

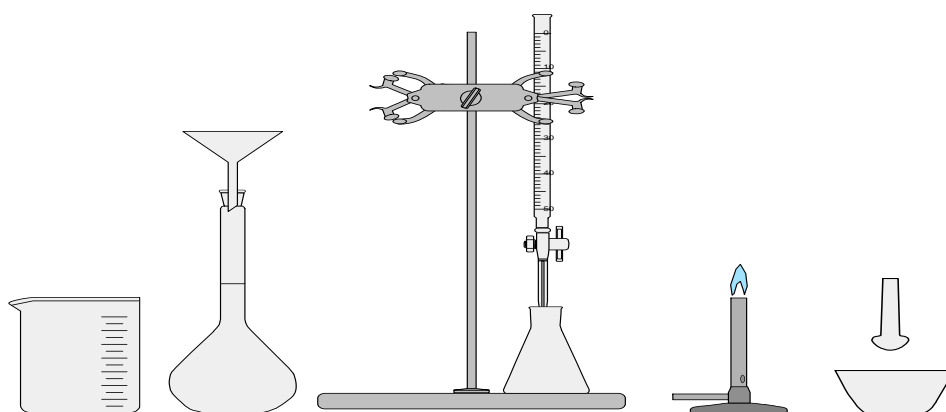
**AMERICAN INTERNATIONAL UNIVERSITY-BANGLADESH (AIUB)**

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

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



# Chemistry Lab Manual



**ACADEMIC YEAR 2021-2022**

2022

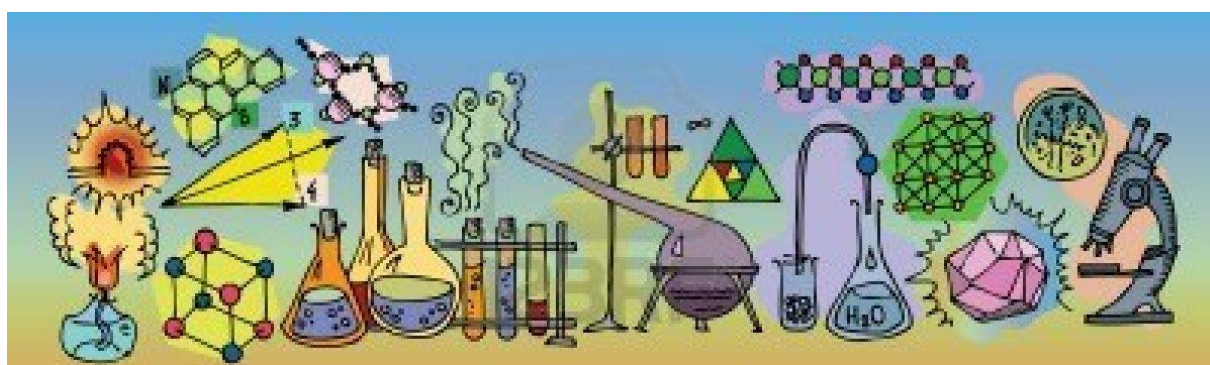
# Preface

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

<i>Serial</i>	<i>Break-up</i>	<i>Marks</i>
1	Class Performance and Attendance	10
2	Quizzes (2/best 1)	30
3	Report Writing	20
4	Viva-Voce (Sessional)	20
5	Lab Exam	20
	<b>Total Marks (Mid/Final)</b>	<b>100</b>
	<b>Contribution for Grade</b>	<b>40%</b>

## 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 ( $\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ ) 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 ( $\text{Na}_2\text{CO}_3$ ) solution.

### (B) Redox (Oxidation-Reduction) Titrations:

1. Standardization of sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ) solution with standard potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) 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 ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) solution.
4. Standardization of potassium permanganate ( $\text{KMnO}_4$ ) solution with standard sodium oxalate ( $\text{Na}_2\text{C}_2\text{O}_4$ ) solution.
5. Determination of ferrous ion in a supplied iron salt solution by standard potassium permanganate ( $\text{KMnO}_4$ ).

### (C) Conductometric Titration:

1. Determination of strength of a weak acid ( $\text{CH}_3\text{COOH}$ ) 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 away 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, it's 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 ( $(\text{NH}_4)_2\text{S}_2\text{O}_8$ ), 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 burettes and pipettes. 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 burettes 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.



## **Report Writing**

A lab report consists of four parts:

- (A) Cover Page,
- (B) Body of the Report,
- (C) Lab-sheet
- (D) Discussion

(A) **Cover Page**: It will be uploaded in student's web-account separately. Students will collect and fill up this before submission of reports. A total of 8-10 copies of cover pages are required throughout the semester.

(B) **Body of the Report**: (*Hand written*)

1. Objective of the Experiment:
2. Theory:
  - ✓ Methods involved (acid-base/redox/conductometric titration etc.)
  - ✓ Reactions
  - ✓ Indicator
3. Name of the Chemicals used with formulae:
4. Name of the Apparatus:

(C) **Lab Sheet**: (*Attach original Lab Sheet signed by your teacher*)

1. Preparation of the standard solution:
2. Experimental Data:
3. Calculations:
4. Results: Briefly mention the observed value(s)
5. Percentage of Error: (if necessary)

$$\frac{(\text{Known value} - \text{Observed value}) \times 100}{\text{Known value}}$$

Lab sheet will also be uploaded in student's web-account separately. Students will collect this before starting laboratory performance and will read it thoroughly before attending a particular experiment. A total of 8-9 lab-sheets are required throughout the semester.

(D) **Discussion**:

- a) **Precautions taken**: What sort of precaution one should take during conducting the experiment. (3 points at least)
- b) **Possible errors**: What are the possible errors that cause the deviation of the result from the expected value. (3 points at least)

## **Presentation and Submission of a Lab Report**

- ✓ Use A4-size Off-set paper for report writing,
- ✓ Write on one-side of the paper keeping appropriate margin,
- ✓ Student will fill up the cover page, write the body of the report and discussion by own hand.

- ✓ Staple all parts of the report- cover page, body of the report, original signed lab-sheet and discussion together sequentially,
- ✓ Submit the report of the experiment performed in the next lab class.

## **Book References**

### **Text**

1. M. Mahbubul Huque and A. Jabber Mian, “Practical Chemistry” 2<sup>nd</sup> 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”, 6<sup>th</sup> 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”, 5<sup>th</sup> 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 as- evaporation, 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 ( $C_2H_2O_4 \cdot 2H_2O$ ); Sodium carbonate ( $Na_2CO_3$ ); Potassium dichromate ( $K_2Cr_2O_7$ ), Sodium oxalate ( $Na_2C_2O_4$ ) 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 (KMnO<sub>4</sub>) 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:**  $20 \text{ g NaCl} / 100 \text{ g solution} \times 100 = 20\% \text{ NaCl solution}$

#### ✓ 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 CaCl<sub>2</sub> is added to water to make 100 ml of solution?

**Solution:**  $1 \text{ mol CaCl}_2 = 111 \text{ gm CaCl}_2$   
So,  $(11 \text{ gm CaCl}_2 / 111 \text{ gm CaCl}_2) / 1 \text{ mol CaCl}_2 = 0.10 \text{ mol CaCl}_2$   
and  $100 \text{ mL} (100 \text{ mL} \times 1 \text{ L} / 1000 \text{ mL}) = 0.10 \text{ L}$   
 $\therefore \text{Molarity of the solution} = 0.10 \text{ mol} / 0.10 \text{ L} = 1.0 \text{ M}$

#### ✓ 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)

$$\text{Gram equivalent wt. of an acid or base is} = \frac{\text{Molecular weight of acid or base}}{\text{No. of active/replaceable H}^+ \text{ ion of acid or base}}$$

## Method 2: (for oxidizing agent or reducing agent)

$$\text{Gram equivalent wt. of oxidant or reductant is} = \frac{\text{Molecular weight of oxidant or reductant}}{\text{No. of electrons gain or lose by oxidant or reductant}}$$

**Example-3:** Concentration of 1M sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) is 2N for acid-base reactions because each mole of sulfuric acid provides 2 moles of H<sup>+</sup> ions. On the other hand, 1 M sulfuric acid provides 1 mole of sulfate ions meaning 1 N for sulfate precipitation.



So, 1N H<sub>2</sub>SO<sub>4</sub> = gm eq.wt. / 1L H<sub>2</sub>SO<sub>4</sub> solution = (98÷2) = 49gm/ 1L H<sub>2</sub>SO<sub>4</sub> solution and 1M H<sub>2</sub>SO<sub>4</sub> = 1mol / 1L H<sub>2</sub>SO<sub>4</sub> sol. = 98gm / 1L H<sub>2</sub>SO<sub>4</sub> 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 ( $\text{pH}=7$ ) solution.
- A strong acid reacts with a weak base to form an acidic ( $\text{pH}<7$ ) solution.
- A weak acid reacts with a strong base to form a basic ( $\text{pH}>7$ ) 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.

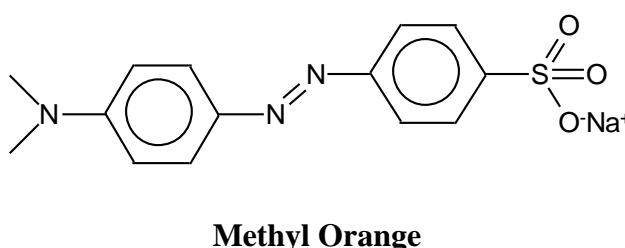
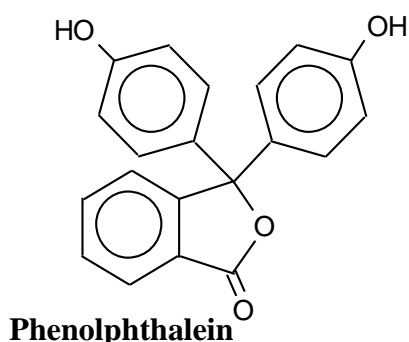
Perform three more titrations, this time more accurately; taking into account we can roughly estimate where the end point will occur. Take note of each of the readings on the burette at the end point, and average these at the end. Endpoint is reached when the indicator just changes color permanently. 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:**

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



### 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.

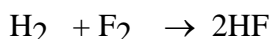
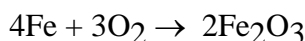
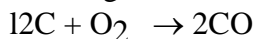
**Example:** An example of a redox titration is treating a solution of iodine with a reducing agent and using starch as indicator. Iodine forms an intensely blue complex with starch. Iodine ( $I_2$ ) can be reduced to iodide ( $I^-$ ) by e.g. thiosulfate ( $S_2O_3^{2-}$ ), and when all iodine is spent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

### Concepts of Oxidation and Reduction

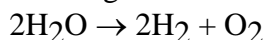
- (a) Classical concept
- (b) Modern concept

#### Classical concept of oxidation:

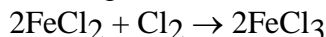
(1) Reactions involving addition of oxygen or of any non-metal element (except hydrogen), e.g.



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

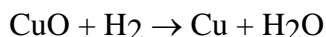


(3) Reactions involving increase of valency of a metal, e.g.

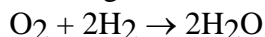


#### Classical concept of reduction:

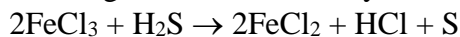
(1) Reactions involving removal of oxygen or of any non-metal element (other than hydrogen), e.g.



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



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

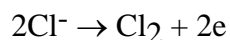
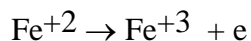
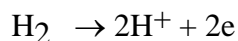
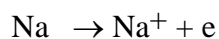


#### Modern concept of oxidation and reduction:

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.

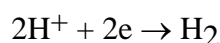
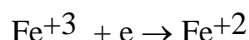
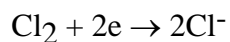


### Examples of oxidation-



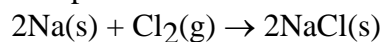
In the above examples, Na, H<sub>2</sub>, Al, Fe<sup>+2</sup>, Cl<sup>-</sup> are said to be **oxidized**. Such equations in which electrons are lost are known as **oxidation half-reactions**.

### Examples of reduction-

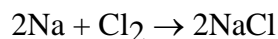
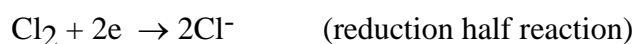
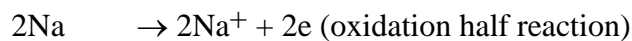


In the above examples, Cl<sub>2</sub>, Fe<sup>+3</sup>, H<sup>+</sup> are said to be **reduced**. Such equations in which electrons are gained are known as **reduction half-reactions**.

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



Two half-reactions are:



**Oxidizing agent-** the reactant that gains electron(s). The oxidizing agent (e<sup>-</sup> 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: K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> (medium), I<sub>2</sub>, CuSO<sub>4</sub>, KMnO<sub>4</sub> (strong), etc.

**Reducing agent-** the reactant that gives up electron(s). The reducing agent (e<sup>-</sup> 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: Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (moderate strong), KI, FeSO<sub>4</sub>, Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub>, etc.

### Titration 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,  $\text{KMnO}_4$ , Potassium dichromate,  $\text{K}_2\text{Cr}_2\text{O}_7$ ) 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  $\text{I}_2$ , not to a change in redox potential. The active fraction of starch is amylose, a polymer of the sugar  $\alpha$ -d-glucose.
- In the presence of starch, iodine forms  $\text{I}_5^-$  chains inside the amylose helix and the color turns dark blue

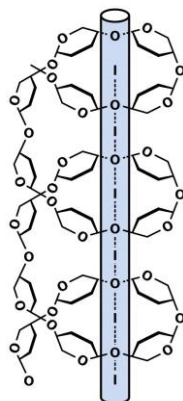


Figure 16-6a  
Quantitative Chemical Analysis, Seventh Edition  
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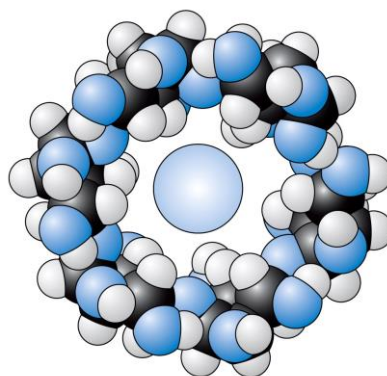


Figure 16-6b  
Quantitative Chemical Analysis, Seventh Edition  
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- **Diphenylamine** is the organic compound with the formula  $(\text{C}_6\text{H}_5)_2\text{NH}$ . It is a colourless solid, but samples are often yellow due to oxidized impurities.

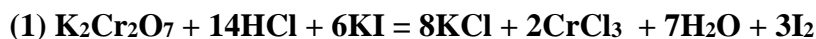
### **How does starch indicate iodine? What properties of starch allow it to be used as an indicator?**

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  $\text{I}_5^-$  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

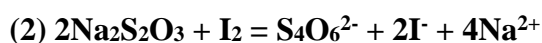
groove of the amylose coil. There is some transfer of charge between the starch and the iodine. That changes the way electrons are confined, and so, changes spacing of the energy levels. The iodine/starch complex has energy level spacings that are just so for absorbing visible light- giving the complex its intense blue color.

### Half Reactions involving Redox Reactions:

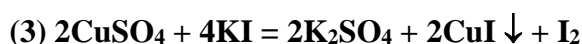
(Experiment 4 & 5)



- (a)  $6\text{I}^-_{(\text{aq.})} \rightarrow 3\text{I}_{2(\text{aq.})} + 6\text{e}^-$  (oxid. half reaction)
- (b)  $\text{Cr}_2\text{O}_7^{2-}_{(\text{aq.})} + 14\text{H}^+_{(\text{aq.})} + 6\text{e}^- \rightarrow 2\text{Cr}^{3+}_{(\text{aq.})} + 7\text{H}_2\text{O}$  (red. half reaction)



- (a)  $2\text{S}_2\text{O}_3^{2-}_{(\text{aq.})} \rightarrow \text{S}_4\text{O}_6^{2-} + 2\text{e}^-$  (oxid. half reaction)
- (b)  $\text{I}_{2(\text{aq.})} + 2\text{e}^- \rightarrow 2\text{I}^-_{(\text{aq.})}$  (red. half reaction)



- (a)  $2\text{I}^-_{(\text{aq.})} \rightarrow \text{I}_{2(\text{aq.})} + 2\text{e}^-$  (oxid. half reaction)
- (b)  $2\text{Cu}^{2+}_{(\text{aq.})} + 2\text{e}^- \rightarrow 2\text{Cu}^+_{(\text{aq.})}$  (red. half reaction)

(Experiment 6)



- (a)  $6\text{Fe}^{2+} \rightarrow 6\text{Fe}^{3+} + 6\text{e}^-$  (Oxid. Half Reaction)
- (b)  $\text{Cr}_2\text{O}_7^{2-} + 14\text{H}^+ + 6\text{e}^- \rightarrow 2\text{Cr}^{3+} + 7\text{H}_2\text{O}$  (Red. Half Reaction)

(Experiment 7 & 10)



- (a)  $5\text{C}_2\text{O}_4^{2-} \rightarrow 10\text{CO}_2 + 10\text{e}^-$  (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)



- (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)

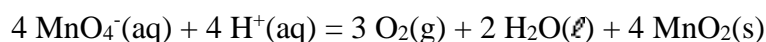
### Acid-base indicator vs. Red-ox indicator

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 change of colour depending on pH	Indicate the end-point of a titration by the change of colour when all liberated iodine is consumed by analyte

## **Titration 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 (KMnO<sub>4</sub>) 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<sub>2</sub> on the skin or fabric surface:



The tiny crystals of MnO<sub>2</sub> 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<sub>2</sub> to water-soluble Mn<sup>2+</sup>:



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 KMnO<sub>4</sub>:**

#### **Advantages of KMnO<sub>4</sub>**

- ✓ KMnO<sub>4</sub> is a strong oxidizing agent,
- ✓ it serves as its own indicator,
- ✓ pink colour being distinguishable even if the solution is very dilute,
- ✓ oldest oxidizing agent (oxidant),
- ✓ Mn<sup>++</sup> ion formed in the reaction act as a catalyst,
- ✓ rate of reaction increases with heating at about 60-70°C.

**If HCl, HNO<sub>3</sub> is used instead of H<sub>2</sub>SO<sub>4</sub>:** KMnO<sub>4</sub> is used up in oxidizing Cl<sup>-</sup> and NO<sub>3</sub><sup>-</sup> to eliminate the interference of these ions.

#### **Disadvantages of KMnO<sub>4</sub>:**

- ✓ It is not a primary standard substance.
- ✓ The strength of the solution decreases gradually when prepared from ordinary distilled water, as permanganate decomposes to MnO<sub>2</sub> due to presence of organic matter in H<sub>2</sub>O. Moreover, MnO<sub>2</sub> acts as catalyst for auto decomposition of permanganates.
- ✓ Hence solution should be kept in dark bottles to avoid light.

### **Estimation of Cu<sup>+2</sup> and Fe<sup>+2</sup> ions:**

- (1) For Cu<sup>+2</sup> in 100 ml of blue vitriol solution =  $0.06354 \times V \times S \times 10 \text{ gm}$   
(V= Volume of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution, S = Strength of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution)
- (2) For Fe<sup>+2</sup> in 100 ml of Mohr's salt solution =  $0.05584 \times V \times S \times 10 \text{ gm}$   
(V= Volume of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution, S = Strength of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> solution)
- (3) For Fe<sup>+2</sup> in 100 ml of Mohr's salt solution =  $0.05584 \times V \times S \times 10 \text{ gm}$   
(V= Volume of KMnO<sub>4</sub> solution, S = Strength of KMnO<sub>4</sub> solution)

### **Calculation of Known Value\* of Cu<sup>+2</sup>/ Fe<sup>+2</sup> ions:**

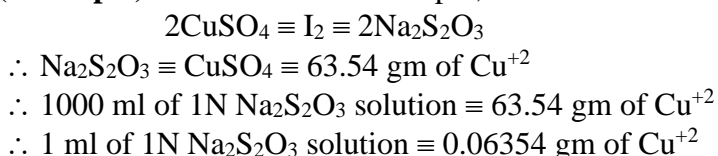
$$\frac{\text{At.wt.of Cu / Fe} \times \text{Amount of salt taken (in gm)}}{\text{Mol.wt.of blue - vitriol / Mohr's salt}}$$

[Atomic weight of Cu = 63.54, Mol. weight of CuSO<sub>4</sub>.5H<sub>2</sub>O = 249.68; Atomic weight of Fe = 55.84, Mol. weight of FeSO<sub>4</sub>.(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>.6H<sub>2</sub>O = 392.14]

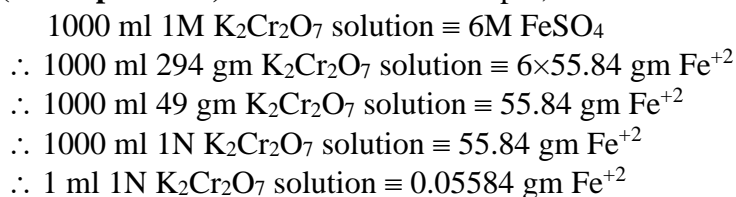
\*Known value calculation is not volume dependant.

### **Theoretical basis of calculation of Cu<sup>+2</sup>, Fe<sup>+2</sup> ions:**

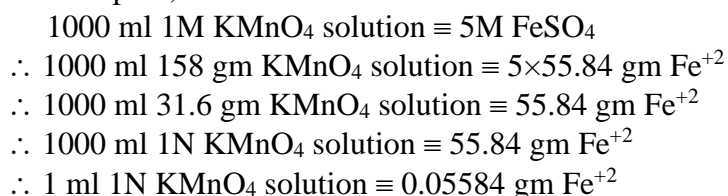
- (1) **For Cu<sup>+2</sup> estimation (see Exp.6):** From reactions of Exp.5, we can write-



- (2) **For Fe<sup>+2</sup> estimation (see Exp.7 & 10):** From reaction of Exp.7, we can write-



Similarly, from reaction of Exp.10, we can write-

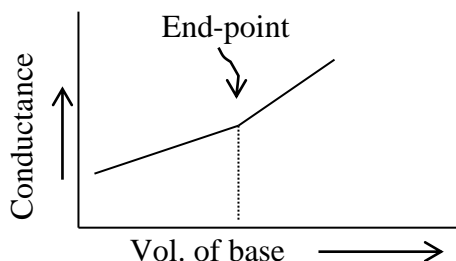
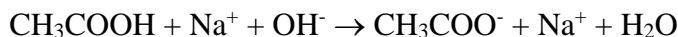
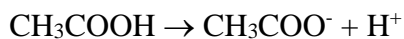


### **Conductometric titration:**

Titration 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<sup>-1</sup> or mho or Siemens (S).

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



**Figure:**  
Conductometric  
titration curve for  
 $\text{CH}_3\text{COOH}$  and  $\text{NaOH}$

- 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.

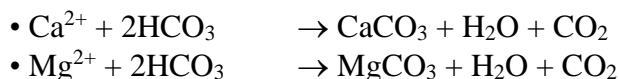
### Hardness of Water

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  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  in the form of the bicarbonate ion  $\text{HCO}_3^-$ , being present in the water. This type of hardness can be treated by boiling the water to expel the  $\text{CO}_2$ , as indicated by the following equation:



Bicarbonate hardness is classified as temporary hardness.

**Permanent hardness** is due to the presence of the ions  $\text{Ca}^{2+}$ ,  $\text{Mg}^{2+}$  in the form of  $\text{Cl}^-$  and  $\text{SO}_4^{2-}$ . 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.

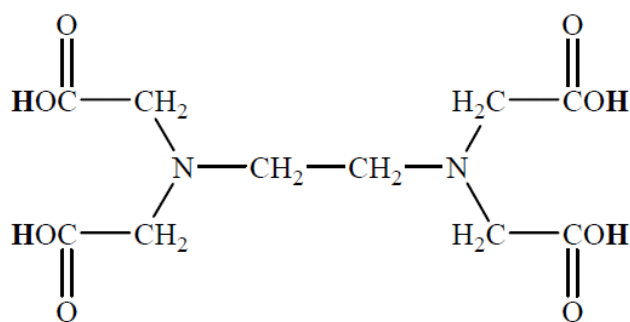
**Problems of hard water:** Originally, water hardness was defined as the measure of the capacity of the water to precipitate soap. It forms scales in the boiler that may cause: (1) Decreasing in heat exchange efficiency, (2) Corrosion takes place, (3) Explosion.

One of the factors that establishes the quality of a water supply is its degree of hardness. Since most analyses do not distinguish between  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$ , 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  $\text{CaCO}_3$  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.  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  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.

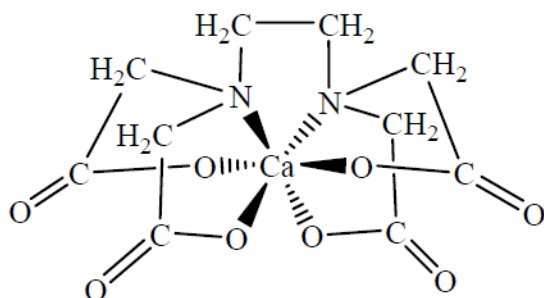
Water hardness can be readily determined by titration with the chelating agent, EDTA (ethylenediaminetetraacetic acid) [Chelation: a type of bonding of ions and molecules to metal ions to form a ring]. This reagent is a weak acid that can lose four H (in bold) on complete neutralization; its structural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



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



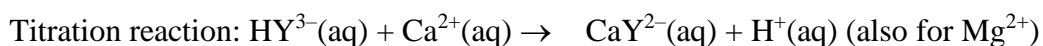
complex is quite stable, and the conditions of its formation can ordinarily be controlled so that it is selective for a particular metal ion.



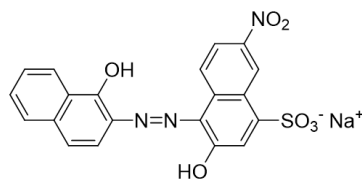
In a titration to determine the concentration of a metal ion, the added EDTA combines quantitatively with the cation to form the complex. The endpoint occurs when essentially all of the cation has reacted.

In this experiment a solution of EDTA will be standardized by titration against a standard solution made from calcium carbonate,  $\text{CaCO}_3$ . The EDTA solution can then be used to determine the hardness of an unknown water sample. Since both EDTA and  $\text{Ca}^{2+}$  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,  $\text{MgIn}^-$ , 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  $\text{Ca}^{2+}$  and  $\text{Mg}^{2+}$  ions, leaving the  $\text{MgIn}^-$  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  $\text{Mg}^{2+}$  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  $\text{NH}_3/\text{NH}_4^+$  buffer, which keeps the EDTA ( $\text{H}_4\text{Y}$ ) mainly in the form  $\text{HY}^{3-}$ , where it complexes the Group 2 ions very well but does not tend to react as readily with other cations such as  $\text{Fe}^{3+}$  that might be present as impurities in the water. Taking  $\text{H}_4\text{Y}$  and  $\text{H}_3\text{In}$  as the formulas for EDTA and Eriochrome Black T, respectively, the equations for the reactions which occur during the titration are:



Since the indicator requires a trace of  $\text{Mg}^{2+}$  to operate properly, a little magnesium ion will be added to each solution. The effect of the added  $\text{Mg}^{2+}$  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, IIB, 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.

1

H

hydrogen

1.0078 1.0082

2

3

Li

lithium

6.938 6.997

4

Be

beryllium

9.0122

11

Na

sodium

22.990

12

Mg

magnesium

24.304 24.307

19

K

potassium

39.098

20

Ca

calcium

40.0784

21

Sc

scandium

44.956

22

Ti

titanium

47.887

23

V

vanadium

50.942

24

Cr

chromium

51.996

25

Mn

manganese

54.938

26

Fe

iron

55.8452

27

Co

cobalt

58.933

28

Ni

nickel

58.693

29

Cu

copper

63.5463

30

Zn

zinc

65.382

37

Rb

rubidium

85.468

38

Sr

strontium

87.62

39

Y

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zirconium

91.2242

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Rh

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Cd

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In

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Sn

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I

iodine

126.905

54

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xenon

131.29

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caesium

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Ba

barium

137.33

57-71

lanthanoids

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Hf

hafnium

178.492

73

Ta

tantalum

180.95

74

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183.84

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186.21

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196.97

80

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200.59

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204.38 204.36

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210

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87

Fr

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223

88

Ra

radium

226

89-103

actinoids

104

Rf

rutherfordium

261

105

Db

dubnium

262

106

Sg

seaborgium

266

107

Bh

bohrium

264

108

Hs

hassium

277

109

Mt

meitnerium

268

110

Ds

darmstadtium

271

111

Rg

roentgenium

272

112

Cn

copernicium

285

113

Nh

nihonium

286

114

Fl

flerovium

289

115

Mc

moscovium

288

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293

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actinoids

104

Rf

rutherfordium

261

105

Db

dubnium

262

106

Sg

seaborgium

266

107

Bh

bohrium

264

108

Hs

hassium

277

109

Mt

meitnerium

26



57 La lanthanum 138.91	58 Ce cerium 140.12	59 Pr praseodymium 140.91	60 Nd neodymium 144.24	61 Pm promethium [145]	62 Sm samarium 150.36	63 Eu europium 151.96	64 Gd gadolinium 157.25	65 Tb terbium 158.93	66 Dy dysprosium 162.50	67 Ho holmium 164.93	68 Er erbium 167.26	69 Tm thulium 168.93	70 Yb ytterbium 173.05	71 Lu lutetium 174.97
89 Ac actinium [227]	90 Th thorium 232.04	91 Pa protactinium 231.04	92 U uranium 238.03	93 Np neptunium [237]	94 Pu plutonium [244]	95 Am americium [243]	96 Cm curium [247]	97 Bk berkelium [247]	98 Cf californium [251]	99 Es einsteinium [252]	100 Fm fermium [257]	101 Md mendelevium [258]	102 No nobelium [259]	103 Lr lawrencium [262]

For notes and updates to this table, see [www.iupac.org](http://www.iupac.org). This version is dated 1 December 2018.  
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www.aiub.edu

# IUPAC PERIODIC TABLE OF ELEMENTS

1 H Hydrogen 1.008	2 He Helium 4.0026																	13 Al Aluminum 26.982	14 Si Silicon 28.086	15 P Phosphorus 30.974	16 S Sulfur 32.06	17 Cl Chlorine 35.45	18 Ar Argon 39.948												
3 Li Lithium 6.941	4 Be Beryllium 9.0122																	19 K Potassium 39.098	20 Ca Calcium 40.078	21 Sc Scandium 44.956	22 Ti Titanium 47.88	23 V Vanadium 50.942	24 Cr Chromium 51.996	25 Mn Manganese 54.938	26 Fe Iron 55.845	27 Co Cobalt 58.933	28 Ni Nickel 58.693	29 Cu Copper 63.546	30 Zn Zinc 65.38	31 Ga Gallium 69.723	32 Ge Germanium 72.630	33 As Arsenic 74.922	34 Se Selenium 78.96	35 Br Bromine 79.904	36 Kr Krypton 83.798
11 Na Sodium 22.990	12 Mg Magnesium 24.305																	37 Rb Rubidium 85.468	38 Sr Strontium 87.62	39 Y Yttrium 88.906	40 Zr Zirconium 91.224	41 Nb Niobium 92.906	42 Mo Molybdenum 95.94	43 Tc Technetium 98.906	44 Ru Ruthenium 101.07	45 Rh Rhodium 102.91	46 Pd Palladium 106.42	47 Ag Silver 107.87	48 Cd Cadmium 112.41	49 In Indium 114.82	50 Sn Tin 118.71	51 Sb Antimony 121.76	52 Te Tellurium 127.60	53 I Iodine 126.90	54 Xe Xenon 131.29
55 Cs Caesium 132.91	56 Ba Barium 137.33	57-71 Lanthanoids	72 Hf Hafnium 178.49	73 Ta Tantalum 180.95	74 W Tungsten 183.84	75 Re Rhenium 186.21	76 Os Osmium 190.23	77 Ir Iridium 192.22	78 Pt Platinum 195.08	79 Au Gold 196.97	80 Hg Mercury 200.59	81 Tl Thallium 204.38	82 Pb Lead 207.2	83 Bi Bismuth 208.98	84 Po Polonium [209]	85 At Astatine [210]	86 Rn Radon [222]																		
87 Fr Francium [223]	88 Ra Radium [226]	89-103 Actinoids	104 Rf Rutherfordium [261]	105 Db Dubnium [262]	106 Sg Seaborgium [266]	107 Bh Bohrium [264]	108 Hs Hassium [277]	109 Mt Meitnerium [268]	110 Ds Darmstadtium [271]	111 Rg Roentgenium [272]	112 Cn Copernicium [285]	113 Nh Nihonium [286]	114 Fl Flerovium [289]	115 Mc Moscovium [288]	116 Lv Livermorium [293]	117 Ts Tennessine [294]	118 Og Oganesson [294]																		
57 La Lanthanum 138.91	58 Ce Cerium 140.12	59 Pr Praseodymium 140.91	60 Nd Neodymium 144.24	61 Pm Promethium [145]	62 Sm Samarium 150.36	63 Eu Europium 151.96	64 Gd Gadolinium 157.25	65 Tb Terbium 158.93	66 Dy Dysprosium 162.50	67 Ho Holmium 164.93	68 Er Erbium 167.26	69 Tm Thulium 168.93	70 Yb Ytterbium 173.05	71 Lu Lutetium 174.97																					
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Updated on December 1, 2018

**AMERICAN INTERNATIONAL UNIVERSITY-BANGLADESH**

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Department of  
**CHEMISTRY**

## Experiment Details

### EXPERIMENT #1

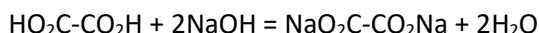
**STANDARDIZATION OF SODIUM HYDROXIDE (NaOH) SOLUTION WITH STANDARD OXALIC ACID (C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>.2H<sub>2</sub>O) SOLUTION.**

### PURPOSE

The strength of supplied NaOH solution (NaOH, being a secondary standard substance) will be measured with standard oxalic acid solution (C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>.2H<sub>2</sub>O, being a primary standard substance) by acid-base titration.

### THEORY

NaOH is a secondary standard substance, a strong base, white solid (mol. wt. 40, gram-equiv. wt. 40) and colorless in water solution. C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>.2H<sub>2</sub>O is a primary standard substance, a weak acid, white solid (mol. wt. 126, gram-equiv. wt. 63) and colorless in water solution. The balanced reaction between NaOH and C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>.2H<sub>2</sub>O is as follows:



Since this reaction is a combination of weak acid and strong base, the choice of indicator is phenolphthalein (C<sub>20</sub>H<sub>14</sub>O<sub>4</sub>). It is a white solid and is dissolved in 50% aqueous ethanol for use during titration.

### APPARATUS

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, stand and clamp etc.

### REQUIRED CHEMICALS

Sodium hydroxide (NaOH), Oxalic acid (C<sub>2</sub>H<sub>2</sub>O<sub>4</sub>.2H<sub>2</sub>O) and Phenolphthalein (C<sub>20</sub>H<sub>14</sub>O<sub>4</sub>).

### PROCEDURE

1. **Preparation of standard solution:** Transfer approx. 0.63 gram of pure oxalic acid (HOOC-COOH.2H<sub>2</sub>O) in a 100 ml measuring/volumetric flask and then dissolve it with distilled water up to the mark. Normality of the standard acid solution will be calculated as follows:

$$\text{The strength of oxalic acid solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.63} \text{ (N)}$$

2. Take 10 mL of NaOH solution in a conical flask by means of a pipette and dilute it to about 50 ml.
3. Add 1-2 drops of phenolphthalein indicator to the solution.
4. Then add standard oxalic acid solution drop by drop from a burette.
5. Shake the flask frequently while adding the acid solution.
6. Stop the addition of oxalic acid solution as soon as the pink color of the solution just disappears.
7. 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.
8. The process should be repeated at least thrice. Take the mean of the readings.
9. Calculate the strength of the supplied NaOH solution using the formulae:

$$V_{\text{NaOH}} \times N_{\text{NaOH}} = V_{\text{Oxalic acid}} \times N_{\text{Oxalic acid}}$$

where  $V_{\text{NaOH}} = 10 \text{ mL}$ ,  $V_{\text{Oxalic acid}}$  = the mean of burette reading,  $N_{\text{Oxalic acid}}$  = strength of oxalic acid and  $N_{\text{NaOH}}$  = strength of NaOH to be calculated.

-----<End of Exp.1>-----

## EXPERIMENT #2

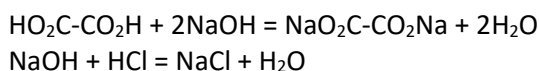
### STANDARDIZATION OF HYDROCHLORIC ACID (HCl) SOLUTION WITH STANDARD SODIUM HYDROXIDE (NaOH) SOLUTION.

#### PURPOSE

The strength of supplied HCl solution (HCl, being a secondary standard substance) will be measured with standard NaOH solution (NaOH, being a secondary standard substance and standardized by standard  $C_2H_2O_4 \cdot 2H_2O$  solution, as Exp.1) by acid-base titration.

#### THEORY

HCl is a secondary standard substance, a strong acid, colorless gas (mol. wt. 36.5, gram-equiv. wt. 36.5) and available in water solution. NaOH is a secondary standard substance, a strong base, white solid (mol. wt. 40, gram-equiv. wt. 40) and colorless in water solution.  $C_2H_2O_4 \cdot 2H_2O$  is a primary standard substance, a weak acid, white solid (mol. wt. 126, gram-equiv. wt. 63) and colorless in water solution. The balanced reactions between NaOH and  $C_2H_2O_4 \cdot 2H_2O$ ; NaOH and HCl are as follows:



Since first reaction is a combination of weak acid and strong base, the choice of indicator is phenolphthalein ( $C_{20}H_{14}O_4$ ). It is a white solid and is dissolved in 50% aqueous ethanol for use during titration. Second reaction is a combination of strong acid and strong base, so the choice of indicator is methyl orange ( $C_{14}H_{14}N_3NaO_3S$ ). It is a red solid and is dissolved in water for use during titration.

#### APPARATUS

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, stand and clamp etc.

#### REQUIRED CHEMICALS

Sodium hydroxide (NaOH), Oxalic acid ( $C_2H_2O_4 \cdot 2H_2O$ ), Hydrochloric acid (HCl), Phenolphthalein ( $C_{20}H_{14}O_4$ ) and Methyl orange ( $C_{14}H_{14}N_3NaO_3S$ ).

#### PROCEDURE

1. **Preparation of standard solution:** Transfer approx. 0.63 gram of pure oxalic acid ( $HOOC-COOH \cdot 2H_2O$ ) in a 100 ml measuring/volumetric flask and then dissolve it with distilled water up to the mark. Normality of the standard acid solution will be calculated as follows:

$$\text{The strength of oxalic acid solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.63} \text{ (N)}$$

2. Calculate the strength of the supplied NaOH solution using the formulae as Exp.1:

$$V_{NaOH} \times N_{NaOH} = V_{Oxalic\ acid} \times N_{Oxalic\ acid}$$

where  $V_{NaOH}$  = 10 mL,  $V_{Oxalic\ acid}$  = the mean of burette reading,  $N_{Oxalic\ acid}$  = strength of oxalic acid and  $N_{NaOH}$  = strength of NaOH to be calculated.

3. **Preparation of approximately 0.1N hydrochloric acid solution:** Take 10 ml conc. HCl in a 1000 ml measuring flask and add distilled water up to the mark.
4. Take 10 mL of NaOH solution in a conical flask by means of a pipette and dilute it to about 50 mL.
5. Add 2-3 drops of methyl orange indicator to the solution and solution just changes to yellow.
6. Then add previously prepared (approx. 0.1N) HCl acid solution drop wise from a burette.
7. Shake the flask frequently during addition of HCl acid.

8. Stop the addition of HCl acid solution as soon as the yellow color of the solution just changes to pink.
9. Note the burette reading. Repeat the process at least twice and take the mean of the readings.
10. Calculate the strength of the dilute HCl solution and from there calculate the strength of commercial HCl using the following formulae:

(A) *The strength of supplied dil. HCl solution:*

$$V_{\text{NaOH}} \times N_{\text{NaOH}} = V_{\text{dil. HCl}} \times N_{\text{dil. HCl to be determined}}$$

(B) *The strength of conc. HCl solution:*

$$V_{\text{dil. HCl}} \times N_{\text{dil. HCl determined}} = V_{\text{conc. HCl taken}} \times N_{\text{conc. HCl to be determined}}$$

----<End of Exp.2>----

### EXPERIMENT #3

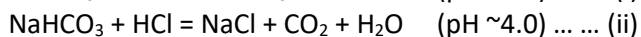
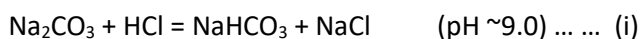
#### STANDARDIZATION OF HYDROCHLORIC ACID (HCl) SOLUTION WITH STANDARD SODIUM CARBONATE (Na<sub>2</sub>CO<sub>3</sub>) SOLUTION.

#### PURPOSE

The strength of supplied HCl solution (HCl, being a secondary standard substance) will be measured with standard Na<sub>2</sub>CO<sub>3</sub> solution (Na<sub>2</sub>CO<sub>3</sub>, being a primary standard substance) by acid-base titration.

#### THEORY

HCl is a secondary standard substance, a strong acid, colorless gas (mol. wt. 36.5, gram-equiv. wt. 36.5) and available in water solution. Na<sub>2</sub>CO<sub>3</sub> is a primary standard substance, a weak base, white solid (mol. wt. 106, gram-equiv. wt. 53) and colorless in water solution. The balanced reaction between Na<sub>2</sub>CO<sub>3</sub> and HCl is as follows:



In the first reaction one mole Na<sub>2</sub>CO<sub>3</sub> is reacted with one mole of HCl to produce NaHCO<sub>3</sub> and NaCl with a pH range ~9.0. So, the choice of indicator is phenolphthalein (C<sub>20</sub>H<sub>14</sub>O<sub>4</sub>) since it works within pH 8.3-10. But sodium bicarbonate is weaker than sodium carbonate, therefore it does not turn the phenolphthalein indicator to pink. In the second reaction one mole NaHCO<sub>3</sub> is reacted with another mole of HCl to produce NaCl, CO<sub>2</sub> and H<sub>2</sub>O with a pH range ~4.0. So, the choice of indicator is methyl orange (C<sub>14</sub>H<sub>14</sub>N<sub>3</sub>NaO<sub>3</sub>S) since it works within pH 3.1-4.4. Phenolphthalein is a white solid and is dissolved in 50% aqueous ethanol for use during titration. Methyl orange is a red solid and is dissolved in water for use during titration.

#### APPARATUS

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, stand and clamp etc.

#### REQUIRED CHEMICALS

Sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>), Hydrochloric acid (HCl), Phenolphthalein (C<sub>20</sub>H<sub>14</sub>O<sub>4</sub>) and Methyl orange (C<sub>14</sub>H<sub>14</sub>N<sub>3</sub>NaO<sub>3</sub>S).

#### PROCEDURE



1. **Preparation of standard solution:** Transfer approx. 0.53 gm of anhydrous  $\text{Na}_2\text{CO}_3$  in a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

$$\text{The strength of sodium carbonate solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.53} \text{ (N)}$$

2. **Preparation of approximately 0.1N hydrochloric acid solution:** Take 10 ml conc. HCl in a 1000 ml measuring flask and add distilled water up to the mark.
3. Take 10 mL of  $\text{Na}_2\text{CO}_3$  solution in a conical flask and dilute it to about 50 mL.
4. Add 1-2 drops of phenolphthalein and titrate against dilute HCl solution contained in a burette.
5. Now note the burette reading when just one drop of HCl discharges the pink color of the solution. This is the first end point.
6. Then add 2–3 drops of methyl orange inside the same conical flask and continue titration against the same HCl solution. The end point reached when the yellow color of the solution just changes to faint pink (or orange). Note the burette reading. This is the second end point.
7. The difference of the burette reading from initial to second end point will be the volume of the acid required for titration.
8. Repeat the whole experiment 2-3 times and take the mean reading initial to second end point.
9. *Take last reading without using phenolphthalein.*
10. Calculate the strength of the dilute HCl solution and from there calculate the strength of commercial HCl using the following formulae:

(A) *The strength of supplied dil. HCl solution:*

$$V_{\text{Na}_2\text{CO}_3} \times N_{\text{Na}_2\text{CO}_3} = V_{\text{dil. HCl}} \times N_{\text{dil. HCl to be determined}}$$

(B) *The strength of conc. HCl solution:*

$$V_{\text{dil. HCl}} \times N_{\text{dil. HCl determined}} = V_{\text{conc. HCl taken}} \times N_{\text{conc. HCl to be determined}}$$

----<End of Exp.3>----

## EXPERIMENT#4

### DETERMINATION OF TOTAL HARDNESS OF WATER USING ERIOCHROME BLACK T (EBT) AS INDICATOR.

#### PURPOSE

To determination of total Hardness of water using Eriochrome Black T (EBT) as indicator.

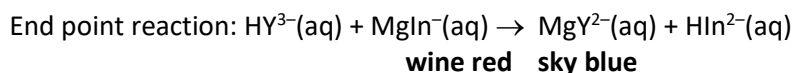
#### THEORY

(i) *Method:* Complexometric titration. 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 Lab Manual page at 21-23).

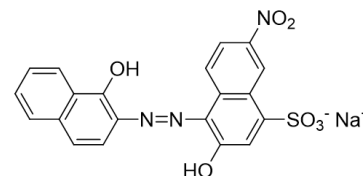
Ethylenediaminetetraacetic acid (EDTA) form a chelated soluble complex when added to a solution of certain metal cations. If a small amount of dye such as Eriochrome Black T (EBT) is added to an aqueous solution containing calcium and magnesium ions at a  $\text{pH}$  of  $10.0 \pm 0.1$ , the solution becomes wine red. If EDTA is added as a titrant, the calcium and magnesium will be complexed, and when all the magnesium and calcium has been complexed the solution turns from wine red to blue, making the end point of the titration.



(ii) *Reaction:* (Y= deprotonated part of EDTA, In=EBT Indicator)



(iii) *Indicator:* Eriochrome Black T (EBT), a complexometric indicator.



## APPARATUS

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, stand and clamp, etc.

## REQUIRED CHEMICALS

Di-sodium ethylenediaminetetraacetic acid (EDTA), Conc. hydrochloric acid (HCl), Sodium hydroxide (NaOH), Buffer solution (pH 10), Eriochrome Black T (EBT) indicator.

## PROCEDURE:

1. **Preparation of 0.01M EDTA solution (100 ml):** Weigh out accurately 0.372 g of di-sodium EDTA ( $\text{C}_{10}\text{N}_2\text{O}_8\text{H}_{14}\text{Na}_2 \cdot 2\text{H}_2\text{O}$ ) powder into a 100ml. volumetric flask and add a little water to dissolve it. Make up to mark with distilled water.

$$\text{Strength of EDTA solution} = \frac{\text{Weight taken (ingm)} \times 0.01}{0.372} \text{ (M)}$$

2. Take the standard EDTA solution into the burette.
3. Take 50 ml. of sample water (tap water) by a volumetric pipette into a conical flask.
4. Acidify the sample water with conc. hydrochloric acid (use a litmus paper) and boil for a minute to drive off carbon dioxide.
5. Cool and neutralize it with sodium hydroxide (use litmus paper).
6. Add about 2 ml of buffer solution (pH 10) and 2 drops of EBT indicator.
7. Titrate the solution with a standard 0.01M EDTA solution until the color changes from wine red to blue.
8. Repeat the titration at least two times and take the mean value.
9. The mean of three readings of EDTA is the volume of EDTA required to calculate the total hardness.

1 ml 0.01 M EDTA solution  $\equiv$  1.00 mg of  $\text{CaCO}_3$  (as shown below)

So, total hardness as mg  $\text{CaCO}_3$  per liter of water =  $(a \times b \times 1000) \div \text{ml of sample taken}$

[where, a = ml. of titrant required (mean volume of EDTA solution)

b = mg  $\text{CaCO}_3$  equivalent to 1.00 ml EDTA titrant.=1mg]

or, b=  $1 \times \text{molarity of EDTA} \div 0.01\text{M}$

-----<End of Exp.4>-----

## EXPERIMENT #5

### STANDARDIZATION OF SODIUM THIOSULPHATE ( $\text{Na}_2\text{S}_2\text{O}_3$ ) SOLUTION WITH STANDARD POTASSIUM DICHROMATE ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) SOLUTION.

#### PURPOSE

The strength of  $\text{Na}_2\text{S}_2\text{O}_3$  solution (being a solution made from secondary standard substance) will be measured against standard  $\text{K}_2\text{Cr}_2\text{O}_7$  solution by iodometric oxidation-reduction titration.

#### THEORY

$\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$  is a secondary standard substance, a reducing agent, white solid (mol. wt. 248, gram-equiv. wt. 248) and colorless in water solution.  $\text{K}_2\text{Cr}_2\text{O}_7$  is a primary standard substance, an oxidizing agent, orange solid (mol. wt. 294, gram-equiv. wt. 49) and orange color in water solution. The balanced reactions of  $\text{K}_2\text{Cr}_2\text{O}_7$  in presence of HCl and KI (white solid, a reducing agent) and of  $\text{Na}_2\text{S}_2\text{O}_3$  with  $\text{I}_2$  are as follows:

1.  $\text{K}_2\text{Cr}_2\text{O}_7 + 14\text{HCl} + 6\text{KI} = 8\text{KCl} + 2\text{CrCl}_3 + 7\text{H}_2\text{O} + 3\text{I}_2$
2.  $2\text{Na}_2\text{S}_2\text{O}_3 + \text{I}_2 = \text{Na}_2\text{S}_4\text{O}_6 + 2\text{NaI}$

#### Redox Half Reactions

For 1: (a)  $6\text{I}^-_{(\text{aq.})} \rightarrow 3\text{I}_{2(\text{aq.})} + 6\text{e}^-$  (oxid. half reaction)

(b)  $\text{Cr}_2\text{O}_7^{2-}_{(\text{aq.})} + 14\text{H}^+_{(\text{aq.})} + 6\text{e}^- \rightarrow 2\text{Cr}^{3+}_{(\text{aq.})} + 7\text{H}_2\text{O}$  (red. half reaction)

For 2: (a)  $2\text{S}_2\text{O}_3^{2-}_{(\text{aq.})} \rightarrow \text{S}_4\text{O}_6^{2-} + 2\text{e}^-$  (oxid. half reaction)

(b)  $\text{I}_{2(\text{aq.})} + 2\text{e}^- \rightarrow 2\text{I}^-_{(\text{aq.})}$  (red. half reaction)

The **iodometric titration** is a general method to determine the concentration of an oxidizing agent in solution. In an iodometric titration, a starch solution is used as an indicator since it can absorb the  $\text{I}_2$  that is released.

**Starch** solution is used in this titration involving iodine because it forms an intense blue complex with even a trace of iodine. But starch is not a redox indicator; it responds specifically to the presence of  $\text{I}_2$ , not to a change in redox potential. The active fraction of starch is amylose, a polymer of the sugar  $\alpha$ -d-glucose. In the presence of starch, iodine forms  $\text{I}_5^-$  chains inside the amylose helix and the color turns dark blue.

*$\text{NaHCO}_3$  (white solid) is used to remove excess of HCl acid in the solution and to create a layer of  $\text{CO}_2$  in the conical flask to reduce loss of volatile  $\text{I}_2$ .*

Solution turns deep brown when  $\text{I}_2$  (brown solid) is produced during first reaction above. With addition of  $\text{Na}_2\text{S}_2\text{O}_3$  solution it turns light yellow due to presence of trace of  $\text{I}_2$ . Starch forms intense blue color complexes with trace of iodine. With further addition of  $\text{Na}_2\text{S}_2\text{O}_3$  solution iodine is completely consumed, end point is achieved, and solution turns light green at the end point due to presence of chromic ions ( $\text{Cr}^{3+}$ ).

#### APPARATUS

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, stand and clamp etc.

#### REQUIRED CHEMICALS

Potassium iodide (KI), Sodium bicarbonate ( $\text{NaHCO}_3$ ), Conc. Hydrochloric acid (HCl), Potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ), Sodium thiosulphate ( $\text{Na}_2\text{S}_2\text{O}_3$ ) and Starch ( $\text{C}_6\text{H}_{10}\text{O}_5$ )<sub>n</sub>.

## PROCEDURE

1. **Preparation of approx. 0.1N potassium dichromate solution:** Transfer approx. 0.49 gram of pure  $K_2Cr_2O_7$  into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

$$\text{The strength of } K_2Cr_2O_7 \text{ solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.49} \text{ (N)}$$

2. Take 4 mL of 12% KI solution in a conical flask and dilute to about 50 mL.
3. Add about one gram of  $NaHCO_3$  and shake the flask until the salt dissolves.
4. Add 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 or dark chamber).
5. Rinse the watch glass and dilute the solution about 100 mL.
6. Titrate the liberated iodine with sodium thiosulphate solution from a burette until the brown color fades (light yellow).
7. Add about 1 mL starch solution and continue titration by adding sodium thiosulphate solution from the burette until one drop of the sodium thiosulphate solution changes the color of the solution from deep blue to light green. This is the end point.
8. Note the burette reading, repeat the whole experiment 2-3 times and take the mean.
9. Calculate the strength of sodium thiosulphate solution using the following formulae:

*The strength of supplied  $Na_2S_2O_3$  solution:*

$$V_{Na_2S_2O_3} \times N_{Na_2S_2O_3} = V_{K_2Cr_2O_7} \times N_{K_2Cr_2O_7}$$

----<End of Exp.5>----

## EXPERIMENT #6

### ESTIMATION OF COPPER (Cu) CONTAINED IN A SUPPLIED SOLUTION OF COPPER SALT BY IODOMETRIC METHOD.

## PURPOSE

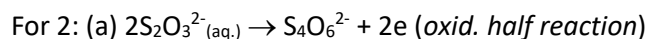
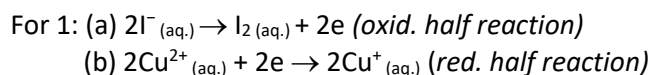
The amount of copper ions in a supplied solution of copper salt will be measured by iodometric oxidation-reduction titration.

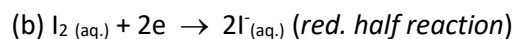
## THEORY

$K_2Cr_2O_7$  is a primary standard substance, an oxidizing agent, orange solid (mol. wt. 294, gram-equiv. wt. 49) and orange color in water solution.  $Na_2S_2O_3 \cdot 5H_2O$  is a secondary standard substance, a reducing agent, white solid (mol. wt. 248, gram-equiv. wt. 248) and colorless in water solution.  $CuSO_4 \cdot 5H_2O$  is an inorganic compound (also called **blue vitriol**, an oxidizing agent, blue colored, mol. wt. 249.68) and blue in water solution. The balanced reactions of  $CuSO_4$  in presence of  $NH_4OH$ ,  $CH_3COOH$  and KI (white solid, a reducing agent) and of  $Na_2S_2O_3$  with  $I_2$  are as follows:

1.  $2CuSO_4 + 4KI = 2K_2SO_4 + 2CuI \downarrow + I_2$
2.  $2Na_2S_2O_3 + I_2 = Na_2S_4O_6 + 2NaI$

### Redox Half Reactions





- 6M NH<sub>4</sub>OH: 4 drops for each reading; *to adjust the acidity or to keep high pH in the solution.*
- CH<sub>3</sub>COOH: 4-5 drops for each reading; *to remove or dissolve the ppt by adjusting acidity.*
- 10% NH<sub>4</sub>SCN: *to separate the adsorbed iodine from ppt (CuI solid).*

**Starch** solution is used in this titration involving iodine because it forms an intense blue complex with even a trace of iodine. But starch is not a redox indicator; it responds specifically to the presence of I<sub>2</sub>, 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<sub>5</sub><sup>-</sup> chains inside the amylose helix and the color turns dark blue.

Solution turns brown when I<sub>2</sub> (brown solid) is produced during first reaction above. With addition of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution it turns light yellow due to presence of trace of I<sub>2</sub>. Starch forms intense blue color complexes with trace of iodine. With further addition of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution iodine is completely consumed, end point is achieved, and solution turns off white at the end point due to presence of insoluble CuI (white color solid).

## APPARATUS

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, stand and clamp etc.

## REQUIRED CHEMICALS

Potassium iodide (KI), Sodium bicarbonate (NaHCO<sub>3</sub>), Conc. Hydrochloric acid (HCl), Potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>), Sodium thiosulphate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>·5H<sub>2</sub>O), Starch (C<sub>6</sub>H<sub>10</sub>O<sub>5</sub>)<sub>n</sub>, Copper sulphate (CuSO<sub>4</sub>·5H<sub>2</sub>O), Ammonium hydroxide (NH<sub>4</sub>OH, 6M), Conc. Acetic acid (CH<sub>3</sub>COOH) and Ammonium thiocyanate (NH<sub>4</sub>SCN).

## PROCEDURE

1. **Preparation of approx. 0.1N potassium dichromate solution:** Transfer approx. 0.49 gram of pure K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

$$\text{The strength of K}_2\text{Cr}_2\text{O}_7 \text{ solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.49} \text{ (N)}$$

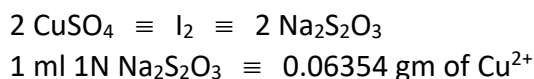
2. **Standardization of supplied Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution:** Calculate the strength of sodium thiosulphate solution using the following formulae as Exp.4:

*The strength of supplied Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution:*

$$V_{\text{Na}_2\text{S}_2\text{O}_3} \times N_{\text{Na}_2\text{S}_2\text{O}_3} = V_{\text{K}_2\text{Cr}_2\text{O}_7} \times N_{\text{K}_2\text{Cr}_2\text{O}_7}$$

3. Pipette out 10 mL of copper salt solution (containing 4 gm blue vitriol in 500 mL) into a conical flask.
4. Add 3-4 drops of 6M NH<sub>4</sub>OH until a faint permanent ppt remain and then add 6-8 drops of conc. CH<sub>3</sub>COOH.

- Now add about 10 ml of 12% potassium iodide (KI) solution and titrate the liberated iodine against the standard sodium thiosulphate solution (standardized previously) until the brown color of iodine changes to light yellow.
- Add approx. 1 mL of starch solution, solution turns intense blue 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 (off-white).
- Note the burette reading. Repeat the process at least twice and take the mean of the readings.
- Calculate the amount of copper present in 500 mL of copper salt solution using the following formula (also observed value of Cu in 500 mL solution):



- Also calculate percentage of error by calculating known value of Cu in the supplied Cu-salt solution.

(a) Known value of Cu in 500 mL Cu-salt solution = (mass of Cu × amount Cu-salt taken) ÷ mol. wt. of Cu-salt = (63.54 × amount Cu-salt taken) ÷ 249.68

(b) % of error =

$$\frac{\text{known value} - \text{observed value}}{\text{known value}} \times 100$$

----<End of Exp.6>----

## EXPERIMENT #7

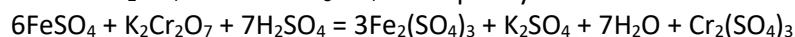
### DETERMINATION OF FERROUS ION ( $\text{Fe}^{2+}$ ) IN A SUPPLIED SOLUTION OF IRON SALT BY STANDARD POTASSIUM DICHROMATE ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) SOLUTION.

#### PURPOSE

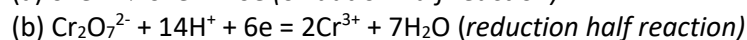
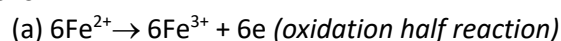
The amount of ferrous ions (iron) in a supplied solution of Mohr's salt (iron salt) will be measured by standard  $\text{K}_2\text{Cr}_2\text{O}_7$  solution.

#### THEORY

$\text{K}_2\text{Cr}_2\text{O}_7$  is a primary standard substance, an oxidizing agent, orange solid (mol. wt. 294, gram-equiv. wt. 49) and orange color in water solution. Iron salt, ferrous ammonium sulphate,  $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$  is an inorganic compound (also called **Mohr's salt**, a reducing agent, blue-green solid, mol. wt. 392.14) and blue-green in water solution. The balanced reactions of  $\text{FeSO}_4$  with  $\text{K}_2\text{Cr}_2\text{O}_7$  in presence of 5%  $\text{H}_2\text{SO}_4$  and conc.  $\text{H}_3\text{PO}_4$  and diphenyl amine:



#### Redox Half Reactions



- Conc.  $\text{H}_3\text{PO}_4$ : It is used to reduce the oxidation potential by forming complexes with  $\text{Fe}^{+3}$  ions produced in the reaction.
- Diphenylamine: It is used as a redox indicator because it shows a very clear color change from green to violet when end point of the titration is reached, 1 gm solid in 100 mL conc.  $\text{H}_2\text{SO}_4$

- 5% H<sub>2</sub>SO<sub>4</sub>: It is the main source of H<sub>3</sub>O<sup>+</sup> which supply proton to remove oxygen, such as those in the dichromate ions.

## APPARATUS

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, stand and clamp etc.

## REQUIRED CHEMICALS

Mohr's salt [FeSO<sub>4</sub>·(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>·6H<sub>2</sub>O], Sulphuric acid (H<sub>2</sub>SO<sub>4</sub>, 5%), conc. Phosphoric acid (H<sub>3</sub>PO<sub>4</sub>), Potassium dichromate (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>) and Diphenylamine [(C<sub>6</sub>H<sub>5</sub>)<sub>2</sub>NH].

## PROCEDURE

1. **Preparation of approx. 0.1N potassium dichromate solution:** Transfer approx. 0.49 gram of pure K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

$$\text{The strength of K}_2\text{Cr}_2\text{O}_7 \text{ solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.49} \text{ (N)}$$

2. Take 10 mL of the supplied iron salt (Mohr's salt) solution in a conical flask.
3. Add 50 mL 5% sulfuric acid and 5 mL of conc. phosphoric acid.
4. Then add 4-5 drops of diphenyl amine indicator and titrate slowly against the standard potassium dichromate solution drop wise 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.
5. Repeat the experiment at least thrice. Calculate the amount of iron per 500 mL of iron salt solution. Calculate the amount of iron present in 500 mL of iron salt solution using the following formula (also observed value of Fe in 500 mL solution):

$$1 \text{ mL } 1\text{N K}_2\text{Cr}_2\text{O}_7 \equiv 0.05584 \text{ gm of Fe}^{2+}$$

6. Also calculate percentage of error by calculating known value of Fe in the supplied iron-salt solution.

$$\text{(a) Known value of Fe in 500 mL iron-salt solution} = (\text{mass of Fe} \times \text{amount iron-salt taken}) \div \text{mol. wt. of iron-salt} = (55.84 \times \text{amount iron-salt taken}) \div 392.14$$

$$\text{(b) \% of error} =$$

$$\frac{\text{known value} - \text{observed value}}{\text{known value}} \times 100$$

-----<End of Exp.7>-----

## EXPERIMENT #8

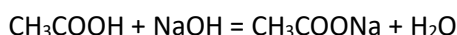
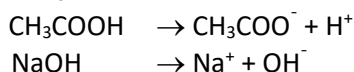
### DETERMINATION OF STRENGTH OF A WEAK ACID (CH<sub>3</sub>COOH) AGAINST A STRONG ALKALI (NaOH) SOLUTION BY MEASURING CONDUCTANCE.

#### PURPOSE

- To draw the titration curves by measuring the conductance
- To find out the end-point of an acid-base reaction
- To know the strength of supplied sample solution (acetic acid)

#### THEORY

NaOH is a secondary standard substance, a strong base, white solid (mol. wt. 40, gram-equiv. wt. 40) and colorless in water solution. CH<sub>3</sub>COOH is also a secondary standard substance, a weak acid, colorless liquid (mol. wt. 60.05, gram-equiv. wt. 60.05) and colorless in water solution. The balanced reaction between NaOH and CH<sub>3</sub>COOH is as follows:



**Conductometric Titration:** The conductance of an acid solution varies with the amount of alkali solution added to it because of the change in the number and nature of the ions in the solution. The curve showing this variation is called the conductance titration curve. At the end-point (also called, neutralizing point) of acid-alkali reaction, there is a sharp change in the conductivity. Therefore, if the conductance titration curve between volume of alkali vs conductance is drawn graphically, the end-point of the titration can easily be determined.

The conductance of a dilute acetic acid solution is due to the small amounts of H<sup>+</sup> ions and acetate ions resulting from the dissociation of the weak acid. When small amount of alkali is added and the H<sup>+</sup> ions are neutralized, an equal amount of H<sup>+</sup> ions are not generated by further dissociation of the weak acid because the acetate ions suppress the dissociation due to common-ion effect. Therefore the conductance decreases. Upon further addition of alkali the conductance increases because of the addition of Na<sup>+</sup> ions and formation of acetate ions. After the end-point, the conductance increases at a sharper rate due to addition of the fast moving OH<sup>-</sup> ions, in addition to the Na<sup>+</sup> ions.

#### APPARATUS

Conductivity meter, Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, stand and clamp etc.

#### REQUIRED CHEMICALS

Sodium hydroxide (NaOH, 0.1N) and Acetic acid (CH<sub>3</sub>COOH, approx. 0.1N).

#### PROCEDURE

1. Take 10 mL of the supplied CH<sub>3</sub>COOH solution in a beaker. Place the previously washed (with hot water) conductance cell in it and add sufficient water (~200 mL) to keep the electrodes of the cell immersed.
2. Measure the conductance of the acid solution (1<sup>st</sup> reading).
3. Fill a burette with the supplied ~ 0.1 N NaOH solution. Add 2/1 mL NaOH solution from the burette, stir the solution and measure the conductance (2<sup>nd</sup> to 12<sup>th</sup> reading) after each addition.



4. Get a graph paper ready for the plotting conductance data. Plot conductance data (in  $\mu\text{s}$ ) against the final volume (in mL) of NaOH solution on graph paper and find the end-point. The end-point gives the required volume of NaOH equivalent to 10 ml of supplied dil.  $\text{CH}_3\text{COOH}$  solution.
5. Now calculate the strength of  $\text{CH}_3\text{COOH}$  solution following the formulae:

$$V_{\text{CH}_3\text{COOH}} \times N_{\text{CH}_3\text{COOH}} = V_{\text{NaOH}} \times N_{\text{NaOH}}$$

where  $V_{\text{NaOH}}$  = from graph (ml),  $V_{\text{Oxalic acid}} = 10 \text{ ml}$ ,  $N_{\text{Oxalic acid}}$  = to be determined and  $N_{\text{NaOH}} = 0.1\text{N}$ .

----<End of Exp.8>----

## EXPERIMENT #9

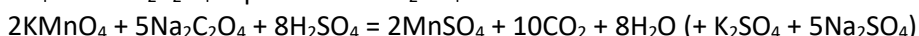
### STANDARDIZATION OF POTASSIUM PERMANGANATE ( $\text{KMnO}_4$ ) SOLUTION WITH STANDARD SODIUM OXALATE ( $\text{Na}_2\text{C}_2\text{O}_4$ ) SOLUTION.

#### PURPOSE

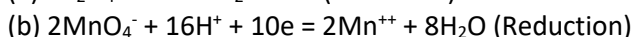
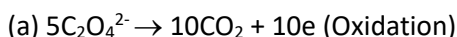
To know the strength of supplied  $\text{KMnO}_4$  solution (being made from a secondary standard substance) by standard  $\text{Na}_2\text{C}_2\text{O}_4$  solution.

#### THEORY

$\text{KMnO}_4$  is a secondary standard substance, a strong oxidizing agent, pink solid (mol. wt. 158, gram-equiv. wt. 31.6) and pink color in water solution.  $\text{Na}_2\text{C}_2\text{O}_4$  is a primary standard substance, a reducing agent, white solid (mol. wt. 134, gram-equiv. wt. 67) and colorless in water solution. The balanced reactions of  $\text{KMnO}_4$  with  $\text{Na}_2\text{C}_2\text{O}_4$  in presence of  $\text{H}_2\text{SO}_4$  are as follows:



#### Redox Half Reactions



#### Some information about $\text{KMnO}_4$ :

##### Advantages of $\text{KMnO}_4$

- ✓  $\text{KMnO}_4$  is a strong oxidizing agent,
- ✓ it serves as its own indicator,
- ✓ pink colour being distinguishable even if the solution is very dilute,
- ✓ oldest oxidizing agent (oxidant),
- ✓  $\text{Mn}^{++}$  ion formed in the reaction act as a catalyst,
- ✓ rate of reaction increases with heating at about  $60\text{--}70^\circ\text{C}$ .

**If HCl,  $\text{HNO}_3$  is used instead of  $\text{H}_2\text{SO}_4$ :**  $\text{KMnO}_4$  is used up in oxidizing  $\text{Cl}^-$  and  $\text{NO}_3^-$  to eliminate the interference of these ions.

##### Disadvantages of $\text{KMnO}_4$ :

- ✓ It is not a primary standard substance.
- ✓ The strength of the solution decreases gradually when prepared from ordinary distilled water, as permanganate decomposes to  $\text{MnO}_2$  due to presence of organic matter in  $\text{H}_2\text{O}$ . Moreover,  $\text{MnO}_2$  acts as catalyst for auto decomposition of permanganates.
- ✓ Hence solution should be kept in dark bottles to avoid light.

## APPARATUS

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, stand and clamp etc.

## REQUIRED CHEMICALS

Potassium permanganate (KMnO<sub>4</sub>), Sodium oxalate (Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub>) and Sulphuric acid (H<sub>2</sub>SO<sub>4</sub>, 2N).

## PROCEDURE

1. **Preparation of approx. 0.1N sodium oxalate solution:** Transfer approx. 0.67 gram of pure Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

$$\text{The strength of Na}_2\text{C}_2\text{O}_4 \text{ solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.67} \text{ (N)}$$

2. Pipette out 10 mL standard oxalate solution in a conical flask.
3. Add 50 mL 2N sulfuric acid solution to it and then add 4-5 drops of KMnO<sub>4</sub> solution from a burette. Shake the solution until the color disappears.
4. Continue to add permanganate solution from the burette at a moderate rate and shake the solution continuously.
5. As the color disappears more slowly, slow down the addition of permanganate solution drop wise until one drop gives a light but definite pink color. This is the end point of the titration.
6. Repeat the process at least twice. Calculate the mean value of burette readings and find out the strength of supplied KMnO<sub>4</sub> solution.
7. Calculate the strength of potassium permanganate solution using the following formulae:

$$\text{The strength of supplied KMnO}_4 \text{ solution: } V_{\text{KMnO}_4} \times N_{\text{KMnO}_4} \text{ (S)} = V_{\text{Na-oxalate}} \times N_{\text{Na-oxalate}}$$

----<End of Exp.9>----

## EXPERIMENT #10

**DETERMINATION OF FERROUS ION (Fe<sup>2+</sup>) IN A SUPPLIED SOLUTION OF IRON SALT BY STANDARD POTASSIUM PERMANGANATE (KMnO<sub>4</sub>) SOLUTION.**

## PURPOSE

The amount of ferrous ions (iron) in a supplied solution of Mohr's salt (iron salt) will be measured by standard KMnO<sub>4</sub> solution.

## INTRODUCTION

KMnO<sub>4</sub> is a secondary standard substance, a strong oxidizing agent, pink solid (mol. wt. 158, gram-equiv. wt. 31.6) and pink color in water solution. Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> is a primary standard substance, a reducing agent, white solid (mol. wt. 134, gram-equiv. wt. 67) and colorless in water solution. Iron salt, ferrous ammonium sulphate, FeSO<sub>4</sub>.(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>.6H<sub>2</sub>O is an inorganic compound (also called **Mohr's salt**, a reducing agent, blue-green solid, mol. wt. 392.14) and blue-green in water solution. The balanced reactions of KMnO<sub>4</sub> with Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub> and FeSO<sub>4</sub> separately in presence of H<sub>2</sub>SO<sub>4</sub> are as follows:

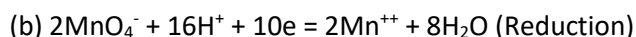
1.  $2\text{KMnO}_4 + 5\text{Na}_2\text{C}_2\text{O}_4 + 8\text{H}_2\text{SO}_4 = 2\text{MnSO}_4 + 10\text{CO}_2 + 8\text{H}_2\text{O} (+ \text{K}_2\text{SO}_4 + 5\text{Na}_2\text{SO}_4)$
2.  $2\text{KMnO}_4 + 10\text{FeSO}_4 + 8\text{H}_2\text{SO}_4 = 2\text{MnSO}_4 + 5\text{Fe}_2(\text{SO}_4)_3 + \text{K}_2\text{SO}_4 + 8\text{H}_2\text{O}$

### Redox Half Reactions

For 1: (a)  $5\text{C}_2\text{O}_4^{2-} \rightarrow 10\text{CO}_2 + 10\text{e}^-$  (Oxidation)

(b)  $2\text{MnO}_4^- + 16\text{H}^+ + 10\text{e}^- = 2\text{Mn}^{2+} + 8\text{H}_2\text{O}$  (Reduction)

For 2: (a)  $10\text{Fe}^{2+} \rightarrow 10\text{Fe}^{3+} + 10\text{e}^-$  (Oxidation)



## APPARATUS

Burette (50mL), pipette (10mL), conical flask (250mL), volumetric flask (100mL), watch glass, pipette filler, dropper, stand and clamp etc.

## REQUIRED CHEMICALS

Potassium permanganate ( $\text{KMnO}_4$ ), Sodium oxalate ( $\text{Na}_2\text{C}_2\text{O}_4$ ), Sulphuric acid ( $\text{H}_2\text{SO}_4$ , 2N) and Mohr's salt [ $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ ].

## PROCEDURE

- 1. Preparation of approx. 0.1N sodium oxalate solution:** Transfer approx. 0.67 gram of pure  $\text{Na}_2\text{C}_2\text{O}_4$  into a 100 mL measuring flask and then dissolve it with distilled water up to the mark.

$$\text{The strength of Na}_2\text{C}_2\text{O}_4 \text{ solution} = \frac{\text{Weight taken (in gm)} \times 0.1}{0.67} \text{ (N)}$$

- 2. Standardization of supplied  $\text{KMnO}_4$  solution:** Pipette out 10 mL standard oxalate solution in a conical flask. Add 50 mL 2N sulfuric acid solution to it and then add 4-5 drops of  $\text{KMnO}_4$  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 solution continuously. As the color disappears more slowly, slow down the addition of permanganate solution drop wise until one drop gives a light but definite pink color. This is the end point of the titration. Repeat the process at least twice. Calculate the mean value of burette readings and find out the strength of supplied  $\text{KMnO}_4$  solution. Calculate the strength of potassium permanganate solution using the following formulae:

$$\text{The strength of supplied KMnO}_4 \text{ solution: } V_{\text{KMnO}_4} \times N_{\text{KMnO}_4} (\text{S}) = V_{\text{Na-oxalate}} \times N_{\text{Na-oxalate}}$$

- Pipette out 10 mL of the iron (Mohr's salt) solution in a conical flask. Add about 6 mL of 2N sulfuric acid solution. Dilute 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 procedure (2).
- Repeat the titration at least twice. Calculate the amount of iron per 500 mL of iron salt solution. Calculate the amount of iron present in 500 mL of iron salt solution using the following formula (also observed value of Fe in 500 mL solution):
 
$$1 \text{ mL } 1\text{N } \text{KMnO}_4 \equiv 0.05584 \text{ gm of Fe}^{2+}$$
- Also calculate percentage of error by calculating known value of Fe in the supplied Fe-salt solution.
  - Known value of Fe in 500 mL iron-salt solution = (mass of Fe  $\times$  amount iron-salt taken)  $\div$  mol. wt. of iron-salt = (55.84  $\times$  amount iron-salt taken)  $\div$  392.14

(b) % of error =

$$\frac{\text{known value} - \text{observed value}}{\text{known value}} \times 100$$

----<End of Exp.10>----

## COMMON LABORATORY APPARATUS

Digital Balance



Funnel



Dropping Bottle



Reagent Bottle



Watch Glass



Volumetric or Measuring Flask



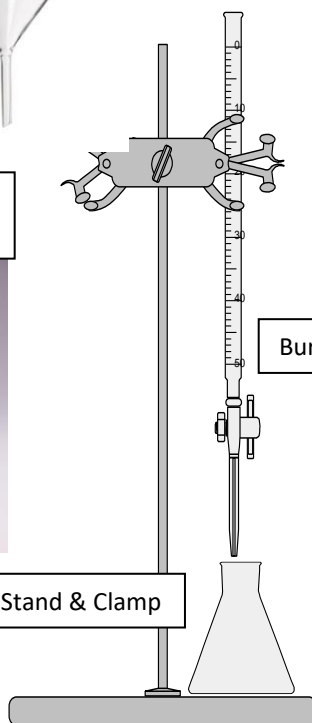
Pipette Filler



Pipette



Stand & Clamp



Measuring Cylinder



Conical Flask



Test Tube



Wash Bottle



Beaker



Glass Rod



Desiccator



Spatula



Weighing Bottle



## 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  $\text{NaHCO}_3$  in this experiment?
7. Mention the colour of the solid and the solution of the following:  $\text{NaOH}$ ,  $\text{HCl}$ ,  $\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{K}_2\text{Cr}_2\text{O}_7$ ,  $\text{KI}$ ,  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ ,  $\text{KMnO}_4$  and  $\text{Na}_2\text{C}_2\text{O}_4$ .
8. Calculate the molecular weight and gram equivalent weight (except some having \*) of the following:  $\text{NaOH}$ ,  $\text{HCl}$ ,  $\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{K}_2\text{Cr}_2\text{O}_7$ ,  $\text{KI}^*$ ,  $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ ,  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}^*$ ,  $\text{H}_2\text{SO}_4$ ,  $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}^*$ ,  $\text{KMnO}_4$  and  $\text{Na}_2\text{C}_2\text{O}_4$ .
9. What are the advantages and disadvantages of using  $\text{KMnO}_4$  solution? Why we don't use  $\text{HCl}$  or  $\text{HNO}_3$  with  $\text{KMnO}_4$  solution?
10. Explain why we use the following: (a)  $\text{H}_3\text{PO}_4$  in Exp.6, (b)  $\text{NH}_4\text{OH}$ ,  $\text{CH}_3\text{COOH}$  &  $\text{NH}_4\text{SCN}$  in Exp.5, (c)  $\text{NaHCO}_3$  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 ( $\text{C}_2\text{H}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ , MW 126) for the preparation of 250 ml 0.1 N oxalic acid solutions.
16. Calculate the normality of  $\text{HCl}$  solution if 5g of  $\text{HCl}$  (MW 36.5) is taken in 250 ml solution.
17. If 15ml 0.25 N  $\text{HCl}$  solutions react with 20ml  $\text{NaOH}$  solution, then calculate the normality of  $\text{NaOH}$  solution.
18. Suppose, 9.5 ml of oxalic acid solution and 11.5 ml of dil.  $\text{HCl}$  solution are required to titrate 10 ml  $\text{NaOH}$  solution separately. Calculate the normality of  $\text{NaOH}$  and dil.  $\text{HCl}$  solutions. [Given that oxalic acid solution is 0.13(N)].
19. Calculate the amount of  $\text{Na}_2\text{CO}_3$  (MW 106) to prepare 0.3 N 200ml  $\text{Na}_2\text{CO}_3$  solution.
20. Complete the following reactions: (a)  $\text{NaOH} + \text{H}_2\text{C}_2\text{O}_4 = \dots\dots\dots$ , (b)  $\text{NaOH} + \text{HCl} = \dots\dots\dots$ , (c)  $\text{Na}_2\text{CO}_3 + \text{HCl} = \dots\dots\dots$ , (d)  $\text{K}_2\text{Cr}_2\text{O}_7 + \text{KI} + \text{HCl} = \dots\dots\dots$ , (e)  $\text{Na}_2\text{S}_2\text{O}_3 + \text{I}_2 = \dots\dots\dots$ , (f)  $\text{CuSO}_4 + \text{KI} = \dots\dots\dots$ , (g)  $\text{KMnO}_4 + \text{Na}_2\text{C}_2\text{O}_4 + \text{H}_2\text{SO}_4 = \dots\dots\dots$ , (h)  $\text{K}_2\text{Cr}_2\text{O}_7 + \text{FeSO}_4 + \text{H}_2\text{SO}_4 = \dots\dots\dots$ , (i)  $\text{KMnO}_4 + \text{FeSO}_4 + \text{H}_2\text{SO}_4 = \dots\dots\dots$
21. How can you establish the relation to calculate the amount of  $\text{Cu}^{+2}$  ions in a supplied solution of blue vitriol: 1 ml 1N  $\text{Na}_2\text{S}_2\text{O}_3 \equiv 0.06354$  gm of  $\text{Cu}^{+2}$ ; Ans: see p.20 (1)

22. Calculate the amount of  $\text{Cu}^{+2}$  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  $\text{Na}_2\text{S}_2\text{O}_3 \equiv 0.06354$  gm of  $\text{Cu}^{+2}$ ). 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  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O} = 249.68$ ). Ans: 0.76345 gm
24. How can you establish the relation to calculate the amount of  $\text{Fe}^{+2}$  ions in a supplied solution of Mohr's salt: 1 ml 1N  $\text{K}_2\text{Cr}_2\text{O}_7 \equiv 0.05584$  gm of  $\text{Fe}^{+2}$ ; Ans: see p.20 (2a)
25. Calculate the amount of  $\text{Fe}^{+2}$  in a 300 ml Mohr's salt solution if 10 ml of this solution is titrated with 4 ml 0.075N  $\text{K}_2\text{Cr}_2\text{O}_7$  solution. (1 ml 1N  $\text{K}_2\text{Cr}_2\text{O}_7 \equiv 0.05584$  gm of  $\text{Fe}^{+2}$ ). Ans: 0.50256 gm
26. How can you establish the relation to calculate the amount of  $\text{Fe}^{+2}$  ions in a supplied solution of Mohr's salt: 1 ml 1N  $\text{KMnO}_4 \equiv 0.05584$  gm of  $\text{Fe}^{+2}$ ; Ans: see p.20 (2b)
27. Calculate the amount of  $\text{Fe}^{+2}$  ions in 500 ml Mohr's salt solution if 10 ml of this solution is titrated with 7.5 ml 0.06N  $\text{KMnO}_4$  solution. (1 ml 1N  $\text{KMnO}_4 \equiv 0.05584$  gm of  $\text{Fe}^{+2}$ ). 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  $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O} = 392.14$ ). 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  $\text{Ca}^{2+}$  ion in the water sample in moles per liter and in parts per million, ppm  $\text{CaCO}_3$ ? Answers: 0.0017 M  $\text{Ca}^{2+}$  and 170 ppm  $\text{CaCO}_3$ . [Hints:  $b=1 \times \text{molarity of EDTA} \div 0.01\text{M}$ ]

## 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  $\text{pK}_a$  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<sup>±</sup> 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),  $\text{H}_2\text{SO}_4$  (sulfuric acid),  $\text{HClO}_4$  (perchloric acid), and  $\text{HNO}_3$  (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  $\text{Ca}^{2+}$  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 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  $\text{Fe}^{2+}$  and titrating the solution with potassium permanganate ( $\text{KMnO}_4$ ), 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).

\*\*\*Nothing Follows\*\*\*