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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 working calibration weights and calibration checks of analytical and top-loading balances.

1.2. Primary standard weights are purchased with a Certificate of Traceability to NIST standards. These weights are used to verify the calibration of working calibration weights annually.

1.3. An outside weighing service provider annually calibrates all balances.

1.4. Working calibration weights, bracketing the range of use, are used to check the balance calibration on each day a balance is used.

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

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

1.5.2. *Calibration of Auto-Pipettes and Dispensers (SOP CM-032-1.0)*

2. ANALYTICAL WEIGHT REQUIREMENTS AND HANDLING INSTRUCTIONS

2.1. Primary standard weights and working calibration 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 calibration 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 at the time of use.

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.

2.4. It is recommended that all weights be constructed of nonmagnetic stainless steel. This type of construction ensures 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.

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Consult your supervisor on replacing or cleaning contamination from calibration weights.

2.6. All weights **must** be handled 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. Ensure that the to-loading or 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.

2.7. The use of antistatic, powder-free gloves is highly recommended, even when handling the weights with forceps.

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 and Maintenance Log(s).

3. BALANCE REQUIREMENTS

3.1. Each balance must be identified by a unique ID 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. To create a new logbook, use the latest version of form CM-BAL-ID. Obtain a unique ID for the notebook in the Laboratory Information Management System's (LIMS) Document Control System module. Fill in the details of the balance and establish the acceptance weighing range as appropriate for the specific balance.

3.3. All balances are professionally inspected, cleaned, and calibrated on an annual basis using an outside weighing service provider. 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.

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

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4. ANNUAL WORKING WEIGHT CALIBRATION CHECK

4.1. The annual calibration check of working calibration weights against the primary calibration standards 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.

4.3. Locate the Balance Weights Calibration Log in the Weights Cabinet in room A311. It is a compilation of form WEIGHTS-CALIB-1.3. Complete the header part of the next available empty form in the logbook.

4.4. 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.

4.5. Inspect the Certificate of Traceability for the primary (NIST-traceable) Class 0 standard weights to make sure they are current.

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. Static removal devices may be used for this purpose.

4.9. Weigh each primary standard and working weight, 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. Calculate the difference between the standard and working weights as instructed on the form. Determine if a working calibration weight is within specification by comparing the weight difference (Wd) to the tolerance (Wt). If the difference is smaller or equal to the tolerance the working weight is in calibration. Note this as Y or N in the appropriate column in the form. Initial your entries. In addition to the form the tolerances or allowable deviations from the nominal weight for different working calibration weights used on laboratory analytical balances are listed in **Table 1** below.

4.11. If the gravimetric method being used has more stringent weight requirements, then the method requirements supersede the criteria described here.

4.12. Report any weight calibration check failures to your supervisor. It may not be possible to weigh the smaller masses accurately on some balances within the laboratory. The weights that fail to pass the calibration check should be rechecked

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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.

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

Table 1: Allowable Working Weight 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. GENERAL BALANCE OPERATION AND CALIBRATION CHECK INSTRUCTIONS

5.1. Analytical balances are delicate instruments and must be used with care. Visually inspect the balance before using it to ensure 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. The balance is level when the bubble is centered in the bubble window.

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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. All balances are subject to **daily calibration checks** before being used to weigh substances. The daily calibration check results are recorded in the Balance Calibration Logbook of each balance. Tare the balance and ensure that the balance zero is stable for at least 15 seconds. There may be a problem with vibrations, air drafts, level of the balance, moisture or with the balance itself if too much time is needed for stabilization. Report any problems to your supervisor. The tolerances or allowable deviations from the nominal weight for different working calibration weights used on laboratory balances during the daily balance calibration checks are listed in the Balance Calibration and Maintenance Logbook of each weighing balance.

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.

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 calibration of the analytical balances must be checked with individual standards, accounting for 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.

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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 or employing an electronic antistatic ionizing device.

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 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. Ensure 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.

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

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

7.4. Comprehensive Quality Assurance Plan, Chemistry Section, Bureau of Labs, DEP, 2018.

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

7.6. Florida Department of Environmental Protection, Standard Operating Procedure **CM-032** *Calibration of Auto-Pipettes and Dispensers.*

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8.0 SOP UPDATE REVISION SUMMARY

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

- Typo corrections.
- Updated references.

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