in use.

6.5 QC Checks: Metal Reference Filters and Environmental Conditions

Filters weighed in the MTL Autohandler Chamber are weighed over an extended period of time, so the QC must be reviewed before any of those filters are processed.

1) Open the Daily Balance QC Review page (location noted in Section 7: Data and Records Management).
2) Begin by selecting the MTL system to review in the dropdown; the default instrument is Luna. Then identify the active test weights that are in use; these with have either a green "OK" button or a red button with a number. Select the active 50mg test weight. Identify the following:
    a. Solid gray line: This marks the Nominal mass, the ideal weight as determined by the certified mass on the most recent certificate.
    b. Dotted lines above and below the Nominal mass line: These mark ± 0.004 mg, the uncertainties of our balance.
    c. Solid blue line: This is a trend line for that time period.
    d. Blue dots: These are all QC measurements for that 50 mg Metal Reference weight that are within acceptable ranges, ± 0.004 mg of the Nominal Mass.
    e. Red "X": These are all QC measurements for the 50 mg Metal Reference weight that are not within acceptable ranges.
    f. "Last Approved" green vertical line: On the QC measurement timeline, this shows when the last QC review was performed, and all measurements were accepted.
3) Review all measurements to the right of the Last Approved line. If any measurements are plotted as an "X", note the date and time for that measurement.
4) Click on the rectangular button to the right of "100 mg" and "200 mg" to review the active 100 mg, and 200 mg weights in the same manner. For each Metal Reference weight, if that button to the left says "OK" and is green, all values for the entire 2-week period have been reviewed and cleared if they exceeded the ± 0.004 acceptance criteria. If that button is red, the number value notes how many points for that same time period have exceeded the ± 0.004 and have not been cleared. Metal Reference Weights are weighed at the beginning and after every four hours that the MTL Autohandler Chamber is operational. For QC to pass, no more than 1 Metal Reference weight measurement may exceed acceptance criteria for each time that the metal reference weights are measured. Reviewing and approving QC validates all measurements taken by the MTL Autohandler chamber and monitor the measurements for unexplained shifts in data or sloping trends. The date range for reviewing data can be modified as necessary. If two or more Metal Reference

Weights from that round exceed acceptance criteria, QC does not pass. If QC does not pass, the following steps must be taken.

a. QC must pass before filters are weighed again. Through the FWS, run the full "QC 3cCheck" procedure. To continue, no more than one round of QC can fail. If two or more rounds of QC fail, air brush the inside of the Mettler balance. Persistent instability or bias may require a Mettler Technician visit. Refer to "Flow Chart 1. Calibration Procedure" and "Flow Chart 2. External test" in *UCD SOP #251: Sample Handling*.

b. All network filters from the last known accepted QC, up until the next accepted QC, must be reweighed.

c. Information for the QC failure and the reweighing of network filters are recorded in the AQRC_Lab_Metadata report (location noted in Section 7: Data and Records Management).

5) If one of the Metal Reference weights consistently does not meet criteria, weigh it on the Manual-weighing balance, and consider sending it for recertification. Refer to "Flow Chart 1. Calibration Procedure" and "Flow Chart 2. External test" in *UCD SOP #251: Sample Handling*.

6) Review temperatures and RH plots in the same manner, according to the guidelines of the Nominal and acceptance limit lines on the plot. Environmental measurements for QC appear as blue circles; these are reviewed for QC purposes. Any measurement appearing as a light blue "x" is for a network filter measurement and is plotted for informational purposes. Barring any unusual circumstances, the Temperature and RH must pass the same criteria as the Metal Reference Weights. If any round of environmental measurements fails QC, the same actions as the Metal Reference weights must be taken. The uncertainty for RH is ± 2% from 39%. The uncertainty for Temperature is ± 1.5 ℃ from 21.5 ℃.

7) If the Metal Reference weights and environmental conditions pass QC, or after all reweighed filters pass QC, click on the "Approved" button on the upper right of the screen. In the following pop-up, enter user initials, clear all applicable invalid data, and input comments, as necessary. Past approvals can be reviewed by pressing the download button on the right.

8) In the upper left-hand corner, select the MTL Autohandler Chamber that has not been reviewed yet and follow procedure 6.5 to review the QC weights.

6.6 Blank and Sampled Reference Filters

Blank and sampled reference filters are weighed once a week on each MTL system. These filters serve as a reference for general filter and balance consistency and are not a component of QC. Note that this procedure will take four to six hours in total for each instrument. Preparations will need to be made the previous night to allow for filter equilibration. Review the procedure in full to confirm that there is enough instrument time on each MTL system to complete the weighing.

1) Retrieve the inter-comparison test filters stored in the Sample Handling Lab. These are stored in a sealed container and include: four lab blanks, three sampled reference filters, and four metal test weights. Filters are stored in their assigned carrier. Allow the filters to equilibrate in either MTL chamber overnight. Routine weighing can proceed while the inter-comparison test filters equilibrate.
2) The following morning, load all inter-comparison test filters into any Silo B through F on the selected MTL system. Run the "Weekly Inter-comparison" procedure through the software. No additional equilibration time is necessary. The MTL software will proceed with routine QC, then measure the inter-comparison filters. No other filters are to be weighed during this procedure.
3) After weighing has completed on the first MTL system, transfer the inter-comparison test filters to the other MTL system, loading any Silo B through F. Run the same "Weekly Inter-comparison" procedure through the software, but allow a 4-hour equilibration time before the procedure start time. After equilibration, the MTL software will proceed with routine QC, then measure the inter-comparison filters. No other filters are to be weighed during this procedure.
4) Upon the procedure's completion, move the inter-comparison test filters back to the storage container in the designated location. Return the petri dishes to the storage container and back to the storage location.
5) These measurements can be reviewed on the same Daily Balance QC Review page under the Intercomparison section. The plotted time period extends to the previous month but can be adjusted as necessary.

7. DATA AND RECORDS MANAGEMENT

The stainless-steel external test weights, blank reference filters, and sampled reference filter weights data is stored in SQL. Data can be reviewed on the Daily Balance QC Review page: https://shiny.aqrc.ucdavis.edu/balance_daily_ops/.

The AQRC_Lab_Metadata report records all internally-recorded and externally-reported equipment, lab utilities, procedure events and changes. The file is located here: U\QualityManagementSystem\Lab Metadata.

8. QUALITY ASSURANCE AND QUALITY CONTROL

Test weights and filters are monitored by laboratory staff to ensure that the balances are both accurate and precise in their measurements. If laboratory personnel notice a consistent, change outside of ± 0.004 mg in weight for a calibration standard, the standard is sent for recertification and an alternate calibration standard is introduced.