Activity 2

BASIC TECHNIQUES AND NUMERACY IN ANALYTICAL CHEMISTRY LABORATORY

BACKGROUND

University level science courses are numerical subjects and simple numerical skills are essential if you are to be able to design and carry out experiments and interpret results. In this session we will:

1. review the concepts of units of volume, mass, molarity and dilution, significant figures and rounding off numbers;
2. practice using three basic pieces of apparatus – digital balance (analytical balance and top loading balance), volumetric flask and graduated pipette; and
3. prepare standard solutions of sodium hydroxide and hydrochloric acid.

1.1 COMMONLY USED UNITS IN ANALYTICAL CHEMISTRY LABORATORY

In analytical chemistry, units of volume are based on the liter (symbol L). This is the same quantity as a dm^3 with which some of you may be more familiar. Since we are often dealing with small quantities, volumes can also be given as:

milliliter (mL)	**= 10^{-3} L**	**= 0.001 L**
microliter (μL)	**= 10^{-6} L**	**= 0.000001 L**

Similarly, weights are given as gram (g) and can also be given as:

milligram (mg)	**= 10^{-3} g**	**= 0.001 g**

microgram (μg)	**= 10^{-6} g**	**= 0.000001 g**

1.2 SOLUTIONS, CONCENTRATIONS, MOLES AND MOLARITY

A solution is a homogeneous mixture of two or more substances. Solutions have two components: (a) the minor species in the solution is called solute, and (b) the major species is called solvent. In this laboratory course, all solutions are referred to as aqueous solutions, in which the solvent is water. The amount of solute that is contained in a given mass or volume of the solution is known as concentration.

1.2.1 MOLE

The mole is defined as Avogadro's number of particles (atoms, molecules, ions, or anything else). A mole is the quantity of a substance whose weight in grams is equal to the molecular weight of the substance. This is conventionally written as 1 mol.

Example: NaOH

Atomic Mass of Na = 23, Atomic Mass of O = 16, Atomic Mass of H = 1,

The molecular weight (MW) of NaOH is the sum of atomic masses of the atoms in the molecule, that is, MW = 23 + 16 + 1 = 40.

Therefore, 1 mole of NaOH is equal to 40 g

A millimole is one-thousandth of a mole, that is 1 millimole (mmol) is the MW in mg.

1.2.2 MOLARITY

A solution made up of 1 mole of a substance dissolved in a total volume of 1 liter is a 1 molar solution. Molarity is symbolized by the capital letter M, thus, 1 molar solution is conventionally written as 1 M. Hence, 1 mmol contained in 1 liter is a 1 millimolar solution and written as 1 mM. Don't confuse mol and M. The first is an amount, the second is a concentration.

To interconvert between volumes, molarities and moles, remember that the molarity (M) of a solution is the number of moles per liter:

$$M = \frac{mol}{liter}$$

There are other ways of expressing units of concentration, particularly if the solute MW is known (e.g., molality, percentage, normality). The detailed discussion of these units will be presented in the lecture course.

1.3 SIGNIFICANT FIGURES AND ROUNDING OFF NUMBERS

1.3.1 SIGNIFICANT FIGURES

All your results and calculations using your measured data should reflect the accuracy of the experiment you carried out. We use significant figures and decimal places to indicate the accuracy of our measurements. For example, a concentration of 25 mM indicates that the true concentration is somewhere between 24 and 26 mM. If, however, the concentration was given as 25.3 mM, this indicates that the true value lies between 25.2 and 25.4 mM.

Problems with significant figures and decimal places often arise when you are carrying out a calculation with your data and the calculator gives a number with many decimal places. Familiarizing with the rules of significant figures and rounding off numbers may help you. When reading a measured value, all nonzero digits should be counted as significant. There is a set of rules for determining if a zero in a measurement is significant or not.

RULE 1. Zeros in the middle of a number are like any other digit; they are always significant. Thus, 94.072 g has five significant figures.

RULE 2. Zeros at the beginning of a number are NOT significant; they act only to locate the decimal point. Thus, 0.0834 cm has three significant figures, and 0.02907 mL has four.

RULE 3. Zeros at the end of a number and *after* the decimal point are significant. It is assumed that these zeros would not be shown unless they were significant. 138.200 m has six significant figures. If the value were known to only four significant figures, we would write 138.2 m.

RULE 4. Zeros at the end of a number and *before* an implied decimal point may or may not be significant. We cannot tell whether they are part of the measurement or whether they act only to locate the unwritten but implied decimal point.

1.3.2 ROUNDING OFF NUMBERS

Once you decide how many digits to retain, the rules for rounding off numbers are straightforward:

RULE 1. If the first digit you remove is 4 or less, drop it and all following digits. 2.4<u>2</u>71 becomes 2.4 when rounded off to two significant figures because the first dropped digit (a <u>2</u>) is 4 or less.

RULE 2. If the first digit removed is 5 or greater, round up by adding 1 to the last digit kept. 4.5<u>8</u>32 is 4.6 when rounded off to 2 significant figures since the first dropped digit (an <u>8</u>) is 5 or greater.

RULE 3. In carrying out a multiplication or division, the answer cannot have more significant figures than either of the original numbers. The final answer should contain the same number of significant figures as there are in the measurement with the fewest significant figures.

RULE 4. In carrying out an addition or subtraction, the answer cannot have more digits after the decimal point than either of the original numbers. The final answer has the same number of decimal places as there are in the measurement with the fewest decimal places.

If a calculation has several steps, it is best to round off at the end. In calculations use all the decimal places that the measurement provides and round off the final number to the correct number of significant figures.

Example 1: 3.463g of substance dissolved in 2.13 L of solution = 3.463/2.13 g/L = 1.625821596 round to 1.63 g/L

Example 2: 3.689g of substance dissolved in 2.17 L of solution = 3.689/2.17 g/L = 1.70 g/L (not 1.7)

1.4 DILUTION OF SOLUTIONS

In teaching and research laboratories, many frequently used solutions like buffers and chemical reagents are made up and stored as concentrated stock solutions and diluted appropriately before use. In dilution, a known volume of the concentrated solution (referred as stock solution) is transferred to clean vessel (such as a volumetric flask) and diluted to the desired final volume by addition of a solvent (i.e., water). Dilutions of solutions is governed by the Dilution formula:

$$C_1 V_1 = C_2 V_2$$

C_1 and C_2 are the initial and final concentrations. V_1 and V_2 are the initial and final volumes.

SAMPLE PROBLEMS

1. What volume of 5 M NaOH is needed to create a 100-mL solution of 1M NaOH?
2. If you have 300 mL of 1.5 M NaCl, how many mL of 0.25 M NaCl can you make?

2.0 PRACTICAL EXERCISES

2.1 INSTRUCTIONS

1. You will be instructed on the proper use of a digital balance, volumetric flask, and Mohr pipette in weighing and transferring required volumes of solutions.
2. Your laboratory instructor will show you how to use the volumetric flask and pipettes.
3. Using a Labster Simulation, you will prepare a 0.300 M aqueous NH_4Cl solution. Access the simulation through Cloud Campus (Blackboard Course Site).
4. **SAFETY NOTES: LABORATORY SAFETY FORM**

   As part of this exercise, you will be introduced to writing safety notes before commencing with the practical work. A template is provided at the end of this activity. Accomplishing this safety form requires a little bit of research using the materials and data sheet (MSDS) of the chemical regents.

2.2 GRADUATED GLASSWARE USED FOR PREPARATION OF SOLUTIONS

IMPORTANT REMINDERS ON HOW TO TAKE CARE OF VOLUMETRIC GLASSWARE

1. Use a clean volumetric glassware before preparing solutions to avoid contamination. Wash glassware with a mild dilute soap solution. Rinse first with tap water, and then deionized (DI) water. Re-wash the volumetric flask if you notice beads of water form on the walls. Allow it to drain and dry in air or use lint-free paper towels.

2. A volumetric glassware is a calibrated glassware. Never dry volumetric glassware in an oven or stream of hot air! The heat will distort the glass and change the calibrated volumes.

3. The glassware must be always rinsed with a small amount of the solvent to be used. This step prevents contamination of the solution from water or other contaminants on the glassware's inside walls and removes the need to dry the flask.

4. Allow heated solutions to cool down to room temperature or completely dissolve solid solutes in the solutions before using a graduated pipet (Mohr or volumetric) to transfer these solutions. The heat will distort the calibrated volume and any undissolved solids can clog the narrow stem and tip.

IMPORTANT REMINDERS ON HOW TO READ THE LEVEL OF LIQUID IN A VOLUMETRIC GLASSWARE

1. The "meniscus" of a liquid is the curvature of the liquid surface in a narrow container. The downward curving (concave) liquid surfaces (e.g., water) is read at the bottom of the meniscus.

2. The upward curving (convex) liquid surfaces (e.g., mercury) is read from the top of the meniscus.

3. The liquid level is read from the edge of the liquid when the shape of the meniscus is difficult to discern such as in highly colored liquids (e.g., purple $KMnO_4$).

4. Read liquid levels by positioning the eye at the same level as the meniscus to avoid parallax error.

2.2.1 THE VOLUMETRIC FLASK

A volumetric flask shown in Figure 1 is a piece of laboratory glassware used to prepare solutions. A volumetric flask contained a specified volume when the liquid level is adjusted to the single graduation mark in the neck of the flask. Volumetric flasks are used to measure volumes more precisely than beakers or Erlenmeyer flasks.

Figure 1. Volumetric flasks

2.2.2 GRADUATED PIPETTES: VOLUMETRIC PIPETTE

A volumetric pipet shown in Figure 2 is an elongated glass bulb with two narrow glass stems at the top and bottom of the bulb. This type of pipette is used "to deliver" a single and fixed volume of liquid from one container to another at a specific temperature (usually 20.0°C).

Figure 2. A volumetric pipette

Similar to volumetric flasks, a single calibration mark on the top stem marks the volume contained at a specific temperature. The top end of the stem is open and is usually attached to a suction bulb to draw liquid into the pipet. **Note: Never pipet by mouth! Always use a pipet bulb to provide the necessary suction.**

HOW TO USE A VOLUMETRIC PIPETTE

1. Rinse the pipet with a small amount of the solution to remove any water film or contaminants from the inside walls.

2. Attach a rubber pipette bulb onto the top end. To fill the pipet, compress the rubber pipet bulb and insert the pipet tip into the liquid. Slowly release the pressure on the pipet bulb. Allow the liquid level to rise above the calibration mark. DO NOT permit the liquid to enter the rubber pipet bulb.

3. Remove the bulb and quickly fit your index finger on top of the stem. Allow the level of liquid to drop until the meniscus is exactly level with the calibration mark by adjusting the pressure of the index finger. Touch off the hanging drop from the tip of the pipet.

4. Simply release finger pressure on the pipet stem and allow the liquid to drain freely to a container.

5. When finished, touch off the drop of liquid hanging at the tip into the transferred liquid (it is part of the delivered volume). DO NOT blow out any liquid remaining inside the tip of the pipette! The pipette has been calibrated to contain this last drop of liquid.

2.2.3 GRADUATED PIPETTES: MOHR PIPETTE

Mohr pipets shown in Figure 3 have graduated volume markings increasing in value going down the pipet. This type of pipette allows variable volumes of the liquid to be delivered. There are two types of Mohr Pipettes: Graduated Tip and Clear Tip.

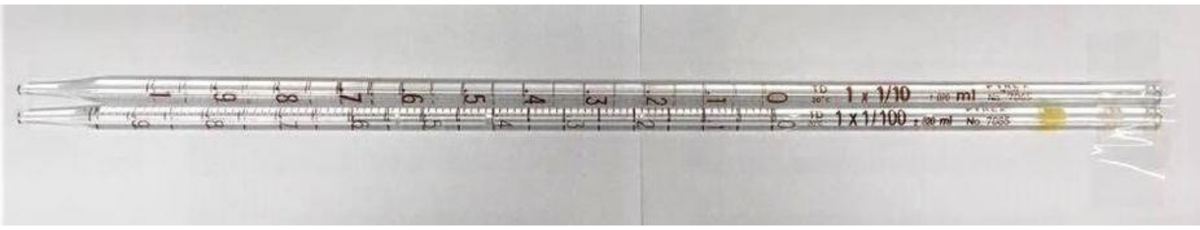


Figure 3: Mohr (Graduated) Pipette

A. GRADUATED TIP

Volume graduations are present all the way down to the tip (so the pipet can be completely drained). Once a suction bulb has been used to fill this type of Mohr pipet, the level of liquid needs to be lowered so its meniscus falls within the markings and then the initial volume of liquid is recorded. The initial volume can be lowered so that the volume left within the pipet is the desired volume that is to be delivered. Because of the way the markings are numbered the initial volume needs to be determine by subtracting the desired volume from the pipet's total volume.

B. CLEAR TIP

Volume markings end with a last marking for the total volume of the pipet (so the liquid must be stopped before it goes below this last marking and the final volume must be recorded from the pipet).

2.3 METHODOLOGY FOR THE PREPARATION OF SOLUTIONS

After doing the simulation, read the following instructions on the preparation of HCl and NaOH standards as done in an actual laboratory. Take note of the safety precautions and best practices.

2.3.1 PREPARATION OF 0.10 M HYDROCHLORIC ACID SOLUTION

1. Pipette ~8.4 mL of concentrated HCl and slowly add this to a 100.0 mL distilled water in a 250 mL beaker. Do this in the fume hood cupboard. Concentrated HCl is a corrosive acid; Be mindful of the hazards when pipetting and transferring the required volume of the acid.

2. Transfer this solution to a 1.00-liter volumetric flask. Rinse the beaker with 100.0 mL of distilled water to the same beaker and transfer the rinsing to the volumetric flask. Repeat this for three times to ensure quantitative transfer.

3. Fill the volumetric flask with distilled water to mark. Place the stopper and mix the solution by carefully inverting the volumetric flask while securing the stopper. Transfer the solution to a clean reagent bottle and cap it tightly. Store this in your locker and use this in the succeeding experiments. The exact concentration of the solution will be determined in another experiment. For the meantime, label your reagent bottle as follows:

M HCl

Group No. ________

Section: __________

Date Prepared: ______________

2.3.2 PREPARATI

1. Boil 1.5 liters of distilled water and cool to room temperature. This is the water that you will use to dissolve the NaOH pellets in Step 3.

2. Weigh approximately 4.00 grams of sodium hydroxide (NaOH) using the top loading balance. Solid NaOH pellets rapidly absorb moisture form the air and turns into a liquid. Avoid too much exposure of NaOH in air and tightly seal NaOH containers once you are done weighing.

> **WARNING!**
> NaOH is corrosive (DO NOT let this compound get into your eyes).

3. Dissolve the pellets with 50 mL of distilled water in a clean 250-mL beaker. Once the pellets are dissolved, transfer the solution in a 1.0–liter volumetric flask and dilute with distilled water to make 1.0-liter solution. This will give a concentration of ~0.1 M NaOH.

4. Transfer the NaOH solution in a clean polyethylene bottle and tightly cap the bottle. Keep this in your locker and use this in the succeeding experiment to determine its exact concentration. For the meantime, label your reagent bottle as follows:

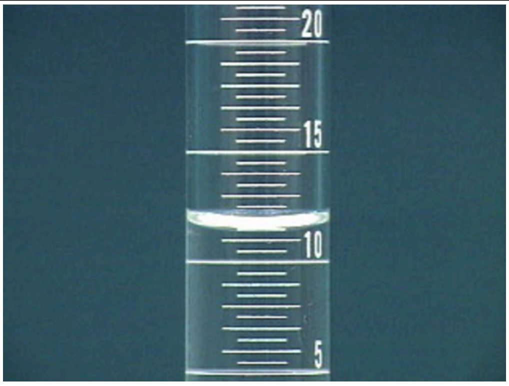
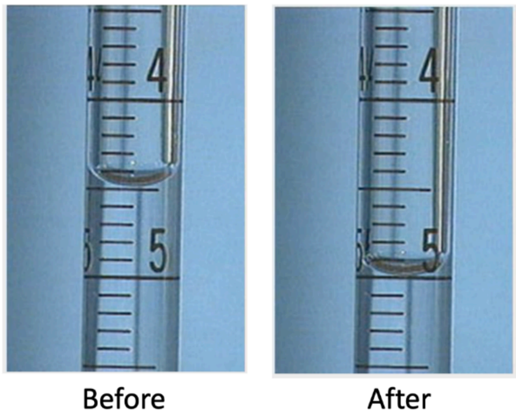
M NaOH

Group No. _________
Section: __________

Date Prepared: ______________

ACTIVITY 2 WORKSHEET

PART 1 – MULTIPLE CHOICE. CHOOSE THE BEST ANSWER. WRITE THE CAPITAL LETTER OF YOUR ANSWER INSIDE EACH BOX.

 1. What is the volume of the liquid being held in this 25-ml graduated cylinder? A. 13 ml B. 11.5 ml C. 10.6 ml D. 10.5 ml	 2. Consider the images of a 10-ml volumetric pipette. What volume of liquid was delivered? A. 4.5 ml B. 5.5 ml C. 0.5 ml D. 1 ml
3. How many significant figures are in 0.0040020? A. 5 B. 7 C. 4 D. 6	4. A quantity of 2,500,000 micrograms converted to grams is… A. 250 g B. 2.5 g C. 0.0025 g D. 0.25 g
5. If 1.14 g/ml is converted to mg/L… A. 1,140 B. 0.0014 C. 114,000 D. 1,140,000	6. In lab glassware, which of the following is completely transparent to UV light? A. standard borosilicate glass B. soda-lime glass C. fused quartz D. milky quartz
7. Which of the following should be used to dispense a precise volume of liquid? A. Dropper B. Graduated cylinder C. Volumetric flask D. Volumetric pipette	8. If 60 g of NaCl (MW = 58.44 g/mol) is in 150 ml of NaCl solution, the molar concentration of the solution is… A. 6.84 B. 0.0068 C. 23.38 D. 6.49
9. If 34.5 ml of 1.88 M stock solution of NaCl was used to prepare 134 ml of diluted solution. What is the concentration of the diluted solution? A. 1.24 M B. 0.48 M C. 0.084 M D. 0.27 M	10. Which of the following should be used in preparing solutions? A. Beaker B. Graduated cylinder C. Volumetric flask D. Erlenmeyer flask

PART 2 – PROBLEM SOLVING. CALCULATE THE UNKNOWN. SHOW YOUR SOLUTIONS AND EXPRESS YOUR ANSWERS IN PROPER UNITS AND CORRECT NUMBER OF SIGNIFICANT FIGURES AND DECIMAL PLACES.

1. Calculate the grams (3 significant figures) of solution necessary to provide 68.5 g of NaCl from a 12.0% NaCl solution. **SOLUTION:**
2. What is the molar concentration (3 significant figures) of a 17.5% aqueous NaOH solution (density = 1.05 g/mL)? **SOLUTION:**
3. If 12.00 L of a 6.00 M HNO_3 solution needs to be diluted to 0.750 M, what will be its final volume in L (2 decimal places)? **SOLUTION:**
4. Typical blood serum is about 0.14M NaCl. What volume in mL (2 decimal places) of blood contains 1.0 mg NaCl? **SOLUTION:**
5. If 0.470 L of water is added to a 30.0 mL of 17.4 M stock solution of acetic acid (CH3COOH), what is the resulting concentration in M (3 significant figures)? **SOLUTION:**