Experiment-1

Title of Experiment: Estimation of CO_2 content in Polluted Water

Introduction: CO_2 present in water is called free Co_2. Free CO_2 reacts with H_2O and form carbonic acid which is titrated with strong base (NaOH) or a weak base (Na_2CO_3).

Carbonic acid converts into neutral sodium bicarbonate, by adding phenolphthalein indicator during titration at pH 8.3 it gives pink colour.

$2NaOH + CO_2 = Na_2CO_3 + H_2O$

$Na_2CO_3 + H_2O + CO_2 = 2NaHCO_3$.

Free CO_2 is titrated by method given below:-

(a) NaOH (b) Na_2CO_3

(a) By NaOH:-

Materials required:

0.05 N NaOH:- 40 gm + 1000 ml DW Heat and then Cool.

Phenolpthalein:- 50 ml 95% Ethanol + 500 mg Phenolpthalien.

Procedure:

1. Collect 250-300 ml sample in Nessler tube carefully.

2. Take 100 ml sample from collected sample in a conical flask.

3. Add few drops of indicator if it shows pink colour then no free CO2 present in sample.

4. If it remains colourless then titrate it up to pink colour appearance.

5.Note the reading and repeat the process three times for better results.

Observation:

Sl.No.	Sample Volume	Burette Reading	Mean
		Start End Total	
1	100		
2	100		

3	100		

Calculation:

Chloride mg/L= A x N of NaOH X 1000 x 44

ml of sample

= A x 0.05 x 1000 x 44

100

Result : A x 22mg/L.

Experiment-2

Title of Experiment: To determine the adsorption of aqueous acetic acid by activated charcoal and to study the adsorption isotherm.

Introduction: Interaction between the adsorbed molecule and the solid surface varies over a wide range from weak non-polar van der Waals' forces to strong chemical bonding. Examples of adsorption where ionic or covalent bonding occurs are the adsorption of chloride ions on silver chloride (ionic) or of oxygen gas on metals where oxygen-metal bonds are formed (covalent). In these cases, the process is called chemisorption, and it is generally characterized by high heats of adsorption (from 10 to 100 Kcal per mole of gas adsorbed). Chemisorption is highly specific and depends on the chemical properties of both the surface molecules and the adsorbed molecules. Adsorption arising from the weaker van der Waals and dipole forces is not so specific and can take place in any system at low or moderate temperatures. This type of adsorption is called physical adsorption and is usually associated with low heats of adsorption (less than about 10 Kcal/mole).

The amount of solute adsorbed by a given quantity of adsorbent increases with the concentration of the solution. In some cases the layer of adsorbed molecules is only one molecule deep, and further adsorption stops when the surface of the crystal lattice is covered. The equilibrium between the dissolved solute and the material adsorbed also depends upon the nature of the solvent and the temperature, the amount adsorbed increasing at lower temperatures. From measurements at a constant temperature, one can obtain a plot of x/m, the number of grams adsorbed per gram of adsorbent, vs. c, the equilibrium solute concentration. This is called an adsorption isotherm.

Material Required: Stoppered bottles, Pipette, burette, beakers, powdered activated Charcoal, glacial Acetic acid, 0.1N NaOH

Procedure: Proceed stepwise as follow:

1. Take 50,40,30,20 and 10 ml of 0.5N acetic acid in stoppered bottles and to these add 0,10,20,30 and 40 ml of distilled water respectively and label them.

2. Transfer 1 gm of activated charcoal to each bottle then shake these vigorously for about one hour and allow them to stand for half an hour to attain the room temperature.

3. Filter off charcoal while filtering discard nearly 4 ml of filtrate in each case.

4. Now take 5 ml of each filtrate and titrate it against standard solution of 0.1 NaOH using phenolphthalein as an indicator.

5. Note the reading in each case.

Observation:

1. Room temperature = 40 Degree C

2. Amount of Charcoal used in each bottle = 1gm

Bottle No.	Volume of 0.5N CH_3COOH (ml)	Volume of water added	Initial normality of the acid C_2	Volume of NaOH used, V
1	50	0	0.50	10.5
2	40	10	0.40	8.3
3	30	20	0.30	6.1
4	20	30	0.20	4.0
5	10	40	0.10	1.8

Calculate the Normality of the acid after adsorption using $N_1V_1 = N_2V_2$

The amount of acid adsorbed, x = N X 60/1000 gm

Result in tabular form:

Bottle No.	Initial normality of acid	Normality of the acid after adsorption	Amount of acid adsorbed x	logx/m	LogC
1	0.52	0.42	315	2.50	3.10
2	0.41	0.32	270	2.43	2.98
3	0.30	0.23	210	2.32	2.85
4	0.20	0.15	150	2.25	2.65
5	0.09	0.06	75	1.87	2.29

Plot the graph between log x/m and log C. The slope is equivalent to 1/n and the intercept to log K.

Result: The trend of the graph is in accordance with Freundlich adsorption isotherm.

Precaution:

1. Shake all the the solutions properly.

2. Filter the solution before proceeding for titration and discard the initial small volume of the filtrate.

3. Do not use wet filter paper in filtration as it may dilute the solution.

4. Avoid to breathe the fumes of acetic acid.

SOP prepared by Department of Chemistry, S.S. khanna Girls Degree College, University of Allahabad for newly introduced experiment under strengthening component of star college scheme of DBT,GOI

Experiment-3

Title of Experiment : To determine the molecular weight of an unknown volatile liquid using the Victor Meyer apparatus.

Introduction: The Victor Meyer method consists of evaporating a unknown liquid of known weight in a chamber maintained at an appropriate and constant high temperature. The air displaced from the chamber by the vaporized sample is cooled to room temperature, and its volume is carefully measured. The substitution of air for vapour makes it possible to determine the volume that the known mass of vapour would occupy at room temperature if it could be cooled without condensation.

The apparatus consists of an inner Victor Meyer tube, the lower end of which is shaped like a pear. At the upper end of the tube is a side tube leading to a trough filled with water. The Victor-Meyer tube is surrounded by an outer jacket. Inside the outer jacket is a liquid that boils at a temperature at least 30 K higher than the substance being tested. The lower end of the Victor-Meyer tube is covered with a small amount of glass wool or asbestos to prevent it from breaking when a glass bottle containing the substance to be tested is placed inside it.

During this method, a known mass of a volatile solid or liquid to be studied is converted to its vapour form by heating in a Victor-Meyer tube. The vapour displaces its own vapour of air. The volume of air displaced at the experimental temperature and pressure is calculated. Then the volume of air displaced at standard temperature and pressure is calculated. From this, the mass of air displaced at $2.24 \times 10\text{-}2\ m^3$ of steam at STP is calculated. This value corresponds to the molecular mass of the substance. It should be noted that it is not necessary that the temperature of the vaporization chamber be known, but it must be constant.

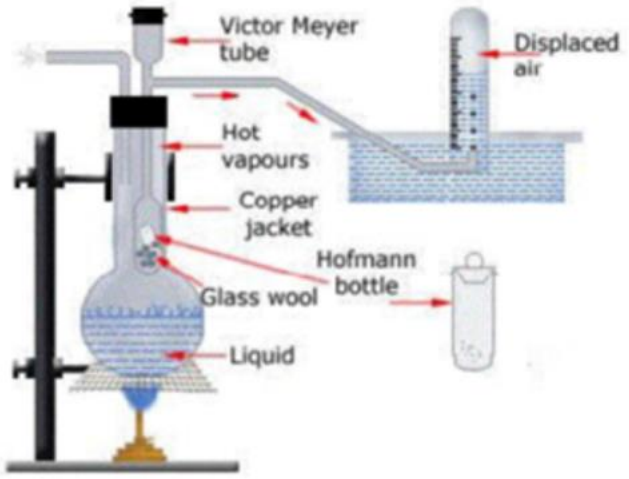


Fig. 1 Victor Meyer Apparatus

Procedure: The apparatus should be assembled as in Figure 1. A round-bottomed flask filled with a liquid whose temperature is 10 °C above that of the volatile liquid serves as the outer glass jacket. A Victor-Mayor tube with an outer tube serves as the inner glass jacket. This outer tube is immersed in a trough filled with water. The bottom of the Victor-Mayors tube is made of Hg or asbestos pieces for cushioning.

A small glass tube is known as Hoffman's bottle with a stopper is cleaned, washed and dried and weighed. Volatile Liquid is taken in Hoffman's bottle with the help of syringe and weighed. A Eudiometer tube filled with water is placed over the tube connected to Victor Mayor apparatus. It is desired to have an amount of liquid which will give a vapour volume of 15 to 20 ml. The liquids which we can use are: dichloromethane, ethyl acetate, and methanol. Here we have used acetone while performing this experiment. Once the vaporizing chamber has reached a constant temperature we are ready to begin the experiment.

Now Hoffman's bottle is dropped in the Victor Mayor's tube. The heat causes the liquid in the bottle to evaporate, blowing off the stopper and displacing the air corresponding to its own volume, which collects in a eudiometer tube by the downward displacement of the water. The volume of displaced air is noted, which in fact corresponds to the volume of vapours. The temperature and pressure of water are also noted.

Observation :

The weight of the Hoffman's Bottle = w_1 gm.

The weight of the Hoffman's bottle + Liquid = w_2 gm.

Weight of the volatile liquid = w_1-w_2 = w gm = 0.1 gm

Pressure = (P – p) mm of Hg = 766- 16 = 750 mm Hg

p = Aqueous tension of water at that temperature = 16 mmHg

Temperature = t °C = T K = 288K

Volume of vapours = V ml = 40 ml

Now the volume of vapours at S.T.P. from the above data is calculated using following formula.

T_0 = Temperature at STP = 273 K

P_0 = Pressure at STP = 760 mm Hg

$$\frac{P_0V_0}{T_0} = \frac{P_1V_1}{T_1} \qquad V_0 = \frac{P_1V_1T_0}{T_1P_0} = \frac{(P-f)V\,T_0}{T\,P_0} = 0.0374 \text{ ml}$$

$$\frac{weight\ of\ volatile\ compound}{Molecular\ weight\ of\ volatile\ compound} = \frac{V_0}{22400}$$

Molecular weight of volatile compound = $\frac{22400}{V_0}$ x weight of volatile compound

Molecular weight of volatile compound = 59.9 g

Result : The molecular weight of an unknown volatile liquid using the Victor Meyer apparatus is 59.9 g .

Precautions :

1. Syringe should be used for pouring unknown volatile compound.
2 . Electronic balance should be properly calibrated before use.

Experiment-4

Title of Experiment: To determine molecular weight of a unknown volatile substance by Duma's method.

Introduction: The molecular weight of any compound is actually the sum of the atomic masses of all its element present . One of the earliest experimental techniques for calculating the molecular weight of a volatile substance is given by Dumas as Dumas method. In this method ideal gas equation is used to calculate the number of moles of a vapour which is being trapped in a flask at a certain temperature and pressure.

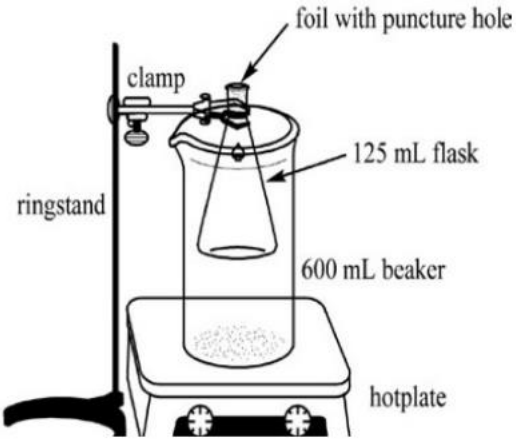
PV=nRT where P is measured in atmosphere, V in litres, R = 0.08206 (L . atom /mol K). T is absolute temperature in Kelvin. Number of moles is actually the weight divided by molecular weight

PV = w . RT / MW or MW = w RT / PV

During the experiment as we make tiny hole on the aluminium foil. Pressure inside the flask will be same as atmospheric pressure and volume of volatile compound occupied will be same as the volume of water it holds.

Procedure :

1. Prepare a hot water bath by keeping water in 500ml beaker and put it on the flame.
2. Take Erlenmeyer flask of 125 ml which was dried in incubator earlier and brought to room temperature for better result.
3. Weigh Erlenmeyer flask and aluminium foil.
4. Now put 5ml of unknown solution with the help of syringe by making a very tiny hole on the aluminium foil.
5. Vaporize the unknown solution completely by putting it inside water bath so formed. Measure the temperature of water bath.
6. At the moment liquid vaporizes completely take it out (approximate time 3-4 min) and put it inside the ice bath to liquefy vapour so present in Erlenmeyer flask .
7. After sometime take it off and wipe the moisture on the flask present on outside surface and bring it to the room temperature and weigh it.
8. Now rinse the Erlenmeyer flask and pour distil water to its maximum capacity. Pour that water into the measuring flask and measure it.



Dumas Experimental Set up

Observation:

Mass of empty Erlenmeyer Flask and Aluminium Foil: 125.7822 g

Mass after condensation : 126.3828 g

Barometric Room Temperature : 754.1 mmHg

Pressure in atmosphere : $\frac{754.1\ mm\ Hg\ \ x\ \ 1\ Atm}{760\ mm\ Hg}$ = 0.9922 Atm

R = 0.08206 L atm / mol K

Temperature of water bath = 100^{o} C = 100 + 273 = 373 K

Volume of the water hold by Erlenmeyer flask = 150.72 ml

Mass of the gas = (mass after condensation) – (initial mass of empty Erlenmeyer flask and aluminium flask) = 126.4108 – 125.7822 =0.6286 g

Putting values in the formula :

Mol. Wt. = $\frac{w\ R\ T}{PV}$ = …….123.7

Result: The unknown compound's molecular weight obtained is 123.7 (unknown compound given $CHCl_3$ (Chloroform) whose molecular weight is 119.38).

Precautions :

1. The Erlenmeyer flask taken should be kept in incubator for drying so that it become completely dried .
2. Very tiny hole should be made on aluminium foil.
3. Syringe should be used for pouring unknown volatile compound.
4. At the moment volatile compound volatizes completely flask should be removed from water bath.
5. Electronic balance should be properly calibrated before use.

Experiment-5

Title of Experiment: To determine molecular weight of a unknown volatile substance by Duma's method.

Introduction: The molecular weight of any compound is actually the sum of the atomic masses of all its element present . One of the earliest experimental techniques for calculating the molecular weight of a volatile substance is given by Dumas as Dumas method. In this method ideal gas equation is used to calculate the number of moles of a vapour which is being trapped in a flask at a certain temperature and pressure.

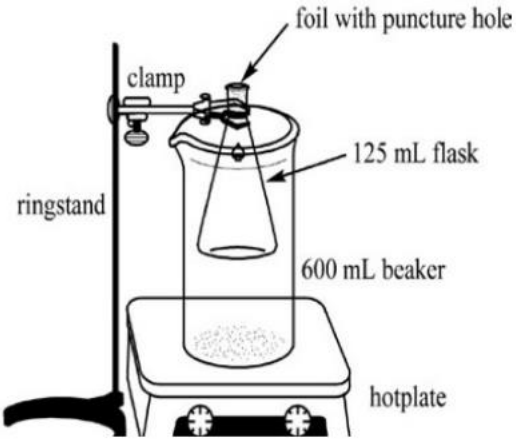
PV=nRT where P is measured in atmosphere, V in litres, R = 0.08206 (L . atom /mol K). T is absolute temperature in Kelvin. Number of moles is actually the weight divided by molecular weight

PV = w . RT / MW or MW = w RT / PV

During the experiment as we make tiny hole on the aluminium foil. Pressure inside the flask will be same as atmospheric pressure and volume of volatile compound occupied will be same as the volume of water it holds.

Procedure :

9. Prepare a hot water bath by keeping water in 500ml beaker and put it on the flame.
10. Take Erlenmeyer flask of 125 ml which was dried in incubator earlier and brought to room temperature for better result.
11. Weigh Erlenmeyer flask and aluminium foil.
12. Now put 5ml of unknown solution with the help of syringe by making a very tiny hole on the aluminium foil.
13. Vaporize the unknown solution completely by putting it inside water bath so formed. Measure the temperature of water bath.
14. At the moment liquid vaporizes completely take it out (approximate time 3-4 min) and put it inside the ice bath to liquefy vapour so present in Erlenmeyer flask .
15. After sometime take it off and wipe the moisture on the flask present on outside surface and bring it to the room temperature and weigh it.
16. Now rinse the Erlenmeyer flask and pour distil water to its maximum capacity. Pour that water into the measuring flask and measure it.



Dumas Experimental Set up

Observation:

Mass of empty Erlenmeyer Flask and Aluminium Foil: 125.7822 g

Mass after condensation : 126.3828 g

Barometric Room Temperature : 754.1 mmHg

Pressure in atmosphere : $\frac{754.1\ mm\ Hg\ \ x\ \ 1\ Atm}{760\ mm\ Hg}$ = 0.9922 Atm

R = 0.08206 L atm / mol K

Temperature of water bath = 100^{o} C = 100 + 273 = 373 K

Volume of the water hold by Erlenmeyer flask = 150.72 ml

Mass of the gas = (mass after condensation) – (initial mass of empty Erlenmeyer flask and aluminium flask) = 126.4108 – 125.7822 =0.6286 g

Putting values in the formula :

Mol. Wt. = $\frac{w\ R\ T}{PV}$ = …….123.7

Result: The unknown compound's molecular weight obtained is 123.7 (unknown compound given $CHCl_3$ (Chloroform) whose molecular weight is 119.38).

Precautions :

6. The Erlenmeyer flask taken should be kept in incubator for drying so that it become completely dried .
7. Very tiny hole should be made on aluminium foil.
8. Syringe should be used for pouring unknown volatile compound.
9. At the moment volatile compound volatizes completely flask should be removed from water bath.
10. Electronic balance should be properly calibrated before use.

Experiment-6

Title of Experiment: Acetylation of Aniline

Introduction:

Acetanilide is synthesized from aniline by acetylating it with acetic anhydride in presence of glacial acetic acid. Aniline or phenylamine is a primary amine and basic in nature. Acetic anhydride, an anhydride of acetic acid, acts as a source of acyl group here. Aniline reacts with acetic anhydride to form Acetanilide by nucleophilic substitution reaction and the reaction is called acetylation. In this reaction, aniline acts as the nucleophile and acyl (CH_3CO-) group from acetic anhydride acts as the electrophile. Here, the hydrogen atom of $–NH_2$ group is replaced by the acyl group.

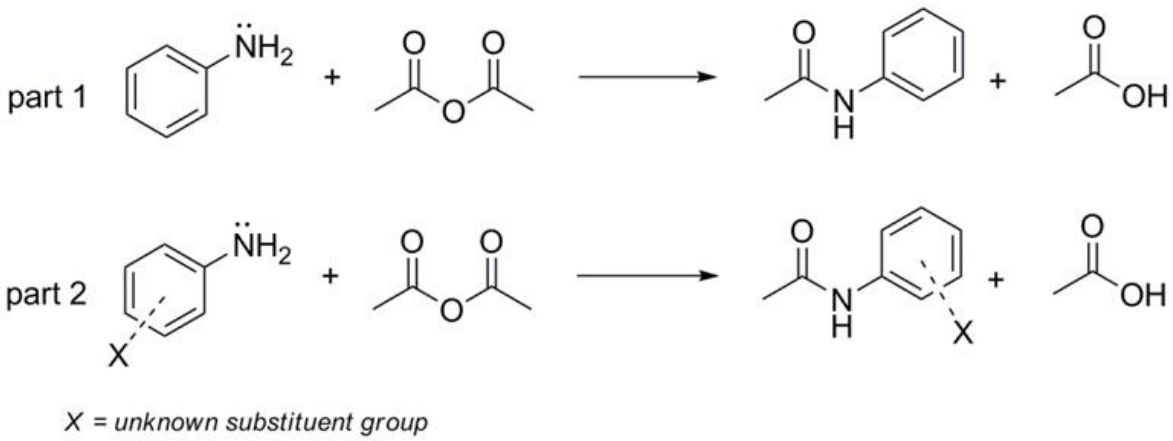


Figure 1

Materials required:

Chemicals: **1. Acetic acid/anhydride mixture – 20 ml**

2. Aniline – 10 ml

Apparatus: **1. Conical flask, 250 ml 2. Reflux water-condenser set 3. Buchner funnel 4. Measuring cylinder 5. Filter paper**

Procedure:

1.Add 20 ml of a mixture of acetic anhydride and glacial acetic acid (equal volumes) to 10 ml (10.3 g) of aniline in a conical flask of 250 ml.

2. Fit a reflux water-condenser to the flask and gently boil the mixture for 10 min. Then pour the hot liquid into 200 ml of cold water with constant stirring.

3.The acetanilide quickly crystallizes. Filter yield by a pump and wash the crude acetanilide well with water. Recrystallizes from about 60 ml of a mixture of one volume of acetic acid and two volumes of water

4. filter off the colorless crystals at the pump, again wash thoroughly with water, drain and dry.

Note: Alternatively, the crude acetanilide may be recrystallised from boiling water, but in this case, a much greater volume (about 300 ml) of the solvent will be required.

Observation:

Molecular formula of acetanilide = C_8H_9ON

Molecular weight of aniline = 93 g/mole

Molecular weight of acetanilide = 135 g/mole

Theoretical yield:

1 g aniline forms 135 g acetanilide

Therefore, 10.3 g (10 ml) aniline will form ……? (X) g acetanilide

X =(135 × 10.3)/93 = 14.95 g

Theoretical yield = 14.95 g

Practical yield = ————- g

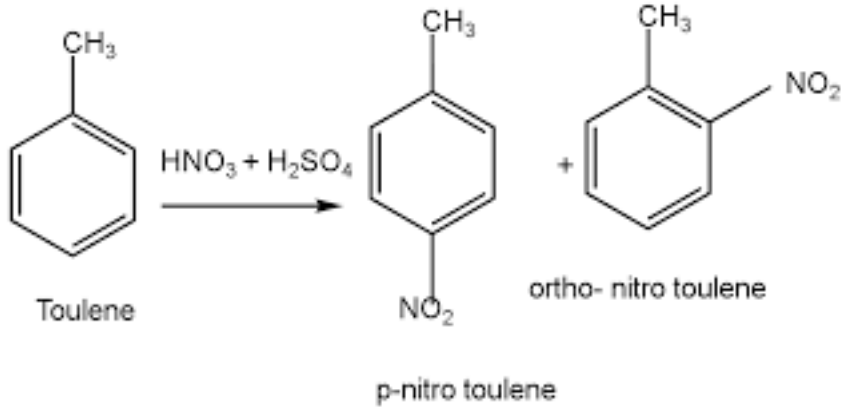
% Yield = (Practical Yield)/(Theoretical Yield) × 100

Result: Acetanilide was synthesized and the percentage yield was found to be….% (M.p. 113°; yield, 10 g.).

Experiment-7

Title of Experiment: Nitration of Toluene

Introduction: Electrophilic aromatic substitution represents an important class of reactions in organic synthesis. In "aromatic nitration," aromatic organic compounds are nitrated via an electrophilic aromatic substitution mechanism involving the attack of the electron rich benzene ring on the nitronium ion. The formation of a nitronium ion (the electrophile) from nitric acid and sulfuric acid is shown below. The sulfuric acid is regenerated and hence acts as a catalyst. It also absorbs water to drive the reaction forward. The mechanism for the formation of a nitronium ion. The methyl group of toluene makes it around 25 times more reactive than benzene in electrophilic aromatic substitution reactions. Toluene undergoes nitration to give ortho and para nitrotoluene isomers, but if heated, it can give dinitrotoluene and ultimately the explosive trinitrotoluene (TNT).



Materials required: Toluene, ice, Nitric acid, Sulphuric acid

Procedure:

1. Place a 5 mL conical vial, equipped with a spin vane, in a crystallizing dish filled with ice water placed on a stirrer.

2. Pour 1.0 mL of concentrated nitric acid into the vial. While stirring, slowly add 1.0 mL of concentrated sulfuric acid.

3. After the addition of sulfuric acid is complete, add 1.0 mL of toluene dropwise and slowly over a period of 5 minutes (slow down if you see boiling). Reaction produces a lot of heat.

4. While Stirring,allow the contents of the flask to reach room temperature. Stir at room temperature for another 5 minutes.

5. Add 10 mL of water into a small separatory funnel. Then transfer the contents of the flask (from step 4) into the funnel. Rinse the contents with 4 mL of diethyl ether and add it to the separatory funnel. Repeat the Ether rinsing one more time with a fresh 4 mL portion of ether.

6. Gently shake the contents of the separatory funnel, let it settle, and remove the aqueous layer.

7. Wash the organic layer with a 10 mL portion of 10% sodium bicarbonate (shake and VENT due to the formation of gas), and finally, wash it with 5 mL of water

8. Discard the aqueous layer and dry the organic layer using anhydrous sodium sulfate

9. Evaporate the solvent (it is critical to keep the temperature as low as possible). (Do not overheat) and obtain the isolated nitrotoluene.

10. Obtain an IR spectrum of the product.

Observation:

Precautions-

1. Minimize or eliminate use and storage of acid whenever possible.

2. Use in ventilated areas and in proximity to eyewash and safety shower stations while wearing compatible gloves, safety goggles, and a lab coat.

3. Avoid contact with metals! Nitric acid is extremely corrosive in the presence of aluminum, copper, and oxides and attacks all base metals.

4. Store in glass containers that are secured, dry, and cool (<23'C/73.4'F), away from sources of ignition, combustible materials, other acids, bases, cyanides, and acetone. Use secondary containers to segregate nitric acid from other acids in your acid cabinet.

5. Storage containers must be dry, as nitric acid can react with water or steam to produce heat and toxic, corrosive, and flammable vapors.

6. Pre-labeled and dated safety-coated glass bottles (PTFE) may be used for nitric acid waste; avoid using empty organic solvent bottles.

7. Proper waste segregation can help avoid laboratory accidents and explosions. Do not mix nitric acid waste with any other waste streams, including other inorganic acids.

8. Segregation of nitric acid waste from different processes or experiments is recommended.

9. In the case of a spill, absorb nitric acid with an inert dry material (earth, sand, or other non-combustible material), place in an appropriate waste container, and neutralize with dilute sodium carbonate.

Experiment-8

Title of Experiment: To determine the transition temperature of Glauber's salt ($Na_2SO_4.10H_2O$) by thermometric method.

Introduction: When temperature is raised, the hydrated salt becomes partially liquid and a point is reached at which temperature of the salt remains practically stationary until conversion is complete. It is due to absorption of heat during transition of hydrated form into another or anhydrous form. This point is transition point. During cooling again a point is reached at which the temperature of salt remains practically stationary for a certain interval. It is due to evolution of heat during the change of anhydrous form to hydrated form. This temperature is also the transition temperature. Hence both the temperature must coincide theoretically

Materials Required: Thermometer (0.1* C), Ordinary thermometer, thin walled glass tube, stirrers, hydrated salt, beaker and burner, etc.

Procedure

1. Take about 40-50 gm of powdered $Na_2SO_4.10H_2O$ in a thin walled test tube

2. Insert a thermometer (0.1*C) and stirrer into the salt.

3. Heat the beaker gently and stir the water regulary

4. Read the temperature of the salt after every minute.

5. When the temperature reaches to 40* C stop heating

Observation

S.No.	Timing (min) (*C)	While heating (*C)	While cooling(*C)
1.	5	28.2	32.4
2.	6	29.1	31.7
3.	7	30.2	30.3
4	8	30.3	30.1
5	9	30.5	29.6
6	10	30.8	28.4
7	11	31.4	27.2

8	12	32.4	26.4

Calculation: Plot temperature on x axis, and time on y axis. The mean position of heating and cooling curve is the transition temperature of Glaubers salt.

Result: The transition temperature of $Na_2SO_4.10H_2O$ is 32 *C

Precautions:

1. The salt should be well powdered

2. Addition of salt should be quick

3. Thermometer reading should be taken carefully

SOP prepared by Department of Chemistry, S.S. khanna Girls Degree College, University of Allahabad for newly introduced experiment under strengthening component of star college scheme of DBT,GOI