Gravimetric Analysis: Analytical Weight and Balance Calibration

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GRAVIMETRIC ANALYSIS: ANALYTICAL WEIGHT AND BALANCE CALIBRATION

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Gravimetric Analysis: Analytical Weight and Balance Calibration

1. SCOPE AND APPLICATION

1.1. This SOP addresses the basic requirements, procedures, and frequency for recalibration of standard weights and calibration checks of micro, analytical, and top-loading balances.

1.2. Traceability of the primary standard weights used to check the calibration of the analytical balances must be verified annually to the specifications established by NIST (see ASTM E617-97(2008)). Procedures to check working weight calibration are described for the typical mass reference standard range of 0.001 mg-to-200 mg for the microbalance, 1 mg-to-250 g for the analytical balance and 0.10 g-to-4.0 Kg for the top-loading balance. These ranges are approximate, but represent the typical capabilities of the instruments available within the laboratory.

1.3. Refer to the following Standard Operating Procedures (SOPs) for additional gravimetric procedural information:

1.3.1. Determination *of $PM_{2.5}$ in Ambient Air by Gravimetric Analysis* (SOP **PM-001**)

1.3.2. *Determination of % Dry Solids by Gravimetric Analysis* (SOP **MT-055).**

2. ANALYTICAL WEIGHT REQUIREMENTS AND HANDLING INSTRUCTIONS

2.1. Primary standard weights and working weights used within the laboratory must meet the tolerances for their Class as established in ASTM E617-97 and must be calibrated to specifications provided by NIST. Only Class weights 0, 1, 1.1, 2, or equivalent are used within the laboratory.

2.2 The NIST traceability for each set of working weights must be renewed annually against a set of primary standard weights with equal or better tolerance specifications. The primary standard weights must have a current Certificate of Traceability

2.3. The annual check of working calibration weights must be performed on a balance with readability equal to or better than the other balances within the laboratory subsection. The current limit of readability for balances in the laboratory is typically 0.1 mg on the analytical balances and 0.001 mg on the microbalances.

2.4. It is recommended that all weights be constructed of nonmagnetic stainless steel. This type of construction insures a more stable mass value over time. Aluminum or brass weights should be avoided or replaced if possible since these materials are more prone to wear and corrosion.

2.5. Working calibration weights must be visually inspected for damage, corrosion or contamination prior to every use. Damaged or corroded weights must not be used. Consult your supervisor on replacing or cleaning contamination from calibration weights.

2.6. Handle all weights with dedicated nonmetallic forceps. Each balance must have a set of available forceps, preferably stored with the weights. Never pick up the weights with your fingers or with metal forceps or tongs. Insure that the analytical balance is clean before placing the weights on the weighing pan. Larger weights may be handled with antistatic, powder-free gloves instead of forceps.

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2.7. The use of antistatic, powder-free gloves are highly recommended, even when handling the weights with forceps. The gloves are required for microbalance measurements.

2.8. Never place the weights on a dusty, dirty or wet surface.

2.9. Always store all weights in their original protective case and place the weights in a dry, cool location. Do not store the weights near chemicals or solutions.

2.10. Working weights should be stored near the Instrument and Calibration Log(s).

3. BALANCE REQUIREMENTS

3.1. Each balance must be identified by a balance number or serial number. This balance ID will be used to identify the balance in sample preparation notebooks where necessary and in the balance calibration log.

3.2. Each balance must be accompanied by a calibration and maintenance logbook. The logbook must contain a full description of the balance, laboratory ID number, instrument acquisition date, logbook start and end date (when closed), balance location, and acceptance criteria limits. Ideally the logbook should be paginated and bound.

3.3. All balances should be professionally inspected, cleaned and calibrated on an annual basis or more often if necessary. Each balance is to be maintained according to the manufacture's recommendations. The analyst should not attempt to repair the balance.

3.4. All balances must be placed on a surface free of excessive vibration, air movement, and temperature and humidity swings. The balance must also be placed away from chemical storage, solvent bottles and potentially corrosive environments. The preferred location is a climate-controlled, draft-free space or room with minimal human traffic. A balance should not be placed in a chemical hood unless there is a compelling reason to do so. The environmental specifications for the microbalances may be found in EPA Quality Assurance Guidance Document 2.12.

3.5. It is recommended that the maximum load limit for each balance be clearly identified in order to avoid damaging the balance from sample overload.

4. ANNUAL STANDARD WEIGHT CALIBRATION CHECK

4.1. The annual calibration check of working weights should be performed with close supervision and consultation with the group supervisor.

4.2. A suitable balance must be identified for the calibration check. The readability of the balance must be equal or better than the application where the weights will be used. Indicate the balance identification on the Annual Weight Calibration Form.

4.3. Visually inspect the working calibration weights carefully. Inform the supervisor if any weight is worn, damaged, corroded or contaminated. Aluminum and brass weights should be replaced if possible. Make note of the condition of the working weights on the annual weight calibration form. Record the identification and Class of the working weights.

4.4. Inspect the Certificate of Traceability for the primary standard weights to make sure they are current. Record the certification number and Class of the primary standard weights in

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the calibration form. The Class of the primary standard weights must be equal to or better than the working calibration weights.

4.5. If necessary, measure and record the starting temperature and humidity of the weighing environment using a NIST traceable unit.

4.6. Tare the balance before starting and ensure the reading is stable for at least 15 seconds.

4.7. Pick up the weights with dedicated nonmetallic forceps. Wear antistatic, powder-free gloves when handling the weights.

4.8. Smaller masses may require static charge removal to obtain a stable reading. The static charge is removed by placing the weight near a polonium strip (NRD StaticMaster Ionizing Unit, Model No. 2U500) for 1 minute prior to taking the measurement or passing the item through an electronic anti-static U-electrode (Mettler Toledo).

4.9. Weigh each primary standard and working weight at least twice, allowing the reading to stabilize before recording the value. Record the weight values in the calibration form. The balance must read zero before and after each weight is placed on the weighing pan.

4.10. Working weights must not deviate more than the tolerances provided in Table 1 from the NIST certified weights. If the gravimetric method being used has more stringent weight requirements, then the method requirements supersede the criteria described here. Report any weight calibration check failures to your supervisor. It may not be possible to weigh all of the smaller masses accurately on some balances within the laboratory. Therefore, it may be necessary to use a microbalance for the calibration check of the smaller weights. The weights that fail to pass the calibration check should be rechecked on a second balance with better readability. If the calibration check fails again, then the failing weights must be sent out for recertification or discarded.

4.11. Return the weights to their protective box when done.

4.12. If necessary, measure and record the ending temperature and humidity of the weighing environment using a NIST traceable unit.

4.13. Sign and date the calibration form.

5. GENERAL BALANCE OPERATION AND CALIBRATION INSTRUCTIONS

5.1. Analytical and microbalances are delicate instruments and must be used with care. Visually inspect the balance before using it to insure that the balance is clean and in working order.

5.2. Check to make sure that the balance is level before use. Most balances are equipped with a bubble window to check the level of the balance.

5.3. Visually inspect the weighing pan and surrounding surfaces for contamination, wear or damage. Report any problems to your supervisor.

5.4. Inspect the balance area for possible interferences such as vibrations, air drafts, human traffic, electrostatic sources, excessive dust, papers, or other materials near the balance. Report any problems to your supervisor.

5.5. Tare the balance and insure that the balance zero is stable for at least 15 seconds. There may be a problem with vibrations, air drafts, moisture or with the balance itself if too

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much time is needed for stabilization. Report any problems to your supervisor. The tolerances or allowable deviations from the nominal weight for different weights used on laboratory analytical balances are listed in **Table 1** below. Tolerances for microbalance weights are described in SOP PM-001, *Determination of Particulate Metter ($PM_{2.5}$) in Ambient Air by Gravimetric Analysis*.

Table 1: Allowable Working Weigh Tolerances

Weight (g)	Tolerance (g)	Multiple	Tolerance (% of weight)
4000	4	1000	0.100%
2000	2	1000	0.100%
300	0.3	1000	0.100%
100	0.1	1000	0.100%
50	0.05	1000	0.100%
20	0.02	1000	0.100%
20	0.02	1000	0.100%
10	0.01	1000	0.100%
5.0	0.005	1000	0.100%
2.0	0.002	1000	0.100%
2.0	0.002	1000	0.100%
1.0	0.001	1000	0.100%
0.50	0.005	100	1.000%
0.20	0.002	100	1.000%
0.20	0.002	100	1.000%
0.10	0.002	50	2.000%
0.050	0.001	50	2.000%
0.020	0.0004	50	2.000%
0.020	0.0004	50	2.000%
0.010	0.0002	50	2.000%
0.0050	0.0002	25	4.000%
0.0020	0.0001	20	5.000%
0.0010	0.0001	10	10.000%

5.6. Samples to be weighed will require environmental conditioning if they were just removed from a freezer, oven or came from outside the laboratory. Samples must be allowed to equilibrate to the ambient weighing environment prior to making weight measurements. For microbalance measurements an equilibration time of 24 hours or more may be required depending on the application.

5.7. Check the certification date on the calibration weights. (The individual weight corrections must always accompany the weight set.)

5.8. The daily balance calibration check must bracket the entire weight range used, including the mass of a weighing boat or other container that may be tarred on the balance. The top-loading balance can be calibrated with the weighing boat or container already tarred on the balance. However, the analytical and microbalances must be calibrated with

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individual standards, taking into account the weight of any containers used in weighing samples.

5.9. Use tweezers or antistatic, powder-free gloves to pick up calibration weights that bracket the expected sample weight. Place the weights on the weighing pan and wait at least 15 seconds for the balance to stabilize. Record the weight values in the appropriate logbook. Note that each analytical balance has its own logbook, identified by a model and serial number.

5.10. Record the date and your initials. Make sure your writing is neat and legible. Mark through any corrections with one line, and then write an explanation, date, and your initials.

5.11. Notify your supervisor immediately if any of the calibration weights differ by more than required criteria from the certified value. The acceptance limits should be documented in the balance logbook.

5.12. Immediately clean up any chemical spills.

5.13. Keep the balance and weighing area clean and tidy. Always return the calibration weights to their storage box.

6. INTERFERENCES

6.1. High humidity and temperature in the weighing environment will interfere with the weight measurement unless tightly controlled. If possible place the balance in an environmentally controlled room.

6.2. Static charge build-up on the sample, weighing pan, gloves, table, and other items can cause balance weight errors. Reduce static charge by grounding equipment, using antistatic solution and using Polonium strips to remove static charge.

6.3. Air movement caused by air condition drafts or traffic areas may result in balance instability. Be aware of air drafts and add draft shields if necessary. Move the balance to an area with low traffic.

6.4. Vibration or movement in the balance environment may results in balance instability. Move the balance to a vibration-free environment.

6.5. The balance must be level in order to work properly. Check to make sure the balance in on a level surface.

6.6. Excessive dust in the environment may result in weighing errors. Insure the balance is placed in a clean, dust-free location.

7. REFERENCES

7.1. ASTM. E617-97(2008)). Standard specification for laboratory weights and precision mass standards. American Society for Testing and Materials. 2008 Annual Book of ASTM Standards, Designation E 617-97.

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7.2. EPA. 1998. Quality Assurance Handbook, Vol II, Part II, "Quality Assuance Guidance Document 2.12 : Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods".

7.3. Harris G. 1993. Ensuring accuracy and traceability of weighing instruments. ASTM Standardization News **21**(4) 44-51.

7.4. Kupper WE. High accuracy mass measurements, from micrograms to tons. Instrument Society of American Transactions **29**(4): 11-20.

7.5. Comprehensive Quality Assurance Plan, Chemistry Section, Bureau of Labs, DEP, 2015.

7.6. Florida Department of Environmental Protection, Standard Operating Procedure **PM-001** *Determination of $PM_{2.5}$ in Ambient Air by Gravimetric Analysis.*

7.7. Florida Department of Environmental Protection, Standard Operating Procedure **MT-055** *Determination of % Dry Solids by Gravimetric Analysis.*

8.0 SOP UPDATE REVISION SUMMARY

The following revisions were made to this SOP from the prior version:

- Incremented revision number and date
- Added table of weight tolerances.
- Updated references.

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