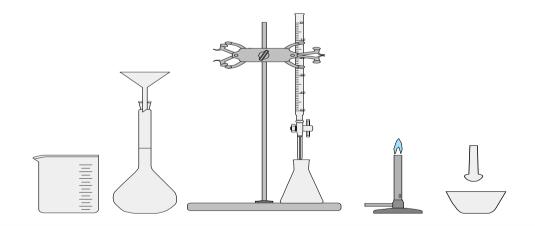
AMERICAN INTERNATIONAL UNIVERSITY-BANGLADESH (AIUB)

Department of Natural Science (Chemistry)
Faculty of Science & Technology

BSc Engineering (EEE, CoE, CSE & IPE) Program



Chemistry Lab Manual



ACADEMIC YEAR: 2021-2022

2021

Preface

Chemistry is a discipline based on observation (as are all sciences). In lecture student will learn the principles and theories that, to date, best explain the observations that have accumulated. The problem is that, if all students have in lecture, then it is all too easy to forget that these theories apply to the 'real world.' The laboratory experience is by design, students' opportunity to see these principles and theories in practice. Keeping this in view, AIUB Chemistry Lab has been in operation since January 2003 for its undergraduate programs B. Sc. in Electrical & Electronic Engineering (EEE), Computer Engineering (CoE), Computer Science & Engineering (CSE) and Industrial Production Engineering (IPE). This handbook having the following contents will help the students during their laboratory performance. Students should carry the manual during every lab class to get the full benefit of this handbook.

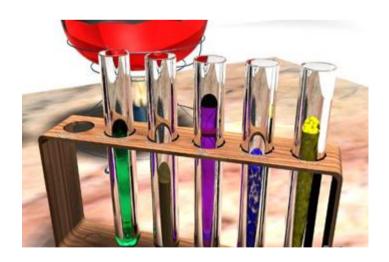
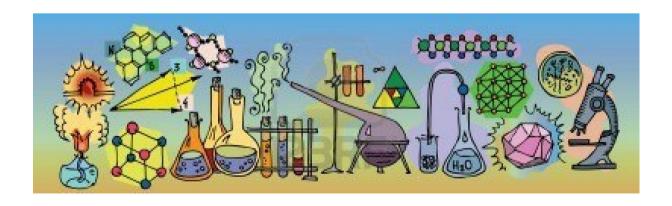


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Marks Distribution

Serial	Break-up	Marks
1	Class Performance and Attendance	10
2	Quizzes (2 Quizzes/Best 1)	30
3	Assignment	20
4	Viva	20
5	Comprehensive Quiz	20
	Total Marks (Mid/Final)	100
	Contribution for Grade	40%

Quantitative Inorganic Analysis

Main Objectives:

- To learn the technique how to determine the amount/concentration of a substance in a sample solution quantitatively.
- To develop basic knowledge of acid-base titration, redox titration and conductometric titration.
- To learn about chemicals and common analytical instruments.
- To know how to prepare solutions and how to handle glassware and instruments.

Fundamental Knowledge:

- 1. Concepts of qualitative analysis and quantitative analysis
- 2. Concentration units and problems related to these units
- 3. Volumetric analysis: acid-base titration, redox titration, conductometric titration volumetric calculations
- 4. Technique and equipments of volumetric analysis
- 5. Primary and secondary standard substances
- 6. Evaluation of analytical data

Some Basic Concepts

- Acid-Base Reactions
- Oxidation-Reduction Reactions
- Conductometric titration behaviour of acids and bases
- Complexometric titration

For Acid-Base Reactions -

- ✓ Definition of acid and base
- ✓ Strength of acid and base
- ✓ Neutralization reactions
- ✓ pH of a solution and pH scale
- ✓ Common-ion effect
- ✓ Buffer solution; action and uses
- ✓ Acid-base titration curves
- ✓ Acid-base indicators
- ✓ Standard substances and Standard solutions
- ✓ Concentration of solutions and their units

For Oxidation-Reduction Reactions –

- ✓ Definition of oxidation and reduction
- ✓ Oxidation number/oxidation state
- ✓ Oxidizing agent (Oxidant) and Reducing agent (Reductant)
- ✓ Types of oxidation and reduction reactions
- ✓ Balancing oxidation-reduction reactions
- ✓ Electro-chemical cells and half reaction
- ✓ Strength of oxidizing-reducing agents

List of the Experiments

(A) Acid-Base Titrations:

- 1. Standardization of sodium hydroxide (NaOH) solution with standard oxalic acid (C₂H₂O₄.2H₂O) solution.
- 2. Standardization of hydrochloric acid (HCl) solution with standard sodium hydroxide (NaOH) solution.
- 3. Standardization of hydrochloric acid (HCl) solution with standard sodium carbonate (Na₂CO₃) solution.

(B) Redox (Oxidation-Reduction) Titrations:

- 1. Standardization of sodium thiosulphate (Na₂S₂O₃.5H₂O) solution with standard potassium dichromate (K₂Cr₂O₇) solution.
- 2. Estimation of copper contained in a supplied solution of copper salt by iodometric method.
- 3. Determination of ferrous ion in a supplied iron salt solution by standard potassium dichromate (K₂Cr₂O₇) solution.
- 4. Standardization of potassium permanganate ($KMnO_4$) solution with standard sodium oxalate ($Na_2C_2O_4$) solution.
- 5. Determination of ferrous ion in a supplied iron salt solution by standard potassium permanganate (KMnO₄).

(C) Conductometric Titration:

1. Determination of strength of a weak acid (CH₃COOH) against a strong alkali (NaOH) solution by measuring conductance.

(D) Complexometric Titration:

1. Determination of total Hardness of water using Eriochrome Black T (EBT) as indicator.

Laboratory operations & safety:

Chemicals

What is the difference between "technical grade" and "commercial grade" chemicals?

Technical grade chemicals are less pure than **reagent grade** chemicals and are usually used in applications where there are no official standards for impurity levels. **Commercial grade** chemicals are prepared in volume for general industrial use. Purity levels vary but they're often comparable in purity to technical grade chemicals.

Glassware

How to tell if glassware is clean -The glass should wet uniformly. If the meniscus pulls way from the side as the glassware drains, it isn't clean.

How to clean glassware -A bottle brush and ordinary lab detergent (trisodium phosphate or organic sulfonate based detergents work great). If you're doing phosphorus assays with the molybdenum blue method, its better not to use a phosphate based detergent.

To clean silicone-based stopcock grease off the glassware- you should soak it in hot saturated NaOH in ethanol for about 15 minutes, and then rinse thoroughly with water.

How to really clean glassware - Sometimes detergent and elbow grease isn't enough, and you have to get mean. Take 20g of ammonium peroxydisulfate (NH₄)₂S₂O₈, put it in a clean 4L glass lidded vat (like a desiccator), and add 1 L of 98% sulfuric acid. A 15-30 minute soak in this stuff will eat anything- dirt, grease, organics, clothes, and you, too. Keep it under the hood and please, wear gloves and eye protection. You want it to contact only the inside surfaces of the glassware; it will attack pigments used for fiducial marks and gradations on burretts and pipetts. Let the solution gravity drain off the glassware, and then rinse it with copious amounts of water. The final rinse should be with distilled water.

Dry glassware before putting it into this stuff. Take apart your stopcocks and degrease them as outlined above, and please make sure the alcoholic NaOH is completely washed off before they come anywhere near the peroxydisulfate solution.

It does weaken over time. You'll have to add a few grams of peroxydisulfate every other week.

This sounds like the cleaning solution from hell, and it is; but it's not as bad as the chromic acid witch's brew chemists used to use, which was just as corrosive and also carcinogenic.

How to dry glassware - Let glassware gravity-drain once it's cleaned. A final rinse with distilled water is always recommended. Don't dry it with an air hose; that can spray microscopic droplets of grease and other gunk from the airline on your clean glass.

You don't need to dry the glassware if you've just cleaned it; just rinse it with the solution you want to fill it with a couple of times. Small rinses are more effective than large ones.

How to store glassware - It's best to just let glassware dry and store it in a closed cabinet so it won't get dusty. Some people fill burretts with distilled water and put an inverted beaker on top and store them that way.

How to grease glassware - Use a silicone lubricant, like Dow Corning stopcock grease. Use as little grease as you possibly can; less is more.

Safety

Why is acid always added to water, and not the reverse?

A large amount of heat is released when strong acids are mixed with water. Addition of more acid releases more heat. If you add water to acid, you form an extremely concentrated solution of acid initially. So much heat is released that the solution may boil very violently, splashing concentrated acid out of the container. If you add acid to water, the solution that forms is very dilute and the small amount of heat released is not enough to vaporize and spatter it. So Always Add Acid to water, and never the reverse.

Laboratory Safety:

Laboratory Safety Equipment: Each student should know the location of the following safety equipment that is nearest his or her laboratory station.

- 1. First aid kit:
- 2. Safety shower:
- 3. Eye wash fountain:
- 4. Fire extinguishers and sand buckets:
- 5. Spill control materials:
- 6. Fume hoods and auxiliary fan switches:
- 7. AIUB Security Contact Number:

Laboratory Safety Guidelines: Each student shall observe the following guidelines unless otherwise instructed.

- 1. Read the experiment, including the procedure, complete the prelab assignment, and be familiar with the chapter in the text that covers what will be done in the lab before attempting the experiment.
- 2. No person is to work alone in a laboratory and all work is to be done during the scheduled laboratory periods. Unauthorized experiments, including variations of those in the lab book, are forbidden.
- 3. Any and all medical problems or situations that may inhibit a student's laboratory performance should be explained to the instructor prior to the laboratory session.
- 4. Allergies (to any chemicals or material to be used in the laboratory) that a student has should be explained to the instructor prior to the laboratory session.
- 5. Safety glasses are to be worn at **ALL** times in the laboratory session.
- 6. Eating, drinking, gum chewing, smoking or any other use of tobacco is **NOT** permitted at any time in the laboratory.
- 7. "Horseplay" is strictly forbidden in the laboratory.
- 8. Unauthorized persons (guests, etc.) are not allowed in the laboratory without the permission of the instructor.

- 9. Sandals and canvas shoes are not permitted. Wear only shoes that shed liquids. **NEVER** attend lab with bear feet.
- 10. Wear clothing that covers your torso and legs.
- 11. Laboratory coats/aprons must be worn to protect your clothing.
- 12. The wearing of rings, bracelets and/or dangling necklaces in the labs is discouraged.
- 13. When working in the lab, confine long hair.
- 14. Turn off bunsen burners when they are not in use and **NEVER** leave one unattended.
- 15. Do not attempt to pick up broken glass with your bare hands.
- 16. Work only in well ventilated areas.
- 17. Clean up chemical or biological material spills only under the direct supervision of your laboratory instructor.
- 18. Be aware of your neighbors' activities for you may be the victim of their mistakes. Advise them of improper techniques or unsafe practices. If necessary, inform the instructor.
- 19. In case of any accident or injury, no matter how minor, notify the instructor at once.

How to safely handle the equipment?

- ❖ Use only the equipment that is clean and in good condition. If your glassware or other equipment is dirty, clean it.
- ❖ Use common sense when working with heating devices and hot objects. In the laboratory burns are one of the most common injuries. To avoid burns follow these guidelines when working with heat producing reactions, hot plates, or Bunsen burners:
 - a) When heating solid or solution, or performing a heat producing reaction in a test tube, use a metal test tube holder. *NEVER use a plastic-coated clamp as a test tube holder!*
 - b) When observing a heat producing reaction in a flat bottom glassware, such as a beaker, place it on a ceramic fiber pad on the lab bench.
 - c) When heating a piece of glassware you also heat the clamps or other devices supporting the glassware. Be aware of this fact when adjusting, moving glassware, or heating apparatus.
 - d) Never leave any heat source unattended.
 - e) Ignite open flame (Bunsen burner) only when no flammable solvents are in the vicinity.

How to safely handle the chemicals?

The general rules for safely handling the chemicals are as follows:

- treat every chemical as though it were hazardous,
- never touch any chemicals with your bare hands,
- use the proper personal protective equipment,
- after handling chemicals, wash your hands,
- use correct laboratory techniques,
- always use common sense.

Assignment Writing

A Lab Assignment is the **Data and Results Entry Form** of an experiment which consists of the following points:

- Students' Name, ID and Section
- Name of the experiment
- Experimental Data (will be supplied)
- Calculations
- Results
- Questions and Answers (if any)

Presentation and Submission of the Assignment:

- 1. The Assignment should be hand-written following Data and Results Entry Form.
- 2. Common experimental data will be supplied to all students, but some variables (amount of primary standard substances) will be assigned to individual student for calculation of the results.
- 3. Students will note down the data, calculate the results during the class.
- 4. 1-2 pages are enough for each of the assignment.
- 5. Students will upload the image of an assignment in Assignment section of MS Team within deadline as per teacher's instruction.

Important points while attending each lab class:

- ✓ Read the Purpose, Introduction, Apparatus, Required Chemicals and Procedure sections for each experiment in your Chemistry Lab Manual.
- ✓ Watch the video/ppt slides on the Demonstration of Reaction.
- ✓ Write the common experimental data given and weight of primary standard substance assigned to individual student to the Data and Results Entry Form.
- ✓ Perform the calculation as instructed in the Lab manual and enter results into the Data and Results Entry Form.
- ✓ Answer the questions that appear at the end of Results (if any).
- ✓ Complete the <u>Assignment</u> by submitting your completed Data and Results Entry Form.

Book References

Text

1. M. Mahbubul Huque and A. Jabber Mian, "Practical Chemistry" 2nd ed. (1972), Student Ways, ISBN: Not found

References

- 1. J. Mendham, R. C. Denney, J. D. Barnes and M. Thomas, "Vogel's Text Book of Quantitative Chemical Analysis", 6th ed. (2000), Pearson Education Ltd, ISBN: 81-7808-538-0;
- 2. G. H. Jeffery, J. Bassett, J. Mendham, R. C. Denney, "Vogel's Text Book of Quantitative Chemical Analysis", 5th ed. (1989), Longman (ELBS), ISBN: 0-582-25167-2

Basics of Chemical Analysis:

Types of Chemical Analysis:

- ✓ Qualitative Analysis: It is concerned with the detection of the elements present in an unknown sample, mainly carried out by *dry-test* with solids and *wet-test* in aqueous solutions.
- ✓ Quantitative Analysis: It is concerned with the determination of concentration or the amount of a known substance quantitatively by *Volumetric Analysis* (Titration). *Gravimetric Analysis* is also a quantitative analysis in which an element or a definite compound of an element is isolated and weighed by different techniques such asevaporation, filtration etc.

Standard Solution:

In analytical chemistry, a standard solution is a solution containing accurately known concentration of a substance in a specific volume. It is prepared using a primary **standard** substance (a compound which is stable, highly pure and soluble in water).

Primary and Secondary Standard Substances:

Substances can be classified into two types: (a) Primary standard substances, and (b) Secondary standard substances.

Primary standard substances:

- The substances which do not react or decompose with the environment or do not absorb moisture easily,
- These are 100% pure,
- Examples are- Oxalic acid (C₂H₂O₄.2H₂O); Sodium carbonate (Na₂CO₃); Potassium dichromate (K₂Cr₂O₇), Sodium oxalate (Na₂C₂O₄) etc.

Secondary standard substances:

- The substances which do react or decompose with the environment or do absorb moisture easily.
- These are not 100% pure
- Examples are- Hydrochloric acid (HCl), Sodium hydroxide (NaOH), Potassium permanganate (KMnO₄) etc.

A solution prepared from a primary standard substance can be called a *standard solution*. A solution prepared from a secondary standard substance is not a standard solution. To make it standard solution, it needs to be standardized by another suitable standard solution.

Units of Concentration

Concentration may be expressed in several different ways, using percent composition by mass, mole fraction, normality, molarity or molality. We would like to discuss percent composition by mass, molarity and normality here:

✓ Percent Composition by Mass (%)

This is the mass of the solute divided by the mass of the solution (i.e. mass of solute plus mass of solvent), multiplied by 100. For example 12% KI solution means 12g KI in 100g solution.

Example-1: Determine the percent composition by mass of a 100 g salt solution which contains 20 g salt.

Solution: 20 g NaCl / 100 g solution \times 100 = 20% NaCl solution

✓ Molarity (M)

Molarity is the number of moles of solute per liter of solution. It is probably the most commonly used unit of concentration.

Example-2: What is the molarity of a solution made when 11 gm CaCl₂ is added to water to make 100 ml of solution?

Solution:

 $1 mol \ CaCl_2 = 111 gm \ CaCl_2$ So, $(11 \ gm \ CaCl_2 / 111 \ gm \ CaCl_2) / 1 mol \ CaCl_2 = 0.10 \ mol \ CaCl_2$ and $100 \ mL \ (100 \ mL \times 1 \ L / 1000 \ mL) = 0.10 \ L$.: Molarity of the solution = $0.10 \ mol \ / \ 0.10 \ L = 1.0 \ M$

✓ Normality (N)

Normality is equal to the gram equivalent weight of a solute per liter of solution. A gram equivalent weight is a measure of the reactive capacity of a given molecule. Normality is the only concentration unit that is reaction dependent. There are two methods to calculate the gram equivalent weight of a substance:

Method 1: (for acid or base)

Gram equivalent wt. of an acid or base is =

Molecular weight of acid or base

No. of active/replaceable H⁺ ion of acid or base

Method 2: (for oxidizing agent or reducing agent)

Gram equivalent wt. of oxidant or reductant is =

Molecular weight of oxidant or reductant

No. of electrons gain or lose by oxidant or reductant

Example-3: Concentration of 1M sulfuric acid (H₂SO₄) is 2N for acid-base reactions because each mole of sulfuric acid provides 2 moles of H⁺ ions. On the other hand, 1 M sulfuric acid provides 1 mole of sulfate ions meaning 1 N for sulfate precipitation.

$$H_2SO_4 \rightarrow 2H^+ + SO_4^{2-} \downarrow$$

So, $1N H_2SO_4 = gm \ eq.wt. / 1L H_2SO_4 \ solution = (98 \div 2) = 49gm/ 1L H_2SO_4 \ solution$ and $1M H_2SO_4 = 1mol / 1L H_2SO_4 \ sol. = 98gm / 1L H_2SO_4 \ solution$.

Formulae to calculate molarity or normality:

$$C = \frac{W \times 1000}{M \times V}$$

Where C =Strength of the solution in molarity (M) or normality (N)

W = Amount of substance e.g. acid, base, oxidizing agent or reducing agent (in gm)

M = Molecular weight (for molarity) or gram-equivalent-weight (for normality) in gm

V = Volume of the solution (in ml)

Volumetric Analysis or Titration:

In this method, the volume of a standard solution (with known concentration) is determined by reaction with a certain volume of a sample solution (under investigation) having unknown concentration.

Titration is a common laboratory method of quantitative chemical analysis which can be used to determine the concentration of a known reactant. As volume measurements play a key role in titration, it is also known as *volumetric analysis*. A reagent, called the *titrant*, (usually a standard solution) of known concentration and volume is used to react with a solution of the analyte, whose concentration is not known in advance. Using a calibrated burette to add the titrant, it is possible to determine the exact amount that has been consumed when the *endpoint* is reached. The *endpoint* is the point at which the titration is complete, as determined by an indicator (see below). This is ideally the same volume as the *equivalence point* - the volume of added titrant at which the number of moles of titrant is equal to the number of moles of analyte, or some multiple thereof (as in polyprotic acids). In the classic strong acid-strong base titration the endpoint of a titration is when the pH of the reactant is just about equal to 7, and often when the solution permanently changes color due to an indicator. There are however many different types of titrations (see below).

Types of Titrations

Titrations can be classified by the type of reaction. Different types of titration reaction include:

- 1. **Acid-base titration** is based on the neutralization reaction between the analyte and an acidic or basic titrant. This titration most commonly use a pH indicator, a pH meter, or a conductance meter to determine the endpoint (see page at 13)..
- 2. A **Redox titration** is based on an oxidation-reduction reaction between the analyte and titrant. This one use a potentiometer or a redox indicator to determine the endpoint. Frequently either the reactants or the titrant have a colour intense enough that an additional indicator is not needed (see page at 15-19).
- 3. Titrations in which conductance measurements are made use of in determining the end-point of acid-alkali reactions, some displacement reactions or precipitation reactions are called **Conductometric titrations** (see page at 20-21).
- 4. A **Complexometric titration** is based on the formation of a complex between the analyte and the titrant. The chelating agent EDTA is very commonly used to titrate

metal ions in solution. These titrations generally require specialized indicators that form weaker complexes with the analyte. A common example is Eriochrome Black T for the titration of calcium and magnesium ions to calculate hardness of water (see page at 21-23).

Acid-base titration

An **acid-base titration** is a method in chemistry that allows quantitative analysis of the concentration of an unknown acid or base solution. It makes use of the neutralization reaction that occurs between acids and bases and the end-point of the reaction. We can find out how the acids and bases will react if we know their formula.

Equipment

The key equipments used in a titration are:

- Burette
- White Tile used to see a colour change in the solution
- Pipette and glass funnel
- Acid/Base Indicator (the one used varies depending on the reactants)
- Volumetric/measuring flask and conical flask
- Standard Solution (a solution of known concentration)
- Solution of unknown concentration

Method

Before starting the titration a suitable pH indicator must be chosen. The endpoint of the reaction, when all the reactants have reacted, will have a pH change depending on the relative strengths of the acids and bases. The pH of the endpoint can be roughly determined using the following rules:

- A strong acid reacts with a strong base to form a neutral (pH=7) solution.
- A strong acid reacts with a weak base to form an acidic (pH<7) solution.
- A weak acid reacts with a strong base to form a basic (pH>7) solution.

When a weak acid reacts with a weak base, the endpoint of the solution will be basic if the base is stronger and acidic if the acid is stronger. If both are of equal strength, then the endpoint pH will be neutral.

A suitable indicator should be chosen that will show a change in color close to the end point of the reaction.

First, the burette should be rinsed with the standard solution, the pipette with the unknown solution, and the conical flask with distilled water.

Secondly, a known volume of the unknown concentration solution should be taken with the pipette and placed into the conical flask, along with a small amount (few drops) of the indicator chosen. The burette should be fitted to the top of its scale filled with the known solution.

The known solution should then be added drop by drop from the burette, into the conical flask. At this stage a rough estimation of the amount of this solution that took to neutralize the unknown solution can be done. Let the solution out of the burette until the indicator changes color and then record the value on the burette.

Perform three more titrations, this time more accurately; taking into account we can roughly estimate where the end point will occur. Take note of each of the readings on the burette at the end point, and average these at the end. Endpoint is reached when the indicator just changes color permanently. This is best achieved by adding the hanging drop of the solution from the tip of the burette into the flask right at the end of the titration.

Indicator

Indicator is a complex organic compound that indicates the end point by changing its colour, at which a chemically equivalent amount of the reagent has been added. Change of color sometime depends on pH, in acid-base reaction. The change in colour of the indicators is caused by a structural change in the molecule of the indicator when pH of the solution changes.

Acid-base indicators:

Indicator	Chemical Name	pH range	Colour in Acid	Colour in Base
Phenolphthalein	Phenol-sulphonephthalein (C ₂₀ H ₁₄ O ₄)	8.3-10	Colourless	Pink
Methyl Orange	Dimethylamine azo-benzene sodium sulphonate (C ₁₄ H ₁₄ N ₃ NaO ₃ S)	3.1-4.4	Red/Pink	Yellow

Phenolphthalein

Methyl Orange

Choice of acid-base indicators:

Strong acid-strong base	Any one (preferably methyl orange)
Strong acid-weak base	Methyl orange or red
Weak acid- strong base	Phenolphthalein
Weak acid-weak base	No suitable indicator

Redox titration

Redox titration (also called *oxidation-reduction titration*) is a type of titration based on a redox reaction between the *analyte* (substance subjected to analysis) and *titrant* (standard solution taken in burette). Redox titration may involve the use of a redox indicator and/or a potentiometer.

Example: An example of a redox titration is treating a solution of iodine with a reducing agent and using starch as indicator. Iodine forms an intensely blue complex with starch. Iodine (I_2) can be reduced to iodide (I^-) by e.g. thiosulfate ($S_2O_3^{2-}$), and when all iodine is spent the blue colour disappears. This redox titration is specifically called iodometric titration.

Concepts of Oxidation and Reduction

- (a) Classical concept
- (b) Modern concept

Classical concept of oxidation:

(1) Reactions involving addition of oxygen or of any non-metal element (except hydrogen), e.g. $12C + O_2 \rightarrow 2CO$

$$4Fe + 3O_2 \rightarrow 2Fe_2O_3$$

$$H_2 + F_2 \rightarrow 2HF$$

(2) Reactions involving removal of hydrogen or of any metal, e.g.

$$2H_2O \rightarrow 2H_2 + O_2$$

(3) Reactions involving increase of valency of a metal, e.g.

$$2\text{FeCl}_2 + \text{Cl}_2 \rightarrow 2\text{FeCl}_3$$
 Classical concept of reduction:

(1) Reactions involving removal of oxygen or of any non-metal element (other than hydrogen), e.g.

$$CuO + H_2 \rightarrow Cu + H_2O$$

(2) Reactions involving addition of hydrogen or of any metal, e.g.

$$O_2 + 2H_2 \rightarrow 2H_2O$$

(3) Reactions involving decrease of valency of a metal, e.g.

$$2FeCl_3 + H_2S \rightarrow 2FeCl_2 + HCl + S$$

Modern concept of oxidation and reduction:

According to electronic interpretation, **oxidation** is a process which involves the loss of one or more electrons, while **reduction** is a process which involves the gain of one or more electrons. **Examples of oxidation**-

Na
$$\rightarrow$$
 Na⁺ + e
H₂ \rightarrow 2H⁺ + 2e
Al \rightarrow Al⁺³ + 3e
Fe⁺² \rightarrow Fe⁺³ + e
2Cl⁻ \rightarrow Cl₂ + 2e

In the above examples, Na, H_2 , Al, Fe^{+2} , Cl⁻ are said to be **oxidized**. Such equations in which electrons are lost are known as **oxidation half-reactions**.

Examples of reduction-

$$Cl_2 + 2e \rightarrow 2Cl^-$$

 $Fe^{+3} + e \rightarrow Fe^{+2}$
 $2H^+ + 2e \rightarrow H_2$

In the above examples, Cl_2 , Fe^{+3} , H^+ are said to be **reduced**. Such equations in which electrons are gained are known as **reduction half-reactions**.

Redox Reaction: For example- formation of sodium chloride from sodium and chlorine.

$$2Na(s) + Cl_2(g) \rightarrow 2NaCl(s)$$

Two half-reactions are:

2Na
$$\rightarrow$$
 2Na⁺ + 2e (oxidation half reaction)
Cl₂ + 2e \rightarrow 2Cl⁻ (reduction half reaction)

 $2Na + Cl_2 \rightarrow 2NaCl$ **Oxidizing agent**- the reactant that gains

electron(s). The oxidizing agent (e⁻ acceptor) becomes reduced by gaining electron(s) and its oxidation number decreases. If a substance gains electrons easily, it is said to be a *strong* oxidizing agent. Example: K₂Cr₂O₇ (medium), I₂, CuSO₄, KMnO₄ (strong), etc.

Reducing agent- the reactant that gives up electron(s). The reducing agent (e⁻ donor) becomes oxidized by losing electron(s) and its oxidation number increases. If a substance gives up electrons easily, it is said to be a *strong reducing agent*. Example: Na₂S₂O₃ (moderate strong), KI, FeSO₄, Na₂C₂O₄ etc.

Titrations involving iodine

What is the difference between 'iodometry' and 'iodimetry'?

When an analyte that is a reducing agent is titrated directly with a standard iodine solution, the method is called "**iodimetry**".

When an analyte that is an oxidizing agent is added to excess iodide to produce iodine, and the iodine produced is determined by titration with sodium thiosulfate, the method is called "iodometry".

Redox Indicators

A **redox indicator** (also called an *oxidation-reduction indicator*) is an indicator that undergoes a definite colour change at a **specific electrode potential**. The requirement for fast and reversible colour change means that the oxidation-reduction equilibrium for an indicator redox system needs to be established very fast. Therefore only a few classes of organic redox systems can be used for indicator purposes.

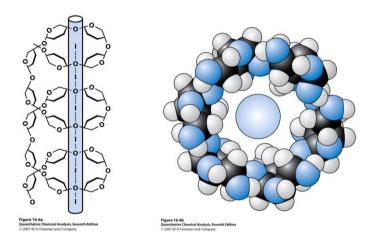
There are two common type of redox indicators:

metal-organic complexes (Ex. phenanthroline) true organic redox systems (Ex. Methylene blue)

Sometimes coloured inorganic oxidants or reductants (Ex. Potassium permanganate, KMnO₄, Potassium dichromate, K₂Cr₂O₇) are also *incorrectly* called redox indicators. They can't be classified as **true** redox indicators because of their **irreversibility**.

Almost all redox indicators with true organic redox systems involve a proton as a participant in their electrochemical reaction. Therefore sometimes redox indicators are also divided into two general groups: independent or dependent on pH.

- Starch is the indicator of choice for those procedures involving iodine because it forms an intense blue complex with iodine. Starch is not a redox indicator; it responds specifically to the presence of I_2 , not to a change in redox potential. The active fraction of starch is amylose, a polymer of the sugar α -d-glucose.
- In the presence of starch, iodine forms I_5^- chains inside the amylose helix and the color turns dark blue



• **Diphenylamine** is the organic compound with the formula $(C_6H_5)_2NH$. It is a colourless solid, but samples are often yellow due to oxidized impurities.

How does starch indicate iodine? What properties of starch allow it to be used as an indicator?

When starch is mixed with iodine in water, an intensely blue colored starch/iodine complex is formed. Many of the details of the reaction are still unknown. But it seems that the iodine (in the form of I_5^- ions) gets stuck in the coils of beta amylose molecules (beta amylose is a soluble starch). The starch forces the iodine atoms into a linear arrangement in the central groove of the amylose coil. There is some transfer of charge between the starch and the iodine. That changes the way electrons are confined, and so, changes spacing of the energy levels. The iodine/starch complex has energy level spacings that are just so for absorbing visible light- giving the complex its intense blue color.

Half Reactions involving Redox Reactions:

(Experiment 4 & 5)

- (1) $K_2Cr_2O_7 + 14HCl + 6KI = 8KCl + 2CrCl_3 + 7H_2O + 3I_2$
 - (a) $6I^{-}_{(aq.)} \rightarrow 3I_{2(aq.)} + 6e$ (oxid. half reaction)
 - (b) $Cr_2O_7^{2-}$ (aq.) + $14H^+$ (aq.) + $6e \rightarrow 2Cr^{3+}$ (aq.) + $7H_2O$ (red. half reaction)

- (2) $2Na_2S_2O_3 + I_2 = S_4O_6^{2-} + 2I^{-} + 4Na^{2+}$
 - (a) $2S_2O_3^{2-}$ (aq.) $\rightarrow S_4O_6^{2-} + 2e$ (oxid. half reaction)
 - (b) $I_{2 \text{ (aq.)}} + 2e \rightarrow 2I_{\text{(aq.)}}$ (red. half reaction)
- (3) $2\text{CuSO}_4 + 4\text{KI} = 2\text{K}_2\text{SO}_4 + 2\text{CuI} \downarrow + \text{I}_2$
 - (a) $2\Gamma_{(aq.)} \rightarrow I_{2(aq.)} + 2e$ (oxid. half reaction)
 - (b) $2Cu^{2+}_{(aq.)} + 2e \rightarrow 2Cu^{+}_{(aq.)}$ (red. half reaction)

(Experiment 6)

(4)
$$6FeSO_4 + K_2Cr_2O_7 + 7H_2SO_4 \rightarrow 3Fe_2(SO_4)_3 + K_2SO_4 + 7H_2O + Cr_2(SO_4)_3$$

- (a) $6\text{Fe}^{2+} \rightarrow 6\text{Fe}^{3+} + 6\text{e}$ (Oxid. Half Reaction)
- (b) $Cr_2O_7^{2-} + 14H^+ + 6e \rightarrow 2Cr^{3+} + 7H_2O$ (*Red. Half Reaction*)

(Experiment 7 & 10)

$$(5) 2KMnO4 + 5Na2C2O4 + 8H2SO4 = 2MnSO4 + 10CO2 + (K2SO4 + 5Na2SO4) + 8H2O$$

- (a) $5C_2O_4^{2-} \rightarrow 10CO_2 + 10e$ (Oxid. Half Reaction)
- (b) $2MnO_4^- + 16H^+ + 10e = 2Mn^{++} + 8H_2O$ (*Red. Half Reaction*)

(6)
$$2KMnO_4 + 10FeSO_4 + 8H_2SO_4 = 2MnSO_4 + 5Fe_2(SO_4)_3 + K_2SO_4 + 8H_2O_4$$

- (a) $10\text{Fe}^{2+} \rightarrow 10\text{Fe}^{3+} + 10\text{e}$ (Oxid. Half Reaction)
- (b) $2\text{MnO}_4^- + 16\text{H}^+ + 10\text{e} = 2\text{Mn}^{++} + 8\text{H}_2\text{O}$ (*Red. Half Reaction*)

Acid-base indicator vs. Red-ox indicator

Acid-base indicator	Red-ox indicator
Complex organic compounds	Complex organic compounds
Weak acid or base	Neutral compounds
Possess different colours	Usually colourless
Indicate the end-point of a titration by the	Indicate the end-point of a titration by the
change of colour depending on pH	change of colour when all liberated iodine is
	consumed by analyte

Titrations with Permanganate ion

How does permanganate cause stains and also remove them? When a redox titration with acidified potassium permanganate and iron (II) sulphate is done, why potassium permanganate stains skin on drying?

Potassium permanganate (KMnO₄) stains fabrics, fingers, and paper a dark brown color. The coffee-colored stains often can't be scrubbed off, and they may persist until the outer layer of stained skin wears off (in a day or two). It's more than just a nuisance for students and laboratory workers. Municipal water supplies are sometimes over treated with permanganate ion to kill microbes and parasites, and when that water is chlorinated, swimming pools and

porcelain can be badly stained. Permanganate itself is a deep purple colored (almost black) ion. The brown stain develops when permanganate decomposes into crystalline manganese dioxide, MnO_2 on the skin or fabric surface:

$$4 \text{ MnO}_4(qq) + 4 \text{ H}^+(qq) = 3 \text{ O}_2(g) + 2 \text{ H}_2O(\ell) + 4 \text{ MnO}_2(s)$$

The tiny crystals of MnO_2 become trapped in the pores and complex surfaces of the skin. The crystals are insoluble, and washing with water will only dislodge a few loose crystals. If the stain is on a nonliving surface, try treating it with a weak hydrochloric acid solution. Hydrochloric acid can reduce the MnO_2 to water-soluble Mn^{2+} :

$$MnO_2(s) + 4 HCl(aq) = MnCl_2(aq) + Cl_2(aq) + 2 H_2O(\ell)$$

Curiously, permanganate's oxidizing power makes it very useful for *removing* stains. Permanganate oxidation breaks the molecules of large, highly colored substances into smaller pieces. The excess permanganate is then removed chemically or by quickly rinsing.

Some information about KMnO₄:

Advantages of KMnO₄

- a) KMnO₄ is a strong oxidizing agent,
- b) it serves as its own indicator,
- c) pink colour being distinguishable even if the solution is very dilute,
- d) oldest oxidizing agent (oxidant),
- e) Mn⁺⁺ ion formed in the reaction act as a catalyst,
- f) rate of reaction increases with heating at about $60-70^{\circ}$ C.

<u>If HCl, HNO₃ is used instead of H₂SO₄</u>: KMnO₄ is used up in oxidizing Cl⁻ and NO₃⁻ to eliminate the interference of these ions.

Disadvantages of KMnO₄:

- a) It is not a primary standard substance.
- b) The strength of the solution decreases gradually when prepared from ordinary distilled water, as permanganate decomposes to MnO_2 due to presence of organic matter in H_2O . Moreover, MnO_2 acts as catalyst for auto decomposition of permanganates.
- c) Hence solution should be kept in dark bottles to avoid light.

Estimation of Cu⁺² and Fe⁺² ions:

- (1) For Cu^{+2} in 100 ml of blue vitriol solution = $0.06354 \times V \times S \times 10$ gm (V= Volume of $Na_2S_2O_3$ solution, S = Strength of $Na_2S_2O_3$ solution)
- (2) For Fe⁺² in 100 ml of Mohr's salt solution = $0.05584 \times V \times S \times 10$ gm

(V= Volume of $K_2Cr_2O_7$ solution, S = Strength of $K_2Cr_2O_7$ solution)

(3) For Fe⁺² in 100 ml of Mohr's salt solution = $0.05584 \times V \times S \times 10$ gm (V= Volume of KMnO₄ solution, S = Strength of KMnO₄ solution)

Calculation of Known Value* of Cu⁺²/ Fe⁺² ions:

 $At.wt.of Cu/Fe \times Amount of salt taken (in gm)$

Mol.wt.of blue – vitriol / Mohr's salt

[Atomic weight of Cu = 63.54, Mol. weight of $CuSO_4.5H_2O = 249.68$; Atomic weight of Fe = 55.84, Mol. weight of $FeSO_4.(NH_4)_2SO_4.6H_2O = 392.14$]

*Known value calculation is not volume dependant.

Theoretical basis of calculation of Cu⁺², Fe⁺² ions:

(1) For Cu^{+2} estimation (see Exp.5): From reactions of Exp.5, we can write-

 $2CuSO_4 \equiv I_2 \equiv 2Na_2S_2O_3$

- :. $Na_2S_2O_3 \equiv CuSO_4 \equiv 63.54 \text{ gm of } Cu^{+2}$
- \therefore 1000 ml of 1N Na₂S₂O₃ solution \equiv 63.54 gm of Cu⁺²
- \therefore 1 ml of 1N Na₂S₂O₃ solution \equiv 0.06354 gm of Cu⁺²
- (2) For Fe⁺² estimation (see Exp.6 & 7): From reaction of Exp.6, we can write-

 $1000 \text{ ml } 1M \text{ K}_2\text{Cr}_2\text{O}_7 \text{ solution} \equiv 6M \text{ FeSO}_4$

- \therefore 1000 ml 294 gm K₂Cr₂O₇ solution \equiv 6×55.84 gm Fe⁺²
- \therefore 1000 ml 49 gm K₂Cr₂O₇ solution \equiv 55.84 gm Fe⁺²
- \therefore 1000 ml 1N K₂Cr₂O₇ solution = 55.84 gm Fe⁺²
- \therefore 1 ml 1N K₂Cr₂O₇ solution \equiv 0.05584 gm Fe⁺²

Similarly, from reaction of Exp.7, we can write-

1000 ml 1M KMnO₄ solution \equiv 5M FeSO₄

- \therefore 1000 ml 158 gm KMnO₄ solution \equiv 5×55.84 gm Fe⁺²
- \therefore 1000 ml 31.6 gm KMnO₄ solution \equiv 55.84 gm Fe⁺²
- \therefore 1000 ml 1N KMnO₄ solution \equiv 55.84 gm Fe⁺²
- \therefore 1 ml 1N KMnO₄ solution \equiv 0.05584 gm Fe⁺²

Conductometric titration:

Titrations in which conductance measurements are made use of in determining the end-point of acid-alkali reactions, some displacement reactions or precipitation reactions are called *conductometric titrations*.

Conductance of electrolytes: The power of electrolytes to conduct electrical currents is termed conductivity or conductance of electrolytes. It is reciprocal of the resistance (R) and denoted by C. So, C=R⁻¹. The unit of conductance is ohm⁻¹ or mho or Siemens (S).

Conductometric titration behavior of a weak acid against a strong base:

$$CH_3COOH \rightarrow CH_3COO^- + H^+$$

 $CH_3COOH + Na^+ + OH^- \rightarrow CH_3COO^- + Na^+ + H_2O$

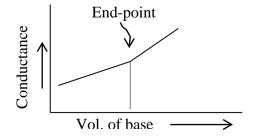


Figure: Conductometric titration curve for CH₃COOH and NaOH

- Initial conductance is low because of the poor dissociation of the weak acid.
- Sodium acetate formed; at first suppress the ionization due to common-ion-effect.
- Conductance begins to increase with the further addition of alkali.

Advantages of conductometric titrations over volumetric titrations:

- (1) coloured solutions where no indicator is found to work satisfactorily can be successfully titrated by this method.
- (2) The method is useful for the titration of weak acids against weak bases which do not give a sharp change of colour with indicators in ordinary volumetric analysis.
- (3) More accurate results are obtained because the end-point is determined graphically.

Precautions:

- (a) Temperature should be kept constant throughout the experiment.
- (b) The titrant (standard solution in burette) should be 10 times stronger so that the volume change is as little as possible.

Hardness of Water

Water can be classified as *soft water* and *hard water*.

Soft water consists of low concentration of calcium and magnesium salts. It gives foam with soap. Examples: Tap water and Drinking water. **Hard water** is due to the presence of high concentration of calcium and magnesium salts that are dissolved in water. It doesn't form foam with soap. Examples: Sea water.

There are two types hardness of water: *Temporary hardness* and *Permanent hardness*.

Temporary Hardness is due to the presence of Ca²⁺, Mg²⁺ in the form of the bicarbonate ion HCO₃⁻, being present in the water. This type of hardness can be treated by boiling the water to expel the CO₂, as indicated by the following equation:

Bicarbonate hardness is classified as temporary hardness.

Permanent hardness is due to the presence of the ions Ca^{2+} , Mg^{+2} in the form of Cl^{-} and SO_4^{2-} . This type of hardness cannot be eliminated by boiling. The water with this type of hardness is said to be *permanently hard*. As it can't be treated easily, so it's treated by chemical treatment such as: ion exchange resin.

Problems of hard water: Originally, water hardness was defined as the measure of the capacity of the water to precipitate soap. It forms scales in the boiler that may cause: (1) Decreasing in heat exchange efficiency, (2) Corrosion takes place, (3) Explosion.

One of the factors that establishes the quality of a water supply is its degree of hardness. Since most analyses do not distinguish between Ca^{2+} and Mg^{2+} , and since most hardness is

caused by carbonate mineral deposits, hardness is usually reported as parts per million (ppm) of calcium carbonate (by weight). A water supply with a hardness of 100 ppm contains the equivalent of 100 g of CaCO₃ in 1 million g of water or 0.1 g in 1 L of water (or 1000 g of water since the density of water is about 1 g/mL).

Water Hardness								
Calcium Carbonate (mg/L)	Designation							
0-60	Soft							
60-120	Moderately hard							
120-180	Hard							
More than 180	Very hard							
(mg/L=ppm)								

Water hardness is usually noticed because of difficulty in lathering soap and the formation of a scum in the bathtub. Ca²⁺ and Mg²⁺ form **insoluble salts** with soaps causing precipitation of the soap scum. Another effect of hard water is "boiler scale". When hard water comes into contact with dissolved carbonates, a precipitate of insoluble calcium carbonate forms. This "scale" can build up on the inside of water pipes to such a degree that the pipes become almost completely blocked.

Water hardness can be readily determined by titration with the chelating agent, EDTA (ethylenediaminetetraacetic acid) [Chelation: a type of bonding of ions and molecules to metal ions to form a ring]. This reagent is a weak acid that can lose four H (in bold) on complete neutralization; its structural formula is:

$$HOC$$
— CH_2
 H_2C — COH
 HOC — CH_2
 H_2C — COH
 HOC — CH_2
 H_2C — COH

The four acid oxygen sites and the two nitrogen atoms have unshared electron pairs, which can form bonds to a metal ion forming a **complex ion** or **coordination compound**. The complex is quite stable, and the conditions of its formation can ordinarily be controlled so that it is selective for a particular metal ion.

$$\begin{array}{c|c} H_2C & CH_2 \\ H_2C & CH_2 \\ \hline \\ H_2C & CH_2 \\ \hline \\ C & CH_2 \\ \hline \\ C & C & CH_2 \\ \hline \\ C & C & CH_2 \\ \hline \\ C & C & C \\ \hline$$

In a titration to determine the concentration of a metal ion, the added EDTA combines quantitatively with the cation to form the complex. The endpoint occurs when essentially all of the cation has reacted.

In this experiment a solution of EDTA will be standardize by titration against a standard solution made from calcium carbonate, CaCO₃. The EDTA solution can then be used to determine the hardness of an unknown water sample. Since both EDTA and Ca²⁺ are colorless, it is necessary to use a special indicator to detect the end point of the titration. The indicator most often used is called **Eriochrome Black T**, which forms a very stable wine-red complex, MgIn⁻, with the magnesium ion. A tiny amount of this complex will be present in the solution during the titration. As EDTA is added, it will complex free Ca²⁺ and Mg²⁺ions, leaving the MgIn⁻ complex alone until essentially all of the calcium and magnesium have been converted to chelates. Once all the divalent ions in solution have reacted with EDTA, the EDTA reacts with the Mg²⁺ ions that are complexed with the Eriochrome Black-T indicator, thus causing the solution to turn sky blue, establishing the end point of the titration.

The titration is carried out at a pH of 10, in a NH₃/NH₄₊ buffer, which keeps the EDTA (H₄Y) mainly in the form HY³⁻, where it complexes the Group 2 ions very well but does not tend to react as readily with other cations such as Fe³⁺ that might be present as impurities in the water. Taking H₄Y and H₃In as the formulas for EDTA and Eriochrome Black T, respectively, the equations for the reactions which occur during the titration are:

Titration reaction:
$$HY^{3-}(aq) + Ca^{2+}(aq) \rightarrow CaY^{2-}(aq) + H^{+}(aq)$$
 (also for Mg^{2+})

End point reaction:
$$HY^{3-}(aq) + MgIn^{-}(aq) \rightarrow MgY^{2-}(aq) + HIn^{2-}(aq)$$
wine red sky blue

Since the indicator requires a trace of Mg^{2+} to operate properly, a little magnesium ion will be added to each solution. The effect of the added Mg^{2+} can be subtracted by titrating a blank.

Eriochrome Black T

FAMILIARIZE WITH MODERN PERIODIC TABLE

In 1869 the Russian chemist Dmitri Mendeleev (1834–1907) and the German chemist J. Lothar Meyer (1830–1895), working independently, made similar discoveries. They found that when they arranged the elements in order of atomic mass, they could place them in horizontal rows, one row under the other, so that the elements in each vertical column have similar properties. A tabular arrangement of elements in rows and columns, highlighting the regular repetition of properties of the elements, is called a **periodic table.**

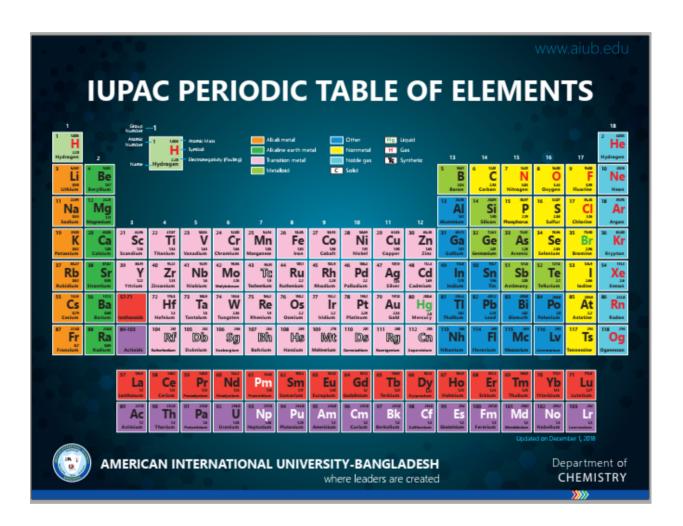
Eventually, more accurate determinations of atomic masses revealed discrepancies in this ordering of the elements. When the elements in the periodic table are ordered by atomic number, such discrepancies vanish. In modern version of the periodic table, each entry lists the atomic number, atomic symbol, and atomic mass of an element.

Patterns in the Periodic Table: Main Features

- 1. The basic structure of the periodic table is its division into rows and columns.
- 2. The columns are called groups. The groups are divided into two sub groups: A and B. These are numbered IA, IIA, IIIB, IVB, VB, VIB, VIIB, VIIIB, IB, IIIA, IIIA, IVA, VA, VIA, VIIA and VIIIA (0) groups. The number of valence electrons equals the Group number. All the members of a Group have the same valence configuration but different principal quantum numbers. Elements within a group share several common properties.
- 3. The rows are called periods (hence periodic table). There are seven periods in periodic table. The period number of an element signifies the highest unexcited energy level for an electron in that element. The period number equals the principal quantum number of the valence shell.
- 4. Group 1 is called The Alkali Metals. They are highly reactive and have a +1 charge.
- 5. Group 2 is called The Alkaline Earth Metals. They have +2 charge.
- 6. Transition metals are in the middle, they have groups from IB to VIIIB.
- 7. The inner transition metals are at the bottom- as lanthanides and actinides.
- 8. Group 7 is called the Halogens. They are also highly reactive and have a -1 charge.
- 9. Group VIIIA or zero is called the Noble Gases/ Inert Gases.
- 10. Hydrogen occupies a unique position at the top of the periodic table. It does not fit naturally into any Group. It has a single positive charge, like the alkali metals, but at room temperature, it is a gas that doesn't act like a metal.
- 11. The elements in group VIIIB consist of three groups of elements at the middle of the periodic table. They contain triads, which are metals with very similar properties, usually found together.

H hydrogen					1	UPAC	Perio	dic Tal	ble of	the Ele	ement						18 Pe He helium
078, 1.0082	2		Key:									13	14	15	16	17	4.0026
3 Li lithium 694 938, 6.997]	Be beryllium 9.0122		Symbo name convertised storic w standard atomic w	ol 								5 B boron 10.81 [10.805, 10.821]	6 C carbon 12.01 [12.009, 12.012]	7 N nitrogen 14.007 [14.006, 14.008]	0 0xygen 15.999 (15.999, 16.000)	F fluorine 18.998	10 Ne neon 20.180
Na sodium	12 Mg magnesium 24.305 [24.304, 24.307]	3	4	5	6	7	8	9	10	11	12	13 AI aluminium 26.962	14 Si silicon 21.005 [28.084, 28.086]	15 P phosphorus 30,974	16 S sulfur 3205 p2.059, 32.076)	17 CI chlorine 35.45 [35.446, 35.457]	18 Ar argon 39.25 [39.792, 39.9
19 K ctassium 39.098	20 Ca calcium 40.078(4)	21 SC scandium 44.956	22 Ti Stanium 47.867	V vanadium	Cr chromium	25 Mn manganese 54,936	26 Fe iron 65.845(2)	27 Co cobalt 58.933	28 Ni nickel ss.ess	29 Cu copper 63,546(3)	30 Zn zinc 65.38(2)	31 Ga gallium 60.723	32 Ge germanium 72.630(8)	33 As amenic 74922	34 Se selenium 78971(8)	35 Br bromine 73904 [79.901, 79.907]	36 Kr krypton 83.798(2)
37 Rb rubidium	38 Sr strontium	39 Y yttrium	40 Zr zirconium	Nb niobium	Mo malybdenum	TC technetium	Ru ruthenium	45 Rh rhedium	46 Pd palladium	Ag silver	48 Cd cadmium	49 In indium	Sn fin	51 Sb antimony	Te tellurium	53 iodine	Xe xenon
55 Cs caesium	56 Ba barium 137.33	57-71 lanthanoids	91.224(2) 72 Hf halnium 178.49(2)	92.908 73 Ta tantalum	9595 74 W tungsten 18384	75 Re menium	76 Os osmium 190.23(3)	77 r ridium 192.22	78 Pt platinum 195.08	79 Au gold 19697	80 Hg mercury 200.59	81 TI shallium 204.39 [204.38, 204.39]	82 Pb lead	83 Bi bismuth	84 Po polonium	85 At astatine	86 Rn radon
87 Fr randum	Ra Ra radium	89-103 actinoids	104 Rf rutherfordium	105 Db dubrium	106 Sg seaborgium	107 Bh bohrium	108 HS hassium	109 Mt meitnerium	DS darmstadtium	Rg roentgenium	Cn copernicium	113 Nh nihonium	114 FI flerovium	115 Mc moscovium	116 Lv Ivermorium	117 TS termessine	Og oganesso





PROBABLE QUESTIONS & PROBLEMS

- 1. Define the following terms with suitable examples: Quantitative analysis, qualitative analysis, primary standard substance, secondary standard substance, standard solution, normality, molarity, gram equivalent weight, strong acid & base, weak acid & base, titration, indicator, redox titration, oxidation, reduction, oxidizing agent, reducing agent, conductometric titration.
- 2. How do we choose an acid-base indicator? What are acid-base indicators? Mention their chemical name, pH range and colour in acid or base solution.
- 3. What are redox indicators? State the differences between acid-base and redox indicators.
- 4. Explain that oxidation-reduction happen simultaneously with one example.

- 5. Can you perform Exp.3 without using phenolphthalein? If answer is 'yes' then explain why we use two indicators in this experiment.
- 6. 'Exp.4 can be performed without indicator'-explain. Why we use NaHCO₃ in this experiment?
- 7. Mention the colour of the solid and the solution of the following: NaOH, HCl, C₂H₂O₄.2H₂O, Na₂CO₃, K₂Cr₂O₇, KI, Na₂S₂O₃.5H₂O, CuSO₄.5H₂O, H₂SO₄, FeSO₄.(NH₄)₂SO₄.6H₂O, KMnO₄ and Na₂C₂O₄.
- 8. Calculate the molecular weight and gram equivalent weight (except some having *) of the following: NaOH, HCl, C₂H₂O₄.2H₂O, Na₂CO₃, K₂Cr₂O₇, KI*, Na₂S₂O₃.5H₂O, CuSO₄.5H₂O*, H₂SO₄, FeSO₄.(NH₄)₂SO₄.6H₂O*, KMnO₄ and Na₂C₂O₄.
- 9. What are the advantages and disadvantages of using KMnO₄ solution? Why we don't use HCl or HNO₃ with KMnO₄ solution?
- 10. Explain why we use the following: (a) H₃PO₄ in Exp.6, (b) NH₄OH, CH₃COOH & NH₄SCN in Exp.5, (c) NaHCO₃ in Exp.4
- 11. Write down the main reactions involving Exp.1 to Exp.10.
- 12. Write down the redox half reactions involving Exp. 4 to Exp.7 and Exp.10.
- 13. Mention the objectives of the experiments: Exp.1 to Exp.10.
- 14. What is volume-concentration relationship? How can you use this to calculate the strength of a solution applying acid-base titration? Give one suitable example.
- 15. Calculate the amount of oxalic acid (C₂H₂O₄.2H₂O, MW 126) for the preparation of 250 ml 0.1 N oxalic acid solutions.
- 16. Calculate the normality of HCl solution if 5g of HCl (MW 36.5) is taken in 250 ml solution.
- 17. If 15ml 0.25 N HCl solutions react with 20ml NaOH solution, then calculate the normality of NaOH solution.
- 18. Suppose, 9.5 ml of oxalic acid solution and 11.5 ml of dil. HCl solution are required to titrate 10 ml NaOH solution separately. Calculate the normality of NaOH and dil. HCl solutions. [Given that oxalic acid solution is 0.13(N)].
- 19. Calculate the amount of Na₂CO₃ (MW 106) to prepare 0.3 N 200ml Na₂CO₃ solution.
- 21. How can you establish the relation to calculate the amount of Cu^{+2} ions in a supplied solution of blue vitriol: 1 ml 1N Na₂S₂O₃ = 0.06354 gm of Cu^{+2} ; Ans: see p.20 (1)
- 22. Calculate the amount of Cu^{+2} in a 150 ml blue vitriol solution if 10 ml of it is titrated with 5 ml 0.039N sodium thiosulphate solution. (1 ml 1N Na₂S₂O₃ \equiv 0.06354 gm of Cu^{+2}). Ans: 0.18585 gm
- 23. Calculate the known value of copper when 3g blue vitriol is dissolved in 100 ml of solution. (Atomic weight of Cu = 63.54, Mol. weight of $CuSO_4.5H_2O = 249.68$). Ans: 0.76345 gm
- 24. How can you establish the relation to calculate the amount of Fe⁺² ions in a supplied solution of Mohr's salt: 1 ml 1N $K_2Cr_2O_7 \equiv 0.05584$ gm of Fe⁺²; Ans: see p.20 (2a)
- 25. Calculate the amount of Fe⁺² in a 300 ml Mohr's salt solution if 10 ml of this solution is titrated with 4 ml 0.075N $K_2Cr_2O_7$ solution. (1 ml 1N $K_2Cr_2O_7 \equiv 0.05584$ gm of Fe⁺²). Ans: 0.50256 gm
- 26. How can you establish the relation to calculate the amount of Fe^{+2} ions in a supplied solution of Mohr's salt: 1 ml 1N KMnO₄ = 0.05584 gm of Fe^{+2} ; Ans: see p.20 (2b)

- 27. Calculate the amount of Fe^{+2} ions in 500 ml Mohr's salt solution if 10ml of this solution is titrated with 7.5ml 0.06N KMnO₄ solution. (1ml 1N KMnO₄= 0.05584 gm of F^{+2}). Ans: 1.256 gm
- 28. Calculate the known value of iron when 2g Mohr's salt is dissolved in 100 ml of solution. (Atomic weight of Fe = 55.84, Mol. weight of FeSO₄.(NH₄)₂SO₄.6H₂O = 392.14). Ans: 0.2847 gm
- 29. Suppose you are using EDTA with a molarity of 0.008 for the titration. You titrate 50.00 mL of a water sample using 10.68 mL of EDTA. Calculate the concentration of Ca²⁺ ion in the water sample in moles per liter and in parts per million, ppm CaCO₃? Answers: 0.0017 M Ca²⁺ and 170 ppm CaCO₃. [Hints: b=1×molarity of EDTA÷0.01M]

GLOSSARY

Indicator. A substance that undergoes a sharp, easily observable change when conditions in its solutions change. See, for example, acid-base indicator and redox indicator.

Acid-base indicator. A weak acid that has acid and base forms with sharply different colors. Change in pH around the acid's pKa are "indicated" by color changes.

Indicator diagram. PV diagram. A plot of pressure vs. volume. Lines or curves on the indicator diagram represent processes. The areas under curves on the indicator diagram are equal to the work released by the process.

Redox indicator or oxidation-reduction indicator. An organic molecule that has reduced and oxidized forms with different colors; interconversion of the reduced and oxidized forms of the indicator must be reversible. Ferroin is an example.

Universal indicator. A universal indicator is an indicator which undergoes several color changes over a wide range of pH. The color is used to "indicate" pH directly. Universal indicators are usually mixtures of several indicators.

Strong acid. Compare with weak acid. A strong acid is an acid that completely dissociates into hydrogen ions and anions in solution. Strong acids are strong electrolytes. There are only six common strong acids: HCl (hydrochloric acid), HBr (hydrobromic acid), HI (hydroiodic acid), H₂SO₄ (sulfuric acid), HClO₄ (perchloric acid), and HNO₃ (nitric acid).

Strong base. A strong base is a base that completely dissociates into ions in solution. Strong bases are strong electrolytes. The most common strong bases are alkali metal and alkaline earth metal hydroxides.

Back titration or indirect titration. Determining the concentration of an analyte by reacting it with a known number of moles of excess reagent. The excess reagent is then titrated with a second reagent. The concentration of the analyte in the original solution is then related to the amount of reagent consumed.

Complexometric titration or Chelometric titration. A titration based on a reaction between a ligand and a metal ion to form a complex. For example, free Ca²⁺ in milk powder can be determined by titrating a milk powder sample with EDTA solution, which chelates calcium

ion. Endpoints in complexometric titrations are often determined using organochromic indicators.

Ligand. A ligand is an ion or molecule that binds to a central metal atom to form a complex. Ligands are usually thought of as electron donors attracted to the metal at the center of the complex. Metals are electron acceptors. **Polydentate Ligand**. A ligand molecule with more than one donor atom is a called a polydentate ligand. These are given specific names, depending on how many donor atoms they contain. EDTA, a hexadentate **ligand**, is an example of a **polydentate ligand** that has six donor atoms with electron pairs that can be used to bond to a central metal atom or ion.

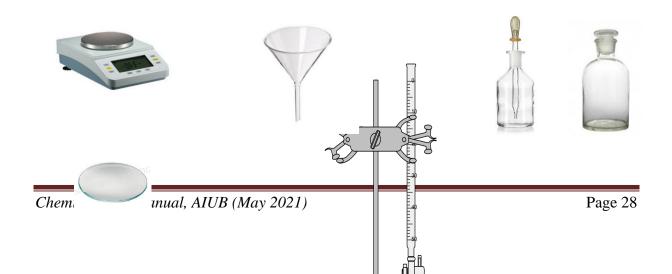
Chelating Agent. A **chelating agent** is a substance whose molecules can form several bonds to a single metal ion. In other words, a **chelating agent** is a multidentate **ligand**. An example of a simple **chelating agent** is ethylenediamine.

Redox titration or oxidation-reduction titration. A titration based on a redox reaction. For example, iron in water can be determined by converting dissolved iron to Fe²⁺ and titrating the solution with potassium permanganate (KMnO₄), a powerful oxidizing agent.

Titration curve. A plot that summarizes data collected in a titration. A linear titration curve plots moles of analyte (or, some quantity proportional to moles of analyte) on the Y axis, and the volume of titrant added on the X axis. Nonlinear plots use the log of the concentration of the analyte instead. Nonlinear titration curves are often used for neutralization titrations (pH vs. mL NaOH solution). Logs are used to exaggerate the rate of change of concentration on the plot, so that the endpoint can be determined from the point of maximal slope.

Titration. A procedure for determining the amount of some unknown substance (**analyte**) by quantitative reaction with a measured volume of a solution of precisely known concentration (**titrant** or standard solution).

COMMON LABORATORY APPARATUS





ANNEXURE (Experimental Details): Please see in a separate file

Nothing Follows