ISLAMIC UNIVERSITY OF TECHNOLOGY (IUT)
Department of Computer Science & Engineering (CSE)

Course: **Chem-4242**
Quantitative Inorganic Analysis **(Credit: 3 hours / alternative week)**.
For First year B. Sc. Engineering (CSE)

PURPOSE

The main purpose of this course is to develop the knowledge of a student regarding the quantitative analysis of a substance. The teacher will deliver necessary lectures to develop the fundamental knowledge of the students regarding the techniques of performing the experiments for analysis. The skill of the students will be developed on the basis of the following topics.

- Concepts of quantitative analysis.
- Solutions: Concentration units, stoichiometric relations, problems related to these concepts.
- Introduction to volumetric analysis: fundamental definitions, titration involving neutralization reactions, oxidation - reduction reactions & calculations involved.
- 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. A serious accident may occur due to carelessness. Understanding of what to do and how to do will surely minimize the danger in the chemistry lab. **The preparation in advance for the sessional class is very much important.** To get good experimental result several things a student should strive to do:

1. Student should read this laboratory manual properly and should use it effectively.

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

3. He should utilize his time effectively in his laboratory class. In the Chemistry Laboratory, students are advised to wear APRON.

4. Students are advised not to pour any reagent / chemical back into a stock solution bottle.

5. A student must not undertake any unauthorized experiment. INFLAMED STICK OF MATCHES MUST NOT BE PUT HERE AND THERE. SMOKING IN THE LABORATORY IS STRICKLY PROHIBITED. All chemicals are toxic/ poisonous to some extent, a student should never eat or drink.

NOTE BOOK:

For recording data obtained during experiment: **"A BOUND NOTE ROOK"** should be used. All primary data must be recorded in the note book. It is a bad practice to record primary data on loose sheet, or perishable paper. Data must be recorded in permanent ink. Teacher may check the students' note book any time. So, a student should always keep it up to date. The preparation of the

reports is accomplished using the material recorded in the laboratory note book. Nothing should be kept on the working table, except instruction manual, note book and calculator.

LABORATORY GLASSWARE:

Since laboratory glassware is very expensive, so student should give proper attention and take special care of it. Needless maltreatment of laboratory equipments may cost student in big amount of money.

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 attack 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 of cleaning purpose. West acetone can be reused effectively several times before it is spent. For troublesome stains and residues which remain adhering to the glassware in spite of your best effort, a cleaning mixture may be used. **The most commonly used cleaning mixture consists of 35 mL of saturated aqueous potassium dichromate solution mixed in one-liter concentrated sulfuric acid solution**. The preparation of cleaning mixture is quite hazardous, it should not be undertaken without careful preparation. Don't allow cleaning mixture to come in contact with your skin or your clothing. Always use small amount of cleaning mixture. Swirl the cleaning mixture in the glassware for a few minutes and pour the remaining cleaning mixture into the stock bottle. Be sure that the flask is rinsed with sufficient amount of tap water and then distilled water.

REPORT WRITING:

Students are advised to follow the following sequences in order to write their report on the experiment done in the class.

l. Introduction:

Theory of the process (students should mention the name and objective of the experiment) the methods, chemical reaction involved in the experiment and the types of indicators used.

2. Chemicals used:

The name of the chemicals / reagents used in the experiment should be mentioned.

3. Experimental data:

Each table will have a title for which it is meant. Student should mention accurately the units of the parameters and experimental conditions like, whether the experiment is carried out in acidic or basic media, experimental temperature etc.

4. Calculations:

The students must have a proper clear concept of units, specially of concentration units used in the calculation of results.

5. Results:

Briefly mention the result / results obtained.

6. Percentage of Error:

$$\text{The percentage (\%) of error} = \frac{\text{Known value - Observed value}}{\text{Known value}} \times 100$$

7. Discussion:

Preference should be given in discussion on what sort of precautions have been taken during conducting the experiment and what are the possible sources of errors that cause the deviation of the result from the expected value. Justification of using a particular indicator / chemical other than the main reactants used should also be mentioned.

8. Books consulted:

The books consulted for the preparation of the report should be mentioned.

Recommended reference books:

1. **Vogel's Text Book of Quantitative Chemical Analysis Forth/Fifth edition, Revised by G. H. JEFFERY and others.**
2. **Practical Chemistry *by*- Mahbubul Huq and Jabbar Mian.**

MARKS DISTRIBUTION:

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

Class Performance and Attendance	**05**
Report writing	**15**
Practical Examination	**15**
Viva Voce	**10**
Quiz Examination	**15 × 2 = 30**
Total Marks	**75**

EXPT. NO.: l.

STANDARDIZATION OF NaOH SOLUTION WITH STANDARD OXALIC ACID SOLUTION:

PREPARATION OF N/10 OXALIC ACID SOLUTION (MW = 126):

Transfer 0.63 g. of pure oxalic acid (HOOC-COOH. $2H_20$) in a 100 mL measuring flask and dissolve in distilled water and make up to the mark. Normality of the prepared acid solution will be calculated as:

$$N_{acid} = \frac{\text{Weight taken in g} \times 0.1\,\text{N}}{0.63\,\text{g}}$$

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 1-2 drops of Phenolphthalein indicator to the solution. Then add standard 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 colour of the solution just disappears. Note the burette reading. The burette reading should be taken carefully at the lower meniscus of the

liquid. Difference of the initial and final burette reading gives the volume of the acid added. The process should be repeated at least three times. These should agree within ± 0.1 mL. Calculate the normality of the supplied NaOH solution by using the following relation.

$$V_{acid} \times S_{acid} = V_{base} \times S_{base}$$

EXPT. NO. 2.
STANDARDIZATION OF HYDROCHLORIC ACID WITH STANDARD NaOH SOLUTION.

(a) Standardize the supplied NaOH solution as in Experiment No.1.
(b) **Preparation of approximately N/10 Hydrochloric acid (HCI) solution.**
Take about 1.2 - 1.3 mL conc. HCl in a 100 mL volumetric flask and add distilled water to make up to the mark.

PROCEDURE*:*
Take 10 mL of standard NaOH solution in a conical flask and dilute it to about 50 mL. Add 2 or 3 drops of methyl orange indicator to the solution. Then add previously prepared (approx. N/l0*)* HCl acid solution drop wise from a burette. Shake the flask frequently during addition of HCI acid. Stop addition of HCI acid solution as soon as the yellow color of the solution just changes to orange. Note the burette reading. Repeat the process at least three times and these should agree within ± 0.1 mL. Calculate the normality of the dilute HCl and then calculate the strength of commercial HCl. Use the following relations:

(a) $V_{HCl} \times N_{HCl} = V_{NaOH} \times S_{NaOH}$
(b) $V_{dilute\ HCl} \times S_{dil.\ HCl} = V_{conc.\ HCl} \times N_{conc.\ HCl}$

EXPT. NO.3
STANDARDIZATION OF HCl WITH STANDARD Na_2CO_3 SOLUTION
PREPARATI0N Of' 0.1N Na_2CO_3 SOLUTI0N (MW =106):
Transfer 0.53 g of anhydrous Na_2CO_3 in a 100 mL measuring flask and then dissolve with distilled water and make up to the mark.

PROCEDURE:

Take 10 mL of standard Na_2CO_3 solution in a conical flask and dilute to about 50 mL. Add one or two drops of phenolphthalein indicator and titrate with HCl solution (prepared as Expt.No.2) from a burette. Stop addition of HCI acid solution as soon as the pink color of the solution is discharged / vanished. This is the first end point. Then add 2-3 drops of methyl orange indicator in the same solution mixture and continue titration with the HCI solution. The end point is reached when the yellow colour 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 / final end point will be the volume of the acid required in the titration. Repeat the titration at least three times and take the mean value. Calculate the strength of dil. HCl acid and then find out the normality of supplied commercial conc. HCl acid.

N.B. Follow the formulae used in Expt. No. 2(a) and (b). to calculated the cone. of HCl.

EXPT. NO. 4
STANDARDIZATION OF SODIUM THIOSULPHATE SOLUTION WITH A STANDARD POTASSIUM DICHROMATE SOLUTION.

PREPARATI0N OF 100 mL 0.1N POTASSIUM DICHROMATE SOLUTI0N:

Transfer 0.49 g pure $K_2Cr_2O_7$ into a 100 mL volumetric flask and then dissolved with distilled water upto the mark.

PROCEDURE:

Take 4 mL of 12% potassium iodide (KI) solution in a conical flask and dilute to about 50 mL. Add about 1 g. of $NaHCO_3$ and shake the flask until the salt dissolves. Add about 4 mL. conc. 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 colour 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 colour of the solution from deep blue to light green or light blue. This is the end point. Calculate the strength of sodium thiosulphate solution using the following equation:

Formulae: $\mathbf{V_1 \times S_1 = V_2 \times S_2}$

EXPT. NO.5
ESTIMATION OF COPPER CONTAINED IN A SUPPLIED SOLUTION BY IODOMETRIC METHOD.

N.B. Standardize Sodium thiosulphate solution as Expt.No.4.

PROCEDURE:

Pipette out 10 mL. of supplied copper salt solution into a conical flask. Add a few drop of dil. Na_2CO_3. A pale greenish precipitate should appear. Dissolve the precipitate by adding few drop 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 (Standardized previously) until the brown colour of iodine changes to light yellow. Add 1 or 2 mL of starch solution and continue titration till the blue colour 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.

HINTS: Try to generate the following equation from which you can calculate the amount of copper per liter.

$$\mathbf{2CuSO_4 \equiv I_2 \equiv 2Na_2S_2O_3}$$

$$\mathbf{1\ mL\ 1N\ Na_2S_2O_3 \equiv 0.06354\ g\ of\ Cu^{2+}}$$