University of Baghdad
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Practical physical chemistry For second year students of Chemistry

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2019-2018

Experiment No. 1

Part (A)

Density

Determination of the relative and absolute densities of a liquid or solution

Introduction:

The absolute density of a liquid is the mass of a unit volume of the substance. The relative density at a given temperature is the density relative to that of a standard substance (water).

It is easily determined by means of a vessel of accurately definite volume.

Experiment Method:

1 . Weight the empty and dried volumetric flask with its cover W_1.
2 . Weight it again after fill it with distilled water W_2.
3 . Remove the water from it, then dried it and rinse with the unknown liquid, weight again W_3. Wash it with water.

Calculation:

Let W_2-W_1 be the weight of distilled water.
Let D_1 be the density of distilled water.
If V is the volume of volumetric flask.
Let W_3-W_2 be the weight of liquid that fills the volumetric flasks.
Let D_2 be the density of this liquid, then:

$$D_1 = \frac{W_2 - W_1}{v} \qquad \text{………… (1)}$$

$$D_2 = \frac{W_3 - W_1}{v} \qquad \text{…………..(2)}$$

The relative density of the liquid= $\frac{W_3 - W_1}{W_2 - W_1}$ ………..(3)

Experiment No. 2
Part (B)

Viscosity

Determination of the relative and absolute viscosities of a liquid and its variation with temperature

Introduction:

A liquid moving through a tube may be considered as composed of concentric layers moving with different velocities. Layers adhering to the sides of the tube are considered to be stationary, and the velocity increases as the middle of the tube is approached.

Hence, there will be a velocity gradient between the layers due to fractional forces acting between the different layers of the liquid. These forces are responsible for the so called viscosity of the liquid.

It has been shown experimentally that the tangential force, f, required to maintain a constant different between the velocities of the parallel layers of liquid moving in the same direction varies directly with the difference in velocity U, and the area A, of the surface of contact of the two layers, and inversely as the distance d, between the layers that is:

$$f = \mu \frac{A\mathrm{u}}{d} \quad \ldots\ldots\ldots\ldots(4)$$

Where μ is a proportionality factor known as the coefficient of viscosity. The unit of viscosity is the poise. This is defined as the force necessary to move a layer of liquid of area $1\mathrm{cm}^2$ with a velocity of $1\ \mathrm{cm}^2$ per second, past another layer at distance of one centimeter. The viscosity of a liquid is generally measured by observing the time required for a definite volume of liquid to flow through a standardized capillary tube under a known deference of pressure. The apparatus commonly used in the laboratory is the outwards viscometer. The low governing the flow of liquids through capillary tubes was discovered by Poiseulle, and is given by the relation:

$$\mu = \frac{\pi p r^4 t}{8V\ell} \quad \ldots\ldots\ldots\ldots\ldots(5)$$

in which V denotes the volume of a liquid of viscosity flowing through a capillary tube of length ℓ, radius r in time t and under the pressure p.

If the time of flow of equal volumes of two liquids through the same capillary are measured under the same experimental conditions, it follows that:

$$\frac{\mu_1}{\mu_2} = \frac{p_1 t_1}{p_2 t_2} = \frac{d_1 t_1}{d_2 t_2} \ldots\ldots\ldots (6)$$

Where μ_1 denote the coefficient of viscosity of unknown liquid and μ_2 denote the coefficient of viscosity of water, d_1 and d_2 denote their density, and t_1 and t_2 denote their times of flow. This equation is used to calculate the so called relative viscosity. Water is quite generally accepted as the standard of reference in determinations of relative viscosity. If the absolute viscosity of water is known, that of liquid can be calculated.

The viscosity of liquids varies with temperature according to the following equation:

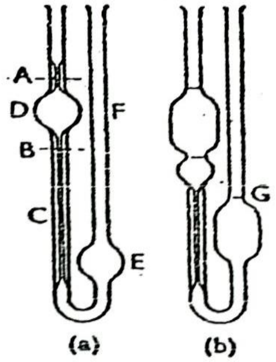
$$\mu = Ae^{E/RT}$$

$$\log \mu = \log A + \frac{E}{2.303RT} \ldots\ldots\ldots(7)$$

A = constant
R = gas constant
T = absolute temperature
E = activation energy that is necessary for the process of flowing of liquids.

Experimental method:

1. Cleaned and dried the viscometer in the same way as described in part A.
2. The viscometer is clamped vertically in a water thermostat. It must be dipped in the thermostat down to the mark (a).
3. A definite volume of the liquid 10 ml is introduced in the wide tube (limb C) of the viscometer and is left until it acquires temperature of the bath.
4. Weight the empty volumetric flask, filled with a liquid, put it with the viscometer in the water bath, leave it for 10 min.
5. The liquid in the viscometer is forced up through the capillary tube by suction through a rubber tube attached to the end (a) of the viscometer until the liquid fills the tube (E) and rises slightly above the mark (0). The volume of the liquid introduced must be sufficient to fill the bulb (E), the lower bend (f) and extends up slightly in to wider tube (C), otherwise, air bubbles will form in the capillary and affect the time of flow.
6. Repeat the above steps on freshly distilled water using exactly the same volume that taken of the liquid and the same temperature.
7. Repeat step 4 and 5 at different temperatures (20,30,40 and 50).



Calculation:

1 . Let the time of flow of the water be t_1 its density d_1, and its viscosity μ_1. The time of flow of liquid be t_2 its density d_2, and its absolute viscosity μ_2. Then:

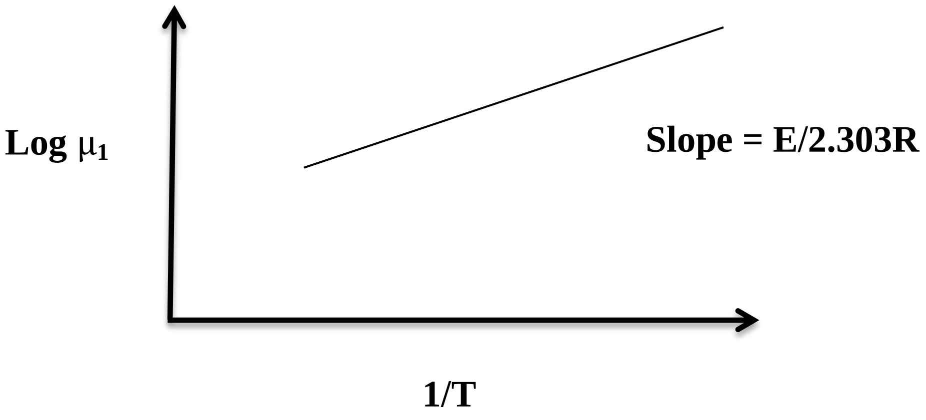
$$\frac{\mu_1}{\mu_2} = \frac{t_1 d_{1\,(water)}}{t_2 d_{2(liquid)}}$$

Poise = dynes /cm^2/unit velocity gradient.
Viscosity of water at 35C° = 0.08937.

2 . Put the results as in the following table:

C°	T (K)	t_1 (sec)	t_2 (sec)	μ_1	μ_2	log μ_2	1/T (K^{-1})

3 . Plot a curve between log μ_1 against 1/T then calculate the activation energy from the slope of the straight line.



Experiment No. 2

Heat of solution

Determination of heat of solution from solubility measurements

Introduction:

When a solution is dissolved in a solvent, heat may be absorbed or evolved; in general, the heat of solution depends on the concentrate of the final solution. The integral heat of solution is the enthalpy change for the solution of 1 mole of solute in 1 mole of solvent.

It might be expected that heat would always be absorbed in overcoming the attraction between the molecules or ions of the solid solute.

Another process which commonly occurs is a strong interaction with the solvent, referred to as solvate, which evolves heat.

With many crystals the heat evolved by solvate is less than the heat absorbed in the separate of the units of the crystal. And so heat is absorbed in the solution process.

The differential heat of solute is the heat of solution of 1 mole of solute in a 1000 ml of solvent so large that the additions of one more mole of solute dose not change the concentration appreciably. The differential heat of solution depends on the concentration of the solution.

The equilibrium between a solid and its saturated solution may be represented as:

Solid solute $\longleftrightarrow$ dissolved solute

Equilibrium constant K_s represented as:

$$K_s = \frac{dissolved\ solute}{solid\ solute}$$

K_s α Dissolved solute C_s

C_s is the molar concentration of dissolved solute. If ΔH is the change in heat content when one mole of solute is dissolved in a large volume of the neatly saturated (heat of solution), then according to Vant Hoff equation:

$$\frac{d\ ln\, K_S}{dT} = \frac{\Delta H}{RT^2} \quad \ldots\ldots\ldots(8)$$

$$\frac{d \ln C_S}{dT} = \frac{\Delta H}{RT^2}$$

And by integration:

$$\ln C_s = \frac{-\Delta H}{RT} + C$$

$$\log C_s = \frac{-\Delta H}{2.303RT} + \text{constant(C)} \ldots\ldots\ldots..(9)$$

$$\log C_s = \frac{\Delta H}{2.303R}\left[\frac{1}{T_m} - \frac{1}{T}\right] \ldots\ldots\ldots\ldots\ldots\ldots.(10)$$

Where T_m melting point of the solute, $\log C_s$ the solubility at any temperature.

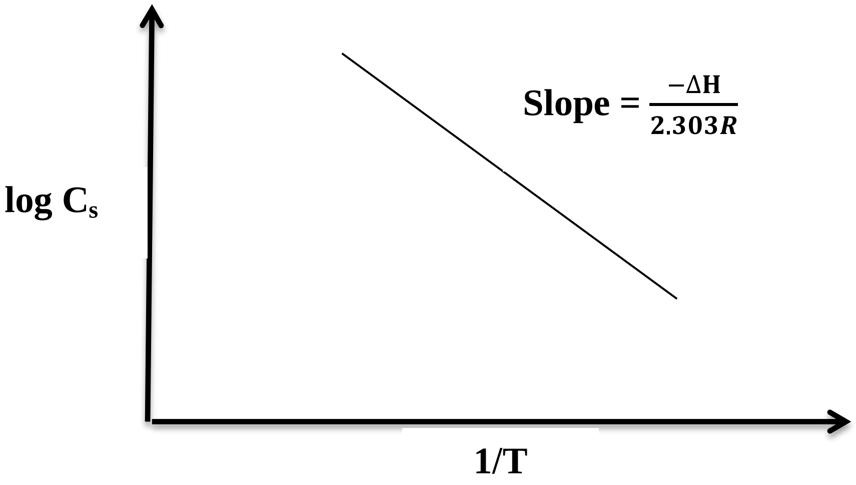


Fig.4: curve between log Cs against 1/T.

C_s = dissolved solute.
T = absolute temperature.
ΔH = heat of solution.
R = gas constant = 8.314 J K^{-1} $mole^{-1}$ or R = 1.987 cal.K^{-1}.$mole^{-1}$.
Integration of Vant Hoff equation between two different temperatures (limited integration) will give the following equation:

$$\log \frac{C_{s(2)}}{C_{s(1)}} = \frac{-\Delta H}{2.303R}\left(\frac{T_1 - T_2}{T_1T_2}\right) \ldots\ldots\ldots\ldots(11)$$

Experiment Method:

1. Prepare 100 ml of standard solution of 0.06 N NaOH.
2. Put 75 ml of distilled water in a large test tube is saturated with benzoic acid crystals at about 80C°, adjust the temperature by the use of thermostat.
3. After equilibrium is reached, at 70C° transfer by means of a pipette 5 ml (which warmed by washing it with hot distilled water) of the clear supernatant liquid into a conical flask, the tip of the pipette is plugged with a small roll of cotton wool so as to prevent withdrawal of fine crystals with the solution, then wash the pipette again with hot distilled water to be sure that all the acid is pour.
4. Titrate the solution against 0.06 N NaOH, using few drops of ph.ph. as an indicator.
5. Repeat the above steps at the temperatures (60C° ,50C° ,40C° ,and 30C°).

Calculation:

1 . Calculate the normality concentration (N)at each temperature.

2 . Arrange your data as the following table:

T (C°)	**T (K)**	**Volume of NaOH (ml)**	**Normality (N)**	**Solubility (C_s)**	**log C_s**	**1/T (K^{-1})**

3 . Calculate the solubility C_s by:

$$N = \frac{wt(C_s)}{eq.wt} * \frac{1000}{V}$$

4 . Plot (log C_s) against (1/T) as in equation No.9.

5 . From the slope of the straight line. Calculate ΔH practically.

6 . Calculate ΔH theoretically from two temperatures by equation No.11 then compare it with the value of ΔH practically.

7 . Calculate the melting point of the solute (T_m) from equation No. 10.

Experiment No. 3

Molecular weight determination

Freezing points, cooling curves, and cryoscopic determination of molecular weight

Introduction:

The freezing point of a liquid is the temperature at which die solid and the liquid are in equilibrium. Freezing points of pure liquids are used to identify materials and as a criteria of purity.

When a liquid consisting of one component (pure) is cooled the plot of temperature versus time (cooling curve) has a nearly constant slope. At die temperature at which the solid crystallized out, the cooling curve becomes horizontal if the cooling is slow enough. The halt in the cooling curve results from the heat evolved when die liquid solidified (fig. 5).

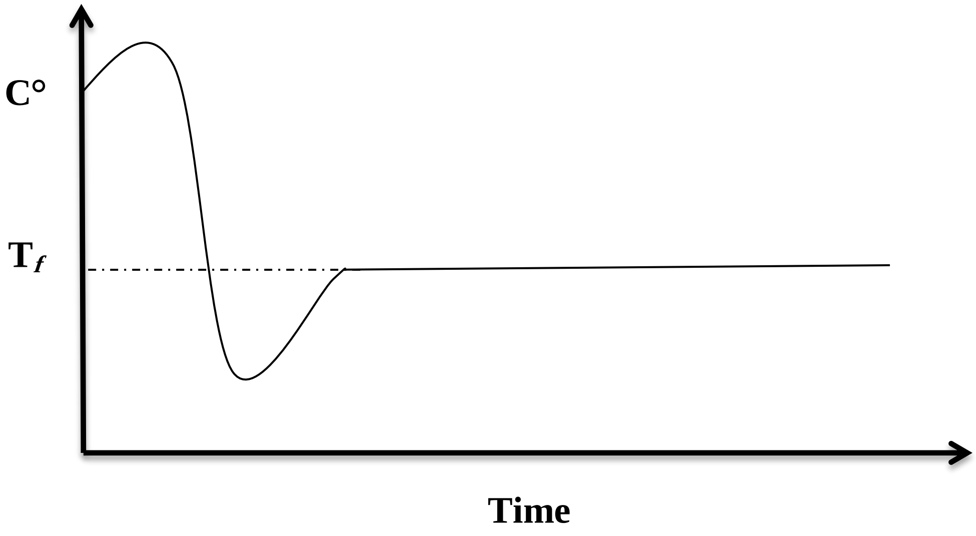


Fig. 5: The halt in the cooling curve results from the heat evolved when the liquid solidified.

The dip below the freezing point is due to super cooling. Freezing points are usually, but not always, depressed by the presence of solutes. This fact is used in molecular weight determinations. If solution is ideal, the following relation may be applied:

$$T_{\circ} - T = \Delta T = K_f * \frac{W_1 * 1000}{W_2 M_1} \text{ ....}(12)$$

$$\Delta T = K_f * m \text{ ....}(13)$$

M_1: molecular weight of solute.
W_1: weight of dissolved solute.
W_2: weight of solvent.
m: Molarity of the solution.
K_f: is called the molar depression constant. It is a constant characteristics of a certain solvent, and is defined as the lowering of the freezing point cause by dissolved 1g molecular of solute in 1000 ml of solvent.

The cooling curve for the solution (fig. 6) shows no horizontal portion, because the solution becomes more concentrated as the solvent freezes out and the freezing point drops continuously. Line a-d is extrapolated back to interact line a-b. The intersection is the true freezing point of the solution.

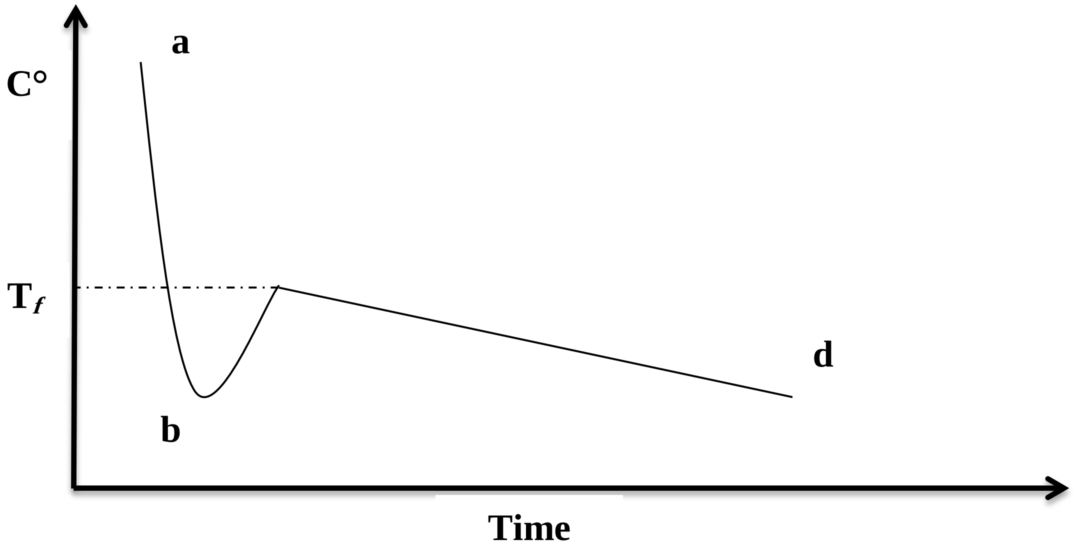


Fig. 6: The cooling curve for the solution shows no horizontal portion.

1. Determination of K_f for benzene:

Apparatus: the apparatus is shown in the figure below:

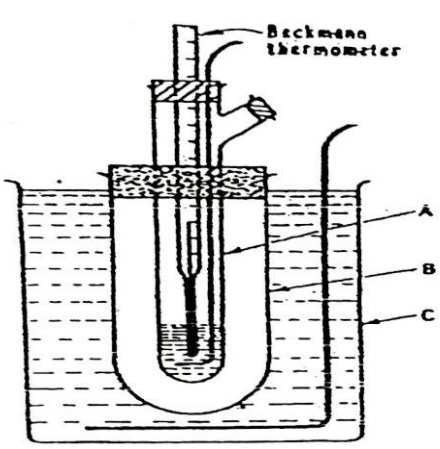


Fig. 7: Beckmann's fp. depression apparatus.

Experiment Method:

1. Introduce 10 ml of water in tube (A) fitted with a Beckmann thermometer as shown in the Fig.. This tube inserted in the air jacket in the air jacket (B), and the whole arrangement is placed in cooling mixture of ice and common salt. The cooling mixture is contained in the glass vessel (C) covered with metallic lid carrying (B) and another stirrer to maintain the temperature uniform.
2. Record the water temperature each minute with stirring it continually.
3. Dissolve about 1g of a known substance, then repeat step 2 and determine the freezing of the solution.
4. Repeat the above steps by using unknown substance.

Calculation:

1 . Plot the cooling curves to determine the freezing points of pure water $T_{\circ}$.

2 . Plot the cooling curves to determine the freezing points of solution T.

3 . Calculate ΔT:

$$\Delta T = T_{\circ} - T$$

4 . Use the formula given above equation (12) to estimate K_f for water.

5 . Repeat steps 2,3. Then calculate the molecular weight of unknown substance.

Experiment No. 4

Density of gases and vapors

Molecular weight determination by Victor Meyer method

Introduction:

The density of a gas or vapor is defined as the weight of unit volume measured under specified conditions of temperature and pressure (0°C and 1 atmospheric pressure).

Relative densities are the ratio of the density of the gas to that of a reference gas, usually Hydrogen or Oxygen at the same temperature and pressure.

Avogadro's hypothesis states that equal volumes of gases at the same temperature and pressure contain equal numbers of molecules; the relative gas density is equal to the ratio of the molecular weights of the two gases.

The two well-known classic methods of determining the density of vapor of a substance which is liquid at normal temperature are those of Durnas and of Victor Meyer.

In Victor Meyer's method the vaporized liquid displaces air from a vessel maintained at a high constant temperature, but the volume of air is measured at ordinary temperature.

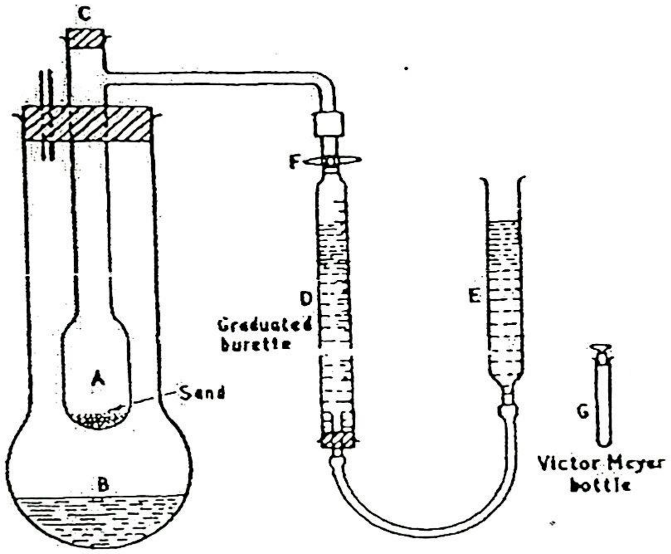


Fig. 8: Victor Meyer's apparatus for determination of vapor densities.

Experiment Method:

1. Take off the stopper (C) as shown in Figure 3B.
2. Boil the water in the outer jacket (B), continue boiling noticing that bubbles are set free at the open end of the side tube (C).
3. Meanwhile, weight the Victor Meyer bottle, first empty, then filled with the given liquid; taking care that no air bubbles should exist after weighting. Let the weight of liquid be $W_{(g)}$.
4. Judging that all excess air in (B) has been expelled, release momentarily stopper (D) and quickly throw Victor Meyer bottle to the bottom of (B). The liquid evaporation, and equal volume of air is displaced up to the graduated jar.
5. When evaporation is complete, both water surface in the two jars are adjusted to the same level. The volume of gas enclosed V is then recorded.
6. The atmospheric pressure P is read on Fortin's barometer, and the vapor pressure of water P° at the working temperature T is found from tables.

Calculation:

$$V_\circ = \frac{P-P^\circ}{760} * \frac{273}{273+tC^\circ} * V_3$$

W_1(g): Weight of Victor Meyer bottle empty.

W_2(g): Weight of Victor Meyer bottle with liquid.

W_2(g)- W_1(g) = W_3(g) Weight of the liquid.

V_1:Initial reading at the graduated bottle.

V_2: Final reading at the graduated bottle.

V_2-V_1=V_3 volume of the displaced air which equal to the volume of liquid vapor.

t C° : Experiment temperature.

P: Atmospheric pressure.

P°: Water vapor pressure at the experimental temperature.

$V_\circ$: The correct volume (at S.T.P).

$W_\circ$:Weight of the same volume of Hydrogen at S.T.P.

$$W_\circ = V_\circ * \frac{0.09g}{1000\ cm^3}$$

$$\text{Relative density} = \frac{Wt\ of\ unknown\ (w_3)}{Wt\ of\ the\ same\ volume\ of\ H_2(w_\circ)}$$

M.wt of unknown = Relative density* M.wt of Hydrogen.

Experiment No. 5:

Part (A):

Refractometry

Determination of refractive index of some alcohols

Introduction:

When a ray of monochromatic light passes from a less dense to a more dense medium, it is bent or refracted towards the normal.

In (fig. 10) if *I* is the less dense and *II* is the more dense medium, a ray of light passing from *I* to *II* will be bent so that the angle of refraction *e* will be less than the angle of incidence i and according to the law of refraction, the relation between these two angles will be such that:

$$\frac{\sin i}{\sin e} = \frac{N}{n}$$

n: Index of refraction of the less dense medium.

N: Index of refraction of the denser medium.

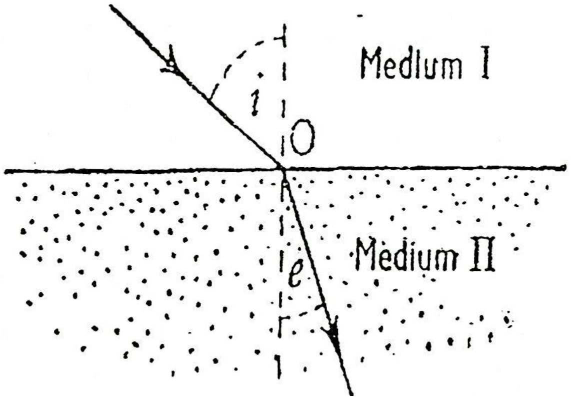


Fig. 10: Refraction and the critical angle principle.

The expression:

$$R = \frac{n^2-1}{n^2+2} * \frac{1}{d}$$

Remains nearly constant at different temperature, whereas the refractive index n of a substance varies with the temperature (d: the density).

The value of *R* dependent only on the nature of the substance and is a characteristic of it, and it is called refractivity of the substance.

If the refractivity is multiplied by the molecular weight of the substance, one obtains the molecular refractivity.

$$[R_D] = \frac{n^2-1}{n^2+2} * \frac{M}{\rho} \text{ ………………..(18)}$$

M:Molecular weight.
R: Have the dimensions of a volume.

Refractometer measurement may be used, very advantageously for the quantitative determination of the composition of binary solutions.

The molecular refractivity is related to the composition of solution according to the following equation:

$$[R_{A,B}] = X_A R_A + X_B R_B$$

$$= X_A R_A + (1-X_n)R_B$$

$$= X_A(R_A - R_B) + R_B \text{ …………………(19)}$$

For solution the molecular refractivity is:

$$[R_{A,B}] = \frac{n_{AB}^2+1}{n_{AB}^2+2} \left[\frac{X_A M_A + X_B M_B}{\rho_{AB}}\right] \text{ ………(20)}$$

R_A: Molecular refractivity of liquid A.
R_B: Molecular refractivity of liquid B.
R_{AB}: Molecular refractivity of liquid solution of (A+B).
X_a, X_b: Mole fractions of *A* and *B* in solution.
$n_{A,B}$: Refractive index of solution of (A+B).
n_A: Refractive index of pure A.
n_B: Refractive index of pure B.

The abbe refractometer is the most commonly used instrument in chemical laboratories. The principle of operation is illustrated diagrammatically. The appearance of the instrument is shown in (fig. 11).

The optical system consist of three parts: a mirror M, a prism-box PQ which can be rotated as a whole by means f a knob- R, and a fixed telescope, T.

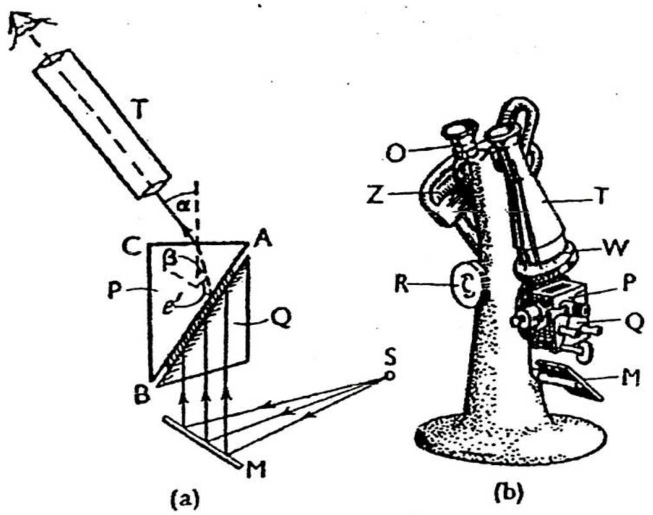


Fig. 11:The Abbe refractometer
(a) Optical principle.
(b) General view of the instrument.

Experiment Method:

1 . Clean the glass surface of refractive index by using alcohol or acetone.
2 . In order to carry out a determination of refractive index of a liquid, open the prism box and place a few drops of the liquid on the ground surface of the lower prism.
3 . Close and fasten- the prism box again, taking care that the liquid dose not flow away. A film of liquid will thus be enclosed between the two prisms.
4 . Focus the cross-wire of the telescope by rotating the eyepiece and adjust so as to got good illumination.
5 . By means of the knob R, turn the prism box slowly backwards and forwards until the field of view becomes partly light and partly dark.
6 . When a white light is used the edge of the light band will show a colored fringe. By the means of the ring W on the telescope, rotate the "compensator" until the fringe disappears and the light band shows a sharp edge.
7 . Now rotate the prism box until this sharp edge is in coincidence with the intersection of the cross-wire in the telescope.
8 . Read off directly the index of refraction on the scale through eyepiece O. The third decimal place can be read directly, and the fourth can be estimated with an accuracy of about ± 0.0002.
9 . Use the density bottle to measure the density of liquid as in experiment No. 1, part (A).
10 . Repeat the above steps by using another alcohol.

Calculation:

1 . Calculate R for each liquid replacing n_D in equation No. 18.
2 . Calculate R for methylene group -CH_2- by subtract value of R_B from value of R_A to get R_{-CH3-}

Experiment No. 5

Part (B):

Determination of molecular refractivity of solutions

Experiment Method:

1 . Propose a series of solution of unknown composition of benzene and carbon tetrachloride according to the following table:

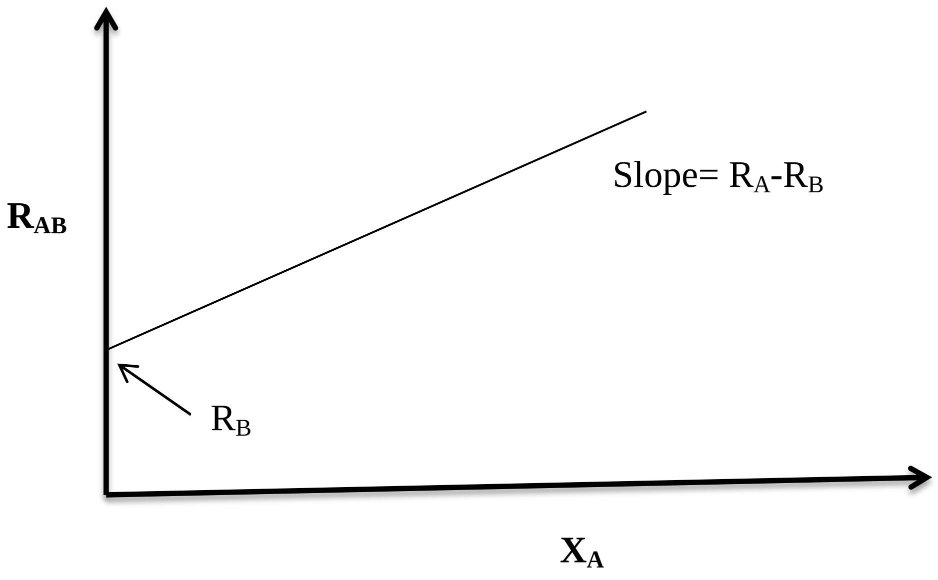
V_A	V_B	W_A	W_B	No. of A moles n_A	No. of B moles n_B	No. of total moles n_{AB}	Molar fraction for X_a	Molar fraction for X_B
1	4							
2	3							
3	2							
4	1							

2 . Read the refractive index of each solution as in part (A) and also determine its density as in experiment no. 1, then arrange your data as the following table:

Mixture AB	Density of mixture ρ_{AB}	η_{AB}	Molecular refractivity of mixture R_{AB}
1			
2			
3			
4			

Calculation:

1. Calculate [$R_{A,B}$] by the use of equation (20).
2. Plot graph between the molecular refractivity of [$R_{A,B}$] as ordinate and the mole fraction X_A as abscissa according to equation (19).
3. From graph calculate the slope and R_B.
4. Compare the result obtained from this plot and the result obtained by the direct calculation of the molecular refractivity of a pure liquid by the use of equation (18).



Experiment No. 6
Part (A)

Thermochemistry

Determination of calorimetric constant

Introduction:

Most chemical process are accompanied by a measurable absorption or evolution of heat. It follow from the first law of thermodynamics that the magnitude of the heat change is proportional to the quantity of substance involved and depends also on the physical state of the reactants and the products, but it is independent on the path by which the reaction is brought about.

The heat change accompanying a physical or chemical process is measured by some form of calorimeter, the measured rise or fall of temperature multiplied by the total heat capacity of the calorimeter gives the quantity of the heat in calories.

$$q = C\Delta T \quad \ldots\ldots\ldots (21)$$

The technique of calorimetry presents two principal difficulties firstly, how to determine the heat capacity of the calorimeter and all its contents, and secondly, how to deal with die inevitable exchange of some heat between die calorimeter and its surroundings.

The calorimeter constant may be measured by carries out a reaction in it and observes the temperature change ΔT for this reaction.

The calorimeter consists of an isolated vessel {thermos} with a compact cover pass through it a thermometer and stirrer.

Exothermic reactions evolve heat and have negative values of ΔH and endothermic reactions absorb heat and have positive values of ΔH.

Experiment Method:

1. Clean Dewor's flask (calorimeter) and put 100 ml of distilled water, stir and record the temperature for 10 minute in a 1 minute interval, be careful that the thermometer bulb immersed in water before starting.
2. Rise up the cover and add quickly 1.5 ml of concentrated sulfuric acid and cover again then record the temperature for 5 minute in a half a minute interval.
3. Plot the temperature as ordinate and the time as abscissa for a graphical correction for the heat exchange in calorimetry (calculate ΔT) as in Fig. 13.

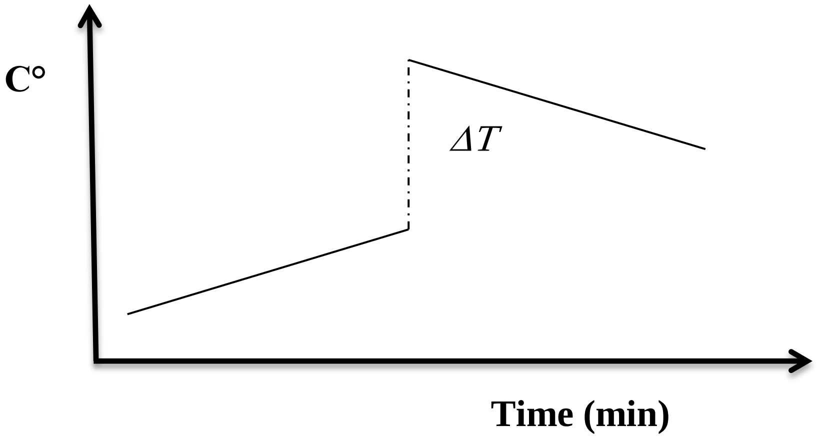


Fig. 13: A graph of the temperature as ordinate and the time as abscissa for a graphical correction for the heat exchange in calorimetry.

4. Draw 10 ml of a solution in the calorimeter, put it in a conical flask, add two drops of phenolphthalein indicator, and titrate with 1N NaOH.
5. To determine ΔH which is equivalent to this normality, plot a graph between ΔH and normality for a data in table. And obtain it from this graph. Fig. 14.

Acid added (ml) D=1.84	Normality of final solution (N)	Heat liberated ΔH (kcal)
3.0	1.100	0.946
2.5	0.918	0.780
2.3	0.842	0.718
1.5	0.552	0.473
0.75	0.227	0.242
0.6	0.217	0.191

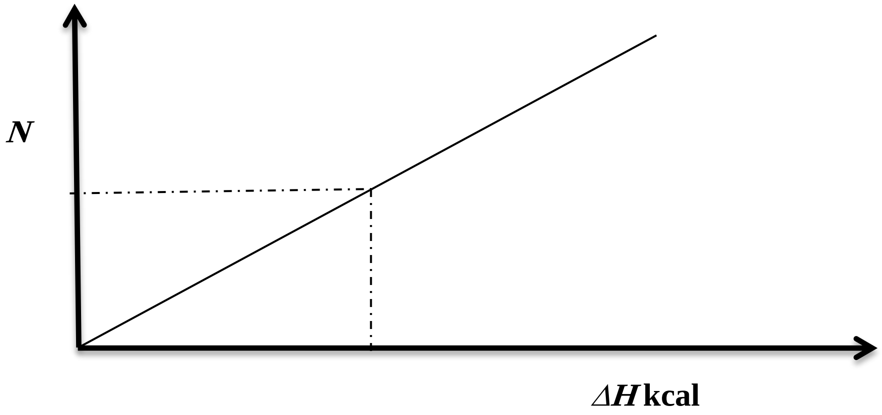


Fig. 14 : A graph between ΔH and normality.

6. Calculate the calorimeter constant from the following equation:

$$C = \Delta H * (V/100) * (1/\Delta T)$$

V: Volume of solution in the calorimeter.
ΔT: The change in temperature.
C: Calorimeter constant.
ΔH: Heat liberated (from calibration curve).

Experiment no. 6

Part (B)

Determination of the heat of solution

Introduction:

The heat of solution of a solid or a liquid substance can be determined in practically the same manner as the employed for the determination of the heat of neutralization and the same apparatus can be employed. Since the heat which is evolved or absorbed on dissolving a substance depends on the amount of water or other solvent employed, the statement of the heat of solution has a definite meaning only when the concentration of the solution formed is given.

Experiment Method:

1. Use the same Dewar's flask as in part A.
2. Place 100 ml distilled water heated to 36-40 C° in a Dewar's flask and record the temperature on the thermometer every minute for 10 minutes interval.
3. Add quickly a weighted quantity of ammonium chloride salt (8g) or any other salt and stir the solution with the continuous recording the temperature every half a minute for another five minutes.

Calculation:

1. Plot a graph between the temperatures as ordinate and the time as abscissa ΔT.

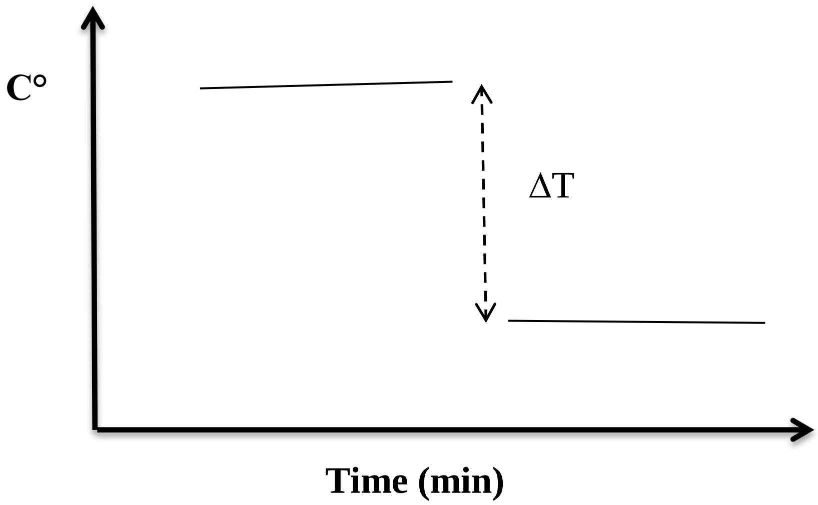


Fig. 14: A graph between the temperature as ordinate and the time as abscissa ΔT.

2. Calculate the heat evolved or absorbed by dissolving this amount of salt using of equation:

$$X = \frac{W_1(T_2 - T_1)C_1}{1000} + C\Delta T$$

W_1: Weight of dissolved salt.
T_1: Laboratory temperature.
T_2: Temperature of the heated water before the addition of the salt.
C_1: Salt heat capacity (0.25).
X: heat evolved or absorbed.
C: Calorimeter constant.
$\Delta T = T_2 - T_1$

3. Calculate the heat of solution that companying dissolving mal as:

$$\Delta H_{solubility} = \frac{X * M.wt_{salt}}{W_1}$$

Experiment No. 7

Equilibrium Constant

Determination the Formula of the Complex Formed Between Copper (II) Ion and Ammonia

Introduction:

The distribution coefficient of ammonia between carbon tetrachloride and water is given by:

$$D = [NH_3]_{org} / \ [NH3]_{aq}$$

Although in the presence of copper sulfate solution some of the ammonia is present as the complex copper ammonium ion, $Cu(NH_3)_n^{++}$, the relationship (1) still applies to the concentration of "free" ammonia. Thus, from a study of the distribution of the ammonia between water and chloroform and an aqueous solution of copper sulfate and chloroform, the formula of the complex ion can be determined, assuming that the equilibrium:

$$Cu^{++} + n\ NH_3 \longleftrightarrow Cu(NH_3)_n^{++}$$

Is completely to the right.

Experiment Method:

1. Prepare 25 ml of 0.1 M hydrochloric acid, then from it prepare 25 ml of 0.01N for titration.
2. Mix (15ml) of carbon tetra chloride CCl_4 with (15ml) of 1M ammonia solution in separating funnel for 20 minute. Leave it for 10 minute to separate into two layers.
3. Drop 5 ml of the organic layer by pipette in conical flask, then add 10 ml distilled water and 8 drops green bromogresol indicator.
4. Titrate against 0.01M HCl, shake well the green yellowish color is appear.
5. Leave the aqueous layer.
6. Mix (25ml) of carbon tetra chloride CCl_4 with (8ml) of 1M ammonia solution and (8ml) of 0.1M copper sulfate in clean and dry separating funnel for 15 minute. Leave it for 10 minute to separate into two layers.
7. Drop 5 ml of the organic layer by pipette in conical flask, then add 10 ml distilled water and 8 drops green bromogresol indicator.
8. Titrate against 0.01M HCl, shake well the green yellowish color is appear.
9. Leave the aqueous layer.

Calculation:

First separation funnel:

1. Calculate the ammonia concentration at organic layer, $[NH_3]_{org}$.
2. Calculate the ammonia concentration at aqueous layer, $[NH_3]_{aq}$ by abstract the ammonia primary concentration from $[NH_3]_{org}$.
3. Calculate the distribution coefficient D from the relationship:

$$D = \frac{[NH_3]_{org}}{[NH_3]_{aq}}$$

Where $[NH_3]_{aq}$ =Ammonia primary concentration-$[NH_3]_{org}$

Second separation funnel:

1. Calculate the ammonia concentration at organic layer, $[NH_3]_{org}$.
2. Calculate the moles number of ammonia at organic layer, X.

$$X = \frac{[NH_3]*V_{organic}(25ml)}{1000}$$

3. Calculate the ammonia concentration $[NH_3]_{aq}$ at aqueous layer, by this equation: $[NH_3]_{aq} = \frac{[NH_3]_{org}}{D}$
4. Calculate the free moles number of ammonia at aqueous layer, Y.

$$Y = \frac{[NH_3]_{aq}*V_{aqueous}(16ml)}{1000}$$

5. Calculate the total moles number of ammonia, Z.

$$Z = \frac{primary\ concentration*V_{mixture}(8ml)}{1000}$$

6. Calculate the associated moles number of ammonia, $\bar{Z}$.

$$\bar{Z} = Z - (X+Y)$$

7. The moles number of copper $=\frac{Conc.primaryCu*V(8ml)}{1000}$
8. $n = \frac{the\ associated\ moles\ number\ of\ ammonia}{the\ moles\ number\ of\ copper}$
9. $K_s = \frac{[\cancel{complex}]}{[\cancel{Cu}][NH_3]^n} = \frac{1}{[NH_3]^n} = \frac{1}{\bar{Z}} = \frac{1}{(\bar{Z}*1000)/16}$

Experiment No. 8:

Properties of dilute solution

Distribution of a solute between immiscible solvents

Introduction:

The reversible reaction:

$$KI + I_2 \longleftrightarrow KI_3$$

Occurs in aqueous solution and according to the law of mass action the equilibrium constant K_C is given by:

$$K_C = \frac{[KI_3]}{[KI][I_2]} \quad \text{.........(27)}$$

The equilibrium may be investigated by studying the distribution of iodine between an organic solvent and water, followed by a similar study for the distribution of iodine between the same organic solvent and an aqueous solution of potassium iodide. The first study enables the distribution coefficient D to be calculated from the relationship:

$$D = \frac{C_{org}}{C_{aq}} \quad \text{..........(28)}$$

Where C is the concentration of the iodine (determined by titration).

Since the distribution law only applied to the species common to both layers, the concentration of free iodine in the aqueous potassium iodide layer $C_{KI,aq}$ can be determined from the relationship:

$$C_{KI,aq} = \frac{C_{org}}{D}$$

The iodine combined with potassium iodide to form may be determined since the total iodine in the aqueous potassium iodide layer may be obtained by titration.

The amount of iodide which has combined with the iodine can then be found and as the original concentration of iodide is known the amount of uncombined iodide may be obtained by difference, hence K_C may be calculated.

Experiment Method:

First separation funnel:

1. 5 ml of saturated solution iodine in carbon tetra chloride is shaken with 50 ml of distilled water in cleaned and dried glass Stoppard for 15 minutes.
2. Leave the solution to separate into two layers at least 15 minutes.
3. Use a pipette to take 20 ml of aqueous layer, then titrate with 0.025 M sodium thiosulphate by using starch solution as an indicator.
4. Use a pipette to take 1 ml of organic layer, add 10 ml of distilled water then titrate with 0.025 M of sodium thiosulphate by using starch solution as an indicator.

Second separation funnel:

1. 5 ml of saturated solution iodine in carbon tetra chloride is shaken with 50 ml of aqueous solution of potassium iodide KI (0.1 M) in cleaned and dried glass Stoppard bottles for 15 minutes.
2. Repeat steps (2, 3, 4) of first funnel.

Calculation:

1. Calculate the distribution coefficient D from the following equation:

$$D = \frac{[I_2]_{org}}{[I_2]_{aq}}$$

2. Calculate equilibrium constant K_C from equation No. (27) by calculate each concentration:

$$\text{uncombined } [I_2]_{aq} = \frac{[I_2]_{org}}{[D]}$$

$$\text{combined } [I_2] = \text{total } [I_2] - \text{uncombined } [I_2]$$

3. Remaining [KI] = total [KI] – combined [KI]

4. $$K_C = \frac{formed[KI_3]_{org}}{remaining[KI] * uncombined[I_2]}$$

Experiment No. 9

Relative molecular Mass

Determine the relative molecular mass of polymer from viscosity measurements

Introduction:

The viscosity of polymer solution is related to the relative molecular mass, M_r of the polymer by equation:

$$\frac{\eta_{sp}}{c} = \frac{\frac{\eta}{\eta_\circ}-1}{c} = KM_r^{\alpha} \quad ....(1)$$

Where η_{sp} is the specific viscosity of the solution, $\eta_\circ$ and η the viscosities of solvent and solution respectively, c/g the weight of polymer in 100 cm^3 of solution and K and α constants. This equation is only valid for low concentrations (<0.5 to 1%) of a linear polymer, dissolved in a solvent in which there is no association. It is thus necessary to extrapolate the curve of C against η_{sp}/c to c=0; the intercept is the intrinsic viscosity$|\ \eta\ |$. Thus, in the limit, equation (1) becomes:

$$|\ \eta\ | = KM_r^{\alpha} \ ....(2)$$

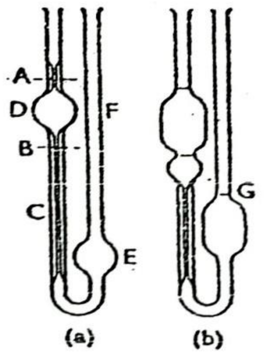
In general, the value of K depends on the type of polymer, the solvent and the temperature, while α is a function of the geometry of the molecule. The viscometric method of determining relative molecular masses is very convenient and gives accurate results K and α are known. These are obtained from samples of polymer of known M_r determined by alternative methods.

To calculate the relative viscosity for the solution, use equation (3).

$$\frac{\eta}{\eta_\circ} = \frac{\rho\, t}{\rho_\circ t_\circ}$$

Experiment Method:

1. Weight the five empty cleaned and dried (25ml) volumetric flask with its cover.
2. Prepare four polymer solutions (2, 4, 6 and 10N).
3. Weight them again after fill one of them with distilled water and the others with polymer solutions above to calculate the density.
4. Cleaned and dried the viscometer then clamped it vertically.
5. A definite volume of the polymer (10 ml) is introduced in the wide tube (limb C) of the viscometer.
6. The liquid in the viscometer is forced up through the capillary tube by suction through a rubber tube attached to the end (a) of the viscometer until the liquid fills the tube (E) and rises slightly above the mark (0). The volume of the liquid introduced must be sufficient to fill the bulb (E), the lower bend (f) and extends up slightly in to wider tube (C), otherwise, air bubbles will form in the capillary and affect the time of flow (t), repeat that twice to get the correct time.
7. Repeat the above steps on freshly distilled water using exactly the same volume (10 ml) to calculate $t_\circ$.



Calculation:

1. Let the time of flow of the polymer be *t* its density *d*, and its viscosity η. The time of flow of water be $t_\circ$ its density $d_\circ$, and its absolute viscosity $\eta_\circ$. Then:

$$\frac{\eta}{\eta_\circ} = \frac{t\,d}{t_\circ d_\circ}$$

Poise= dynes /cm^3/ unit velocity gradient.

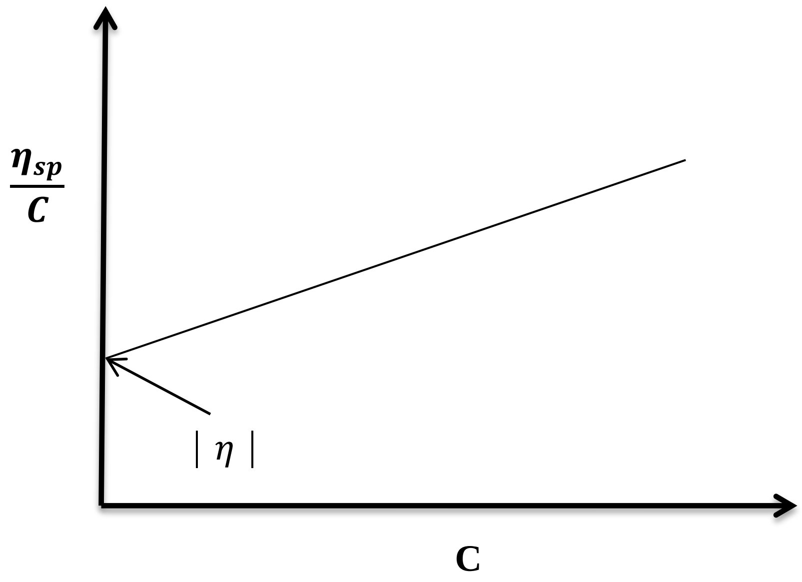
Take the viscosity of water at laboratory temperature from the table.

2. Put the results as in the following tables:

C (N)	D (g/cm^3)	t (sec)	$\frac{\eta}{\eta_\circ}$	$\frac{\eta}{\eta_\circ}-1$ (η_{sp})	$\frac{\eta_{sp}}{C}$

3. Plot a curve between (η_{sp}/c) against C from the equation:

$$\frac{\eta_{sp}}{C} = \frac{\frac{\eta}{\eta_\circ}-1}{C} = KM_r^{\alpha} \quad \ldots\ldots\ldots..(1)$$



4. Find the value $|\ \eta\ |$ from the intercept, then find the relative molecular mass of polymer (M) from the equation (2), $\alpha = 0.76$ and $K = 13*10^{-5}$.

$$|\ \eta\ | = KM_r^{\alpha} \ldots\ldots\ldots\ldots\ldots\ldots(2)$$

Experiment No. 10

A Three Component Liquid System

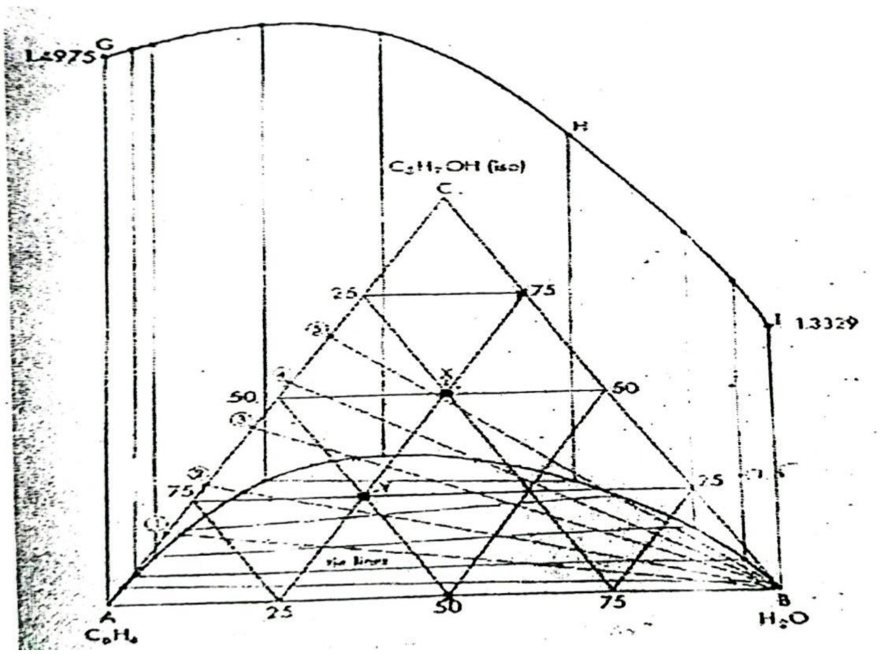
The Triple System

Introduction:

In many cases, the addition of a third component to a pair of almost immiscible liquids dose not appreciably increase their solubility in each other. However, the addition of a third liquid intermediate in polarity may have a large effect, and such a case is illustrated in this experiment.

The solubility relationships of any three substances can be represented with the aid of an equilateral triangle as shown in Fig. 38. To illustrate how such a diagram is used, points X and Y have been chosen. The coordinates of point Y are A= 50, B= 25, C= 25 percent, and chose of X are A= 25, B= 25, C= 50 percent. For any lines, such as those joining points 1-5 and B, the ratio of the quantities A and C are constant while the amount of B varies. Similar relation holds for line drawn from the opposite side to one of the apices.

This principle is applied in constructing the solubility curves. Mixtures of liquids A and C of various proportions are prepared corresponding to the points marked 1, 2, etc. Component B is added until the solubility curve is intersected, whereupon a second conjugate ternary solution appears, its composition being given by the tie line.



Experiment Method:

1. Put in five clean, dry round bottle flask benzene and ethanol as shown in the table to get a clear mixture:

No. of flask	Benzene	Ethyl alcohol
1	8	3.8
2	5.7	6.4
3	3.7	8.9
4	1.2	10.9
5	0.5	12.0

2. Fill the burette with water.
3. Add to each flask the water drop after drop until get turbid mixture, then record the volume of water.

Calculation:

1. The density of Ethanol is 0.789 gm/ml, water is 0.996 gm/ml and benzene is 0.87 gm/ml.
2. Calculate weight percentage for each substance in the mixture, then fill the table:

Flask number	Weight % of benzene	Weight % of ethanol	Weight % of water	Total Weight %
1				
2				
3				
4				
5				

3. Plot the triangular diagram.
4. Find the number of phases on the triangular diagram, above it and under it.