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A Central University established by the Central University Act 2009 No. 25 of 2009



**Chemistry Laboratory Manual**  
**For**  
**U.G. and P.G.**



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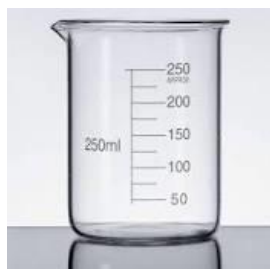


## GENERAL CHEMISTRY LABORATORY RULES

- You are responsible for all safety rules in the laboratory.
- Keep the apparatus scrupulously clean.
- Keep the reagents in their proper places after use. Do not alter their position.
- Do not contaminate the reagents.
- Keep gas taps and water taps closed when not in use.
- Use either spirit lamp or candle to light the burner. Do not use paper torches.
- Do not throw any waste paper /litmus paper etc. into the sink. Throw them into the dust bin.
- Do not pour concentrated acids into the sink. If they are to be poured, flush them with water (liberally).
- To be a better analyst, understand the theory of the experiments you conduct.
- Record your observations as and when you proceed (and not after completion) in a note book and keep it away from reagents and sink.
- You have to wear a Lab Coat and Safety Goggles and also have a Lab Manual and Calculator while performing experiments in the laboratory. Without them you are not allowed to enter the lab section.
- Read the lab experiments and any suggested additional reading(s), before coming to lab.
- Eating, Drinking, Smoking, and Cellphones are Forbidden in the laboratory at all times. Avoid unnecessary movement and talk in the laboratory.
- Any accident involving even the most minor injury must be reported to the lab assistants.
- Do not attempt any unauthorized experiment. Perform only lab operations and activities.

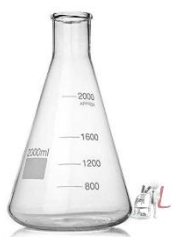
## LABORATORY EQUIPMENTS

### BEAKER



A beaker is a cylindrical glass or plastic vessel used for holding liquids. It is a multi-purpose piece of equipment used for containing a chemical reaction, measuring liquids, heating them over a Bunsen burner's flame or collecting them in a titration experiment.

### ERLENMAYER FLASK



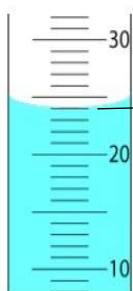
Erlenmeyer flasks are conical shaped flasks, which are made of Pyrex, are safe for chemical storage, heating solutions and recrystallizations. Because of its conical shape they are best for using with equipment's with stopcocks such as separatory funnels and burettes. They are also, like beakers, for containing and transferring liquids not for making measurements.

### GRADUATED CYLINDER



Graduated cylinders are used for measuring the volumes of liquids from a few milliliters to many liters. It is important to choose the graduated cylinder according to the amount of liquid to be measured for more accurate measurements. Always read meniscus point for graduated cylinders, pipettes, volumetric flasks and burettes.

#### What is Meniscus Point and how to read it?



24 mL not 25 mL

Meniscus point occurs when liquid molecules adhere themselves onto the walls of the glassware. This phenomenon is known as adhesion. Always read the value right at the bottom of the meniscus

## VOLUMETRIC FLASK



Volumetric flasks are used for measuring very precise and accurate amount of a liquid and is used for such when the amount is too much for pipette or burette. They are also used for solution preparation.

## PIPETTE



Pipette is a glass tube used for the delivery of a measured quantity of liquids. There are two kinds of pipettes:

**(a) Graduated (Measuring) Pipette:** has many graduated marks, much like a graduated cylinder that can deliver moderately accurate volumes (left picture)

**(b) Volumetric (Transfer) Pipette:** a kind with a bulbous middle section that has a single mark for the quantitative delivery of a single volume of liquid each time (middle and right picture)

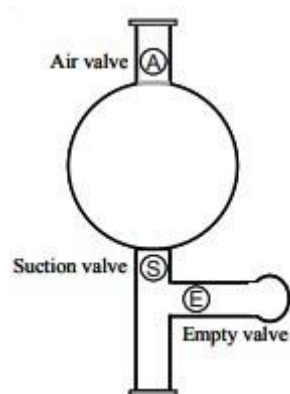
## SUCTION DEVICE



A suction device or rubber bulb is a device to place on top of pipettes for generation of vacuum to transfer known volume of liquid, usually from one container to another.

### Use of a suction device with a pipette:

Before using the suction device deflate the air inside by pressing button A. Then connect the suction device on to a pipette. Press the button S for suction. Measure desired amount of liquid with pipette by taking meniscus point into account. While emptying use button E



### STAND



A metal rod attached to a heavy metal base. The heavy base keeps the stand stable, and the vertical metal rod allows for easy height adjustment of the iron ring/clamp.



### BURETTE



Burette is a vertical cylindrical piece of laboratory glassware with a volumetric graduation on its full length and a stopcock on the bottom. It is used to dispense known amounts of a liquid reagent in a titration experiment.

## BURETTE CLAMP



Burette clamp is used to fix the burette onto the stand.

## RING SUPPORT



An iron ring/clamp that holds glassware such as a funnels and separatory funnels, and it can be attached to an iron stand.

## FUNNEL



Funnels are used for pouring liquids from one container to another. In addition, with the aid of filter paper, they can be used as separation devices to separate liquids from solids. It is fixed by a ring support on a stand.

## FILTER PAPER



Filter paper is used to separate solid particles from liquids. They can have different size with different pore size.

Solids that remain on filter paper can later be dried on a watch glass or in an oven.

## WATCH GLASS



Watch glass is used to allow crystals to dry after they have been filtered. They can be used as an evaporating surface or to cover a beaker that can be heated to very high temperatures.

## SEPARATORY FUNNEL



Separatory funnel is used in liquid-liquid extractions to separate the components of a mixture between two immiscible solvent phases of different densities. It is fixed on a stand by a ring support.

## OVEN



An oven is an enclosed chamber in which heat is produced to dry chemicals or laboratory equipment's.

## EVAPORATING DISH



The evaporating dishes are made of porcelain or ceramic material to heat and evaporate solutions to dryness.



## ROUND-BOTTOM FLASK



Round bottom flasks are used for heating or boiling of a liquid, in distillation procedures and to carry out chemical reactions. Their two or three-necked versions are also available and usually more suitable for carrying out reactions.

## TEST TUBE



Test tubes are used as containers for solids and liquids to perform quick tests for properties such as solubility, effect of heat, etc. They can also be used as centrifuge tubes when a separation of solid and liquid is necessary.

## TEST TUBE RACK



The test tube racks provide places to hold the test tubes vertical so that chemicals are not spilled out.

## TEST TUBE BRUSH



Test tube brush is a long and narrow equipment to clean the inside of glassware particularly test tubes.

## THERMOMETER



A thermometer is a device used to measure temperatures or temperature changes.

## SPATULA



Spatula or spoons are hand tools used to weigh solids in balance machine.

## BALANCE Machine



A balance machine is a scale for weighing; to obtain mass of various objects.

## STIRRING ROD



A stirring rod is made of solid glass and used to stir liquids in flasks or beakers.

## MAGNETIC BAR



Magnetic bars employ rotating magnetic field to mix a chemical mixture or a reaction continuously once they are placed in. For a magnetic bar to work a stirrer must be placed below or a heater with a stirrer feature.

## HEATER



A heater is a device that heats water or other solutions to a desired temperature. They usually have a stirrer feature to use with magnetic bars while carrying out a reaction.

## BUNSEN BURNER



Bunsen burner is used for heating when no flammable material is present. The burner can be regulated by changing the air and gas mixture.

## WASH BOTTLE



Wash bottles usually contain distilled water or acetone and provide a convenient method to wash out laboratory glassware.

## STOPPER



Stoppers are used to close flasks and test tubes to protect them from the environment.

## FUME HOODS



Fume hoods protect laboratory workers from fumes and potentially dangerous chemical reactions by continuously vacuuming air out of the lab and providing a glass shield.

## CENTRIFUGE

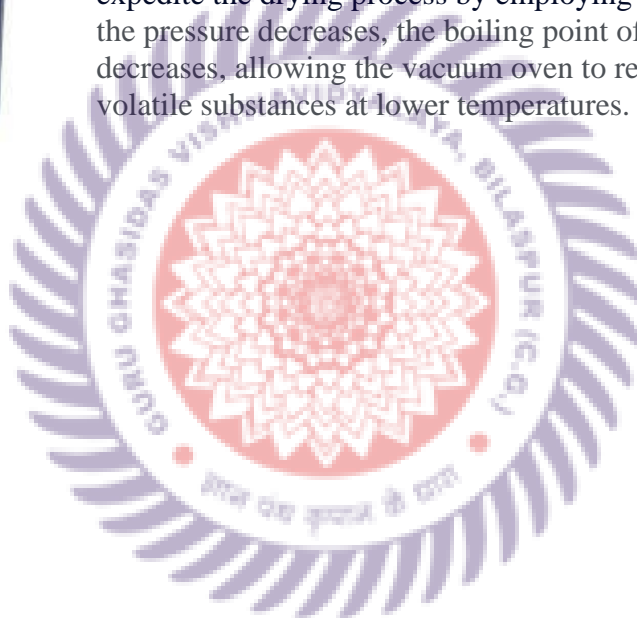


Centrifuge, any device that applies a sustained centrifugal force that is, a force due to rotation. Effectively, the centrifuge substitutes a similar, stronger, force for that of gravity. Every centrifuge contains a spinning vessel; there are many configurations, depending on use.

## VACUUM OVEN



A vacuum oven, or vacuum drying oven, is primarily used to expedite the drying process by employing vacuum and heat. As the pressure decreases, the boiling point of the solvent also decreases, allowing the vacuum oven to remove moisture and volatile substances at lower temperatures.



## 1. Aim: Iodometric titration of potassium dichromate and sodium thiosulphate.

### Requirements

**Apparatus:** Conical flask, Burette, Pipette, Volumetric flask, Stand, Beaker.

**Chemicals:**  $\text{Na}_2\text{S}_2\text{O}_3/\text{NaHCO}_3$ ,  $\text{K}_2\text{Cr}_2\text{O}_7$ , KI,  $\text{H}_2\text{SO}_4$ , Distilled water, Starch.

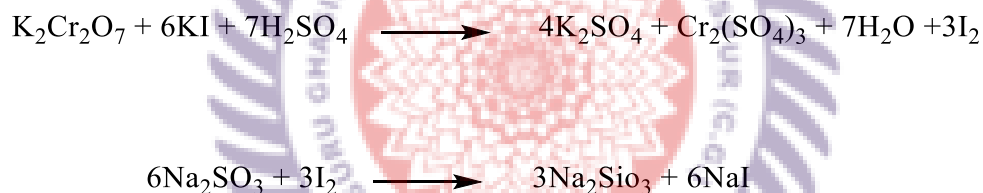
**Theory:** In this reaction, the strength of sodium thiosulphate is determined by titration with standardized potassium dichromate. This is oxidation reduction as well as iodometric titration.

Titration involving with iodine or dealing with iodine liberated in chemical reaction is called iodometric and iodometric titration respectively. This reaction is iodometric because iodine is get from KI.

Here  $\text{K}_2\text{Cr}_2\text{O}_7$  is an oxidizing agent while iodine is a reducing agent. In last step of reaction, iodine is oxidizing agent while thiosulphate ion is reducing agent. In the 2nd step of reaction, specific indicator is used that is Starch which has significant characteristic on iodine.

If to a solution containing a little iodine, some starch is added and  $\text{Na}_2\text{S}_2\text{O}_3$  is run in from burette. The blue color of starch iodine complex will disappear from the solution as soon as all the iodine has been reduced to iodine ion.

### Chemical reaction:



### Procedure:

1. Prepare 10% solution of  $\text{K}_2\text{Cr}_2\text{O}_7$  by dissolving 10g of potassium dichromate in 100ml of water.
2. Add 10ml of potassium dichromate in a flask and add half test tube of  $\text{H}_2\text{SO}_4$  in it.
3. Add 10-20ml of 5% KI solution in above flask and dilute the mixture of flask with 15-20ml of distilled water.
4. Titrate that solution against  $\text{Na}_2\text{SO}_3$ .
5. When color of solution changes to yellow, use 1ml of starch as an indicator.
6. Appearance of blue color indicate end point. Note the volume of sodium thiosulphate used till end point.
7. Repeat the process thrice for better results.

### Blank Experiment (procedure):

1. Take 10ml of distilled water in place of potassium dichromate, and add half test tube of

sulphuric acid.

2. Add 10-20ml of 5% KI solution in the above flask and dilute the solution mixture of flask with 15-20ml of distilled water.
3. Titrate the solution against  $\text{Na}_2\text{SO}_3$ .
4. When color of solution changes to transparent, use 1ml of starch as an indicator.
5. Appearance of milky color indicate end point. Note the volume of sodium thiosulphate used till end point.
6. Repeat the process thrice for better results.
7. Take difference of volume of sodium thiosulphate used in procedure of potassium dichromate and blank experiment and use that volume in calculations.

**Calculation:**

Volume of  $\text{K}_2\text{Cr}_2\text{O}_7$  used = 10ml

S. No.	Initial Volume (ml)	Final Volume (ml)	Volume used (ml)
1.			
2.			
3.			

Volume of  $\text{Na}_2\text{SO}_3$  used for burette according to experimental values =  $V_s = 22.3\text{ml}$

**Blank Experiment**

S. No.	Initial Volume (ml)	Final Volume (ml)	Volume used (ml)
1.			
2.			
3.			

Average volume of  $\text{Na}_2\text{SO}_3$  used during blank experiment =  $V_y = 13\text{ml}$

Volume of  $\text{Na}_2\text{SO}_3$  used =  $V_2 = 22.3 - 13 = 9.9\text{ml}$

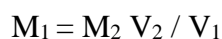
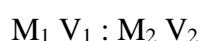
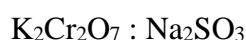
Volume of  $K_2Cr_2O_7 = V_1 = 10ml$

Molarity of  $K_2Cr_2O_7 = M_1 =$

No. of moles of  $K_2Cr_2O_7 = n_1 =$

Molarity of  $Na_2SO_3 = M_2 =$

No. of moles of  $Na_2SO_3 = n_2 =$



**Result:** The strength of  $K_2Cr_2O_7$  solution is \_\_\_\_\_ M.

**Precautions:**

1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

**2. Titrimetric Analysis (Redox Reaction):**

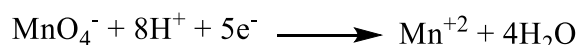
**Aim:** To determine the concentration/molarity of  $KMnO_4$  solution by titrating it against a 0.1 M standard solution of oxalic acid.

**Requirements**

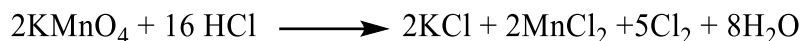
**Apparatus:** Measuring flask, Burette, Burette stand, Pipette, Conical flask, Funnel.

**Chemicals:** Oxalic acid, Potassium permanganate solution, 1M Sulphuric acid.

**Theory:** In the present experiment, potassium permanganate acts as a powerful oxidizing agent. Although  $KMnO_4$  acts as an oxidizing agent in alkaline medium also, for quantitative analysis mostly acidic medium is used. The oxidizing action of  $KMnO_4$  in the acidic medium can be represented by the following equation:



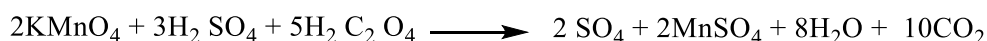
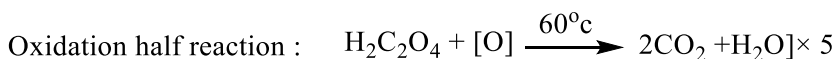
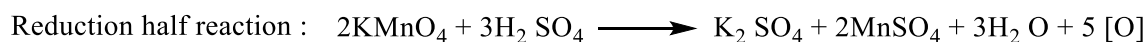
The acid used in this titration is dilute sulphuric acid. Nitric acid is not used as it is itself an oxidizing agent and hydrochloric acid is usually avoided because it reacts with  $KMnO_4$  according to the equation given below to produce chlorine and chlorine which is also an oxidizing agent in the aqueous solution.



Since, oxalic acid acts as a reducing agent, it can be titrated against potassium permanganate in the acidic medium according to the following equation:

### Reactions of oxalic acid:

#### A. Chemical equations



#### B. Ionic equation



In these equations,  $\text{MnO}_4^-$  is reduced to  $\text{Mn}^{2+}$  and  $\text{C}_2\text{O}_4^{2-}$  is oxidised to  $\text{CO}_2$ . The oxidation number of carbon in  $\text{C}_2\text{O}_4^{2-}$  changes from +3 to +4. In these titrations, potassium permanganate acts as a self-indicator. Initially colour of potassium permanganate is discharged due to its reduction by oxalic acid. After complete consumption of oxalate ions, the end point is indicated by the appearance of a light pink colour produced by the addition of a little excess of unreacted potassium permanganate. Further, during the titration of oxalic acid against potassium permanganate, warming of oxalic acid solution ( $50^\circ\text{--}60^\circ\text{C}$ ) along with dilute  $\text{H}_2\text{SO}_4$  is required. This is essential because the reaction takes place at higher temperature. During the titration, first manganous sulphate is formed which acts as a catalyst for the reduction of  $\text{KMnO}_4$  by oxalic acid. Therefore, in the beginning the reaction rate is slow and as the reaction proceeds, the rate of the reaction increases.

### Procedure:

#### A. Preparation of 0.1 M standard solution of oxalic acid

Prepare 0.1M oxalic acid solution as mentioned in experiment 2.1(Unit 2, Class XI, Laboratory Manual)

#### B. Titration of oxalic acid solution against potassium permanganate solution

1. Rinse and fill a clean burette with potassium permanganate solution. Remove the air bubble, if any, from the nozzle of the burette by releasing some solution through it. The burette used in the permanganate titration must have a glass stop cock as rubber is attacked by permanganate ions.

2. Take 10 mL of 0.1 M oxalic acid solution in a conical flask and add half of the test tube full ( $\approx 5\text{ mL}$ ) of 1.0 M  $\text{H}_2\text{SO}_4$  to it to prevent the formation of any precipitate of manganese dioxide during the course of the titration.



3. Heat the oxalic acid solution up to 50°– 60°C before titrating it with potassium permanganate solution taken in the burette. To increase the visibility of the colour change, place the conical flask containing the solution to be titrated over a white glazed tile kept below the nozzle of the vertically fitted burette.

4. Note the initial reading of the volume of permanganate solution in the burette and add it in small volumes to the hot oxalic acid solution while swirling the contents of the flask gently. The violet colour of permanganate solution is discharged on reaction with oxalic acid. The end point is indicated by the appearance of permanent light pink colour due to a slight excess of permanganate solution.

5. Repeat the titration till three concordant readings are obtained. Since the solution of  $\text{KMnO}_4$  is of dark colour, the upper meniscus should be considered for noting the burette readings.

6. Record the readings as shown in observation Table and calculate the strength of potassium permanganate solution in mols/litre.

**Table:** Titration of potassium permanganate solution against standard oxalic acid solution.

S, No.	Volume of Oxalic acid in ml	Burette Reading		Volume (V) of $\text{KMnO}_4$ used $V = (y-x)$ ml
		Initial (x)	Final (y)	

**Calculations:**

The strength of the unknown solution in terms of molarity may be determined by the following equation.

$$a_1M_1V_1 = a_2M_2V_2$$

For oxalic acid vs potassium permanganate titration:

$a_1 = 2$ , (the number of electrons lost per formula unit of oxalic acid in a balanced equation of half-cell reaction)

$a_2 = 5$ , (the number of electrons gained per formula unit of potassium permanganate in the balanced equation of half-cell reaction)

$M_1$  and  $M_2$  are the molarities of oxalic acid and potassium permanganate solutions used in the titration.

$V_1$  and  $V_2$  are the volumes of oxalic acid and potassium permanganate solutions.

On putting the value of  $a_1$  and  $a_2$  in equation, we get Oxalic acid :  $\text{KMnO}_4$

$$2M_1V_1 = 5M_2V_2$$

$$M_2 = \frac{2M_1V_1}{5V_2}$$

We can calculate the molarity of potassium permanganate solution by using Strength of the solution is given by the following equation:

$$\text{Strength} = \text{Molarity} \times \text{Molar mass}$$

**Result:** Molarity of KMnO<sub>4</sub> solution is .....  
Strength of KMnO<sub>4</sub> solution is .....

### 3. Determination of a mixture of sodium carbonate and sodium hydroxide by using double indicator method.

**Objective:** The objective of this experiment is to determine the concentration of NaOH and Na<sub>2</sub>CO<sub>3</sub> in a mixture by using double indicator method and titration.

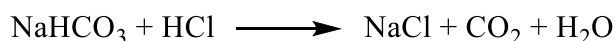
**Introduction:** It is known that NaOH absorbs CO<sub>2</sub> from the atmosphere and converts in to carbonate according to the following equations:



Therefore, sodium hydroxide solution always contains an amount of sodium carbonate. The titration of a mixture of NaOH and Na<sub>2</sub>CO<sub>3</sub> with a standardized HCl solution has two equivalence points. The first equivalence point is due to the conversion of all the carbonate to bicarbonate and sodium hydroxide to sodium chloride as follows:



The pH of the resulting solution is around (8) therefore phenolphthalein could be used as an indicator. The second equivalence point is due to the reaction of the resulting bicarbonate with an excess of HCl solution as follows:



In this stage methyl orange is used to find the equivalence point because the pH of the solution at this point will be around (3.8).

#### Procedure:

1. Pipette 10 mL of the mixture (NaOH and Na<sub>2</sub>CO<sub>3</sub>) into a conical flask and add three drops of phenolphthalein indicator.
2. Titrate the mixture with standard HCl solution from burette until the pink color disappears. Record the volume (V<sub>1</sub>) of the consumed HCl solution.

3. Add 3 drops of methyl orange indicator to the same mixture and continue the titration until the color changes from yellow to pale pink. Find the volume ( $V_2$ ) of the consumed acid in this step.

4. Calculate the amount of the sodium carbonate and sodium hydroxide as follows:

$$(N \times (V_1 - V_2))_{\text{HCl}} = (N \times V)_{\text{NaOH}}$$
$$(N \times 2V_2)_{\text{HCl}} = (N \times V)_{\text{Na}_2\text{CO}_3}$$

### Preparing methyl orange indicator

50 mg of methyl orange in 100 mL solution (distilled water as solvent).

### Result:

### Precautions:

1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

## OXIDATION REDUCTION TITRATION

### 4. Determination of $\text{Fe}^{+2}$ Concentration in an Unknown Sample by Redox Titration.

**Objective:** The objective of this experiment is to determine the ferrous ( $\text{Fe}^{2+}$ ) ion concentration in a sample by redox titration.

**Introduction:** Oxidation reduction (Redox) titrations depend on the transfer of electrons between the two reacting species in solution. A substance that is the electron donor (or has lost or donated electrons) is said to be oxidized, resulting in an increase in its oxidation number. The process is called oxidation. Conversely, a substance that is an electron acceptor (or has gained or accepted electrons) is said to be reduced, resulting in a decrease of its oxidation number. This process is called reduction.

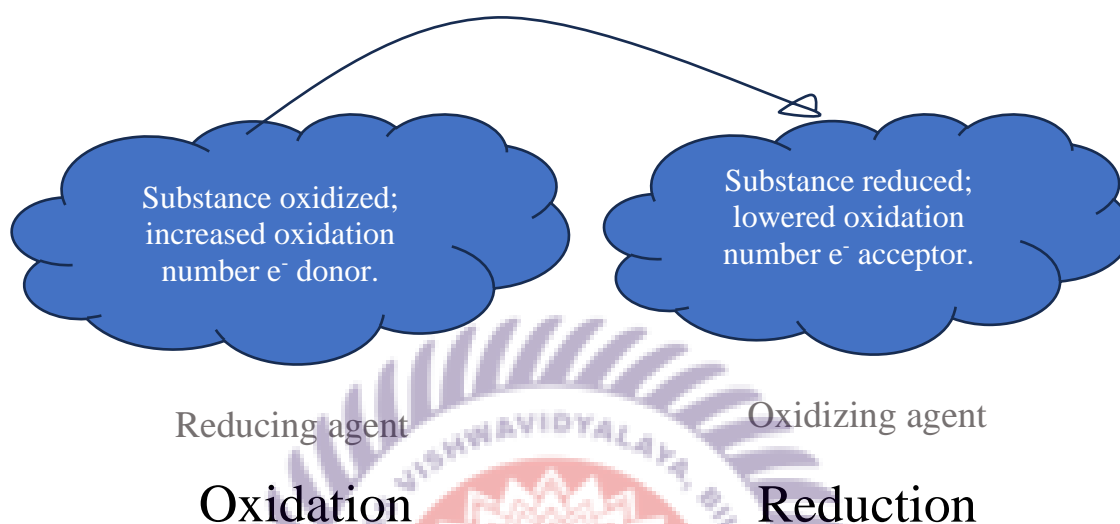
In all redox reactions, the substance that is the electron donor (it is oxidized) donates its electrons to the electron acceptor (to be reduced). Therefore, the substance being oxidized causes a reduction and is thus called a reducing agent. Conversely, the substance that is the electron acceptor (to be reduced) accepts its electrons from the electron donor (it is oxidized). Therefore, this substance being reduced causes an oxidation and is thus called an oxidizing agent.

**Oxidation:** a process whereby a substance loses electrons but increases in oxidation number.

**Reduction:** a process whereby a substance gains electrons but decreases in oxidation number.

**Reducing agent:** a substance that donates electrons, causing reduction of another substance to occur (the reducing agent is therefore oxidized).

**Oxidizing agent:** a substance that accepts electrons, causing oxidation of another substance to occur (the oxidizing agent is therefore reduced).



**Precautions:**

1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

**5. Quantitative estimations of Cu +2 as copper thiocyanate**

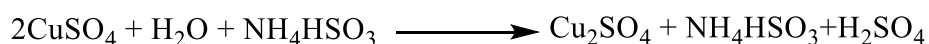
**Aim:** Find out gravimetrically the percentage of Cu in given solution of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , 20g of which has been dissolved per liter.

**Requirements:**

**Apparatus:** Beaker, watch glass, pipette, glass rod.

**Chemical:** Ammonium thiocyanate solution, Saturated solution of  $\text{NH}_4\text{HSO}_3$ , Solution of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ .

**Reactions:**



In this experiment firstly, cupric salt is reduced to cuprous salt either by using sulphurous acid or ammonium bisulphate solution. Then cuprous ions are precipitated by ammonium thiocyanate ( $\text{NH}_4\text{SCN}$ ). After then precipitates formed are filtered, washed dried and weighed using sintered glass crucible. From the weight of precipitates formed percentage of copper is determined.

**Procedure:**

1. Take 20 ml of given solution of copper sulphate in a clean 400 ml beaker.
2. Add few drops of HCl, followed by addition of 25 ml of 10%  $\text{NH}_4\text{HSO}_3$  Solution.
3. Dilute the above solution by adding 150 ml of distilled water. Boil the solution.
4. Now put the beaker on an asbestos sheet, and then add drop wise 10% ammonium thiocyanate solution with constant stirring till the supernatant solution becomes colourless. (To check for complete precipitation, add few drops more of ammonium thiocyanate).
5. Cover the beaker and allow the contents to stand for 2-3 hours preferably overnight.
6. Filter the precipitates using G-4 crucible, wash the precipitates using 1% cold dilute solution of  $\text{NH}_4\text{HSO}_3$  till the filtrate is free from  $\text{SCN}^-$  ions.
7. Finally wash the precipitates with 20% alcohol to remove  $\text{NH}_4\text{SCN}$ .
8. Heat the crucible in oven at  $110^\circ\text{C}$ - $120^\circ\text{C}$  to constant weight.

**Observations:**

Volume of given solution = 20ml

Strength of given Copper sulphate solution = 20 g/litre

Weight of sintered glass crucible Before experiment i.e., empty =  $W_1$  gm

Weight of sintered glass crucible and  $\text{Cu}_2(\text{SCN})_2$  =  $W_2$  g

Weight of  $\text{Cu}_2(\text{SCN})_2$  formed =  $W_2 - W_1 = W$  gm

**Calculations:**

1. From 20 ml of given solution weight  $\text{Cu}_2(\text{SCN})_2$  formed =  $W$  gm

From 1 ml of given solution weight =  $W/20$

From 1000 ml given solution weight =  $W/20 \times 1000 = 50 W$  gm

2.  $\text{Cu}_2(\text{SCN})_2 = 2 \text{ Cu}$

243 gm  $\text{Cu}_2(\text{SCN})_2$  is formed from copper = 127 gm

1 gm  $\text{Cu}_2(\text{SCN})_2$  is formed from copper =  $127/243$

50w gm of  $\text{Cu}_2(\text{SCN})_2$  is formed from copper =  $127/243 \times 50 w = a \text{ gm litre (say)}$

3. In 20 gms of copper sulphate, actual amount of Cu present = a gms

In 1 gms of copper sulphate,  
Actual amount of Cu present =  $a/20$

In 100 gms of copper sulphate,  
Actual amount of Cu present =  $a/20 \times 100 = 5a$

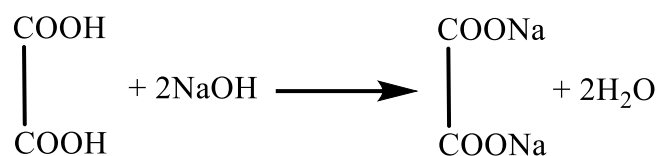
**Result:** Percentage of Cu =  $5a$

**Precautions:**

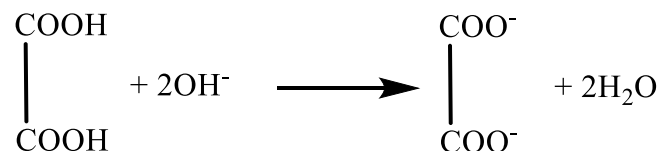
1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.
6. Precipitate i.e.  $\text{NH}_4\text{SCN}$  should not added in excess to avoid solubility of  $\text{Cu}_2(\text{SCN})_2$  as complex ion.
7. Washing of the precipitates is to be done with dilute solution of  $\text{NH}_4\text{HSO}_3$  to avoid oxidation of Cu(I) to Cu (II).

6. **Aim:** To determine volumetrically the strength of given sodium hydroxide solution; prepare a standard oxalic acid solution.

**Principle:** Prepare a standard solution of oxalic acid and then titrate it against sodium hydroxide solution, using phenolphthalein as in indicator.



or



**Procedure:** It involves the following steps:

1. Preparation of 0.1N oxalic acid: Weigh out accurately  $x$  g (= 1.575 g or some other weight) in a weighing bottle. Transfer it to 250 ml measuring flask and add distilled water to it. Now shake the flask to dissolve the acid. Then make the solution up to the mark with distilled water. Shake the flask well for homogenization.

2. Titration Rinse and fill the burette with the prepared oxalic acid solution. Pipette out 20 ml of sodium hydroxide solution into a conical flask. To it add a few drops of phenolphthalein. Now run oxalic acid solution from the burette into the flask until the solution becomes just colorless. Repeat the titration to get at least three concordant readings.

**Calculations:**

Suppose weight of oxalic acid in 250 ml of solution =  $x$  gm

Weight of oxalic acid in 1000 ml of solution =  $4x$  g

Now, the normality of oxalic acid =  $4x / 63$  N

Suppose 20 ml NaOH =  $V_1$  ml of  $4x / 63$  N of oxalic acid

$$20 \times N_{\text{NaOH}} = V_1 \times 4x / 63 \text{ N}$$

$$\text{or } N_{\text{NaOH}} = V_1 / 20 \times 4x / 63 \text{ N}$$

Hence,

The strength of given NaOH =  $V_1 / 20 \times 4x / 63 \text{ N} \times 40 \text{ g/litre}$

**7. Aim:** Conductometric Titration of Mixture of Strong Acid (HCl) and Weak Acid

(CH<sub>3</sub>COOH) With a Strong Base.

**Principle:**

When a mixture of a strong acid and a weak acid is titrated with a strong base or a weak base. In this curve there are two break points. The first break point corresponds to the neutralization of strong acid. When the strong acid has been completely neutralized only then the weak acid starts neutralizing. The second break point corresponds to the neutralization of weak acid and after that the conductance increases due to the excess of OH<sup>-</sup> ions in case of a strong base as the titrant. However, when the titrant is a weak base, it remains almost constant after the end point.

**Requirements:**

**Apparatus:** Conical flask, Burette, Pipette, Volumetric flask, Stand, Beaker.

**Chemicals:** Sodium hydroxide, Hydrochloric acid and acetic acid

**Solution provided:**

1. 0.1 M Sodium hydroxide solution which was standardized with standard oxalic acid.
2. Mixture of ~ 0.01 M HCl (15 cm<sup>3</sup>) and ~ 0.01 M acetic acid (15 cm<sup>3</sup>)

**Procedure:**

1. Take 30 cm<sup>3</sup> of given solution [mixture of ~ 0.01 M HCl (15 cm<sup>3</sup>) and ~ 0.01 M acetic acid (15 cm<sup>3</sup>)] in a 50 cm<sup>3</sup> beaker and dip the conductance cell into it.
2. Take NaOH solution in the burette.
3. Connect the conductometer to the mains and to the conductance cell. Switch on the instrument keeping the meter switch at 'CAL'.
4. Calibrate the meter keeping the selector knob at "20 m S" by rotating the sensitivity knob till the meter reads 1.0.
5. Shift the meter switch to 'READ'. Read the conductance of the solution. Record this value in observation Table



Volume of NaOH added(cm <sup>3</sup> )	Conductance (mS)
0.00	
0.30	
0.60	
0.90	
1.20	
1.80	
2.10	
2.40	
2.70	
3.30	
3.60	
3.90	
4.20	
4.50	
5.10	

6. Make additions of NaOH from the burette as given in Observation Table After each addition, stir the solution well and read the conductance. Enter all the conductance values in observation Table.

7. Plot conductance versus volume of NaOH on a graph sheet and calculate the volume of NaOH used for the neutralization of HCl and acetic acid respectively.

**Observations:**

**Observation Table:** Conductometric titration of a mixture of a strong acid and a weak acid vs. a strong base.

**Calculations:**

Volume of the acid mixture taken = 30 cm<sup>3</sup> [mixture of ~ 0.01 M HCl (15 cm<sup>3</sup>) and ~ 0.01 M acetic acid (15 cm<sup>3</sup>)] Molarity of standardized NaOH solution = = M<sub>1</sub> mol dm<sup>-3</sup> = ..... mol dm<sup>-3</sup>.

1. From the graph, the first change in slope of the conductometric curve gives the first equivalence point (obtained after complete neutralization of HCl present in the acid mixture). From which volume of NaOH corresponding to this first equivalence point  $V_1 \text{ cm}^3$  can be determined.

2. Similarly, the volume of NaOH, corresponding to the second equivalence point  $V_2 \text{ cm}^3$  (total volume of NaOH used for the complete neutralization of both the acids 40 Spectroscopic Methods Lab. can be determined from the graph. Volume NaOH used for the neutralization of weak acid =  $(V_2 - V_1) \text{ cm}^3$

The molarity of strong acid (HCl) =  $M_2 \dots \dots \text{ Mol dm}^{-3}$  Volume of the strong acid taken =  $15 \text{ cm}^3$   
molarity of the weak acid ( $\text{CH}_3\text{COOH}$ ) =  $M_3 \text{ mol dm}^{-3}$  Volume of the weak acid taken =  $15 \text{ cm}^3$

**Results:**

Molarity of HCl =  $M_1 V_1 / 15 \text{ mol dm}^{-3} \dots \dots \dots \text{ mol dm}^{-3}$

Molarity of  $\text{CH}_3\text{COOH}$  =  $M_1 (V_2 - V_1) / 15 \text{ mol dm}^{-3} \dots \dots \dots \text{ mol dm}^{-3}$

**Precautions:**

1. After switching on the instrument (conductometer), it should be allowed to stabilize prior starting the experiment.
2. The conductance cell must always be dipped either in solution or in distilled water.
3. The platinum electrodes of the conductance cell must be completely immersed in the solution during the measurement of conductance.
4. There should be no air bubble between the two electrodes.
5. The titrant must be at least ten times more concentrate than the analyte.

## Inorganic Preparations

**8. Aim:** To prepare pure crystals of tetra ammine copper (II) sulphate.

**Requirements:**

**Apparatus:** Measuring flask, Beakers, Glass Rod, Filter Paper, Burner, Funnel.

**Chemicals:**

1. Copper sulphate = 5gm
2. 1: 1 Ammonia = 20ml
3. Ethyl Alcohol = 20-25ml
4. Conc.  $\text{H}_2\text{SO}_4$  = 1-2ml

**Principle:**

Tetra ammine copper sulphate is a complex of Cu(II) with coordination no. 4. When  $\text{NH}_4\text{OH}$  is gradually added to an aqueous solution of  $\text{CuSO}_4$ ,  $\text{Cu}(\text{OH})_2$  is first precipitated and the precipitate then dissolves, yielding a solution of a bright blue colour which is due to formation of tetra ammine copper (II) ion,  $[\text{Cu}(\text{NH}_3)_4]^{2+}$ . The removal of practically the whole cupric ion from the solution shifts the equilibrium of equation (2) towards the right, until the cupric hydroxide has completely passed into solution. The deep blue solution containing tetra ammine copper (II) ion is known as Schweizer's reagent. It has an ammoniacal odor, and crystals of the complex are obtained by adding ethyl alcohol to the above solution.

**Procedure:**

1. Take 5gm powdered copper sulphate in a 250ml clean beaker and dissolve it in a minimum amount of water. Add 1-2ml conc.  $\text{H}_2\text{SO}_4$  to make the solution clear.
2. Now pour 1:1  $\text{NH}_4\text{OH}$  very slowly into the beaker with constant stirring till a ppt. of  $\text{Cu}(\text{OH})_2$  first formed is redissolved yielding a deep blue solution due to formation of  $[\text{Cu}(\text{NH}_3)_4]^{2+}$  and smell of ammonia is present due to slight excess of  $\text{NH}_4\text{OH}$ .
3. Now to the blue solution, add 20-25ml of ethanol dropwise with constant stirring. Add about 2ml of  $\text{NH}_4\text{OH}$  and heat the beaker in a water bath at  $60-70^\circ\text{C}$  for about 15-20 minutes.
4. Stop heating, cover the beaker with clock glass and allow it to stand undisturbed for 2-3 hours (preferably overnight).
5. Long needle shaped blue crystals of complex are formed. Filter, wash with little alcohol and dry the crystals gently by pressing in between the folds of the filter paper or by placing in a desiccator.

**Observations: -**

1. Yield= about 5gm
2. Colour = Blue
3. Shape = Needle-shaped crystals

**Calculations:**

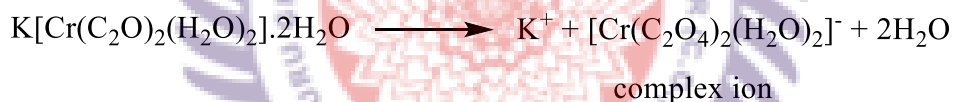
1. Amount of aqua- tetra ammine copper (II) sulphate = \_\_\_ gm
2. Percentage yield of = (Experimental yield/Theoretical yield) \*100

**Results:**

- 1.Amount of aqua-tetra ammine copper (II) sulphate = \_\_\_ gm
2. Percentage yield of aqua-tetra ammine copper (II) sulphate obtained = \_\_\_ %

**9. Aim:** Preparation of cis-and trans-Potassium Dioxalato Diaquo chromate  $K[Cr(C_2O_4)_2(H_2O)_2] \cdot 2H_2O$ .

**Principle:** The complex  $K[Cr(C_2O_4)_2(H_2O)_2] \cdot 2H_2O$  ionises as



In the complex ion  $[Cr(C_2O_4)_2(H_2O)_2]^-$ , chromium has coordination number six and thus the complex ion is octahedral and is of the type  $[M(AA)_2X_2]$  where  $M = Cr$ ,  $AA = (C_2O_4)_2$  (oxalate ion);  $X_2H_2O$ . Therefore, the complex ion is expected to exist in cis- and trans- forms known as geometrical isomers. The structures of the two isomers are given in Fig.

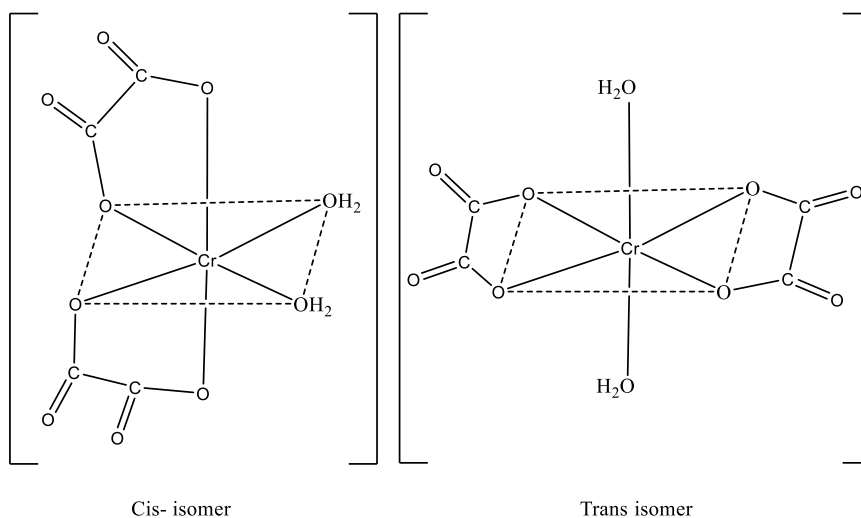


Fig. *cis*- and *trans* isomer of  $[Cr(C_2O_4)_2(H_2O)_2]^-$  ion

Therefore, it will be quite interesting to prepare cis- and trans- isomers of potassium dioxalatodiquochromate (III).

**cis-Potassium dioxalatodiaquochromate (III),  $K[Cr(C_2O_4)_2(H_2O)_2]$**

**Principle:** It may be prepared by the reaction of potassium dichromate with oxalic acid



**Requirements**

Oxalic acid crystals 6 g

Potassium dichromate crystals 2g

Ethanol 40 ml

**Procedure:**

1. Take a dry mortar and put 6 g of oxalic acid crystals and 2 g of potassium dichromate crystals in it.
2. Gently grind the mixture with a pestle till the powders are quite intimately mixed.
3. Take a china dish (10 cm diameter) and moisten it with water. Drain away the excess of water.
4. Introduce the mixture at the centre of the china dish in a compact (v) Cover the china dish with a clock glass.
5. Heat the contents in the china dish gently on a low flame (or a small microscale burner).
6. After a few minutes, a vigorous reaction will set in accompanied with frothing due to evolution of carbon dioxide and water vapours.
7. Soon the reaction will spread throughout the mixture which will become deeply coloured liquid.
8. Without waiting for the thick liquid to cool, pour about 20 ml ethanol over this liquid.
9. Using a metallic spatula, triturate the contents until a solid is formed.
10. In case complete solidification is not achieved, decant the liquid and add to it another lot of 20 ml of ethanol.
11. Warm and resume trituration until the product is in the form of granular crystals.
12. The crystals will look black in diffused day light and deep purple in artificial light.
13. Weigh and record the yield.

**Precautions:**

1. Pestle and mortar must be dry otherwise a reaction may set in.
2. Heating should be very gentle otherwise the reaction may go out of control and cause explosion.
3. Since ethanol has to be added to the contents, naked flame should be kept away as far as is possible.

It is safer to use hot plate instead of burner in this preparation.

**trans-Potassium dioxalatodiaquochromate (III),  $K[Cr(C_2O_4)_2(H_2O)_2]$**

**Principle:** It is prepared as follows:



**Requirements:**

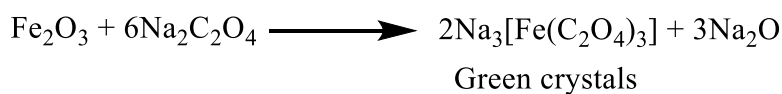
Oxalic acid crystals 6 g.

Potassium dichromate crystals 2 g.

**9. Aim:** Sodium tri oxalato ferrate tri hydrate,  $\text{Na}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ .

**Principle:**

It is prepared by the action of sodium oxalate on hydrated ferric oxide.



**Reagents:**

Ferric chloride = 10 g

Sodium hydroxide = 11.5 g

Oxalic acid = 12 g

**Procedure:**

Dissolve 10 g of ferric chloride in 10 ml of water and dissolve 7.5 g of sodium hydroxide in 5 ml water. To the solution of ferric chloride, add sodium hydroxide solution in small quantities, resulting in the formation of ferric hydroxide. Now filter the ppt. of  $\text{Fe}(\text{OH})_3$  through Buchner funnel and wash it several times with small quantities of hot water.

In another beaker, dissolve 12 g of oxalic acid in 50 ml of water. To this solution, add the pellets of sodium hydroxide (4 g), resulting in the formation of sodium oxalate. Now add the washed hydrated ferric oxide to the hot sodium oxalate solution with constant stirring by a glass rod. Filter the solution and reject any residue if present on the filter paper, and concentrate the green filtrate in a silica dish on a water bath to get the green crystals of sodium ferrioxalate. Wash these crystals first with ice cold water and then with alcohol. Dry the crystals in a desiccator.

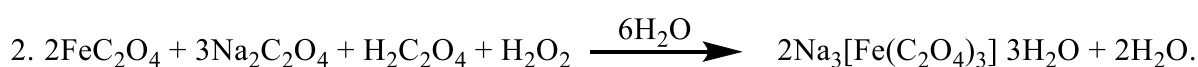
**Expected yield** = 15 g

**Alternative procedure**

**Reagents:**

FeSO<sub>4</sub>.(NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>.6H<sub>2</sub>O = 3 g  
10% H<sub>2</sub>C<sub>2</sub>O<sub>4</sub> solution = 100 ml  
Na<sub>2</sub>C<sub>2</sub>O<sub>4</sub>.2H<sub>2</sub>O = 10 g  
4 N H<sub>2</sub>SO<sub>4</sub> = 1 ml  
Ethyl alcohol  
Acetone  
20 volume H<sub>2</sub>O<sub>2</sub> = 25 ml

**Reactions:**



**Procedure:**

1. Dissolve 3 g or the supplied quantity of ferrous ammonium sulphate in 50 ml of hot distilled water, ml beaker. containing about 1 ml of 4 N H<sub>2</sub>SO<sub>4</sub> in a 250 ml beaker.
2. Stir the solution and add very slowly, 75 ml of 10% to it. oxalic acid solution to it.
3. Heat the solution on a sand bath to boiling, stirring it all the time with a glass rod.
4. Yellow precipitate of ferrous oxalate forms which is allowed to settle down. Decant the supernatant solution completely without disturbing the precipitate as far as possible.
5. Now, add 50 ml of hot distilled water to the precipitate and stir it well. Allow the precipitate to settle down once again and decant the supernatant solution very carefully.
6. Dissolve 10 g of sodium oxalate in 30 ml of hot distilled water and add slowly to the ferrous oxalate precipitate in a beaker.
7. Keep the beaker in a water bath and add slowly 25 ml of 20 volume H<sub>2</sub>O stirring the solution all the time (maintain the temperature of the solution to about 40°C, by keeping the beaker in the water bath).
8. The solution may contain traces of ferric hydroxide precipitate at this stage, so place the beaker on a wire gauze and heat the solution to boiling.
9. Stir the boiling solution continuously and add very rapidly, 20 ml of 10% oxalic acid solution from the burette.
10. Then, add drop by drop 5 ml more of 10% oxalic acid to it, from the burette.
11. Concentrate and filter the hot solution. Collect the filtrate and add 30 ml of warm ethyl alcohol to it. (Any crystals formed, may dissolve in it.)
12. Keep the filtrate for cooling in dark, i.e. cool it for crystallisation.

13. Crystals obtained are filtered through the previously weighed sintered glass crucible. Wash them with 50% ethyl alcohol and then with little quantity of acetone.

14. Dry and then weigh the product.

**Precautions:**

1. Always wear appropriate eye protection (i.e., chemical splash goggles) in the laboratory.
2. Wear disposable gloves, when handling hazardous materials.
3. Wear a full-length, long-sleeved laboratory coat or chemical-resistant apron.
4. Wear shoes that adequately cover the whole foot; low-heeled shoes with non-slip soles are preferable.
5. Clean apparatus before and after use.

