CHEMISTRY LABORATORY NOTE

Course No. CHEM 112

Fall 2018

Department of Computer Science and Engineering

Name	
Student ID	
Department	
Section	

Class duration: 3 h (1.5 Credits)

PURPOSE OF CHEM112

The main purpose of this course is to develop the basic knowledge of a student dealing with chemical reagents. The student will learn how to handle equipments (basic to every research laboratory). The student will also learn techniques, how to determine the amount of a substance quantitatively. The teacher will deliver some lectures to develop the fundamental knowledge of the students regarding the techniques of performing the experiments. The skill of the students will be developed on the basis of the following topics.

- ◈ Elementary concepts of quantitative analysis.
- ◈ Chemical composition of solutions: weights and concentration units, stoichiometric relations, problems related to these concepts.
- ◈ Introduction to volumetric analysis: fundamental definitions, titration curves of acid-base and oxidation - reduction reactions, volumetric calculations.
- ◈ Techniques and tools of volumetric analysis.
- ◈ Primary standard and Secondary standard substances.
- ◈ Evaluation of analytical data.

ADVICE TO THE STUDENTS:

Chemistry laboratory is a place where seriousness is very much essential. Any time a serious accident may happen due to carelessness of the student. If there is an accident it will never be reversible. Student will not get a second chance to recover it. Understanding of what to do, will serve well in minimizing the danger for the student. **Advance preparation for the sessional class is very much important.** To get good result in the sessional course several things a student should to do.

Firstly, student should understand this Laboratory manual clearly and how to use it effectively. It is his duty to learn it carefully.

Secondly, he must try to understand both the purpose and the principle behind each of the experiment he will do.

Thirdly, he will organize his time effectively in advance of each experiment. As in the Chemistry Laboratory a student has to work with various hazardous chemicals, he should wear an APRON during his sessional work.

To avoid unnecessary hazards of explosion a student should never pour any reagent back into a stock solution bottle. If you do this then a student may introduce impurities which could spoil the experiment for the person using the stock reagent after him. So pouring things back into bottle is not only a dangerous practice but also unconsidered.

A student should not undertake any unauthorized experiment. The chances of an accident are high particularly with an experiment which has not been completely checked to reduce the hazard. Because a chemistry laboratory course deals with flammable solvent at all times the danger of fire is always present. Because of this danger **DO NOT PUT INFLAMED STICK OF MATCHES HERE AND THERE. SMOKING IN THE LABORATORY IS STRICTLY PROHIBITED.**

Because all chemicals are toxic to some extent, a student should never eat or drink in the laboratory. There is always the possibility that whatever a student eats or drinks it may become contaminated with the toxic material.

LABORATORY GLASSWARE:

Since laboratory glassware is very expensive and the person who is working with this glassware is responsible for it, student should give proper attention and take special care of it. Needless maltreatment of laboratory equipments may cost student in big amount of money.

Mistreatment of equipment can also cause of losing time in the laboratory. A student should be familiar with the equipments of his experiment.

CLEANING OF GLASSWARE:

Glassware can be cleaned more easily if it is cleaned immediately after use. It is good practice to wash the equipment right away. With time the materials left in the apparatus begin to stick on the surface of the glass. Various washing powder and cleaning agents may be used as aids in the washing glassware, synthetic detergents may also be used. Organic solvent may be used in cleaning if the adhering substance is organic one. When solvents are used in cleaning glassware caution should be taken since the solvents are hazardous. Acetone is a commonly used solvent for cleaning glassware, but it is expensive. Don't use reagent grade acetone for cleaning purpose. Waste acetone can be reused effectively several times before it is spent. For troublesome stains and residues which remain adhering to the glassware inspite of your best effort, a cleaning solution may be used. **The most commonly used cleaning solution consists of 35 mL of saturated aqueous potassium dicromate solution dissolved in one liter concentrated sulfuric acid**. A cleaning solution is to be prepared by dissolving sufficient dichromate in one-liter conc. sulfuric acid to prepare a saturated solution. It is much more hazardous to add the aqueous solution to concentrated acid since spattering may result. The preparation of cleaning solution is quite hazardous, it should not be undertaken without careful preparation. Don't allow cleaning solution to come in contact with your skin or your clothing. Always use small amount of cleaning solution. Swirl the cleaning solution in the glassware for a few minutes and pour the remaining cleaning solution into the stock bottle. Be sure that the flask is rinsed with sufficient amount of water.

REPORT WRITING:

Students are advised to follow the following sequences in order to submit their report on the experiment that will be carried out in this course:

1. Objective: Aim and objective of the experiment

2. Introduction:

Theory of the process (students should mention the name and objective of the corresponding experiment), the methods, chemical reaction(s) involved in the corresponding experiment, types of indicators if necessary for a particular experiment should be included in this session.

2. Name of the chemicals:

With formula used in each experiment.

3. Experimental data:

The student should prepare a table (one must mention properly the title i.e. what one is going to be determine. Student should also mention accurately the units of its parameters and experimental conditions like, whether the experiment is carried out in acidic or basic media, experimental temperature etc.).

4. Calculations:

Students are advised to report their calculations in terms of Normality as well as Molarity. It is advisable that the students must have a proper clear concept of UNITS, specially of Concentrations Units.

5. Results:

Briefly mention the observed value(s).

6. Percentage of Error:

One should calculate % of error by using the following formulae:

$$\frac{\text{Known value} - \text{Observe value}}{\text{Known Value}} \times 100$$

7. Discussion:

Preference should be given on what sort of precautions should one take during conducting the experiment; what are the possible errors that cause the deviation of the result from the expected value.

MARKS DISTRIBUTION:

The performance of a student will be evaluated according to the following:

Attendance	**20%**
Report writing	**20%**
Quiz Examination	**20%**
Lab Exam	**20%**
Viva	**20%**
Total	**100%**

REF. BOOKS:

1. **A Text Book of Quantitative Inorganic Analysis, *by*- A.I. Vogel.**
2. **Practical Chemistry, *by*- Mahbubul Huq and Jabbar Mian.**

SOME BASIC CONCEPTS REQUIRED FOR THIS COURSE:

For acid- base reactions:

⇨ Definition of acid and base
⇨ Strength of acid and base
⇨ Neutralization reaction
⇨ The pH of the solution and pH scale
⇨ Ion - product constant of water
⇨ Acid - base - ionization equilibrium
⇨Buffer solution and buffer action and their uses Henderson equations and their use
⇨Acid-base titration curves, Acid-base indicator and their uses
⇨Definition of titrant, titrand, titration equivalence point, etc.
⇨Type of titration
⇨Standard substances and standard solutions
⇨Concentration of solutions and their units

For Oxidation - Reduction reactions:

⇨ Definition of oxidization-reduction (Redox) reactions.
⇨ Oxidation number and oxidation state.
⇨ Oxidizing agent/oxidant and reducing agent /reluctant.
⇨ Oxidation number.
⇨ Type of oxidation-reduction reactions and balancing oxidation-reduction reactions.
⇨ Electro-chemical cells and half- reaction.
⇨ Potential difference and electromotive force (EMF) and standard electrode-potentials.
⇨ Strength of oxidizing - reducing agents.
⇨ Example of some important application of redox reactions.

List of Experiments

Experiment No.	Title of the Experiment
01	**Standardization of hydrochloric acid with standard sodium hydroxide solution**
02	**Standardization of sodium hydroxide solution by titration with standard oxalic acid solution**
03	**Standardization of hydrochloric acid with standard sodium carbonate solution**
04	**Standardization of sodium thiosulphate solution with standard potassium dichromate solution**
05	**Estimation of copper in a supplied solution by iodometric method**
06	**Determination of ferrous ion by titration with standard potassium dichromate solution**
07	**Standardization of potassium permanganate solution by titration with standard oxalate solution**
08	**Determination of ferrous ion in a supplied solution by titration with standard potassium permanganate solution**

Experiment No. 01	Standardization of hydrochloric acid with standard sodium hydroxide solution

Name	
Student ID-	Section-
Group No-	Date-

Procedure:

Take 10 mL of NaOH in a conical flask and add about 50 mL of distilled water. Add 2 or 3 drops of methyl orange indicator to the solution. Then titrate the solution with HCl solution drop wise from a burette. Shake the flask frequently during addition of NaOH. Stop addition of acid solution as soon as the color of the solution changed from yellow to orange color. Note the burette reading. Repeat the process at least two times and these should agree within ± 0.1 mL. Calculate the molarity of the dilute HCl solution.

Table- Determination of the volume of hydrochloric acid solution required for the titration of supplied standard sodium hydroxide soltution

Observation Number	Volume of NaOH Solution Taken (mL)	Burette Reading of HCl Solution (mL)			Required Volume of HCl Solution (mL)
		Initial Burette Reading	Final Burette Reading	Difference	
1					
2					
3					

Calculation:

Experiment No. 02	Standardization of sodium hydroxide solution with standard oxalic acid solution

Name	
Student ID-	Section-
Group No-	Date-

Procedure*:*
Take 10 mL of supplied NaOH solution in a conical flask by means of a pipette and dilute it to about 50 mL. Add one drop of phenolphthalein indicator to the solution. Then add prepared oxalic acid solution drop by drop from a burette. Shake the flask frequently while adding the acid solution. Stop the addition of oxalic acid solution as soon as the pink color disappears. Note the burette reading. The burette reading should be taken carefully at the lower meniscus of the liquid. Difference between the initial and final burette reading gives the volume of the acid required for the titration. The process should be repeated at least twice. These data should agree within ± 0.1 mL. Calculate the molarity of the supplied NaOH solution.

Table- Determination of the volume of the standard oxalic acid solution required for the titration of supplied sodium hydroxide

Observation Number	Volume of NaOH Taken (mL)	Burette Reading of Oxalic Acid Solution (mL)			Required Volume Oxalic Acid (mL)
		Initial Burette Reading	Final Burette Reading	Difference	
1					
2					
3					

Calculation:

Experiment No. 03	Standardization of hydrochloric acid with standard sodium carbonate solution

Name	
Student ID-	Section-
Group No-	Date-

Procedure:

Take 10 mL of standard Na_2CO_3 solution in a conical flask and dilute it to about 50 mL. Add one drop of phenolphthalein and titrate against HCl from a burette. Now note the burette reading when just one drop of HCl discharges the pink color of the solution. This is the first end point. Then add 3-4 drops of methyl orange inside the same conical flask and continue titration against the same HCI acid. The end point reached when the yellow color of the solution just changes to faint pink (Orange). Note the burette reading. This is the second end point. The difference of the burette reading from initial to second end point will be the volume of the acid required in the titration. Repeat the whole experiment twice and these should agree within ± 0.1 mL initial to second end point. Calculate the strength of dil. HCl acid and then find out the normality of commercial conc. HCl acid.

Table- Determination of the volume of dilute hydrochloric acid solution required for the titration of standard sodium carbonate solution

Obs. No.	Volume of Sodium Carbonate Solution (mL)	Burette Reading of Dilute Hydrochloric Acid Solution (mL)				Required Volume of HCl for Final Titration (mL)
		Initial Burette Reading	Final Burette Reading		Difference	
			Phenolphthalein	Methyl orange		
1						
2						
3						

Calculation:

Experiment No. 04	Standardization of sodium thiosulphate solution with standard potassium dichromate solution

Name	
Student ID-	Section-
Group No-	Date-

Procedure:

Take 4 mL of 12 % potassium iodide (KI) solution in a conical flask and dilute it to about 50 mL. Add about 1 g of $NaHCO_3$ and shake the flask until the salt dissolves. Add about 4 mL concentrated HCl acid and then add 10 mL standard $K_2Cr_2O_7$ solution by means of a pipette in the same flask. Shake the flask and cover it with a watch glass, allow the solution to stand for about five minutes in the dark (inside the desk). Rinse the watch glass and dilute the solution about 100 mL. Titrate the liberated iodine with sodium thiosulphate solution from a burette until the brown color fades (light yellow). Add about 1 mL starch solution and continue titration by adding sodium thiosulphate solution from the burette until one drop of the sodium thiosulfate solution changes the color of the solution from deep blue to light green or light blue. This is the end point. Calculate the strength of sodium thiosulphate solution.

Table- Measurement of the volume of sodium thiosuphate for the iodometric titration of standard potassium dichromate solution

Observation Number	Volume of $K_2Cr_2O_7$ Taken (mL)	Burette Reading of Sodium Thiosuphate Solution (mL)			Required Volume of $Na_2S_2O_3$ (mL)
		Initial Burette Reading	Final Burette Reading	Difference	
1					
2					
3					

Calculation:

Experiment No. 05	Estimation of copper in a supplied solution by iodometric method

Name	
Student ID-	Section-
Group No-	Date-

Procedure:

Pipette out 10 mL of supplied copper salt solution into a conical flask. Add a few drops of $NaHCO_3$ in to the solution. A pale greenish precipitate should appear. Dissolve the precipitate by adding few drops of acetic acid (CH_3COOH). Add about 10 mL of 10 % potassium iodide (KI) solution and titrate the liberated iodine against the standard thiosulphate solution until the brown color of iodine changes to light yellow. Add 1 mL of starch solution and continue titration till the blue color begins to fade. Now add few drops of 10 % Ammonium thiocyanate solution and continue titration until the blue color is just discharged. Calculate the amount of copper present in one liter of the supplied solution.

Table- Measurement of the volume of sodium thiosulphate required for the titration of supplied copper sulphate solution

Observation Number	Volume of CuSO4 Taken (mL)	Burette Reading of $Na_2S_2O_3$ Solution (mL)			Required Volume of $Na_2S_2O_3$ Solution (mL)
		Initial Burette Reading	Final Burette Reading	Difference	
1					
2					
3					

Calculation:

Experiment No. 06	Determination of ferrous ion by titration with standard potassium dichromate solution

Name	
Student ID-	Section-
Group No-	Date-

Procedure:

Take 10 mL of the supplied iron (Mohr's salt) solution in a conical flask. Add 50 mL 1 M sulfuric acid and 5 mL of concentrated phosphoric acid. Then add 4-5 drops of diphenylamine indicator and titrate slowly against standard potassium dichromate solution dropwise maintaining an interval of few seconds between each drop until the addition of one drop causes the formation of intense purple or violet blue coloration which remains permanent and is unaffected by further addition of dichromate solution, Repeat the experiment at least twice. These should agree within ± 0.1 mL. Calculate the amount of iron per liter of solution.

Table- Measurement of the volume of potassium dichromate solution required for the titration of supplied Morh's salt

Observation Number	Volume of Morh's Salt Taken (mL)	Burette Reading of $K_2Cr_2O_7$ Solution (mL)			Required Volume of $K_2Cr_2O_7$ (mL)
		Initial Burette Reading	Final Burette Reading	Difference	
1					
2					
3					

Calculation:

Experiment No. 07	**Standardization of potassium permanganate solution by titration with standard oxalate solution**

Name	
Student ID-	**Section-**
Group No-	**Date-**

Procedure:

Pipette out 10 mL 0.05 M standard oxalate solution in a conical flask. Add about 50 mL 1 M sulfuric acid solution to it. Heat the solution upto 60 to 70 ^{o}C and then add 4-5 drops of $KMnO_4$ solution from a burette. Shake the solution until the color disappears. Continue to add permanganate solution from the burette at a moderate rate and shake the clear solution continuously. As the color disappears more slowly, slow down the addition of permanganate solution dropwise until one drop gives a light but permanent pink color. This is the end point of the titration. Repeat the experiment thrice and calculate the molarity of permanganate solution.

Table- Measurement of the volume of potassium permanganate solution required for the titration of standard sodium oxalate solution

Observation Number	Volume of $Na_2C_2O_4$ Taken (mL)	Burette Reading of $KMnO_4$ Solution (mL)			Required Volume of $KMnO_4$ (mL)
		Initial Burette Reading	Final Burette Reading	Difference	
1					
2					
3					

Calculation:

Experiment No. 08	**Determination of ferrous ion in a supplied solution by titration with standard potassium permanganate solution**

Name	
Student ID-	**Section-**
Group No-	**Date-**

Procedure:

Pipette out 10 mL of iron solution (Morh's salt) in a conical flask. Add about 6 mL of 1 M sulfuric acid solution. Dilute it to about 50 mL distilled water. Titrate the resulting solution with standard potassium permanganate solution. The end point is indicated by the first appearance of permanent light pink color as in Expt. No. 7. Repeat the titration at least thrice. Calculate the amount of iron of the supplied solution.

Table- Measurement of the volume of potassium permanganate solution required for the titration of supplied Mohr's salt solution

Observation Number	Volume of Morh's Salt Taken (mL)	Burette Reading of $KMnO_4$ Solution (mL)			Required Volume of $KMnO_4$ (mL)
		Initial Burette Reading	Final Burette Reading	Difference	
1					
2					
3					

Calculation: