CEAC 103
GENERAL CHEMISTRY

Experiment 1

Introductory to Laboratory Techniques

Purpose: To learn how to use common laboratory equipments and to practice the procedures commonly used in laboratory.

Theory

Separation of Substances

Every substance has a large number of physical and chemical properties. Physical properties include color, smell, taste, solubility, density, electrical conductivity, heat conductivity, melting and boiling points. When a physical change is observed, the substance retains its chemical identity.

In contrast to physical changes, when chemical changes are observed, new substances are formed. Chemical properties include decomposition by heating, and reactions of the substance with water, oxygen, acids, bases, etc.

So, physical changes are reversible, chemical changes are irreversible (not reversible).

Solutions

When a solid is mixed with a liquid, the resultant mixture may still be another liquid. This liquid may contain no visible solid particles although its color may be different.

For the process described above, the resultant mixture is called as solution, the solid used is a solute, and the liquid originally used is solvent. Solutions are homogenous mixtures. When a solution forms, it can be stated that "The solid dissolves in the liquid" or "The solid is soluble in the liquid".

Solubility and Miscibility

Solubility can be defined as the quantity of a particular substance that can dissolve in a particular solvent. More precisely, percent solubility is the maximum amount of a particular substance that can be dissolved in 100 mL of pure water at a particular reference temperature.

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$CuSO_4$ (solute), is soluble in water (solvent) and the solution formed is blue.

After mixing a solid and a liquid, if the solid remains in its original physical form, we state that "The solid does not dissolves in this liquid "or "The solid is INSOLUBLE in this liquid".

In Part A, water will be separated from potassium permanganate by *distillation*. When the potassium permanganate solution is heated, water vapor will be driven off first, because the boiling point of potassium permanganate is much higher than that of water.

In Part B, The behavior of several substances with different solvents will be tested. For this, solubility of chemicals in water and in nitric acid will be examined. Furthermore, behavior of these substances on heating will be observed.

In Part C, it will be investigated that how two water soluble salts can be separated on the basis of their solubility differences at room temperature. When sodium dichromate ($Na_2Cr_2O_7$) and potassium chloride (KCl) are added to water, a solution which contains K^+, Na^+ , Cl^- and $Cr_2O_7^{2-}$ ions are formed. When this solution is cooled, the most insoluble salt will precipitate. In Table 1 solubilities of all related compounds in water are given.

Table 1: Solubilities of related compounds in water.

Temperature ^0C	NaCl g / 100 g H_2O	KCl g / 100 g H_2O	$Na_2Cr_2O_7.2H_2O$ g / 100 g H_2O	$K_2Cr_2O_7$ g / 100 g H_2O
0	35.7	27.6	143	5
20	36.0	34	178	12
40	36.6	40	223	26
60	37.3	45.5	280	43
80	38.4	51.1	376	61
100	39.8	56.7	---	80

Pre-laboratory Work:

Before the experiment in the laboratory, you should be able to answer these questions:

1. List five physical properties.
2. What are the differences between physical and chemical changes?
3. Define solubility and discuss the factors affecting solubility.
4. What is density? How do you calculate it?

Materials

Potassium permanganate ($KMnO_4$)	Sodium dichromate dihydrate ($Na_2Cr_2O_7.2H_2O$)	50 mL beakers
Starch	Ethyl alcohol	Balance
Sodium carbonate (Na_2CO_3)	Distilled Water	Graduated cylinder
Sodium sulfate (Na_2SO_4)	Diethyl ether	Ring, clamp
Nitric acid (HNO_3)	Boiling chips	Filter paper
Sulphuric acid (H_2SO_4)	Bunsen burner	Thermometer
Potassium chloride (KCl)	One holed rubber stopper	

Procedure

Part A: Separation by Distillation	
1. Pour 30 mL potassium permanganate solution (solid $KMnO_4$ dissolved in water) into a 100 mL round bottom flask. Add some boiling chips into the flask to make solution boil calmly.	
2. Set a simple distillation apparatus by inserting the short end of the glass tubing acting as condenser in a one-holed rubber stopper.	
3. Ask your assistant how to use the Bunsen burner. Light it and adjust until you have a small and continous hot flame.	
4. Heat the $KMnO_4$ solution and observe the hot solvent vapors of the solution are cooled and dripped into the test tube. Continue distillation process until about 10 mL of liquid have distilled over. Observe the differences in color between the distillate and the original solution; write these observations on your data sheet.	

Part B: Identifying Substances by Their Properties

B.1. Solubility in Water

1. Fill a test tube to the half width distilled water and add a sample of the substance.

2. Shake the test tube several times and observe whether or not the substance dissolves. Some substances dissolve slowly, so you may have to wait a few minutes to be sure.

B.2. Behavior on Heating

1. Place a small quantity of a substance on a clean piece of porcelain and heat moderately

2. Record all changes like color, gas evolution, melting, odor, etc.

3. Be sure that you have cooled the hot porcelain by placing it under flowing cold water after your test.

B.3. Solubility in Nitric Acid (HNO_3)

1. Fill a test tube to the half with dilute HNO_3 and test the solubility of the substances as you did with water in test 1.

Part C: Fractional Crystallization and Recrystallization

1. Put about 3 g of the sodium dichromate dihydrate, $Na_2Cr_2O_7.2H_2O$ into a beaker. Add 4 mL water to the $Na_2Cr_2O_7.2H_2O$ and heat the solution just to the boiling point. Stir until the solid dissolves completely.
2. Put 5 g KCl into a flask. Add 13 mL distilled water to the KCl and heat the solution just to the boiling point. Stir until the solid dissolves completely.
3. Mix the $Na_2Cr_2O_7.2H_2O$ solution with the KCl solution well by swirling. Record the volume of the solution.
4. Cool the solution by passing cold water over the outside of the flask until no more crystals appear to form. Measure the temperature of the solution.
5. Pour the solution through a filter. Your assistant will show you how to use filter paper and funnel (see Figure 1).
6. Which salt is precipitated? (Hint: See Table 1)

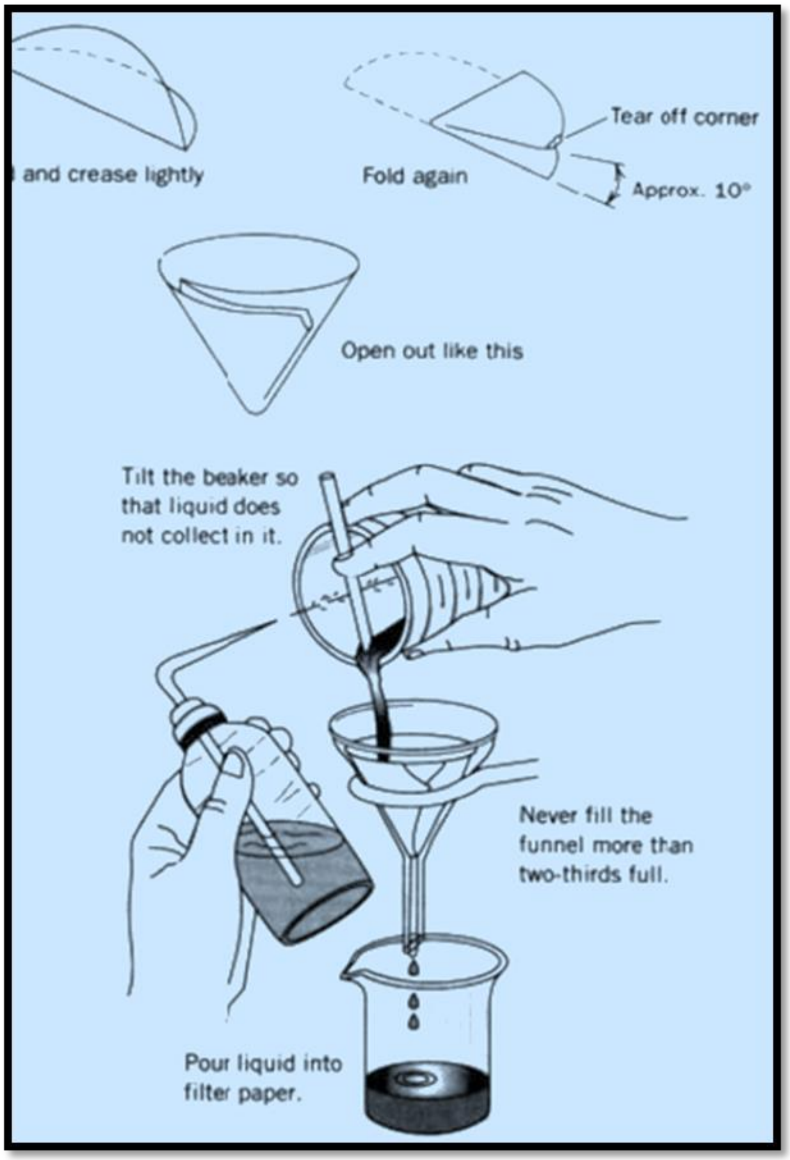

Figure 1: Technique for folding filter paper and transfer of precipitate from the beaker into the filter paper.

DATA SHEET

Introductory to Laboratory Techniques

Student's Name : Date:

Laboratory Section/Group No :

Assistant's Name and Signature:

A. Separation by Distillation

i. What was the color of the original ($KMnO_4$) solution?

ii. What is the color of the distillate which is collected in the test tube?

iii. Why did we collect water as distillate in test tube? (Hint: Consider the boiling point differences of distinct substances)

B. Identifying Substances by their Properties

	B.1.Solubility in water	**B.2.Behavior on heating**	**B.3.Solubility in HNO_3 solution**
$CuSO_4$			
Starch			
Na_2CO_3			
$BaSO_4$			
Na_2SO_4			
Sugar			
MgO			
Unknown			

C. Fractional Crystallization and Recrystallization

i) Volume of solution:..................mL

ii) Temperature of solution:…………°C

iii) The compound that is precipitated is..........................

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Experiment 2

The Law of Definite Proportions

Purpose: To understand " the law of definite proportions" concept and to learn how to make gravimetric analysis (analysis by weighing) calculations.

Prelaboratory Work

Before the experiment in the laboratory, you should be able to answer these questions.

1. Define the terms "compound" and "catalyst".

2. How do empirical and molecular formulas differ?

3. Calculate the percent composition of HNO_3? (H: 1 g/mol; N: 14 g/mol; O: 16 g/mol)

4. What is law of definite proportions?

5. How many lithium atoms are present in 0.01456 g of lithium?

6. What is percent oxygen by weight in water molecule?

7. Given that zinc chloride has a formula weight of 136.28 g/mol, what is its formula?

Theory

The law of definite proportions states that a chemical compound always contains exactly the same **proportion** of elements by mass. Law of definite proportions shows a good way to find percent weight or exact weight of a desired element in a compound. It also gives useful information to find empirical or molecular formula for a compound and percent weight of a compound in an unknown mixture.

Example: What is the % O by weight inV_2O_5? (Atomic weights are; V: 50.9 g/mol, O: 16 g/mol)

Solution: First, we must find the total weight of the compound. Then we will divide the desired element's weight by the total weight as in follow:

$$O\% = \frac{5\times(16.0)}{2\times(50.9)+5\times(16.0)}\times 100 = 44.0\%$$

In today's experiment, potassium chlorate ($KCI0_3$) will be decomposed into potassium chloride (KCI) and oxygen (0_2) by heating ($Mn0_2$ will be used as a catalyst to speed up the reaction without being consumed.) :

$$2KClO_3(s) \xrightarrow{\Delta} 2KCl(s) + 3O_2(g)$$

As it is seen from the reaction equation, oxygen gas releasing upon decomposition results with the weight loss of initial compound. In other words, the weight difference gives the weight of oxygen in the compound.

At the end of the experimental part, theoritical and experimental percent oxygen by weight for $KCIO_3$will be calculated and compared. Comparison will give the Percent Error for the experiment.

Percent error is the ratio of the absolute value of the error to the theoritical value and multiplied by 100.

Error: experimental value – theoritical value

% Error: $\frac{|\text{experimental} - \text{theoritical}|}{\text{theoritical}}\times 100$

Example: A chemical compound theoritically contains 39.2 % O by weight. In a laboratory, % O by weight for this compound was found as 36.3 %. Calculate the error and % error of this experiment.

Solution:

Error $= 36.3 - 39.2 = -2.9$

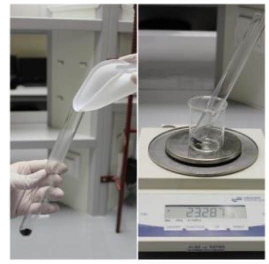
% Error = $\frac{|36.3 - 39.2|}{39.2} \times 100 = 7.4\%$

Materials

Manganese (IV) oxide (MnO_2)	Test tube	Bunsen Burner
Potassium chlorate ($KClO_3$)	Balance	Wooden Tongs
Potassium chloride (KCl)		

Procedure

Part A: Percent Oxygen in Potassium Chlorate	
Drying the Catalyst	
1. Put about a tea-spoon of MnO_2 in a dry test tube. Heat the test tube in order to remove the moisture of the catalyst. Move the test tube continuously on the flame.	
2. After test tube is cooled to room temperature, weigh it (W_1).	

Decomposition Reaction	
1. Add about between 2 - 4 g of $KClO_3$ into the test tube and weigh again (W_2). Calculate the weight of $KClO_3$ (W_3=W_2-W_1).	
2. Start to heat the test tube in a diagonal position first gently, then more strongly. Heat the entire test tube to redness, and maintain the temperature for fifteen minutes. The mixture will first melt, then effervesce (produce gas) strongly, and finally solidify. **DON'T KEEP OPEN SIDE OF THE TEST TUBE TOWARDS YOUR AND YOUR LAB-MATES FACES! Oxygen release can sputter very hot content as well! Move the test tube continuously on the flame, otherwise the glass may melt.**	
3. Cool the test tube slowly and weigh (W_4).	
4. Heat the test tube and the contents to redness for additional five minutes. Cool and reweigh (W_4).	
5. Repeat Step 8 until your last weight will be the same with previous one. Your last weighing is W_f. Same weight means; you removed all of the oxygen from your compound. Calculate the weight of oxygen given off, W_{ox}. Calculate experimental percent oxygen by weight in $KClO_3$. Calculate the theoretical percent of oxygen in $KClO_3$. The atomic weights are as follows: O=16.0 g/mol; Cl=35.5 g/mol; K=39.l g/mol. Calculate % Error as explained in theoritical part.	

Part B: Analysis of a $KCIO_3$ - KCI Sample	
1. The composition of an unknown $KClO_3$ – KCl will be determined with the same procedure as in Part A. Take your unknown sample from your assistant. Follow the same procedure used in Part A with the unknown mixture instead of pure $KClO_3$. Use the same notations. (Calculate the percent $KClO_3$ by weight in your unknown sample (see your data sheet).	

Questions

1. How many kilograms of copper sulfide could be formed from the reaction of 2.70 mol of copper with excess sulfur?
2. Given that zinc chloride has a formula weight of 136.28 g/mol, what is its formula?
3. Calculate the percent composition of HNO_3? (H: 1 g/mol; N: 14 g/mol; O: 16 g/mol)

DATA SHEET

The Law of Definite Proportions

Student's Name : Date:

Laboratory Section/Group No :

Assistant's Name and Signature:

A. Percent Oxygen in Potassium Chlorate

1. Weight of test tube and catalyst (W_1) .... g
2. Weight of test tube, catalyst and $KClO_3$ (W_2) .... g
3. Weight of $KClO_3$ (W_2-W_1= W_3) .... g
4. Weight of the test tube and the contents after first heating (W_4) .... g
5. Weight of the test tube and the contents after second heating (W_5) .... g
6. Weight of the test tube and the contents after third heating (W_6) .... g
7. Weight of the test tube and the contents after last heating (W_f) .... g
8. Weight of oxygen given off ($W_2 – W_f= W_{ox}$) .... g
9. Experimental % of oxygen [(W_{ox} / W_3) × 100] .... %
10. Theoretical % of oxygen by weight in $KClO_3$ .... %
11. Percent error .... %

B. Analysis of a $KClO_3$ - KCl Sample

12. Weight of test tube and catalyst (W_1) .... g
13. Weight of test tube, catalyst and unknown (W_2) .... g
14. Weight of unknown ($W_2 – W_1= W_3$) .... g
15. Weight of the test tube and the contents after first heating (W_4) .... g
16. Weight of the test tube and the contents after second heating (W_5) .... g
17. Weight of the test tube and the contents after third heating (W_6) .... g
18. Weight of the test tube and the contents after last heating (W_f) .... g
19. Weight of oxygen given off (W_2-W_f = W_{ox}) .... g
20. Percent oxygen by weight in unknown .... %
21. % $KClO_3$ in sample .... %

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Experiment 3

Stoichiometry: The Reaction of Iron with Copper(II) Sulfate

Purpose: To enhance the understanding of stoichiometry, a reaction between iron and copper (II) sulfate solution will be conducted. This will help you to differentiate limiting and excess reactant in a chemical reaction. Finally the theoretical and percent yield of this reaction will be calculated.

Theory

Stoichiometry is the measurement of quantitative relationships in chemical formulas and equations. In this experiment stoichiometric principles will be used to obtain the appropriate equation between the reaction of iron metal and copper(II) sulfate solution. After the reaction is taking place, the formation of metallic copper, which is seen precipitating as a finely divided reddish-orange powder will be observed. This reaction is one of the example of single substitution reaction in which one element "displaces" from a compound by another element. The element which has ability of displacing other element from compound is said to be "more active" than the displaced metal. In this experiment, iron is more active than copper.

Two distinct forms of iron are present, namely Fe^{2+} and Fe^{3+}. Stoichiometric principles will be used to determine which reaction is more dominant compared to other one by examining the reaction between iron and copper (II) sulfate solution. If Fe^{2+} is formed, then **equation (1)** is dominant, while **equation (2)** will be selected if Fe^{3+} is formed. This can be determined according to mole ratio of copper to iron. If the moles of copper is equal to the moles of iron, then equation (1) has taken place. If you obtain 1.5 moles of copper per mole of iron, in this case equation (2) should be selected Find out which equation is corresponding to the results of the experiment you have done.

Equation (1) $Fe_{(s)} + CuSO_{4(aq)} \rightarrow FeSO_{4(aq)} + Cu_{(s)}$, other representation is

$$Fe_{(s)} + Cu^{2+}{}_{(aq)} \rightarrow Fe^{2+}{}_{(aq)} + Cu_{(s)}$$

Equation (2) $2\ Fe_{(s)} + 3\ CuSO_{4(aq)} \rightarrow Fe_2(SO_4)_{3(aq)} + 3\ Cu_{(s)}$, other representation is

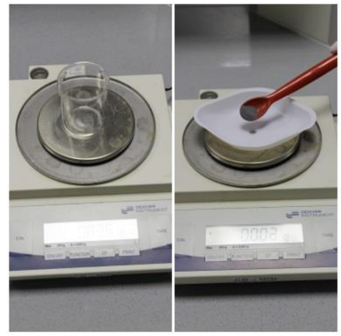
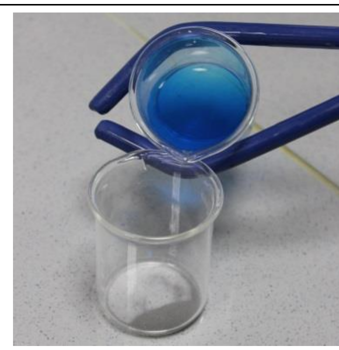
$$2\ Fe_{(s)} + 3\ Cu^{2+}{}_{(aq)} \rightarrow 2\ Fe^{3+}{}_{(aq)} + 3\ Cu_{(s)}$$

To the known amount of iron, excess of copper (II) sulfate solution will be added. The purpose of using excess solution is owing to provide the complete reaction of iron. The metallic copper produced will be weighed after washing and drying processes and these weighings will be used to calculate the moles of iron used and the moles of copper formed.

Materials

Fe powder	Acetone	Glass stick
Cupper (II) Sulfate ($CuSO_4$)	Beaker	Bunsen burner

Procedure

The Reaction of Iron with Copper(II) Sulfate	
1. Weigh a dry and clean 100 or 250 mL beaker and record the weight of it onto your data sheet. then, accurately weigh 1.00 gram of iron powder into this beaker. Do not exceed 1.01 grams.	
2. Measure 30 mL of 1.0 M $CuSO_4$ solution by using a graduated cylinder. Pour this solution into another beaker, and heat gently to almost boiling.	
3. Slowly add hot $CuSO_4$ solution to the beaker that contains the iron powder. Stir the mixture a few times until completeness of the reaction. You should see copper forming. When the reaction has finished, allow the copper product to cool.	
4. Then carefully decant the liquid from the copper into the waste container. Be careful not to lose any copper.	

5. Add about 10 mL of distilled water to the solid copper and swirl to wash any remaining ions from the copper. Decant the wash water from the copper and add 10 more mL of distilled water, swirl and decant again. Wash copper particles finally with several mL of acetone (**CAUTION**-Acetone is very flammable). Swirl and allow to stand a few minutes. Decant off the acetone.	
6. The acetone readily dissolves the water and helps the removal of it from the medium. Swirl the beaker gently on low heat flame. Copper product should be spread in a single layer on the bottom of the beaker. Grinding of aggregates with a spatula makes the copper easy to dry. Be sure not to remove any copper from the beaker.	
7. After drying, allow copper to cool and weigh the beaker plus copper to calculate the mass of copper formed. Record the mass on your data sheet. Finally, calculate the moles of iron used and the moles of copper formed to determine which reaction of iron is taking place, reaction (1) or reaction (2).	

DATA SHEET

Stoichiometry: The Reaction of Iron with Copper(II) Sulfate

Student's Name : Date:

Laboratory Section/Group No :

Assistant's Name and Signature:

Data and Calculations

Mass of empty beaker :

Mass of iron used :

Moles of iron used :

Mass of beaker plus copper :

Mass of copper formed :

Moles of copper formed :

Moles of Cu divided by moles of Fe :

Reaction Equation :

Limiting Reagent :

Theoretical and Percent Yield :