

GMP 10

Good Measurement Practice for Understanding Factors Affecting Weighing Operations

Good laboratory techniques and practices, as well as good standards and balances, are required to obtain good mass measurements. Errors can arise from a variety of sources, such as balance drift, air currents, lack of thermal equilibrium, magnetic or electrostatic fields, and manipulative skills while using the balance. The magnitude and significance of these errors depends upon the laboratory environment, the balance, and the accuracy required for the measurement. Different methods of weighing are used to eliminate or reduce the effects of sources of error. Sources of error and ways to eliminate the errors are discussed with the weighing procedures. The accuracy required for a measurement and the criticality of the measurement often influence the choice of the weighing method used in the measurement. Regardless of the procedure used, several practices should be followed to promote good measurements.

1 Environment and Preparation

First, the balance should be installed in a laboratory having good temperature and humidity control. The requirements for temperature stability increase as more precision is needed in the measurement. For high precision measurement, the temperature should not vary by more than ± 1 °C in a 24 h period throughout the year and should vary by less than 0.5 °C during any one measurement series (typically less than 1 h). Automated systems will require tighter limits to environmental variations, possibly over a longer period. General laboratory measurements can probably withstand temperature variations of ± 2 °C per day. Varying temperatures result in balance drift and in unequal temperatures for the air, balance, and objects to be weighed. The relative humidity should be maintained between 40 % and 60 % which is the recommended limits provided by most balance manufacturers and by OIML R 111. If the relative humidity falls significantly below 40 %, electrostatic discharge may be generated both in and near the balance. This can result in erratic readings and make precision weighing impossible. If precision mass standards are cleaned, they should stabilize in the laboratory environment for at least seven to ten days.

Thermal and environmental equilibrium are critical for mass calibrations. Therefore, each mass SOP provides instruction that the environment must be stable and the weights set in or near the balance for 24 hours prior to a calibration. The test objects, balances, reference standards, and air should all be in thermal equilibrium. When possible, the objects to be weighed and the reference standards should be placed either in the balance chamber or adjacent to the balance, so all can come to the same thermal equilibrium. If there is a lack of thermal equilibrium, convection currents will be generated when an object is on the balance pan and an erroneous reading may result. These types of errors are likely to go unnoticed when check standards are handled the same way, so care must be taken to reduce the likelihood of their occurrence. Tests have shown that these types of errors depend upon the temperature differences among the objects and the balance, and on the geometry of the objects being weighed. On 20 g standards of greatly differing geometry, studies have shown that errors as large as 0.2 mg can occur for a 4 °C temperature difference among the standards and the balance. Errors as large as 3 mg have also been observed at 1 kg loads when

standards were stored in a cabinet and unknown test items near the balance where a temperature gradient of 2 °C was present (when the expanded uncertainty was less than 0.1 mg). When weights are delivered to the laboratory, care must be taken to ensure adequate environmental equilibration, especially when outside conditions deviate from those in the laboratory by more than 10 °C. The presence of frost, ice, or condensation on the weights is a common occurrence and may not be readily visible.

The balance must be installed in an area free from air currents. Air flow should be less than 2 m/s in mass calibration areas. Balances should be installed away from heating/cooling vents. It is not adequate to merely close vents when weighing because disrupting the temperature control system may result in temperature changes near the balances that exceed limits specified in the procedures and/or documentary standards.

2 Handling Weights

The masses of standard weights or objects weighed can be affected significantly by the way they are handled. Human contact can leave grease or oily films that affect the mass at the time of measurement and can even cause permanent damage due to corrosion.

Small weights should never be touched by hand, but handled using forceps, clean gloves, or swatches of cloth. In the latter case, the cloth must be lint free. Instructions for cleaning weights and for removing adhering foreign material are described in GMP 5.

Large weights of lower tolerance classes (NIST Class F) may be handled by bare hands. Large weights are a source of special problems. Fork lifts, portable cranes, hoists, or even overhead cranes may have to be used to move the weights within the laboratory and on or off the balances. Laboratory personnel must become expert in using such equipment, as necessary, to avoid damage to the laboratory facilities, to the balances used, and even to the weights themselves. Special hoist/crane hydraulics or multi-speed systems are available to gently set large weights in place on large comparators to avoid damage. The problem of temperature equilibrium for large weights is qualitatively the same as for small weights and needs consideration on an individual basis.

Large weights must be clean at the time of use, but cleaning may be a problem. Minimally, they should be inspected to ensure that foreign material is not present. Cleaning is addressed further in GMP 5.

Cleanliness of weight handling materials is just as critical as their use. Forceps, gloves, and other lifting materials are prone to contamination and must be regularly cleaned. Care must be taken when using gloves to ensure that heat and moisture are not transferred to the weights, that other laboratory materials (balances, pens, computers) are not handled that will cause contamination, and that gloves are not used simply to keep hands clean!

3 Balance and Weighing Operations

Analytical balances are high precision instruments and should be operated carefully. Excessive shock can damage a balance. Avoid shock loading the balance. Gently place all weights in the center of the weighing pan/platform. The dials on mechanical balances should be turned slowly and gently. Careful balance operation will improve the repeatability of measurements.

Mechanical analytical balances are provided with partial and full release positions. The partial release position is used when determining if an unknown load will provide an on-scale reading. The balance beam has limited movement in this position. The partial release position provides some protection to the balance when the dialed-in weights are not close to the actual mass placed on the pan. It is good practice to arrest the pan each time a dial is being changed to protect the balance from shock loading. It is acceptable to change the dial representing the smallest built-in weights when in the partial release position because the small weight changes should not result in shock loading of the balance.

When releasing the pan to either the full or partial release position, the action should be done slowly and carefully. The objective is to minimize disturbances to the balance as much as possible.

Similarly, all loads should be placed on the balance pan carefully and centered on the pan.

When a mechanical balance is released, the beam goes through a series of oscillations. The frequency of these oscillations diminishes as time passes until they are almost imperceptible to the naked eye. At this point, optimal stabilization is achieved. This stabilization of the balance typically lasts for a very short period, after which the balance reading will usually drift. A similar situation occurs when a mass is placed on the pan of an electronic balance. Therefore, readings should be taken at the precise moment of achieving balance stability. This interval between the releasing of a pan on a mechanical balance, or the placing of a mass on an electronic balance, and the reading of the observation, varies from balance to balance. Stabilization time differs for all balances, even among those of the same type and model. Knowledge of the operating characteristics of each instrument is critical to correctly establish this time interval. Although manufacturers will usually state this value, it is necessary for the metrologist to verify its reliability. Many electronic balances have a stability signal incorporated into the design, but stability also must be verified. All measurements in a calibration should be performed at the same time interval, and within the shortest time possible.

All balances should be exercised before taking readings. A load equal to the load to be measured should be placed on the balance, a reading taken, and the pan arrested, if appropriate, or the weight removed from electronic balances. This operation should be repeated several times before readings are taken for measurement purposes. Once the balance has been "warmed-up", better repeatability will be achieved. Balances can be very accurate even when used without being exercised first, but improved results can be obtained by going through a "warm-up" procedure. If the larger variation present in initial weighings on a balance that has not been exercised is not significant to the measurement, the warm-up procedure may be minimized. The warm-up description provided here is not related to ensuring an instrument is plugged in and turned on, although following manufacturers recommendations regarding electronic warm-up periods are also important.

To determine the repeatability of measurements when a balance has not been exercised versus its performance after being exercised, and to determine how much warm-up time or exercising is required, begin measurements using the process that will be used starting with the unexercised balance and record the readings. Repeat a series of measurements until you have obtained several measurements after the balance has been exercised. This process can be repeated over several days using the same objects and the results compared. The readings obtained when using an unexercised balance are likely to show a slightly larger variation than those obtained after the balance has been

exercised. Balance drift is likely to be larger initially and then reach a steady state when the balance has been exercised.

4 Comparison (Substitution) Weighing - Mechanical Balance

For mass calibrations, the unknown object must be compared to, or substituted with, a mass standard with known calibration values. Comparison weighing (also called substitution weighing) eliminates the errors of the built-in weights, reduces disturbances during the measurement because dial settings are not changed during the measurement, and can minimize the effects of drift by selecting the appropriate weighing design and appropriate sensitivity weight(s). Comparing the unknown, X , to a standard, S , eliminates the built-in weights (and associated calibrations) from the measurement: thus, the built-in weights act only as counterweights; they do not affect the difference measured between X and S . Consequently, the dial settings must not be changed during a comparison measurement; otherwise the built-in weights would be part of the measurement.

When comparison measurements are made on a single pan mechanical balance, all readings are taken from the optical scale. The unknown and the standard must have nearly the same mass so that the difference between them can be measured on the optical scale. If the masses of the unknown and the standard are significantly different, small mass standards are used as tare weights with either the unknown or the reference standard or both to obtain an observed mass difference that is significantly less than one-fourth the range of the optical scale.

As part of a comparison measurement, the mass value of a scale division is determined by performing a sensitivity determination. The small mass standard used as part of the weighing design to calibrate the optical scale is called the sensitivity weight and should have a mass between $1/5$ and $1/2$ the range of the optical scale, inclusive. Additionally, the mass of the sensitivity weight should have a mass that is at least twice the observed mass difference between the standard and the unknown. Since the maximum size of the sensitivity is limited to one-half the range of the optical scale, it may be necessary to carry tare weights with the standard and the unknown to ensure that the observed difference between them is less than one-half the mass of the sensitivity weight. The value of the sensitivity weight should include the correction plus its nominal value. For high precision calibrations, the inclusion of the air buoyancy correction for the sensitivity weight is critical and the sensitivity weight usually needs to be four times the difference between the standard and unknown (with any associated tare weights). See SOP 34 for detailed guidance on the selection of sensitivity weights.

The first readings for the standard and the unknown in a comparison on a single pan balance should fall in the first quarter of the optical scale, but well ahead of zero, so the balance drift will not result in negative values for any readings. Although negative numbers may be used in calculations, they are avoided to simplify calculations and reduce calculation errors. Because the sensitivity weight may have a mass as large as one-half the range of the optical scale and the measured difference between the standard and the unknown may be as large as one-fourth the range of the optical scale, it is necessary to obtain the first two readings in the first quarter of the optical scale so all readings will remain on-scale for the measurement. In this way it is not necessary to change the dial settings to measure the difference between the standard and the unknown.

5 Comparison Weighing - Electronic Balance

Measurements made on a full electronic balance are simplified because there are no built-in weights to consider. Although many electronic balances are equipped with a built-in calibration weight, the internal weights are not involved in the comparison (substitution) weighing.

The principles for comparison weighing on a full electronic balance are the same as when using a single pan mechanical balance. The balance indications are used to measure the mass difference between the standard and the unknown, and a sensitivity weight is used to establish the mass value for a digital division on the balance. Since there are no built-in weights in the full electronic balance, the entire range of the digital indications can be considered for "optical scale" of the balance.

For comparison weighing the standard and the unknown should be equally nominal or "nearly the same mass." Since a full electronic balance has a much larger range for indicating mass values, the masses do not have to be as close together as when a mechanical balance is being used. When using an electronic balance, the difference in mass between the standard and unknown should be less than 0.05 % of the balance capacity. Tare weights that are known standards should be used if the masses are not sufficiently close together. The sensitivity weight used to determine the mass per digital division should have a mass that is at least 10 to 20 times the mass difference between the standard and the unknown but not exceeding 1 % of the balance capacity. For high precision weighing, air buoyancy corrections must be made for all objects used in the intercomparison. See SOP 34 for detailed guidance on the selection of sensitivity weights.

6 Magnetic and Electrostatic Fields

Care must be taken when weighing magnets or objects having electrostatic charges. A magnetic field will likely affect results of a mass measurement. A magnet is attracted to ferromagnetic materials in the balance and balance chamber. The magnetic field may also affect the magnetic field generated in an electronic balance that utilizes the principle of magnetic force restoration as its method of measurement.

Weights made of ferromagnetic material can become magnetized during manufacture or during use if they are placed in a strong magnetic field. Magnetized weights can result in measurement errors that may go undetected. If a measurement problem is suspected, the weights should be checked for magnetism and may have to be rejected if excessively magnetized.

If magnets or magnetized material must be weighed, the material should be placed in a magnetically shielded container to prevent the magnetic field from generating measurement errors. If balance design and conditions permit, an alternative is to position the magnetized material a relatively large distance from the balance pan using a non-ferromagnetic object on the pan to serve as a platform for the magnetic. Since the strength of the magnetic field drops off at a rate of the cube of the distance from the magnetic, it may be possible to effectively isolate the magnet from other ferromagnetic material in the balance.

Electrostatic fields can also cause errors in measurements. If there is a static charge in a mechanical balance, the balance may give erratic readings and lack repeatability. If the object being weighed has a significant electrostatic charge, it may result in measurement errors and may leave a static

charge on the balance. Electrostatic charges are of concern when plastic containers are placed on the balance.

Care should be taken to remove electrostatic charges from objects being weighed by grounding the objects, if necessary, before placing them on the balance. To prevent the build-up of static electricity in a balance, the relative humidity in the laboratory should be maintained between 40 % and 60 % which are also the general manufacturer's specified limits for precision balance operations. The water vapor in the air will serve to drain off electrostatic charges from the balance.

Balances utilizing the magnetic force restoration principle for weighing should be checked to verify that the magnetic field generated by the magnetic cell in the balance does not exist around the balance pan. If the shielding of the magnetic cell is inadequate, measurement errors may occur when weighing ferromagnetic objects or when the balance is placed on a surface comprised of ferromagnetic material.