⟨1251⟩ WEIGHING ON AN ANALYTICAL BALANCE

Weighing is a frequent step in analytical procedures, and the balance is an essential piece of laboratory equipment in most analyses. In spite of this, weighing is a common source of error that can be difficult to detect in the final analytical results. The procedure described here applies directly to electronic balances; therefore, certain portions of the procedure are not applicable to other types of balance. The weighing procedure can be separated into three basic steps: planning, checking the balance, and weighing the material.

PLANNING

The initial step is to assemble the proper equipment, such as containers for weighing, receiving vessels, forceps, pipets, spatulas of proper size, and so forth. Use containers of size such that the loading capacity of the balance is not exceeded. Make sure that the containers selected to receive the weighed material are clean and dry. Assemble the necessary chemicals if solutions or reagents are required.

Preparation of the material to be weighed is often necessary. The material may require grinding or drying. Some materials may have been heated or stored in a refrigerator. Materials must be brought to the temperature of the balance before they are weighed. To avoid condensation of moisture, refrigerated materials must be allowed to come to room temperature before the container is opened.

CHECKING THE BALANCE

In the next step it is important to remember that, unless the balance is checked before each weighing operation is performed, errors can easily occur, resulting in faulty analytical data. The balance user should check the *Balance Environment*, *Calibration*, and *Balance Uncertainties*. Do not assume that the balance has been left in the proper operating condition by the previous user.

Balance Environment

The balance is placed in a suitable location with sufficiently low levels of vibration and air current. It must have a constant electrical supply. The balance and the surrounding work area have to be kept neat and tidy. It is good practice to use a camel's hair brush or its equivalent to dust the balance pan before any weighing so as to remove any materials that may have been left by the previous operator. [NOTE—Individuals must clean up debris, dispose of any spilled materials or paper, and remove the vessels and apparatus used in making the measurements.] When a balance is moved, it must be allowed to adjust to the temperature of its new environment and be recalibrated.

Calibration

If necessary, turn on the power, and allow the balance to equilibrate for at least 1 hour before proceeding with the calibration. (Microbalances may require up to 24 hours to reach equilibrium.) If the balance power has gone off and then has come back on, as in a power outage, certain types of balance may display a message indicating that the balance must be calibrated before a weighing is made. If the operator touches the balance bar, the message may be cleared and the balance may display zeros; however, the balance will not give the correct weighing until it has been calibrated. Electronic analytical balances have an internal calibration system based on an applied load. The calibration applies for the current ambient temperature.

Balance Uncertainties

DRIFT REDUCTION

Drift is one of the most common errors, and it is also one of the easiest to reduce or eliminate. Balance drift can be present without the operators being aware of the problem. Check the sample, the balance, and the laboratory environment for the following causes of errors, and eliminate them:

1. A balance door is open.
2. Temperatures of the balance and the material to be weighed are not the same.
3. The sample is losing or gaining weight.
4. The balance has been recently moved but has not been allowed to equilibrate to its surroundings or has not been recalibrated.
5. Air currents are present in the laboratory.
6. Temperatures in the laboratory vary.
7. The balance is not properly leveled.
8. Laboratory operations are causing vibration.
9. Hysteresis of the mechanical parts occurs during weighing.

MECHANICAL HYSTERESIS

Hysteresis in the balance is caused by excessive stretching of the springs, and it is primarily due to overloading or to the accidental dropping of an object onto the pan. Microbalances are very sensitive to overload and shock. When using a microbalance, set the lever to the rest position when adding or removing material; turn the lever to the weigh position to register the weight. In some cases, drift due to hysteresis can be eliminated by allowing the balance to stand without weighing long enough for it to recover. If stretching of the springs is excessive, an expensive balance overhaul may be needed. In the case of electronic force restoration balances, springs are replaced by flextures, and the term *creep* is more appropriate than *hysteresis.*

QUALITY ASSURANCE PROCEDURE FOR MEASUREMENT OF BALANCE DRIFT

Over an extended period of time, balance drift and other day-to-day variations are monitored by weighing a fixed check-weight on a regular basis; this check should be performed after the balance has been calibrated at the ambient laboratory temperature. The check should be made before the first weighing of the day or after any event that might disturb the balance's calibration (power failure, moving the balance to a new location, etc.). The check-weight may be any object whose mass remains constant and does not exceed the load limit of the balance. A balance weight makes a reliable check-weight. Each balance should be provided with a check-weight, which should be stored in a protective container near the balance.

Perform the following procedures to reduce balance errors and the possibility of an incorrect reading because of drift:

1. Make certain that the electrical power to the balance is on and that the level bubble is in the center of the indicator.
2. Calibrate the analytical balance or the microbalance. [NOTE—Some balances have a calibration lever, which must be returned fully to its original weighing position. Do not depend upon any prior calibration.]
3. The first person to use the balance each day should weigh the check-weight and record the weight in the

log book for comparison with previous readings. If a deviation greater than those indicated below for *Analytical Balances* and *Microbalances* is observed, the balance should be reported for service. [NOTE—Check-weights tend to gain weight upon standing because of mishandling and exposure to contaminants in the atmosphere. These weights can be cleaned by wiping with a lint-free cloth moistened with a small amount of an appropriate solvent such as diethyl ether.]

Analytical Balances—Select a check-weight of an appropriate mass to examine an analytical balance. If possible, set the balance to read to 5 decimal places. Follow the manufacturer's operating instructions. Pick up the check-weight with a forceps, place it carefully on the balance pan, and weigh it. [NOTE—Do not drop the weight on the balance pan, because damage to the balance could result.] Place the weight in the center of the pan to eliminate corner-weighing differences. The accuracy of the weight is not important: the only factor of interest is whether any drift has occurred. If no drift has taken place, the value should remain constant. Periodic weighing of a fixed weight will determine whether the boards (or knife edges in mechanical balances) in the instrument are defective. The check for drift at the most sensitive position will show whether a problem exists; the variation in the observed weight does not exceed ±0.2 mg. For example, with a 20-g weight, if the mean value of the readings were 19.9984, the tolerance would be from 19.9982 to 19.9986 g. Thus, several readings must be taken before one can establish a tolerance. [NOTE—The check-weight need not be of high accuracy, but it is essential that its mass remain constant. In addition, the tolerance does not correspond to the value of 0.1%, specified under *Weights and Balances* ⟨41⟩, for weighing material accurately. Rather, the tolerance is purposefully tight to reveal possible drift or calibration errors; this tolerance is readily achievable with modern electronic balances.]

Microbalances—Proceed as directed for *Analytical Balances,* but use a check-weight appropriate for the particular balance. For example, a 100-mg check-weight might be selected for a balance that has a load limit of 150 mg; or a 10-mg check-weight might be used for an ultramicrobalance with a load limit of 15 mg. (The operator must know the maximum capacity of the balance to select the correct check-weight.) The balance indicates the weight in milligrams. Record the weight as soon as the reading is stable for a few seconds. The variation in weighings ought to be within a range commensurate with the specifications given by the balance manufacturer, but not greater than 0.1% of the amount of material typically weighed on the particular balance. For example, if 10-mg samples are routinely weighed, the variation in the weighings of the check-weight cannot exceed 0.01 mg.

WEIGHING THE MATERIAL

In this final step, select the number of decimal places required for the analytical procedure. In most pharmaceutical analyses small quantities of material are used, requiring the balance reading to be set to the fifth decimal place to achieve the necessary accuracy. Weighing read with four decimal places is preferred for weighing near-gram quantities. Do not allow the material to remain on the balance for an extended period of time because changes, caused by interaction with atmospheric water or carbon dioxide, may take place.

Load Limit

Select the appropriate balance for the quantity and accuracy needed. Each balance has a load limit, which should not be exceeded. Each balance manufacturer supplies the maximum loading condition, and this limit varies with the type of balance. The operator should know this limit so that the balance will not be damaged. [NOTE—Electronic balances operate on a "load cell" principle that produces an electrical output proportional to the movement of the strain gauge and is linear over the range.]

Receivers

The proper receiver for the material must be selected. The receiver's weight plus the weight to be measured must not exceed the maximum load for the balance; the size and shape of the receiver should permit it to fit into the space and on the balance pan without interfering with any operation. It is important that the receiver be clean and dry. Common receivers are weighing bottles, weighing funnels, flasks, and weighing paper. The correct receiver depends upon the quantity and type of material (liquid, solid, or powder) to be weighed. All other things being equal, a vessel of low mass should be chosen when small amounts of material are to be weighed. It is recommended that gloves, forceps, or another type of gripping device be used when handling receivers, because oils from the hands will add weight.

The weighing funnel is often the most satisfactory receiver, because it can function as both a weighing dish and a transfer funnel, allowing easy transfer to volumetric flasks. Weighing funnels come in various sizes; the size suitable for the operation should be selected.

Weighing paper may be used for solids. Paper receivers must be handled by hand, and great care must be used to prevent spills.

Weighing by Difference

Weighing is usually done by difference. The following methods are acceptable for good analytical results.

METHOD 1

Tare the empty receiver as follows. Place the receiver on the balance in the center of the pan, and press the appropriate tare key on the balance. This operation electrically sets the signal from the strain gauge to zero so that the weight of the receiver is no longer indicated. Add the material to the receiver, and record the weight. Transfer the weighed material to the final flask or receiver; then reweigh the original weighing receiver by placing it in the same position on the pan. [NOTE—Do not change the set tare of the balance between these two weighings.] The second weight represents the untransferred material and is subtracted from the total material weight to determine the weight of the transferred material.

METHOD 2

If the empty receiver is not going to be tared, add the material to the receiver, and place the receiver on the balance in the center of the pan. Record the weight, and transfer the weighed material to the final flask or receiver; then reweigh the original weighing receiver by returning it to the same position on the pan. The second weight represents the sum of the weights of the receiver and the untransferred material; subtract this sum from the sum of the total material weight and the receiver weight to determine the weight of the transferred material.

METHOD 3

This method may be described as quantitative transfer. The material is added to the tared receiver, the amount is determined by difference, and then the whole amount is

transferred quantitatively (e.g., by using a solvent) to the final receiver.

Materials-Handling Safety Procedures

The operator must be familiar with precautions described in the Material Safety Data Sheet for the substance before weighing it. Hazardous materials must be handled in an enclosure having appropriate air filtration. Many substances are extremely toxic, are possibly allergenic, and may be liquids or finely divided particles. A mask that covers the nose and mouth should be used to prevent any inhalation of chemical dust. Gloves should be used to prevent any contact with the skin. [NOTE—The use of gloves is good practice for handling any chemical. If it is necessary to handle the container being weighed, the operator should put on gloves, not only for self-protection but also to prevent moisture and oils from being deposited on the weighed container.] During a weighing, the operator may be exposed to high concentrations of the pure substance; therefore, the operator must carefully consider these possibilities at all times.

Weighings are made on many different types of materials, such as large solids, finely divided powders, and liquids (viscous or nonviscous, volatile or nonvolatile). Each type of material requires its own special handling.

Weighing Solids

Solids come in two forms: large chunks, with or without powdery surface, and finely divided powders or small crystals. If large chunks with a powdery surface are to be weighed, at least a piece of weighing paper must be placed on the balance pan to protect it from damage. Large nonreactive chunks that have no powdery surface may be placed directly on the pan (for example, a coated tablet). [NOTE—Solid pieces must be handled with forceps, never by hand.]

STATIC CHARGE

Fine powders have a tendency to pick up static charge, which will cause the particles to fly around. This static charge must be eliminated before a suitable weighing can be made. An antistatic device may be used to minimize this problem. [NOTE—Such devices may use piezoelectric components or a very small amount of a radioactive element (typically polonium) to generate a stream of ions that dissipate the static charge when passed over the powder to be weighed.] The static charge depends upon the relative humidity of the laboratory, which in turn depends upon the atmospheric conditions. In certain conditions, static charge is caused by the type of clothing worn by the operator; this charge causes large errors in the weighing when discharged.

WEIGHING PROCEDURE

Place the receiver on the balance pan, close the balance door, and weigh as indicated for *Weighing by Difference*, with the following additions. Carefully add the powdered material from a spatula until the desired amount is added. Use care to avoid spilling. Close the balance door, and record the weight as soon as the balance shows a stable reading.

SPILLS

If solids are spilled, remove the receiver, and sweep out all of the spilled material from the balance. The spilled material must be properly disposed of and must not be swept out onto the balance table where other operators may come in contact with the chemical. Then either start the process over or reweigh the remaining material. [NOTE—Never return any excess material to the original container. Any excess material must be disposed of in a proper manner.]

Weighing Liquids

Liquids may be volatile or nonvolatile and viscous or nonviscous. Each type requires special attention.

WEIGHING PROCEDURE

Weigh as directed for *Weighing by Difference*, with the following additions. Liquids should always be weighed into a container that can be closed so that none of the material is lost. It is best if the liquid can be added to its receiving container outside the balance because of the possibility of a spill. [NOTE—Liquids spilled within the balance housing can cause serious damage to the balance, and they may be difficult to remove.]

Nonviscous liquids can be handled with a Pasteur capillary pipet equipped with a small rubber bulb such as a medicine dropper bulb. The liquid is discharged into its receiver, the top is closed or stoppered, and the receiver and contents are weighed. Small quantities of viscous liquids can be handled by touching a glass stirring rod to the surface of the liquid and then carefully touching the rod to the side of the receiving vessel, which allows some of the material to be transferred.

Weighing Corrosive Materials

Many chemicals, such as salts, are corrosive, and materials of this nature should not be spilled on the balance pan or inside the balance housing. Extreme care is essential when materials of this nature are being weighed.

CONCLUSION

By carefully following the procedures outlined above, laboratory personnel will eliminate many errors that might be introduced into weighing procedures. However, it is important for each balance to be serviced and calibrated regularly by a specially trained internal or external service person. The balance should be tested using weights traceable to standardization by the National Institute of Standards and Technology. No repairs should be made to any balance by anyone other than a qualified maintenance person.

⟨1265⟩ WRITTEN PRESCRIPTION DRUG INFORMATION—GUIDELINES

The purpose of these guidelines—comprising format, content, and accessibility of prescription drug leaflets—is to help ensure that leaflets are useful. In this context, "useful" means that recipients receive, understand, and are motivated to apply written information about their medicines to achieve maximum benefit and minimize harm. Dispensers, prescribers, health care providers who counsel patients about their medicines, and the patients themselves are intended to be the primary beneficiaries for these guidelines.