Chapter 5 Gravimetric Analysis

Gravimetric analysis involves the use of the analytical balance. Common laboratory practices include weighing chemicals for standard and reagent preparation and solids analysis such as Total Suspended Solids, Mixed Liquor Suspended Solids, and Total Dissolved Solids.

Weighing Solids

One of the basic skills required by a lab technician is the preparation of reagents used in a variety of analyses. To weigh a chemical on an analytical balance, the technician should first place a "tare" on the balance. A "tare" can be any clean, dry object capable of holding the chemical. Common objects that can be used as tares are weighing paper, weighing bottles, weighing boats, or even beakers.

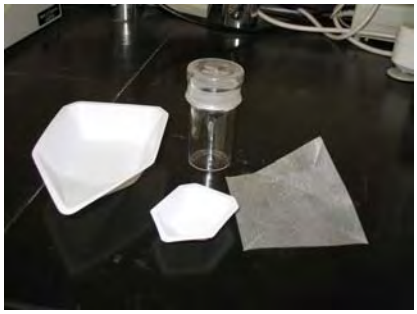

Figure: Various tare materials

If the weighing is only a one time operation, the tare can be zeroed by pressing the zero button/bar. The tare weight will now read zero.

Figure: Tare zeroed

The chemical can now be carefully added directly to the tare using a small spatula. Place the bottle containing the dry chemical through one door of the balance and the spatula through the other door to reduce spilling. After the dry powder has been measured, the balance doors should be closed to verify the weight is correct. Add or remove small amounts of chemical by using a narrow-tipped spatula. To avoid contamination of the original bottle, excess solids should not be returned to the bottle but rather be discarded appropriately.

Transferring Solids

Once the solids have been accurately weighed, they must be transferred to a receiving container without loss. Accurate weighing is only useful if all the solids are transferred. Carrying a weighing boat across the room is poor procedure. It is better to remove the tare and solids from the balance and bring the receiving container to the balance area. Small amounts of the weighed powder may be lost by carrying solids across the room. Place a small funnel into the neck of the receiving flask to avoid loss of the powder.

Figure: Use a funnel to transfer solids

Pour and wash the solids into the funnel using a deionized water wash bottle. Avoid splashing or spilling the powder. Recover any remaining powder by rinsing the tare several times with deionized water. Pour the rinses through the funnel. Rinse the funnel with several small

volumes of deionized water. Remove the funnel and now rinse the neck of the container with small volumes of deionized water. All the weighed solids should now be in the container.

Figure: Rinse to recover all the weighed solids

If the container is a volumetric flask, add a magnetic stirrer to the flask and dissolve the powder. Use a magnet retriever to remove the magnet and rinse the magnetic retriever with deionized water as it is removed from the flask. Once the magnet is removed, fill the volumetric flask to the mark with deionized water.

The magnet can now be put back into the volumetric flask again to finish mixing the solution completely.

Weighing Liquids

Liquids cannot be measured accurately on an analytical balance. The analytical balance is so sensitive; it will measure evaporation of the liquids. If measuring liquids, the first weight displayed should be used.

Transferring Liquids

Liquids which have been weighed are transferred using the same technique as solids. The liquid should be poured into a funnel; the container, the funnel and the walls of the container should be rinsed down with deionized water.

Reagent Preparation

Reagents are chemical solutions used in the measurement of some specific analyte (what your testing for). Analytical balances are commonly used to measure the amount of chemical called for in the recipe found in *Standard Methods for the Examination of Water and Wastewater*. Most reagent preparation is straight forward and uneventful, however, it is critical that the lab technician read the complete procedure before beginning preparation. Sometimes the last sentence is the first thing you do!!

Standard Preparation

Concentrations of solutions are usually written in one of 3 forms - Molarity (M), Normality (N), and mg/L. For most laboratories, it is more economical to purchase standard solutions than prepare them in-house. Major suppliers like Fisher Scientific, VWR, and Ricca have a large variety of solutions commonly used in routine laboratory analysis. While these solutions come pre-standardized, the laboratory may have to verify the concentration upon arrival.

Molarity

Molarity is a form of concentration not typically encountered in wastewater analysis. It stands for moles/liter and is occasionally found in *Standard Methods* as part of a chemical recipe.

Normality

Normality is a form of concentration commonly describing the concentration of acids and bases. For example, a 0.02 N concentration of hydrochloric acid is weaker than a 1 N concentration. The normality of a solution is measured by a process called standardization. Standardization is a chemical method of

calibration. In the standardization process, the concentration of an unknown solution is compared against a known concentration using a procedure called a titration. This will be discussed later.

Mg/L

The most common form of concentration used in water and wastewater analysis is milligrams per liter (mg/L). A milligram per liter would be defined as the weight of a solid component dissolved in one liter of liquid, namely water. For example, a 40 mg/L total suspended solids concentration would mean that there were 40 milligrams of solid material in each liter of wastewater. A 0.15 mg/L sulfate concentration would mean that 0.15 milligrams of sulfate were dissolved in 1 liter of water.

This is the common unit used for analysis of water and wastewater and would be similar to $1 if we were measuring money. It is seldom feasible to test 1 liter of sample but the answer is always reported as if l liter was tested.

For example, suppose you filtered 100 milliliters (0.1 liter) dried the filter then weighed 20 mg of dry solids using the analytical balance.

The answer could be: $\frac{\text{20 milligrams}}{\text{0.1 liter}}$

However, convention calls for the answer to be reported as mg/L. This would make the reportable answer:

$$\frac{\text{20 milligrams}}{\text{0.1 liter}} = \frac{\text{200 milligrams}}{\text{1 liter}}$$