GUIDELINES FOR LABORATORY SAFETY

All instructors, students and technicians who work in the laboratory are duty bound to prevent exposure of themselves and others to unreasonable risks. The primary causes of accidents in the laboratory are **careless techniques, unawareness of potential danger**, **improper procedures, sloppy work habits and lack of sufficient concern for others**.

Laboratory experiments can be performed with a minimum of accidents by following established procedures.

EYE PROTECTION *must be worn at ALL TIMES in areas where chemicals are being used or stored.* Safety glasses should have shatterproof lenses with splash shields. Contact lenses are generally not safe since they can absorb chemicals that irritate the eye. If chemicals get in your eyes **flood them immediately with water** and then seek medical attention.

KNOW WHAT TO DO IN THE CASE OF AN ACCIDENT. *Acquaint yourself with the LOCATION and PROPER USE of emergency equipment such as* first-aid kit, fire extinguishers, fire blankets, safety showers and eyewash stations.

READ CAREFULLY THE EXPERIMENTAL PROCEDURE PRIOR TO CLASS
Make note of safety warnings and precautions to be taken, equipment and techniques to be used and the nature of the chemical substances i.e. whether they are corrosive, volatile or poisonous. In the event of a spill apply proper procedure for cleanup and disposal. Carefully follow the recommended manner of waste disposal of chemical reagents and products.

USE THE FUMEHOOD WHEN HANDLING VOLATILE CHEMICALS WITH TOXIC AND/OR IRRITATING FUMES OR WHEN CONDUCTING EXPERIMENTS INVOLVING SUCH VOLATILE FUMES.

REPORT ALL ACCIDENTS IMMEDIATELY TO THE LECTURER IN CHARGE OR ALERT THE NEAREST PERSON IN CASE OF AN EMERGENCY.

NEVER WORK ALONE IN THE LABORATORY OR WITHOUT IMMEDIATE ACCESS TO ANOTHER PERSON.

KEEP YOUR WORK SPACE **ORDERLY AND UNCLUTTERED**.

WASH YOUR HANDS FREQUENTLY DURING AND AT THE END OF THE LABORATORY SESSION. Fumes and chemicals may produce skin irritations after long exposure.

READ LABEL OF CHEMICAL AT LEAST THREE TIMES: when **OBTAINING** chemical, when **MEASURING** and when **RETURNING** to its place of origin.

ALWAYS REMEMBER TO REPLACE STOPPER OR LID OF CHEMICAL CONTAINER AFTER USE AND TO WIPE OUTSIDE OF CONTAINER AND WORK AREA IF ANY SPILLS HAVE OCCURRED.

NO FLAMES SHOULD BE USED DURING SESSIONS INVOLVING THE USE OF FLAMMABLE MATERIALS, ESPECIALLY THOSE WITH LOW BOILING POINTS.

NEVER POINT THE OPEN END OF TEST TUBES WITH REACTION MIXTURES AT ANYONE, INCLUDING YOURSELF.

NEVER ADD WATER TO CONCENTRATED ACID. THE ACID SHOULD ALWAYS BE ADDED SLOWLY TO THE WATER WHILE MIXING, SO THAT ANY HEAT GENERATED CAN BE ABSORBED AND DISSIPATED.

ALWAYS BE PROPERLY ATTIRED; USE LABORATORY COATS, COMFORTABLE WHOLE SHOES (TOES AND INSTEP COVERED) AND KEEP HAIR TIED BACK. USE DISPOSABLE GLOVES WHEN ADVISED TO DO SO.

DO NOT EAT, DRINK OR SMOKE IN THE LABORATORY.

REGARD EVERY CHEMICAL AS POTENTIALLY HAZARDOUS AND VIGOROUSLY FOLLOW EVERY SAFETY PROCEDURE DESCRIBED; THEY ARE NOT SUGGESTIONS, THEY ARE MANDATORY.

BE IN ATTENDANCE FOR LABORATORY INSTRUCTIONS AT THE BEGINNING OF EACH SESSION AND NOTE SPECIAL PRECAUTIONS TO BE TAKEN AND/OR CHANGES TO THE PROCEDURE, IF ANY.

ACCIDENTS ARE USUALLY PREVENTABLE. BE ALERT IN THE LAB. Understand experimental procedures and be aware of any hazards. ASK YOUR INSTRUCTOR TO EXPLAIN OPERATIONS YOU DO NOT UNDERSTAND. DO NOT PROCEED UNTIL YOU KNOW WHAT YOU NEED TO DO.

BEFORE LEAVING THE LABORATORY ENSURE THAT ALL EQUIPMENT AND ALL UTILITIES ARE TURNED OFF AND THAT YOUR WORK AREA IS TIDY.

IN CASE OF ACCIDENT

Always call or notify the laboratory supervisor as soon as possible.

Fire

1. ***Burning Reagents:*** Immediately extinguish any gas burners in the vicinity. Fire extinguishers are available in various parts of the laboratory. For burning oil use **powdered sodium bicarbonate.**

2. ***Burning Clothing:*** Avoid running (which fans the flame) and take great care not to inhale the flame. Rolling on the floor is often the quickest and best method for extinguishing a fire on one's own clothing. Smother the fire as quickly as possible using wet towels, laboratory coats, heavy (fire) blankets, or carbon dioxide extinguisher.

3. ***Treatment of Small Burns:*** Submerge the burned area in cold water until the pain subsides. Blot the area dry, gently, with sterile gauze and apply a dry gauze as a protective bandage. In small second or third degree burns in which blisters have formed of broken, or in which deep burns are encountered, see a physician as soon as possible.

4. ***Extensive Burns:*** These require special treatment to avoid serious or fatal outcome---*summon medical treatment at once.* Combat the effects of shock by keeping the patient warm and quiet.

Injuries and Chemical Burns

5. ***Reagents in the Eye:*** Wash immediately with a large amount of water, using the ordinary sink hose, eye-wash fountain, or eye-wash bottle----*do not touch the eye.*
   After the eye has been washed thoroughly for 15 minutes, if any discomfort remains, see physician.

6. ***Reagents on the Skin:*** *Acids---Wash* immediately with a large amount of water, then soak the burned part in ***sodium bicarbonate solution*****.** Cover the burned area with a dressing bandage and see a physician.

   Alkali---Wash immediately with a large amount of water, then soak the burned area in ***1% boric acid solution*** to neutralize the alkali. Cover the burned area with a dressing and see a physician.

   Bromine---Wash immediately with a large amount of water, then soak the burned area with a dressing in ***10% sodium thiosulphate***, or cover with a wet sodium thiosulphate dressing, for at least 3 hours and see a physician.

Organic Substances---Most organic substances can be removed from the skin by washing immediately with ordinary ***ethanol***, followed by washing with soap and warm water. If the skin is burned (as by phenol), soak the injured part in water for at least 3 hours and see a physician.

7. ***Cuts:*** Wash the wound with sterile gauze, soap, and water. Cover with a sterile dressing and keep dry.

WASTE DISPOSAL

IN AN EFFORT TO MINIMIZE THE AMOUNT OF HAZARDOUS WASTES GENERATED IN ACADEMIC LABORATORIES CERTAIN PROCEDURES HAVE BEEN INCORPORATED INTO THE EXPERIMENTS TO ACCOMPLISH THIS.

1. **THE SCALE OF THE EXPERIMENTS HAS BEEN REDUCED.**

2. **HAZARDOUS CHEMICALS HAVE BEEN REPLACED BY LESS HAZARDOUS CHEMICALS**

3. **INSTRUCTIONS HAVE BEEN GIVEN FOR RECOVERING CHEMICALS FOR RE-USE, WHENEVER PRACTICAL**.

SPECIAL INSTRUCTIONS FOR THE DISPOSAL OF WASTES ARE PROVIDED WITH THE EXPERIMENTS, WHERE NECESSARY.

SOLUTIONS ACCEPTABLE FOR SINK DISPOSAL SHOULD BE DILUTED ONE HUNDREDFOLD BEFORE BEING SLOWLY POURED DOWN THE DRAIN. ALTERNATIVELY THE SOLUTION MAY BE POURED DOWN THE SINK ALONG WITH A STREAM OF WATER.

SMALL AMOUNTS OF WATER-SOLUBLE ORGANIC SOLVENTS MAY BE DILUTED AND FLUSHED DOWN THE DRAIN.

WHEN IN DOUBT, CHECK WITH YOUR DEMONSTRATOR BEFORE DISPOSING ANY CHEMICAL.

LARGE AMOUNTS OF ORGANIC SOLVENTS SHOULD BE PLACED IN PROPERLY LABELLED BOTTLES FOR RECYCLING OR DISPOSAL.

BROKEN GLASS APPARATUS SHOULD BE PLACED IN SPECIALLY DESIGNATED BINS FOR RECYCLING.

POINTS FOR GRADING.

1. Ensure that you properly clean your **workspace**, the **fume hoods** and the **centre bench** (which usually has the balances and communal reagents) at the end of the lab. A majority of students forget the fume hood as well as the centre bench. However significant marks in technique will be deducted if a clean work environment is not maintained at the end of a lab session. If the fume hoods and centre bench are left in an unclean state at the end of the laboratory session, marks will be deducted from the entire class. Marks will also be deducted for leaving reagent bottles uncovered as well as leaving your apparatus at the centre bench or fume hood. You thus have significant control in maintaining your technique marks.

Remember that persons are not attracted to dirty Pharmacies!

2. Never begin any experiment unless you understand its overall purpose and reasons for each operation involved in the experiment.

3. Always be neat and careful in handling chemicals, and in the assembly of apparatus.

4. Cultivate the habit of being observant during all stages of an experiment. (Was there a fleeting colour change when a drop of reagent was added to the solution? Was a precipitateformed? Is the reaction exothermic? Is your neighbour's technique "better" than your own?)

5. Record your observations and results in a **bound notebook.** Write in ink. Number your pages of your notebook.

6. Your notebook should show the date and title of each experiment. Your report should show the quantities of material used, the melting (or boiling) point of the product obtained, the weight of the product and any other relevant data.

7. Your notebook should show each new experiment on a new page.

8. Save the final products from each experiment for examination by your demonstrator.

9. When you have completed your lab, and properly cleaned your workstation, fume hood and centre bench area, ensure that the lecturer (or instructor/demonstrator/technician/lab assistant) signs your results. Also hand in a single Master Copy of the lab results to the demonstrator.

MEASUREMENT OF MASS

Your Lecturer, demonstrator or technician will demonstrate **the correct use of the balances** – top loading and analytical – and will help you understand the principles of operation. The general rules to follow when making weighings are:

i. Use an assigned balance for all your determinations.
ii. Always level and zero balance before use.
iii. Consult an instructor if you need assistance.
iv. Clean up any spilled chemicals on balance immediately.
v. Leave the balance area free and clean of all chemicals, containers, paper etc.

MEASUREMENT OF VOLUME

The volume of a liquid can be measured easily and accurately by means of specialized laboratory glassware called volumetric glassware: graduated cylinders, volumetric flasks, pipettes, burettes. The proper use of glassware will be demonstrated to you.

SEPARATION TECHNIQUES

Separation may be defined as an operation that results in the isolation and purification of a single chemical constituent or phase. The process of separation may be divided into two general categories depending upon the complexity of the method used. The first category includes separation carried out by a mechanical operation such as distillation, solvent extraction, sieving, centrifugation and filtration. The second category of separations includes those carried out with the aid of chemical methods.

There are two kinds of filtration: GRAVITY FILTRATION
VACUUM FILTRATION

Vacuum filtration is more rapid and efficient than gravity filtration. In this technique the filter paper rests flush against the flat bottom of a Buchner funnel. The funnel is sealed to the top of a filtration flask by a rubber stopper or gasket ring. The sidearm of the flask is connected to a water aspirator or vacuum line by heavy-walled tubing. This connection is sometimes made through a "trap" jar to prevent the direct transfer of liquids between the flask and the source of vacuum. The vacuum source greatly reduces the presence in the filtration flask, causing the air pressure above the funnel to push the mixture through the filter paper. The resulting force, causing the liquid to pass through the filter paper, is much greater than the gravitational force used alone in gravity filtration. The advantages are that large quantities of solid can be removed in less time than by gravity alone.

FUNDAMENTALS OF MEASUREMENT AND DATA EVALUATION

All measurements are subject to error. Experimental results are only as precise as the measurements used to obtain them. Reported results should indicate the estimated magnitude of the experimental error.

Experimental errors may be classified as belonging to one of the following three types:

1. **Systematic errors** result from faulty calibration of a measurement device. Misapplication of a measurement technique, or use of an inappropriate mathematical model. A meter stick might be 90 cm long yet still be divided into 100 cm units; the mass of an object might be read directly from a balance that has not been previously zeroed; the area of a triangle might be incorrectly calculated as the product of base times height. The effects of systematic errors are predictable and correctable if the existence of the errors is recognized.

2. **Random errors** result from a large number of unknown and unpredictable variations in measurement. They include the effects of observer judgements in deciding when and how to read scales, temperature fluctuations, and mechanical vibrations. Random errors often follow simple laws. The presence of random errors can be detected and their effects minimized by replicate measurements.

3. **Careless errors** are made by the inattentive observer who misreads graduated glassware, copies data incorrectly and makes mistakes in mathematical calculations. These errors are totally avoidable and must be eliminated.

ACCURACY AND PRECISION

Precision and accuracy are two terms which have quite different meanings. The **precision** of a measurement is the degree of agreement between it and other measurements made under the same conditions. The **accuracy** of a measurement is how closely it agrees with the accepted or true value.

The precision of a measurement is affected by random errors, which are caused by the inability of a person to read and instrument accurately. The errors arise in the interpolation between the scale divisions. The readings are either higher or lower than the actual value. The precision of some instruments is given as follows:

0.02 mL for the 50 mL burette; 0.2 mL for the 50 mL graduated cylinder;

0.2° for the 110°C thermometer; 0.03 mL for the 25 mL transfer pipette;

0.2 mL for the 250 mL volumetric flask.

Results may have a high degree of precision without being accurate. But an accurate result must be obtained from measurements made with high precision. **Hence you should strive for both precision and accuracy in the laboratory.** In order to illustrate this idea where three separate analyses were made by each of students A and B on a sample containing 53.26% iron (Fe). Student A obtained values of 55.86, 55.92 and 55.89 or a mean value of 55.89% with average deviation of ± 0.02% of the measurements from the mean value. Student B obtained values of 53.09, 56.28 and 50.56 or a mean value of 53.31% with average deviation of ± 1.98%.

From such data, student A's precision is high, but his average is by no means accurate. Student B's average is very close to the true percentage, but precision is poor. Therefore student B's result, though accurate, is **NOT** reliable.

SIGNIFICANT FIGURES

One method of indicating uncertainty is to include only **significant figures or digits** in numerical expressions. Only the last digit in a number should be in doubt. Zeros are assumed to be significant only when they are not needed in order to position the decimal point. Table 1 includes numerous examples of determining the number of significant figures in experimental data.

TABLE 1 Significant Figures (digits)

Measurement	Significant Digits	Comments
5.630	4	Trailing 0 counted
0.270	3	Leading 0 not counted
1.0060	5	Each 0 counted
0.0004	1	Leading 0's place holders
-5002	4	–
8.1 x 10^7	2	first factor only
9.65	3	–
0856	3	leading 0 not counted
3 x 10^{-6}	1	first factor only
3600	2,3, or 4	0's may be holding decimal place
3.60 x 10^3	3	–

After all computations, the final answer should contain the proper number of significant figures in order to express the degree of reliability of the result obtained.

In addition and subtraction, the last digit retained in the answer should correspond to the first uncertain decimal place in any of the numbers. For example the sum of the following numbers, namely, 32.7, 1.38, 0.423 is 34.5 (Since 32.7 + 1.4 + 0.4 = 34.5).

In rounding off numbers, increase the last digit by 1 if the digit which is to be discarded is 5 or greater. If the rejected digit is less than 5, do not change the last significant digit.

In multiplication and division, the answer should not have more significant figures in any of the numbers entering into the computation. For example, the product of 0.112 x 14.73 x 23.169 is 38.1 (The number with the largest relative deviation is the first number and the value is 1/112 times 100 = 0.9%. This means that the product can vary by 9 parts per thousand or by 0.3 in 38.1).

ABSOLUTE AND RELATIVE ERRORS

In reporting the result of an experiment, the mean value or average of a number of determinations is normally used, because it is more accurate than the individual determinations.

The difference between the mean value obtained experimentally and the true value is the absolute error. When this error is expressed in terms of percentage or parts per thousand, it is called the relative error.

THE STUDENT MUST ALWAYS USE THESE CONCEPTS TO FACILITATE HIS WORK

EXPERIMENT 1 STANDARDIZATION OF HYDROCHLORIC ACID (USING SODIUM CARBONATE)

Objectives:

At the end of this session you should be familiar with the following:

i) The principles upon which volumetric analyses are based.
ii) The characteristics of primary standard solution.
iii) The mole concept.

Task:

In today's laboratory session you are required to perform the following:

1. Prepare a standard solution of anhydrous sodium carbonate.
2. Standardize a solution of hydrochloric acid using the sodium carbonate solution in A.

A. Preparation of a Standard Solution of Sodium Carbonate

Procedure:

Weigh out accurately, approximately 1.3 g. of anhydrous sodium carbonate into a volumetric flask (250 mL). To do this, first weigh out approximately 1.3 g of the sodium carbonate in a sample bottle using the top loading balance provided. Then using the analytical balance weigh accurately to four decimal places, sample bottle and contents. Transfer quantitatively into volumetric flask by decanting small amounts of sample from bottle into a funnel and washing carefully into flask with distilled water. Shake to completely dissolve the sodium carbonate and then carefully make up to the graduation mark with distilled water. Re-weigh sample bottle, record all mass in your note-book and determine mass of sodium carbonate used by the method of difference.

Results

Mass of bottle and salt =

Mass of bottle =

Mass of salt =

B. Standardization of Hydrochloric Acid

Procedure:

Pipette 25 mL aliquots of the standard sodium carbonate solution prepared in Exercise A above into conical flasks (250 mL). Titrate these samples with the hydrochloric acid in the burette using methyl orange as indicator. Repeat until concordant results are obtained. Record your results in tabular form in your note-book, as indicated below.

BURETTE READINGS	**ROUGH**	**1**	**11**
Final volume (mL)			
Initial volume (mL)			
Volume acid used			

WORKSHOP SESSIONS

PHARMACEUTICAL CALCULATIONS

The Properties of solutions: Measures of Concentration

I Normality and Molarity

A <u>normal</u> solution is one which contains one gramme-equivalent of the active substance in 1000 mL. An equivalent (eq) in neutralization is the mass of acid that yields 1 mol of H^+ or the mass of base that reacts with 1 mol of H^+

e.g. $H_2SO_4 + 2NaOH \longrightarrow Na_2SO_4 + 2H_2O$

Each mole of H_2SO_4 supplies 2 mol of H^+. Because the <u>molar mass</u> is 98.1g, an equivalent of H_2SO_4 in this reaction equals 98.1g/2 = 49.0g. Because each mole of NaOH reacts with 1 mole H^+, an equivalent of NaOH = the <u>molar mass</u> (40.0g). One eq of H_2SO_4 reacts with one eq of NaOH.

However, in the reaction

$$H_2SO_4 + NaOH \longrightarrow NaHSO_4 + H_2O$$

each mole H_2SO_4 supplies only 1 mol H^+. Therefore an equivalent of H_2SO_4 in this reaction equals the molar mass of 98.1g.

An equivalent (eq) in a <u>redox reaction</u> is the mass of oxidizing or reducing agent that uses or provides one mole of electrons. Thus one eq of an oxidizing agent reacts with one eq of reducing agent. For example the permanganate ion, MnO^{4-} gains 5 electrons when it is reduced to Mn^{2+} in acidic solution

$$MnO_4^- + 8H^+ + 5e \longrightarrow Mn^{2+} + 4H_20$$

An eq of $KMnO_4$ is obtained by dividing its molar mass (158.0g) by the number of electrons gained (5), i.e. 158.0g/5 = 31.6g

The <u>Normality</u> of a solution is the number of equivalents of a substance dissolved in a litre of solution. It equals the <u>molarity</u> of the solution times the number of equivalents per mole. For example, if the reaction is an oxidation by $KMnO_4$ in acidic solution, the normality of 0.10 M $KMnO_4$ is

$$\text{Normality} = \underbrace{\frac{\text{moles of substance}}{\text{Litres solution}}}_{\text{M}} \times \frac{\text{equivalents substance}}{\text{moles substance}}$$

$$= 0.1 \frac{\text{mol } KMnO_4}{\text{L solution}} \times \frac{5 \text{ eq } KmnO_4}{\text{mol } KmnO_4}$$

$$= 0.50 \text{ N } KMnO_4$$

A molar solution (designated M) is one which contains the gram-molecular weight of the substance in one litre of solution. The strengths of molar solutions are independent of the reaction under consideration.

II Volume percent composition and mass percent composition

Volume percent composition

Many solutions are prepared by measuring the volume of a solute, instead of by its mass, and mixing it with the solvent. A convenient expression of the resulting composition is in terms of the volume percent composition., the volume of a substance present in a solution expressed as a percentage of the total volume. Volume percent is the basis of the measurement of alcohol (ethanol) content of beers, wines and spirits. A typical wine has about 15% by volume alcohol (i.e. 100 mL wine contains 15 mL ethanol). The proof scale equates 100 proof with 50% by volume ethanol.

When there is only a small quantity of solute in a solution, and particularly when the solution is gaseous (e.g. a pollutant in air) the composition is often expressed in parts per million by volume (ppm by volume) , which is 10^6 times the ratio of the volume of the solute to the volume of the sample. It follows that parts per million by volume is numerically equal to the volume percentage composition multiplied by 10^4. Thus a SO_2 concentration expressed as 42 ppm by volume would correspond to 42 mL of SO_2 in a sample of volume 10^3 L.

Mass percentage composition

The mass of one substance present in a mixture expressed as a percentage of the total mass:

$$\text{Mass percentage of A} = \frac{\text{mass of A in solution}}{\text{total mass of solution}} \quad \text{X} \quad 100\%$$

$$= \frac{m_A}{m_A + m_B + \ldots} \quad \text{X} \quad 100\%$$

This measure is often used when reporting the composition of a solution of a solid solute. For example, we may report the composition of a solution of sucrose in water as being 22.1% by mass of sucrose. When the mass of solute is very small, a widely used measure of composition is parts per million by mass (ppm by mass). For the analysis of pollutants in water, part per million is 10^6 times the ratio of the mass of the solute to the mass of the sample. In practice it is the mass of solute in milligrams per kilogram of sample (mg/kg).

III Mole Fraction and Molarity

The mole fraction, x, of a species in a mixture is the number of moles of that species expressed as a fraction of the total number of moles of ions and molecules in the mixture.

For solute molecules in a nonelectrolyte solution, mole fraction is

$$x_{solute} = \frac{n_{solute}}{n_{solvent} + n_{solute}}$$

where n_{solute} is the number (in moles) of a solute present and $n_{solvent}$, the amount (in moles) of solvent. A similar equation defines the mole fraction of the solvent molecules $x_{solvent}$. A useful relation between the two is

$$x_{solvent} + x_{solute} = \frac{n_{solvent}}{n_{solvent} + n_{solvent}} + \frac{n_{solute}}{n_{solvent} + n_{solute}} = 1$$

A mole fraction must lie in the range $x_{solute} = 0$ (no solute) to $x_{solute} = 1$ (pure solute).

For an electrolyte solution, the mole fraction is calculated by treating the cations and anions as individual particles: hence

$$x_{cations} = \frac{n_{cations}}{n_{solvent} + n_{solvent} + n_{anions}}$$

IV Molality

The molality of a solution also emphasizes the relative numbers of solute and solvent molecules. The <u>molality</u> of a solution is the amount of solute per kilogram of solvent:

$$\text{Molality} = \frac{\text{amount (mol) of solute}}{\text{mass (kg) of solvent}}$$

The units of molality, moles per kilogram of solvent (mol/kg), are often abbreviated as *m* and read “molal”. The emphasis on solvent in the definition should be noted. It means that to prepare a 1 *m* $NiSO_4$ (aq) solution, 1 mol of $NiSO_4$ is dissolved in 1 kg of water. Because 1 kg of solvent consists of a definite number of moles of molecules (55.5 mol H_2O per kilogram of water), the higher the molality of a given solution, the higher the proportion of solute molecules.

The molality is a conversion factor from the mass of solvent in a sample to the number of moles of solute in it:

$$\text{amount of solute (mol)} = \text{mass of solvent (kg)} \times \frac{\text{amount (mol) of solute}}{\text{mass (kg) of solvent}}$$

The molality of a solution must be carefully distinguished from its molarity (its molar concentration). The molality is the moles of solute per kilogram of solvent. The molar concentration is the moles of solute per liter of solution. There are two good reasons for introducing molality in addition to molar concentration. One is that the molality makes it very easy to calculate the relative numbers of solute and solvent molecules in a solution. The second is the molality is independent of temperature, whereas the molar concentration is not (because the volume of the solution increases as the temperature is raised).

The relation between the volume of a solution and the mass of solvent present in it is obtained by using the density of the solution(to obtain the total mass of the solution), the mass of solute (which is known from the molar concentration and the molar mass of the solute), and the relation

$$\text{mass of solution} = \text{mass(solvent)} + \text{mass(solute)}$$

to obtain the mass of solvent.

V Percentage Concentrations

Before we use mole fractions or molalities in a study of certain physical properties of solutions, we will complete the list of the chief ways by which chemists describe concentrations. The rest of the list are various kinds of percent concentrations.

In most chemical laboratories, a percent concentration means percent by weight or weight/weight percent. The symbol for it is usually % (w/w). This is the number of grams of solute per 100 grams of solution (not solvent). A solution labeled “0.9% (w/w) NaCl”, for example, is one in which the ratio of solute to solution is 0.9g of NaCl to 100g of solution.

EXAMPLES

USING WEIGHT/WEIGHT PERCENTS

Problem How many grams of a 4.00% (w/v) solution of NaCl is needed to obtain 0.500 g of NaCl?

Solution The given concentration gives us the following conversion factors:

$$\frac{4.00 \text{ g NaCl}}{100 \text{ g solution}} \quad \text{and} \quad \frac{100 \text{ g solution}}{4.00 \text{ g NaCl}}$$

We want 0.500 g of NaCl from this solution, so we use the second conversion factor.

$$0.500 \text{ g NaCl} \ \text{X} \ \frac{100 \text{ g solution}}{4.00 \text{ g NaCl}} = 12.5 \text{ g of } 4.00\% \text{ (w/w) solution}$$

Thus if we take 12.5 g of the 4.00% (w/w) NaCl solution we will also be taking 0.500 g of NaCl.

PREPARING WEIGHT/WEIGHT PERCENT SOLUTIONS

Problem "White vinegar" can be made by preparing a 5.0% (w/w) solution of acetic acid in water. How would you make 500 g of such a solution?

Solution The mass of acetic acid first has to be calculated. Then it has to be combined with enough water to make the total solution have a mass of 500 g. To find the mass of solute, we use the given concentration to give us our choice of conversion factors. Thus, 5.0% (w/w) acetic acid means:

$$\frac{5.00 \text{ g acetic acid}}{100 \text{ g solution}} \quad \text{and} \quad \frac{100 \text{ g solution}}{5.00 \text{ g acetic acid}}$$

We have to use the first factor to get the units to work out.

$$500 \text{ g solution} \ \text{X} \ \frac{5.0 \text{ g acetic acid}}{100 \text{ g solution}} = 25 \text{ g acetic acid}$$

Thus if we dissolve 25 g acetic acid in enough water to make the final mass equal to 500 g , we would have a 5.0% (w/w) solution of acetic acid – the same as white vinegar. This, of course, would require 475 g of water (500 g –25 g).

NOTE More than two-significant-figure precision is not often sought when solutions are made up to a percent concentration.

Because we know that the density of water at room temperature is (to two significant figures) 1.0 g/mL, we can easily see that 475 g of water needed in this example is the same as 475 mL of water. When greater precision is not needed, a chemist will generally make this mental calculation and then mix 25 g of acetic acid with 475 mL of water to make the 5.0% (w/w) acetic acid solution. It is just easier to measure a large volume than a large mass because large graduated cylinders are readily available.

When both solute and solvent are liquids, or both gases, then it can be converted to use percent by volume – symbolized as % (v/v) – which is the number of volumes of one component in 100 volumes of the entire solution. (Notice again that the emphasis is on the volume of the final *solution*.) The unit of volume can be any unit, provided that the same unit is used for both solute and solution. (True percentages, of course, have no formal units because they cancel in the final calculation, but we still have to remember how each kind of percent concentration is defined.)

USING VOLUME/VOLUME PERCENTS

Problem A 40% (v/v) solution of ethylene glycol in water gives an antifreeze that will protect a vehicle's cooling system to -24°C (-12°F). What volume of ethylene glycol has to be used to make 5 quarts of this solution?

Solution First, we use the given concentration to construct conversion factors. Thus, "40% (v/v) ethylene glycol " means:

$$\frac{\text{40 vol ethylene glycol}}{\text{100 vol solution}} \quad \text{and} \quad \frac{\text{100 vol solution}}{\text{40 vol ethylene glycol}}$$

We have to use the first factor to get the answer in units of ethylene glycol. And we substitute "quarts" for "vol" now.

$$\text{5 quarts solution} \quad \text{X} \quad \frac{\text{40 quarts ethylene glycol}}{\text{100 quarts solution}} = \text{2 quarts ethylene glycol}$$

> Thus if we dissolve 2 quarts of ethylene glycol in enough water to make the final volume of the solution 5 quarts, the antifreeze will be 40% (v/v) ethylene glycol. (Incidentally, we could not assume that all we had to do was add 2 quarts of ethylene glycol to 3 quarts of water to make 5 quarts of solution. When two liquids mix, the final volume is sometimes less than the sum of their initial volumes, and sometimes more. Sometimes it is the same, but this cannot be assumed. It depends on how the individual molecules find room for each other in the solutions.)

Some fields of science employ still other kinds of percent concentrations, but they are not true percents (the units do not easily cancel) and they are not preferred by chemists. In case you might run into them we have described a few in the Special Topic.

SPECIAL TOPIC: SOME HYBRID PERCENT CONCENTRATIONS

By "hybrid percent concentration" we mean one in which the units do not cancel. For example, a **weight/volume percent concentration** gives the grams of solute in 100 mL of *solution*. When the solvent is water and the solution is dilute, the numerical value of a weight/volume percent concentration is virtually the same as that of a weight/weight percent.

People in the health fields sometimes use a **milligram percent concentration**, which gives the number of milligrams of solute in 100 mL of solvent.
Whenever a percent concentration is given without any specification about its type, assume that it means a weight/weight percent.

CONVERTING AMONG CONCENTRATION UNITS

Many made-up solutions are available from chemical supply houses and stockrooms, as you have probably already seen in the lab. Sometimes in working with a solution whose molarity is known we run into a situation where we would like to know its molarity or its percent (w/w) concentration, too. Is there any way we can calculate one kind of concentration from another?

The only additional information we need for some calculations are formula weights and densities, which are often given in tables in reference handbooks for chemistry. These conversions are easy, ***provided you know the definitions of each kind of concentration*** and can prepare conversion factors from given concentrations. In fact, working through these examples and doing practice exercises is a superb way to get a final and lasting grasp of all the concentration expressions.

EXAMPLES

CONVERTING FROM WEIGHT PERCENT TO MOLE FRACTIONS

Problem What are the mole fractions and mole percents of the components in 10.0% (w/w) NaCl, the same solution we used in the previous example?

Solution Our goal is to move the units from those of percent (w/w),

$$\frac{\text{g solute}}{\text{100 g solution}}$$

to those implied by a mole fraction,

$$\frac{\text{mol solute}}{\text{total moles}}$$

CONVERTING FROM WEIGHT PERCENT TO MOLALITY

Problem What is the molality of a 10.0% (w/w) NaCl solution?

Solution We are given a ratio of 10.0 g of NaCl to 100 g of *solution*. This is what the percent (w/w) means. But the units of molality are

$$\frac{\text{mol of solute}}{\text{kg of solvent}}$$

What we need to do, then, is use the units of the percent concentration to work out the units of molality. The steps are

Step 1. Calculate the moles of NaCl in 10.0 g of NaCl.
Step 2. Calculate the kilograms of H_2O in 100 g of this *solution*. (Remember it is grams of *solution* with percent concentrations, but kilograms (or1000 g) of *solvent* with molality.)
Step 3. Calculate the ratio of these, because molaltiy is the ratio of moles of solute to kilograms of solvent.

We find the moles of NaCl in 10.0 g of NaCl in the usual way, using the molar mass of NaCl, 58.5 g NaCl/mol NaCl.

$$10.0\text{ g NaCl} \quad \text{X} \quad \frac{1\text{ mol NaCl}}{58.5\text{ g NaCl}} = 0.171\text{ mol NaCl}$$

Next, step 2, we note that 100 g of this *solution*, which has 10.0 g of solute, must therefore have 90.0 g of solvent (100 g – 10 g). So the kilograms of solvent are found by

$$90.0 \text{ g } H_2O \quad \text{X} \quad \frac{1 \text{ kg } H_2O}{1000 \text{ g } H_2O} = 0.0900 \text{ kg } H_2O$$

Remember, both numbers in 1 kg = 1000 g are defined as exact.

Finally, step 3, we calculate *m*, the ratio of moles of solute to kilograms of solvent.

$$\frac{0.171 \text{ mol NaCl}}{0.0900 \text{ kg } H_2O} = 1.90\ m$$

Thus, 10.0% (w/w) NaCl is also 1.90 *m* NaCl.

The amount of solute from the previous example is 0.171 mol of NaCl. We also found in that example that there is 90.0 g H_2O in 100 g of solution. And the moles of water in 90.0 g of H_2O (at 18.0 g H_2O/mol H_2O) is

$$90.0 \text{ g } H_2O \quad \text{X} \quad \frac{1 \text{ kg } H_2O}{18.0\text{g } H_2O} = 5.0 \text{ mol } H_2O$$

So the total number of moles of both solute and solvent in 10.o% (w/w) NaCl is

0.171 mol + 5.0 mol = 5.17 mol (correctly rounded)

Now we can calculate mole fractions of solute and solvent.
For *NaCl*

$$X_{NaCl} = \frac{0.171 \text{ mol}}{5.17 \text{ mol}} = 0.0331 \text{ (or 3.31 mole percent)}$$

For *H_2O*

$$X_{H2O} = \frac{5.00 \text{ mol}}{5.17 \text{ mol}} = 0.967 \text{ (or 96.7 mole percent)}$$

For a two component system like this, we would not need this last calculation. The mole fractions must add up to 1 (or the percents, to 100%), so we could have found the mole fraction of water simply by subtracting the mole fraction of NaCl from 1.00. (Or we could have calculated the mole percent of water by subtracting the mole percent of NaCl from 100.)

CALCULATING PERCENTS BY WEIGHT FROM MOLE FRACTIONS

Problem So-called "100 proof" alcohol has the following mole fractions of components: for water 0.765, and for ethyl alcohol, 0.235. Calculate the percents (w/w) of water and ethyl alcohol in 100 proof ethyl alcohol. The formula weights are: H_2O, 18.0; and ethyl alcohol, C_2H_5OH, 46.1.

Solution As in all kinds of calculations, we work from the meanings of the given concentration units toward the final units. To work toward percents by weight, we have to work toward a ratio calculated from *grams* of components. Let us start with water. When the mole fraction of water is 0.765, we know that there are 0.765 mol of H_2O in a total of 1.00 mol of all components. So, in terms of grams of water, at 18.0 g H_2O/mol H_2O, we have

$$0.765 \text{ mol } H_2O \quad X \quad \frac{18.0 \text{ g } H_2O}{1 \text{ mol } H_2O} \quad = 13.8 \text{ g } H_2O$$

Next, we take ethyl alcohol. When its mole fraction is 0.235, we know that there is 0.235 mol of C_2H_5OH in 1.00 mol of all its components. So, in terms of grams of C_2H_5OH, at 46.1 g C_2H_5OH/mol C_2H_5OH, we have

$$0.235 \text{ mol } C_2H_5OH \quad X \quad \frac{46.1 \text{ g } C_2H_5OH}{1 \text{ mol } C_2H_5OH} = 10.8 \text{ g } C_2H_5OH$$

Thus, in "1.00 mol" of this ethyl alcohol solution there are 13.8 g of H_2O and 10.8 g of C_2H_5OH. To convert to percents by weight, we need the total mass of the solution, which is 13.8 g + 10.8 g = 24.6 g. For the 13.8 g of water, its percentage of 24.6 g is

$$\frac{13.8 \text{ g}}{24.6 \text{ g}} \text{ X } 100 = 56.1\% \text{ (w/w)}$$

APPENDIX I

CONCENTRATIONS OF COMMERCIAL REAGENTS

Concentrated hydrochloric acid	1.19 g cm^{-3} is	12 M
Concentrated nitric acid	1.42 g cm^{-3} is	16 M
Concentrated sulphuric acid	1.84 g cm^{-3} is	18 M
Glacial acetic acid	1.05 g cm^{-3} is	17 M
'0.880' ammonia	0.88 g cm^{-3} is	15 M
'20 volume' hydrogen peroxide (fresh) is approx.		1.8 M
Fresh sodium hypochlorite (15% w/v) is approx.		2 M

APPENDIX 11

Relative Atomic Weights ($^{12}C=12$)

ELEMENT	SYMBOL	ATOMIC NUMBER	ATOMIC WEIGHT
Bromine	Br	35	79.9
Calcium	Ca	20	40.08
Carbon	C	6	12.01
Cerium	Ce	58	140.12
Chlorine	Cl	17	35.45
Chromium	Cr	24	51.99
Copper	Cu	29	63.54
Fluorine	F	9	18.99
Hydrogen	H	1	1.01
Iodine	I	53	114.82
Iron	Fe	26	55.85
Lead	Pb	82	207.19
Magnesium	Mg	12	24.31
Nitrogen	N	7	14.01
Oxygen	O	8	15.99
Phosphorus	P	15	30.97
Potassium	K	19	39.1
Silver	Ag	47	107.87
Sodium	Na	11	22.99
Sulfur	S	16	32.06
Thallium	Tl	81	204.37
Tin	Sn	50	118.69
Zinc	Zn	30	65.370

APPENDIX III

BIBLIOGRAPHY

Beckett, A.H. and Stenlake. Practical Pharmaceutical Chemistry, Third Edition : Part 1

Bentley and Driver. Textbook of Pharmaceutical Chemistry, Eight Edition

Education in Chemistry **(bimonthly)**

Hunt, H.R. and Block, T.F. Laboratory Experiments for General Chemistry

Journal of Chemical Education **(monthly)**

Masterton, W.L. and Hurley, C.N. Principles and reactions, Second Edition.

Milio, F.R. ; Debye, N.W.G. ; Metz, C. Experiments In General Chemistry

U.W.I. Department of Chemistry C10 Laboratory Course

Vogel, A.I. "A Textbook of Macro and Semimicro Qualitative Inorganic Analysis" (Longmans).

Vogel, A.I. "A Textbook of quantitative Inorganic Analysis" (Longmans).

Vogel, A.I. "Elementary Practical Organic Chemistry" (Longmans).