Title: Calibration and Use of Analytical Balances

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References: Current version USP <1251> Weighing on an Analytical Balance.
Current version USP <41> Weights and Balances

Rev. No.	Effective Date	Revision Summary
1.	09-06-91	Original SOP
2.	10-25-91	Weight for calibration of semimicro balance changed to 2 g
3.	09-30-93	New Format
4.	12-09-93	Added CLP Requirements
5.	12-14-94	Minor revisions, 5.2: re-certification of 1 and 50 g Class S weights
6.	12-17-97	"for 2 weeks" deleted from 5.1 and OOS investigation added
7.	07-08-98	5.1 The QAU responsible for both re-calibration of balances if results exceed control limits as well as coordination of balance inspections by an outside service. The results from weekly weighings are documented in the QA Logbook instead of on 3 X 5" cards, and personnel responsible for conducting weekly checks updated.
8.	12-06-99	Changed to current SOP format. 5.1: semi-micro checked with additional 10 or 20 mg weight. 5.1.1.2: added
9.	05-01-00	Sec 1.0, 2.0, 3.0: scope widened to include use of balances. Sec. 4.1, 5.0: added.
10.	02-21-02	Sec. 2.0: Changed balance check from weekly to daily, changed reference form Sec.5.2 to 5.0 and added reference to Sec. 6.0. Section 6.1: Added check with 50g weight remainder of week. Sec. 6.1.2: Changed file cabinet to office. Sec. 6.1.3: Changed weekly to daily. Added reference to current version USP.
11.	02-14-03	Changed weight class from "S" to "2" throughout per current USP <41>.

Rev. No.	Effective Date	Revision Summary
12.	09-03-09	Sec 4.0 added antistatic devices; Sec 5.1 requires max load, sensitivity, and min weight (for assay) to be posted on balances. Restated USP definitions for "about" and "accurately weighed"; Sec 5.2 emphasized clean up after weighing; Sec 5.6.1 add statement to record weight as soon as stable reading is obtained; Sec 5.9 requires weighing of hazardous materials into a closed tared container; Sec 5.12 requires re-weighing if spill occurs during transfer; Sec. 5.14 recommends use of gloves or forceps to transfer weighing containers for analytical balances; Sec. 5.16 allows use of antistatic gun in addition to antistatic brush to eliminate static charge; Entire Sec 6 was revised. 6.1 Balance Calibration, 6.2 Weight Calibration, 6.3 QA Balance Monitoring, Deleted reference to SOP 1000 (retired), Added 6.4 Records, Deleted Appendix 1, added Appendix A for minimum weight determination. Added worksheet for balance failure investigation.
13.	AUG 19 2010	Sec 6.1: Allows use of Exova tolerances for annual balance certification. 6.3.2.1 Requires minimum weight determination (for USP assay) to be performed annually. Sec. 6.3.6.4: Expanded on how to conduct failure investigation and allows use of internal weight calibration. Revised App. B Balance Investigation Form. Added Balance Use Log Template. 6.3.6.5 now requires use log for analytical balances.

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1.0 SCOPE AND APPLICATION

This SOP covers the operation, calibration, and calibration checks of electronic analytical balances used for sample and standard weighings in the laboratory.

2.0 SUMMARY OF METHOD

A variety of types of electronic balances are used to determine the weight of samples and reagents. Proper use of these balances is described (See Section 5.0). Each balance is checked daily against Class 2 weights and inspected annually by an outside ISO 17025 certified service provider. (See Section 6.0)

3.0 INTERFERENCES

3.1 Air drafts, spilled substances, vibrations, improper leveling, and temperature fluctuations should be eliminated prior to any weighings requiring precision greater than 0.1 g.

3.2 Static electricity can be reduced using an anti-static brush or an antistatic gun.

4.0 APPARATUS AND MATERIALS

4.1 Glassine weighing paper, weighing funnels, or weighing boats

4.2 Various calibrated weights, ANSI/ASTM E617 Class 0, 1, and 2

4.3 Electronic balances, various brands and models

4.4 Antistatic Brush, Staticmaster Model 3C500 or equivalent

4.5 Antistatic Gun, Milty Zerostat 3 or equivalent

5.0 USE OF BALANCES

5.1 Choose a balance that is appropriate for the range of the object to be weighed and the precision of a particular analysis. The maximum load and sensitivity are posted on each balance. Use the following guidelines to choose a balance:

- USP work that requires weighing "about," indicates a quantity within 10% of the specified amount.
- For USP assays that require a substance to be "accurately weighed", the weighing is to be performed on a balance whose measurement of uncertainty is not greater than 0.1% of the amount weighed. The minimum weight for USP assay as determined according to Section 6.3.2 is posted on each analytical balance.
- For limit and impurity testing, the term "accurately weighed" is not defined in the USP and should be specified by the appropriate SOP or USP monograph. For quantitative work, the measurement of uncertainty should be appropriate for the level of accuracy required by the product specifications. Typical uncertainty measurements for top loading balances (See Appendix C) are summarized below:

Balance Sensitivity	Weight (g)	Typical Measurement of Uncertainty
0.01 gram	0.50	~5%
0.01 gram	1.00	1 - 2%
0.01 gram	5.00	~0.5%
0.01 gram	10.00	0.1 - 0.2 %
0.01 gram	20.00	0.1%

5.2 Prior to use, the balance should be checked for spilled substances and to ensure that the balance is level, on a stable surface, and free from drafts or air currents. Spilled solids should be brushed away from the balance pan and properly disposed of to prevent other operators from coming in contact with the spilled material. The weighing table or bench area should be clean after weighing is complete.

5.3 The pan of the balance should be checked to ensure that it is properly seated on the pan mechanism.

5.4 Turn on the balance. If an error message is received instead of the tare value, label the balance "Out of Service" and notify the Quality Assurance Unit (QAU).

5.5 Press the tare button and wait until a stable zero point reading is obtained. Re-press the tare button if necessary.

5.6 Place the object to be weighed on the pan, in the center area.

5.6.1 Allow the reading to stabilize and record the weight. For analytical balances, close the balance door and record the weight as soon as the balance shows a stable reading.

5.6.2 If a container or weighing paper is to be used, press the tare button and wait until a stable zero point reading is obtained. Add the substance to be weighed, allow the reading to stabilize, and record the weight.

5.7 Allow samples/containers to reach room temperature before weighing. Hot samples will generate an upward convection of warm air that will cause an inaccurate reading.

5.8 Samples that are extremely hygroscopic or lose water rapidly must be weighed in a closed container.

5.9 Volatile liquids must be weighed in a closed container or with a trapping solvent. Nonviscous liquids may be handled with a Pasteur pipette equipped with a small rubber bulb. Corrosive, hazardous or unpleasant smelling compounds must be transferred into a closed tared container in the fume hood and then carried to the balance for weighing by difference.

5.10 "Lumps" are often caused by moisture or electrostatic charge and may not constitute a representative sample. Agitate or grind the sample in a clean dry mortar and pestle to create a homogeneous sample if possible. Otherwise note in the logbook or worksheet the appearance of the sample.

5.11 When weighing a sample or reagent, discard excess material that has been removed from the original container. Do not return material to its original container.

5.12 Do not use regular paper for weighing. Use glassine paper or weighing boats or tare an appropriate container. When using weighing paper or weighing funnels, great care must be used to prevent spills. If a spill occurs during transfer, then start the process over and reweigh the material.

5.13 Do not weigh containers containing a magnetic stir bar. The magnetic field generated may cause unpredictable effects on the electronic balance.

5.14 Fingerprints may cause an inaccurate value. For accurate weighing on an analytical balance, it is recommended that gloves, forceps, or another type of gripping device be used when handling weighing containers, because oils from the hands will add weight.

5.15 Be sure to use a clean and dry spatula or other transfer device to avoid contamination of the article to be weighed.

5.16 Some samples have a tendency to pick up static charge which will cause particles to fly around. Charged materials can result in unpredictable balance behavior and weighings. An antistatic brush or antistatic gun may be passed over the sample to be weighed to dissipate the static charge.

6.0 QUALITY ASSURANCE FOR WEIGHTS AND BALANCES

6.1 Balance Calibration

Balances are certified annually by an outside ISO 17025 certified service provider. Tolerances for the annual calibration can be set by Exova. Tolerances required for 5 and 6 place analytical balances for the annual balance certification must be NMT 0.002% of the weighed amount for weights that are 10-100% of the balance capacity. All tolerances must meet the requirement of NMT 0.1% error of the amount weighed. The QAU is responsible for scheduling the annual balance calibrations. All balances are listed on the master calibration schedule.

6.2 Weight Calibration

6.2.1 Weights used to check for drift are calibrated annually by an approved ISO 17025 certified service supplier according to the appropriate weight class. The QAU is responsible for scheduling the annual weight calibrations. Calibrated weights used for drift and accuracy checks are listed on the master calibration schedule.

6.2.2 When certain weights are out for calibration, other calibrated weights may be substituted for the drift checks. For example, when the 10 mg, 1 g and 50 g weights are out for re-certification, the 20 mg, 2g, and 20 g weights, respectively, will be substituted.

6.2.3 Traceability of weights to their current calibration certificates are maintained by placing a label on the weight storage container.

6.2.4 Calibrated weights are to be stored in the QA office.

6.3 QA Balance Monitoring

6.3.1 Balance List

The QAU creates monitoring plans for each individual balance. Each new balance is assigned a unique ID and added to the balance list located in Master Calibration Schedule. The list contains the following information for each balance: room location, maximum load, sensitivity, and the minimum weight for assay if applicable. The QAU is responsible for establishing weight tolerances and creating an appropriate form for QA monitoring of each individual balance.

6.3.2 Measurement Uncertainty and Minimum Sample Weight

6.3.2.1 The measurement of uncertainty and minimum sample weight for USP assay is determined annually (after the annual balance certification) and are posted on each analytical balance. Sample and standard weights used in a USP assay are not to exceed the minimum weight requirement.

6.3.2.2 To determine the measurement of uncertainty, 10 replicate weighings are obtained using the worksheet provided in Appendix A. The measurement of uncertainty is defined as 3 x the standard deviation of 10 replicate weighings divided by the amount weighed. The 10 replicate weights are repeated with progressively higher or lower weights until the lowest passing minimum weight has been determined. The measurement of uncertainty is acceptable if this value does not exceed 0.1% of the amount weighed for USP assays.

6.3.3 Responsibility for QA Balance Checks

6.3.3.1 Monitoring includes daily checks for drift (precision) and weekly calibration verification for accuracy. The ASTM Class of weights are chosen so that the tolerance of the weight does not exceed 0.1% of the amount weighed. If the appropriate weight class is not available, the actual value from the weight calibration certificate may be used to set the acceptance range for the balance check. The balance monitoring forms are compiled into a QA Balance Logbook.

6.3.3.2 The QAU designates the person responsible for conducting balance checks and recording the results in the QA logbook. Balance cleanliness and the balance level indicator (if applicable) are also checked each work day.

6.3.4 Accuracy Calibration Verification

At the beginning of each work week, balances are checked for calibration verification (accuracy) at a minimum at two levels in the range for which the balance is used. For check weights that are above the established minimum weight for USP assay, weight tolerances for accuracy are based on at least 0.1% of the amount weighed. Tolerances for top loading balances may be limited by balance sensitivity.

6.3.5 Drift Checks (Precision)

Balances are to be checked at the beginning of each work day for drift (precision) at one level within the range of operation with a Class 2 weight. The purpose of the drift check is to indicate whether mechanical or calibration problems have occurred. Drift tolerances are purposely tighter than tolerances for calibration verification.

Drift tolerance is defined as the average of weight obtained from 10 previous weighings ± the limits listed below.

Balance Sensitivity	Drift Tolerance Average Wt. (n=10) Limit (± g)
0.1	0.2
0.01	0.02
0.001	0.002
0.0001	0.001
0.00001	0.0002
0.000001	0.00002

6.3.6 Balance Check Failures

6.3.6.1 Failures during balance checks are to be noted in the back of the QA balance logbook in the Balance Maintenance Log. The balance is labeled as "Out of Service" and the QAU is notified.

6.3.6.2 The QAU will open an NCR investigation and consider the following possible causes:

- the balance has been turned off and is not yet equilibrated
- the balance door was open
- excessive air currents in the laboratory
- improper leveling
- nearby vibrations
- hysteresis of the mechanical parts

6.3.6.3 If the QAU is unable to remedy the situation, an outside service will be called to inspect the balance. The balance will be not be placed back in service until properly calibrated and re-qualified.

6.3.6.4 Appendix B is a balance investigation form to be used as a part of the NCR investigation. As found data is recorded by the investigator. If the balance is out of tolerance, an internal weight calibration may be performed. The balance is then re-qualified and the data is recorded on the investigation form. As part of the NCR investigation, potentially affected data is reviewed. A determination will be made on whether or not clients need to be notified following an impact assessment.

6.3.6.5 Analysts are responsible for recording the Job Number for any associated work performed on analytical balances. Standard preparation can be tracked through standard logbooks and does not need to be included. The purpose of the use log is to facilitate investigation of balance excursions.

6.3.7 Change Control for Balances

Any changes that will affect the performance of balance require submission of a change control form according to SOP 220. These changes may include moving a balance from one bench to another, moving the balance for cleaning purposes, or changing settings that will affect the performance such as repeatability settings.

6.4 Record Location - The working QA Logbook, Balance Measurement Uncertainty Worksheets, balance calibration records, and weight calibration records are stored in the QA office.

APPENDIX A

Balance Measurement Uncertainty Worksheet

Balance ID: _____ Analyst: _____

Date Tested: _____ Room Location: _____

Weight SN's: _____

Replicate	Test#1	Test#2	Test #3
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			
Weight (g):			
STD Dev:			
3XSTD Dev : Weight			
Pass/Fail			

Acceptance Criteria: NMT 0.001

Minimum Weight For Assay:_____

APPENDIX B

Balance Failure Investigation

Balance ID: _____ Room Location: _____ Date of Failure:_____

Type of failure:_____ Failing Result:_____ Tolerance:_____

Investigation Date: _____ Investigator Initials:_____ NCR#: _____

AS FOUND: Low Check: _____ Drift Check:_____ High Check: _____

AS LEFT

Replicate	Check Wt	
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		
		Pass / Fail
Avg Weight (g):		
STD Dev:		N/A
High Check Tolerance:		
Low Check Tolerance:		

Was the balance level?_____ Was the balance clean?_____
Were there excessive air currents or vibrations?_____
Comments:_____

_____

_____

Corrective Action:_____

_____

_____

APPENDIX C

Measurement Uncertainty Calculation for Top Loading Balances

Balance ID: B-28
Analyst: JLH
Date Tested: 10-2-09

	Test #1 5.00g	Test #2 10.00g	Test #3 1.00g
#1	5.00	10.00	1.00
#2	5.00	10.00	1.00
#3	4.99	10.01	0.99
#4	5.00	10.00	1.00
#5	5.01	10.01	1.00
#6	5.00	10.00	1.00
#7	4.99	10.00	1.00
#8	5.00	10.00	1.00
#9	5.00	10.00	1.00
#10	4.99	10.00	1.00
STD Dev:	0.006325	0.004216	0.003162
(3 x STD Dev)/Amt Wt:	0.0038	0.0013	0.0095

Balance ID: B-31
Analyst: JLH
Date Tested: 10-2-09

	Test #1 5.00g	Test #2 10.00g	Test #3 1.00g
#1	5.00	10.00	1.00
#2	5.00	9.99	1.01
#3	5.01	9.99	1.02
#4	4.99	10.00	1.01
#5	5.00	10.00	1.01
#6	4.99	10.00	1.01
#7	4.99	10.00	1.00
#8	5.00	9.99	1.01
#9	5.00	9.99	1.00
#10	5.00	10.00	1.00
STD Dev:	0.006325	0.005164	0.006749
Result (3 x STD Dev)/Amt Wt:	0.0038	0.0015	0.0202

Analytical Balance Use Log Book #: _____
Balance ID: _____

Initials/Date	Job Number	Analysis	Comments