Part 1 General Principles in Chemical Laboratory

Chapter 1 Chemical Laboratory Safety Rules

Accidents in a chemical laboratory usually are resulted from improper judgment of the victim or one of his/her neighbors. Learn and observe the safety and laboratory rules listed below before starting to do chemical experiments.

Precautions

(1) Maintain a wholesome, businesslike attitude in the laloratory. Horseplay and other acts of carelessness are prohibited.

(2) Never work in the laloratory without the instructor present. This includes setting up equipment.

(3) Wear suitable clothing. Wear clothing that will protect you against spilled chemicals or flaming liquids. Hard-soled, covered footwear must be worn in the laboratory at all times——**no sandals allowed**.

(4) Eating, drinking, and smoking are strictly prohibited in the laboratory at all times because of the possibility of chemicals getting into the mouth or lungs through contamination. The chief hazard with smoking is fire or explosion.

(5) Know the ways to put out a fire.

a) If it is open fire, such as a large chemical spill on a laloratory bench, the correct extinguisher should be used as follows:

- Pull the pin.
- Point the extinguisher (if dry) or hose (if CO_2) at the base of the fire.
- Squeeze the handle while moving the extinguisher back and forth.

NOTE: Be careful not to spread the fire by getting the nozzle of the extinguisher too close——the material being emitted is under pressure.

b) If it is a small contained fire, such as in a flask or beaker, cover the container with a piece of ceramic, cutting off the supply of oxygen to the fire and thus putting it out.

(6) Wash chemicals from skin.

a) If you get a chemical burn from a caustic material, i.e. acid or base, immediately wash the burned area with large amount of water. Ask another student to inform the laloratory instructor.

b) Wash your hands and face quickly and thoroughly whenever they come into contact with chemicals.

c) Always wash your hands, before leaving the laloratory since toxic chemicals may be transferred to your mouth at a later time.

d) Chemicals spilled over a large part of the body require immediate action. Remove all contaminated clothing and use the safety shower, flooding the burned area. Do not use salves, creams, lotions, etc. Get medical attention.

Preparation before the Class

A detailed preparation is highly requested before each single experiment. The experimental instructor may list the order of all the experiments before the first class.

Rules during the Experiment

(1) Keep your workspace orderly.

a) Place tall items, such as graduated cylinders, toward the back of the workbench so they will not be overturned by reaching over them.

b) Clean up all chemical spills, scraps of paper, and glasswares immediately.

c) Keep drawers closed while working and the aisles free of any obstructions.

d) Never place coats, books, and other belongings on the laboratory bench where they will interfere with the experiment and are likely to be damaged.

(2) Always read the label first. Identify the chemicals before use. Read the label carefully, *read it twice*, before taking anything from a bottle. Many chemicals have similar names, such as sodium sulfate and sodium sulfite. Using the wrong reagent can spoil an experiment or can cause a serious accident.

(3) Assume that a particular reagent is hazardous unless you know for sure it is not.

a) Never taste a chemical unless specifically instruction to do so.

b) If you are instructed to smell a chemical, point the vessel away from your face and carefully fan the vapors toward your face with your hand and sniff gently.

c) Material Safety Data Sheets are available.

(4) Never point a test tube toward your neighbors or yourself when:

a) Heating a test tube over a burner.

b) Carrying out a reaction in a test tube.

(5) Dilute concentrated acids and bases by pouring the reagent into water (room temperature or lower) while stirring constantly. Never pour water into concentrated acids; the heat of solution will cause the water to boil and the acid to splatter. To help you remember——"*Do as you oughter, pour acid into water*."

Be careful with flames. A lighted gas burner can be a major fire hazard.

a) General Precautions:

- The burner should be burning only for the period of time in which it is actually utilized.
- Before lighting your burner carefully, position it on the desk away from flammable materials, overhanging reagent shelves, flammable

reagents such as acetone, toluene, and alcohol on neighboring desks.

- Be careful not to extend your arm over a burner while reaching for something.

b) Personal Precautions:

- Keep long hair tied back so that it cannot fall forward into a flame.
- Keep beards away from flames.

(6) Assemble safe apparatus. Always assemble an apparatus as outlined in your instructions. Makeshift equipment and poor apparatus assemblies are the first step to an accident.

(7) Avoid rubbing your eyes unless you know your hands are clean.

(8) Do not put hot objects on the desktops. Place hot objects on a wire gauze or ceramic pad.

(9) Never throw lighted matches into a sink. They may ignite a discarded flammable liquid.

(10) Perform only authorized experiments. Unless authorized to do so by the experimental instructor, a student will be subject to immediate and permanent expulsion from the laloratory if:

a) Attempting to conduct unauthorized experiments.

b) Attempting variations of the experiment in the laloratory manual.

Performing unauthorized experiments are dangerous. Students lack the experience to recognize whether or not the chemicals and techniques are safe.

(11) Clean up your workspace at the end of each laboratory period.

a) Wash and wipe off your desktop.

b) Be sure gas and water is turned off.

c) Return all special equipment to the stockroom.

(12) Avoid using excessive amounts of reagent.

a) Never use more than called for in the experiment.

b) Do not return any excess chemical to the reagent bottle; share it with another student or dispose of it according the in the instructions listed in (13).

c) If you are uncertain how to dispose of an excess of a specific chemical,

consult your experimental instructor.

(13) Discard waste chemicals as follows:

Waste Chemical's Proper Disposal.

a) Non-flammable water-soluble liquids discard into liquid wastes bottle.

b) Chemical solids, contaminated paper, and contaminated broken glassware, solid wastes bottle.

c) Discard paper products into trash can.

d) Discard organic solvents into organic waste bottle (Do not put acids in the organic waste bottle).

e) Discard glass tubing waste or broken glass wares into broken glass wooden box.

(14) Always add a reagent slowly——never "dump" in. There're two reasons:

a) Some reactions give off a lot of heat, and unless adding slowly, can become too vigorous and out of control.

b) If you make a mistake and choose the wrong chemical, adding slowly decreases the possibility of causing a serious accident.

(15) Treat chemical spills as follows:

a) Alert your neighbors and your experimental instructor.

b) Clean up the spill as directed by your experimental instructor.

(16) Never fill a vessel more than about 70% capacity if you plan to heat it, unless specifically told to do so.

(17) Be aware of your neighbors' activities; you may be a victim of their mistakes. If you observe improper techniques or unsafe practices:

a) Advise your neighbors.

b) Advise your experimental instructor if necessary.

(18) Observe all specific precautions and modifications mentioned in each experiment.

(19) Do not remove any chemicals from the laboratory.

(20) For reasons of safety, you may not be allowed to enter the laboratory if you are late.

Chapter 2 General Operations

1. Washing Glassware

In order to obtain a precise results, cleanliness of glassware is highly required.

(1) Chemical stains are often cleaned up with acids, alkalis, or organic solvents.

(2) For example, the stain of the azo dyes (Methyl Red and Methyl Orange) prepared in “Organic Chemistry Experiment” can be easily dissolved in an alkaline solution.

(3) Do not scrub the inside of volumetric glassware such as burettes, volumetric pipettes, measuring flasks, and conical measures with a brush. This could cause a volumetric disorder. Volumetric glassware should be rinsed repeatedly with tap water.

(4) For the same reason, volumetric glassware should not be dried by heating.

(5) Wash off chemical stains with a brush and sodium hydrogen carbonate.

(6) Dissolve stains of organic substances with a small amount of ethanol. Pour the washing waste of ethanol into the specific waste container.

2. Liquid Wastes

Laboratory liquid wastes are classified into organic and inorganic wastes. Pour liquid wastes into the designated liquid wastes container according to textbooks and experimental instructor. Rinse the glassware with the minimum amount of water, and pour into the liquid wastes container.

3. Volumetric pipette

The volume of a volumetric pipette (Fig. 1-1) is the volume of a liquid going out completely from the pipette which is filled up to the marked line. In order to avoid volume disorder, volumetric laboratory glassware such as volumetric pipettes should not be dried by heating. The wet glassware are rinsed with the liquid to be used just before use.

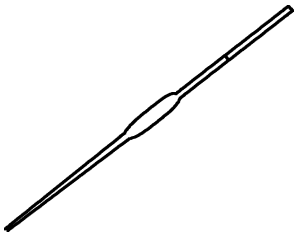

Fig. 1-1 Volumetric pipette

Volumetric Pipette Operation Procedures

(1) Insert the tip of the pipette into the liquid deeply, and draw up it 1-2 cm above the marked line.

(2) After pulling out the tip of the pipette above the surface, drop the inside liquid slowly to adjust the meniscus precisely to the marked line.

(3) Next, put the tip of the pipette on the inside surface of the container to remove a remaining droplet.

(4) Keep the pipette perpendicularly and allow the liquid to fall naturally into the container.

(5) After almost liquid has been drained, hold the bulge of the pipette in your palm during which all the valves of the filler are closed. Then, the inside air expands and all of the liquid falls.

4. Operation of Volumetric Flasks

A volumetric flask is employed when a solution of a definite concentration is prepared. The volume of a volumetric flask is the volume of a liquid in the flask which is filled up to the marked line. When a standard solution is prepared from a

solid sample, it is not dissolved in a volumetric flask but in another container. Then, the solution is transferred into the volumetric flask. Because the volume may change at mixing of a sample and a solvent, the sample should not be diluted directly to the marked line without stopping. After the final dilution, the solution is mixed thoroughly, by inverting the flask and shaking. The mixing is not enough when it is shaken without inverting. The way of determine the finishing point in volumetric flask is shown in Fig. 1-2.

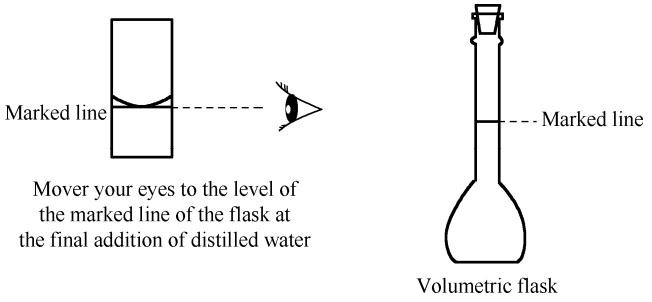

Fig. 1-2 The way of determine the finishing point in volumetric flask

Volumetric Flask Operation Procedures

(1) To make up a standard solution, add first a less amount of distilled water than required to the solid sample weighted precisely.

(2) Next, stir it to be dissolved completely using a stirring stick.

(3) Transfer the solution into the volumetric flask with a stirring stick or a funnel.

(4) In order to transfer the remaining solution in the container and on the stirring stick, rinse them some times with distilled water.

(5) Shake the flask gently to make the solution homogeneous.

(6) Repeat the operation of adding a small amount of distilled water and shaking until the height of the solution reaches just below the marked line.

(7) Move your eyes to the level of the marked line of the flask at the final addition of distilled water. Adjust the meniscus of the solution to the line by adding distilled water dropwise with a transfer pipette.

(8) After the final dilution, mix the solution thoroughly by inverting the flask and shaking with holding tightly the stopper.

Tuning procedures as using volumetric flask is shown in Fig. 1-3.

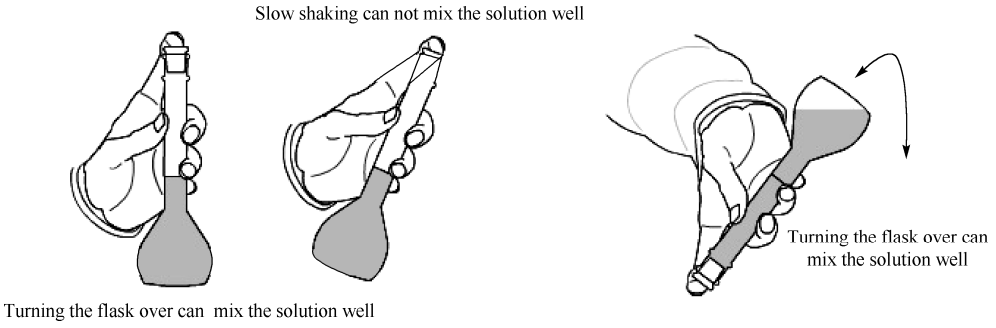

Fig. 1-3 Tuning procedures as using volumetric flask

5. Burette

Burettes are graduated cylinders that measure the volume of the drained liquid. Burettes should not be dried by heating, and thus you may use immediately after rinsing with small amounts of the solution to be measured.

Two approaches of burette operations are shown in Fig. 1-4.

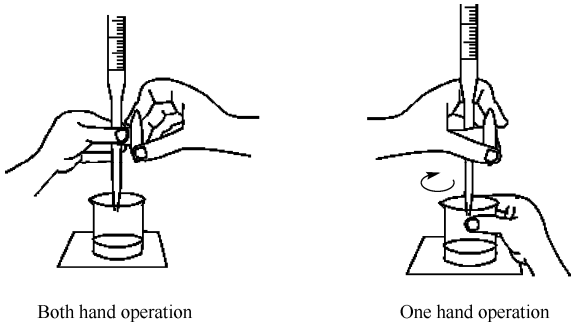

Fig. 1-4 Two approaches of burette operations

Burette Operation Procedures

(1) To transfer the titration solution into a burette, close the stopcock at the bottom and use a funnel. Do not forget to remove the funnel after transferring the solution.

(2) Drain a small amount of the solution to expel air bubbles at the tip of the burette. You do not need to adjust the liquid surface to 0.00 mL.

(3) Move your eyes to the level of the liquid surface and read the value of the bottom of the meniscus with 1/10 of the smallest scale marked on the burette.

6. Separation of Precipitates - Filtration Techniques

(A) Gravity Filtration

Gravity filtration is a common approach which applied for separating a precipitate from a solution using a filter paper and funnel. The method of folding filter paper is introduced in Fig. 1-5.

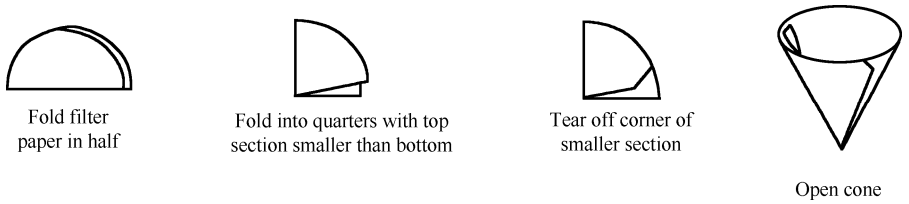

Fig. 1-5 The method of folding filter paper

Gravity Filtration Operation Procedures (Fig. 1-6)

(1) Choose a filter paper. Fold the filter paper into four and fit it into the funnel.

(2) When the filter paper does not fit well into the funnel, slightly adjust the folding in order to fit it perfectly.

(3) In order to make the filter paper fit perfectly, cut the edge of the over lapping filter paper in contact with the funnel obliquely. Wet the paper with the solvent and press with fingers to fit the paper in.

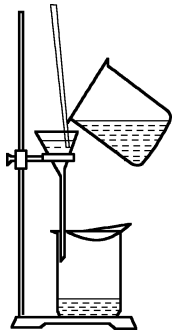

Fig. 1-6 Gravity filtration operation procedures

(4) Place a container in a manner that the leg of the funnel touches its inside wall.

(B) Vacuum Filtration

Vacuum filtration rather than gravity filtration is suitable for the separation of a precipitate consisting of light and fine particles. The vacuum filtration apparatus could be illustrated as shown in Fig. 1-7.

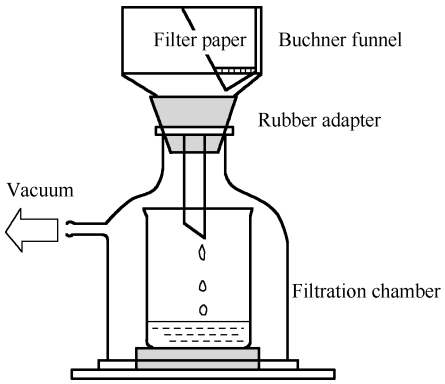

Fig. 1-7 The vacuum filtration apparatus

Vacuum Filtration Operation Procedures

(1) Connect a filtration chamber and an aspirator with thick wall rubber tubing through a three-way stopcock.

(2) Attach a rubber adapter to a Buchner funnel and set the funnel at the top of the filtration chamber.

(3) Put a receiving container in the filtration chamber. If the container is too far from the tip of the funnel, place a rubber board underneath.

(4) Wet the contact between the bottom rim of the filtration chamber and the coaster glass plate. Slightly rubbing their ground glass faces to obtain a good contact.

(5) Put a filter paper slightly smaller than the inside of the funnel and add a small amount of a solvent to wet the filter paper.

(6) Turn on the aspirator and open the three-way stopcock to fit the filter paper to the funnel.

Chapter 3 Data and Error Analysis

Performing the experiment and collecting data are only the beginning of the process of completing an experiment in science. Understanding the results of any given experiment is always the central goal of the experiment. Presenting those results in a clear concise manner completes the experiment. This overview of the complete process is as valid in an instructional laboratory course as in a research environment.

1. Error

The words “error” and “uncertainty” are used to describe the same concept in measurement. It is unfortunate that the term, “error” is the standard scientific word because usually there is no mistake or error in making a measurement. Frequently, the uncertainties are dominated by natural irregularities or differences in what is being measured.

Errors may arise from three sources:

(1) Careless errors: These are due to mistakes in reading scales or careless setting of markers, etc. They can be eliminated by repetition of readings by one or two observers.

(2) Systematic errors: These are due to built-in errors in the instrument either in design or calibration. Repetition of observation with the same instrument will not show a spread in the measurements. They are the hardest source of errors to detect.

(3) Random errors: These always lead to a spread or distribution of results on repetition of the particular measurement. They may arise from fluctuations in either the physical parameters due to the statistical nature of the particular

phenomenon or the judgment of the experimenter, such as variation in response time or estimation in scale reading.

For each measured value A, there is an estimated error ΔA. The complete result is given by $A \pm \Delta A$. This means that the "true value" probably lies between a maximum value of $A+\Delta A$ and a minimum value of $A-\Delta A$. Sometimes the terms relative error and percent error are used, where:

$$\text{Relative Error} = \frac{\text{Estimated Error}}{\text{Estimated Value}} \times 100\% = \frac{\Delta A}{A}\%$$

2. Accuracy and Precision

Firstly, When one considers the quality of a measurement there are two aspects to consider. The first is if one were to repeat the measurement, how close would new results be to the old, i.e., how reproducible is the measurement? Scientists refer to this as the precision of the measurement.

Secondly, a measurement is considered "good" if it agrees with the true value. This is known as the accuracy of the measurement. But there is a potential problem in that one needs to know the "true value" to determine the accuracy.

3. Significant Figures

Measured quantities are often used in calculations. The precision of the calculation is limited by the precision of the measurements on which it is based. The concept of significant figures is introduced to define the real precision of measurements.

The rules for identifying significant digits when writing or interpreting numbers are as follows:

(1) Non-zero digits are always significant.

(2) All zeros between other significant digits are significant.

The number of significant figures is determined starting with the leftmost non-zero digit. The leftmost non-zero digit is sometimes called the most significant digit or the most significant figure.

For example, in the number 0.004205, the “4” is the most significant digit. The left-hand '0's are not significant. The zero between the “2” and the “5” is significant.

The rightmost digit of a decimal number is the least significant digit or least significant figure. Another way to look at the least significant figure is to consider it to be the rightmost digit when the number is written in scientific notation. Least significant figure is still significant! In the number 0.004205 (which may be written as 4.205×10^{-3}), the “5” is the least significant figure. In the number 43.120 (which may be written as 4.3210×10), the “0” is the least significant figure.

If no decimal point is present, the rightmost non-zero digit is the least significant figure. In the number 5800, the least significant figure is “8”.

Rules When Significant Digit Involves in Calculation

1. Addition and Subtraction

When measured quantities are used in addition or subtraction, the uncertainty is determined by the absolute uncertainty in the least precise measurement (not by the number of significant figures). Sometimes this is considered to be the number of digits after the decimal point.

Example: A: 32.01 m; B: 5.325 m; C: 12 m.

When added the previous data together (A+B+C), you will get 49.335 m, but the sum should be reported as “49” m.

2. Multiplication and Division

When experimental quantities are multiplied or divided, the number of significant figures in the result is the same as that in the quantity with the smallest number of significant figures. For example, a density calculation is made in which 25.624 grams is divided by 25 mL, the density should be reported as 1.0 g/ mL, not as 1.0000 g/ mL or 1.000 g/ mL.