

WEIGHING BY DIFFERENCE **(From Dr. David Chesney)**

Weighing by difference is the most accurate method to measure quantitatively the mass of a solid sample. This procedure involves repetitive weighings of a weighing bottle containing a quantity of solid reagent. As the solid is removed to another vessel, the mass of the weighing bottle contents decreases. Upon reweighing, a lower mass is found. The difference in the two masses represents the mass of solid reagent transferred to the vessel. Hence, the phrase "weighing by difference".

PREPARATION FOR WEIGHING

Place in a clean, dry glass weighing bottle an amount of solid reagent estimated to be sufficient for all necessary weighings.

Dry the weighing bottle and contents as instructed. Leave the weighing bottle stopper tilted on its side to allow vaporized water to escape.

Cool the weighing bottle and contents in a desiccator with fresh desiccant. Be careful to avoid contaminating the outside of the weighing bottle with stopcock grease from the lid of the desiccator. Tilt the stopper of the weighing bottle on its side - **do not store a closed weighing bottle in the desiccator.**

Calculate the allowable mass range of your solid reagent appropriate for the determination at hand.

NOTE: Determine what you will accept as the smallest and largest amount of reagent which is consistent with subsequent steps in the experiment. For example, in the standardization of NaOH solution with KHP, the procedure calls for about 0.70 g. of KHP per determination. This amount will require about 35 mL of NaOH titrant for neutralization. However, any titration volume from 25-45 mL will provide the desired degree of accuracy for the standardization. Figure out the corresponding mass range of KHP for this range of titrant volume and use a sample size within the acceptable limits.

THE WEIGHING PROCESS

1. Take the following to the balance room.

1. Desiccator with weighing bottle inside.
2. A number of beakers or Erlenmeyer flasks equal to the number of determinations to be done.
3. Your laboratory notebook.

2. Always use the balance assigned to you.

3. Place the weighing bottle carefully on the balance pan.

NOTE: If your hands are clean and dry, you may not leave a weighable fingerprint on the weighing bottle. However, it is prudent to use a folded piece of paper (clean, dry and lint-free) to handle the weighing bottle, thus preventing contact with your fingers. DO NOT use a pair of tongs to handle the weighing bottle, as the likelihood of slippage and/or breakage goes up dramatically.

NOTE: The decision whether or not to stopper the weighing bottle is usually determined by the hygroscopic (ability to adsorb atmospheric water vapor) nature of the solid reagent. If your reagent is hygroscopic, it is best to minimize contact with water vapor by keeping the stopper on the weighing bottle as much as possible. If your reagent is not appreciably hygroscopic, the stopper may be left off for the duration of the weighing process.

Keep the stopper clean and fingerprint-free.

4. Record the mass in your notebook.

5. Remove the weighing bottle and transfer a quantity of the solid reagent to the beaker or flask in which you will perform the determination.

NOTE: The transfer must be quantitative. Make certain that all the solid reagent removed from the weighing bottle ends up in the beaker or flask. DO NOT use a funnel or piece of paper to effect the transfer; solid reagent may remain on the surface. Perform a direct transfer of the solid reagent from the weighing bottle to the determination vessel by "rolling" the weighing bottle on its side over the mouth of the vessel. In a pinch, a clean, dry spatula may be used to facilitate the transfer, provided that steps are taken to prevent any reagent from remaining on the spatula (rinsing, etc.).

6. Replace the weighing bottle on the balance pan and record the new mass. Subtract this new, smaller mass from the previous mass and compare the result to the desired mass range for the determination (as calculated previously).

NOTE: The amount of material to transfer must always be an estimation, particularly for the first weighing. It may be necessary to transfer reagent a number of times until you obtain the proper amount. Do not attempt to transfer solid reagent back into the weighing bottle if you overshoot. This will lead to an additional transfer and another source of possible error. If the amount transferred is so large as to be unusable, simply discard the solid reagent properly (see instructor), rinse out the vessel, and begin the weighing and transfer process over again. Once you have an appropriate amount of reagent transferred, use this amount as a visual guide to estimate subsequent samples.

7. Repeat these steps for each sample. Record your masses in your notebook in the following manner. Include the appropriate mass unit at all times.

$$\begin{array}{r} \text{Initial (larger) weight} \\ - \text{Final (smaller) weight} \\ \hline \text{Difference} \end{array}$$

You should be able to accurately weigh three samples in ten minutes. Since there will be more than one student assigned to each balance, please be advised that access to the balances is on a first-come, first-served basis. However, **if you take longer than ten minutes at a balance station, it is likely that you will be asked to leave to allow someone else the use of the balance.**

AFTER WEIGHING

Replace the weighing bottle (stopper on its side) in the desiccator. Do not discard any of your solid reagent until you are **ABSOLUTELY CERTAIN** you will not need any more.

Clean up your balance station.

WEIGHING BY DIFFERENCE EXERCISE

Equipment required: Weighing bottle, two beakers, NaCl, notebook.

Half-fill a clean weighing bottle with NaCl (table salt).

Using the technique of weighing by difference, weigh out a 0.7 g sample of NaCl. The sample should be transferred to a beaker.

Record the sample weight data in your notebook in proper format.

Repeat three additional times. Attempt to get samples within 100 mg of the weight of the original sample. These samples may be transferred to a common beaker, since they will not be individually used.

Calculations:

In one of your first lab experiments, you will be asked to standardize 0.1 M NaOH titrant with KHP, a primary (acid) standard. KHP is a monoprotic acid (HA), so one mol of KHP reacts quantitatively with one mole of NaOH. The molecular weight of KHP is 204.23 g/mol.

- 1) You have weighed out 0.7005 g KHP. Calculate how many mL of 0.1000 M NaOH will be required to exactly neutralize this amount of KHP.
- 2) Your second weighing results in a mass of 0.8004 g KHP. Given the fact that you have only a 50-mL buret, will you exceed this volume using 0.0800 M NaOH titrant?
- 3) How many grams of primary standard grade KHP will be neutralized by 30.07 mL of 0.1000 M NaOH?

You must be able to perform calculations like these for this class. Do these calculations now and verify the answers with the TA.