# DEPARTMENT OF SCIENCE AND HUMANITIES 

I SEMESTER - R 2017

BS8161 CHEMISTRY LABORATORY

## LABORATORY MANUAL

Name

Register No $\qquad$

Section $\qquad$

## VISION

College of Engineering is committed to provide highly disciplined, conscientious and enterprising professionals conforming to global standards through value based quality education and training.

## MISSION

- To provide competent technical manpower capable of meeting requirements of the industry
- To contribute to the promotion of Academic Excellence in pursuit of Technical Education at different levels
- To train the students to sell his brawn and brain to the highest bidder but to never put a price tag on heart and soul


## PROGRAMME EDUCATIONAL OBJECTIVES (PEOs)

## 1. Fundamentals

To provide students with a solid foundation in Mathematics, Science and fundamentals of engineering, enabling them to apply, to find solutions for engineering problems and use this knowledge to acquire higher education

## 2. Core Competence

To train the students in chemistry practicals so that they apply their knowledge and training to compare, and to analyze various engineering industrial problems to find solutions

## 3. Breadth

To provide relevant training and experience to bridge the gap between theory and practice this enables them to find solutions for the real time problems in industry, and to design products

## 4. Professionalism

To inculcate professional and effective communication skills, leadership qualities and team spirit in the students to make them multi-faceted personalities and develop their ability to relate engineering issues to broader social context

## 5. Lifelong Learning/Ethics

To demonstrate and practice ethical and professional responsibilities in the industry and society in the large, through commitment and lifelong learning needed for successful professional career

## PROGRAMME OUTCOMES (POs)

To demonstrate and apply knowledge of Mathematics, Science and engineering fundamentals in the following experiments.

1 .To estimate HCl using $\mathrm{Na}_{2} \mathrm{CO}_{3}$ as primary standard and Determination of alkalinity in water sample.
2. To determine total, temporary \& permanent hardness of water by EDTA method.
3. To determine DO content of water sample by Winkler's method.
4. To determine chloride content of water sample by Argentometric method.
5. To estimate copper content of the given solution by lodometry.
6. To determine strength of given hydrochloric acid using pH meter.
7. To determine strength of acids in a mixture of acids using conductivity meter.
8. To determine iron content of the given solution using potentiometer.
9. To estimate iron content of the water sample using spectrophotometer (1, 10-Phenanthroline / thiocyanate method).
10. To estimate sodium and potassium present in water using flame photometer.
11. To determine molecular weight of polyvinyl alcohol using Ostwald viscometer.
12. To determine Pseudo first order kinetics -ester hydrolysis.
13. To determine Corrosion experiment tweightloss method.
14. To estimate CMC.
15. To estimate Phase change in a solid.
16. To determine strength of strong acid vs strong base using conductivity meter.

## BS8161 - CHEMISTRY LABORATORY SYLLABUS

## COURSE OBJECTIVES

- To make the student to acquire practical skills in the determination of water quality parameters through volumetric and instrumental analysis.
- To acquaint the students with the determination of molecular weight of a polymer by viscometery.


## LIST OF EXPERIMENTS:

1. Estimation of HCl using $\mathrm{Na}_{2} \mathrm{CO}_{3}$ as primary standard and Determination of alkalinity in water sample.
2. Determination of total, temporary \& permanent hardness of water by EDTA method.
3. Determination of DO content of water sample by Winkler's method.
4. Determination of chloride content of water sample by Argentometric method.
5. Estimation of copper content of the given solution by lodometry.
6. Determination of strength of given hydrochloric acid using pH meter.
7. Determination of strength of acids in a mixture of acids using conductivity meter.
8. Estimation of iron content of the given solution using potentiometer.
9. Estimation of iron content of the water sample using spectrophotometer
(1, 10-Phenanthroline / thiocyanate method).
10. Estimation of sodium and potassium present in water using flame photometer.
11. Determination of molecular weight of polyvinyl alcohol using Ostwald viscometer.
12. Pseudo first order kinetics -ester hydrolysis.
13. Corrosion experiment -weight loss method.
14. Determination of CMC.
15. Phase change in a solid.
16. Conductometric titration of strong acid vs strong base.

## COURSE OUTCOMES

- The students will be outfitted with hands-on knowledge in the quantitative chemical analysis of water quality related parameters.

INDEX[SEMESTER - I]

| S.No. | Date | Experiment | Date of submission | Mark | Signature |
| :---: | :---: | :---: | :---: | :---: | :---: |
| 1. |  | Estimation of HCl using $\mathrm{Na}_{2} \mathrm{CO}_{3}$ as primary standard and Determination of alkalinity in water sample. |  |  |  |
| 2. |  | Determination of total, temporary \& permanent hardness of water by EDTA method. |  |  |  |
| 3. |  | Determination of chloride content of water sample by argentometric method. |  |  |  |
| 4. |  | Determination of strength of given hydrochloric acid using pH meter. |  |  |  |
| 5. |  | Determination of strength of acids in a mixture of acids using conductivity meter. |  |  |  |
| 6. |  | Estimation of iron content of the given solution using potentiometer. |  |  |  |
| 7. |  | Conductometric titration of strong acid Vs strong base. |  |  |  |
| 8. |  | Estimation of copper content of the given solution by lodometry |  |  |  |
| 9. |  | Estimation of sodium and potassium present in water using flame photometer |  |  |  |

## CONTENTS

| S.No. | NAME OF EXPERIMENT | PAGE <br> No. |
| :---: | :--- | :---: |
| SEMESTER - I | Estimation of HCl using $\mathrm{Na}_{2} \mathrm{CO}_{3}$ as primary standard and Determination of alkalinity in <br> water sample. | 09 |
| 2. | Determination of total, temporary \& permanent hardness of water by EDTA method. | 15 |
| 3. | Determination of chloride content of water sample by Argentometric method. | 21 |
| 4. | Determination of strength of given hydrochloric acid using pH meter. | 25 |
| 5. | Determination of strength of acids in a mixture of acids using conductivity meter. | 29 |
| 6. | Estimation of iron content of the given solution using potentiometer. | 33 |
| 7. | Conductometric titration of strong acid Vs strong base. | 39 |
| 8 | Estimation of copper content of the given solution by lodometry | 43 |
| 9 | Estimation of sodium and potassium present in water using flame photometer |  |

## SHORT PROCEDURE:

| CONTENTS | TITRATION |
| :---: | :--- |
|  | Estimation of alkalinity in water sample <br> (Std HCl Vs water sample) |
| Burette solution | Std. HCl |
| Pipette solution | Water sample |
| Indicator | (i) $\quad$Phenolphthalein <br> (ii) Methyl orange <br> End point |
| (i) Disappearance of pink colour |  |
| (ii) Appearance of reddish orange colour |  |


| Relationship between P \& M | Nature of alkalinity | Amount of individual alkalinities | $\mathrm{OH}^{-}$ | $\mathrm{HCO}_{3}{ }^{-}$ | $\mathrm{CO}_{3}{ }^{\text {2- }}$ |
| :---: | :---: | :---: | :---: | :---: | :---: |
| $P=M$ | only $\mathrm{OH}^{-}$ | P or M | - | 0.0 | 0.0 |
| $2 \mathrm{P}=\mathrm{M}$ | only $\mathrm{CO}_{3}{ }^{2-}$ | 2P or M | 0.0 | 0.0 | - |
| $\mathrm{P}=0, \mathrm{M} \neq 0$ | only $\mathrm{HCO}_{3}{ }^{-}$ | M | 0.0 | - | 0.0 |
| $2 \mathrm{P}>\mathrm{M}$ | $\mathrm{OH}^{-} \& \mathrm{CO}_{3}{ }^{2-}$ | $\begin{gathered} \mathrm{OH}^{-}=2 \mathrm{P}-\mathrm{M} \\ \mathrm{CO}_{3}{ }^{2-}=2(\mathrm{M}-\mathrm{P}) \end{gathered}$ | - | 0.0 | - |
| P < $1 / 2 \mathrm{M}$ | $\mathrm{HCO}_{3}{ }^{-} \mathrm{CO}_{3}{ }^{-{ }^{-}}$ | $\begin{gathered} \mathrm{HCO}_{3}^{-}=\mathrm{M}-2 \mathrm{P} \\ \mathrm{CO}_{3}^{2-}=2 \mathrm{P} \end{gathered}$ | 0.0 | - | - |

## Expt. No. 1 DETERMINATION OF ALKALINITY IN WATER

## AIM

To determine the type and amounts of alkalinity in the given water sample. A standard solution of $\mathrm{Na}_{2} \mathrm{CO}_{3}$ of strength $\qquad$ $N$ is given.

## PRINCIPLE

Alkalinity in water is due to the presence of soluble hydroxides, bicarbonates and carbonates. Determination of various types and amounts of alkalinity is easily carried out by titration with standard HCl employing the indicators phenolphthalein and methyl orange independently or in succession.

The following reactions occur when different types of alkalinity are neutralized with acid.

$$
\begin{align*}
& \mathrm{OH}^{-}+\mathrm{H}^{+} \rightarrow \mathrm{H}_{2} \mathrm{O} \text { completed at pH 8.2-9.0 }  \tag{1}\\
& \mathrm{CO}_{3}^{2-}+\mathrm{H}^{+} \rightarrow \mathrm{HCO}_{3}^{-}  \tag{2}\\
& \mathrm{HCO}_{3}^{-}+\mathrm{H}^{+} \rightarrow\left(\mathrm{H}_{2} \mathrm{CO}_{3}\right) \rightarrow \mathrm{H}_{2} \mathrm{O}+\mathrm{CO}_{2} \text {, completed at pH 4.2-5.5 } \tag{3}
\end{align*}
$$

Neutralisation (1) \& (2) will be notified by phenolphthalein end-point while all the three will be accounted by methyl orange end-point. Bicarbonate in eqn. (3) may be due to the existence of soluble free bicarbonate salts and bicarbonates resulting from half neutralization of soluble carbonates (eqn. (2))

## Various steps to be followed

a) A known volume of water sample is titrated against std. HCl using first phenolphthalein indicator till end-point $(P)$ and the titration is continued without break using methyl orange indicator till the endpoint (M).

Titration
Estimation of Alkalinity in water sample
Standard HCl Vs Water sample

Indicator: 1. Phenolphthalein
2. Methyl Orange

| S.No. | Volume <br> of water <br> sample (ml) | Burette Reading |  |  |  | (nitial |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  | Phenolphthalein End <br> Point (P) | Methyl Orange End <br> Point (M) | Concordant value |  |
| 2 |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |

## CALCULATION

Hydroxide ion alkalinity
Volume of $\mathrm{HCl}(2 \mathrm{P}-\mathrm{M})=$ ml

Strength of $\mathrm{HCl}=$ $\qquad$ N

Volume of water sample = $\qquad$ ml

Strength of water sample $=\frac{\text { Volume of } \mathrm{HCl}(2 \mathrm{P}-\mathrm{M}) \mathrm{X} \text { Strength of } \mathrm{HCl}}{\text { Volume of water sample }}$
$=$
$\therefore$ Hydroxide ion alkalinity in terms of
$\mathrm{CaCO}_{3}$ equivalent $=$ Strength of water sample X Equivalent weight of $\mathrm{CaCO}_{3} \mathrm{X} 1000$
$=$ Strength of water sample $\times 50 \times 1000$
$=$ $\qquad$ .mg/l
b) From the magnitudes of the $P$ \& $M$, the nature of alkalinity can be arrived as follows:
(i) $\mathrm{P}=\mathrm{M} \quad \Rightarrow \quad$ Presence of only $\mathrm{OH}^{-}$
(ii) $2 \mathrm{P}=\mathrm{M} \quad=>\quad$ Presence of only $\mathrm{CO}_{3}{ }^{2-}$
(iii) $\mathrm{P}=0, \mathrm{M}=0 \quad \Rightarrow \quad$ Presence of only $\mathrm{HCO}_{3}{ }^{-}$
(iv) $2 \mathrm{P}>\mathrm{M} \quad \Rightarrow \quad$ Presence of $\mathrm{OH}^{-} \& \mathrm{CO}_{3}{ }^{2-}$
(v) $2 \mathrm{P}<\mathrm{M} \quad \Rightarrow \quad$ Presence of $\mathrm{HCO}_{3}{ }^{-} \& \mathrm{CO}_{3}{ }^{2-}$
(Mixture of $\mathrm{OH}^{-} \& \mathrm{HCO}^{-}$is not listed since they do not exist together and are considered equivalent to $\mathrm{CO}_{3}{ }^{2}$ ).

## PROCEDURE

TITRATION

## ESTIMATION OF ALKALINITY IN WATER SAMPLE

Exactly 20 ml of water sample is pipetted out into a clean conical flask. Few drops of phenolphthalein indicator are added and titrated against a standardized HCl taken in the burette. The end point is the disappearance of pink colour, which is noted as $P$. Few drops of methyl orange indicator is added to the same solution. The solution turns to yellow. The titration is continued further by adding same HCl without break till the end point is reached. The end point is the colour change from yellow to reddish orange.

The experiment is repeated to get concordant values. From the magnitudes of $P \& M$ values, the type of alkalinity present in the water sample is inferred and the individual amounts are calculated and reported.

Carbonate ion alkalinity
Volume of $\mathrm{HCl} 2(\mathrm{M}-\mathrm{P})=$ $\qquad$ .ml

Strength of HCl
$=$ $\qquad$ .N

Volume of water sample
$=$ $\qquad$ .ml

$\therefore$ Carbonate ion alkalinity
in terms of $\mathrm{CaCO}_{3}$ equivalent $=$ Strength of water sample X Equivalent weight of $\mathrm{CaCO}_{3} \mathrm{X} 1000$
$=$ Strength of water sample $\times 50 \times 1000$
$=$ mg/l

## RESULT



## VIVA-VOCE

1. What is meant by alkalinity?
2. What are the ions responsible for alkalinity?
3. Why $\mathrm{OH}^{-}$and $\mathrm{HCO}_{3}{ }^{-}$ions cannot exist together in water?
4. What are the ions responsible for alkalinity when $2 P>M$ ?
5. What is the pH range for both in acidic and basic solutions?
6. What is the colour of phenolphthalein in an acid and in an alkaline solution?
7. What is the purpose of using two indicators in same titration?
8. What are the possible combinations of ions for alkalinity?
9. How is the amount of alkalinity expressed?
10. What are the types of indicator used in this experiment?

SHORT PROCEDURE

| CONTENTS | TITRATION I | TITRATION II | TITRATION III |
| :--- | :---: | :---: | :---: |
|  | Standardisation of <br> EDTA <br> (EDTA Vs Std <br> Hard water) | Determination of <br> total hardness <br> (Std EDTA Vs Hard <br> water sample) | Determination of <br> permanent hardness <br> (Std EDTA Vs Boiled <br> Hard water sample) |
|  | EDTA solution | EDTA solution | EDTA solution |
|  | 20 ml Std. hard water | 20 ml hard water sample | 20ml boiled hard water sample |
| Indicator | 2 drops of EBT | 2 drops of EBT | 2 drops of EBT |
| Additional soln. | Ammoniacal Buffer <br> solution (5ml) | Ammoniacal Buffer solution <br> $(5 \mathrm{ml})$ | Ammoniacal Buffer solution <br> $(5 \mathrm{ml})$ |
| End point | Wine - red to Steel blue | Wine - red to steel blue <br> Wine - red to Steel blue |  |

## STANDARDISATION OF EDTA

Titration - I
Std. HARD WATER Vs EDTA
Indicator: EBT

| S. No. | Volume of std hard <br> water (ml) | Burette Reading |  |  | Volume of <br> EDTA |
| :--- | :---: | :---: | :---: | :---: | :---: |
|  |  |  | Initial | Final | Concordant value |
|  |  |  |  |  |  |


| Volume of std .hard water taken | 20 ml |
| :---: | :---: |
| Volume of EDTA consumed ( $\mathrm{V}_{1}$ | -----ml |
| 1 ml of std.hard water | $=1 \mathrm{mg}$ of $\mathrm{CaCO}_{3}$ |
| 20 ml of std hard water | $=20 \mathrm{mg}$ of $\mathrm{CaCO}_{3}$ |

$\mathrm{V}_{1} \mathrm{ml}$ of EDTA solution containing 20 mg of $\mathrm{CaCO}_{3}$
1 ml of EDTA solution $\quad=\quad 20 / \mathrm{V}_{1}{\mathrm{mg} \text { of } \mathrm{CaCO}_{3}}^{2}$
$=----------------------m g$ of $\mathrm{CaCO}_{3}$

## Expt. No. 2 DETERMINATION OF TOTAL, TEMPORARY AND PERMANENT HARDNESS OF WATER

## AIM

To estimate the amount of total, temporary \& permanent hardness in the given sample of water.

## PRINCIPLE

The estimation is based on the complexometric titration.
(i) Total hardness of water is estimated by titrating against EDTA using EBT indicator.
$\mathrm{EBT}+\mathrm{M} \rightarrow[\mathrm{EBT}-\mathrm{M}]$ (unstable complex)
(EBT-M) (Unstable complex) +EDTA $\rightarrow$ [EDTA-M] (stable complex) +EBT
EBT indicator forms wine- red colored complex with metal ions present in water. On addition of EDTA metal ions preferably forms complexes with EDTA and steel blue EBT indicator is set free. Therefore change of colour from wine- red to steel blue denotes the end point.
(ii) Temporary hardness is removed by boiling the water.

$$
\begin{aligned}
& \mathrm{Ca}\left(\mathrm{HCO}_{3}\right)_{2} \rightarrow \mathrm{CaCO}_{3}+\mathrm{CO}_{2} \uparrow+\mathrm{H}_{2} \mathrm{O} \\
& \mathrm{Mg}\left(\mathrm{HCO}_{3}\right)_{2} \rightarrow \mathrm{Mg}(\mathrm{OH})_{2}+2 \mathrm{CO}_{2} \uparrow
\end{aligned}
$$

The precipitate is filtered and the remaining permanent hardness is estimated using EDTA.

## PROCEDURE

## TITRATION - I

## sTANDARDISATION OF EDTA

Pipette out 20 ml of standard hard water into a 250 ml conical flask. Add 5 ml of buffer solution and 3 drops of eriochrome black $T$ indictor. Titrate the solution with EDTA from the burette until the colour changes from wine- red to steel blue at the end point. Repeat the titration for concordant values. Let the titre value be $\mathrm{V}_{1} \mathrm{ml}$.

Titration-II
SAMPLE HARD WATER Vs Std EDTA
Indicator: EBT

| S.No. | Volume of sample <br> hard water (ml) | Burette Reading |  |  | Volume of <br> EDTA <br> $\left(\mathrm{V}_{2}\right)(\mathrm{ml})$ |
| :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  | Initial | Final |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

## CALCULATION

## Calculation of total hardness

| Volume of EDTA consumed ( $\mathrm{V}_{2}$ ) | -- ml |
| :---: | :---: |
| 1 ml of EDTA | $=20 / \mathrm{V}_{1} \mathrm{mg}$ of $\mathrm{CaCO}_{3}$ |
| $V_{2} \mathrm{ml}$ of EDTA | $=20 / \mathrm{V}_{1} \times \mathrm{V}_{2} \mathrm{mg}$ of $\mathrm{CaCO}_{3}$ |

( i.e), If 20 ml of sample hard water contains $20 \times \mathrm{V}_{2} \mathrm{mg}$ of $\mathrm{CaCO}_{3} / \mathrm{V}_{1}$

Then 1000 ml of water sample $20 \times \mathrm{V}_{2} \times 1000 / \mathrm{V}_{1} \times 20 \mathrm{mg}$ of $\mathrm{CaCO}_{3}$
$=1000 \times \mathrm{V}_{2} / \mathrm{V}_{1} \mathrm{mg}$ of CaCO 3

Total hardness of given water sample = ppm

## DETERMINATION OF TOTAL HARDNESS

Pipette out 20 ml of sample hard water into a clean conical flask. Add 5 ml of buffer solution and $4-5$ drops of Eriochrome black -T indicator. Titrate the wine - red coloured solution with EDTA from the burette until the colour steel blue appears at the end point. Repeat the titration for concordant values. Let the titre value be V2ml.

## DETERMINATION OF PERMANENT HARDNESS

Take 100 ml of hard water sample in a 500 ml beaker and boil gently for about 1 hour. Cool, filter it into a 100 ml standard flask and make the volume up to the mark. Take 20 ml of this solution and proceed it in the same way as in titration (I). The volume of EDTA consumed corresponds to the permanent hardness of the water sample. Let the titre value be $\mathrm{V}_{3} \mathrm{ml}$. Temporary hardness is calculated by subtracting permanent hardness from total hardness.

## DETERMINATION OF PERMANENT HARDNESS <br> BOILED SAMPLE WATER Vs Std. EDTA Indicator: EBT

| S.No. | Volume of boiled <br> hard water (ml) | Burette Reading (ml) |  |  | Volume of <br> EDTA <br> (mI) |
| :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | Initial | Final | Concordant value |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

## CALCULATION OF PERMANENT HARDNESS

Volume of EDTA consumed $\mathrm{V}_{3}$

1 ml of EDTA
$V_{3} m l$ of EDTA
(i.e) 20 ml of boiled sample hard water

Then 1000 ml of boiled water sample
permanent hardness of boiled water sample

Temporary hardness of the given water sample
= ------------------m|
$=20 / \mathrm{V}_{1} \mathrm{mg}$ of $\mathrm{CaCO}_{3}$
$=20 / \mathrm{V}_{1} \times \mathrm{V}_{3} \mathrm{mg}$ of $\mathrm{CaCO}_{3}$
$=20 / \mathrm{V}_{1} \times \mathrm{V}_{3} \mathrm{mg}$ of $\mathrm{CaCO}_{3}$
$=20 / \mathrm{V}_{1} \times \mathrm{V}_{3} / 20 * 1000 \mathrm{mg}$ of $\mathrm{CaCO}_{3}$
$=1000 \times \mathrm{V}_{3} / \mathrm{V}_{1} \mathrm{mg}$ of $\mathrm{CaCO}_{3}$
= -----------------ppm
= Total hardness - Permanent hardness

## RESULT

(i) Total hardness of the given sample water $=-------------\mathrm{ppm}$.
(ii) Permanent hardness of the given sample water $=--------------\mathrm{ppm}$.
(iii) Temporary hardness of the given sample water $=---------------$-ppm.

## VIVA-VOCE

1. Define - Hard water
2. What is the structure of EDTA?
3. How will you calculate temporary hardness?
4. What is the role of the buffer in EDTA titration?
5. What is the permissible limit of hardness indrinking water?
6. Name the hardness producing ions present in water.
7. Name two salts which are responsible for temporary hardness.
8. Why is $\mathrm{CaCO}_{3}$ taken as the standard?
9. Write the expansion of EDTA.
10. What is meant by EBT?

## SHORT PROCEDURE

| CONTENTS | TITRATION I | TITRATION II |
| :--- | :---: | :---: |
|  | Standardisation of <br> $\mathrm{AgNO}_{3}$ | Estimation of Chloride |
| Burette solution | $\mathrm{AgNO}_{3}$ solution | Std. $\mathrm{AgNO}_{3}$ solution |
| Pipette solution | 20 ml std KCl solution | 20 ml water sample |
| Indicator | $1 \mathrm{ml} \mathrm{K}_{2} \mathrm{CrO}_{4}$ | $1 \mathrm{ml} \mathrm{K}_{2} \mathrm{CrO}_{4}$ |
| End point | yellow to reddish brown | yellow to reddish brown |

Titration I
Std. $\mathrm{AgNO}_{3} \mathrm{Vs} \mathrm{KCl}$
Indicator: Potassium Chromate

| S.No. | Volume of KCl <br> (ml) | Burette Reading (ml) |  | Volume of $\mathrm{AgNO}_{3}(\mathrm{ml})$ | Concordant Value |
| :---: | :---: | :---: | :---: | :---: | :---: |
|  |  | Initial | Final |  |  |
|  |  |  |  |  |  |

## CALCULATION

Volume of KCl
$\left(V_{1}\right)=$
------- ml
Strength of KCl
$\left(\mathrm{N}_{1}\right)=$
------- N

Volume of Silver Nitrate
$\left(V_{2}\right)=$ $\qquad$
Strength of Siver Nitrate

$$
N_{2}=\frac{V_{1} N_{1}}{V_{1}}
$$

$$
=\quad--------------------N
$$

## Expt.No. 3 ESTIMATION OF CHLORIDE CONTENT IN WATER (ARGENTOMETRIC METHOD)

AIM
To estimate the amount of chloride present in given water sample by Argentometric method (Mohr's method). You are provided with standard solution of NaCl of strength ------N and a approximately $\mathrm{N} / 10$ solution of Silver nitrate.

## PRINCIPLE

The Chloride ion in the form of $\mathrm{NaCl}, \mathrm{KCl}, \mathrm{CaCl}_{2}, \mathrm{MgCl}_{2}$ etc., present in the water sample can be estimated by Argentometric method or known as Mohr's method. It is an example for precipitation titration. The reaction between std. Sodium chloride and Silver nitrate is given below.

$$
\mathrm{AgNO}_{3}+\mathrm{NaCl} \rightarrow \mathrm{AgCl} \downarrow+\mathrm{NaNO}_{3}
$$

The completion of reaction can be observed by employing potassium chromate as the indicator. The end point is the colour change from yellow to reddish brown due to the following reaction.

$$
2 \mathrm{AgNO}_{3}+\mathrm{K}_{2} \mathrm{CrO}_{4} \rightarrow \mathrm{Ag}_{2} \mathrm{CrO}_{4} \downarrow+2 \mathrm{KNO}_{3}
$$

Potassium chromate indicator will not be precipitated as silver chromate until all the chlorides in the solution have been precipitated as AgCl Since a precipitation reaction takes place in preference, this reaction takes place only at the end point.

## PROCEDURE

## TITRATION I: STANDARDISATION OF SILVER NITRATE

Pipette out 20 ml of standard sodium or potassium chloride solution into a clean conical flask. Add 1 ml of $5 \%$ $\mathrm{K}_{2} \mathrm{CrO}_{4}$ indicator, as the solution turns yellow in colour. Titrate it against silver nitrate solution taken in the burette. Observe the nearness of the end point through the formation of coagulated silver chloride precipitate at the bottom of the flask. Now add the silver nitrate drop by drop until the solution gets a faint reddish brown tinge. Repeat the titration for concordant value. Let the volume of silver nitrate be $\mathrm{V}_{2} \mathrm{ml}$.

TITRATION - II
Std $\mathrm{AgNO}_{3}$ Vs WATER SAMPLE
Indicator: Potassium Chromate

| S.No. | $\begin{array}{c}\text { Volume of water } \\ \text { sample (ml) }\end{array}$ | Burette Reading |  |  | $\begin{array}{c}\text { Volume of } \\ \mathrm{AgNO}_{3}(\mathrm{ml})\end{array}$ |
| :--- | :---: | :---: | :---: | :---: | :---: |
|  |  |  | Initial | Concordant |  |
|  |  |  |  |  |  |$]$| Final |
| :--- |
|  |

## CALCULATION

Volume of Silver Nitrate
$\left(V_{3}\right)=----------m l$
Strength of Siver Nitrate
$\left(\mathrm{N}_{3}\right)=$ $\qquad$ N

Volume of Water sample
$\left(V_{4}\right)=\cdots------>-$
Strength of Water sample


Amount of Chloride ion present in 1000 ml of the water sample $=$ Equivalent mass of HCl X 1000

$$
\begin{aligned}
& =N_{4} \times 35.46 \times 1000 \\
& =---------------m g / l
\end{aligned}
$$

## TITRATION - II ESTIMATION OF CHLORIDE ION

Pipette out 40 ml of the given water sample into a clean conical flask. To this sample add 1 ml of $5 \% \mathrm{~K}_{2} \mathrm{CrO}_{4}$ indicator and titrate it against the standardized silver nitrate solution taken in the burette and continue the titration until the solution produces a reddish brown colour. Note down the end point and repeat the titration for concordance. Let the volume be $\mathrm{V}_{3} \mathrm{ml}$.

## RESULT

The amount of chloride ion present in the water sample $=---------------\mathrm{mg} / \mathrm{l}$.

## VIVA-VOCE

1. Why is this method known as Argentometric method?
2. What is the chemical reaction between $\mathrm{AgNO}_{3}$ and $\mathrm{Cl}^{-}$ions present in water?
3. What is the form of Chloride ion present in water?
4. What is the reaction between $\mathrm{AgNO}_{3}$ and $\mathrm{K}_{2} \mathrm{CrO}_{4}$ ?
5. What is the name of the method used in this titration?
6. Name the link solution used in the determination.
7. Name the solution used to standardize $\mathrm{AgNO}_{3}$.
8. What is the indicator used in the determination?
9. How will you calculate the amount of chloride ion in the given solution?
10. What is the colour change in the titration?
www.FirstRanker.com

## SHORT PROCEDURE

| CONTENTS | TITRATION I | TITRATION II |
| :--- | :---: | :---: |
| Burette solution | Std Sodium Hydroxide | Std Sodium Hydroxide |
| Pipette solution | 20 ml hydrochloric acid | 20 ml hydrochloric acid |
| Additional solution | 20 ml distilled water | 20 ml distilled water |
| End point | Sudden increase in pH | pH gradual increase |
| Equivalent Weight of $\mathrm{HCl}=36.5$ |  |  |

ROUGH GRAPH


Volume of NaOH
(ml)

FAIR GRAPH


## Expt. No. 4 DETERMINATION OF STRENGTH OF HCI by pH METRY

## AIM

To determine the strength of given HCl by pH metry. A standard solution of 0.1 N NaOH is provided.

## PRINCIPLE

The pH of a solution is related to $\mathrm{H}^{+}$ion concentration by the following formula.

$$
\mathrm{pH}=-\log \left[\mathrm{H}^{+}\right]
$$

pH of a solution is indirectly related to $\mathrm{H}^{+}$ion concentration. When NaOH is added slowly to $\mathrm{HCl}, \mathrm{H}^{+}$ions get neutralized by $\mathrm{OH}^{-}$ions. The pH increases slowly.

$$
\mathrm{H}^{+}+\mathrm{Cl}^{-}+\mathrm{Na}^{+}+\mathrm{OH}^{-} \rightarrow \mathrm{Na}^{+}+\mathrm{Cl}^{-}+\mathrm{H}_{2} \mathrm{O}
$$

When all $\mathrm{H}^{+}$ions of HCl are neutralized at the end point, addition of NaOH causes high increase in pH because of the addition of excess $\mathrm{OH}^{-}$ions.

## MATERIALS REQUIRED

pH meter, Glass electrode, Burette, Pipette, Std. NaOH , approximately $\mathrm{N} / 10 \mathrm{HCl}$.

## PROCEDURE

The burette is washed with distilled water, rinsed with the given std. Sodium hydroxide and filled with the same solution. Exactly 10 ml of the given HCl solution is pipetted out into a clean beaker. It is then diluted to 50 ml with distilled water. A glass electrode is dipped into the solution and its terminals are connected to a pH meter.

## TITRATION - I

| S.No | Volume of <br> $\mathrm{NaOH}(\mathrm{ml})$ | pH |
| :--- | :---: | :---: |
| 1 |  |  |
| 2 |  |  |
| 3 |  |  |
| 4 |  |  |
| 5 |  |  |
| 6 |  |  |
| 7 |  |  |
| 8 |  |  |
| 9 |  |  |
| 10 |  |  |


| S.No | Volume of <br> $\mathrm{NaOH}(\mathrm{ml})$ | pH |
| :--- | :---: | :---: |
| 11 |  |  |
| 12 |  |  |
| 13 |  |  |
| 14 |  |  |
| 15 |  |  |
| 16 |  |  |
| 17 |  |  |
| 18 |  |  |
| 19 |  |  |
| 20 |  |  |

## TITRATION - II

| $\mathrm{S} . \mathrm{No}$ | Volume of <br> $\mathrm{NaOH}(\mathrm{ml})$ | pH | $\Delta \mathrm{pH}$ | $\Delta \mathrm{V}$ | $\Delta \mathrm{pH} / \Delta \mathrm{V}$ |
| :--- | :---: | :---: | :---: | :---: | :---: |
| 1. |  |  |  |  |  |
| 2. |  |  |  |  |  |
| 3. |  |  |  |  |  |
| 4. |  |  |  |  |  |
| 5. |  |  |  |  |  |
| 6. |  |  |  |  |  |
| 7. |  |  |  |  |  |
| 8. |  |  |  |  |  |
| 9. |  |  |  |  |  |
| 10. |  |  |  |  |  |

## CALCULATION



Now NaOH is gradually added from the burette to HCl taken in the beaker. pH of the solution is noted for each addition of NaOH . This process is continued until 5 readings are taken after the end point.

A fair titration is performed to find the exact end point.

## RESULT

$\begin{array}{ll}\text { (i) Strength of the given } \mathrm{HCl} \text { solution } & =--------------\mathrm{N} \\ \text { (ii) Amount of } \mathrm{HCl} \text { present in a litre of the solution }=-----------\mathrm{g}\end{array}$

## VIVA-VOCE

1. What is meant by pH ?
2. What is the name of the reference electrode used?
3. Why does pH value increase suddenly at the end point?
4. What is the indicator used in the titration?
5. What is the pH range of human blood?
6. What is the unit of pH ?
7. How will you calculate the amount of HCl ?
8. How is glass electrode represented?
9. Why is glass electrode known as ion selective electrode?
10. What is the chemical reaction involved in this titration?

## SHORT PROCEDURE

| CONTENTS | TITRATION |
| :---: | :---: |
| Burette solution | Std Sodium Hydroxide |
| Pipette solution | 20 ml mixture of acid ( $\mathrm{HCl}+\mathrm{CH}_{3} \mathrm{COOH}$ ) |
| Additional solution | 20 ml distilled water |
| End point | Conductivity decreases-slowly increases-Sudden increase |
| Equivalent Weight of $\mathrm{HCl}=36.5$ | Equivalent Weight of $\mathrm{CH}_{3} \mathrm{COOH}=60$ |

## MODEL GRAPH



# CONDUCTOMETRIC TITRATION OF MIXTURE OF ACIDS 

AIM
To determine the amount of a strong acid and a weak acid present in the given solution.

## PRINCIPLE

Solution of electrolytes conducts electricity due to the presence of ions. Since specific conductance of a solution is proportional to the concentration of ions in it, conductance of the solution is measured during the titration.

$$
\mathrm{HCl}+\mathrm{NaOH} \rightarrow \mathrm{NaCl}+\mathrm{H}_{2} \mathrm{O} \text { (I neutralization) }
$$

$\mathrm{CH}_{3} \mathrm{COOH}+\mathrm{NaOH} \rightarrow \mathrm{CH}_{3} \mathrm{COONa}+\mathrm{H}_{2} \mathrm{O}$ (H) neutralization)

When a solution of HCl is treated with NaOH the fast moving hydrogen ions are progressively replaced by slow moving sodium ions. As a result conductance of the solution decreases. This decrease will take place until the first neutralisation point is reached. Further addition of alkali results in formation of sodium acetate.

Since sodium acetate is stronger than acetic acid conductivity slowly increases until all acetic acid is completely neutralized.(II Neutralisation) This is due to the presence of fast moving $\mathrm{OH}^{-}$ions. Anymore addition of alkali increases the conductance sharply.

## PROCEDURE

The burette is filled with NaOH solution upto zero mark. The given unknown solution (mixture of a weak \& a strong acid) is transferred into a 100 ml standard flask and made upto the mark with distilled water. 20 ml of the made up solution is pipetted out into a clean 100 ml beaker. The solution is diluted to 20 ml using distilled water. A conductivity cell is dipped into the solution and the terminals are connected to a conductivity meter.

## TITRATION

| S.No | Volume of NaOH <br> $(\mathrm{ml})$ | Conductance (mho) |
| :---: | :---: | :--- |
| 1 |  |  |
| 2 |  |  |
| 3 |  |  |
| 4 |  |  |
| 5 |  |  |
| 6 |  |  |
| 7 |  |  |
| 8 |  |  |
| 9 |  |  |
| 10 |  |  |
| 11 |  |  |
| 12 |  |  |
| 13 |  |  |


| S.No | Volume of NaOH <br> $(\mathrm{ml})$ | Conductance (mho) |
| :---: | :---: | :---: |
| 14 |  |  |
| 15 |  |  |
| 16 |  |  |
| 17 |  |  |
| 18 |  |  |
| 19 |  |  |
| 20 |  |  |
| 21 |  |  |
| 22 |  |  |
| 23 |  |  |
| 24 |  |  |
| 25 |  |  |
| 26 |  |  |

## CALCULATION OF STRENGTH OF HCl

| Volume of NaOH | $\left(\mathrm{V}_{1}\right)$ | $=\cdots$ |
| :--- | :--- | :--- |
| Strength of NaOH | $\left(\mathrm{N}_{1}\right)$ | $=\cdots-\cdots$ |
| Volume of HCl | $\left(\mathrm{V}_{2}\right)$ | $=-\cdots$ |



## CALCULATION OF STRENGTH OF $\mathrm{CH}_{3} \mathrm{COOH}$

| Volume of NaOH | ( $\mathrm{V}_{1}$ ) | $=$ | ------------ ml (titre value) |
| :---: | :---: | :---: | :---: |
| Strength of NaOH | $\left(\mathrm{N}_{1}\right)$ | = | ------------ N |
| Volume of $\mathrm{CH}_{3} \mathrm{COOH}$ | $\left(\mathrm{V}_{2}\right)$ |  | ------------ ml |
| Strength of $\mathrm{CH}_{3} \mathrm{COOH}$ | $\left(\mathrm{N}_{2}\right)$ | $=$ | ------------ N |
|  | $\mathrm{N}_{2}$ |  | / $\mathrm{V}_{2}$ |

Strength of $\mathrm{CH}_{3} \mathrm{COOH}$
$=-----------N$

The burette solution is added to the unknown solution in the beaker in 0.5 ml increments, the solution is stirred using a glass rod, and the observed conductance values are read from the meter. The conductance values show decrease in the initial values, then gradually increases and finally shows a steep increase. The titration hence shows two end-points (i.e) I neutralization (weak acid) \& II neutralization (strong acid).

The accurate end-point is obtained by plotting a graph between observed conductance Vs volume of NaOH added.

RESULT
The strength of acids present in the given unknown solution are:
a) Strong acid
= --------------- $N$
b) Weak acid
= -----------------N

## VIVA-VOCE

1. What is weak acid?
2. How many end points are obtained from the graph? Comment two end points.
3. Why is the conductance increase in the first end point?
4. What mixture of acids used in this titration?
5. Write the neutralization reaction between $\mathrm{CH}_{3} \mathrm{COOH}$ and NaOH .
6. What is the solution present in the conductivity cell?
7. What are strong and weak electrolytes?
8. Give an example for strong acid and weak acid.
9. What is the strong electrolyte of weak electrolyte?
10. Give an example for strong base.

## SHORT PROCEDURE

| CONTENTS | TITRATION I | TITRATION II |
| :---: | :---: | :---: |
| Burette solution | Std Potassium dichromate | Std Potassium dichromate |
| Pipette solution | 10 ml of FAS | 10 ml of FAS |
| Additional <br> solution | 20 ml distilled water+ <br> 10 ml of dil $\mathrm{H}_{2} \mathrm{SO}_{4}$ | 20 ml distilled water+ <br> 10 ml of dil $\mathrm{H}_{2} \mathrm{SO}_{4}$ |
| End point | Sudden increase in EMF | EMF increases and decreases |
| Equivalent Weight of Ferrous ion $=55.85$ |  |  |

## ROUGH GRAPH



## Expt. No. 6 ESTIMATION OF FERROUS ION BY POTENTIOMETRIC TITRATION

## AIM

To estimate the amount of ferrous ion present in whole of the given solution potentiometrically. A standard solution of potassium dichromate of strength ----------N is provided.

## PRINCIPLE

Potentiometric titrations depend on measurement of emf between reference electrode and an indicator electrode. When a solution of ferrous iron is titrated with a solution of potassium dichromate, the following redox reaction takes place.

$$
6 \mathrm{Fe}^{2+}+\mathrm{Cr}_{2} \mathrm{O}_{7}^{2-}+14 \mathrm{H}^{+} \rightarrow 6 \mathrm{Fe}^{3+}+2 \mathrm{Cr}^{3+}+7 \mathrm{H}_{2} \mathrm{O}
$$

During this titration $\mathrm{Fe}^{2+}$ is converted in to $\mathrm{Fe}^{3+}$, whose concentration increases. At the end point, there will be a sharp change due to sudden removal of all $\mathrm{Fe}^{2+}$ ions.

The cell is set up by connecting this redox electrode with a calomel electrode as shown below:

$$
\mathrm{Pt}, \mathrm{Fe}^{2+}, \mathrm{Fe}^{3+}, I I \mathrm{KCl}, \mathrm{HgCl}_{2(\mathrm{~s})}, \mathrm{Hg}
$$

A graph between EMF measured against the volume of potassium dichromate added is drawn and the end point is noted from the graph.

## PROCEDURE

10 ml of Ferrous solution is pipetted out into a 100 ml beaker. 10 ml of dil. $\mathrm{H}_{2} \mathrm{SO}_{4}$ and 20 ml of distilled water are added. A platinum electrode and a calomel electrode are dipped into this solution and connected to the potentiometer. Then 1 ml of potassium dichromate is added to the solution and stirred well for 30 seconds. The EMF is measured and the titration is continued by adding potassium dichromate in 1 ml increments till five measurements after the end point.

## ROUGH TITRATION

| S.No. | Volume of dichromate (ml) |  |
| :--- | :--- | :--- |
| 1. |  |  |
| 2. |  |  |
| 3. |  |  |
| 4. |  |  |
| 5. |  |  |
| 6. |  |  |
| 7. |  |  |
| 8. |  |  |
| 9. |  |  |
| 10. |  |  |
| 11. |  |  |
| 12. |  |  |
| 13. |  |  |
| 14. |  |  |
| 15. |  |  |
| 16. |  |  |
| 19. |  |  |
| 20. |  |  |
| 21. |  |  |
| 10. |  |  |

A graph is drawn by plotting the emf against the volume of potassium dichromate and the end point range is fixed.

About 20 ml of Ferrous solution is pipetted out and the titration is continued by adding 0.1 ml increaments of potassium dichromate in the end point range. The emf is measured for each 0.1 ml after stirring the solution well.

A graph is plotted between emf and the volume of potassium dichromate and also a first derivative graph is plotted ( $\mathbb{E} / \Delta$ against vol. of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ ). The strength of Ferrous solution and the amount of Ferrous ion present are calculated from the end point.

## FAIR TITRATION

| S.No | Volume of <br> Dichromate (ml) | Emf (mV) | $\Delta \mathrm{E}$ | $\Delta \mathrm{V}$ | $\Delta \mathrm{E} / \Delta \mathrm{V}$ |
| :--- | :--- | :--- | :--- | :--- | :--- |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

## CALCULATION

| Volume of Potassim dichromate | ( $\mathrm{V}_{1}$ ) | = | ----- ml |
| :---: | :---: | :---: | :---: |
| Normality of Potassium dichromate | $\left(N_{1}\right)$ |  | -- N |
| Volume of Ferrous sulphate | $\left(\mathrm{V}_{2}\right)$ |  | - ml |
| Normality of Ferrous sulphate | $\left(\mathrm{N}_{2}\right)$ |  | ---------- N |

Amount of Ferrous ion present in 1000 ml of the solution $=$ Normality X Equivalent weight of ferrous ion
= $\qquad$

## RESULT

The amount of Ferrous ion present in the whole of the given solution
$=$
g.

## VIVA-VOCE

1. What is EMF?
2. What is the basic principle of potentiometric titration?
3. Why does EMF increase during the titration?
4. Write the cell representation of the tifration.
5. What is redox reaction?
6. What is the purpose of adding sulphuric acid?
7. What are the advantages of potentiometric titration?
8. How will you obtain the end point in the titration?
9. What is the name of the indicator electrode used in this titration?
10. What is the name of the reference electrode used?

## SHORT PROCEDURE

| CONTENTS |  |
| :--- | :--- |
| Burette solution | Sodium Hydroxide |
| Pipette solution | 20 ml HCl |
| Additional solution | 20 ml distilled water |
| End point | Decrease in conductance- increase in conductance |
| Equivalent Weight of $\mathrm{HCl}=36.5$ |  |



## Expt.No. 7 CONDUCTOMETRIC TITRATION OF STRONG ACID WITH STRONG BASE

AIM

To determine the strength of a strong acid by titrating a strong acid and strong base conductometrically. A standard solution of NaOH of known strength ---------N is provided.

## PRINCIPLE

Solution of electrolytes conducts electricity due to the presence of ions. The specific conductance of a solution is proportional to the concentration of ions in it. The reaction between HCl and NaOH may be represented as,

$$
\mathrm{HCl}+\mathrm{NaOH} \rightarrow \mathrm{NaCl}+\mathrm{H}_{2} \mathrm{O}
$$

When a solution of hydrochloric acid is titrated with NaOH , the fast moving hydrogen ions are progressively replaced by slow moving sodium ions. As a result conductance of the solution decreases, this decrease in conductance will take place until the end point is reached. Further addition of alkali raises the conductance sharply, as there is an excess of hydroxide ions.

A graph is drawn between volume of NaOH added and the conductance of solution. The exact end point is intersection of the two lines.

## PROCEDURE

The given HCl is made up in a 100 ml standard flask. 10 ml of this solution is pipetted out in a beaker. The burette is filled with standard sodium hydroxide upto the mark.

A conductivity cell is dipped into the beaker with HCl solution and connected to the terminals of the conductivity bridge. About 1 ml of STD NaOH is added and stirred well for 30 seconds. The conductance is measured and the titration is continued till five measurements after the endpoint. A graph is plotted for the conductance values against the volume of NaOH and the endpoint range is fixed.

## TABLE

| S.NO | Volume of NaOH <br> $(\mathrm{ml})$ | Conductance <br> $(\mathrm{mho})$ |
| :---: | :---: | :---: |
| 1 |  |  |
| 2 |  |  |
| 3 |  |  |
| 4 |  |  |
| 5 |  |  |
| 6 |  |  |
| 7 |  |  |
| 8 |  |  |
| 9 |  |  |
| 10 |  |  |
| 11 |  |  |
| 12 |  |  |
| 13 |  |  |
| 14 |  |  |
| 15 |  |  |
| 16 |  |  |
| 18 |  |  |
| 19 |  |  |
| 20 |  |  |
| 21 |  |  |
| 22 |  |  |
| 24 |  |  |
| 25 |  |  |

## CALCULATION

| Volume of Sodium Hydroxide | $\left(\mathrm{V}_{1}\right)$ |  |  |
| :---: | :---: | :---: | :---: |
| Normality of Sodium Hydroxide | $\left(\mathrm{N}_{1}\right)$ |  |  |
| Volume of Strong acid | $\left(\mathrm{V}_{2}\right)$ | $=$ |  |
| Normality of Strong acid | $\left(\mathrm{N}_{2}\right)$ | = | $\mathrm{V}_{1} \mathrm{~N}_{1}$ |
|  |  |  | $\mathrm{V}_{2}$ |

The strength of the given strong acid
= ------------- N

The above titration is repeated again and the exact endpoint is found out by adding in increments of 0.1 ml of NaOH in the end point range and continued after endpoint upto five readings.

A graph is plotted with the conductance values against the volume of NaOH and then the strength of the strong acid is found out.

## RESULT

The strength of the given strong acid is found to be = $\qquad$ N

## VIVA-VOCE

1. What is the basic principle of conductometric titration?
2. Why is conductance decreased and increased during the titration?
3. What is conductance mention its unit?
4. Give some examples for Strong acids and Strong bases.
5. Write the reaction between HCl and NaOH .
6. What is meant by strong base?
7. Explain why HCl is a strong acid?
8. Explain why the strength of burette solution is always greater than the pipette solution in conductometric titration.
9. What is the equivalent weight of HCl and NaOH ?
10. Write the formula that is used to calculate the amount of HCl .

## ESTIMATION OF COPPER

Alloy solution Vs Std. $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ (Thio)

| S.No. | Volume of <br> Alloy Solution <br> $(\mathrm{ml})$ | Burette Reading (ml) |  | Volume of <br> Thio <br> $(\mathrm{ml})$ | Concordant value <br> $(\mathrm{ml})$ |
| :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  |  |  |
| 2. |  |  |  |  |  |
| 2. |  |  |  |  |  |
| 3. |  |  |  |  |  |

## CALCULATION



## Expt.No. 8 ESTIMATION OF COPPER IN BRASS

AIM
To estimate the amount of copper present in the given sample of brass.

## PRINCIPLE

Brass is an alloy with $55 \%$ copper and $33 \%$ zinc and small amount of lead and aluminum. Brass is dissolved in conc. nitric acid to convert copper present in the alloy to cupric ions. Then copper is estimated iodometrically by titrating the iodine that was liberated against standard sodium thio sulphate solution using starch as indicator.

$$
\begin{aligned}
& 2 \mathrm{Cu}^{2+}+4 \mathrm{I}^{-} \rightarrow 2 \mathrm{CuI}+\mathrm{I}_{2} \\
& \mathrm{I}_{2}+2 \mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3} \rightarrow \mathrm{Na}_{2} \mathrm{~S}_{4} \mathrm{O}_{6}+2 \mathrm{NaI}
\end{aligned}
$$

## CHEMICALS REQUIRED

0.1 N Potassium dichromate, 0.1 N sodium thio sulphate, 0.01 M EDTA, Con. $\mathrm{HNO}_{3}, \mathrm{KI}, \mathrm{EBT}$, $\mathrm{NH}_{3} \mathrm{OH} / \mathrm{NH}_{4} \mathrm{Cl}$ buffer, Starch.

## PROCEDURE <br> PREPARATION OF BRASS ALLOY SOLUTION

About 0.2 g of the sample of brass was weighed accurately and transferred into a clean dry 250 ml beaker. To this about 10 ml of con. Nitric acid was added and allowed to boil for a small interval of time till the alloy was dissolved. This was cooled, diluted with water and made upto 250 ml in a standard flask.

## TITRATION I STANDARDIZATION OF $\mathrm{Na}_{2} \mathrm{~S}_{2} \mathrm{O}_{3}$ WITH $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ SOLUTION

20 ml of the standard potassium dichromate solution is pipetted out into a conical flask. 20 ml of dil.sulphuric acid and 10 ml of $10 \% \mathrm{KI}$ are added. The liberated iodine is titrated against sodium thio sulphate taken in the burette till the colour changes to yellowish green. 1 ml of starch is added till the colour changes from blue to light green colour. Titration is repeated for concordance.

## CALCULATION



## TITRATION II

## ESTIMATION OF COPPER

The brass solution is transferred into the 100 ml standard flask and 10 ml of dil. sulphuric acid is added and made up to the mark using distilled water.

20 ml of the alloy solutionis pipetted out into a conical flask. To this aqueous ammonia is added in drops till a blue precipitate of $\mathrm{Cu}(\mathrm{OH})_{2}$ is formed. One or two drops of acetic acid is added to dissolve the precipitate. To this 10 ml of $10 \% \mathrm{KI}$ is added and titrated against standard thio sulphate using starch as indicator. The end point is the disappearance of blue colour and appearance of green colour.

## RESULT

The given sample of brass was found to contain copper = \%

## VIVA-VOCE

1. What is the composition of brass?
2. What is meant by iodometry?
3. What is the principle used in $t$ his method?
4. What is the role of $\mathrm{K}_{2} \mathrm{Cr}_{2} \mathrm{O}_{7}$ ?
5. What is the colour change in this titration?
6. Write the chemical reactions involved in this experiments?
7. How will you find out $\%$ of copper in brass?
8. What is the role of indicator?
9. What is the equivalent weight of copper?
10. What is the role of adding aqueous ammonia?

## SHORT PROCEDURE

Standard solutions are prepared by the dilution of the given stock solution. The flasks are numbered and the solutions are prepared according to the following table using different glass pipettes.

| Flask number | $\mathbf{1}$ | $\mathbf{2}$ | $\mathbf{3}$ | $\mathbf{4}$ | $\mathbf{5}$ | $\mathbf{6}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| Vol. of pipette to use | 1 | 1 | 2 | 10 | 10 | 10 |
| Vol. of Na stock solution to pipette | 0.5 | 1 | 2 | 4 | 6 | 8 |
| Conc. of solution obtained ( $\mu \mathrm{g} / \mathrm{ml}$ ) | 5 | 10 | 10 | 40 | 60 | 80 |

MODEL GRAPH

ABSORBANCE


CONCENTRATION

## Expt. No 9 ESTIMATION OF SODIUM BY USING FLAME PHOTOMETER

## AIM

To estimate the amount of sodium present in whole of the given solution.

## APPARATUS REQUIRED

1. Flame photometer FLAPHO or Eppendorf.
2. Stock solutions of $\mathrm{Na}+$ and $\mathrm{K}+, \mathrm{C}=1 \mathrm{mg} / \mathrm{ml}$.
3.6 numbered 100 ml volumetric flasks.
3. Glass pipettes: $1,2,10 \mathrm{ml}$.

## MEASUREMENT PROCEDURE

1. The flame photo meter is switched on.
2. The instrument is kept for warming.
3. Distilled water is fed into the instrument.
4. The element Na is selected by turning the selector "Elementwahl".
5. The outer knob "Messbereich" is turned into position "100". The "Kompensaton l" knob is pulled slightly out and adjusted to 0 . The "Kompensation l"knob is pressed back. 0 reading is adjusted with "Kompensation II" if necessary.
6. The most concentrated standard solution (solution number 6) is aspirated and is adjusted to 350 (on uppermost scale) using inner "Messbereich" knob.
7. Distilled water is aspirated till instrument reads 0 .
8. The standard solutions no. 1, 2, 3, test solution, and then standards 4,5,6 are aspirated.

The results are recorded.
8. Distilled water is aspirated for at least 5 minutes to clean the system(FLAPHO flame photometer). (FLAPHO is a dual channel instrument, which measures concentrations of Na simultaneously).

## TABULATION

| Flask No. | Concentration | Absorbance |
| :---: | :---: | :---: |
| 1 |  |  |
| 2 |  |  |
| 3 |  |  |
| 4 |  |  |
| 6 |  |  |
| Unknown Solution |  |  |

## CALCULATION

1. The calibration curves are drawn for sodium and potassium on a sheet of millimeterpaper. The concentrations are used as abscissa and instrument readouts as ordinate values.
2. The concentration of sodium and potassium ions in test solution are found from calibration curves.

## RESULT

The amount of sodium present in whole of the given solution $=$

## VIVA - VOCE

1. What is meant by flame photometry?
2. How will you prepare blank solution?
3. Define -Absorbance
4. State Beer Ł_amberts law.
5. What is the absorbance of sodium?
6. How will you find out unknown concentration of solution?
7. What is meant by wavelength?
8. How will you calibrate the instrument?
9. What is meant by flame emission spectroscopy?
10. Distinguish between absorption and emission spectrum.

## LIST OF PROJECTS

1. Photo catalysis and water purification and electricity generation
2. The effect of acid rain on marigold plants
3. Using microorganisms (green algae) to bioremediate a source of waste water.
4. The effect of water hardness on the germination of marigold seeds
5. Turn milk into plastic!
6. Find out which household organic waste produce more biogas
7. What makes food decay faster?
8. Obesity: how greasy are your potato chips?
9. The effects of ph level on apple juice fermentation
10. Why does organic milk last so much longer than regular milk? [
11. How sugar content changes in ripening fruit
12. Do oranges lose or gain vitamin c after being picked?
13. Measuring glucose in your food
14. Determine if turmeric can prevent the growth of bacteria which cause foodborne illnesses.
15. What is the effect of antioxidants (vitamins a, e, c) on cells?
16. Will antioxidant supplements help you live longer?
17. Can cloning make a better plant?
18. Biotechnology in the food industry
19. Build a simple burglar alarm
20. Build and demonstrate a homemade fuel cell.
21. Demonstrate the energy stored inside a peanut
22. Increasing the useful lifetime of solar panels
23. The effect of fertilizers on algal growth
24. Determine the effects of neem tree extracts as antibacterial and antifungal agents as well as a pesticidal agent.
25. Compare short term memory according to gender and age.
26. Which recipe will create the biggest bubbles?
27. Do twins have identical or similar fingerprints?
28. How do plants grow in different environments?
29. Reusable water-powered battery
30. Build a solar toy car and explain its operation.
31. Demonstrate the greenhouse effect in a jar.
32. Study the toxicity of energy drinks.
33. Formulate a non-toxic or biodegradable ink.
34. Compare fruits for their sugar content.
35. Using solar energy to purify polluted or salt water
36. Test for the amylase enzyme in your saliva
37. The effects fat has on freezing milk
38. How vehicles affect the ph properties of snow
39. Is radiation level elevated nearby our school?
40. Examines the levels of harmful bacteria that grow in baby food after it has been opened and used in both refrigerated and room temperature environments.
