Introduction to Lab Techniques, Apparatus and Preparation of solution

INTRODUCTION

Chemistry is an experimental science. It depends upon careful observation and the use of good laboratory techniques. Most of the experiments in the chemistry laboratory involve quantitative analytical procedures. It involves the use of common glassware for example burette, pipette, volumetric flask, etc. Glassware is used to measure the volume of solutions at a certain temperature. The volume of a liquid changes with temperature. To get the accuracy, the apparatus has to be calibrated before being used.

Mistakes and errors can happen during an experiment. A mistake is a blunder or unintentional action with measurement. The error may be either systematic or random. A systematic error can happen when an apparatus that is not calibrated is used. The measurement will always be too large or too small. A systematic error will influence the accuracy of a measurement, that is the agreement between a measured value of a quantity and its true value. A random error will be evidence of undesirable consequences. Error, on the other hand, accounts for the range of values obtained from successive measurements of the same quantity, even though there was no mistake in any of them when a measuring device, even a very accurate one, is used a number of times to make the same measurement. Both errors can be reduced by using a calibrated apparatus and by being careful when conducting experiments.

Volumetric Flask

A volumetric flask is a glassware designed to deliver the standard solution at precise known volume of liquid at a given temperature. The actual volume of liquid in the flask can be determined by weighing the flask when it is empty and when it is filled with distilled water. The difference between both readings is equal to the mass of water. The volume of water in the flask can be calculated by referring to Table 1. Volumetric flasks are used to make solutions of known concentration by the dissolution of a known mass of solid or the dilution of a more concentrated solution.

Before use, always wash the flask and then pre-rinse it with a solvent. Some frequently used volumes in chemistry laboratories are 10.00, 25.00, 50.00, 100.00, and 250.00 mL flasks. At times the zeros to the right of the decimal point are omitted. However, these zeros must always be considered in calculations, as they indicate the accuracy of the volume measurement.

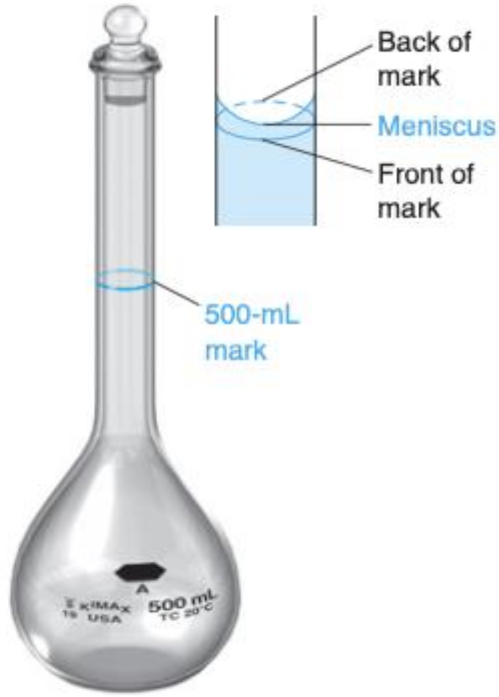

Figure 1: 500 mL of volumetric flask and the meniscus level.

b) Burette

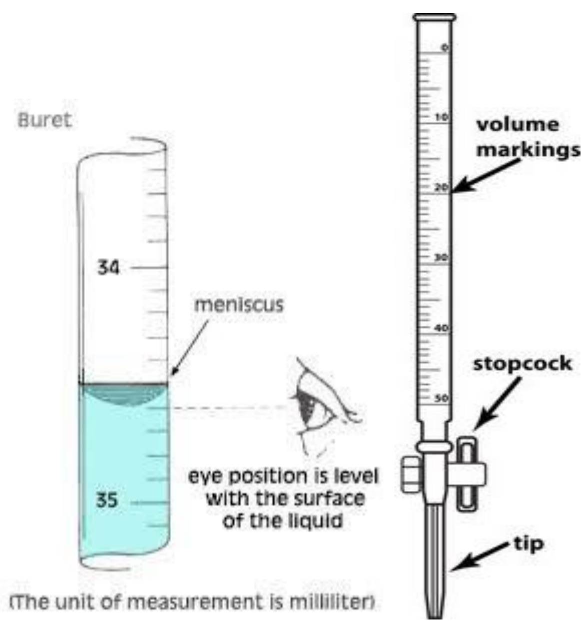

Figure 2: 50 mL pipette and the meniscus level.

A burette is a vertical cylindrical piece of laboratory glassware with a volumetric graduation on its full length and a precision tap, or stopcock, at the bottom. It is used to dispense the known amount of a liquid reagent in experiments for which precision is necessary, such as a titration experiment. Burettes are extremely accurate - 50 mL burette has a tolerance of 0.1 mL (class B) or 0.06 mL (class A). The difference between the initial and final volume is the amount dispensed. The spacing between the lines will allow you to estimate the volume to the nearest 0.01 mL. Thus, typical burette readings would be two decimal points eg 9.34 mL or

17.60 mL reading such as 9.3 mL or 17.6 mL is not acceptable. The following are steps that will help you to have a burette that operates as it should:

1. Clean and rinse the burette with tap water, then distilled water.
2. Rinse the burette with about 5-10 mL of solution.
3. Fill the burette above the zero mark with the stop cork closed.
4. Open the stopcock fully so that the liquid drains rapidly to release an air bubble at the tip of the burette.
5. Drain the liquid until the meniscus rests at a certain number, e.g. 1 mL marks (or 0 mL).
6. Read the burette to two decimal places with your eyes at the same level at the meniscus.
7. To obtain the volume of the solution (liquid) that you use in titration, subtract the initial reading from the final reading.

To calibrate the burette, transfer several volumes of solution from the burette and weigh an accurately. From the density of the solution, we can calculate the volume of the solution that has been transferred.

Table 1.1 : Density (g/mL) of Water at Various Temperature(ºC)

Temperature (ºc)	Density of Water (g/mL)
22	1.0032
23	1.0034
24	1.0037
25	1.0039
26	1.0042
27	1.0045
28	1.0047
29	1.0050
30	1.0053
35	1.0059

Standard solution:

A standard solution is a solution whose concentration is known accurately. Its concentration is usually given in mol dm^{-3}. When making up a standard solution it is important that the correct mass of substance is accurately measured. It is also important that all of this is successfully transferred to the volumetric flask used to make up the solution

Preparing Solutions:

Solutions of known concentration can be prepared in a number of different ways depending on the nature of the analyte and/or the concentration required:

- Weighing out a solid material of known purity, dissolving it in a suitable solvent and diluting to the required volume
- Weighing out a liquid of known purity, dissolving it in a suitable solvent and diluting to the required volume
- Diluting a solution previously prepared in the laboratory
- Diluting a solution from a chemical supplier.

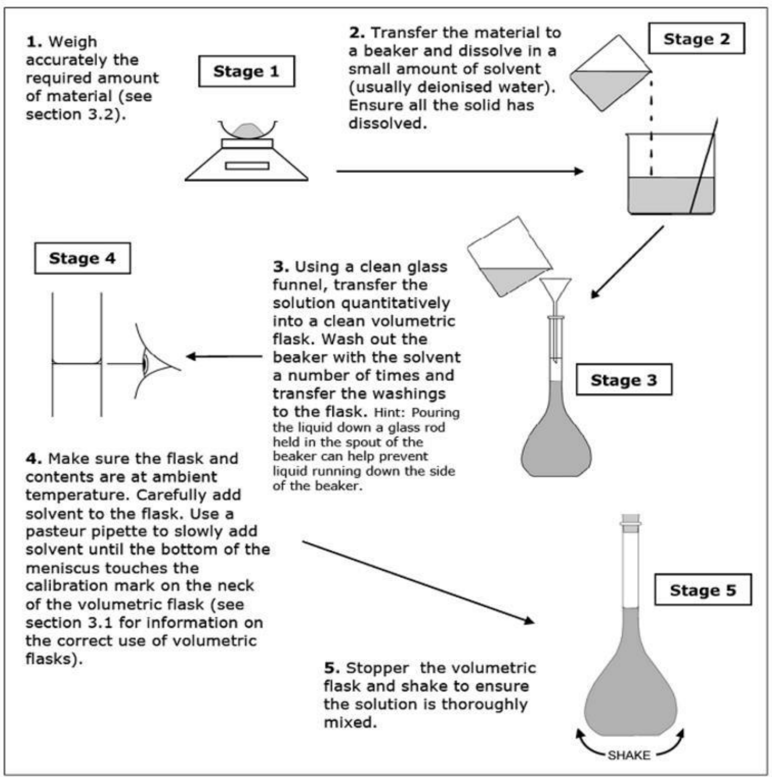

Figure 3: Procedure for preparing a solution of known concentration from a known amount of a solid material

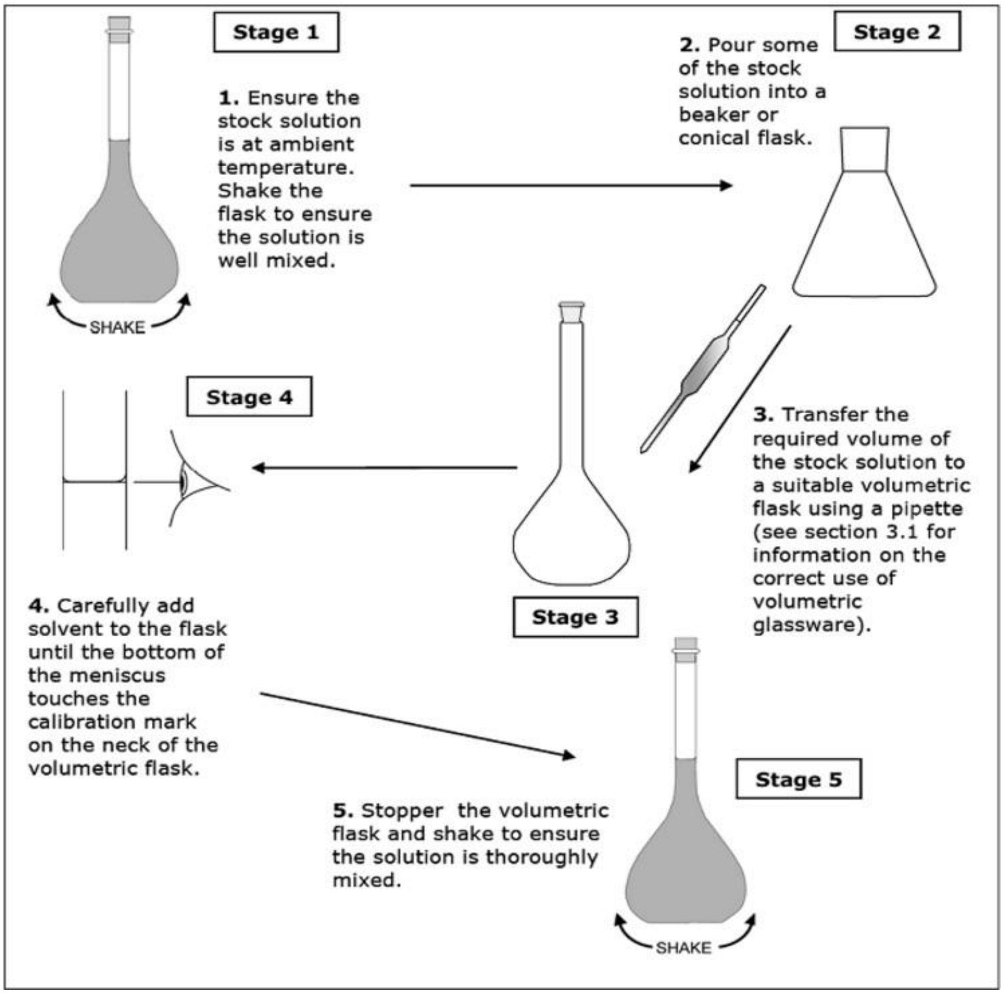

Figure 4: Procedure for preparing a solution of known concentration by dilution.

Aquous solutions can be prepared by percentage weight/volume (w/v%) or by percentagevolume/volume (v/v%)

Using percentage by weight (w/v%)

The formula for weight percent (w/v%) is:

Mass of solute (g)/ Volume of solution (mL) x 100

e.g A 10% NaCl solution has 10g of NaCl dissolved up to 100mL of solution.

Using percentage by weight (v/v%)

When the solute is a liquid, v/v% is expressedThe

formula for volume percent (v/v%) is:

Volume of solute (mL)/ Volume of solution (mL) x 100

e.g: A 10%v/v is prepared by diluting 10 mL of acetic acid with 90 mL of water.

Molar solutions:

Molar solutions are the most useful in chemical reaction calculations because theydirectly relate the moles of solute to the volume of solution.

The formula for molarity (M) is:

Moles of solute / 1 liter of solution

Or

Gram-molecular masses of solute / 1 liter of solution.

- e.g: The molecular weight of a sodium chloride molecule (NaCl) is 58.44, so one gram-molecular mass (=1 mole) is 58.44 g. We know this by looking at the periodic table.
- The atomic mass (or weight) of Na is22.99, the atomic mass of Cl is 35.45, so 22.99 + 35.45 = 58.44.
- If you dissolve **58.44g** of NaCl in a final volume of **1 liter**, you have made a **1M NaCl** solution, a 1 molar solution.

Some common terminologies you will come across are:

Solute The substance which dissolves in a solution

Solvent The substance which dissolves another to form a solution. For example, in asugar and water solution, water is the solvent; sugar is the solute.

Solution A mixture of two or more pure substances. In a solution one pure substanceis dissolved in another pure substance homogenously. For example, in a sugar and water solution, the solution has the same concentration throughout, ie. it is homogenous.

Mole A fundamental unit of mass (like a "dozen" to a baker) used by chemists. This term refers to a large number of elementary particles (atoms,molecules, ions, electrons, etc) of any substance. 1 mole is 6.02 x10^{23} molecules of that substance. (Avogadro's number).

OBJECTIVES

1. To learn the qualitative and quantitative aspects of common laboratory equipment
2. To expose students to the factors that affect the accuracy of an experiment
3. Recall the definitions of: standard solution, molar solution, molarity, mole, w/w% and v/v%
4. State the relationship between concentration, molarity, molecular mass, number of moles, volume, mass
5. Prepare a standard solution using volumetric glassware
6. Correctly use a volumetric pipette
7. Accurately prepare solutions that may be used in clinic (such as w/v%, v/v%, molar)
8. Understand and able to carry out calculations and conversions of solid and liquid substances into dilute and standard solutions

APPARATUS

1. Analytical balance
2. Burette (50 mL)
3. Volumetric flask (250 mL)
4. Beaker (100mL)
5. Thermometer
6. Retort stand
7. Burette clamp
8. Dropper

CHEMICAL

Distilled water, sodium chloride, NaCl

PROCEDURE

(A) Calibration of Volumetric Flask

1. Clean and dry a 100 mL volumetric flask and weigh accurately using analytical balance. Record the weight.
2. Fill the volumetric flask with distilled water and weigh again (use the same balance).
3. Record the weigh.
4. Record the temperature of the distilled water.
5. From Table 1.1 , determine the actual volume of the volumetric flask.

(B) Calibration of Burette

1. The 50mL beaker was cleaned and weighed accurately using an analytical balance. The mass of the empty beaker was recorded.
2. The burette was cleaned and raised using distilled water and filled in the burette with the distilled water until the zero mark. (Make sure there are no bubbles in the tip of the burette).
3. 5mL of the water from the burette was drained into the beaker and weighed as soon as possible. The mass was recorded.
4. Step 3 was repeated by draining water from the burette until the following burette reading became 10mL,15mL, and 20mL. (Each time 5 mL distilled water was added from the burette). Every time after adding 5mL of water mass (distilled water+ beaker) was recorded.
5. The temperature of the distilled water was recorded.
6. From Table 1.1, the actual volume of added addition of 5mL of distilled water was determine.

(C) Preparation of 100ml of 1% NaCl solution using percentage by weight (w/v%)

Remember: The formula for weight percent (w/v%) is:

Mass of solute (g)/ Volume of solution (mL) x 100

1. Accurately weigh approximately 1g of sodium chloride. Record your exact weight to 2 decimal places in **RESULTS**.

2. Pour the sodium chloride in the volumetric flask containing about 60mL of water.

3. Once the sodium chloride has dissolved completely (swirl the flask gently if necessary), add water to bring the volume up to the final 100 mL mark using Pasteur pipette.

   Caution: Do not simply measure 100mL of water and add 10g of sodium chloride. This will introduce error because adding the solid will change the final volume of thesolution and throw off the final percentage.

RESULTS

(A) Calibration of Volumetric Flask

Weight of empty volumetric flask	=	__________	g
Weight of volumetric flask + water	=	__________	g
Weight of water	=	__________	g
Temperature of the distilled water	=	__________	°C
Volume of the volumetric flask	=	__________	mL

(B) Calibration of the Burette

Weight of empty beaker	=	__________	g
Weight of beaker + water at 10mL	=	__________	g
Weight of beaker + water at 15 mL	=	__________	g
Weight of beaker + water at 20 mL	=	__________	g
Weight of water	=	__________	g
Temperature of the distilled water	=	__________	°C
Actual volume of the burette at 10 mL water	=	__________	mL
Actual volume of the burette at 15 mL water	=	__________	mL
Actual volume of the burette at 20 mL water	=	__________	mL

(C) Preparation of 100ml of 1% NaCl solution using percentage by weight (w/v%)

Mass of NaCl	=	__________	g
Number of moles in 1g of NaCl	=	__________	mol
Molarity of the prepared solution	=	__________	Mol dm^{-3}

DISCUSSION

Interpret data results in the context of the specific question you set out to address in this study. *Consider the following issues:*

- *What did you expect to find, and why?*
- *How did your results compare with those expected? State your expectations explicitly, and back up your statements with a reference.*
- *List any weaknesses in the experimental design and how these weaknesses may have affected your results. Since your laboratory investigation was subject to limitations of time and facilities, you did not do a "perfect" experiment. It is important for you to understand and acknowledge this in your conclusion. Also, consider how you might be able to get more specific and more reliable results by changing certain aspects of the experiment.*

CONCLUSION

QUESTIONS

1. How to overcome or reduce the problems of random error and systematic error while doing an experiment.

2. In what situation do you use a volumetric flask, conical flask , pipette and graduated cylinder? Explain your answer from the accuracy aspects of these apparatus.

3. Explain how to read the burette. What are the factors to be considered while using burette?

$$2KClO_3 \text{ ---> } 2KCl + 3O_2$$

4. 50 mol of $KClO_3$ decomposes. How many grams of O_2 will be produced?

5. If 80.0 grams of O_2 was produced, how many moles of $KClO_3$ decomposed?

6. We want to produce 2.75 mol of KCl. How many grams of $KClO_3$ would be required?