

CHEMISTRY LAB

# Chemistry Lab Manual

Govt. College of Engineering Kalahandi, Bhawanipatna

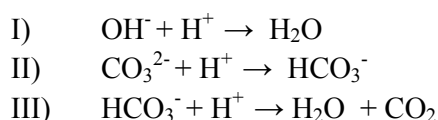
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## **EXPERIMENT No-01: Determination of the amount of NaOH and Na<sub>2</sub>CO<sub>3</sub> present in a solution mixture.**

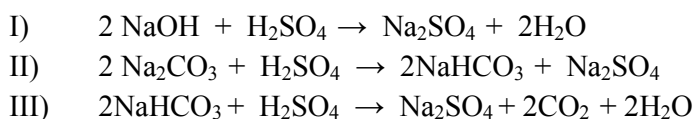
**Aim of the Experiment:** To determine the amount of NaOH and Na<sub>2</sub>CO<sub>3</sub> present in a solution mixture.

### **Theory:**

As the given solution contains NaOH and Na<sub>2</sub>CO<sub>3</sub>, the solution is alkaline in nature. Hence, the amount of NaOH and Na<sub>2</sub>CO<sub>3</sub> can be determined by titration with a standard acid solution, like H<sub>2</sub>SO<sub>4</sub>. The following reactions take place during the titration.



The corresponding molecular reactions can be written as:



The reaction (I) and (II) take place in the alkaline pH range (pH=8-9.8). So the completion of the reaction may be detected by using phenolphthalein indicator. Whereas, the reaction (III) takes place in the acidic pH range (pH=3.1-4.5). So the completion of this reaction may be detected from the colour change of the methyl orange indicator.

### **Procedure: (Write in passive sentence in lab record)**

- i) Clean the burette and Rinse with H<sub>2</sub>SO<sub>4</sub> acid solution and fill it. Fix on the stand after removing air bubbles and note down IBR (initial burette reading).
- ii) Pipette out 10 ml of the given solution and transfer it to a clean conical flask.
- iii) Add 1-2 drop of phenolphthalein indicator. The colour changes to pink.
- iv) Add dilute H<sub>2</sub>SO<sub>4</sub> solution from burette drop-wise into the flask with constant shaking till the pink colour disappears. Note down the FBR-1 (final burette reading-1).
- v) To this solution mixture in the flask, add 1-2 drop of methyl orange so that the solution colour changes to yellow.
- vi) Add the dilute H<sub>2</sub>SO<sub>4</sub> acid again from burette with constant shaking till a faint pink colour appears in the solution.
- vii) Note down FBR-2.
- viii) Repeat the titration with fresh solution till three concordant readings are obtained.

**Titration Table-1: (Using Phenolphthalein indicator)**

Sl. No.	Volume of given solution mixture (ml)	IBR (ml)	FBR (ml)	Difference (=FBR - IBR) in ml
1	10			
2	10			
3	10			

**Titration Table-2: (Using Methyl Orange indicator)**

Sl. No.	Volume of given solution mixture (ml)	IBR (ml)	FBR (ml)	Difference (=FBR - IBR) in ml
1	10			
2	10			
3	10			

**Calculation :**

Volume of the acid consumed in the 1<sup>st</sup> part of the titration = A ml (say)

Volume of the acid consumed in the 2<sup>nd</sup> part of the titration = B ml (say)

**Calculation for the estimation of NaOH**

We know that  $N_1V_1=N_2V_2$

Where,  $N_1$  = Normality of the acid solution (known value)

$V_1$  = Volume of the acid solution consumed for the complete neutralisation of NaOH only = (A - B) ml

$N_2$  = Normality of the sample solution with respect to NaOH only = ?

$V_2$  = Volume of the sample solution = 10 ml

Now,  $N_2 = N_1V_1/V_2 = x$  N (say)

So amount of NaOH present in 1L of the given sample solution mixture = (Normality) x (equivalent weight of NaOH) =  $x \times 40$  g

**Calculation for the estimation of Na<sub>2</sub>CO<sub>3</sub>**

$N_3V_3=N_4V_4$

Where,  $N_3$  = Normality of the acid solution (Known value)

$V_3$  = Volume of the acid solution consumed for the complete neutralisation of Na<sub>2</sub>CO<sub>3</sub> only = 2B ml

$N_4 =$  Normality of the sample solution w.r.t.  $\text{Na}_2\text{CO}_3$  only = ?

$V_4 =$  Volume of the sample solution mixture = 10 ml

Now,  $N_4 = N_3 V_3 / V_4 = y N$  (say)

Amount of  $\text{Na}_2\text{CO}_3$  present in 1L of sample solution mixture

= (Normality) x (Eq. Wt. of  $\text{Na}_2\text{CO}_3$ )

=  $y N \times 53 \text{ g}$

**Conclusion:**

Thus, from the titration, the amount of NaOH and  $\text{Na}_2\text{CO}_3$  present in 1L of the given sample solution mixture are found to be \_\_\_\_\_ g and \_\_\_\_\_ g respectively.

## **EXPERIMENT No -02: Estimation of Hardness of a given Water Sample by EDTA Method.**

**Aim of the Experiment:** To estimate the hardness of a given sample of water by using EDTA method.

### **Chemical required:**

CaCO<sub>3</sub> solution, EDTA solution, EBT (Erichrome Black-T) solution, Buffer solution (pH=10)

### **Apparatus required:**

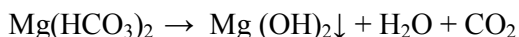
Burette, Burette stand, Pipette, Conical flask, Measuring flask, watch glass.

### **Theory:**

Hardness of water can be defined as the measure of its inability to form lather with soap. In other words, it can be defined as soap consuming capacity of water. Hardness in water generally develops due to the presence of soluble salts of Ca and Mg such as, carbonate bicarbonate, chlorides, and sulfates of Ca and Mg. Some other ions also contribute towards hardness of water.

Hardness of water can be classified into two types.

(a) **Temporary hardness**, which is developed due to presence of carbonate, hydroxide, bicarbonates of Ca and Mg. It can be removed by heating the water so that decomposition of the salts takes place resulting in precipitations as per the following reactions.



(b) **Permanent hardness** (non-carbonate hardness)

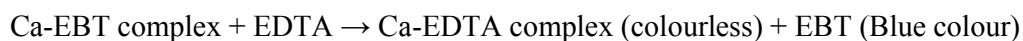
This is caused due to the presence of non-carbonate and non-hydroxide salts of Ca and Mg such as CaCl<sub>2</sub>, CaSO<sub>4</sub>, MgSO<sub>4</sub>, Ca(NO<sub>3</sub>)<sub>2</sub> etc. This cannot be removed by heating, but can be removed by ion-exchange method.

### **Disadvantages of Hard water:**

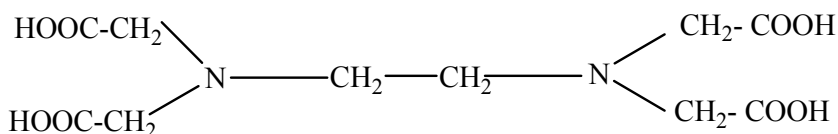
- (i) Hard water is not suitable for domestic purposes like washing, bathing, cooking and drinking.
- (ii) It is also not suitable for paper, pharmaceutical, sugar, textile industries.
- (iii) It cannot be used in industries where boiler is used, since it causes problems like scale and sludge formation, boiler corrosion, caustic embrittlement priming and foaming etc.

Owing to these disadvantages, it is necessary to estimate the hardness of water. It can be performed by titration with EDTA solution using EBT (Eriochrome Black-T) indicator. For convenience, it is assumed that the water sample contains only  $\text{Ca}^{+2}$  ions. The calcium ions form complex with EBT, whose colour is wine-red. When EDTA solution is added, the EBT in Ca-EBT complex is replaced by EDTA to form Ca-EDTA complex and makes EBT free. As the Ca-EDTA complex is colourless, the colour of the solution at the end point is blue, which is the colour of EBT.

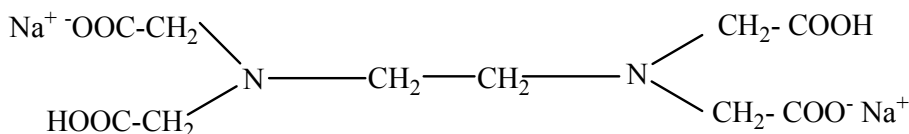
Thus, the reactions taking place during the titration can be written as:



**Structure of EDTA (Ethylene Diamine Tetracetic Acid):**



EDTA is a hexadentate ligand. However, this is not soluble in water. Hence, disodium salt of EDTA, a water soluble salt, is generally used. The **structure of disodium salt of EDTA** is:



The EDTA solution is not of primary standard, so it is first standardized with standard solution like  $\text{CaCO}_3$  solution.

**Procedure: (For Standardisation)**

- (i) Clean the burette with water and then rinse and fill it with supplied EDTA solution. Keep on the stand after removing air bubble and note down IBR (initial burette reading).
- (ii) Pipette out 10 ml of standard  $\text{CaCO}_3$  solution and add 2 ml of buffer solution to it. Then add 1-2 drops of EBT indicator so that the colour of the solution changes to wine red.
- (iv) To this wine red solution, add EDTA solution drop wise from burette with constants shaking till the colour changes to blue. Note down FBR (final burette reading).
- (v) Repeat the titration with fresh solution till three concordant readings are obtained.

**Procedure: (For Estimation)**

Repeat the above titration procedure with supplied water sample (in place of CaCO<sub>3</sub> solution) to get three concordant readings.

**Titration Table-1: (For Standardization)**

Sl. No.	Volume of CaCO <sub>3</sub> solution (ml)	IBR (ml)	FBR (ml)	Difference (=FBR-IBR) in ml
1	10			
2	10			
3	10			

**Titration Table-2: (For Estimation)**

Sl. No.	Volume of water sample (ml)	IBR (ml)	FBR (ml)	Difference (=FBR-IBR) in ml
1	10			
2	10			
3	10			

**Calculation: (Standardization)**

We know that:  $M_1V_1 = M_2V_2$

Where,  $M_1$  = Molarity of EDTA solution = (to be found)

$V_1$  = Volume of EDTA solution = (from burette reading difference in standardization titration)

$M_2$  = Molarity of CaCO<sub>3</sub> solution = known

$V_2$  = Volume of CaCO<sub>3</sub> solution = 10 ml

$M_1 = M_2V_2/V_1 = x$  M (say)

**Calculation: (Estimation)**

Hardness is always calculated in terms of CaCO<sub>3</sub>, although hardness is never present in the form of CaCO<sub>3</sub>. Because calculation becomes easy as molecular weight of CaCO<sub>3</sub> is 100 g and it is the most insoluble salt that can be precipitated in water treatment.

$M_3V_3 = M_4V_4$

$M_3$  = Molarity of EDTA solution =  $x$  M

$V_3$  = Volume of EDTA solution = (from burette reading difference in estimation titration)

$M_4$  = Molarity of water sample w.r.t.  $\text{Ca}^{+2}$  ion = ?

$V_4$  = Volume of water sample = 10 ml

$M_4 = M_3 V_3 / V_4 = y$  M (say)

Amount of  $\text{Ca}^{+2}$  ion in terms of  $\text{CaCO}_3$  in 1L of the given water sample

= (Molarity) X (mol. wt of  $\text{CaCO}_3$ )

=  $y$  M X 100 g

Hardness of water sample =  $100 y \times 1000$  (mg / L)

=  $y \times 10^5$  mg/L

=  $y \times 10^5$  ppm

**Conclusion:**

Using EDTA method, the hardness of the given water sample is estimated to be \_\_\_\_\_ ppm.



## **For more Studies**

### **Disadvantages of hard water:**

(i) In industries like textile, paper, sugar, dyeing, pharmaceutical etc. A large quantity of water is used for steam generation.

a) The use of hard water in boiler causes problems like scale and sludge formation, boiler corrosion, caustic embrittlement priming and foaming etc.

b) In sugar industries, salts dissolved in water cause problems in crystallisation of sugar.

c) The cations present in hard water react with chemicals involved in different steps of paper manufacturing and produces various unwanted side products and undesirable effects like loss of gloss and smoothness, colour change.

d) The sticking of Mg and Ca salts to the fabric surfaces causes undesirable colour change and texture of fabric.

e) In pharmaceutical industries, use of hard water lead to production of undesirable products that may be harmful or even poisonous.

### **(2) In domestic usages:**

a) Drinking:- Taste of soft water is better than hard water. The dissolved Calcium in hard water can produce strong teeth and bones in children. However, hard water can have bad effect on appetite and digestive system. Sometimes it produces Calcium oxalate that causes different urinary problems.

b) Cooking:- The boiling point of hard water increases due to presence of dissolved salts . This causes wastage of fuels and time.

c) Bathing and washing:- hard water produces sticky precipitates of Ca and Mg with soap. So a lot of soap gets wasted and the sticky precipitates adhere on the clothes giving stains and streaks and spots.

## **EXPERIMENT No-04: Determination of ferrous ion in Mohr's salt by potassium permanganate.**

### **Aim of the Experiment:**

To determine the amount of Ferrous ( $\text{Fe}^{+2}$ ) ions present in the supplied Mohr's salt solution.

### **Apparatus required:**

Burette, Burette stand, Pipette, Conical flask, measuring flask.

### **Chemical required:**

Mohr's salt solution, sodium oxalate solution, 6N  $\text{H}_2\text{SO}_4$  and  $\text{KMnO}_4$  solution

### **Theory:**

Mohr's salt is a double salt having molecular formula  $(\text{NH}_4)_2\text{SO}_4 \cdot \text{FeSO}_4 \cdot 6\text{H}_2\text{O}$  (Ferrous ammonium sulphate)

$\text{Fe}^{+2}$  (ferrous) ions can be oxidised to  $\text{Fe}^{+3}$  (ferric) ions by an oxidising agent like  $\text{KMnO}_4$ . Thus,  $\text{Fe}^{+2}$  ions present in Mohr's salt can be determined by titrating with standard  $\text{KMnO}_4$  solution in an acidic medium.

However, the  $\text{KMnO}_4$  solution is not of primary standard. So it has to be standardized by titrating against a primary standard solution of  $\text{Na}_2\text{C}_2\text{O}_4$  (sodium oxalate).

### **Reactions involved:**

#### **During standardisation titration**

Oxidation reaction:  $\text{C}_2\text{O}_4^{2-} \rightarrow 2 \text{CO}_2 + 2\text{e}^-$  ] (multiplied by 5)

Reduction reaction:  $\text{MnO}_4^- + 8 \text{H}^+ + 5\text{e}^- \rightarrow \text{Mn}^{2+} + 4 \text{H}_2\text{O}$  ] (multiplied by 2)

Overall reaction:  $5 \text{C}_2\text{O}_4^{2-} + 2 \text{MnO}_4^- + 16\text{H}^+ \rightarrow 10 \text{CO}_2 + 2 \text{Mn}^{2+} + 8 \text{H}_2\text{O}$

The corresponding balanced molecular equation can be written as

$$5 \text{Na}_2\text{C}_2\text{O}_4 + 2 \text{KMnO}_4 + 8 \text{H}_2\text{SO}_4 \rightarrow 10 \text{CO}_2 + 2 \text{MnSO}_4 + 8 \text{H}_2\text{O} + 5 \text{Na}_2\text{SO}_4 + \text{K}_2\text{SO}_4$$

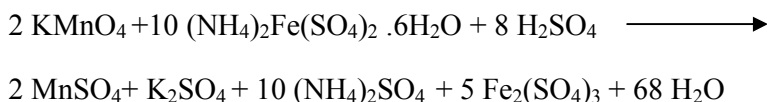
#### **Reaction involved during estimation titration:**

Oxidation reaction:  $\text{Fe}^{2+} \rightarrow \text{Fe}^{3+} + \text{e}^-$  ] (multiplied by 5)

Reduction reaction:  $\text{MnO}_4^- + 8 \text{H}^+ + 5\text{e}^- \rightarrow \text{Mn}^{2+} + 4 \text{H}_2\text{O}$

Overall reaction:  $5 \text{Fe}^{2+} + \text{MnO}_4^- + 8 \text{H}^+ \rightarrow 5 \text{Fe}^{3+} + \text{Mn}^{2+} + 4 \text{H}_2\text{O}$

The corresponding balanced molecular equation can be written as



**Procedure:(for standardization)**

- (i) Clean the Burette with water, then rinsed with  $\text{KMnO}_4$  solution and fill with it. Remove air bubble and note down IBR.
- (ii) Pipette out 10ml of Sodium oxalate solution (known conc.) and transfer to a clean and dry conical flask.
- (iii) Add 10 cc of 6N  $\text{H}_2\text{SO}_4$  acid solution to it and titrate against  $\text{KMnO}_4$  solution till a faint permanent pink colour is obtained.
- (iv) If the pink colour does not persist, warm the solution in conical flask and then add few drops of  $\text{KMnO}_4$  solution to get a permanent faint pink colour. Note down FBR.
- (v) Repeat the titration with fresh solution to get three concordant readings.

**Procedure:(for estimation)**

- (i) Note down IBR.
- (ii) Pipette out 10ml of Mohr's salt solution and transfer it to a clean conical flask.
- (iii) Add 10 cc of 6N  $\text{H}_2\text{SO}_4$  to it and titrate against  $\text{KMnO}_4$  solution by adding drop wise from burette till a faint permanent pink colour is obtained.
- (iv) Note down FBR.
- (v) Repeat the titration with fresh solution to get three concordant readings.

**Titration Table-1: (Standardization)**

Sl. No.	Volume of sodium oxalate solution (ml)	IBR (ml)	FBR (ml)	Difference=(FBR - IBR) in ml
1	10			
2	10			
3	10			

**Titration Table-2: (Estimation)**

Sl. No.	Volume of Mohr's salt solution (ml)	IBR (ml)	FBR (ml)	Difference=(FBR - IBR) in ml
1	10			
2	10			
3	10			

**Calculation: (for standardization of KMnO<sub>4</sub>)**

From the balanced chemical equation, it is found that 2 mole of KMnO<sub>4</sub> oxidises 5 mole of Sodium oxalate.

$$M_1V_1/n_1 = M_2V_2/n_2$$

$$M_1 = \text{Molarity of KMnO}_4 \text{ solution} = ?$$

V<sub>1</sub> = Volume of KMnO<sub>4</sub> solution = (from burette reading difference in standardisation titration)

$$n_1 = 2 \text{ mole}$$

$$M_2 = \text{Molarity of Sodium oxalate solution} = (\text{known value})$$

$$V_2 = \text{Volume of Sodium oxalate solution} = 10 \text{ ml}$$

$$n_2 = 5 \text{ mole}$$

$$\text{So } M_1 = (M_2V_2/n_2) \times (n_1/V_1) = x \text{ M (say)}$$

**Calculation: (for estimation)**

From the balanced chemical equation, it is found that 2 moles of KMnO<sub>4</sub> oxidises 10 moles of Mohr's salt.

$$M_3V_3/n_3 = M_4V_4/n_4$$

$$\text{Where, } M_3 = \text{Molarity of KMnO}_4 \text{ solution} = x \text{ M}$$

V<sub>3</sub> = Volume of KMnO<sub>4</sub> solution (from burette reading difference in estimation titration)

$$n_3 = 2 \text{ mole}$$

$$M_4 = \text{Molarity of Mohr's salt solution} = ?$$

$$V_4 = \text{Volume of Mohr's salt solution} = 10 \text{ ml}$$

$$n_4 = 10 \text{ mole}$$

$$\text{Hence, } M_4 = (M_3V_3/n_3) \times (n_4/V_4) = y \text{ M (say)}$$

So amount of Fe<sup>+2</sup> (ferrous) ions present in 1L of the given Mohr's salt solution is

$$= (\text{molarity}) \times (\text{molecular weight of Fe}^{+2} \text{ ion})$$

$$= y \times 56.58 \text{ g} = \text{----- g}$$

**Conclusion:** From the titration, it is found that 1L of the supplied Mohr's salt solution contains ----- g of ferrous ions.