## INSTITUTE OF AERONAUTICAL ENGINEERING

Dundigal, Hyderabad -500 043.

## FRESHMAN ENGINEERING

# ENGINEERING CHEMISTRY

# LAB MANUAL

2000

IARE

EDUCATION FOR LIBERATION

# INSTITUTE OF AERONAUTICAL ENGINEERING

(Affiliated to JNTU Hyderabad and Approved by AICTE)
Dundigal (Village), Quthbullapur (Mandal), Hyderabad - 43, A.P., INDIA.
Ph.: 08418-257181/82 FAX: 08418-257202

ICS
ISO 9001:2000
Reg. No.: RQ91/4199

**ISO 9001 : 2000 Certified College**

JAS-ANZ

## CERTIFICATE

*This is to Certify that it is a bonafied record of Practical work done by*

*Sri / Kum.* ________________________________ *bearing*

*the Regd. No.* ____________ *of* ____________ *Class* ________________

*branch in the* ______________________________ *laboratory during the*

*Academic year* ____________________ *under our supervision.*

Signature of Head of the Dept.

Signature of Lecture In-charge

Signature of External Examiner

Signature of Internal Examiner

## Chemistry Lab Do’s and Don’ts

The chemistry laboratory must be a safe place in which to work and learn about chemistry. Most of these involve just using common sense.

1. Wear chemical splash goggles at all times while you are in the laboratory.
2. Wear a chemical-resistant apron.
3. Be familiar with your lab assignment **before** you come to lab. Follow all written and verbal instructions carefully. Observe the safety alerts in the laboratory directions. If you do not understand a direction or part of a procedure, ask the teacher before proceeding.
4. When entering the lab/classroom, do not touch any equipment, chemicals, or other materials without being instructed to do so. Perform only those experiments authorized by the instructor.
5. No student may work in the laboratory without an instructor present. Work only with your lab partner(s). Do not venture to other lab stations for any reason.
6. Do not wear bulky or dangling clothing.
7. Never eat or drink in the laboratory. Don't chew on the end of a pen which was lying on the lab bench.
8. Wash acid, base, or any chemical spill off of yourself immediately with large amounts of water. Notify your teacher of the spill.
9. Clean up spills immediately. If you spill a very reactive substance such as an acid or base, notify the people in the area and then obtain assistance from your teacher. Acid spills should be neutralized with baking soda, base spills with vinegar before cleaning them up.
10. If chemical substances get in your eye, wash the eye out for 15 minutes. Hold your eye open with your fingers while washing it out.
11. If you take more of a chemical substance from a container than you need, you should not return the excess to the container. This might cause contamination of the substance remaining. Dispose of the excess as your teacher directs.
12. When weighing never place chemicals directly on the balance pan. **Never weigh a hot object**.
13. Never smell anything in the laboratory unless your teacher tells you it is safe. Do not smell a substance by putting your nose directly over the container and inhaling. Instead, waft the vapors toward your nose by gently fanning the vapors toward yourself.
14. Do not directly touch any chemical with your hands. Never taste materials in the laboratory.
15. If you burn yourself on a hot object, immediately hold the burned area under cold water for 15 minutes. Inform your teacher.
16. Observe good housekeeping practices. Work areas should be kept clean and tidy at all times. Only lab notebooks or lab handouts should be out on the table while performing an Experiment. Books and book bags should not be on the lab table. Passageways need to be clear at all times.
17. Always replace lids or caps on bottles and jars.
18. If your Bunsen burner goes out, turn the gas off immediately.
19. Constantly move a test tube when heating it. Never heat a test tube that is not labeled Pyrex and never point the open end at anyone.
20. Always add acid to water and stir the solution while adding the acid. Never add water to an acid.
21. Report all accidents to your teacher.
22. Absolutely no running, practical jokes, or horseplay is allowed in the Laboratory.
23. Thoroughly clean your laboratory work space at the end of the laboratory Session. Make sure that all equipment is clean, and returned to its original place.

## Importance of Engineering Chemistry Laboratory:

The experiments to be performed in the Engineering Chemistry Laboratory as per the curriculum of JNTU-H regulations are very useful to the students of I B.Tech of different disciplines. The various experiments included in the syllabus are the basics of Engineering Chemistry that are useful for different industries. To mention, Complexometric titrations to find Hardness of water, Colorimetric estimations to find the amount of iron in the cement, understanding the basic concepts of Electrochemistry like Conductometry and Potentiometry, importantly preparation of a pain killer Aspirin, preparation of a industrially useful polymer (Thiokol) rubber . In all the above experiments, the students would learn about handling of the equipment and conceptualization of certain fundamental aspects of Chemistry.

## JAWAHARLAL NEHRU TECHNOLOGICAL UNIVERSITY HYDERABAD

| **I Year B.Tech.** | **T/P/D C** |
|---|---|
| | **- -/3/- 4** |

## ENGINEERING CHEMISTRY LAB

List of Experiments (Any 12 of the following):

**Titrimetry:**

1. Estimation of ferrous iron by dichrometry.
2. Estimation of hardness of water by EDTA method.

**Mineral analysis:**

3. Determination of percentage of copper in brass.
4. Estimation of manganese dioxide in pyrolusite.

**Instrumental Methods:**
**Colorimetry:**

5. Determination of ferrous iron in cement by colorimetric method
6. Estimation of copper by colorimetric method.

**Conductometry:**

7. Conductometric titration of strong acid vs strong base.
8. Conductometric titration of mixture of acids vs strong base.

**Potentiometry:**

9. Titration of strong acd vs strong base by potentiometry.
10. Titration of weak acid vs strong base by potentiometry.

**Physical properties:**

11. Determination of viscosity of sample oil by redwood / Oswald’s viscometer.
12. Determination of Surface tension of lubricants.

**Preparations:**

13. Preparation of Aspirin
14. Preparation of Thiokol rubber

**Adsorption:**

15. Adsorption of acetic acid on charcoal.

**TEXT BOOKS:**

1. Practical Engineering Chemistry by K. Mukkanti, etal, B.S. Publications, Hyderabad.

# INDEX

| S. No | NAME OF THE EXPERIMENT | DATE | REMARKS |
|---|---|---|---|
| 1 | | | |
| 2 | | | |
| 3 | | | |
| 4 | | | |
| 5 | | | |
| 6 | | | |
| 7 | | | |
| 8 | | | |
| 9 | | | |
| 10 | | | |
| 11 | | | |
| 12 | | | |

# PREPARATION OF ASPIRIN

## AIM:

To prepare Aspirin from salicylic acid.

## APPARATUS:

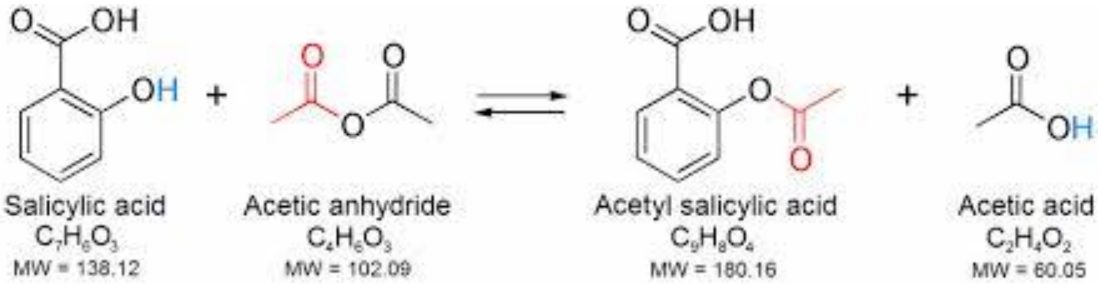
Conical flask, Beakers, glass rod, funnel etc.

## CHEMICALS REQUIRED:

1. Salicylic acid - 2.5gm (MW= 138)
2. Acetic anhydride - 3.5ml
3. Con.$H_2SO_4$ - 3-4drops

## PRINCIPLE:

Salicylic acid is a phenolic acid. The phenolic group can be easily acetylated using acetic anhydride. This is an example of Nucleophilic substitution reaction. Phenolic hydroxyl group of salicylic acid acts as a Nucleophile and lone pair of electrons on the Oxygen atom attacks the carbonyl group of acetic anhydride to form Aspirin.



## PROCEDURE:

Take all the three chemical compounds in a given 250ml conical flask. Shake the mixture thoroughly and warm the reaction mixture at 50-60°C for 15 min. on a water bath with continuous stirring with glass rod and allow the reaction mixture to cool. Add nearly 100ml of distilled water. Stir thoroughly and filter the product.

## RECRYSTALIZATION:

Dissolve the product in 20ml alcohol and pour the solution into warm water, If solids separate warm it to dissolve the solids and clear the solution and allow it to cool slowly to get beautiful needles of Aspirin(MW=180) is formed.

## RESULT:

Percentage yield of product ______%

# PREPARATION OF THIOKOL RUBBER

## AIM:

To synthesize Thiokol rubber using sodium polysulphide with 1, 2-Dichloroethane.

## APPARATUS:

Beakers, glass rod, funnel etc.

## CHEMICALS REQUIRED:

1. Sodium hydroxide
2. Powered sulphur
3. 1, 2-Dichloroethane
4. 5% $H_2SO_4$, etc

## THEORY:

It is a rubbery white substance and is obtained by treating sodium polysulphide with 1, 2-Dichloroethane.

$$S_8 + 2\ NaOH \rightarrow Na_2S_8$$

$$n(Cl\text{-}CH_2\text{-}CH_2\text{-}Cl) + nNa_2S_8 \rightarrow [\text{-}CH_2\text{-}CH_2\text{-}S\text{-}S\text{-}]_n + 2n\ NaCl$$

## PROCEDURE:

1. In a 100ml beaker dissolve 2gms NaOH in 50-60 ml of water.
2. Boil the solution and to this add in small lots with stirring 4 gms of powdered sulphur. During addition and stirring, the yellow solution turns deep red.
3. Cool it to 60-70 $^0$C and add 10ml of 1, 2-Dichloroethane with stirring. Stir for an additional period of 20 min White rubber polymer separated out as lump.
4. Pour out the liquid from the beaker in the sink to obtain Thiokol rubber. Wash under the tap
5. Dry in the fold of filter paper, the yield is about 1.5 gms. Determine the solubility of the polymer in Benzene, Acetone, 5% $H_2SO_4$ and $HNO_3$ etc.

## RESULT:

Yield obtained = _________ gms

# DETERMINATION OF VISCOSITY OF LIQUIDS BY OSWALD'S VISCOMETER

## AIM:

To determine the absolute viscosity of a liquid by using Oswald’s viscometer.

## APPARATUS:

Oswald’s viscometer, stop watch, density bottle, rubber bulbs, Beakers, etc.

## CHEMICALS REQUIRED:

Standard liquid (water), test liquid etc.

## PRINCIPLE: (POISEUILLE'S PRINCIPLE)

If a liquid flows with in a uniform velocity at a rate of 'V'in 't' seconds through a capillary tube of radius 'r' and length 1cm under a driving pressure 'p' dynes/ $cm^2$. Then,

The co-efficient of viscosity is given as =

$$\boxed{\eta = \pi r^4 t \Delta P / 8VL}$$

$\eta \Rightarrow$ Viscosity of liquid in poise
$\Delta P \Rightarrow$ pressure head i.e. dynes/ $cm^2$.
r $\Rightarrow$ radius of inner layer of capillary tube
L $\Rightarrow$ length of capillary tube
V $\Rightarrow$ volume of capillary tube
t $\Rightarrow$ flow time in seconds

The poiseulles law is applicable only to linear flow or stream line flow. For a given Oswald’s viscometer the length, radius and volume of liquids are constants and at end are combined to a single constant. The above equation can be written as

$\eta = k t \Delta P$ In this eq. $\Delta P$ depends on

1. Density of liquid to be measured
2. Acceleration due to gravity
3. the difference due to gravity is constant Then

The viscosity of liquid may be expressed as
$\eta_1 = kt_1 \rho_1$ (viscosity Standard liquid (water))
$\eta_2 = kt_2 \rho_2$(viscosity of test liquid)
Relative viscosity $\Rightarrow \eta_1/\eta_2 = t_1 \rho_1 / t_2 \rho_2$
Units: (CGS) $\Rightarrow$ dynes-sec/$cm^2$ or poise

## PROCEDURE:

Clean thoroughly and dry the Oswald’s viscometer, a definite volume of standard liquid is allow to flow into 'A ' arm such that it raises above the values X and Y. The same procedure is repeated with the test liquid and note the time by stop clock

## CALCULATIONS:

| S. No | Standard liquid ($t_1$) | Test liquid ($t_2$) |
|---|---|---|
| TRIAL-I | | |
| TRIAL-II | | |
| TRIAL-III | | |

Weight of empty density bottle ($W_1$) = _________ gm

Weight of empty bottle + water ($W_2$) = _________ gm

Weight of empty bottle +liquid ($W_3$) = _________ gm

Density of water ($\rho_{1}$) = _______

Density of liquid ($\rho_2$) = _______

Viscosity Standard liquid ($\eta_1$) at 25°C= 1.0019cps

$\eta_2 = t_2 \rho_2 / t_1 \rho_1 \times \eta_1$

## RESULT:

Absolute viscosity of a given liquid ($\eta_2$) = ________ cps

# DETERMINATION OF SURFACE TENTION OF LIQUIDS

## AIM:

To determine surface tension of liquids by using stalagmometer.

## APPARATUS:

**S**talagmometer, Density bottle, Thermometer, Beakers, Burette Stand etc.

## CHEMICALS REQUIRED:

Distilled water, Acetone (or) Acetic acid etc.

## PRINCIPLE:

The force in dynes acting on a surface at right angles at any line of unit length is called Surface tension.

$$\text{Surface tension } (\gamma) = F/L$$

## THEORY:

## DROP NUMBER METHOD:

It is the simplest method for determination of surface tension of liquids in the laboratory. This method is based on the principle that a fixed volume (weight of liquid W) of the liquid is delivered as freely falling from Capillary tube held vertically. Surface tension is directly proportional to weight i.e. $W \alpha \gamma$

Circumference of drop = $2\pi r$ is equal to length of the bar .Hence, the equation is represented as

$$\gamma = F/2\pi r$$

$$\gamma = mg/2\pi r$$

If ‘V’ volume contains 'n' drops then weight of a single drop is $\gamma\, 2\pi r = \rho\, vg/n$

If density of two liquids $\rho_{1\text{and}}\, \rho_2$ the no. of drops of two liquids be $n_1$ and $n_2$ of the same volume of liquids from two fixed points respectively. Applying equation and radius of tube is same.

$$\gamma = v\rho g/2\pi r n$$

$$\gamma_1 = v\rho_1 g/2\pi r n_1\text{-----------} (1)$$

$$\gamma_2 = v\rho_2 g/2\pi r n_2\text{-----------} (2)$$

$$\boxed{\text{Formula } \gamma_1 / \gamma_2 = \rho_1 n_2 / \rho_2 n_1}$$

$\rho_1 \Rightarrow$ density of water (0.998 at $25^oC$)

$\rho_1 \Rightarrow$ density of given sample

$\gamma_1 \Rightarrow$ Surface tension of water at room temp. i.e. 72.8 dynes/cm

$\gamma_2 \Rightarrow$ Surface tension of liquid to be determined

$n_1, n_2 \Rightarrow$ no. of drops of water and liquid by stalagmometer.

## PROCEDURE:

a) Thoroughly clean the density bottle and stalagmometer using chromic acid and purified water
b) Stalagmometer must be mounted in vertical plane using burette stand
c) Fill the purified water in the instrument and count the number of drops falling between two points of instrument as shown in the figure.
d) Repeat this step (iii)at least three times
e) Rinse the stalagmometer using the same liquid whose surface tension is to be determined
f) Fill the stalagmometer by liquid and count the number f drops falling down between the two points as in step (iii)
g) Repeat step (v) at least three times
h) Density of water and liquid is determined using density bottles

## CALCULATIONS:

| S. No. | SAMPLE | NUMBER OF DROPS | | | | DENSITY | SURFACE TENSION |
|---|---|---|---|---|---|---|---|
| | | I | II | III | MEAN | | |
| | | | | | | | |
| | | | | | | | |

Weight of empty density bottle ($W_1$) = _________ gm

Weight of empty bottle + water ($W_2$) = _________ gm

Weight of empty bottle +liquid ($W_3$) = _________ gm

Density of water ( $\rho_1$) = _______
Density of liquid ( $\rho_2$) = _______
Specific gravity ( $\rho_2 / \rho_1$) = $W_3$-- $W_1$ / $W_2$--$W_1$

$$\gamma_2 = \rho_2 n_1 \gamma_1 / \rho_1 n_2$$

## RESULT:

Surface tension of given liquid at room temperature = ________ dynes/cm

# ESTIMATION OF HARDNESS OF WATER BY EDTA METHOD

**AIM:**

To estimate the total hardness, permanent hardness and temporary hardness of water by using standard solution of EDTA

**APPARATUS:**

Burette, pipette, Conical flask, Beakers, Standard flask, Burette stand and funnel etc..

**CHEMICALS REQUIRED:**

Buffer sol$^n$, EDTA sol$^n$, Eriochrome black-T, Magnesium sulphate etc.

**PRINCIPLE:**

Hard water which contains $Ca^{2+}$ and $Mg^{2+}$ ions which forms wine red color complex with the indicator

$Ca^{2+}$ (or) $Mg^{2+}$ + EBT ———> Ca-EBT (or) Mg-EBT
(Wine red colour complex)

EDTA forms a colour less complex with the metal ions ($Ca^{2+}$ and $Mg^{2+}$)

Ca-EBT (or) Mg-EBT + EDTA———> Ca-EDTA (or) Mg-EDTA + EBT
(Wine red colour complex) (Colorless stable complex) (Blue)

When free ions are not available, EDTA extracts the metal from (ion) metal ion indicator complex, there by releasing the free indicator.

**PROCEDURE:**

**STEP-I**

**PREPARATION OF STANDARD SOLUTION OF $MgSO_4$:**

Weigh the approx 0.25gm of $MgSO_4$ and transfer into 100ml standard flask through the funnel and dissolve in minimum quantity of distilled water. Make up the solution up to the mark with distilled water and shake the flask well for uniform concentration then calculate the Molarity of $MgSO_4$

M. Wt. of $MgSO_4$ = 246.48gm
Molarity of $MgSO_4$=0.01M

**STEP-II**

**STANDARDISATION OF EDTA SOLUTION:**

Pipette out 20ml of $MgSO_4$ sol. into a clean conical flask. Add 2ml of buffer sol. and add 2 to 3 drops of EBT indicator and it gets wine red color sol. Take EDTA solution in a burette after titrate with EDTA solution till wine red color changes to blue color. Note the burette reading and repeat the titration to get concurrent values.

| S. No | Volume of $MgSO_4$ in ml | Burette Reading | | Volume of EDTA consumed |
|---|---|---|---|---|
| | | Initial | Final | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

$M_1$ = $MgSO_4$ molarity $M_2$ = EDTA molarity
$V_1$ = volume of $MgSO_4$ $V_2$ = volume of EDTA consumed

$$M_1V_1 = M_2V_2$$

## STEP-III

### STANDARDISATION OF HARD WATER:

Pipette out 20ml of tap water into a 250ml conical flask add 2 ml of buffer sol. and add 2-3drops of EBT indicator. Titrate the wine red color sol. with EDTA taken in burette, till a blue color end point is obtained. Repeat the titration to get concurrent values.

| S. No | Volume of Hard water in(ml) | Burette Reading | | Volume of EDTA consumed |
|---|---|---|---|---|
| | | Initial | Final | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

$M_3$ = molarity of hard water  $M_2$ = EDTA molarity
$V_3$= volume of Hard water  $V_2$ = volume of EDTA consumed

$$M_3V_3 = M_2V_2$$

Total hardness= $M_3$X100X1000 = -----------PPM

## STEP-IV

### STANDARDISATION OF PERMANENT HARDNESS OF WATER:

Pipette out 100ml of hard water sample into a beaker containing 250ml and boil the water till volume reduces to 50ml (all the bicarbonates of $Ca^{2+}$, $Mg^{2+}$ decomposes to$CaCO_3$and $Mg(OH)_2$ respectively). Cool the solution and filter the water into beaker then pipette out 20ml of this cool water sample in to 250ml conical flask add 2ml of buffer sol. and 2-3 drops of EBT indicator. Titrate the wine red color solution with EDTA taken in the burette, till a blue colored solution end point is obtained. Repeat the titration to get concurrent values.

| S. No | Volume of Hard water in (ml) | Burette Reading | | Volume of EDTA consumed |
|---|---|---|---|---|
| | | Initial | Final | |
| 1 | 20 | | | |
| 2 | 20 | | | |
| 3 | 20 | | | |

$M_4$ = molarity of hard water  $M_2$ = EDTA molarity
$V_4$ = volume of hard water  $V_2$ = volume of EDTA consumed

$$M_4V_4 = M_2V_2$$

Permenent hardness = $M_4$X100X1000 = --------------PPM

## RESULT:

1) Total Hardness in _______ PPM

2) Permanent Hardness in _______ PPM

3) Temporary hardness in _______ PPM

# ESTIMATION OF MANGANESE DIOXIDE IN PYROLUSITE

## AIM:

To estimate the amount of $MnO_2$ present in the given sample of pyrolusite.

## APPARATUS:

Burette, pipette, conical flask, Standard flask, Burette, stand, Funnel and Beaker etc.

## CHEMICALS REQUIRED:

Sodium Oxalate, Potassium permanganate, Sulphuric acid, pyrolusite sample and $MnO_2$ ....

## PRINCIPLE:

The $MnO_2$ present in the pyrolusite sample is reduced by a known excess of standard Sodium Oxalate solution and the uncreated oxalate is titrated against a standardized solution of $KMnO_4$, where $KMnO_4$ acts as self indicator. Pale pink color is the end point of the titration.
$MnO_2 + Na_2C_2O_4 + 2\ H_2SO_4$ --------> $MnSO_4 + Na_2SO_4 + 2CO_2 + 2H_2O$
(From pyrolusite)
$Na_2C_2O_4 + KMnO_4 + 8H_2SO_4$------->$2MnSO_4 + K_2SO_4 + 5\ Na_2SO_4 + 10CO_2 + 8H_2O$
(Unreacted)

## PROCEDURE:

1) **PREPARATION OF STANDARD SODIUM OXALATE SOLUTION:**
Weigh 0.67 gram of sodium oxalate into a clean 100 ml standard flask, dissolve in small amount of water and make up the solution up to the mark with distilled water. Shake the flask well for uniform concentration.

2) **PREPARATION OF $KMNO_4$ SOLUTION:**
Dissolve 0.79 gram of $KMnO_4$ into a 250 ml standard flask in little distilled water and make up the solution up to the mark with distilled water. Shake the flask well for uniform concentration.

3) **PREPARATION OF 0.1N SULPHURIC ACID:**
Dissolve 0.72 ml of concentrated $H_2SO_4$ slowly drop by drop in 100 ml of distilled water (the acid must be added to water not water to acid)

4) **STANDARDIZATION OF $KMNO_4$ SOLUTION:**
Rinse the burette with $KMnO_4$ and fill the burette with $KMnO_4$ up to zero mark, pipette out 10ml of sodium oxalate into a clean 250 ml of conical flask and add 5 ml of 0.1N $H_2So_4$ and keep the solution till fumes come out of the solution (70-80$^oC$). Titrate the hot solution with $KMn0_4$ till pale pink color is observed in the solution Repeat the solution to get concurrent values. Let the titre value of the sample be X ml.

5) **STANDARDIZATION OF PYROLUSITE SAMPLE:**
Weigh out accurately about 0.20-0.25gm of sample into a 250 ml dry conical flask and exactly 40 ml of sodium oxalate with the help of pipette and 40 ml of 0.1N $H_2SO_4$ solution. Put a small funnel in the neck of the flask and heat the solution on a small flame. Till all the black particles of pyrolusite disappear in the conical flask. Now wash the funnel and the side of the conical flask with little distilled water in to the flask and titrate the unreacted oxalic acid present in hot solution with $KMn0_4$ solution taken in the burette. Pale pink color is the end point of the titration. Let the titre value with unreacted oxalic acid be Y ml.

   **Note:** The titration of unreacted oxalic must be carried out in hot condition i.e. 60-70 $^oC$.

6) **STANDARDIZATION OF BLANK SOLUTION:**
Pipette out 40 ml of sodium oxalate solution into a 250 ml of conical flask and add 50 ml of 0.1N $H_2SO_4$. Keep a small funnel in the neck of the flask and heat to 70-80$^oC$, rinse the funnel and neck of the flask with distilled water and titrate the hot solution with $KMn0_4$ till pale pink colored end point is obtained the titre value z ml indicates the total amount of potassium permanganate required for the complete reaction of the 40 ml of sodium oxalate.

**CALCULATIONS:**

Weight of pyrolusite = ---------- gm

Normality of $KMnO_4$ = 0.1N

Titre value of pyrolusite(Y) = …………ml

Blank titre value (Z) = ………ml

Volume = Blank titre value (Z) – Titre value of pyrolusite(Y) **(Eq. wt. of $MnO_2$ = 43.46gm)**

Amount of $MnO_2$ present in sample= Volume **X** Normality of $KMnO_4$ **X** Eq. wt. of $MnO_2$ / 1000

% of $MnO_2$ present in given sample = Amount of $MnO_2$ X 100 / Wt. of sample

**RESULT:**

The amount of manganese dioxide present in given sample of pyrolusite is --------%

# ESTIMATION OF COPPER BY COLORIMETRIC METHOD

**AIM:**

To estimate the amount of copper in the given solution by colorimetric method

**APPARATUS:**

Colorimeter, Standard volumetric flask, Test tubes, Measuring jar, Cuvettes etc.

**CHEMICALS REQUIRED:**

Copper Sulphate, $H_2SO_4$ soln…

**PRINCIPLE:**

Colorimetric method depends on the measurements of quantity of light absorbed by a coloured solution according to Beers law

$$\text{i.e. } \log (I_o/I_t) = \in.c.l \text{ (or) } A= \in.c.l \text{ (or) } O.D = \in.c.l$$

Where

A or O.D = absorbents
$I_o$ = intensity of incident light
$I_t$ = intensity of transmitted light
$\in$ = Molar absorption co-efficient
L = path length or thickness of the medium
C = Concentration of solution
Since $\log(I_o/I_t)$ = absorbents or Optical density of medium

**PROCEDURE:**

Take 11 clean test tubes and all the test tubes are labeled as A,B,C,D,E,F,G,H,I,J,K. Now the standard Copper sulphate solution (0.5M) and distilled water are taken into test tubes by using 10ml measuring jar as indicated in the table. Now the solutions are ready for measuring the absorbents value.

Measurement of absorbents

The Measurement of absorbents with colorimeter is a two step process:
(1) Selection of proper filter
(2) Determination of absorbents

In Measurement of absorbents a blank solution is used. The blank solution consists of all reagents used in developing color expect the one responsible for color. The measurement with colorimeter is done by first adjusting the absorbents to zero with the blank solution in the path of light. Now replace the blank solution with sample solutions and absorbents will be displayed. Follow the same procedure for every measurement

**(1) Selection of proper filter:**

The Colorimeter cuvettes are cleaned. The blank solution (distilled water) and the sample solution are taken into two different cuvettes. One is used to select the filter and the other for adjusting absorbents to zero with distilled water. Power is supplied to Colorimeter and by using with distilled water, set the absorbents of the filter to zero and then measure the absorbents by using standard copper sulphate solution. Follow the same procedure at all filters. The results are incorporated in the table.

From the table the filter which gives maximum absorbance is noted. This filter is used for Measurement of absorbents of all the same sample solutions.

**(2) Determination of absorbents:**

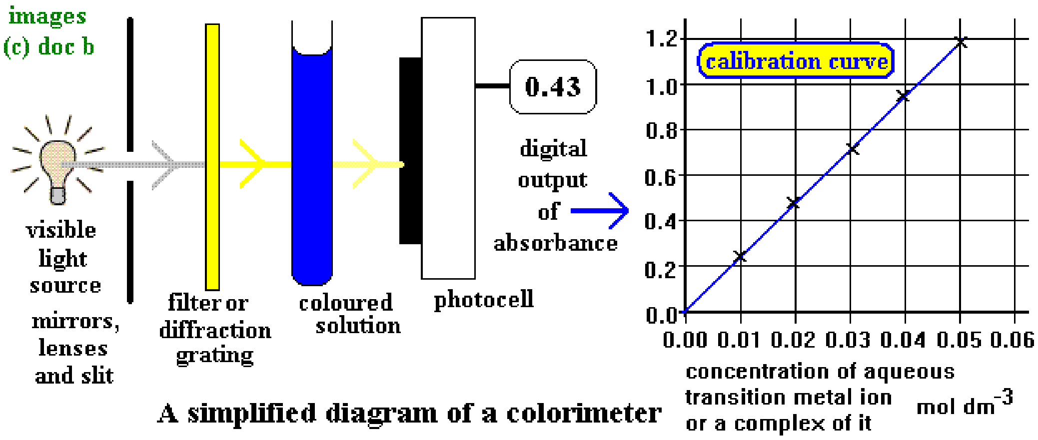
Initial reading of colorimeter of the selected filter is adjusted to zero absorbance (100% Transmittance) with the distilled water as blank solution in the cuvettes, now measure the absorbance of sample solutions at the same filter. The results are incorporated in the table.

Taking the concentration of copper sulphate on X-axis and volume of absorbance on Y-axis a graph is plotted. It is a straight line.

**Measurement of absorbance for unknown sample solution:**

Measures the absorbance for unknown sample solution at the same filter from the graph using absorbance determine the concentration of unknown solution.

**GRAPH:**



A simplified diagram of a colorimeter

**TABLE-I**
**SELECTION OF PROPER FILTER:**

| S. No | FILTER NUMBER | ABSORBANCE |
|---|---|---|
| 1 | 40 | |
| 2 | 42 | |
| 3 | 48 | |
| 4 | 50 | |
| 5 | 52 | |
| 6 | 54 | |
| 7 | 62 | |
| 8 | 68 | |

**TABLE-II**
**DETERINATION OF ABSORBENTS:**

| Test Tube label | (Volume of $CuSO_4$) + (Volume of distilled water) | Absorbance |
|---|---|---|
| **A** | (0+10)ml (0M) | |
| **B** | (1+9)ml (0.05M) | |
| **C** | (2+8)ml (0.10M) | |
| **D** | (3+7)ml (0.15M) | |
| **E** | (4+6)ml (0.20M) | |
| **F** | (5+5)ml (0.25M) | |
| **G** | (6+4)ml (0.30M) | |
| **H** | (7+3)ml (0.35M) | |
| **I** | (8+2)ml (0.40M) | |
| **J** | (9+1)ml (0.45M) | |
| **K** | (10+0)ml (0.50M) | |

**RESULT:**

Weight of copper present in given unknown solution =_________

i.e. $\dfrac{M \times M.Wt\ of\ CuSO_4 \times V}{1000}$

# DETERMINATION OF PERCENTAGE OF COPPER IN BRASS

**AIM:**

To determine the percentage of copper in a sample of brass in iodimetry

**APPARATUS:**

Burette, pipette, beakers, watch glass, conical flask, volumetric flask etc…

**CHEMICALS REQUIRED:**

Ammonia, Acetic acid, Potassium iodide, Sodium thiosulphate($Na_2S_2O_3$), Starch indicator, 6N $HNO_3$, Con.$H_2SO_4$, 6N $H_2SO_4$, $H_3PO_4$(ortho phosphoric acid), KSCN etc…..

**THEORY:**

Any cupric salt in neutral medium when heated with potassium iodide forms white ppt. of cuprous iodide. The liberated iodine is treated against hypo using starch as the indicator.

$CuSO_4 + 2KI \longrightarrow CuI_2 + K_2SO_4$

Cuprous iodide being unstable decomposes as follows

$2CuI_2 \longrightarrow Cu_2I_2 + I_2$

$I_2 + Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2NaI$

There fore

$2CuSO_4 = I_2 = Na_2S_2O_3$

The equivalent weight of copper sulphate is 63.54 gm

**PROCEDURE:**

1) Dry and clean a piece of brass cut into small piece and weigh about 0.20gm of the sample accurately to the fourth decimal place. Put it in a conical flask and add about 10ml of 6N $HNO_3$. Warm the soln. under a hood until the brass is completely dissolved.
2) Add 10ml of Con.$H_2SO_4$ and evaporate on sand bath till it gives copious white fumes
3) Allow the mixture to cool and then add carefully 20ml of water. Boil for 20sec and cool.
4) While vigorously shaking, add liquor ammonia drop wise until the first blue colour of cupric ammonia complex disappears.
5) Add 6N $H_2SO_4$ drop wise until the dark blue colour just disappears. Then add 2ml syrupy phosphoric acid ($H_3PO_4$).
6) Cool it to room temp. And transfer the solution to a 100ml volumetric flask. Make the volume up to 100ml.
7) Take 25ml of this solution in a125mlConical flask and add about 2ml of 5% KI.
8) Titrate immediately with standard Hypo solution until there is a faint color of iodine.
9) Add 3ml of 1% starch solution and titrate until the blue color begins to fade. Add 3 ml of KSCN and complete the titration.
10) Repeat the titration at least twice by taking 25ml of the solution each time.

**OBSERVATIONS AND CALCULATIONS:**
**$Cu^{+2}$ solution Vs Hypo solution**

| **S. No** | **Volume of $Cu^{+2}$ solution** | **Burette reading** | | **Volume of Hypo consumed** |
|---|---|---|---|---|
| | | **Initial** | **Final(ml)** | |
| 1 | 25ml | | | |
| 2 | 25ml | | | |
| 3 | 25ml | | | |
| | | Average the titre value | | (Yml) |

Weight of the Brass sample = _________ (W) gm

Strength of Sodium thiosulphate (Hypo) solution = _______ N (PN)

Normality of $Cu^{+2}$ in solution = P X Y/ 25 = _______ (ZN)

Here, equivalent weight of copper = Atomic weight

Amount of copper per liter = Z X Atomic weight (63.5gm)

Amount of copper per 100ml = Z X Atomic weight X100/1000

= ________ (Bgm)

% of copper in brass = B X 100/ W

= _________%

**RESULT:**

% of copper in brass = ________

# CONDUCTOMETRIC TITRATION OF STRONG ACID VS STRONG BASE

**AIM:**

To determine the strength of the strong acid by titration with strong base Conductometrically.

**APPARATUS:**

Conductivity Bridge, Conductivity cell, Burette, Beakers, Standard flask, pipette, Burette Stand etc.

**CHEMICALS REQUIRED:**

Sodium hydroxide, Hydrochloric acid

**PRINCIPLE:**

At first solution contain $H^+$ and $Cl^-$ ions. Since $H^+$ions posses greater mobility it follows that the conductivity is mainly due to $H^+$ ions. The addition of NaOH is represented by the equation.

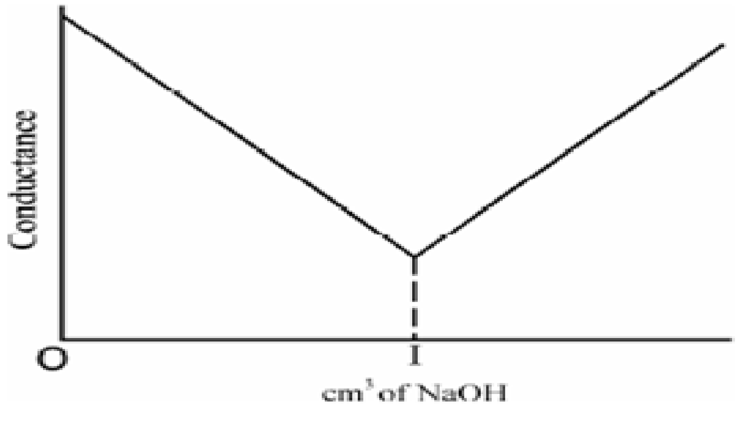
$H^+ + Cl^- + Na^+ + OH^-$ ————> $Na^+ + Cl^- + H_2O$

As NaOH is added the $H^+$ ions are removed. The conductivity decreases as $Na^+$ ions do not process much mobility. As the neutralization point and solutions contains $Na^+$ions and $Cl^-$ ions and will have minimum conductance value. If NaOH is further added this will add $OH^-$ ions and so the conductivity increases.

**PROCEDURE:**

A standard solution of 0.2N NaOH is prepared. Similarly 0.1N HCl is prepared. 20 ml of HCl is taken in a 100 ml beaker and to it 20 ml of distilled water is added and kept in a thermostat. The conductivity cell is washed with distilled water and rinsed with acid soln. The cell is kept in acid containing beaker and it is connected to the bridge. The conductivity of the soln is measured by adjusting the reading. NaOH soln is taken into burette and add 1 ml of soln to acid, stirred well and conductance is measured. Each time 1 ml of base is added to acid stirred well and the conductance is measured. For every instance. Equal numbers of values are taken on either side of the point of maximum. Repeat the procedure of addition of 1 ml NaOH and noting the conductivity of the resulting solution. Take 20-25 readings

**GRAPH:**



**CALCULATIONS:**

| **S. No** | **Volume of NaOH** | **Observed conductance** |
|---|---|---|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |
| 6 | | |
| 7 | | |
| 8 | | |
| 9 | | |
| 10 | | |
| 11 | | |
| 12 | | |
| 13 | | |
| 14 | | |
| 15 | | |
| 16 | | |
| 17 | | |
| 18 | | |
| 19 | | |
| 20 | | |
| 21 | | |
| 22 | | |
| 23 | | |
| 24 | | |
| 25 | | |

**FORMULA:**

$$N_1V_1 = N_2V_2$$

**RESULT:**

The normality of strong acid (HCl) determined by titrating against a strong base (NaOH) =________N

# CONDUCTOMETRIC TITRATION OF MIXTURE OF ACIDS VS STRONG BASE

**AIM:**

To determine the strength of the mixture of acids by titration with strong base Conductometrically.

**APPARATUS:**

Conductivity Bridge, Conductivity cell, Burette, Beakers, Standard flask, pipette, Burette Stand etc.

**CHEMICALS REQUIRED:**

Sodium hydroxide (NaOH), Hydrochloric acid(HCl) and Acetic acid($CH_3COOH$) etc…

**PRINCIPLE:**

The conductivity of the solution is related to the mobility of ions which in turn related with the size of the ions. When a mixture of acids like a strong acid (HCl) and weak acid (acetic acid) are titrated against a strong base (NaOH), strong acid reacts first followed by a weak acid. When the titration of strong acid and strong base are carried out, there is a decrease in conductivity as highly mobilized hydrogen ions are replaced by sodium ions.

$$\mathbf{NaOH + HCl \text{ -------> } NaCl + H_2O}$$

When the whole strong acid is consumed, base reacts with weak acid and conductivity increases as unionized weak acid becomes the ionized salt.

$$\mathbf{CH_3COOH + NaOH \text{----------> } CH_3COONa + H_2O}$$

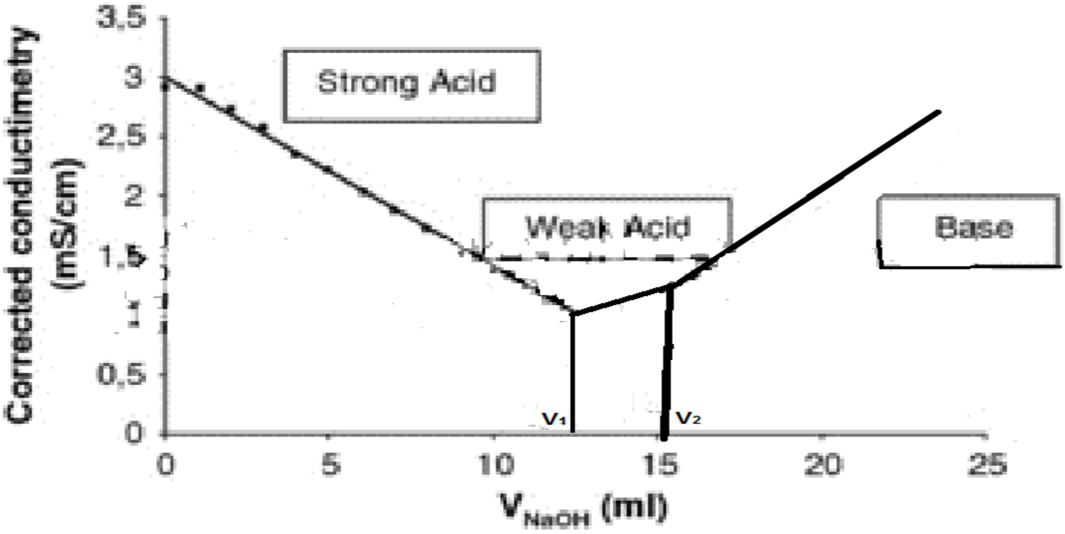
After both the acids are consumed, there is a steep increase in conductivity which gives the end point and this increase in conductivity is due to the fast moving hydroxyl ions from the base. From this, amount of base consumed for acid and in turn, the amount of acids present is calculated.

Let $V_1$ and $V_2$ ml be the volumes of alkaline corresponding beaker resp. Then $V_1$ ml of NaOH equivalent to HCl, $(V_2\text{-}V_1)_S$ ml of NaOH equivalent to $CH_3COOH$

**FORMULA:**

$$\mathbf{N_1V_1 = N_2V_2}$$

**GRAPH:**



**PROCEDURE:**

1) Fill the burette with standard 0.2N NaOH solution
2) Take the given mixture (25 ml of $CH_3COOH$ + 25 ml of HCl) in a small beaker and titrate against NaOH Conducto metrically.
3) Add 1ml of NaOH from burette and stir well and note down the conductance of solution.
4) Repeat the determination by stirring well after each addition of 1ml of NaOH until add up to 25-30 ml of NaOH.
5) Plot a graph between volume of NaOH Vs Conductance.

## OBSERVATIONS AND CALCULATIONS:

| **S. No** | **Volume of NaOH** | **Observed conductance** |
|---|---|---|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |
| 6 | | |
| 7 | | |
| 8 | | |
| 9 | | |
| 10 | | |
| 11 | | |
| 12 | | |
| 13 | | |
| 14 | | |
| 15 | | |
| 16 | | |
| 17 | | |
| 18 | | |
| 19 | | |
| 20 | | |
| 21 | | |
| 22 | | |
| 23 | | |
| 24 | | |
| 25 | | |

The volume of NaOH corresponding to the first point of inflectionsis $V_1 = X_1$ ml and that corresponding to the second point of inflectionis $V_2 = X_2$ ml.

$V_2 - V_1 = X_3$ ml

Hence the HCl and $CH_3COOH$ in their mixture of volume (50ml) consume respectively $X_1$ml and $X_3$ ml

There fore

(a). Strength of HCL $= \dfrac{X_1\text{ml x con. of NaOH}}{25\text{ml}}$ = ----------N

(b). Strength of $CH_3COOH$ $= \dfrac{X_3\text{ml x con. of NaOH}}{25\text{ml}}$ = --------N

## RESULT:

1) Strength of given HCl = ________________N

2) Strength of given $CH_3COOH$ = ______________N

# POTENTIOMETRIC TITRATION OF STRONG ACID VS STRONG BASE

**AIM:**

To determine the strength of the strong acid by titration with strong base Potentiometrically.

**APPARATUS:**

Potentiometer, Platinum electrode, Calomel electrode, Burette, Beakers, Standard flask, pipette, Burette Stand etc.

**CHEMICALS REQUIRED:**

Sodium hydroxide, Hydrochloric acid and Quinhydrone powder etc.

**PRINCIPLE:**

The cell will have certain emf depending upon pH value of the solution, on adding small portions of alkali to an acid. The acid potential changes slowly at first since the change in electrode depends on the fraction of hydrogen ions removed. After addition of certain amount of alkali the titration of hydrogen ions removed by alkali increases, correspondingly there is a rapid decrease in emf on addition of excess of alkali. The emf again shows a flow change. If a graph is plotted by taking volume of alkali added on X- axis and change in emf by point of intersection on Y-axis, a curve is obtained.

$$H^+ + Cl^- + Na^+ + OH^- \longrightarrow Na^+ + Cl^- + H_2O$$

The cell can be represented as

$$H_2, Pt/Acid\ soln\ //\ KCl_{aq}\ /\ Calomel\ electrode$$

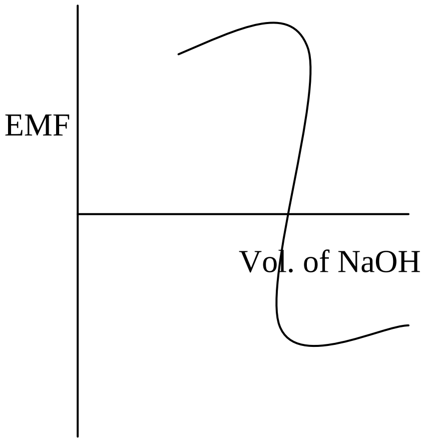
**PROCEDURE:**

Calibrate the instrument before starting the experiment. Approximately 0.1N HCl is prepared and standard decinormal solution of NaOH is prepared. Exactly 20 ml of the acid is pipette out into a clean 100ml of beaker and a pinch of Quinhydrone is added which acts as indicator. Platinum electrode and calomel electrodes are dipped in the solution.

The alkali (NaOH) against which the acid is being titrated is taken in burette. The solution is stirred well with a glass rod. The end reading is taken after adding definite amount of alkali. Finally after knowing the range in which the end point can be located, the whole experiment is repeatedly adding in steps of 1 ml in the end point.

**GRAPH:**

Two graphs are plotted of which one is between volume of alkali and observed emf and other is between volume of alkali and $\Delta E/\Delta V$ Sigmoid curve



## CALCULATIONS:

| S. No | Volume of NaOH | Observed EMF |
|---|---|---|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |
| 6 | | |
| 7 | | |
| 8 | | |
| 9 | | |
| 10 | | |
| 11 | | |
| 12 | | |
| 13 | | |
| 14 | | |
| 15 | | |
| 16 | | |
| 17 | | |
| 18 | | |
| 19 | | |
| 20 | | |

## FORMULA:

$$N_1V_1 = N_2V_2$$

## RESULT:

The normality of strong acid (HCl) determined by titrating against a strong base (NaOH) =________N

# POTENTIOMETRIC TITRATION OF WEAK ACID VS STRONG BASE

## AIM:

To determine the normality of Acetic acid by titration with strong base Potentiometrically.

## APPARATUS:

Potentiometer, Platinum electrode, Calomel electrode, Burette, Beakers, Standard flask, pipette, Burette Stand etc.

## CHEMICALS REQUIRED:

Sodium hydroxide (NaOH), Acetic acid ($CH_3COOH$) and Quinhydrone powder etc.

## PRINCIPLE:

The cell will have certain emf depending upon pH value of the solution, on adding small portions of alkali to an acid. The acid potential changes slowly at first since the change in electrode depends on the fraction of hydrogen ions removed. After addition of certain amount of alkali the titration of hydrogen ions removed by alkali increases, correspondingly there is a rapid decrease in emf on addition of excess of alkali. The emf again shows a flow change. If a graph is plotted by taking volume of alkali added on X- axis and change in emf by point of intersection on Y-axis, a curve is obtained.

$$CH_3COOH + NaOH \text{ ------------> } CH_3COONa + H_2O$$

The EMF of the cell represented as

$H_2$,Pt/Acid soln // KClaq / Calomel electrode

$$E = E^0 + 0.059pH$$

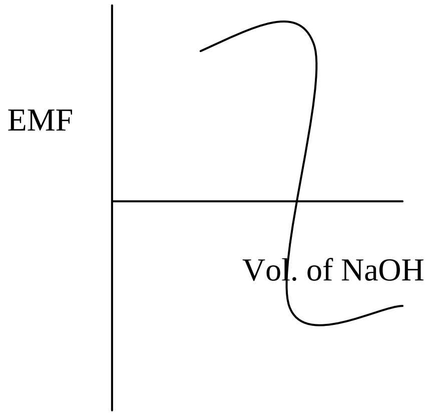
## PROCEDURE:

Calibrate the instrument before starting the experiment. Approximately 0.1N $CH_3COOH$is prepared and standard decinormal solution of NaOH is prepared. Exactly 20 ml of the acid is pipette out into a clean 100ml of beaker and a pinch of Quinhydrone is added which acts as indicator. Platinum electrode and calomel electrodes are dipped in the solution.

The alkali (NaOH) against which the acid is being titrated is taken in burette. The solution is stirred well with a glass rod. The end reading is taken after adding definite amount of alkali. Finally after knowing the range in which the end point can be located, the whole experiment is repeatedly adding in steps of 1 ml in the end point.

## GRAPH:

Two graphs are plotted of which one is between volume of alkali and observed emf and other is between volume of alkali and $\Delta E/\Delta V$ Sigmoid curve



**CALCULATIONS:**

| **S. No** | **Volume of NaOH** | **Observed emf** |
|---|---|---|
| 1 | | |
| 2 | | |
| 3 | | |
| 4 | | |
| 5 | | |
| 6 | | |
| 7 | | |
| 8 | | |
| 9 | | |
| 10 | | |
| 11 | | |
| 12 | | |
| 13 | | |
| 14 | | |
| 15 | | |
| 16 | | |
| 17 | | |
| 18 | | |
| 19 | | |
| 20 | | |

**FORMULA:**

$$N_1V_1 = N_2V_2$$

**RESULT:**

The normality of Weak acid ($CH_3COOH$) determined by titrating against a strong base (NaOH) =________N