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Weighing on an Analytical Balance

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Weighing is a frequent step in analytical procedures, and the balance is an essential piece of laboratory equipment in most analyses. Yet weighing is a common source of error which 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 balances.

The weighing procedure can be separated into three basic steps, namely, planning, checking the balance for proper operation, and the actual weighing.

Planning

Assemble the proper equipment, such as containers for weighing, receiving vessels, forceps, pipettes, proper sized spatulas, etc. Use containers of such size that the loading capacity of the balance is not exceeded. Make sure that the containers to be used 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.

Balance Check

Unless the balance is checked each time a weighing is made, errors can easily occur, resulting in faulty analytical data. Do not assume that the balance has been left in the proper operating condition by the prior user.

Check the balance location and mounting. The balance table must be free of vibration and located in a place with minimum air current to reduce or eliminate weighing errors. The balance must have a constant electrical supply. The working area must be kept clean, free of debris and clutter. The operator must have a suitable place to work and be seated in a comfortable position. Weighing is best done alone; if others are present, they might cause interference by air currents or

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distract the operator.

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 operator 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 and 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.

Hysteresis in the balance is caused by excessive stretching of the springs, and is primarily due to overloading or to accidentally dropping 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 was excessive, an expensive balance overhaul may be needed.

If for any reason the balance power has been off, allow at least 1 hour for the balance to equilibrate after turning it on. (Microbalances require 24 hours to reach equilibrium). If the power to the balance has gone off and then back on, such as in a power outage the operator is not aware of, the balance will typically display a "calibrate" message. This message means the balance must be calibrated before a weighing is made. If the operator touches the balance bar, the "calibrate" message is typically cleared and the balance display zeros, but the balance will not give the correct weighing. *CALIBRATE BEFORE YOU DO ANYTHING ELSE.*

When a balance is moved, it must be allowed to adjust to the temperature of its new environment and be recalibrated. It also must be in a location that is free of vibration and air currents. Turn on the power and allow the same time as described above before proceeding with a calibration. Electronic analytical balances have an internal calibration system based on an applied load. The calibration applies for the current ambient temperature.

Quality Assurance Procedure for Measurement of Balance Drift

Balance drift or other changes from day to day over an extended period are monitored by

weighing a fixed check weight on a daily basis after the balance is calibrated at the ambient laboratory temperature (1). The check weight can be any object which remains constant and whose mass is not greater than the mass of the internal calibration weight. A balance weight is a good check weight.

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 with the internal standard weight before making any weighing. Some balances have a calibration lever which must be returned fully to its original weighing position. Do not depend upon any prior calibration.
3. Each balance should be provided with a check weight which should be stored inside the balance housing. The first person to use the balance each day should weigh the check weight and record the weight in the balance's log book for comparison with previous readings. If a deviation of more than 1 is observed in the decimal place indicated below for the type of balance, it should be reported for service. Check weights will tend to gain weight upon standing because of mishandling and exposure to the contaminated atmosphere. These weights can be cleaned by wiping with a lint-free cloth moistened with a small amount of diethyl ether.
    A. Analytical balance (4 and 5 places): A check weight of 20 g is an appropriate mass with which to check either a 4- or 5-place analytical balance. Set the balance to read 5 places. With the balance pan in the locked position, pick up the 20-g check weight with forceps, place it carefully on the balance pan, and weigh it. *CAUTION: Do not drop the weight on the pan balance because damage to the balance could result.* Place the weight in the center of the pan, so as 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 knife edges or boards in the instrument are defective. The check for drift at the most sensitive position will show if a problem exists; there should be no variation in the observed weight in the first four places to the right of the decimal point.
    B. Microbalance: Proceed as in A, above, but use a check weight of 100 mg for balances that have a maximum loading of 150 mg. Some ultra microbalances have a maximum loading of 15 mg, in which case the check weight should be 10 mg. *(CAUTION: The operator must know the maximum capacity of the balance in order to select the correct check weight.)* The balance indicates the weight in milligrams, and there should be no variation in the first three places to

the right of the decimal point.

4. Record the weight as soon as the reading is stable for a few seconds. Do not allow the material to remain on the balance for an extended period because of possible changes due to interaction with atmospheric water or carbon dioxide.

Weighing the Material

The balance pan and the area inside the balance housing must be kept clean. It is good practice to use a camel's hair brush before any weighing to remove any dirt, dust, or materials that may have been left by the previous operator because dust can easily collect on the pan without being noticed. The area around the balance must also be kept clean. Each operator must clean up his or her own debris, dispose of any spilled materials or paper, and remove all the vessels and apparatus used in making the measurements. The entire area must be returned and maintained as a cleaned area.

Select the number of places required for the analytical procedure. Most pharmaceutical analyses use small quantities of the material, which requires the balance to be set to read the fifth place in order to achieve the necessary accuracy. Weighing to the fourth place is preferred for weighing near-gram quantities. Top-loading balances can be used for weighing when the weight does not enter into the quantitative calculation.

The balance selected should be the proper one 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 be sure to know this limit so that the balance will not be damaged. Electronic balances operate on a "load cell" principle which produces an electrical output proportional to the movement of the strain gauge, and is linear over the range.

The proper receiver for the material must be selected. The receiver s weight must not exceed the maximum load for the balance; it must be one that will fit into the space and fit on the balance pan without interfering with any movement. 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 (liquids, solids, or powders) to be weighed.

The weighing funnel is often the most satisfactory receiver, since it can function as both a weighing dish and a transfer funnel; it allows easy transfer to volumetric flasks. Weighing funnels come in various sizes; the size suitable for the operation should be selected. Weighing funnels, boats, and other such receivers should not be touched by bare hands, since oils from the hands will add weight. Gloves or forceps should be used to handle receivers of this type.

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

Weighing is usually done by difference. Optionally, the empty receiver may be tared as follows: Place the receiver on the balance in the center of the pan, and press the "T" or the "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. Do not change the set tare of the balance between these two weighings. The second weight represents the untransferred material and is subtracted. If the empty receiver is not tared, subtract the weight of the container from the sum of the weight of the material plus the weight of the container to give the weight of the material. Both methods are acceptable for good analytical results.

The operator must be familiar with precautions described in the Material Safety Data Sheet for the substance before weighing it. Many substances are extremely toxic, possibly allergenic, and may be liquids or finely divided particles. A mask which covers the nose and mouth should be used to prevent any inhalation of dusts. Gloves should be used to prevent any contact with the skin. The use of gloves is good practice for handling any chemical. If it is necessary to handle the container that is 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 and nonviscous, volatile and 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 without a receiver, a piece of weighing paper must be placed on the balance pan to protect it from damage. Some large chunks that have no powdery surface may be placed directly on the pan (for example, a coated tablet). Solid pieces must be handled with forceps, never by hand.

Fine powders have a tendency to pick up static charge, which will cause the particles to fly around. This static pickup must be eliminated before a suitable weighing can be made. An antistatic device should be located at each balance station. 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. Static

pickup depends upon the relative humidity of the laboratory, which in turn depends upon the atmospheric conditions. In certain conditions, static pickup will be caused by the type of clothing worn by the operator; this causes large errors in the weighing when the charge is discharged.

Place the receiver on the balance pan, close the balance door, and optionally tare the weight so that the balance will read zero. Carefully add the powdered material from a small 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. Transfer the contents of the receiver into an appropriate vessel, and reweigh the receiver to make a correction for any material which may have adhered to it.

Solids may be spilled accidentally; if this happens, remove the receiver and sweep out all 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 start either the process over or reweigh the remaining material. 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, viscous or nonviscous. Each type requires special attention. Liquids should always be weighed into a container which 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 of the balance because of the possibility of a spill. Liquids spilled within the balance housing can cause serious damage to the balance, and may be difficult to remove.

Nonviscous liquids can be handled with a Pasteur capillary pipette 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.

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 to be weighed.

General Comments

Each balance should be serviced and calibrated regularly by a specially trained person (either an internal or an external service person). The balance should be tested with weights traceable to standardization by the National Institute of Standards and Technology. No repairs should be

made on any balance by any other than a qualified maintenance person.

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Reference

(1). Kenyon A.S, Black J.C, and Layloff T.P, Quality Assurance in Weighing. *J.AOAC International*, *78* (4), 1109, 1995.