Gen Chem Lab

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Collection edited by: Mary McHale

Content authors: Mary McHale, Alvin Orbaek, and Andrew Barron

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Objective

- To help you become familiar with the layout of the laboratory including safety aids and the equipment that you will be using this year.
- To make an order-of-magnitude estimate of the size of a carbon atom and of the number of atoms in a mole of carbon, based on simple assumptions about the spreading of a thin film of stearic acid on a water surface

Grading

- Lab Report (90%)
- TA points (10%)

Before coming to Lab

Read the following:

- Lab instructions
- Background Information
- Concepts of the experiment

Print out the lab instructions and report form.

Read and sign the equipment responsibility form and the safety rules. Email Ms. Duval, at **nduval@rice.edu**, to confirm completing this requirement by noon on September 14 th.

Introduction

Since chemistry is an empirical (experimental) quantitative science, most of the experiments you will do involve measurement. Over the two semesters, you will measure many different types of quantities – temperature, pH, absorbance, etc. – but the most common quantity you will measure will be the *amount* of a substance. The amount may be measured by (1) weight or mass (grams), (2) volume (milliliters or liters), or (3) determining the number of moles. In this experiment we will review the methods of measuring mass and volume and the calculations whereby number of moles are determined.

Experimental Procedure

1. Identification of Apparatus

On the tray (in DBH 214) we have a number of different pieces of common equipment. We will, identify and sketch each - I know this may sound a trivial exercise but it is necessary so that we are all on the same page.

1. beaker
2. Erlenmeyer flask (conical flask)
3. side-arm Erlenmeyer flask
4. graduated (measuring) cylinder
5. pipettes, both types graduated and bulb
6. burette
7. Bunsen burner
8. test tubes
9. watch glass
10. volumetric flask

2. Balance Use

In these general chemistry laboratories, we only use electronic balances – saving you a lot of time. However, it is important that you become adept at using them.

Three aspects of a balance are important:

1. The on/off switch. This is either on the front of the balance or on the back.
2. The "Zero" or "Tare" button. This resets the reading to zero.
3. **CLEANLINESS**. Before and after using a balance, ensure that the entire assembly is spotless. Dirt on the weighing pan can cause erroneous measurements, and chemicals inside the machine can damage it.

Balance Measurements:

1. Turn the balance on.

2. After the display reads zero, place a piece of weighing paper on the pan.

3. Read and record the mass. (2)

4. With a spatula, weigh approximately 0.2 g of a solid, common salt NaCl. The excess salt is discarded, since returning it may contaminate the rest of the salt.

5. Record the mass (1). To determine how much solid you actually have, simply subtract the mass of the weighing paper (2) from the mass of the weighing paper and solid (1). Record this mass (3). You have just determined the mass of an "unknown amount of solid."

6. Now place another piece of weighing paper on the balance and press the Zero or Tare button then weigh out approximately 0.2 g of the salt (4). Thus, the zero/tare button eliminates the need for subtraction.

3. Measuring the volume of liquids

When working with liquids, we usually describe the quantity of the liquid in terms of volume, with the usual units being milliliters (mL). We use three types of glassware to measure volume – (1) burette, (2) bulb pipette, and (3) graduated cylinder. A volumetric flask will also allow for a high degree of accuracy and precision in the measurements of any liquids, so a 100 mL volumetric flask will contain precisely 100.0 mL of solution when filled to the line marked on the neck of the flask.

- Examine each piece of equipment. Note that the sides of each are graduated for the graduated cylinder and the burette. The bulb pipette delivers a specific volume, 10.00 mL. The burette will be used to deliver a variable volume of solution and will also be precise to two decimal places.

- Put some water into the graduated cylinder. Bend down and examine the side of the water level. Note that it has a "curved shape." This is due to the water clinging to the glass sides and is called the meniscus. When reading any liquid level, use the center of the meniscus as your reference point.

Graduated cylinder

Look at the graduations on the side of the cylinder. Note that they go from 0 on the bottom and increase upwards. Since volumes in graduated cylinders are only precise to one decimal place, *a graduated cylinder is generally only used when a high degree of precision is not required.*

1. Using your 10mL graduated cylinder, add water up to the 10 mL line as accurately as possible.

2. Dry a small beaker and weigh it (2).

3. Pour the 10 mL of water from the cylinder into the beaker. Reweigh (1).

4. Subtract the appropriate values to get the weight of the water (3).

Bulb Pipette

1. Half-fill a beaker with water.

2. Squeeze the pipette bulb and attach to the top of the pipette. Put the spout of the pipette under water and release the bulb. It should expand, drawing the water into the pipette. **Do not let the water be drawn into the bulb.**

3. When the bottom of the meniscus is above the line on the pipette, remove the pipette from the water.

4. Squeeze the bulb to run the extra water into a waste container until the bottom of the meniscus is level with the line on the pipette.

5. Add 10 mL of water to a pre-weighed dry beaker (5).

6. Weigh (4).

7. Subtract to get the weight of the water (6).

Burette

1. Examine the graduations. Note that 0 is at the top. Note that the stopcock is horizontal to close the burette and vertical to open it.

2. First, lower the burette so that the top is easy to reach and make sure the burette is closed. Using a funnel, add about 10 mL of water.

3. Open the burette and run a little water into a waste container. Then turn the burette upside down and allow the rest of the water to run into the container.

4. You have just rinsed your burette. This should be done every time before using a burette – first rinse with water, then repeat the process using whatever liquid is needed in the experiment.

5. Fill the burette to any convenient level (half-way is fine). It is a good technique to add more liquid than you need, and allow some liquid to run into a waste container until you reach the appropriate level so that you fill the space from the top to the tip of the burette.

6. Dry a beaker and weigh (8).

7. Add 10 mL of water to a pre-weighed dry beaker (7).

8. Subtract to get the weight of the water (9).

4. Estimation of Avogadro's number

Briefly, as a group with your TA, you will make an approximate (order of magnitude) estimate of Avogadro's number by determining the amount of stearic acid that it takes to form a single layer (called a monolayer) on the surface of water. By making simple assumptions about the way the stearic acid molecules pack together to form the monolayer, we can determine its thickness, and from that thickness we can estimate the size of a carbon atom. Knowing the size of a carbon atom, we can compute its volume; and if we know the volume occupied by a mole of carbon (in the form of a diamond), we can divide the volume of a mole of carbon by the volume of an atom of carbon to get an estimate of Avogadro's number.

Procedure

Special Supplies: 14 cm watch glass; cm ruler; polyethylene transfer pipets; 1-mL syringes; pure distilled water free of surface active materials; disposable rubber gloves (for cleaning own watch glasses in 0.1 M NaOH in 50:50 methanol/water): 13 100 mm test tubes with rubber stoppers to fit.

Chemicals: pure hexane, 0.108 g/L stearic acid (purified grade) solution in hexane. 0.1 M NaOH in 50:50 methanol/water used for washing the watch glasses, dye.

SAFETY PRECAUTIONS: Hexane is flammable! There must be no open flames in the laboratory while hexane is being used.

WASTE COLLECTION: At the end of the experiment, unused hexane solvent and stearic acid in hexane solution should be placed in a waste container, marked "Waste hexane/stearic acid solution in hexane."

Measurement of the volume of stearic acid solution required to cover the water surface

Your TA will do this as a group demonstration:

1. Fill the clean watch glass to brim with deionized water. One recommended way to do this is to set up your 25 mL burette on a ring stand.

2. Using a transfer pipette, obtain about 3-4 mL 0.108 g/L stearic acid solution in hexane in a clean, dry 13 100 mm test tube. Keep the tube corked when not in use.

3. Obtain more distilled water and fill the burette. Place your watch glass directly under the burette (about 1 inch or less from the tip) and dispense the water until the entire watch glass is full. You may have to refill the burette 4 or 5 times to do this. With careful dispensing, the surface tension of the water should allow you to fill the entire watch glass with relative ease.

4. Carefully measure the diameter of the water surface with a centimeter ruler. It should be close to 14 cm, + or - a couple of millimeters. Next, rinse and fill your 1 mL syringe with stearic

acid solution, taking care to eliminate bubbles in the solution inside the syringe.

5. Read and record the initial volume of the syringe (1 mL is always a good place to start.)

6. Then add the stearic acid solution drop by drop to the water surface. Initially, the solution will spread across the entire surface, and it will continue to do so until a complete monolayer of stearic acid has been formed. If your first few drops do not spread and evaporate quickly, either your water or watch glass is still dirty. As this point is approached, the spreading will become slower and slower, until finally a drop will not spread out but will instead sit on the surface of the water (looking like a little contact lens). If this "lens" persists for at least 30 s, you can safely conclude that you have added 1 drop more than is required to form a complete monolayer.

7. Now, read and record the final volume reading of the syringe.

When you have completed all of your measurements, rinse your syringe with pure hexane, and dispose of all the hexane-containing solutions in the waste collection bottle provided.

Calculation of Avogadro's Number

The calculation proceeds in several steps.

- We calculate the volume of stearic acid solution in hexane required to deliver enough stearic acid to form a monolayer.

- All of the hexane evaporates, leaving only the thin monolayer film of stearic acid, so we next calculate the actual mass of pure stearic acid in the monolayer.

- We calculate the thickness of the stearic acid monolayer, using the known density of stearic acid and the area of the monolayer.

- Assuming the stearic acid molecules are stacked on end and are tightly packed, and knowing that there are 18 carbon atoms linked together in the stearic acid molecule, calculate the diameter and volume of a carbon atom.

- Calculate the volume of a mole of carbon atoms in diamond; divide the molar volume of carbon (diamond) by the volume of a single carbon atom to obtain an estimate of Avogadro's number. Remember that the units of Avogadro's number are mol^{-1}, so you can use unit analysis to check your answer.

Solutions

Chapter 2. Stoichiometry: Laws to Moles to Molarity

Experiment 3: Stoichiometry: Laws to Moles to Molarity

Objective

- To determine the mass of a product of a chemical reaction
- To make a solution of assigned molarity
- To test the solubility of Peeps, yes Peeps, in various solvents

Grading

- Lab Report (90%)
- Quiz(10%)

Before Coming to Lab...

- Read the lab instructions
- Complete the online quiz, this is part of your grade NOT bonus

Introduction

The word stoichiometry derives from two Greek words: *stoicheion* (meaning "element") and *metron* (meaning "measure"). Stoichiometry deals with calculations about the masses (sometimes volumes) of reactants and products involved in a chemical reaction. Consequently, it is a very mathematical part of chemistry.

In the first part of this lab, sodium bicarbonate is reacted with an excess of hydrochloric acid.

$$\mathrm{NaHCO_3(s) + HCl(aq) \rightarrow NaCl(aq) + CO_2(g) + H_2O} \quad (2.1)$$

By measuring the mass of $NaHCO_3$ and balancing the equation (above), the mass of NaCl expected to be produced can be calculated and then checked experimentally. Then, the actual amount of NaCl produced can be compared to the predicted amount.

This process includes molar ratios, molar masses, balancing and interpreting equations, and

conversions between grams and moles and can be summarized as follows:

1. Check that the chemical equation is correctly balanced.

2. Using the molar mass of the given substance, convert the mass given in the problem to moles.

3. Construct a molar proportion (two molar ratios set equal to each other). Use it to convert to moles of the unknown.

4. Using the molar mass of the unknown substance, convert the moles just calculated to mass.

In the second part of this lab, since a great deal of chemistry is done with solutions, a solution will be prepared of allocated molarity. Molarity, or more correctly molar concentration, is defined to be the number of moles of solute divided by the number of liters of solution:

$$c_M = \frac{n_{substance}}{V_{solution}}$$

Figure 2.1.

with units of [mole/L]. However molar concentration depends on the temperature so a higher temperature would result in an increased volume with a consequential decrease in molar concentration. This can be a significant source of error, of the same order as the error in the volume measurements of a burette, when the temperature increases more than 5 ºC.

Steps to preparing a solution of a certain concentration:

1. First, you need to know the formula for the solute.

2. Next, you need to calculate the molecular weight of the solute by adding up the atomic weights of the elements present in the correct ratios.

3. Then, based on the volume of solution you are making, calculate the mass of solute needed to dissolve in the solution volume. Usually, deionised water is the solvent.

4. Remember to ensure that all the solute is dissolved before finally filling to the mark on the volumetric flask. If there is any undissolved solute present in the solution, the water level will go down slightly below the mark, since the volume occupied by the solute differs from the actual volume it contributes to the solution once it is dissolved.

Example solution preparation: potassium chromate

1. The formula for potassium chromate is K_2CrO_4.

2. The elements present are potassium, chromium, and oxygen with atomic masses of 39.10

2. The elements present are potassium, chromium, and oxygen with atomic masses of 39.10, 52.00 and 16.00 respectively. Adding up these numbers in the correct ratios dictated by the formula yields the following: 2 x 39.10 + 1 x 52.00 + 4 x 16.00 = 194.20 g/mol.

3. For one liter of solution use a 1000 mL volumetric flask. So a 1M solution would require 194.2g of solid K_2CrO_4 in 1 L, 0.1M 19.42g of solid K_2CrO_4 and so on.

Your teaching assistant will check the accuracy of the solution that you have made by titration, which is a method of **quantitatively** determining the **concentration** of a **solution**. A **standardsolution** (a solution of known **concentration**) is slowly added from a **buret**te to a **solution** of the analyte (a solution of unknown **concentration** – your solution) until the **reaction** between them is judged to be complete (**equivalence point**). In colorimetric **titration**, some **indicator** must be used to locate the **equivalence point**. One example is the addition of **acid** to **base** using phenolphthalein (**indicator**) to turn a pink **solution** colorless in order to determine the **concentration** of unknown **acid**s and **base**s. Record your TAs value of the molarity of your solution on your report form along with your percent error.

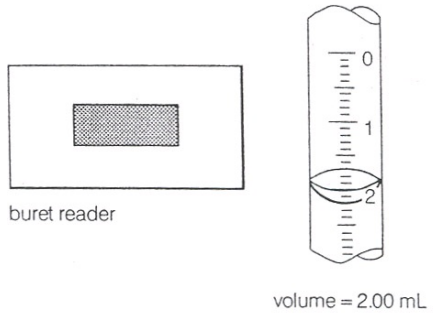

Figure 2.2.

Figure 1: Reading the Burette

When an acid is neutralized by a base, there are stoichiometrically equal amounts of acid and base and the pH = 7. It is possible to accurately determine the concentration of either the acid or base solution.

Moles of a substance = Concentration of solution (moles/L) x Volume (L)

We can calculate the concentration of the acid or base in the solution by using the following equation where balance base and balance acid refer to the stoichiometric ratio of the base and acid to each other.

$$\text{Balance Base (Bb)} \times \text{Moles of Acid} = \text{Moles of Base} \times \text{Balance Acid (Ba)} \qquad (2.2)$$

$$B_b \times C_a \times V_a = B_a \times C_b \times V_b \quad (2.3)$$

Titration Calculations:

Step 1: Balance the neutralization equation. Determine Balance of Acid and Base.

Step 2: Determine what information is given.

Step 3: Determine what information is required.

Step 4: Solve using the equation below.

$B_b \times C_a \times V_a = B_a \times C_b \times V_b$

Example:

Calculate the concentration of a nitric acid solution HNO_3 if a 20 mL sample of the acid required an average volume of 55 mL of a 0.047 mol/L solution of $Ba(OH)_2$ to reach the endpoint of the titration.

Step 1: $2HNO_3 + Ba(OH)_2 \rightarrow Ba(NO_3)_2 + 2H_2O$ Balance Base = 1 Balance Acid = 2

Step 2: Given information Volume Acid = 20 mL Volume Base (average) = 55 mL Concentration of Base = 0.047 mol/L

Step 3: Required information Concentration of Acid **Step 4:** Solve using the equation. $B_b \times C_a \times V_a = Ba \times C_b \times V_b$ $1 \times Ca \times 20mL = 2 \times 0.047 mol/L \times 55mL$ Ca = 0.2585 mol/L (considering significant figures 0.26 mol/L)

Procedure

Materials List

sodium bicarbonate $NaHCO_3$

3M hydrochloric acid (HCl) solution

Part 1

1. Weigh an empty 150-mL beaker on the electronic balance. Record this value in your data table.