B.Sc. CHEMISTRY LAB MANUAL

3rd Semester

Prepared By

Pure & Applied Science Dept.

Chemistry

MIDNAPORE CITY COLLEGE

MIDNAPORE CITY COLLEGE

Department of Pure and Applied Sciences

B.Sc. in Chemistry

Semester: III

Paper Title: Physical Chemistry-I (Practical)

Paper Code: CEMHMJ03P

Course contents:

1. Calibration of various apparatus such as burette, pipette, volumetric flux, measuring cylinder etc.

2. Concepts of molecular weight, equivalent weight, various concentration terms, primary and secondary standard solutions and their preparations with proper explanations of types of apparatus to be used to prepare these solutions.

Prepare the following solutions

(i) Primary standard oxalic acid solution by accurate weighing

(ii) Primary standard potassium dichromate solutions by accurate weighing

(iii) Secondary standard sodium hydroxide solutions by approximate weighing

(iv) Secondary standard sodium thiosulphate solutions by approximate weighing

(v) Secondary standard potassium permanganate solution by approximate weighing

(vi) Secondary standard sulphuric acid, hydrochlroric acid and acetic acid solutions of various strength from the supplied concentrated solutions.

3. Study of kinetics of acid-catalyzed hydrolysis of an ester

4. Study of kinetics of decomposition of H2O2

5. Determination of specific rotation of cane sugar using polarimeter

6. Study the kinetics of inversion of cane sugar using polarimeter.

Experiment 1: Calibration of various apparatus such as burette, pipette, volumetric flask, measuring cylinder, etc.

Introduction:

You have studied the use of various volume measuring devices such as measuring cylinders, burettes, pipettes, and volumetric flasks previously. Measuring cylinders are used for approximate measurement of volume for various purposes and do not require any calibration. Burettes, pipettes, and volumetric flasks are used for accurate quantitative measurements. When using apparatus of standard quality. it is not desirable to attempt calibration of such apparatus for three reasons. Firstly. the process of calibration is time-consuming. Secondly, due to the possibility of introduction of errors in calibrating the apparatus, the apparatus thus calibrated by an inexperienced beginner may be less accurate than the apparatus calibrated by the manufacturer. Thirdly, the error permitted in a determination may take care of any inaccuracies in the calibration of the apparatus. However, if the apparatus being used is not of standard quality. it is advisable to check the calibration of the apparat& before performing any measurements with such an apparatus. In this experiment. you will perform the calibration of a pipette, a burette, and a volumetric flask.

Objectives:

After performing this experiment, you should be able to:

- Justify the need for calibration of apparatus
- State the steps involved in the calibration of volumetric apparatus, calibrate burettes, and volumetric flasks.

Principle

Calibration of a burette and pipette is carried out by measuring the mass of a known volume of water delivered by the burette or the pipette. By knowing the temperature and the density of water at that temperature, the true volume delivered by the burette or the pipette is calculated. This will be the volume delivered by the burette or the pipette and not the one which is etched on the apparatus. The volumetric flasks are calibrated by filling them using a calibrated pipette.

Requirements

Apparatus: Analytical balance -1, Beaker -1, Burette -1, Conical flask- 4, Cork/stoppers- 4, Pipette -1, Thermometer -1, Volumetric flask -1

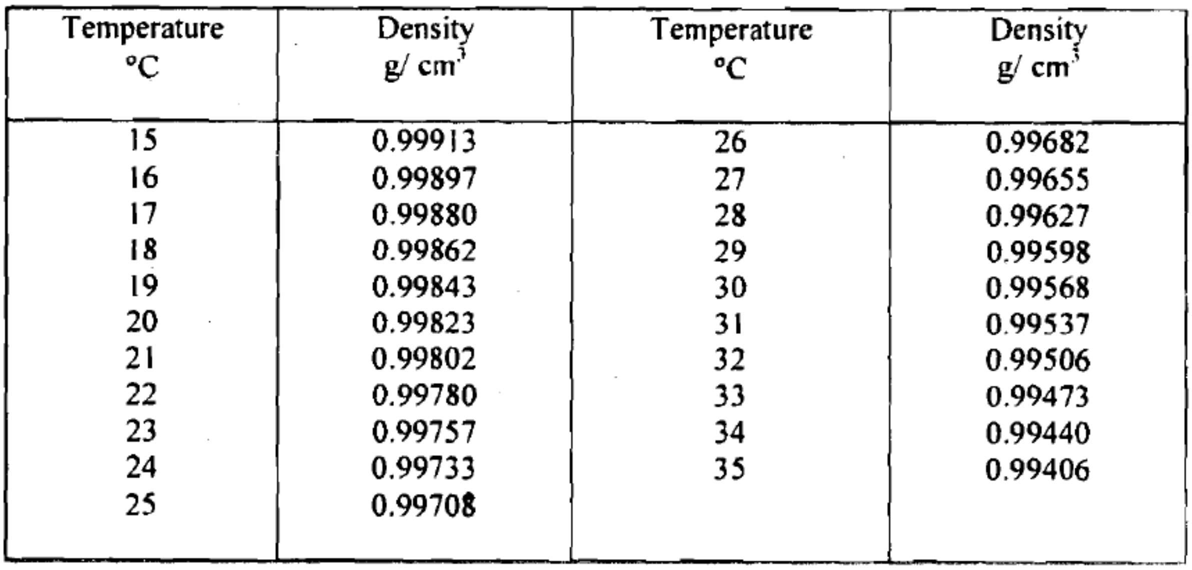
Chemicals: Distilled water

Procedure

Calibration of a Pipette:

Take a clean 100 cm3 (mL) conical flask fitted with a cork or stopper. Weigh the conical flask with cork accurately on an analytical balance and record its mass in your notebook. Pipette out 10/20/25 cm3 (equal to the capacity of the pipette u have) of distilled water from a beaker into the conical flask and weigh the flask with its contents. Record the temperature of the water with a thermometer. Calculate the mass of water delivered from the difference between the two readings. Repeat the procedure 3-4 times. Take the average mass and divide it by the density of water at room temperature to calculate the actual capacity of the pipette.

Values of the density of water at different temperatures are below



Temperature °C	Density g/ cm^3	Temperature °C	Density g/ cm^3
15	0.99913	26	0.99682
16	0.99897	27	0.99655
17	0.99880	28	0.99627
18	0.99862	29	0.99598
19	0.99843	30	0.99568
20	0.99823	31	0.99537
21	0.99802	32	0.99506
22	0.99780	33	0.99473
23	0.99757	34	0.99440
24	0.99733	35	0.99406
25	0.99708		

Calibration of a Burette

Take a clean 100 cm3 (mL) conical flask fitted with a cork. Measure its mass accurately to the nearest milligram on an analytical balance and record the mass. Clean the burette thoroughly and grease the stop-cock properly. Fill up the burette with distilled water and note the room temperature. Ad-just the water level to the zero mark. Touch the tip of the burette to the wall of a beaker to remove any adhering drop. Take the initial reading of the water level in the burette. Run 10 cm3 of distilled water into the conical flask. Touch the tip of the burette I to the wall of the flask. Cork the flask and weigh it accurately on an analytical balance and record the mass. The difference between this mass and the initial mass of the flask gives the mass of water delivered. Calculate the volume of water actually delivered by dividing the mass by the density of water. Repeat the process with 20 cm3, 30 cm3, 40 cm3, and 50 cm3 water, thus calibrating the burette over its entire length.

Calibration of a Volumetric Flask

Volumetric flasks may also be calibrated like that described for burettes and pipettes. But their large volume requires the use of an oversized analytical balance. Therefore, the volumetric flasks are calibrated by the procedure described below.

Take a clean 250 cm3 volumetric flask. Using a calibrated 25 cm3 pipette, carefully transfer 25 cm3 of distilled water 10 times in succession, to the flask. If the level of water in the flask does not correspond with the mark on the neck of the flask. paste a label with its upper edge on a level with the bottom of the meniscus of water. If you want to find the true volume of the water in the flask up to the graduation mark shown on the flask, you may do so with the help of a calibrated burette by transferring water exactly up to the graduation mark.

Observations

Calibration of a Pipette

i) Mass of the empty corked conical flask = $W_1\ g$

ii) Mass of the corked conical flask + distilled water = $W_2\ g$

iii) Temperature of water ………….. = t°C

iv) Density of water at t°C = d

Calibration of a Burette

i) Temperature of water = t°C

ii) Density of water at t°C = d

iii) Mass of the empty corked conical flask = $w_1\ g$

iv) Mass of the corked conical flask + 10 cm^3 distilled water = $w_2\ g$

v) Mass of the corked conical flask + 20 cm^3 distilled water = $w_3\ g$

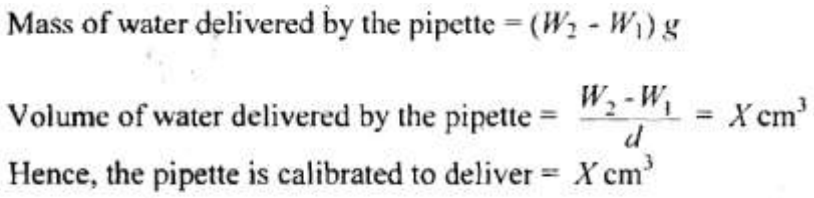
vi) Mass of the corked conical flask + 30 cm^3 distilled water = $w_4\ g$

vii) Mass of the corked conical flask + 40 cm^3 distilled water = $w_5\ g$

viii) Mass of the corked conical flask + 50 cm^3 distilled water = $w_6\ g$

CALCULATIONS:

Calibration of pipette

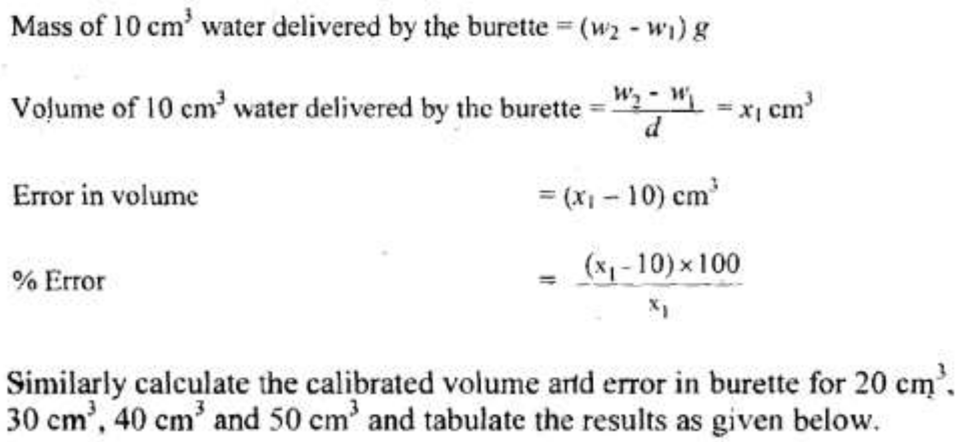


Mass of water delivered by the pipette = $(W_2 - W_1)\ g$

Volume of water delivered by the pipette = $\dfrac{W_2 - W_1}{d}$ = $X\ \text{cm}^3$

Hence, the pipette is calibrated to deliver = $X\ \text{cm}^3$

Calibration of burette



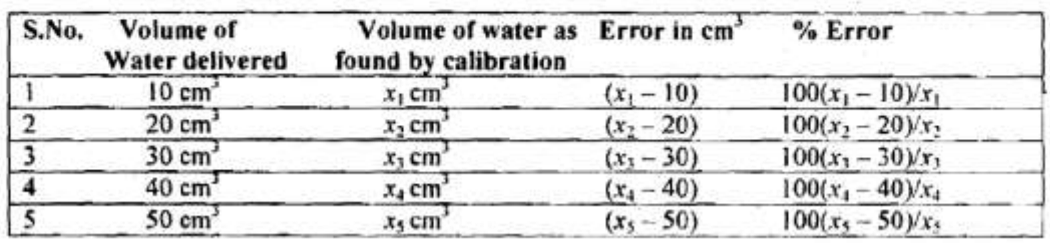
Mass of 10 cm^3 water delivered by the burette = $(w_2 - w_1)\ g$

Volume of 10 cm^3 water delivered by the burette = $\dfrac{w_2 - w_1}{d}$ = $x_1\ \text{cm}^3$

Error in volume = $(x_1 - 10)\ \text{cm}^3$

% Error = $\dfrac{(x_1 - 10) \times 100}{x_1}$

Similarly calculate the calibrated volume and error in burette for 20 cm^3, 30 cm^3, 40 cm^3 and 50 cm^3 and tabulate the results as given below.



S.No.	Volume of Water delivered	Volume of water as found by calibration	Error in cm^3	% Error
1	10 cm^3	$x_1\ \text{cm}^3$	$(x_1 - 10)$	$100(x_1 - 10)/x_1$
2	20 cm^3	$x_2\ \text{cm}^3$	$(x_2 - 20)$	$100(x_2 - 20)/x_2$
3	30 cm^3	$x_3\ \text{cm}^3$	$(x_3 - 30)$	$100(x_3 - 30)/x_3$
4	40 cm^3	$x_4\ \text{cm}^3$	$(x_4 - 40)$	$100(x_4 - 40)/x_4$
5	50 cm^3	$x_5\ \text{cm}^3$	$(x_5 - 50)$	$100(x_5 - 50)/x_5$

Summary:

In this experiment, you would have performed the following: calibrated a pipette, calibrated a burette, and calibrated a volumetric flask.

Experiment 2: Concepts of molecular weight, equivalent weight, various concentration terms, primary and secondary standard solutions and their preparations with proper explanations of types of apparatus to be used to prepare these solutions.

Prepare the following solutions

(i) Primary standard oxalic acid solution by accurate weighing

(ii) Primary standard potassium dichromate solutions by accurate weighing

(iii) Secondary standard sodium hydroxide solutions by approximate weighing

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(v) Secondary standard potassium permanganate solution by approximate weighing

(vi) Secondary standard sulphuric acid, hydrochlroric acid and acetic acid solutions of various strength from the supplied concentrated solutions.

Experiment 3: Study of kinetics of acid-catalyzed hydrolysis of ester

Theory:

The acid catalysed hydrolysis reaction of ester can be represented as

$$R_1COOR_2 + H_2O + H^+ \rightleftarrows R_1COOH + R_2OH$$

The rate equation is represented as

$$-\frac{d[R_1COOR_2]}{dt} = k[R_1COOR_2][H^+][H_2O]$$
$$= k_1[R_1COOR_2],$$

where $k_1 = k[H^+][H_2O]$ = constant, (in large excess of water, $[H_2O]$ remains constant and H^+ being the catalyst $[H^+]$ is constant)

Therefore the reaction becomes first order w.r.t ester. Integration of this equation with the boundary conditions, when t= 0, $[R_1COOR_2] = C_0$ and at time t, $[R_1COOR_2] = C$, gives

$$K_1 = (2.303/t)\log(C_0/C)$$

The progress of the catalysed reaction may be studied by withdrawing measured volume of aliquot from the reaction mixture at different intervals of time and titrating with standard alkali solution using phenolphthalein indicator. The volume of alkali required at any instant is equivalent to be the sum of weak acid (produced as a result of hydrolysis) and the acid used as catalyst (a constant quantity).

If V_0, V_n and V_∞ be the volumes of alkali required for the same volume of aliquot at the beginning, at time $t = t_n$ and at the end of the reaction (at infinite time) respectively then,

$$C_0 \propto (V_\infty - V_0) \text{ and } C \propto (V_\infty - V_n)$$

$$\text{Then, } k_1 = \frac{2.303}{t_n} \log \frac{(V\infty - V0)}{(V\infty - Vn)}$$

To avoid the measurement of V_0 the equation may be represented in the form

$$\Delta t_n = t_n - t_1 = \frac{2.303}{k_1} \log \frac{(V\infty - V1)}{(V\infty - Vn)}$$

Where V_n and V_1 are the volumes of alkali required at times t_n and t_1 respectively.

Plot of $\log \frac{(V\infty - V1)}{(V\infty - Vn)}$ vs Δt_n will give a straight line passing through the origin and the slope k_1 may be determined by

$$K_1 = \text{slope} \times 2.303$$

Apparatus required:

1) 100 mL dry conical flask-1
2) 500 mL bottle for NaOH.
3) 2 mL pipette; 5 mL pipette-1 each
4) 250 mL conical flask-5
5) Sufficient ice cold water; water bath
6) Stop watch

Chemicals required: Methyl acetate, NaOH, and Phenolphthalein

Procedure:

1) Prepare 250 mL of approximately 0.1(N) NaOH solution.
2) Prepare 100 mL 1(N) HCl solution
3) Pipette out 50 mL of prepared HCl solution (catalyst solution) in a 100 mL dry conical flask, and add 5 mL of ester using a pipette. Start the stopwatch at the time of half discharge. Mix the solution thoroughly by a swirling motion.
4) At 5-7 minute intervals, take a 2 mL aliquot and add to 50 mL ice-cold water taken in a 250 mL conical flask. Note the time of half discharge. Titrate rapidly against the prepared~ 0.1(N) NaOH solution taken in a burette, using phenolphthalein as an indicator. Take at least 6 readings.
5) The remaining solution is heated at about 60ºC in a water bath~for 40 minutes with an air condenser fitted in the mouth of the conical flask. The solution is allowed to cool to room temperature. Pipette out 2 mL of it in 50 mL of water taken in a 250 mL conical flask and titrate with~0.1(N) NaOH solution, using phenolphthalein as an indicator. The titre value corresponds to V∞.

6) Plot a graph of $log\frac{(V\infty - V1)}{(V\infty - Vn)}$ vs Δt_n and draw the best fit straight line passing through the origin.
7) Calculate the value of 'k_1' from the slope of the graph.

Experimental result:

1. Room temperature

2. Preparation of 250 mL~0.1(N) NaOH solution: Dissolve 1 g NaOH in 250 mL deionized water.

3. Preparation of 100 mL 1(N) HCl solution: Dissolve~9 mL conc. HCl in 91 mL deionized water.

4. Recording of data for ester hydrolysis:

Time(t)	Time(t_n)in sec	$\Delta t_n = t_n - t_1$	Volume of NaOH (V_n mL)	(V_∞-V_1) mL	(V_∞-V_n) mL	$log\frac{(V\infty - V1)}{(V\infty - Vn)}$

5. Determination of V_∞:

V_∞ = Volume of NaOH required to standardize 2 mL reaction mixture after heating at about 60ºC in a water bath.

Plotting of graph

$log\frac{(V\infty - V1)}{(V\infty - Vn)}$						
Δt_n(Sec)						

Calculation: Observed rate constant, k_1=slope×2.303

Conclusion: Therefore observed rate constant for the acid catalyzed hydrolysis of methyl acetate at …..ºC is…..sec^{-1}.

Experiment 4: Study of kinetics of decomposition of H_2O_2

Theory:

The reaction between H_2O_2 and KI in dilute H_2SO_4 medium is represented as

$$H_2O_2+2KI+H_2SO_4=I_2+2H_2O+K_2SO_4$$

Suggested reaction mechanism is

$$H_2O_2+I^-=H_2O+OI^- \quad \text{….(slow)}$$

$$OI^-+2H^++I^-=H_2O+I_2 \quad \text{…….(fast)}$$

The first step being the slowest step is the rate determining step. The differential rate becomes $-\frac{d[H_2O_2]}{dt}=k[H_2O_2][I^-]$, where k is the rate constant and other terms have their usual significance.

Therefore, kinetically the reaction is of second order (overall). But if the concentration of iodide ion is kept constant [by adding $Na_2S_2O_3$ continuously whereby the $S_2O_3^-$ ions react with the liberated iodine (I_2) and regenerate I^- according to the equation, $I_2+2\,S_2O_3^{2-}=S_4O_6^{2-}+2I^-$] the reaction becomes kinetically first order w.r.t.[H_2O_2] only. Under this condition the rate equation may be expressed as

$$-\frac{d[H_2O_2]}{dt}=k_1[H_2O_2],\ \text{where } k_1=k[I^-]$$

If the initial (at time t=0) concentration of H_2O_2 is a and at time t, $[H_2O_2]=(a-x)$, (where, x= amount of H_2O_2 reacted), integration of the above equation gives

$$K_1=\frac{2.303}{t}\log\frac{a}{a-x}$$

If both ‘a’ and (a-x) are represented in terms of their $Na_2S_2O_3$ equivalent, we have $V_0 \propto a$ (where V_0 is the titre value of thiosulphate for the total iodine liberated by a fixed volume of H_2O_2 solution of concentration ‘a’ after complete decomposition).

And $V_t \propto x$ (where V_t is the titre value of the same thiosulphate solution for the iodine liberated by the same H_2O_2 solution undergoing reaction at time t).

The rate equation may then be expressed as

$$K_1=\frac{2.303}{t}\log V_0/V_0-V_t$$

$$\text{Or, } \log V_0/V_0-V_t=k_1t/2.303$$

A plot of V_0/V_0-V_t vs. t will give a straight line passing through the origin and from the slope k_1 may be determined, k_1= slope× 2.303.

Apparatus required:

1) 500 mL conical flask-2
2) 500 mL glass bottle for thiosulphate-1
3) Burette-1
4) 250 mL volumetric flask for KI-1
5) 10 mL pipette
6) Watch glass
7) Stop watch

Chemicals required: H_2O_2, Sodium thiosulphate, KI, 12N H_2SO_4, 1% Ammonium molybdate solution, Starch

Procedure:

1. Prepare 100 mL ‘2 volume’ H_2O_2 solution. in a glass bottle.

2. Prepare 250 mL of 0.1(N) sodium thiosulphate solution in a glass bottle. Fill the burette with sodium thiosulphate solution.

3. Take 10 mL 12 (N) H_2SO_4 in a 500 mL conical flask. Add 10 mL of prepared H_2O_2 solution followed by addition of 50 mL of water. Then add approximately 2 gm soli KI. Add 2 mL 1% ammonium molybdate solution. Cover the conical flask with a watch glass and keep it in dark for 1 minute. Add about 50 mL of water and titrate the liberated iodine with sodium thiosulphate solution using starch as an indicator. Record the volume of thiosulphate (V_0).

4. Take 250 mL of 0.4% KI solution (dissolve 1 gm KI in 250 mL of deionized water) in a 500 mL conical flask. Add 15 mL 12(N) sulphuric acid and 5 L of freshly prepared starch solution. Add 10 mL of prepared H_2O_2 solutions and at the time of half discharge start stop watch. Run immediately sodium thiosulphate solution from the burette from the burette into the mixture in excess to discharge the blue colour. Wait for appearance of blue colour and record the time (t) of reappearance of colour, volume of thiosulphate used (V_t). Again run thiosulphate into the mixture in excess to discharge the blue colour, record time (t) of reappearance of colour, and total volume of thiosulphate used (V_t).Take at least 6 readings.

5. Plot log [V_0/(V_0-V_t)] vs. t in millimeter graph paper and draw the best fit straight line passing through the origin. Calculate ‘k_1’ value from the slope of the graph.

Experimental result:

1) Room temperature
2) Preparation of 250 mL~0.1(N) sodium thiosulphate solution: Dissolve~6.2 gm $Na_2S_2O_3$ in 50 mL deionized water.
3) Determination of V_0:

   V_0 = titre value of thiosulphate for the total iodine liberated by 10 mL of''2 volume' H_2O_2 solution after complete decomposition.
4) Titre value of thiosulphate at different times:

Time(t)	Time (t) in sec	Volume of $S_2O_3^{2-}$ used (V_t mL)	(V_0-V_t) mL	log V_0/V_0-V_t

5. Plotting of graph:

log V_0/V_0-V_t						
t(sec)						

Calculation: Observed rate constant, k_1=slope× 2.303

Conclusion: Therefore, the observed rate constant for the decomposition of H_2O_2 at ….. ⁰C is …….Sec^{-1}.

Experiment 5: Determination of specific rotation of cane sugar using polarimeter

Object:

To determine the specific rotation of cane sugar solution using Polarimeter.

Specific Rotation:

Specific rotation is the rotation produced by a column in a solution of an optically active substance one centimeter long, the concentration of the substance being one gram per c.c.

Apparatus Used:

Polarimeter, Electric bulb with housing, sugar, balance, graduated cylinder and thermometer.

Formula:

The specific rotation S of the plane of polarization of sugar dissolved in water.

$$S = \theta/lxc = \theta\ x\ v/l\ x\ m$$

Where, c = Concentration of the solution m/v = ….gm/cc

θ = Rotation produced in degree

l = Length of the tube in cm

m = Mass of sugar in g dissolved in water

v = Volume of sugar solution

Method:

1. Weigh 5 gm of sugar and carefully dissolve it is water to make up to 100cc of solution. The solution should be well mixed by pouring it from one jar to another so that it becomes of one uniform concentration. This gives a 5% solution of sugar.

2. Clean the glass tube, fill it with water and close the end, see that there are no air-bubbles in the tube and no water is sticking on the outside of the end glasses. Put the tube in position and rotate the analyzer. A till the field of view is completely and uniformly dark i.e. both halves are equally dark. Read the position of analyzer on the circular scale, provided for the purpose. A further rotation through 180o will again produce an extinction.

3. Remove the tube, empty it, rinse it with solution and then fill it completely, removing all bubbled. Clean the ends and put the tube in position. The two halves of the field will appear unequally dark.

4. Rotate the analyzer to produce extinction again and read the scale. This difference gives the rotation produced. Get a second position of extinction (equal darkness) by a further rotation of 180o and take mean for the rotation produced.

5. Now, to 50 c.c. of solution add 50 c.c of water and mix thoroughly. This gives a 5% solution and mix thoroughly. This gives a 5% solution. Take readings with it and then mix 50 c.c. of this solution with 50 c.c. of water again. This gives a 2.5% solution. Take readings with this also. Repeat with 1.25% solution.

Table for θ

Value of one division of mains scale =

No. of division on Vernier scale =

Least count of Vernier analyzer =

Table -1

S.NO.	% solution i.e. reading of analyzer with water						Mean $a = \frac{X+Y}{2}$
	Clockwise			Anticlockwise			
	M.S.	V.S.	Total 'X'	M.S.	V.S.	Total 'Y'	
1.							
2.							
3.							

Table – 2

S.NO.	Concentration of solution	% solution i.e. reading of analyzer with water						Mean $b = \frac{X'+Y'}{2}$	$(\theta) = a - b$
		Clockwise			Anticlockwise				
		M.S.	V.S.	Total 'X'	M.S.	V.S.	Total 'Y'		
1.	5% solution								
2.	2.5% solution								
3.	1.25% solution								

Calculations:

1. Draw a graph between θ and concentrations. The graph will be a straight line as shown in Fig.1.

2. Find out the value of θ for particular value of concentrations.

3. Calculate the value of specific rotations S as

S = $\theta\ x\ v/l\ x\ m$ ...°/cm/gm/cc.

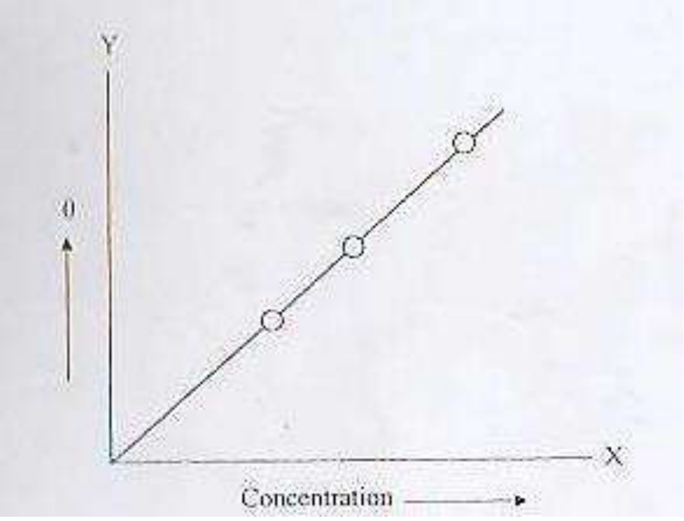


Fig. 2

Result:

1. The specific rotation of sugar solution (Solvent water) = …o/cm/gm/cc at ºC at λ = 5880 Ao.

2. Standard value of specific rotation of water = 66.5º.

3. Standard value of specific rotation of Glucose (Solvent water) = 52º.

Precautions and Source of Error:

1. The polarimeter tube should be well cleaned.

2. Water and sugar used should be dust free.

3. Whenever a solution is changed, risen the tube will new solution under concentration or clean the tube will water and dry it.

4. There is should be no bubble inside the tube.

5. The position of analyzer should be set accurately.

6. Readings should be taken when halves of the field of view becomes equally illuminated.

Experiment 6: Study the kinetics of inversion of cane sugar using polarimeter

Theory:

if a compound when placed in the path of plane of polarization it is said to be optically active and this property of rotating the plane of polarized light is called optical activity. If a compound rotates the plane of polarization in clock clockwise direction, facing the beam, it is called dextrorotatory, and if it rotates in an anti-clockwise direction, it is called laevorotatory. A solution of sucrose is optically active and dextrorotatory, while glucose and fructose are dextrorotatory and laevorotatory.

Principle:

An important characteristic of reaction is that sucrose is dextrorotatory, whereas glucose and fructose are dextrorotatory and laevorotatory. Further, the laevorotation of fructose is more (being-92º) than the dextro rotation of glucose (being -52.50), so that the mixture as a whole is laevorotatory. It means the product has a resultant levorotatory effect. Thus on hydrolysis, the dextrorotatory sucrose gradually changes into the levorotatory mixture. It is for this reason that the reaction is called the Inversion of Sucrose. The inversion of cane sugar is a pseudo-first-order reaction and represented by

$$C_{12}H_{22}O_{11} + H_2O \rightarrow C_6H_{12}O_6 + C_6H_{12}O_6$$

And the angle of rotations of Sucrose is (+66.6º), while that of Glucose and Sucrose are (-52.5º) and (-92º) respectively.

Materials:

Polarimeter, sample tube, flasks, beakers, sodium lamp and stirrer.

Chemicals:

Sugar solution and HCl.

Procedure:

Firstly, prepared 5% sucrose solution and 0.1 N HCl solution in separate flasks and placed them in water bath to maintain the temperature. Flasks should be closed with stopper to avoid any fumes. Then mixed equimolar solution of both HCl and sucrose. Temperature should be maintained. Took sample tube, cleaned its lens and poured sample solution in it form its one end and placed the sample tube in polarimeter, applied light source from one end and note the reading from the main scale and Vernier scale at time 0,7.18, 27.05 min and infinity, and noted the specific rotations at different time intervals.

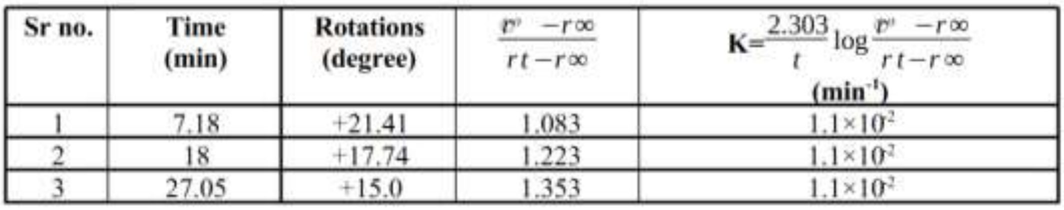
Observation and Calculation:

Reading of polarimeter at any time = r_t

Reading of polarimeter at zero time = r_0=+24.09

Reading of polarimeter at infinite time = r_∞ = -10.74

Table: Record of data to determine the rate constant for inversion of cane sugar.



Sr no.	Time (min)	Rotations (degree)	$\frac{r^0 - r\infty}{rt - r\infty}$	$K=\frac{2.303}{t}\log\frac{r^0 - r\infty}{rt - r\infty}$ (min^{-1})
1	7.18	+21.41	1.083	1.1×10^{2}
2	18	+17.74	1.223	1.1×10^{2}
3	27.05	+15.0	1.353	1.1×10^{2}

The average value of rate constant k = 1.1×$^{-2}$ min^{-1}.

Result:

As the value of k is constant, it indicates that the inversion of cane sugar is first order reaction.

Paper Title: Organic Chemistry-III (Practical)

Paper Code: CEMHMJ04P

Course contents:

Experiment: Qualitative Analysis of Single Solid Organic Compounds

a) Detection of special elements (N, S, Cl, Br) by Lassaigne's test

b) Solubility and classification (solvents: H2O, 5% HCl, 5% NaOH and 5% NaHCO3)

c) Detection of the following functional groups by systematic chemical tests: aromatic amino (-NH2), aromatic nitro (-NO2), amido (-CONH2, including imide), phenolic –OH, carboxylic acid (-COOH), carbonyl (-CHO and >C=O).

d) Melting point of the given compound

Qualitative Analysis of Single Solid Organic Compounds

(A) Physical Characteristics

(i) State of aggregation: solid

(ii) Texture: Crystalline or amorphous

(iii) Colour:

(iv) Odour:

(B) Melting Point:

(C) Solubility Test

Cold H_2O	Hot H_2O	dil. HCl	dil. $NaHCO_3$	dil. NaOH

Conclusion:

- From solubility and melting point inference can be drawn for the given organic sample.
- If the sample is soluble in dil. NaOH. The compound may contain the functional group –COOH or phenolic –OH or –COOH and phenolic –OH both.
- As the sample effervesces with $NaHCO_3$ solution i.e the compound is soluble in $NaHCO_3$ solution then the sample may contains –COOH group with or without other functional groups.

- As the sample is soluble in only dil. HCl then the compound is amine with or without other functional groups.
- As sample is soluble in both dil. HCl and dil. NaOH the compound may be aminophenol or amino acid.
- As the sample is not soluble in dil acid or alkali but only soluble in concentrated H_2SO_4. The compound may contain –NO_2 and or >C=O group.

(D) Detection of Special Elements

Lassaigne's Test:

Take a small piece of dry sodium metal in a fusion tube and heat it gently till the metal melts or fuses. Add equal quantity of compound to this fused metal. Heat it gently then strongly till it becomes red hot. Plung the red hot tube in 10 ml distilled water taken in a porcelain dish, crush the fusion tube completely. Carry out more fusion in the similar way. Boil the extract for five minutes, reduce the volume to about 5 ml and filter. Perform following test using this filtrate.

Test	Observation	Inference
Test for Nitrogen: Filtrate + freshly prepared FeSO4 soln. + boiled, cooled + acidified with dil. H2SO4 soln.	Prussian blue or green ppt or colour.	N-present
Test for Sulphur: 1 ml of filtrate + 1 drop of sodium nitroprusside solution.	A violet or purple coloration	S-present.
Test for Halogen: 1 ml of filtrate + conc. HNO_3, boiled, cooled, + $AgNO_3$ soln.	White ppt. which is soluble in dil. NH_4OH but reappears when acidified with HNO_3	Cl-present.

Conclusion: From the above experiment, is concluded the supplied organic sample contain N, S, and Cl as special elements.

(E) Systematic analysis of the functional groups:

1. Detection of Nitrogenous functional groups

Experiment	Observation	Inference

Test for aromatic-NH_2 group: Dye test: Solution-I: Sample is dissolved in dil. HCl and cooled in an ice-bath. Solution-II: Dil. $NaNO_2$ soln. is cooled in an ice-bath. Solution-III: Alkaline β-napthol soln. is cooled in an ice-bath. The mixture of solution-I & II is cooled in an ice-bath. After some time alkaline β-napthol soln. is added to the mixture of solution-I & II.	Red or orange–red dye	Ar-NH_2 group confirmed.
Test for -NO_2 group: Mulliken and Barker's test: Alcoholic soln. of sample + solid NH4Cl + Zn-dust boiled for few minutes, cooled allowed to stand for 5 minutes, and filtered. Filtrate + Tollen's reagent, warmed in a water bath.	Silver mirror or black or grey ppt.	Ar-NO_2 group confirmed.
Test for -$CONH_2$ group: Hydrolysis test: sample + Conc. NaOH solution, heated.	Characteristic smell of NH3 which turns mercurous nitrate paper black.	-$CONH_2$ group confirmed.

2. Detection of Non-Nitrogenous functional groups:

Experiment	Observation	Inference
Test for -COOH group: (i) NaHCO3 Test: Saturated solution of sodium bicarbonate under heating, cool + pinch of sample. **(ii) Esterification test**: 1.0 g sample + 5 ml ethanol + 2-3 concentrated H_2SO_4, boiled in a water bath for 10 minutes. The mixture is then poured into 150 ml water.	Effervescence of CO_2 gas. A sweet smell of ester.	-COOH group may be present. -COOH group is present and confirmed.
Test for phenolic-OH group: (i) **$FeCl_3$ Test:** alcoholic or aqueous solution of sample + $FeCl_3$ solution.	Violet or green or blue colouration.	Phenolic-OH group may be present.

(ii) Back dye test: Solution-I: small amount of aniline is dissolved in dil. HCl, cooled in ice-bath. Solution-II: $NaNO_2$ solution, cooled in ice-bath. Solution-III: alkaline solution of sample, cooled in ice-bath. The solution-I & II are mixed and kept for some time. The solution-III is added to the mixture of solution-I & II.	Orange-red dye.	Phenolic-OH group present and confirmed.
Test for carbonyl group: 2 g sample is dissolved in minimum quantity of ethanol + 5 cc saturated solution of 2,4-dinitrophenylhydrazone, warmed, scratched with glass rod.	Yellow or orange ppt.	Carbonyl (>C=O or –CHO) group is present and confirmed.
Test for -CHO group: (i) Fehlings's test: sample + Fehlings's solution (Fehlings-I & Fehlings-II), heated in water bath. **(ii) Tollen's Test:** sample + Tollen's reagent, heated in water bath.	Yellow or red ppt. Silver mirror or grey ppt.	-CHO group present and confirmed. -CHO group present and confirmed.

Highlights:

1. During Lassaigne's test a dark coloured sodium extract is obtained either due to incomplete fusion of the organic compound or when it is taken in excess of the sodium taken. Again if sodium is not used in excess during the experiment, on pouring the fusion tube into water no sodium hydroxide will be formed and the solution will not be alkaline. So green precipitate of ferrous hydroxide will not be observed during Prussian blue test and the test for N will fail.

2. If the solution is not alkaline sodium nitroprusside test will also fail.

Above two tests can be performed by adding sodium hydroxide solution to the filtrate if the solution is not alkaline.

3. The solution will give a black precipitate on addition of ferrous sulphate if sulphur is present in the organic sample.

4. The ferrous sulphate solution should be freshly prepared. A little amount of ferrous sulphate is washed with distilled water to remove ferric sulphate is more soluble than ferrous sulphate. Next the sulphate is dissolved to get freshly prepared ferrous sulphate solution. A dilute $FeSO_4$ on standing is oxidized to basic Ferric Sulphate which becomes useless in analysis.

5. The addition of Ferric chloride solution and HCL is avoided during Prussian blue test as it forms a green precipitate or colour. Concentrated sulphuric acid, in turn, will give a fine blue precipitate or colouration as concentrated sulphuric acid will oxidise the excess ferrous sulphate present in solution to ferric sulphate and those ferric icons will yield Prussian blue in fine blue colour.

6. It is essential to boil the sodium extract with HNO_3 to remove CN- as voilatole HCN. Otherwise it will give give ppt of AgCN similar to AgCl and misguide the analysis.

$$NaCN + AgNO_3 = AgCN + NaNO_3$$

$$NaCN + HNO_3 = HCN + NaNO_3$$

Paper Title: Preparation of Household Chemicals (Practical)

Paper Code: CEMSEC03

Course contents:

Part A:

(i) Preparation of liquid disinfectant

(ii) Preparation of Liquid Detergent

(iii) Preparation of Dish wash Liquid Soap

(iv) Preparation of Hand Sanitizer

(v) Preparation of simple organophosphates, phosphonates and thiophosphates

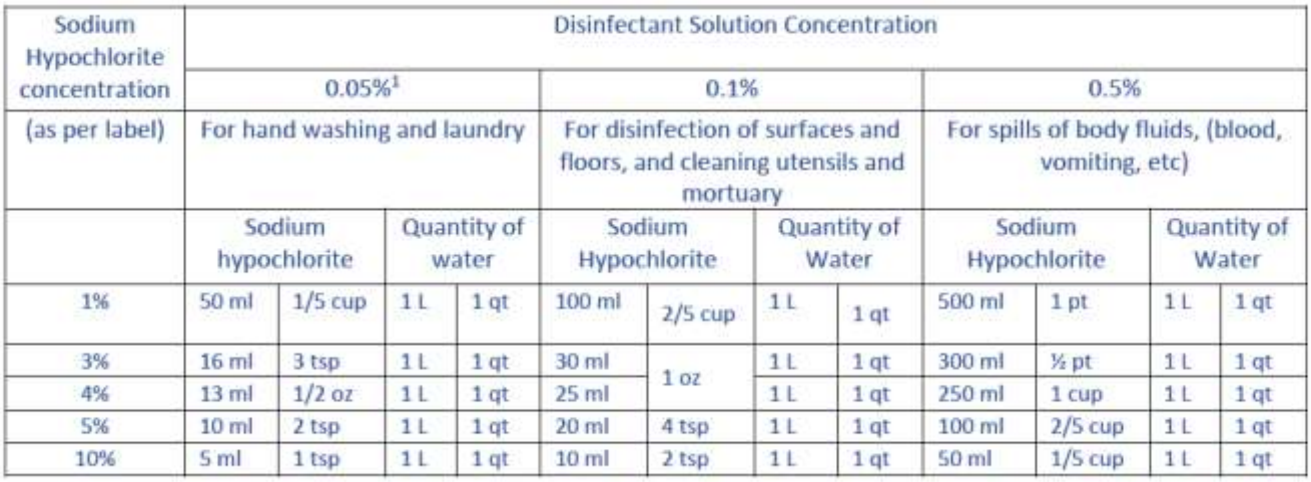
Part B: Field visit and submission of the report

(i) Preparation of liquid disinfectant

HOW TO PREPARE DISINFECTANT SOLUTIONS

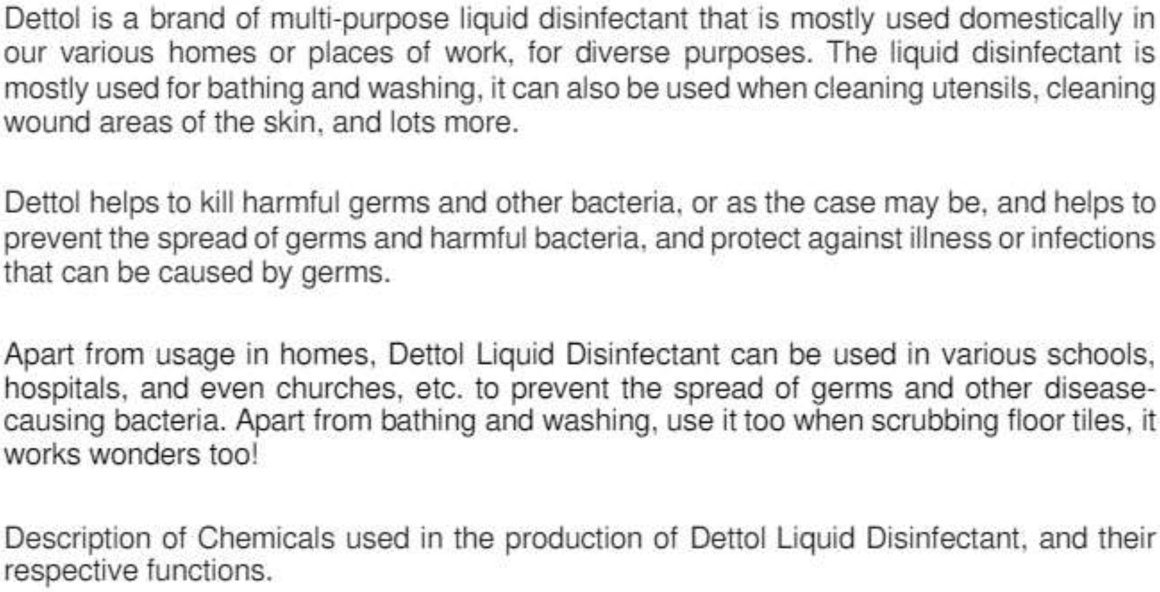
- Use the concentration of sodium hypochlorite available in the country, as indicated on the label of the container, to mix the concentration of disinfection solution according to the intended use, following the indications in the table below.
- Use protective equipment such as gloves, mask, eye protection, and apron for mixing the solutions.
- Prepare solutions daily. If you don't need a liter per day, make adjustments to the amounts in the table below.
- Use a dedicated container for the prepared solutions, and mark (label) the container with the concentration clearly.
- Do not reuse packaging of edible products to prepare the disinfectant solution to avoid accidental poisoning.
- Add the quantity of sodium hypochlorite to the quantity of water as indicated in the table below.
- Do not mix sodium hypochlorite with soaps or cleaners.

- ➢ Store sodium hypochlorite out of the reach and sight of children, in a safe place that is not exposed to light and heat.
- ➢ Use sodium hypochlorite products without added fabric softeners, colors, or perfumes.



Sodium Hypochlorite concentration	Disinfectant Solution Concentration											
	0.05%[1]				0.1%				0.5%			
(as per label)	For hand washing and laundry				For disinfection of surfaces and floors, and cleaning utensils and mortuary				For spills of body fluids, (blood, vomiting, etc)			
	Sodium hypochlorite		Quantity of water		Sodium Hypochlorite		Quantity of Water		Sodium Hypochlorite		Quantity of Water	
1%	50 ml	1/5 cup	1 L	1 qt	100 ml	2/5 cup	1 L	1 qt	500 ml	1 pt	1 L	1 qt
3%	16 ml	3 tsp	1 L	1 qt	30 ml	1 oz	1 L	1 qt	300 ml	½ pt	1 L	1 qt
4%	13 ml	1/2 oz	1 L	1 qt	25 ml		1 L	1 qt	250 ml	1 cup	1 L	1 qt
5%	10 ml	2 tsp	1 L	1 qt	20 ml	4 tsp	1 L	1 qt	100 ml	2/5 cup	1 L	1 qt
10%	5 ml	1 tsp	1 L	1 qt	10 ml	2 tsp	1 L	1 qt	50 ml	1/5 cup	1 L	1 qt

What is Dettol and what is it used for?



Dettol is a brand of multi-purpose liquid disinfectant that is mostly used domestically in our various homes or places of work, for diverse purposes. The liquid disinfectant is mostly used for bathing and washing, it can also be used when cleaning utensils, cleaning wound areas of the skin, and lots more.

Dettol helps to kill harmful germs and other bacteria, or as the case may be, and helps to prevent the spread of germs and harmful bacteria, and protect against illness or infections that can be caused by germs.

Apart from usage in homes, Dettol Liquid Disinfectant can be used in various schools, hospitals, and even churches, etc. to prevent the spread of germs and other disease-causing bacteria. Apart from bathing and washing, use it too when scrubbing floor tiles, it works wonders too!

Description of Chemicals used in the production of Dettol Liquid Disinfectant, and their respective functions.

1. Texapon:

The Taxapon is a jellylike and pasty substance, it functions as a foaming agent in the production. The Texapon is a detergent that produces micelles that allows non-polar substance such as oils to be dissolved in water.

2. Phenol:

Phenol, also known as hydroxyl benzene is a colorless aromatic compound with a very sweet smell (it sometimes, comes in brownish color too). It is one of the disinfectants in used in the production of Dettol; however, in small concentrations. Phenol can also be used as a mouth wash.

.3. **Chloroxylenol**

This is also known as para-chloro-meta-xylenol (or PCMX), it is the main antiseptic agent in the production of Dettol, and helps to kill germs and for disinfection of the skin. Also used together with alcohol for cleaning and disinfecting surgical instruments.

In higher concentrations, Chloroxylenol may cause some form of irritation to the skin and inflammation, so care must be taken to ensure the measurement during the production of the Dettol.

4. **Isopropyl Alcohol**:

Isopropyl Alcohol also known as IPA, is also one of the vital ingredients used in the production of Dettol Liquid Disinfectant. It is a colorless compound, with a very strong smell. Its powerful antiseptic effects make Dettol have the ability to fight and kill bacteria, fungi, and viruses, especially in wound treatment.

5. **Pine oil**:

Pine oil is an extract from Pine trees. It is also an antiseptic and disinfectant substance with a very pleasant smell and can also be used as a healing oil for cold. Pine oil also helps to relieve skin inflammation. When in water, it emulsifies (i.e. forms a cloudy solution.) It makes the Dettol form that cloudy solution when dropped in water.

The use of Pine oil is very verse and not limited to the above-mentioned, it can also be used for bathing antiseptic soaps, bath oils, etc. It can be effective too in the healing of arthritis.

Required Measurement For The Production Of 5 Liters Of Dettol Liquid Disinfectant

1. TEXAPON ¼ kg
2. PINE OIL ½ liter
3. PHENOL 1/8 liter
4. CHLOROXYLENOL ¼ liters
5. ISOPROPYL ALCOHOL (IPA) 2 liters
6. WATER (Distilled) 2litres
7. COLORANT as desired.

Note, emplace of water, you may use methanol or ethanol; or the mixture of both (i.e. water plus methanol). We only need them to properly dissolve other constituents in the production of Dettol Liquid Disinfectant

More so, if you desire to produce more volumes of the Dettol Liquid Disinfectants, all you need do is to play around with your ratios. For example, you can double the above measurements for a 10 Liters production of the Dettol Liquid Disinfectant, and so on. Consider what we have as the basic measurement.

Production Procedure For The Dettol Liquid Disinfectant.

Safety Tips During Production:

- Please do note that the production should never be so urgent or so important that you don't take the time to carry it out safely.
- Make sure you put on the right protections before commencing the production. The need for use of rubber hand gloves, nose masks, face shields, etc., cannot be overemphasized.
- Carry out all production and an open and well-ventilated environment. Also, make sure it is well illuminated.
- In the event of a chemical splash to the skin or other part of the body exposed, quickly wash with clean running water, then proceed immediately to see medical personnel for further checks.
- Please do not taste any chemical, or directly inhale it.
- Please keep all chemicals away from the reach of children.

Equipment & Tools

- Clean Bowl that can contain 5liters volume without spillage. Preferably a plastic bowl or container.
- A wooden or plastic stirrer
- Funnel
- Packaging containers

Simple Step-By-Step Guide

Step 1: Pour the Texapon into empty clean bowl.

Step 2: Add the Pine oil and stir properly until the Texapon is dissolved completely.

Step 3: Add Phenol to the mixture and stir properly

Step 4: Add Chloroxylenol and stir properly. Note the mixture will turn slightly brownish.

Step 5: Gently pour the amount of water and stir gradually. The mixture will immediately become cloudy or whitish; this is normal.

Step 6: Now add the Isopropyl Alcohol (IPA). Continue to stir until the mixture turns brownish again, to get a perfect blend.

Step 7: Dissolve the color (brown) in a little quantity of water and mix well. Then add bit by bit into the main bowl, while stirring continuously until the desired coloration is reached. Note that this should be the last constituent to be added. Stir very well.

(ii) Preparation of Liquid Detergent

Introduction

Liquid Detergent is generally a cleaning chemical that comes in the form of liquid. It is Easy and convenient to use, Liquid Detergents are generally meant for cleaning of clothes. They are one of the most popular and widely used cleaning liquid for Clothes & some other purpose. Liquid Detergent are chemical solutions used as cleaning agents. They are generally formulated using one or more surfactants to improve their cleaning performance. Liquid Detergent have become an essential commodity in our daily lives. Cleaning products play an essential role in our daily lives. By safely and effectively removing soils, germs and other contaminants, they help us to stay healthy, care for our homes and possessions.

Qualities of good Liquid Detergent

1. It must have attractive scent or perfume.
2. It must not itch your hand or body.
3. Remove germs from the clothes.

Formulation

Major Component of liquid detergent is based on surfactants that remove soil from the fabric and clean it. A combination of anionic and non-ionic surfactants is commonly used for optimum performance. In general, anionic are good for particulate soil removal and non-ionic are good for greasy soil removal. Other components of a liquid laundry detergent formula are detailed in this article. Thus, a liquid laundry detergent formula can be as simple and economical as say 5% active surfactant solution in water with preservatives. The formula will foam and do some cleaning of the clothes. From here one can improve this basic formula to any desired level in order to enhance the performance and position it for various categories such as economical, mid-tier, premium, ultra, and 2X, 3X, 4X etc. Liquid Detergent is formulated using batch or continuous process of Liquid making. These cleansing products contain different ingredients that are used to improve their cleaning performance. The surfactant plays an important role in improving the cleansing action of liquid by reducing the surface tension of wash liquid.

Ingredients:

The important ingredients of Liquid detergent includes- Acid Slurry, caustic soda, AOS, Perfume for fragrance, water.

Liquid Detergent Manufacturing Process:

It is a neutralization process of acid slurry by caustic soda keeping PH 7 to 9.

- Firstly transfer water into the stainless steel tank manually or through pump.
- Now add acid slurry into the tank to turn water into desired colour.
- After that it is neutralized by the aqua solution of caustic soda to get the required PH.
- Now AOS is added into the solution, it is used as a foam booster.
- While urea is used as a buffering agent and also create transparency.
- Lastly add colours & perfume.
- Filling of Liquid detergent in bottle.
- Packaging of goods.

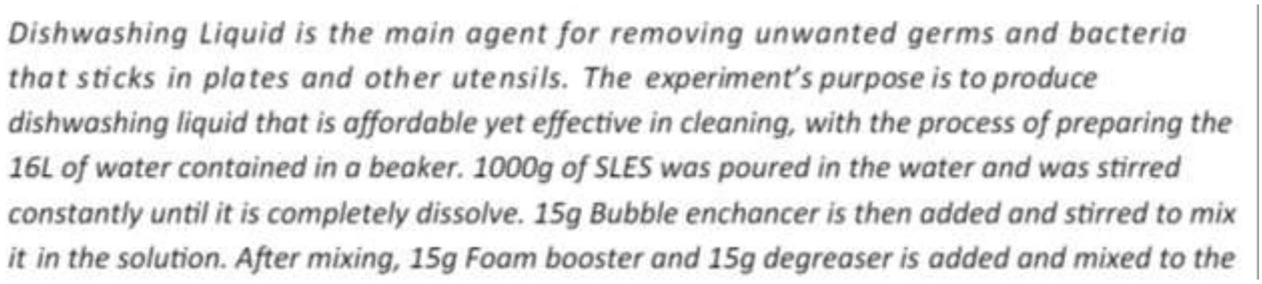
Raw Material Requirement of Liquid Detergent

- Acid Slurry (alkyl benzene sulphonate (ABS) or linear alkyl benzene sulphonic acid (LABSA)
- Caustic Soda
- AOS (Alpha Olefin Sulfonate)
- Urea
- Colour
- Perfume
- Corrugated Boxes
- Water

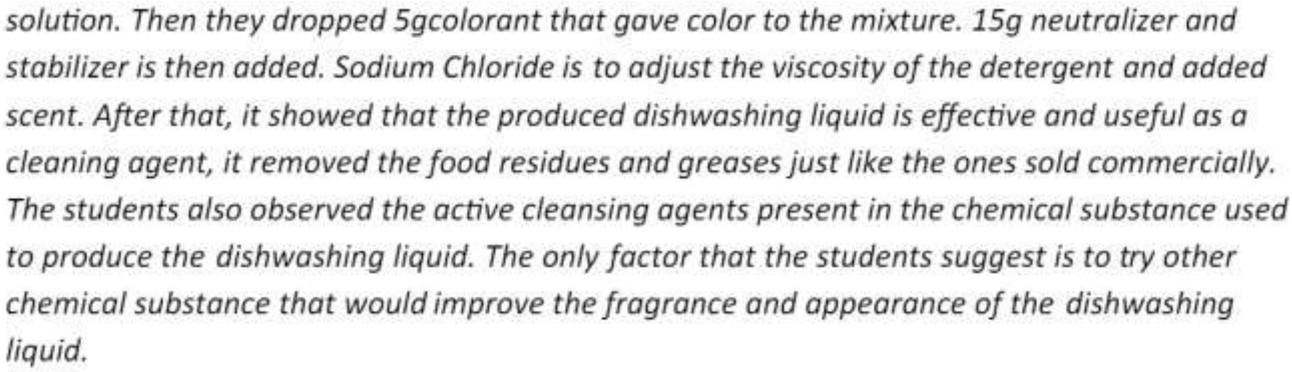
Conclusion:

After completion of manufacturing process, product is ready to sell in the market. Liquid Detergent are used for daily routine activities. This machine can be installed with low investment & one can earn a good Margin of profit by doing this business.

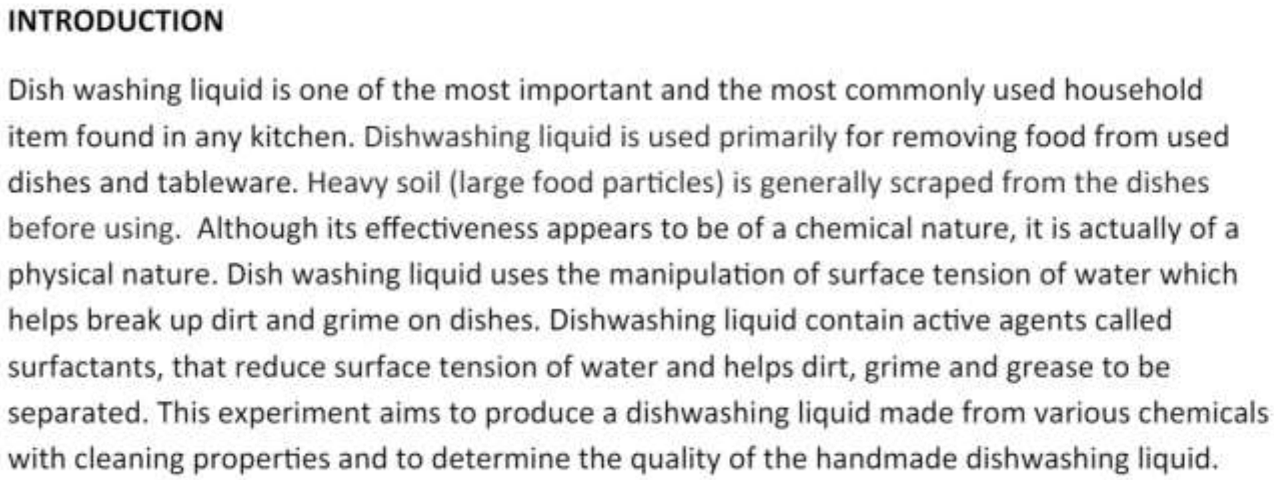
(iii) Preparation of Dish Wash Liquid Soap



Dishwashing Liquid is the main agent for removing unwanted germs and bacteria that sticks in plates and other utensils. The experiment's purpose is to produce dishwashing liquid that is affordable yet effective in cleaning, with the process of preparing the 16L of water contained in a beaker. 1000g of SLES was poured in the water and was stirred constantly until it is completely dissolve. 15g Bubble enchancer is then added and stirred to mix it in the solution. After mixing, 15g Foam booster and 15g degreaser is added and mixed to the



solution. Then they dropped 5gcolorant that gave color to the mixture. 15g neutralizer and stabilizer is then added. Sodium Chloride is to adjust the viscosity of the detergent and added scent. After that, it showed that the produced dishwashing liquid is effective and useful as a cleaning agent, it removed the food residues and greases just like the ones sold commercially. The students also observed the active cleansing agents present in the chemical substance used to produce the dishwashing liquid. The only factor that the students suggest is to try other chemical substance that would improve the fragrance and appearance of the dishwashing liquid.



INTRODUCTION

Dish washing liquid is one of the most important and the most commonly used household item found in any kitchen. Dishwashing liquid is used primarily for removing food from used dishes and tableware. Heavy soil (large food particles) is generally scraped from the dishes before using. Although its effectiveness appears to be of a chemical nature, it is actually of a physical nature. Dish washing liquid uses the manipulation of surface tension of water which helps break up dirt and grime on dishes. Dishwashing liquid contain active agents called surfactants, that reduce surface tension of water and helps dirt, grime and grease to be separated. This experiment aims to produce a dishwashing liquid made from various chemicals with cleaning properties and to determine the quality of the handmade dishwashing liquid.

To make your own dishwashing liquid, simply mix the following ingredients in a clean container:

How to make a liquid dishwashing soap?

- 1 cup of distilled water.
- 1/4 cup of liquid castile soap.
- 1 tablespoon of white vinegar.
- 1 tablespoon of washing soda.
- 5-10 drops of essential oil (optional, for fragrance)

OR

What is the formula for making dishwashing liquid?

In a large bowl, mix together 1 tablespoon (2 g) of Borax, 1 tablespoon (14 g) of washing soda, 2 tablespoons (30 ml) of white distilled vinegar, and ½ cup (120 ml) liquid castile soap. Stir well to ensure that all of the ingredients are well blended.

How to make dishwashing liquid at home: Procedure

- Add your washing soda or baking soda into a bowl with the grated soap. ...
- Heat water to a boil and pour it into the bowl of washing soda and grated soap.
- Whisk the mixture until it is blended thoroughly. ...
- Add a few drops of lemon and lavender essential oil.

What makes a good dishwashing liquid?

For clean dishes and a happy planet, a good dishwashing liquid should be hypoallergenic and free of phosphates, and triclosan. It should not be tested on animals and the packaging should be made from recycled materials or come in a bulk container.

INGREDIENTS:

- Water
- Sodium Hydroxide (Caustic Soda)
- BIO-SOFT® S-118
- STEOL® CS-230
- AMMONYX® LMDO
- Citric Acid, 50% Solution
- Sodium Chloride
- Preservative, Dye, Fragrance, etc.

(iv) Preparation of Hand Sanitizer:

This formulation prescribed the requirement for alcohol based hand sanitizers (hand rub) taking into the WHO recommendation. This does not cover non-alcohol based hand sanitizer.

Alcohol-based hand rubs are the only known means for rapidly and effectively inactivating a wide array of potentially harmful microorganisms on hands. WHO recommends alcohol-based hand rubs based on the following factors:

- Evidence-based, intrinsic advantages of fast-acting and broad-spectrum microbicidal activity with a minimal risk of generating resistance to antimicrobial agents;
- Suitability for use in resource-limited or remote areas with lack of accessibility to sinks or other facilities for hand hygiene (including clean water, towels, etc.);
- Capacity to promote improved compliance with hand hygiene by making the process faster and more convenient;
- Economic benefit by reducing annual costs for hand hygiene, representing approximately 1% of extra-costs generated by Health Care-Associated Infection;
- Minimization of risks from adverse events because of increased safety associated with better acceptability and tolerance than other products.

Health-care a setting currently using commercially-available hand rubs should continue to use them, provided that they meet recognized standards for microbicidal efficacy and are well accepted/tolerated by Health Care-Workers. It is obvious that these products should be regarded as acceptable, even if their contents differ from those of the WHO-recommended formulations.

Ingredients for 10,000 mL

- Ethanol 96% 8333 mL or
- If Ethanol is used, denaturing is recommended

- Isopropyl Alcohol 99% 7576 mL or
- Isopropyl Alcohol 91% 8242 mL

  - When the concentration of alcohol (e.g., ethanol or isopropyl alcohol) in the starting ingredient is not exact, the calculation should be adjusted accordingly to ensure a final concentration of at least 80% ethanol or 75% isopropyl alcohol
  - Volume of starting ingredient required = (final % alcohol) × (final volume of preparation) /(starting % alcohol)
  - Methanol contamination of Alcohol
  - Serious safety concern
  - Substantial methanol exposure can result in nausea, vomiting, headache, blurred vision, permanent blindness, seizures, coma, permanent damage to the nervous system or death
  - Methanol must not be used as an ingredient or as a denaturant
  - Methanol content must not exceed 630 ppm
  - Methanol NMT 630 ppm
  - Benzene NMT 2 ppm
  - Acetaldehyde NMT 50 ppm
  - Acetal (1,1-diethoxyethane) NMT 50 ppm
  - Sum of all other impurities NMT 300 ppm

Composition of Alcohol-Based Hand Rubs Formulations:

The following two formulations are recommended for local production with a maximum of 50 liters per lot to ensure safety in production and storage.

Formulation I:

Final concentrations of ethanol 80% v/v, glycerol 1.45% v/v, hydrogen peroxide 0.125% v/v. Pour the following ingredients as mentioned in table-1 into a graduated flask/ container:

a. Ethanol (with a purity 96% v/v),

b. Hydrogen Peroxide 3% v/v,

c. Glycerol 98% v/v,

Top up the flask to volume required with distilled water or water that has been boiled and cooled; shake the flask gently to mix the content.

Formulation II:

Final concentrations of isopropyl alcohol 75% v/v, glycerol 1.45% v/v, hydrogen peroxide 0.125% v/v: Pour the following ingredients of required volume as mentioned in table-2 into graduated flask/ container.

a. Isopropyl alcohol (with a purity of 99.8% v/v),

b. Hydrogen Peroxide 3% v/v, c. Glycerol 98% v/v,

Top up the flask to volume required with distilled water or water that has been boiled and cooled; shake the flask gently to mix the content. The % purity of the ingredients may vary. For volume of the ingredients to be taken is calculated by using the following formula.

$$\text{Volume of ingredients to be taken in ml} = \frac{(\text{\% value of ingredients to be made available in Sanitizer}) \times (\text{Volume of Sanitizer to be Prepared in ml})}{\text{Purity \% value}}$$

The examples of calculation by using above formula for volume of 1000 ml, 10000 ml and 50000 ml of preparation of hand sanitizers are given in the table 1 and 2.

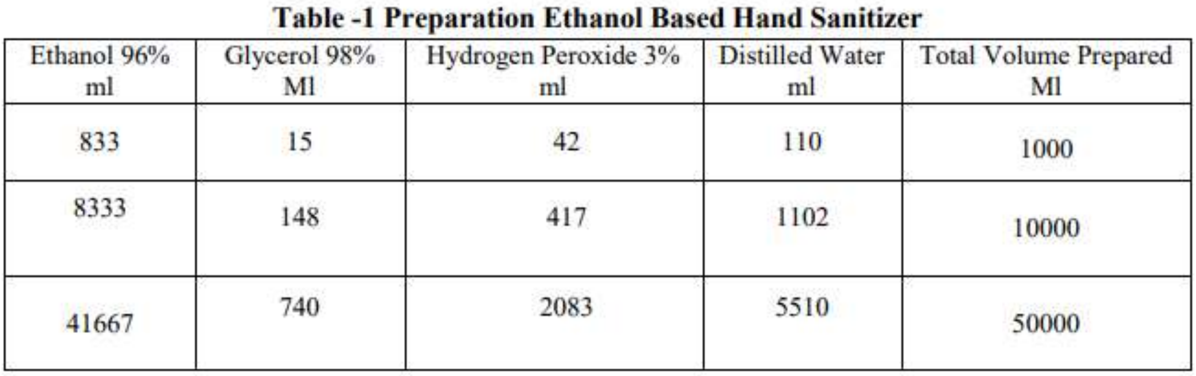


Table -1 Preparation Ethanol Based Hand Sanitizer

Ethanol 96% ml	Glycerol 98% Ml	Hydrogen Peroxide 3% ml	Distilled Water ml	Total Volume Prepared Ml
833	15	42	110	1000
8333	148	417	1102	10000
41667	740	2083	5510	50000

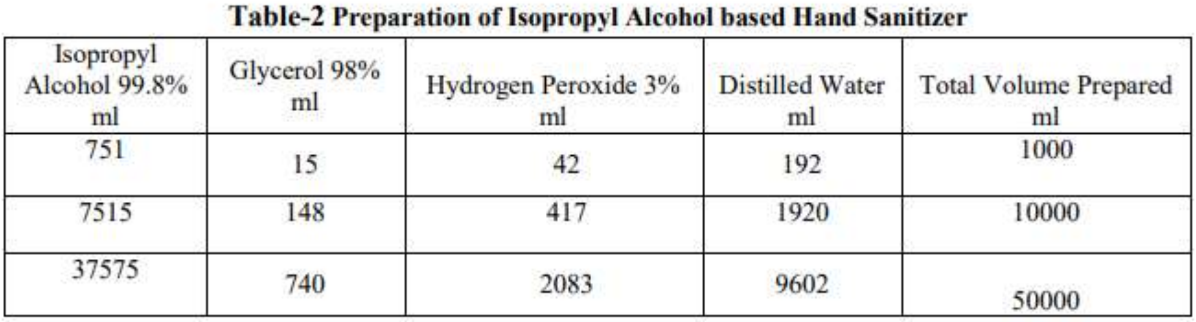


Table-2 Preparation of Isopropyl Alcohol based Hand Sanitizer

Isopropyl Alcohol 99.8% ml	Glycerol 98% ml	Hydrogen Peroxide 3% ml	Distilled Water ml	Total Volume Prepared ml
751	15	42	192	1000
7515	148	417	1920	10000
37575	740	2083	9602	50000

Only pharmacopoeia quality reagents should be used.

Production Method

Volume of Production, Containers

10-litre preparations: glass or plastic bottles with screw threaded stoppers can be used.

50-litre preparations: large plastic (preferably polypropylene, translucent enough to see the liquid level) or stainless steel tanks with an 80 to100 liters capacity should be used to allow for mixing without overflowing. The tanks should be calibrated for the ethanol/isopropyl alcohol volumes and for the final volumes of either 10 or 50 liters. It is best to mark plastic tanks on the outside and stainless steel ones on the inside.

Preparation

a. The alcohol for the chosen formulation is poured into the large bottle or tank up to the graduated mark.

b. H_2O_2 is added using the measuring cylinder.

c. Glycerol is added using a measuring cylinder. As the glycerol is very viscous and sticks to the walls of the measuring cylinder, it can be rinsed with some sterile distilled or cold boiled water to be added and then emptied into the bottle/tank.

d. The bottle/tank is then topped up to the corresponding mark of the volume (10-litre or 50-liter) to be prepared with the remainder of the distilled or cold, boiled water.

e. The lid or the screw cap is placed on the bottle/tank immediately after mixing to prevent evaporation.

f. The solution is mixed by gently shaking the recipient where appropriate (small quantities), or by using a wooden, plastic or metallic paddle. Electric mixers should not be used unless "EX" protected because of the danger of explosion.

g. After mixing, the solution is immediately divided into smaller containers (e.g. 1000, 500 or 100 ml plastic bottles). The bottles should be kept in quarantine for 72 hours. This allows time for any spores present in the alcohol or the new or re-used bottles to be eliminated by H_2O_2.

Quality Control

If concentrated alcohol is obtained from local production, verify the alcohol concentration and make the necessary adjustments in volume to obtain the final recommended concentration. An alcoholmeter can be used to control the alcohol concentration of the final use solution; H O concentration can be measured by titrimetric (oxidation-reduction reaction by iodine in acidic conditions). A higher level quality control can be performed using gas chromatography and the titrimetric method to control the alcohol and the hydrogen peroxide content, respectively.

Moreover, the absence of microbial contamination (including spores) can be checked by filtration.

Labelling

Labels should include the following information:

- Name of producer
- Date of production and batch number
- Composition: ethanol or isopropanol, glycerol and hydrogen peroxide (% *v/v* can also be indicated) and the following statements:
- For external use only
- Avoid contact with eyes
- Keep out of reach of children
- Use: apply a palmful of alcohol-based hand rub and cover all surfaces of the hands. Rub hands until dry. Flammable: keep away from flame and heat.

Ingredients

a. Hydrogen Peroxide

While alcohol is the active component in the formulations, certain aspects of other components should be respected. All raw materials used should be preferably free of viable bacterial spores. The low concentration of H O is incorporated in the formulations to help eliminate contaminating spores in the bulk solutions and excipients and is not active substance for hand antisepsis. While the use of H_2O_2 adds an important safety aspect, the use of 3–6% of H_2O_2 for the production might be complicated by its corrosive nature and by difficult procurement in some countries. Further investigation is needed to assess H O availability in different countries as well as the possibility of using a stock solution with a lower concentration.

b. Glycerol

Glycerol is added to the formulation as a humectant to increase the acceptability of the product. Other humectants or emollients may be used for skin care, provided that they are affordable, available locally, miscible (mixable) in water and alcohol, non-toxic, and hypoallergenic. Glycerol has been chosen because it is safe and relatively inexpensive. Lowering the percentage of glycerol may be considered to further reduce stickiness of the hand rubs.

c. Other Additives

It is strongly recommended that no ingredients other than those specified here be added to the formulations. In the case of any additions, full justification must be provided together with documented safety of the additive, its compatibility with the other ingredients, and all relevant details should be given on the product label.

A colorant may be incorporated to differentiate the hand rub from other fluids as long as such an additive is safe and compatible with the essential components of the hand rubs. However, the H O in the hand rub may tend to fade any coloring agent used and prior testing is recommended. The addition of fragrances is not recommended because of the risk of allergic reactions. To further reduce the risk of abuse and to respect cultural and religious sensitivities, product containers may be labelled simply as “antimicrobial hand rub.

d. Water

While sterile distilled water is preferred for making the formulations, boiled and cooled tap water may also be used as long as it is free of visible particles.

Production and Storage

Special requirements apply for the production and stock piling of the formulations, as well as for the storage of the raw materials. Because undiluted ethanol is highly flammable and may ignite at temperatures as low as 10°C, production facilities should directly dilute it to the above-mentioned concentration. The flash points of ethanol 80% (*v/v*) and isopropyl alcohol 75% (*v/v*) are 17.5°C and 19°C, respectively and special attention should be given to proper storage in tropical climates. Production and storage facilities should be ideally air-conditioned or cool rooms. Open flames and smoking must be strictly prohibited in production and storage areas. Pharmacies and small-scale production centers supplying the WHO-recommended hand rub formulations are advised not to manufacture locally batches of more than 50 liters at a time. For safety reasons, it is advisable to produce smaller volumes and to adhere to local and/or national guidelines and regulations. The production should not be undertaken in central pharmacies lacking specialized air conditioning and ventilation.

Distribution

To avoid contamination with spore-forming organisms, disposable bottles should preferably be used. To prevent evaporation, containers should have a maximum capacity of 500 ml and possibly fit into a wall dispenser. Leakage-free pocket bottles with a capacity of no more than 100 ml should also be used. The production or re-filling unit should follow norms on how to clean and disinfect the bottles (e.g. autoclaving, boiling, or chemical disinfection with chlorine). Autoclaving is considered the most suitable procedure. Reusable bottles should never be refilled until they have been completely emptied and then cleansed and disinfected. Chemical disinfection should include soaking the bottles in a solution containing 1000 ppm of chlorine for a minimum of 15 minutes and then rinsing with sterile/cooled boiled water. After thermal or chemical disinfection, bottles should be left to dry completely upside-down, in a bottle rack. Dry bottles should be closed with a lid and stored, protected from dust, until use.

(v) Preparation of simple organophosphates, phosphonates and thiophosphates:

Preparation of organophosphates:

In organic chemistry, organophosphates (also known as phosphate esters, or OPEs) are a class of organophosphorus compounds with the general structure $O=P(OR)_3$, a central phosphate molecule with alkyl or aromatic substituents.[1] They can be considered as esters of phosphoric acid. Organophosphates are best known for their use as pesticides.

Simple organophosphates can be synthesized via alcoholysis of phosphorus oxychloride ($POCl_3$), esterification of phosphoric acid, or oxidation of phosphite esters. The most common industrial method is alcoholysis, where POCl3 reacts with alcohols to form organophosphates, often with the aid of catalysts like aluminum trichloride.

Alcoholysis of $POCl_3$

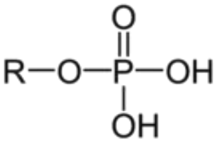
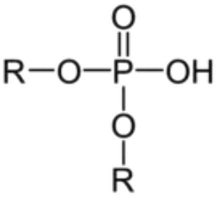
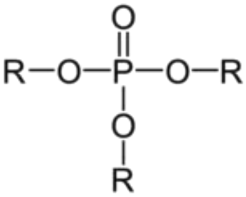
Phosphorus oxychloride reacts readily with alcohols to give organophosphates. This is the dominant industrial route and is responsible for almost all organophosphate production. Aluminum trichloride or magnesium chloride are often employed as catalysts.

Reaction: $POCl_3 + 3ROH \rightarrow O=P(OR)_3 + 3HCl$

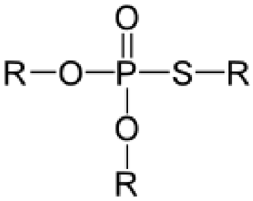
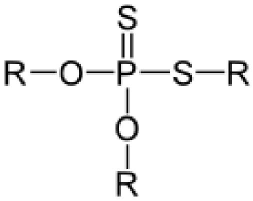
Esterification of Phosphoric Acid:

Reaction: $OP(OH)_3 + ROH \rightarrow OP(OH)_2(OR) + H_2O$, followed by further esterification

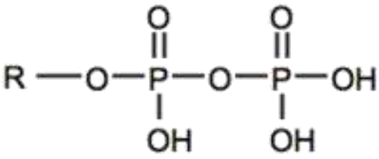
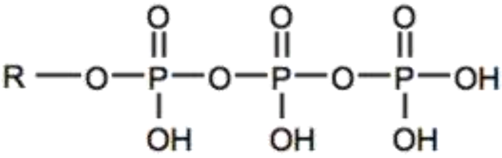
Organophosphates are a class of compounds encompassing a number of distinct but closely related function groups. These are primarily the esters of phosphoric acid and can be mono-esters, di-esters or tri-esters depending on the number of attached organic groups (abbreviated as 'R' in the image below). In general man-made organophosphates are most often triesters, while biological organophosphates are usually mono- or di-esters. The hydolysis of triesters can form diesters and monoesters.

In the context of pesticides, derivatives of organophosphates such as organothiophosphates (P=S) or phosphorodiamidates (P-N) are included as being organophosphates. The reason is that these compound are converted into organophosphates biologically.

In biology the esters of diphosphoric acid and triphosphoric acid are generally included as organophosphates. The reason is again a practical one, as many cellular processes involve the mono-, di- and tri- phosphates of the same compound. For instance, the phosphates of adenosine (AMP, ADP, ATP) play a key role in many metabolic processes.

Synthesis

Alcoholysis of $POCl_3$

Phosphorus oxychloride reacts readily with alcohols to give organophosphates. This is the dominant industrial route and is responsible for almost all organophosphate production. Aluminum trichloride or magnesium chloride are often employed as catalysts.

$O{=}PCl_3 + 3\ ROH \rightarrow O{=}P(OR)_3 + 3\ HCl$

When aliphatic alcohols are used the HCl by-product can react with the phosphate esters to give organochlorides and a lower ester.

$O{=}P(OR)_3 + HCl \rightarrow O{=}P(OR)_2OH + RCl$

This reaction is usually undesirable and is exacerbated by high reaction temperatures. It can be inhibited by the use of a base or the removal of HCl through sparging.

Esterification of phosphoric acid and P_2O_5

Esterifications of phosphoric acid with alcohols proceed less readily than the more common carboxylic acid esterifications, with the reactions rarely proceeding much further than the phosphate mono-ester. The reaction requires high temperatures, under which the phosphoric acid can dehydrate to form poly-phosphoric acids. These are exceedingly viscous and their linear polymeric structure renders them less reactive than phosphoric acid. Despite these limitations the reaction does see industrial use for the formation of monoalkyl phosphates, which are used as surfactants. A major appeal of this route is the low cost of phosphoric acid compared to phosphorus oxychloride.

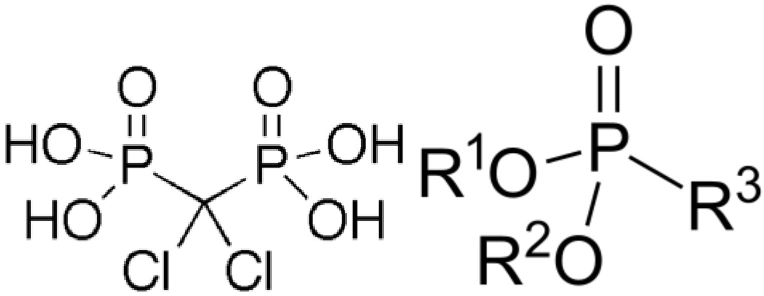
$OP(OH)_3 + ROH \rightarrow OP(OH)_2(OR) + H_2O$

P_2O_5 is the anhydride of phosphoric acid and acts similarly. The reaction yields equimolar amounts of di- and monoesters with no phosphoric acid. The process is mostly limited to primary alcohols, as secondary alcohols are prone to undesirable side reactions such as dehydration.

Preparation of simple phosphonates:

In organic chemistry, **phosphonates** or **phosphonic acids** are organophosphorus compounds containing $C{-}PO(OR)_2$ groups, where R is an organic group (alkyl, aryl). If R is hydrogen then the compound is a dialkyl phosphite, which is a different functional group. Phosphonic acids, typically handled as salts, are generally nonvolatile solids that are poorly soluble in organic solvents, but soluble in water and common alcohols.

Many commercially important compounds are phosphonates, including glyphosate (the active molecule of the herbicide Roundup), and ethephon, a widely used plant growth regulator. Bisphosphonates are popular drugs for treatment of osteoporosis.



Clodronic acid is a bisphosphonate used as a drug to treat osteoporosis. In biochemistry and medicinal chemistry, phosphonate groups are used as stable bioisosteres for phosphate, such as in the antiviral nucleotide analog, Tenofovir, one of the cornerstones of anti-HIV therapy. And there is an indication that phosphonate derivatives are "promising ligands for nuclear medicine.

Synthesis

Most processes begin with phosphorous acid (aka phosphonic acid, H_3PO_3), exploiting its reactive P−H bond.

From phosphonic acid

Phosphonic acid can be alkylated via the Kabachnik–Fields reaction or Pudovik reaction to give aminophosphonate, which are useful as chelating agents. One example is the industrial preparation of nitrilotris(methylenephosphonic acid):

$NH_3 + 3\ H_3PO_3 + 3\ CH_2O \rightarrow N(CH_2PO_3H_2)_3 + 3\ H_2O$

Phosphonic acid also can be alkylated with acrylic acid derivatives to afford carboxyl functionalized phosphonic acids. This reaction is a variant of the Michael addition:

$CH_2{=}CHCO_2R + 3\ H_3PO_3 \rightarrow (HO)_2P(O)CH_2CH_2CO_2R$

In the Hirao coupling dialkyl phosphites (which can also be viewed as di-esters of phosphonic acid: ($O{=}PH(OR)_2$) undergo a palladium-catalyzed coupling reaction with an aryl halide to form a phosphonate.

From phosphorus trichloride

Vinylphosphonic acid can be prepared by the reaction of PCl_3 and acetaldehyde:

$PCl_3 + CH_3CHO \rightarrow CH_3CH(O^-)PCl^+_3$

This adduct reacts with acetic acid:

$CH_3CH(O^-)PCl^+_3 + 2\ CH_3CO_2H \rightarrow CH_3CH(Cl)PO(OH)_2 + 2\ CH_3COCl$

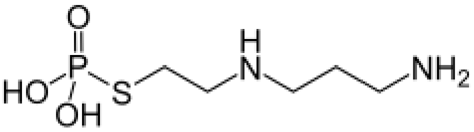
This chloride undergoes dehydrochlorination to afford the target:

$CH_3CH(Cl)PO(OH)_2 \rightarrow CH_2{=}CHPO(OH)_2 + HCl$

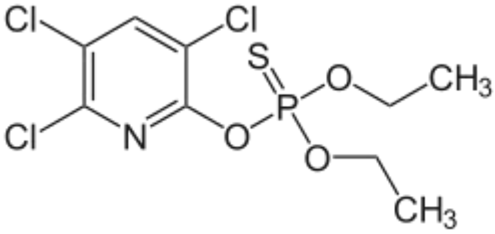
Preparation of simple Thiophosphates:

Thiophosphates (or phosphorothioates, PS) are chemical compounds and anions with the general chemical formula $PS_{4-x}O^{3-}_{x}$ (x = 0, 1, 2, or 3) and related derivatives where organic groups are attached to one or more O or S. Thiophosphates feature tetrahedral phosphorus(V) centers.

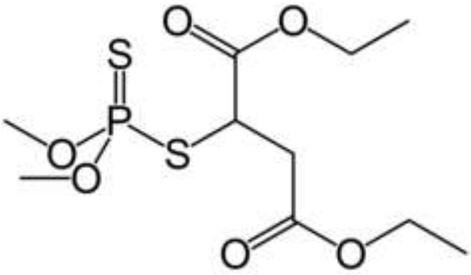
Organothiophosphates are a subclass of organophosphorus compounds that are structurally related to the inorganic thiophosphates. Common members have formulas of the type $(RO)_{3-x}(RS)_xPS$ and related compounds where RO is replaced by RS. Many of these compounds are used as insecticides, some have medical applications, and some have been used as oil additives.



Amifostine, which is used in cancer chemotherapy.



Chlorpyrifos, a popular insecticide.



Malathion, a popular insecticide.

Synthesis

Dithiophosphate has the formula $[PO_2S_2]^{3-}$, which has C_{2v} symmetry. Sodium dithiophosphate, which is colorless, is the major product from the reaction of phosphorus pentasulfide with NaOH:

$P_2S_5 + 6\ NaOH \rightarrow 2\ Na_3PO_2S_2 + H_2S + 2\ H_2O$

Dithiophosphoric acid is obtained by treatment of barium dithiophosphate with sulfuric acid:

$Ba_3(PO_2S_2)_2 + 3\ H_2SO_4 \rightarrow 3\ BaSO_4 + 2\ H_3PO_2S_2$

Both $Na_3PO_2S_2$ and especially $H_3PO_2S_2$ are prone toward hydrolysis to their monothio derivatives.

Part B: Field Visit Report on the Preparation of Household Chemicals and its Applications

Introduction:

Household chemicals are non-food chemicals that are commonly found and used in almost every place like homes, schools, offices and markets. They are designed particularly to assist cleaning house and back garden maintenance, pest control and for other hygiene purposes. Cleaning is the process of removing unwanted substances such as dirt, stains, infectious agents and other impurities from an object or environment So Cleaning is very essential to stay fit & healthy. Cleaning also protect the health of human beings by controlling the transmission of diseases.

For Cleaning we require different types of cleaning agents and disinfectants. A cleaning agent is not the instrument used for cleaning but rather the chemicals in liquid or solid forms actually does the cleaning. They are either natural or synthetic substances that are used to aid the cleaning process. The toxicity of a chemical substance depends on the strength and nature as well as how long the person is in contact with it. So it is important to know the various aspects of their uses. This course is designed to learn about Preparation of household cleaning agents and disinfectants.

LEARNING OUTCOMES:

- The students should learn fundamentals of household chemicals.
- The students should define various processes of house hold chemicals.
- The students should explain preparations, uses and reactions of household chemicals.
- The students should define preparation of different strength cleaning agents and hand Sanitizer.
- The students should prepare various composition of Liquid Detergent, liquid disinfectant and Dish washes Liquid Soap.
- The students should explain preparations and reactions of simple organophosphates, phosphonates and thiophosphates.
- The students should learn technology of detergent.
- Students should define herbal product, extraction techniques.
- Preparation of hand sanitizer of different flavor and fragrances.

Objectives:

- To study the Preparation of Household Chemicals and its Applications.
- To collect information regarding various use and its chemical hazards.
- To identify traditional use in public health and safety.

Observation:

Name of place: Insert image

Description:

Chemical constituents:

Traditional use:

Paper Title: Inorganic Chemistry –II & Organic Chemistry-II (Practical)

Paper Code: CEMMIN03P

Course Content:

Inorganic Chemistry Laboratory

1. Estimation of Fe(II) and Fe(III) in a given mixture.

2. Estimation of Fe(III) and Cu(II) in a mixture.

Organic Chemistry Lab

A. Separation, based upon solubility, by using common laboratory reagents like water (cold, hot), dil. HCl, dil. NaOH, dil. NaHCO3, etc., of components of a binary solid mixture; purification of any one of the separated components by crystallization.

The composition of the mixture may be of the following types: Benzoic acid/p-Toluidine; p-Nitrobenzoic acid/p-Aminobenzoic acid; p-Nitrotolune/p-Anisidine; etc.

B. Identification of a pure organic compound

Solid compounds: oxalic acid, tartaric acid, succinic acid, resorcinol, urea, glucose, benzoic acid and salicylic acid.

Liquid Compounds: methyl alcohol, ethyl alcohol, acetone, aniline, dimethylaniline, benzaldehyde, chloroform and nitrobenzene.

Organic Chemistry Lab

1. Separation

Basic Concept: Synthesis of organic compounds often involves reactions that result in the formation of a mixture of products. When some parts of a tree are extracted in an attempt to isolate a targeted compound, invariably a mixture of compounds appear. Isolation of the desired compound, therefore, needs exclusion of the unwanted companions. This constitutes the principle of separation of a mixture of compounds into its components. Wide variety of techniques, including solvent extraction, fractional crystallisation and steam distillation, chromatography etc are employed for this purpose. An increase in the number of components in the mixture requires a technique with enhanced sophistication and clinical accuracy for separation. In the following section separation of a mixture of only two components utilising the difference in solubility of components in a suitable solvent is discussed.

If one component dissolves in a particular solvent and the other does not, the mixture may be shaken with the appropriate solvent to dissolve the soluble component. Filtration separates the insoluble component from the soluble one. Then the soluble component has to be regenerated from its solution. Acidification, basification, evaporation etc may be needed for this regeneration purpose.

General Procedure of Separation:

1. Test the solubility of the supplied Binary Mixture by adding a pinch of the sample in (-2 mL) each of the following two solvents and shake well to find the solvent in which a part of the mixture is soluble.

Solvents: (i) 10% NaOH solution, (ii) 6 (N) HCI.

2. Add (-25 mL) of the appropriate solvent (identified in step 1) to the rest of the Mixture taken in a 100 mL beaker and stir well with a glass rod for 15 - 20 minutes.
3. Filter under suction and collect the filtrate in a 250 mL beaker.
4. Wash the residue obtained in step 3 with the same solvent used in step 2. Then wash the residue with water, dry and collect as the ‘Insoluble Part’

5. Precipitate the soluble component from the filtrate obtained in step3 either by acidifying with conc. HCl (if 10% NaOH solution is used for dissolution) or basifying with 20% NaOH solution (if 6(N) HCl solution is used for dissolution) as the case may be. Filter off the precipitate, wash with water, dry and collect the solid as the soluble parts.

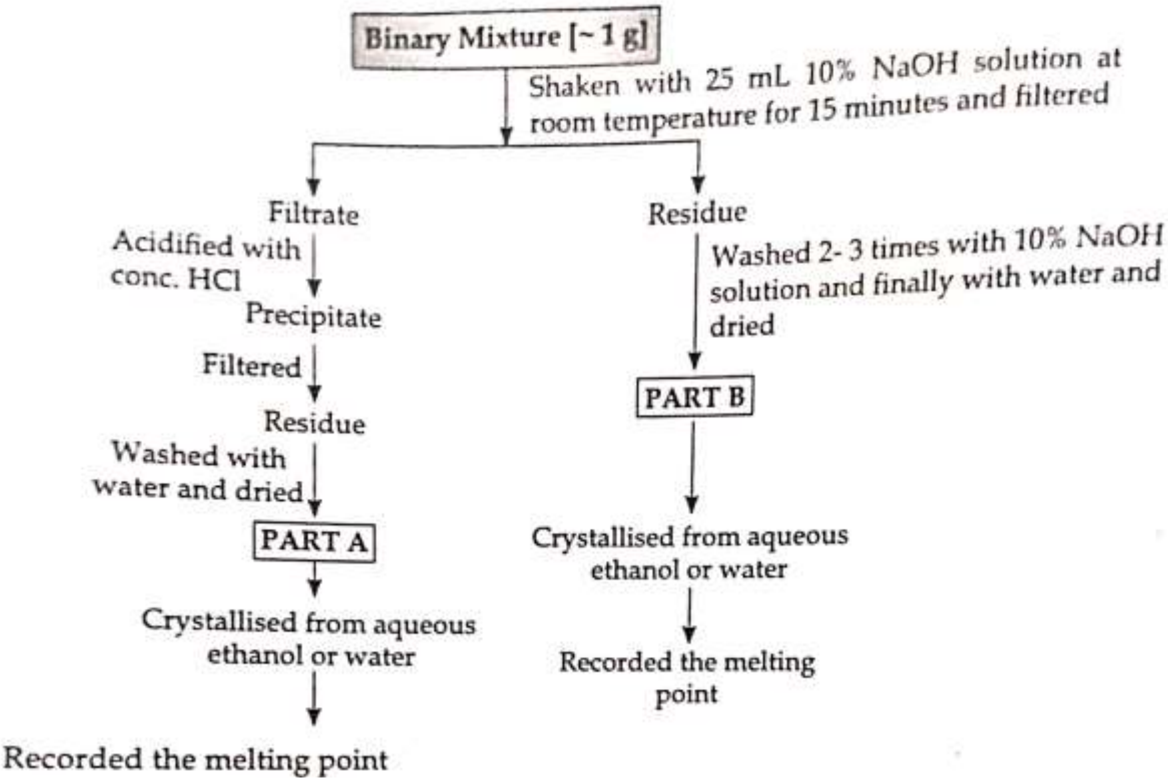
Reporting of Separation of Components of Unknown Binary Solid Mixture:

Chemical required	**Apparatus required**
6(N) HCl	Test tube
Conc. HCl	100 ml beaker
10% NaOH	Measuring cylinder

Chemical required	**Apparatus required**
20% NaOH	Glass rod
Distilled water	Suction filtering apparatus with small Buchner funnel

TYPE I [Mixture of Acidic (PART A) and Neutral (PART B) Components] : 0.2 g of the given binary mixture (preferably in powdered form, if crystalline sample is given, it should be ground thoroughly with the pestle to make it powder) is vigorously shaken separately with 2-3 mL of each of 6(N) HCl and 10% NaOH solution at room temperature for 5 minutes.

6(N) HCl	**10% NaOH**
Insoluble	Partly Soluble

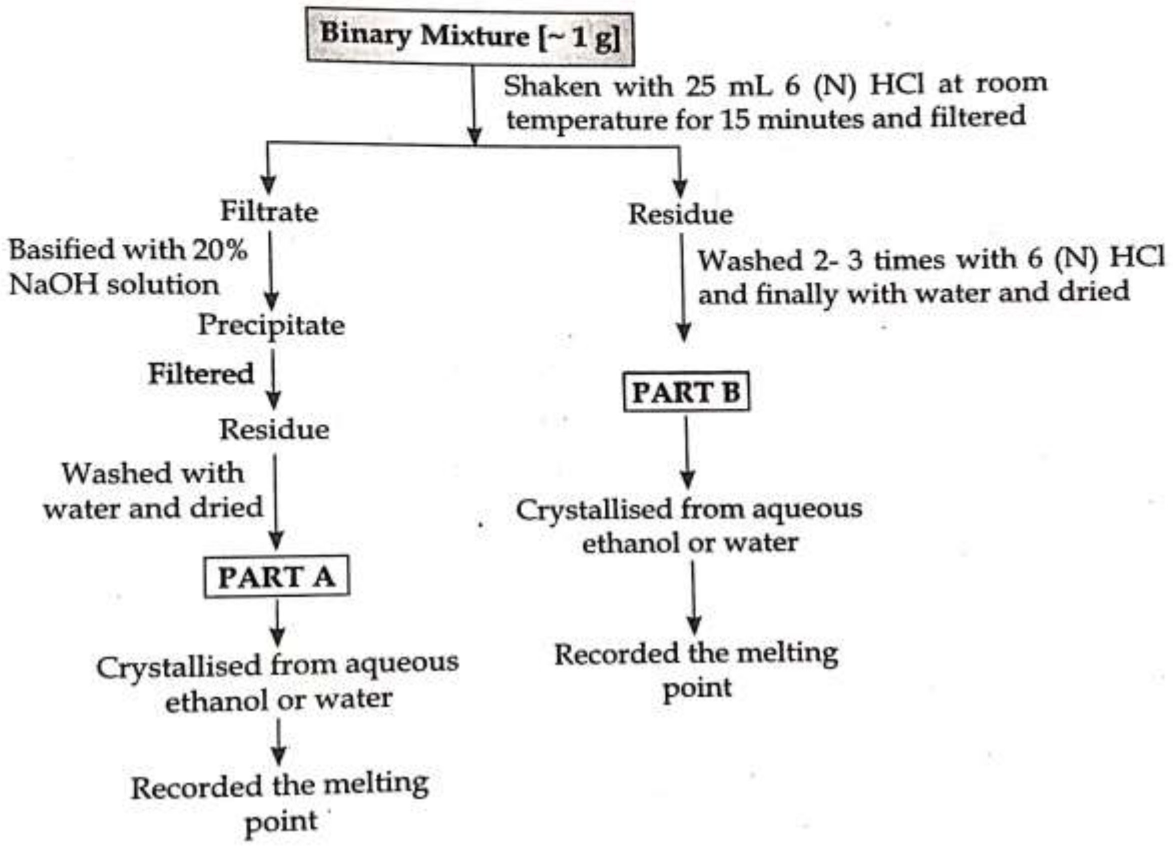


Melting point of part A=

Melting point of part B=

TYPE II [Mixture of Basic (PART A) and Neutral (PART B) Components] : 0.2 g of the given binary mixture (preferably in powdered form, if crystalline sample is given, it should be ground thoroughly with the pestle to make it powder) is vigorously shaken separately with 2-3 mL of each of 6(N) HCl and 10% NaOH solution at room temperature for 5 minutes.

10% NaOH	**6(N) HCl**
Insoluble	Partly Soluble



Melting point of part A=

Melting point of part B=

Identification of a Pure Organic Compound

The sample supplied may be any one of the following types:

1. Pure solids
2. Pure liquid

The following preliminary tests will be performed with the given sample;

Solid compounds

Oxalic acid

Physical characteristics and preliminary test :

1. State : Solid
2. Colour : Colorless
3. Texture : Crystaline

3. Odour : Odourless
4. Solubility : Soluble in cold water
5. Litmus : Blue litmus turns red
6. Action of heat : Volatilises completely without charring when heated slowly and a sublime is formed but decomposed with evolution of gases when heated strongly
7. Ignition test : Blue non-sooty flame

Experiment	Observation
1. H_2SO_4 test: A small amount of O.S. is heated with conc. H_2SO_4 and the evolved gas is passed into lime water.	1. Lime water turns milky.
2. Soda-lime test: A small amount of O.S. is heated in a hard glass test tube with sodalime.	2. The gas burns at the mouth of the test tube ñb sound.
3. Calcium chloride test: Calcium chloride solution is added to neutral solution of O.S. The ppt is divided into two parts (i) Dil. acetic acid is added to one portion. (ii) Dil. HC or dil. HNO3 is added to another portion.	3. A white ppt. forms immediately. (i) The ppt does not dissolve. (ii) The ppt. dissolves
4. Silver nitrate test : $AgNO_3$ sOlution iS added to neutral solution of O.S. The ppt. is divided into three parts : (il Dil. NAOH is added to one portion. (ii) Dil. HNO_3 is ildded to another portion. (iii) The third portion is warmed, dried and heated strongly in a dry test tube	4. A white crystalline ppt (i) The ppt. dissolves (ii) The ppt. dissolves (iii) Metallic silver is formed
5. Denige's test (C.T.): Denige's reagent is added to neutral solution or aqueous solution of O.S.	5. White ppt. in the cold which does not dissolve
6. Potassium permanganate test: A little of dil. H_2SO_4 is added to a solution of O.S or neutral soln. of O.S. The solution is warmed and dil. $KMnO_4$ Soln. is added drop by drop with shaking.	6. The pink colour of permanganate disappears.

Succinic acid

Physical characteristics and preliminary test :

1. State : Solid
2. Colour : Colorless
3. Texture : Crystaline
3. Odour : Odourless
4. Solubility : Soluble in cold water

5. Litmus : Blue litmus turns red
6. Action of heat : Melts and then boils giving off extremely irritating vapors. A sublimate is forme d at the cooler part of the test tube
7. Ignition test : Blue non-sooty flame

Experiment	Observation
1. H_2SO_4 test: Few crystals of O.S. is warmed conc. H_2SO_4 and then heated saongly.	1. O.S dissolve without charring. Slight chairring occurs on strong heating and the solution turn brown with evolution of SO_2.
2. Calcium chloride test: An equal volume of Calcium chloride solution is added to neutral solution of O.S. The mixture is shaken vigorously inner side of the test tube is scratched with glass rod and finally boiled.	2. A white ppt. soluble in acetic acld.
3. Silver nitrate test : $AgNO_3$ solution is added to neutral solution of O.S.	3. Buff-colored ppt soluble in dil. HCl.
4. Denige's test (C.T.): Denige's reagent is added to neutral solution or aqueous solution of O.S. and then a drop of 2% $KMnO_4$ solution is added.	4. No ppt. in the cold but the color of permanganate presists.

Resorcinol

Physical characteristics and preliminary test :

1. State : Solid
2. Colour : Colorless
3. Texture : Crystaline
4. Odour : Odourless
5. Solubility : Soluble in cold water
6. Litmus : feebly acidic
7. Action of heat : Melts and volatiles
8. Ignition test : Yellow sooty flame

Experiment	Observation
1. H_2SO_4 test: Few crystals of O.S. is warmed with conc. H_2SO_4.	1. O.S dissolve without charring.
2. Ferric chloride test: A few drops of $FeCl_3$ solution is added to aqueous solution of O.S.	2. Bluish-violet color.

3. Bromine writer test: Bromine writer is added to aq. solution of O.S. and shaken.	3. White ppt.
4. Tollen's test: Aqueous solution of O.S is added to Tollen’s reagent and warmed.	4. Black ppt of silver mirror.
5. Fluorescein test: A small quantity of O.S. is mixed with succinic acid and the test performed.	5. An intense green fluorescein.

Urea

Physical characteristics and preliminary test :

1. State : Solid
2. Colour : Colorless
3. Texture : Crystaline
4. Odour : Odourless
5. Solubility : Soluble in cold water
6. Litmus : Neutral
7. Action of heat : Melts and gives off characteristic smell of NH_3
8. Ignition test : Blue non-sooty flame

Experiment	Observation
1. Nitrous acid test: Dil. HCl and dil. NaNO2 solution is added to aq. Solution of O.s in a test tube and shaken.	1. Effervescence with evolution of gas.
2. Nitric acid test: 2 mL of conc. HNO3 is added to aqueous concentrate solution of O.S.	2. A white crystalline ppt.
3. Oxalic acid test: A concentrate solution of oxalic acid is added to conc. solution of O.S.	3. A white crystalline ppt.
4. Biuret test: About 0 5 g of O.S. is heated gently to melt. The heating is continued till the molten mass solidifies at once when test tube is taken out of flame. If there is no solidification the process is repeated. The residue is cooled and dissolved in I ml of NaOH solution by warming and shaking. The solution is cooled again and a drop or two of a very dilute copper sulphate solution is added to it.	4. A pink or violet colour develops.

Benzoic acid

Physical characteristics and preliminary test :

1. State : Solid
2. Colour : White
3. Texture : Plate-shaped or needle shaped

Crystal
4. Odour : Odourless
5. Solubility : Insoluble in cold water but Soluble in hot water
6. Litmus : Blue litmus turns red
7. Action of heat : Melts and sublimates
8. Ignition test : Yellow sooty flame

Experiment	Observation
1. H_2SO_4 test: A small amount of O.S. is warmed with conc. H_2SO_4. **2. Soda-lime test**: A small amount of O.S. is heated in a hard glass test tube with sodalime. **3. Denige's test:** Denige's reagent is added to neutral solution of O.S. **4. Ferric chloride test: (**a) A drop or two of freshly prepared solution of Fecl$_3$ is added to neutral solution of O.S. (b) Dilute HCl is added to it. **5. Esterification test:** About 0.1 g of O.S and 2 mL of dehydrated alcohol are taken in a clean test tube and shaken well. Then a few drops of conc. H_2SO_4 is added and the test tube is waremd in a hot water bath. The test tube is cooled and content of the test tube is poured into 50 mL of dilute sodium cxarbonate solution taken in a beaker.	1. Dissolves without charring. 2. Characteristics smell of benzene. 3. White ppt. dissolves on boiling but appears on cooling. 4. (a) a buff-colored ppt. (b) buff-colored ppt dissolves with appearance of white ppt. 5. characteristics fruity smell of ester.

Salicylic acid

Physical characteristics and preliminary test :

1. State : Solid
2. Colour : White
3. Texture : needle-shaped Crystal
4. Odour : Odourless
5. Solubility : Sparingly soluble in cold water but readily Soluble in hot water
6. Litmus : Blue litmus turns red
7. Action of heat : Melts and sublimes
8. Ignition test : Yellow sooty flame

Experiment	Observation
1. H_2SO_4 test: A small amount of O.S. is warmed with conc. H_2SO_4.	1. The solid dissolved, charring occurs after some time, solution darkens and gas evolve.
2. Soda-lime test: A small amount of O.S. is heated in a hard glass test tube with sodalime.	2. Characteristics smell of phenol.
3. Denige's test: Denige's reagent is added to neutral solution of O.S.	3. White ppt. dissolves on boiling but appears on cooling.
4. Ferric chloride test: A drop or two of freshly prepared solution of $Fecl_3$ is added to neutral solution of O.S. (a) Dilute HCl is added to it. (b) acetic acid is added to another part.	4. An intense violet color (a) The color discharges (b) The color persists
5. Oil of winter test: About 0.1 g of O.S and 1 mL of methanol and few drops of conc. H_2SO_4 are warmed in aclean dry test tube.	5. A characteristics pungent fragnant odor which intensifies on pouring the mixture to dil. Sodium carbonate solution.

Tartaric acid

Physical characteristics and preliminary test :

1. State	: Solid
2. Colour	: Colorless
3. Texture	: Crystalline
4. Odour	: Odourless
5. Solubility	: Soluble in cold water
6. Litmus	: Blue litmus turns red
7. Action of heat	: Charrs and gives off smell of burnt sugar
8. Ignition test	: Blue non-sooty flame

Experiment	Observation
1. H_2SO_4 test: A small amount of O.S. is warmed with conc. H_2SO_4.	1. charrs immediately.
2. Calcium chloride test: Excess of calcium chloride solution is added to	2. A white crystalline ppt. soluble in hot dilute acetic acid.

neutral solution of O.S. shaken and inside of the test tube is scratched with a glass rod.	
4. Silver nitrate test : (a) $AgNO_3$ solution is added to neutral solution of O.S. (b) Addition of AgNO3 solution is continued till the precipitation is complete. Then dil. NH4OH is added dropwise with constant shaking till the ppt. almost and not completely dissolved as revealed by slight turbidity.	4. (a) A white crystalline ppt. (b) shining silver mirror forms at the inner of the test tube.
5. Denige's test (C.T.): Denige's reagent is added to neutral solution or aqueous solution of O.S.	5. Permanganate color discharges immediately without turbidity.

Glucose

Physical characteristics and preliminary test :

1. State : Solid
2. Colour : Colorless
3. Texture : Crystalline
4. Odour : Odourless
5. Solubility : Soluble in cold water
6. Litmus : Neutral
7. Action of heat : Charrs and gives off smell of burnt sugar
8. Ignition test : Blue non-sooty flame

Experiment	Observation
1. H_2SO_4 test: A small amount of O.S. is warmed with conc. H_2SO_4.	1. No charring in cold but darkens on heating immediately.
2. Sodium hydroxide test: 2 ml of 3% solution of NaOH is added to conc. Solution of O.S. and the mixture is heated. The mixture is then acidified with dil. HNO_3.	2. The solution turns first yellow and then raddish brown.
3. Tollen's test : 5 ml of aqueous solution of o.s is added to aeual volume of Tollen's reagent taken in a clean test tube and then the test tube is placed in a beaker of boiling water for few minutes.	3. Black or grey precipitate or shining silver mirror.
	4. Brick red precipitate.

4. Fehling's test: The sample is added to a mixture of Fehling A and Felling B and heated for 1 minute. **5. Lead acetate test:** Lead acetate solution is added to solution of o.s and the mixture is boiled for few seconds. Then dil. NH_4OH solution is added dropwise till just sufficient to produce a permanat white ppt. then the mixture is boiled again.	5. The white ppt. turns salmon-pink in color.

Liquid compounds

Methyl Alcohol

CH_3-OH

Physical characteristics and preliminary test :

1. State : Liquid
2. Colour : Colorless
3. Odour : Pungent but faint alcoholic
4. Miscibility : Miscible With water
5. Litmus : Neutral
6. Action of heat : Volatilises
7. Ignition test : Blue non-sooty flame

Special Tests

Experiment	Observation
1. Oxidation test : A Cu-spiral is made repeatedly red hot and introduced into 2 ml of O.S. kept in a test tube dipped in a beaker containing cold Water. The solution is then divided into four pan ts. **(a) Schiffis test :** One part is added to Schiff s. **(b) Tollen's test:** Another part is added to Tollen's reagent and heated in a water-bath. **(d) Resorcinol test:** A mixture of 0.5 ml of oxidised liquid and a drop of 0.5% aq. Solution of resorcinol is added carefully down the side of the test tube held in an inclined position containing 2ml of conc. H_2SO_4. **2. Denige's reagent:** To about 5ml of aq. Solution of o.s. in a porcelain basin	1. (a) Pink color which deepens slowly. (b) Bright mirror of silver on the side of the test tube. (c) Intense indigo blue color.

placed on ice-water, 2-3 ml of 2.5% soln. of $KMnO_4$ soln. is added followed by few drops of conc. H_2SO_4 when a brown color developes. The brown color is destroyed by addition of saturated soln. of oxalic acid. A freshly prepared soln.of schiffs reagent is added dropwise to it with stirring. **3. Oil of wintergreen test:** 0.5 ml of o.s. and 3 drops of conc. H2SO4 are added to 0.5 g of salicylic acid and heated for one minute and finally the reaction mixture is poured into 50 ml of water taken in a beaker.	(d) A reddish -violet ring appears at the junction of two liquid layers. A white ppt. changing to reddish-violet appear in the aq. Layer above the ring after a little while. 2. A violet color develops deepening on standing. 3. Characteristics smell of methyl salicylate.

Ethanol

CH_3CH_2OH

Physical characteristics and preliminary test :

1. State	**: Liquid**
2. Colour	**: Colorless**
3. Odour	**: Pungent but faint alcoholic**
4. Miscibility	**: Miscible With water**
5. Litmus	**: Neutral**
6. Action of heat	**: Volatilises**
7. Ignition test	**: Blue non-sooty flame**

Special Tests

Experiment	Observation
1. Oxidation test : A Cu-spiral is made repeatedly red hot and introduced into 2 ml of O.S. kept in a test tube dipped in a beaker containing cold Water. The solution is then divided into four pan ts.	1.
(a) Schiffis test : One part is added to Schiff s.	(a) Pink color which deepens slowly.
(b) Tollen's test: Another part is added to Tollen's reagent and heated in a water-bath.	(b) A silver mirror is disposited on the walls of the test tube.
(c) To another part equal volume of very	

dilute solution of sodium nitroprusside solution is added followed by a few drops of NaOH solution. (d) To another part a very dilute solution of sodium nitroprusside solution is added followed by a few drops of piperidine. **2. Ethyl ester formate:** About 0.2 ml of o.s an equal volume of glacial acetic acid or 0.2g of fused sodium acetate and a little conc. H2SO4 are taken in a test tube and warmed in a water-bath for about 5min. the mixture is then poured into water taken in a beaker to which a little Na2CO3 has been added. **3. Iodoform test: (a)** To 2 ml of aq. Soln. of o.s. an equal volume of a conc. Solution of iodine in potassium iodide is added and then NaOH solution is added dropwise with stirring until violet color of iodine dis appears. Then the mixture is warmed and cooled under tap with shaking. (b) The iodoform test is repeated by adding NH4OH instead of NaOH.	(c) Wine-red color develops. (d) A deep red color develops. 2. Pleasant fruity smell. 3. (a) Characteristics smell of methyl salicylate. (b) No ppt. of iodoform forms.

Chloroform
CHCl$_3$

Physical characteristics and preliminary test :

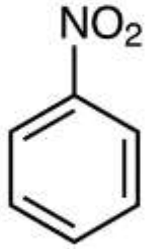
1. State	**: Liquid**
2. Colour	**: Colorless**
3. Odour	**: Sweet**
4. Miscibility	**: Immiscible With water**
5. Litmus	**: Neutral**
6. Action of heat	**: Volatilises**
7. Ignition test	**: Yellow sooty flame**

Special Tests

Experiment	Observation

1. Hydrolysis test: o.s. is treated with aq. KOH or NaOH solution and boiled. The mixture is acidified with conc. HNO3 and then AgNO3 is added.	1.Curdy white ppt. soluble in NH4OH but reappears on addition of conc. HNO3.
2. Fehlings test: o.s and Fehlings solution is warmed with constant shaking.	2. A yellowish red ppt.
3. Resorcinol test: A little powdered resorcinol and few drops of O.S. is taken in a basin, about 1 ml of conc. solution of NaOH is added to it, then the mixture is warmed gently.	3. A brilliant reddish colouration is developed in aq. Layer.
4. Carbylamine test: Few drops of O.S. aniline and alc. KOH are warmed in a diy test tube and the ensuing gas is smelt by placing the thumb at the mouth of the test tube and then holding tfie thumb near the nostrils.	4. Intolerable obnoxious smell of carbylamine.

Nitrobenzene



Physical characteristics and preliminary test :

1. State : Liquid
2. Colour : Pale yellow
3. Odour : Characteristic smell of bitter almonds
4. Miscibility : Immiscible With water
5. Litmus : Neutral
6. Action of heat : Volatilises without leaving any residue
7. Ignition test : Yellow sooty flame

Special Tests

Experiment	Observation

1. Caustic soda test : About 0.2 ml of O.S. is heated with conc. solution of caustic soda.	1.The color darkens.
2. Reduction and diazocoupling test: 0.5 ml is reduced by tin or zinc and dil. HCl for 5 minutes. The decanted solution is cooled and very dilute cold solution of NaNO2 is added to it. The diazotised soln. is added to cold alkaline soln. of. β-naphtol.	2. Brilliant scarlet-red dye.
3. Muliken-Barker test: An aq. Ethanolic solution of few drops of given sample is boiled with a pinch of zinc dust and little solid NH4Cl. The mixture is cooled and filtered into Tollen’s reagent.	3. Grey or black ppt..

Acetone

CH_3COCH_3

Physical characteristics and preliminary test :

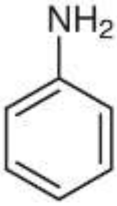
1. State : Liquid
2. Colour : Colorless
3. Odour : Pleasant ethereal odour
4. Miscibility : Miscible With water
5. Litmus : Neutral
6. Action of heat : Completely Volatilises without leaving any residue
7. Ignition test : Blue non-sooty flame

Special Tests

Experiment	Observation
1. Oxidation test : A small amount of O.S. is added to 2-4-dinitrophenylhydrazine solution.	1. Yellow ppt. on mixing.
2. Denige’s test: An equal volume of Denige’s reagent is added to an aqueous solution of O.S and the test tube is then kept in a boiling water bath for few minutes.	2. Heavy white ppt. of double compound of acetone and basic mercuric sulphate.
3. Legal’s test: A few drops of a very dil. solution of sodium nitroprusside is added to 2-3 ml of aqueous solution of O.S. followed by a few drops of dil. NaOH solution.	3. A ruby red (reddish-purple) color develops which disappears on warming but reappears on cooling.
4. Iodoform test: (a) iodoform test is performed with I2 in KI solution and NaOH	4. (a) Yellow ppt. of CHI_3 having characteristics sweeet smell. (b) Yellow ppt. of CHI_3 having

solution. (b) the iodoform test is repeated by adding NH_4OH solution instead of NaOH solution.	characteristics sweeet smell.

Aniline



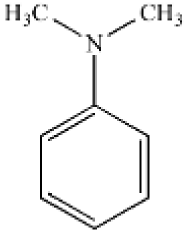
Physical characteristics and preliminary test :

1. State	: Liquid
2. Colour	: Colorless when freshly distilled but turns brown on exposure to light and air
3. Odour	: Characteristic aromatic smell
4. Miscibility	: Immiscible with water but soluble dil.HCI
5. Litmus	: Neutral
6. Action of heat	: Completely Volatilises without leaving any residue
7. Ignition test	: Yellow sooty flame

Special Tests

Experiment	Observation
1. Bleaching powder test: A few drops of bleaching powder solution is added to a dilute solution of O S. in HCl.	1. Purple-violet colouration.
2. Potassium dichromate test: A drop of O.S. is added to 5 to 6 drops of conc. H_2SO_4 taken in a spot plate and the mixture is stirred well with glass rod. Then a drop of $K_2Cr_2O_7$ solution is added to it.	2. Intense blue colour develops.
3. Diazocoupling test: 5-6 drops of O.S are dissolved in dil. HCl in a test tube and cooled in ice-water. Then 3-4 drops of very dilute ice cold solution of sodium nitrite are added to it. Then the solution is added to 1mL of ice-cold alkaline solution of β-naphtol.	3. Brilliant scarlet-red dye.

Dimethylanilinebenzaldehyde



Physical characteristics and preliminary test :

1. State	: Liquid
2. Colour	: Colorless when freshly distilled but turns brown on exposure to light and air
3. Odour	: Characteristic bad smell
4. Miscibility	: Immiscible with water but soluble dil.HCI
5. Litmus	: Neutral
6. Action of heat	: Completely Volatilises without leaving any residue
7. Ignition test	: Yellow sooty flame

Special Tests

Experiment	Observation
1. Bleaching powder test: A few drops of bleaching powder solution is added to a dilute solution of O S. in HCl.	1. Purple-violet colouration.
2. Potassium dichromate test: A drop of O.S. is added to 5 to 6 drops of conc. H_2SO_4 taken in a spot plate and the mixture is stirred well with glass rod. Then a drop of $K_2Cr_2O_7$ solution is added to it.	2. Intense blue colour develops.
3. Diazocoupling test: 5-6 drops of O.S are dissolved in dil. HCl in a test tube and cooled in ice-water. Then 3-4 drops of very dilute ice cold solution of sodium nitrite are added to it. Then the solution is added to 1mL of ice-cold alkaline solution of β-naphtol.	3. Brilliant scarlet-red dye.

Benzaldehyde

CH_3CHO

Physical characteristics and preliminary test :

1. State : Liquid
2. Colour : Colorless
3. Odour : Characteristic smell of bitter almonds
4. Miscibility : Immiscible with water
5. Litmus : Neutral
6. Action of heat : Volatilises
7. Ignition test : Yellow sooty flame

Special Tests

Experiment	Observation
1. 2,4-D.N.P test: A few drops of 2,4-D.N.P solution is added to 1 mL alcoholic solution given sample. bleaching powder solution is added to a dilute solution of O S. in HCl. **2. Tollen's test:** An alcoholic solution of the given sample is added to 1 mL of Tollen's reagent warmed gently. **3. Malachite green test:** 0.5 ml of O.S is heated with 1ml of dimethyl aniline and a small bit of anhydrous $ZnCl_2$ in a dry test tube for one minute.The leucobase produced is oxidised with lead dioxide in a solution of acetic acid and excess conc. HCl is added.	1. reddish yellow ppt. forms immediately simply on mixing. 2. Black ppt. 3. A intense green coloration.

Inorganic Chemistry Laboratory

Experiment 1: Estimation of Fe(II) and Fe(III) in a given mixture.

AIM:

To estimate the amount of ferrous and ferric ions in a given mixture by making use potassium dichromate

Chemicals: potassium dichromate, Stannous chloride, Mercurous chloride, phenyl amine

Preparation of solutions:

Standard potassium dichromate:

Weigh out accurately weighted 0.245 gm potassium dichromate into a 100 ml standard flask, dissolve in little amount of water, and make up the solution with distilled water. Shake the flask well for uniform concentration.

Stannous chloride solution:

Dissolve 150 g of SnCl2.2H2O in 100 mL concentrated HCl and dilute with water to 1 L

Mercuric chloride solution:

Dissolve 60 g of HgCl2 in hot water and dilute to 1 litre.

Acid mixture:

Dissolve 150 ml of concentrated H2SO4 and 50 ml of H3PO4 in a reagent bottle and stopper it.

Phenylamine:

Dissolve 1 gm of di Phenyl amine in 100ml of concentrated H2SO4.

Procedure:

Standardisation of total iron:

Make up the given mixture of Fe^{+2} and Fe^{+3} given in 100 ml standard flask up to the mark withdistilled water and shake the flask well for uniform concentration. Pippette out 20 ml of the iron solution in to a conical flask, Add 100 ml concentrated HCl and heat the solution to bolingtill the colour of the solution changes yellow. Add stannous chloride drop by drop to the hot solution till the yellow colour disappears. Cool the solution under tap and add 100 ml mercuricchloride in one portion silky white ppt of mercurous is obtained. Add 5 ml of acid mixture and3- 4 drops of diphenylamine indicator .Titrate the solution against standard potassium dichromate taken in a burette till the green colour changes to blue violet. Repeat the titration toget concurrent values. Let the titre value be x ml.

Standardization of ferrous iron:

Pipette out 20 ml of the total iron solution into a 250 ml conical flask, add 5 ml of acid mixture and 2 drops of di-phenylamine indicator. Titrate the resulting solution with potassium dichromate taken in a burette till a blue colour is obtained. Note the burette readings and repeat the titration for concurrent values. Let the titre value be y ml

CALCULATIONS:

Normality of potassium dichromate N_1= Weight of potassium dichromate
×10/49Normality of total iron = $V_1N_1=V_2N_2$
V_1= Volume of dichromate

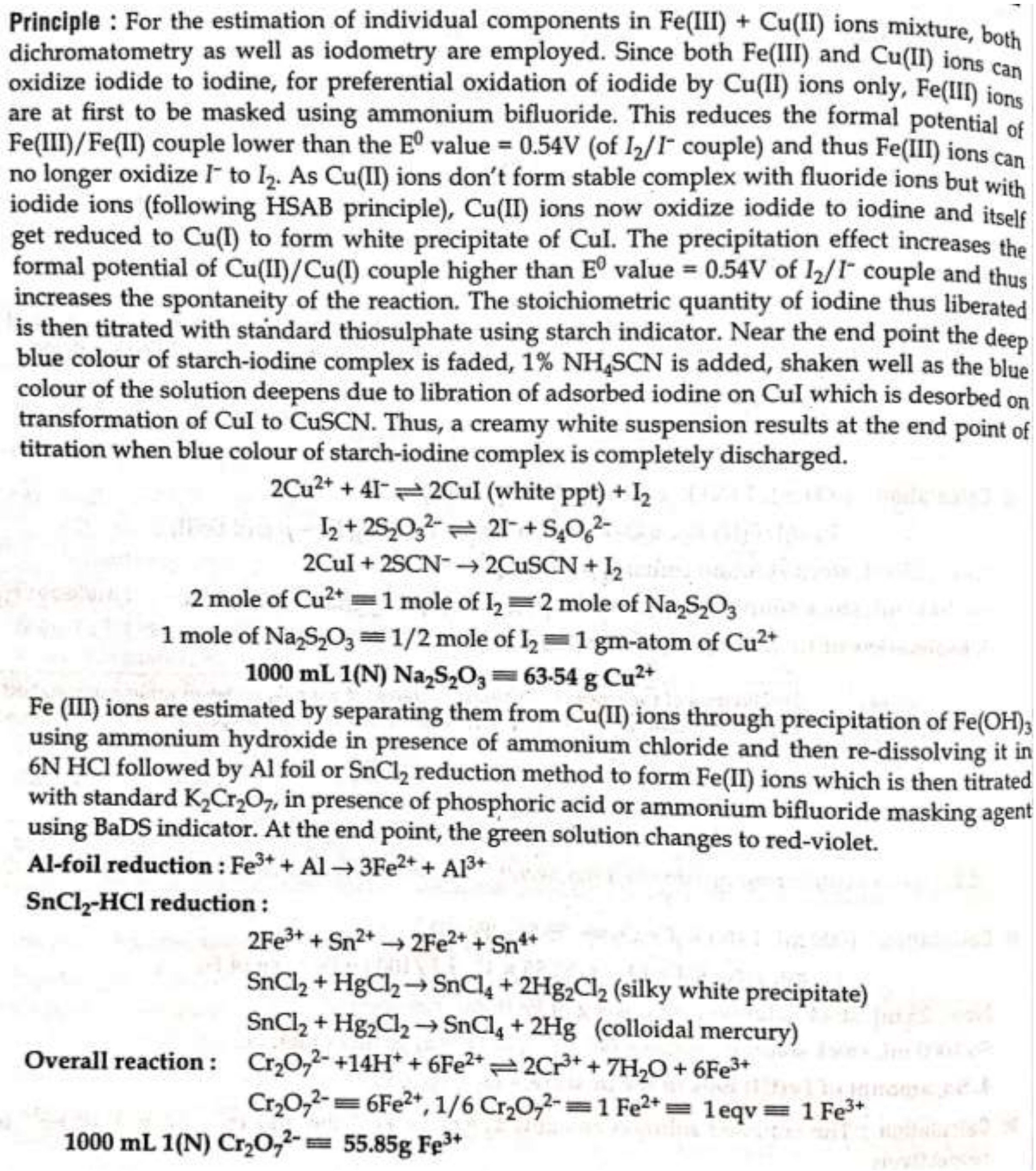
V_2 = Volume of dichromate consumed for
total ironN_2 = Normality of total iron
N_1 = Normality of dichromate
$N_2 = V_1N_1/ V_2$= titre value be x ml ×
N_1/20 Amount of total iron = $\frac{N_2 \times 55.85}{10}$ =
p gms /100 ml

Normality of ferrous iron Fe^{+2}=
$V_1N_1=V_3N_3$ V_1 = Volume of
dichromate
N_1 = Normality of
dichromate V_3 = Volume
of Fe^{+2} solution N_3 =
Normality of Fe^{+2}
solution
$N_3 = \underline{V_1N_1}$ = titre value be y ml
×N_1/20V_3
Amount of Ferrous iron = $\frac{N_3 \times 55.85}{10}$= q gms /100ml

RESULT:

Amount of Ferrous iron =Amount of total iron-amount of Fe^{+2} = p — q/100 gms

Experiment 2: Estimation of Fe(III) and Cu(II) in a mixture



Principle : For the estimation of individual components in Fe(III) + Cu(II) ions mixture, both dichromatometry as well as iodometry are employed. Since both Fe(III) and Cu(II) ions can oxidize iodide to iodine, for preferential oxidation of iodide by Cu(II) ions only, Fe(III) ions are at first to be masked using ammonium bifluoride. This reduces the formal potential of Fe(III)/Fe(II) couple lower than the E^0 value = 0.54V (of I_2/I^- couple) and thus Fe(III) ions can no longer oxidize I^- to I_2. As Cu(II) ions don't form stable complex with fluoride ions but with iodide ions (following HSAB principle), Cu(II) ions now oxidize iodide to iodine and itself get reduced to Cu(I) to form white precipitate of CuI. The precipitation effect increases the formal potential of Cu(II)/Cu(I) couple higher than E^0 value = 0.54V of I_2/I^- couple and thus increases the spontaneity of the reaction. The stoichiometric quantity of iodine thus liberated is then titrated with standard thiosulphate using starch indicator. Near the end point the deep blue colour of starch-iodine complex is faded, 1% NH_4SCN is added, shaken well as the blue colour of the solution deepens due to libration of adsorbed iodine on CuI which is desorbed on transformation of CuI to CuSCN. Thus, a creamy white suspension results at the end point of titration when blue colour of starch-iodine complex is completely discharged.

$$2Cu^{2+} + 4I^- \rightleftharpoons 2CuI \text{ (white ppt)} + I_2$$

$$I_2 + 2S_2O_3^{2-} \rightleftharpoons 2I^- + S_4O_6^{2-}$$

$$2CuI + 2SCN^- \rightarrow 2CuSCN + I_2$$

$$2 \text{ mole of } Cu^{2+} \equiv 1 \text{ mole of } I_2 \equiv 2 \text{ mole of } Na_2S_2O_3$$

$$1 \text{ mole of } Na_2S_2O_3 \equiv 1/2 \text{ mole of } I_2 \equiv 1 \text{ gm-atom of } Cu^{2+}$$

$$\mathbf{1000\ mL\ 1(N)\ Na_2S_2O_3 \equiv 63{\cdot}54\ g\ Cu^{2+}}$$

Fe (III) ions are estimated by separating them from Cu(II) ions through precipitation of $Fe(OH)_3$ using ammonium hydroxide in presence of ammonium chloride and then re-dissolving it in 6N HCl followed by Al foil or $SnCl_2$ reduction method to form Fe(II) ions which is then titrated with standard $K_2Cr_2O_7$, in presence of phosphoric acid or ammonium bifluoride masking agent using BaDS indicator. At the end point, the green solution changes to red-violet.

Al-foil reduction : $Fe^{3+} + Al \rightarrow 3Fe^{2+} + Al^{3+}$

$SnCl_2$-HCl reduction :

$$2Fe^{3+} + Sn^{2+} \rightarrow 2Fe^{2+} + Sn^{4+}$$

$$SnCl_2 + HgCl_2 \rightarrow SnCl_4 + 2Hg_2Cl_2 \text{ (silky white precipitate)}$$

$$SnCl_2 + Hg_2Cl_2 \rightarrow SnCl_4 + 2Hg \text{ (colloidal mercury)}$$

Overall reaction : $Cr_2O_7^{2-} + 14H^+ + 6Fe^{2+} \rightleftharpoons 2Cr^{3+} + 7H_2O + 6Fe^{3+}$

$$Cr_2O_7^{2-} \equiv 6Fe^{2+}, 1/6\ Cr_2O_7^{2-} \equiv 1\ Fe^{2+} \equiv 1 \text{ eqv} \equiv 1\ Fe^{3+}$$

$$\mathbf{1000\ mL\ 1(N)\ Cr_2O_7^{2-} \equiv 55.85g\ Fe^{3+}}$$

Chemicals required:

① Standard (N/20) $K_2Cr_2O_7$ solution
② 4N H_2SO_4 solution
③ Syrupy phosphoric acid or ammonium bifluoride solid
④ 0.1% BaDS indicator
⑤ Concentrated HCl
⑥ Al foil or $SnCl_2$ solution [150g of $SnCl_2 \cdot 2H_2O$ in 500mL conc. HCL and dilute to 1 L with distilled water].
⑦ 10% $HgCl_2$ solution
⑧ 20 mL 10% KI solution
⑨ 1% starch solution
⑩ NH_4Cl
⑪ 1:1 (v/v) Ammonia
⑫ Fe(III) + Cu(II) stock solution: (i) Dissolve 24g ferric alum in 500 mL 2(N) sulphuric acid (ii) Dissolve 12g of $CuSO_4 \cdot 5H_2O$ salt solution in in 500mL 2(N) sulphuric acid. Add (i) and (ii) mixtures to prepare stock solution
⑬ 1% NH_4SCN (w/v) in distilled water.
⑭ (N/20) $Na_2S_2O_3$ solution **(See section 2.5 for details)**
⑮ Concentrated Nitric acid

Procedure :

Step-1 : Preparation of 250 mL standard N/20 $K_2Cr_2O_7$ solution.

(Follow the procedure as discussed in section- 2.4.1)

Step-2 : Standardization of $Na_2S_2O_3$ solution by standard (N/20) $K_2Cr_2O_7$ solution.

(Follow the procedure as discussed in section- 2.5.1, step-2)

Step-3 : Estimation of Cu(II) ions : 25 mL of supplied stock solution is pipetted out in a 250 mL conical flask and neutralized by 1 : 1 NH_4OH till a faint permanent bluish white turbidity appears. Now add 2 g of NH_4HF_2 to dissolve the precipitate, followed by 2 g of solid KI or 20 mL 10% KI solution and dilute to 100 mL. The solution turns brown immediately due to iodine liberation which is then titrated with standard $Na_2S_2O_3$ solution to straw yellow colour. Now add 2 mL of 1% starch solution, as the solution turns deep blue. Continue titration with standard $Na_2S_2O_3$ solution till the blue colour of starch is faded away. Further add 10 mL 1% NH_4SCN, shake well as the blue colour of the solution deepens and continue the titration till blue colour is completely discharged and creamy white solution results. Record the titre value and repeat the experiment.

Step-4 : Estimation of Fe(III) ions : 25 mL of Fe(III) ions stock solution is pipetted out in a 500 mL beaker and diluted to 100 mL by water. Now 2 g of NH_4Cl is added, heated to boiling and 1:1 (v/v) ammonia is added dropwise till smell of ammonia persists. Reddish brown precipitate to ferric hydroxide appears and is allowed to settle down on a hot asbestos board, till the supernatant liquid shows no further precipitation of ferric hydroxide. Filter hot the resultant solution through Whatman-41 filter paper, wash with 1% NH_4Cl aqueous solution containing few drops of ammonia. Now the precipitate is dissolved in minimum volume of hot, 1:1 (v/v) HCl, followed by hot water and is collected in the same beaker where initial precipitation was carried out. (Re-precipitation process is carried out to remove unwanted, adsorbed Cu(II) ions).

Reduce the solution by Al foil and heat the solution carefully (avoid boiling), shake the solution continuously till the yellow colour is discharged.

[The reduction can also be carried out by adding the $SnCl_2$ reagent dropwise with constant stirring. When the yellow colour of the solution is fully discharged, add 1 drop of extra diluted $SnCl_2$ (1 : 1 HCl solution) and cool the solution under tap to room temperature. Then add 10 mL 10% $HgCl_2$ to the solution all at a time and swirl the solution and wait for 10 minutes. A silky white precipitate appears due to formation of Hg_2Cl_2. Excess reagent may otherwise transform Hg_2Cl_2 to colloidal mercury that may interfere in the titration and impart error to analysis.]

After reduction to Fe(II) ions, cool the solution and add 2g NH_4HF_2 or 5 mL phosphoric acid followed by 4-5 drops BaDS indicator. Titrate with standard (N/20) $K_2Cr_2O_7$ solution till the colour of the solution just changes from green to violet. Record the titre value.

Results :

Estimation of Cu(II) ions :

1. Preparation of 250 mL standard (N/20) $K_2Cr_2O_7$ solution :

Weight of $K_2Cr_2O_7$ taken (g)	Weight of $K_2Cr_2O_7$ to be taken (g)	Strength of $K_2Cr_2O_7$ solution
w_k	0.6129	$F = \left(\frac{w_k}{0.6129 \times 20}\right)$(N)

2. Standardization of $Na_2S_2O_3$ solution by standard (N/20) $K_2Cr_2O_7$:

No. of titrations	Volume of F(N) standard $K_2Cr_2O_7$ solution	Volume of thiosulphate $S_2O_3^{2-}$ consumed	Mean volume of consumed thiosulphate solution (mL)
1	25.0 mL	*X* mL	$V_2 = (X + Y)/2$
2	25.0 mL	*Y* mL	

N.B. : If consecutive readings do not differ by ± 0.1 mL, then a third set of titration to be performed.

So, strength of $Na_2S_2O_3$ solution $(S_t) = [25 \times F / V_2]$ (N)

3. Estimation of Cu^{2+} ions :

No. of titrations	Volume of stock solution	Volume of thiosulphate consumed	Mean volume of consumed thiosulphate solution (mL)
1	25.0 mL	*A* mL	$V_t = (A + B)/2$
2	25.0 mL	*B* mL	

N.B. : If consecutive readings do not differ by ± 0.1 mL, then a third set of titration to be performed.

■ **Calculation :** 1000 mL 1 (N) $Na_2S_2O_3 \equiv$ 63.54 g Cu

V_t mL S_t(N) $Na_2S_2O_3$ solution $\equiv [(63.54/1000) \times V_t \times S_t]$ g of Cu

Now , 25 mL stock solution contains $\equiv [(63.54/1000) \times V_t \times S_t]$ g of Cu

Therefore, 1000 mL - - - $\equiv [(40 \times 63.54)/1000 \times V_t \times S_t] = h$ g of Cu

Conclusion : The given/supplied solution contains ...*h*...g/L of Cu^{2+} ion.

4. Estimation of Fe^{3+} ions :

No. of titrations	Volume of stock solution	Volume of standard [F(N)] $K_2Cr_2O_7$ solution.	Mean volume of standard [F(N)] $K_2Cr_2O_7$ solution.
1	25 mL	V_{Fe} mL	V_{Fe} mL

- **Calculation :** 1000 mL 1 (N) $K_2Cr_2O_7 \equiv$ 55.85 g Fe

V_{Fe} mL F(N) $K_2Cr_2O_7 \equiv [55.85 \times V_{Fe} \times F/1000]$ g Fe = z g of Fe

Now , 25 mL stock solution contains z g of Fe

So 1000 mL stock solution contains (40 x z) g of Fe

- **Conclusion :** The supplied solution contains ....g/lit of Fe^{3+} ion
- **Overall conclusion :** Thus, the supplied mixture solution contains ....g/lit of Fe^{3+} ion and .... g/lit of Cu^{2+} ion respectively.

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