Safety Rules and Guidelines

* Handling Chemicals

a) Work with hazardous or toxic materials must not be undertaken without proper precautions.

b) The reagents should be properly used and must be placed at the prescribed place after use.

c) All the bottles and packages should be clearly labeled.

d) Never return unused chemicals to their original containers.

e) Never taste a chemical. Use your hand to waft the odor to your nose.

f) Do not bring inflammable liquids like alcohol, either and benzene near the flame.

* Personal Safety

a) There must be punctuality among the students in the laboratory.

b) Students and staff must wear safety glasses and laboratory coats while working in the laboratory.

c) Proper footwear must be worn in the laboratories. The whole foot must be protected / covered.

d) Smoking is prohibited.

e) Eating and drinking in ongoing laboratories are prohibited.

f) Switch off the burner if not in use and help to save LPG (gas).

g) Heat the reagents in a test tube by keeping it's mouth away from yourself and others.

h) Report any defect breakage of apparatus to the teacher/lab Assistant.

* **Safety Precautions at lab**

1. Burns

a) **Acids:** wash off corrosive substances by flooding with tap water. After washing, rub a paste of sodium bicarbonate and water into skin.

b) **Alkalis:** After washing thoroughly with water, apply a 1% solution of acetic acid or 1% boric acid solution.

c) **Dry heat:** a) Very small burns: Hold the burn in cold saturated (8%) sodium bicarbonate solution for sometime then cover freely with zinc oxide ointment and bandage to exclude air.

d) **Large burns:** Always apply acriflavine emulsion freely and without delay.

e) **Boiling water:** Apply acriflavine/picric acid.

f) **Sodium burns:** Wash with water and then 1% dilute acetic acid and cover with gauze soaked in olive oil. For serious burns apply the acriflavine ointment.

2. Eye Accidents

a) **Acids:** splash water in eyes and then wash with 1% sodium bicarbonate solutions.

b) **Alkali:** wash eyes with water and then 1% boric acid solution.

c) **Glass:** Remove glass pieces either with forceps or by washing with water in an eye bath. If glass is not removed, place patient on his back hold eye gently open until doctor arrives.

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3. Cuts

Wash well in 1% aqueous chloramines-T solution or in 2% iodine solution. Remove dirt or glass. Wash again and apply sterilized dressing firmly bandaged.

4. Fires

a) **Burning cloths**: Prevent person from running and fanning flames. He or she should be down and wrap fireproof blanket around until fire is extinguished.

b) **Burning reagents**: Cover the opening of vessels with a duster for large fire, use dry send and commercial fire extinguisher.

5. Electric shock

Switch off and treat for burns and shock.

6. Explosions

a) Faulty condensation of a heavy inflammable vapour, such as ether.

b) Igniting an inflammable gas before all air has been removed from the containing vessel.

c) Experiments in which metallic sodium has been used, and in which the product has subsequently to be treated with water. Great care should be taken to ensure that no unchanged sodium remaining when the water is added.
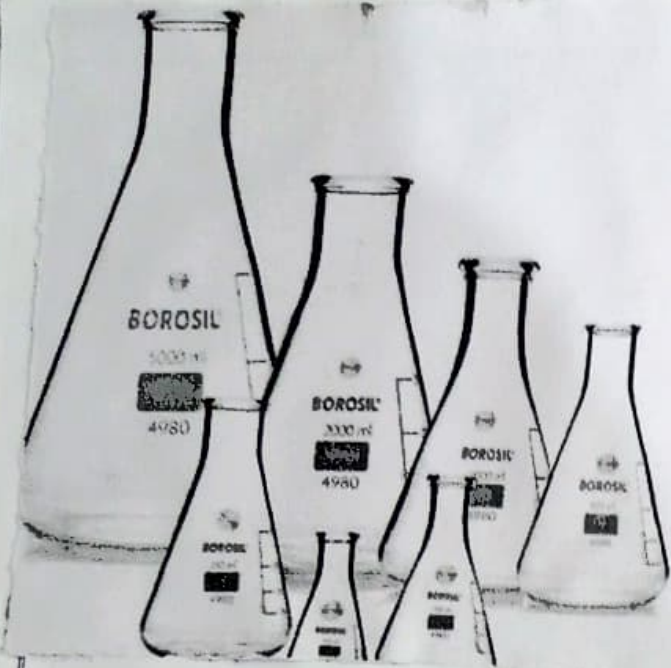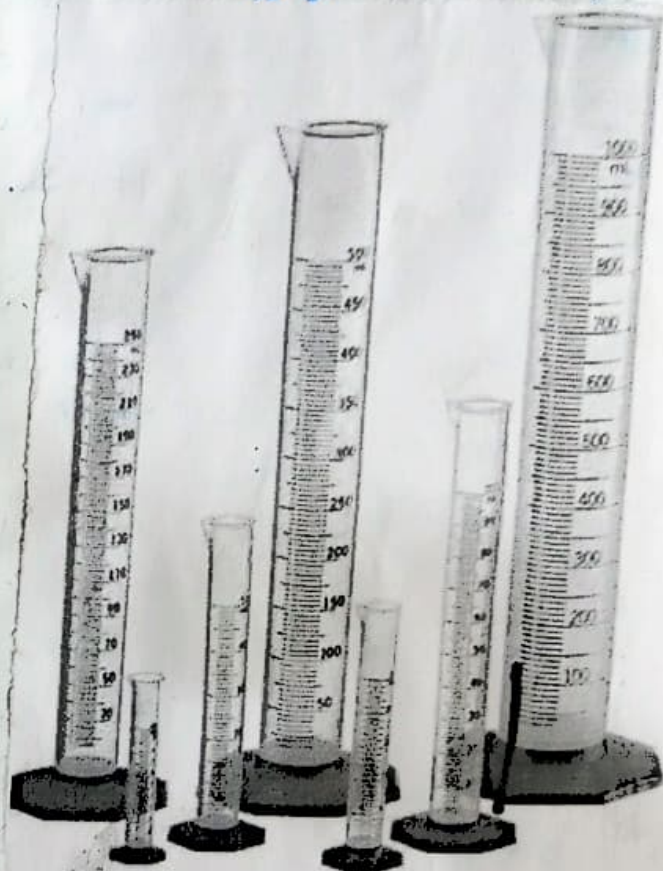
conical flasks
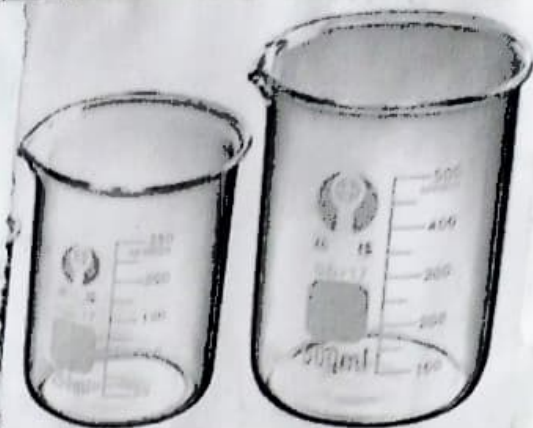
Beakers

Test tubes

Graduated cylinders

Glass Rods

Glassware and Glass Apparatus in chemistry Laboratory

* **Erlenmeyer (conical) flasks** are used to measure, mix and store liquids. Most erlenmeyer flasks are made of borosilicate glass so that they can be heated over a flame or autoclaved.

* **Beakers** are used for routine measuring and mixing in the lab. They are used to measure volumes to within 10% accuracy.

* **Test tubes** are round-bottom cylinders, usually made of brosilicate glass so that they can withstand temperature changes and resist reaction with chemicals. In some cases, test tubes are made from plastic.

* **Volumetric flasks** are used to prepare solutions accurately in chemistry laboratory. This piece of glassware is characterized by a long neck with a line for measuring a specified volume.

* **Graduated cylinders** are used to measure volumes accurately. The can be used to calculate the destiny of an object if it's mass is known.

* **A funnel** is a conical piece of glass or plastic that is used help transfer chemicals from one container to another. Some funnels act as filters, either because of their design or a sieve is placed on the funnel.
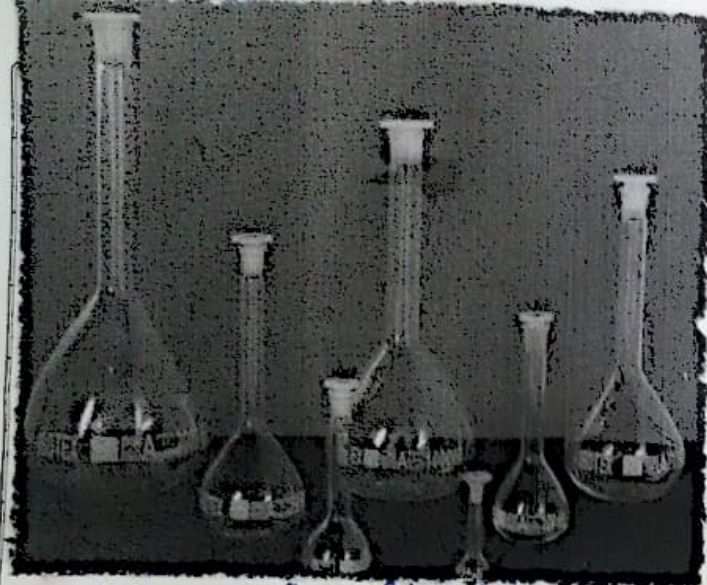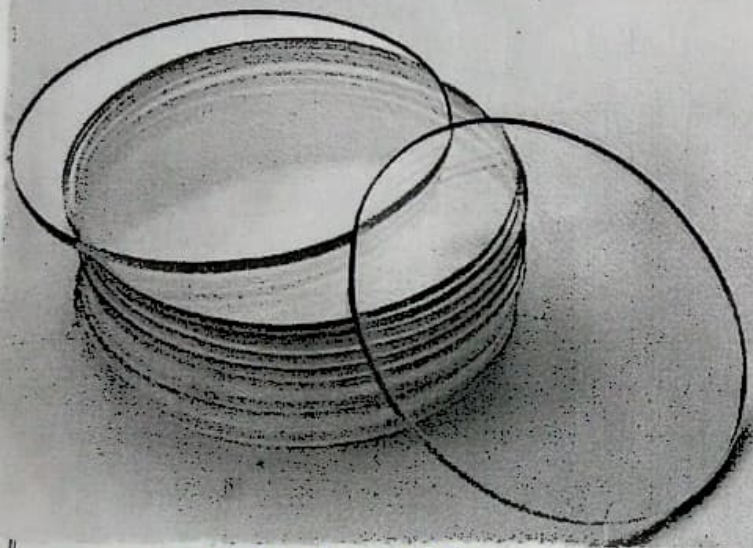
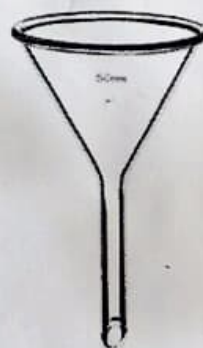Volumetric flasks

100 ml   50 ml
Glass Bottle

Watch Glass

100mm
75mm
Funnel

Pippette
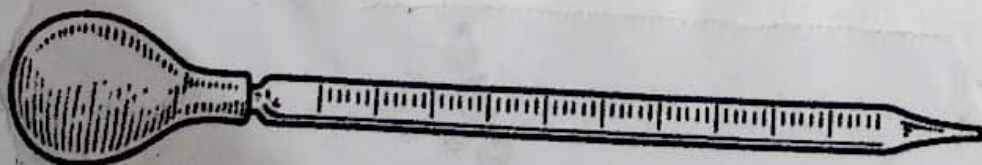
* Pipettes or pipettes are droppers calibrated to deliver a specific volume. Some pipettes are marked like graduated cylinder. Other pipettes are filled to a line to reliably deliver one volume again and again.

* Glass bottles with ground glass stoppers are often used to store stock solutions of chemicals. To avoid contamination, it helps to use one bottle for one chemical.

* Watch glasses have all kinds of uses. They are concave "dishes" that can be used as beaker lids; to hold protests and other invertibrates for viewing under a microscope; or to dissolve materials such as crystals and powders.

* Glass rod is used for stirring.

$$\log\left(I_0/I_t\right) = A = EcL$$
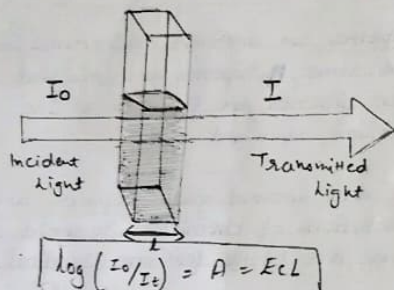
* Schematic Representation of Apparatus and Functioning

(Source)   (Monochromator)   (sample holder)   (Detector)   (Recorder)
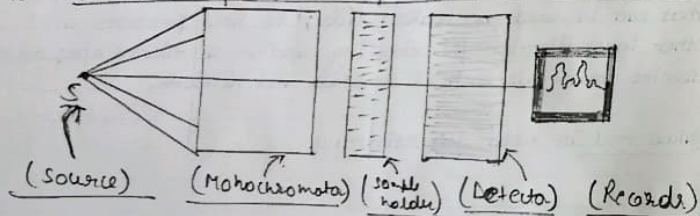
Experiment No. → 1

* AIM

To verify Lambert - Beer's law for $KMnO_4$ colorimetrically.

* Theory

The Lambert - Beer's law states that the absorbance of a solution is [directly] [proportional] to the concentration of the absorbing species in the solution and the path length. Thus, for a fixed path length (cuvette length), UV/V is spectrabrance changes with concentration, A higher concentration of the colored solution absorbs more light (and transmits less) than a solution of lower concentration.

$$\log\left(I_0/I_t\right) = A = EcL$$

where,
I_0 and I_t are the incident and transmitted intensities,
A = absorbance and
c is a constant i.e. absorptivity.

* Requirements

UV-V is spectrophotometer, cuvette, six test tubes.
Two Burettes or graduated cylinders two 100 ml beakers.
$KMnO_4$, distilled water, test tube rack

* Precautions

One should note that the Beer - Lambert law is obeyed by many substances mainly at low to moderate concentration; therefore, dilute concentrations of the absorbing species

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* observation:-

Test Tube	0.0004 M KMnO₄ (ml)	Distilled H₂O (ml)	Conc (m)	Absorbance.
1.	2	8	0.00008	0.223
2.	4	6	0.00016	0.267
3.	6	4	0.00024	0.402
4.	8	2	0.00032	0.570
5.	10	0	0.00040	0.806

Graph)
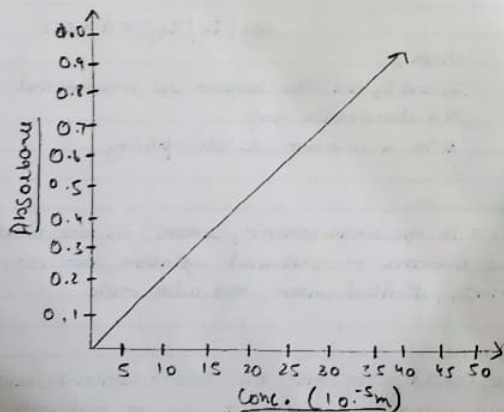
should be measured. In practise it is advisable to measure absorbances in the range $0.1 < A < 1.0$. Care must be taken to avoid any kind of chemical associations/dissociations of the absorbing species.

* Results-
A linear curve is obtained between absorbance v/s concentration that prove the existence of Lambert - Beer law.

* Source of Error :-
i) The solution of KMNO₄ might not be prepared of derived concentration for the experiment.
ii) Error might occur at the time of dilution of solution.
iii) Instrument error while recording the absorbance.

chemical Reactions Involved

$$(COOH)_2 + 2NaOH \longrightarrow 2(COONa) + 2H_2O$$

$$HCL + NaOH \longrightarrow NaCl + H_2O$$

Experiment no. → 2

AIM:-

To determine the normality of 10ml unknown HCL solution when normality of oxalic acid is given.

Material Required:-

- Solutions and Reagents — 0.05N of 10ml oxalic acid, unknown NaOH solution, phenolphthalein, unknown HCL solution.

- Apparatus — Burette, Pipette, conical flask, Funnel, measuring flask, weighing bottle, stirrer, burette stand, beaker, dropper.

Theory:-

i) Initially the estimation involves titration of a weak acid (oxalic acid) against a strong base (sodium Hydroxide) phenolphthalein as an indicator of choice.

ii)

ii) Since sodium hydroxide solution but a standard solution of oxalic acid is prepared and used for standardisation of sodium Hydroxide.

iii) In weak acid — strong base titration at the end - point the amount of acid becomes chemically equivalent to the amount of base present. Phenolphthalein turns pink due to some extra base present in it.

Observation Table

For standard oxalic acid solution of 0.05N and 10 ml and unknown NaOH solution.

Sr. No.	Initial Reading of Burette	Final reading of Burette	Volume of NaOH used
1.	19.50 ml	25.00 ml	5.50 ml
2.	25.60 ml	28.35 ml	3.35 ml
3.	28.85 ml	33.85 ml	5.50 ml
4.	33.85 ml	38.35 ml	5.50 ml

concurrent reading = 5.50 ml

Now, for standard NaOH solution of 0.09N and 10 ml and unknown HCl solution

Sr. No.	Initial Reading of Burette (in ml)	Final Reading of Burette (in ml)	Volume of NaOH used (in ml)
1.	38.35 ml	42.00 ml	3.65 ml
2.	42.00 ml	46.00 ml	4 ml
3.	46.00 ml	50 ml	4 ml

concurrent reading = 4 ml

---

iv) After obtaining a standardised solution of sodium hydroxide, it titrated with unknown solution of hydrocloric acid which is prepared primary as non-standard.

iv) After obtaining a standardised solution of sodium hydroxide, it titrated with unknown solution of hydrocloric acid which is prepared primary as non-standard.

v) In case of strong base titrated at the end point of the solution. The solution becomes neutral. The indicator phenalph-thalin turns the solution pink after some extra base is added to it.

* Calculations :-

$$N_1 V_1 = N_2 V_2$$
$$0.05 \times 10 = N_2 \times 5.50$$
$$\frac{0.5}{5.5} = N_2$$
$$\boxed{N_2 = 0.09 \, N}$$

$N_1 : 0.05N$
(oxalic acid)
$V_1 : 10 \, ml$
$V_2 : 5.50 \, ml$
(vol. of NaOH)

$$N_2 V_2 = N_3 V_3$$
$$0.09 \times 4 = 10 \times N_3$$
$$0.36 = 10 \times N_3$$
$$\boxed{0.04 = N_3}$$

$N_2 : 0.09 \, N$
(NaOH solution)
$V_2 : 4 \, ml$
$V_3 : 10 \, ml$
(vol. of HCl)

* Results:-

The normality of HCl solution calculated is 0.04 N when 10 ml of solution is taken.

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* Precautions :-

i) NaOH solution in burette must be read from lower meniscus.

ii) Rinse the pipette and burette before use.

iii) Use dropper to add phenelphthalin

iv) Clean all the apparatus before use and rinse them with distilled water before starting the experiment

v) Take accurate readings once it reaches the end - point.

vi) The normality of unknown solution must be taken to upto two - decimal places only.

* Sources of Error :-

i) The solutions must be prepared carefully so that error could be minimized.

ii) Measuring the correct amount of solution will reduce error.

iii) Error can be made while taking observations.

Date ___

Expt. No. ___   Page No. ___ 6 ___

Experiment No. → 3

* Aim :-

To determine the strength of unknown oxalic acid solution by titrating against potasium permagnet solution.

* Material Required :-

~~Soulution and Reagents:~~ oxalic acid, potasium permagnet soulution, dilute sulpheric acid.

~~Appratus Required :~~ Buvette, pipette, conical flask, funnel, measuring flask, Weighing bottle.

* Theory:-

i) In present experiment, potasium permagnet acts as a powerful oxidising agent. Although, KMNO4 acts as an oxidising agent in alkaline medium, also, for quantitative analysis mestty acidic medium is used.

ii) The oxidising agent KMNO4, in the acidic medium can be represented by the following reaction equation:

$$MnO_4^- + 8H^+ + 5e^- \longrightarrow Mn^{+2} + 4H_2O$$
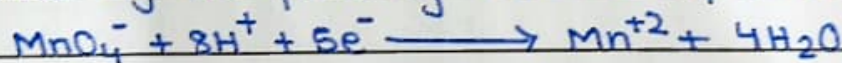
iii) The acid used in titration is dilute sulpheric acid. Nitric acid is not used as it itself an oxidising agent and hydrocloric acid is usally avoided because it reacts with KMNO4 according to the equation given below to produce chlorine is an oxidising agent in aquoeus solution.

$$2KMNO_4 + 16HCL \longrightarrow 2KCL + 2MnCl_2 + 5Cl_2 + 8H_2O$$
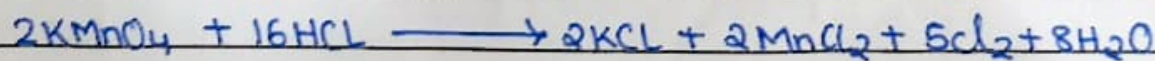
iv) Since, oxalic acid acts as a reducing agent, it can be

titrated against potasium permagnet in the acidic medium according to following given equation.

v) In the equations, MnO_4^- is reduced to Mn^{+2} and $C_2O_4^{2-}$ is oxidised to CO_2. The oxidation number of carbon in $C_2O_4^{2-}$ changes ~~so so~~ from +3 to +4.

vi) In these titration, potasium permagnet acts as a self indicator. Initially colour of potasium permangnet is discharged due to its reduction by oxalic acid.

* Calculations :-

$$N_1 V_1 = N_2 V_2$$

$$0.05 \times 10 = N_2 \times 9.0 ml.$$

$$N_2 = \frac{0.05 \times 10}{9}$$

$$\boxed{N_2 ~ 0.06 N}$$

$N_1 : 0.05$ (oxalic)

$V_1 : 10 ml$

$V_2 : 9 ml$

(Vol. of $KMnO_4$)

$$N_2 V_2 = N_3 V_3$$

$$0.06 \times 6.1 = N_3 \times 10$$

$$\boxed{0.0366 = N_3}$$

$$N_3 ~ 0.04 N$$

$N_2 : 0.06 N$ ($KMnO_4$)

$V_2 : 6.1 ml$

$V_3 : 10 ml.$

* Result :-

The normality obtained of unknown oxalic acid is 0.04N.
The strength obtained is 2.52g/L

* Precautions :-

i) Potasium permagnet is dark in colour, so always read the upper meniscus.

* Observation Table :-

For standard Oxalic acid solution of 0.05N and 10ml vol of Unknown KMnO₄ sol.

S.No	Initial Reading of Burette	Final Reading of Burette	Volume of KMnO₄ used
1.	9.1ml	18.0ml	8.9ml
2.	18.0ml	27.ml	9.0ml
3.	27.0ml	36.0ml	9.0ml

Concurrent Reading = 9.0ml.

Now, Standard KMnO₄ sol. of 0.06N & 10ml Unknown Oxalic acid sol.

S.No	Initial Reading of Burette	Final Reading of Burette	Volume of KMnO₄ used
1.	3.9ml	10ml	6.1ml
2.	10ml	15.8ml	5.8ml
3.	15.8ml	21.9ml	6.1ml

ii) Rinse the pipette and burette before use.
iii) Use dilute sulphuric acid for acidifying the KMnO₄.
iv) Take accurate readings once it reaches the end-point.
v) Don't go with average reading.

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Experiment No. → 4

* Aim :-

To determine the pH of a given buffer soulution by colour matching of indicator.

* Material Required :-

Soultion and Regents: 200 ml of 0.4N CH_3COOH (acetic acid) 200 ml of 0.4N NaOH (Sodium hydroxide), methyl orange as indicator.

Apparatus Required: test tubes, pipette, beakers, pH meter

* Theory :-

i) The pH of an aqueous soulution is the negative of logarithm to base 10 (log_{10}) of hydrogen activity (a_{H^+})

$$pH = -log_{10} a_{H^+}$$

ii) for dilute solutions, activity coefficients (f) are nearly unity and so the activity may be replaced by the numerical value of the molar concentration (c). for such a dilute soulution,

$$pH = log_{10} c_{H^+}$$

iii) A buffer solulution is a mixture of a weak acid and its salt or of a weak base and its salt. pH of buffer solution have definite values depending upon the ionization constants (pK) of the constituent acids or bases and the ratios of acid : salt as the case may be.

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iv) Buffer solution have the ability to resist the change of pH when small amounts of acids and bases are added to them. pH of a buffer solution consisting of a weak acid and its salt with a strong base is expressed by the Hendersen equation.

$$pH = pKa + log \frac{[Salt]}{[acid]}$$

v) Ka is the ionisation constant of the weak acid and [ ]'s represent the molar concentration. Thus, a series of buffer solution of known pH values may be obtained by mixing known amounts of a weak acid with known known amounts of its salt.

vi) Acid-base indicators exhibit distinguishable colours in distinctly acidic and distinctly alkaline solution, the actual shade of colour, of cource, depends up on the ratio of the concentration of the acidic and basic form of the indicator, which in turn depends upon the pH of solution.
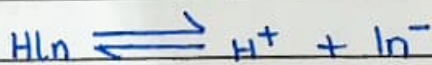
$$HIn \rightleftharpoons H^+ + In^-$$

for which ionisation constant Kin, is given by

$$Kin = \frac{[H^+][In^-]}{[HIn]}$$

* Result :-

The colour of the test tube matches with the given colour of solution of test tube.

The test tube 1 which has pH = 3.53 matches in colour.

* Observation Table :-

S.No	Vol. of CH_3COOH (ml)	Vol. of NaO4	Vol of H_2O	Total Vol of Test tube	PH
1.	5.0	0.5	4.5	10ml	3.58
2.	5.0	1.0	4.0	10ml	3.94
3.	5.0	1.5	3.5	10ml	4.18
4.	5.0	2.0	3.0	10ml	4.34
5.	5.0	2.5	2.5	10ml	4.47
6.	5.0	3.0	2.0	10ml	4.66

* Precautions :-

i) Rinse the test tube and pipette before use.

ii) Always try to take accurate reading of pH from pH meter and no to go with average reading.

iii) Prepare the 10ml solution in test tube carefully while taking volume of solution in desired quantities.

iv) The pH reading of the solution must be taken carefully so that no solution comes out of test tube.

* Sources of Error :-

i) Error can be made while recording the pH reading.

ii) Error can be made while preparing the 10ml solution in test tube as the required volume must not be taken.

Experiment no. → 5

* **Aim :-**

To find the heat of neutralisation of strong acid (HCL) and strong base (NaOH).

* **Material Required :-**

Solution and Reagents: Thermo flask, Glass   , Hydrocloric acid, sodium hydroxide,

Apparatus Required: Thermo flask, Glass   , hydroclovie thermometer stop watch, beaker, measuring cylinder, calorimeter , heater.

* **Theory :-**

Water equivalent of calorimeter :

i) The water equivalent of colorimeter or heat capacity of the colorimeter as the mass of water which will be heater through 1°c by the same amount of heat required to rise the temprature of the body through 1°c.

ii) $g = ms\theta$

where,

     θ is temprature difference

     s is specific heat of substance.

     m is mass of water

     g is quantity of heat lost or gained during the reaction.

     $ms (=w)$ is water equivalent of calorimeter.

iii) Measurement of W (i.e. water equivalent of calorimeter) is necessary because during heat exchange, the calorimeter also takes some amount of heat evolved and hence should taken into

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account.

Heat of Neutralisation of HCL and NaOH

i) The enthalpy of neutralisation (ΔH neut) of an acid can be defined as the enthalpy change associated with the complete neutralisation of its dilute aqueous solution containing one Mole H^+ ions by a dilute aqueous solution containing one mole OH^- ions.

ii) Consider the neutralisation of hydrochloric acid and sodium hydroxide. HCL is a strong acid and NaOH is a strong base. This means that both HCL and NaOH are completely.

iii) The neutralisation of a strong base with a strong acid can be considered as the combination of reaction $[H^+]$ (aq.) ion an $[OH^-]$ ions.

* **Calculations:-**

Temp. of room temp. water = 15°C

Temp. of Hot water = 62°C

Temp. of mixed water = 34°C

Heat lost = Heat gained

$25(62-34) = 25(34-15) + W(34-15)$

$25 \times 28 = 25 \times 19 + W \times 19$

$25 \times 9 = W \times 19$

$\dfrac{25 \times 9}{19} = W$

$$\boxed{11.84 \ cal/°c = W}$$

$Q = ms \ \Delta\theta$

$m = 25g$

$v = 25ml$

density of H_2O

$= 1g/ml$

$S = 1 cal/°c$

$\Delta\theta = (62-34)°c$

water equivalent / heat capacity of calorimeter = 11.84 cal/°c

$\Delta H = -[$Heat gained by the calorimeter + Heat gained by the solution$]$.

$$\Delta H = -[cp(c)(Tm - Ta) + V base \times Sx (Tm - Tn)$$
$$+ V acid \times Sx (Tm - Tn)]$$

$\Delta H = -[11.84 \times (18 - 15) + 25(18 - 15) + 25(18 - 15)]$ cal/mol

$\Delta H = -[35.52 + 50 \times 3]$ cal/mol.

$\Delta H = -[35.52 + 150]$ cal/mol

$\Delta H = -185.52$ cal/mol

(It is calculated for 0.5m solution of NaOH and HCl with 25ml of Volume).

ΔH_{neut} (Standard) $= -185.52 \times 2 \times 40$ cal/mol

$\qquad = -14841.6$ cal/mol

$\qquad = -14.84$ Kcal/mol

(The standard value is obtained for 1m of Solution of NaOH and HCl with 1000 ml of volume).

* **Percentage Error :-**

original ΔH_{neut} (theoritical) $= -13.70$ Kcal/mol

obtained ΔH_{neut} (experimental) $= -14.84$ Kcal/mol

Percentage error $= \dfrac{1.14}{13.7} \times 100\% = 8.32\%$

* **Result :-**

The heat of neutralisation of Solution obtained is $\boxed{-14.84}$ Kcal/mol and water equivalent obtained was $\underline{-14.84}$ cal/°c

* Observation Table :-

- vol. of water at room tmp = $\frac{50}{2}$ml = 28ml

- vol. of hot water = $50/2$ml = 25ml.

(water equivalent of colorimeter)

Room temp water		Hot water.		Mixture.	
Time	Temp (c)	Time	Temp (c)	Time	Temp(c)
30s	15°C	30s	62°C	30's	24°C
60s	15°C	60s	62°C	60s	34°C
90s	15°C	90s	62°C	90s	34°C

- Conc. of HCl used = $1m/2$ = 0.5M

- Conc. of NaOH used = $1m/2$ = 0.5M

(Heat of Neutralisation)

| HCL		NaOH		Mixture.	
Time	Temp °C	Time	Temp (c)	Tim	Temp(°c)
30s	15°C	30s	15°C	30s	18°c
60s	15°C	60s	15°C	60s	18°C
90s	15°C	90s	15°C	90s	15°C.

* Precautions :-

i) Rinse all the equipment before use.

ii) Carefully handle the acid and base.

iii) The stopwatch must show accurate time so as to minimise the error.

iv) The mercury level in the thermometer must be stable to get accurate reading.

v) The calorimeter and stirrer must be handled carefully.
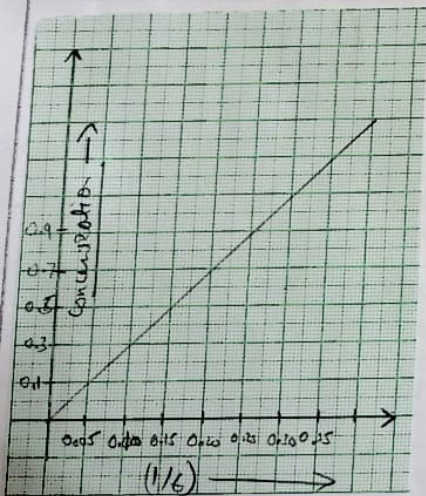
Experiment No. → 6

* Aim :-

To study the kinetics of decomposition of sodium thiosulphate by a mineral acid.

* Material Required :-

Solution and Reagents : Water (H_2O), Hydrochloric acid (HCL), sodium thiosulphate ($Na_2S_2O_3$).

Apparatus Required : beakers, stirrer, stopwatch, measuring cylinder.

* Theory :-

The decomposition of sodium thiosulphate in hydrochloric acid takes place according to give reactions. The precipitation of sulphur is marked by turbidity in its solution and this indicates the progress of reaction.

The acid taken in large excess to keep its concentration more or less constant and solutions of various concentration of sodium thiosulphate to study the reaction kinetics.

* Result :-

The graph obtained is a straight line between the concentration and reciprocal of time. This means that decomposition follows first order kinetics.

* Precautions :-

(a) Measure the right weight of sodium thiosulphate and correct volume of HCl.

✳ Observation Table

Amount of Sodium thiosulphate (in gm)	Volume of 1/2 HCl added (in ml)	time required for turbidity (in min)	Reciprocal of time (1/t)
0.1	20ml	30.85	0.032
0.3	20ml	10.28	0.97
0.5	20ml	6.17	0.162
0.7	20 ml	4.40	0.227
0.9	20ml.	3.43	0.291

(b) Rinse all the equipments before use.

(c) The time all the equipm should be noted from stopwatch carefully.

(d) The temprature should be constant throughout the process until turbidity is reached.

✳ Source of Error :-

(a) The stopwatch can cause error while generating times.

(b) Not exact weight of sodium thoisulphate is taken.

(c) The turbidity of solution can be affected as the temprature can got changed while stirring.

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Experiment No. → 7

* **Aim:-**
  To prepare benzoic acid from benzaldehyde by oxidation.

* **Material Required :-**
  <u>Reagents and Solution</u> → potassium permagnate, sodium carbonate
  benzaldehyde, water, hydrochloric acid.
  <u>Appratus required</u> → conical flask, beaker, filter paper, stirrer.

* **Theory :-**
  Required volume of chemicals taken in experiment
  2g of $KMnO_4$ in 20mL of water.
  2g of Na_2CO_3 in 20 mL of water.
  $N/5$ of HCl solution
  2-3mL of benzaldehyle

  i) Benzaldehyle can be oxidised to benzoic acid by reaction with potassium
  permanganate in the presence of sodium carbonate by given reaction.
  ii) Oxidation of benzaldehyde to benzoic acid requires ~ 15-20 minutes of
  reflexing with potassium permanganate   solution.
  iii) Excess of unreacted potassium permagnate and manganese dioxide
  formed during the reaction, react with sodium sulphit in acidic
  medium as follows.

* **Result :-**
  The powdered salt obtained after filtration is benzoic acid
  white in colour

* Precautions:-

i) Prepare right volume of solution at constant temprature so that concentration doesn't get affected.

ii) Add KMnO₄ gradually to avoid any error.

iii) Heat the solution continuously while adding KMnO₄ until right colour is obtained.

iv) Cool the solution to room temprature before filtering it out.

16/2/23