C 141 (Experiment No._________)
NAME : ______________________________ **BATCH : ________**
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DETERMINATION OF CRITICAL SOLUTION TEMPERATURE (CST) FOR PHENOL – WATER SYSTEM AND TO STUDY THE EFFECT OF ADDED IMPURITY (NaCl) ON CST

AIM

To determine the Critical Solution temperature (CST) for the phenol-water system, and to study the effect of added impurity (NaCl) on the CST.

THEORY

The number of homogeneous, mechanically separable and physically distinct parts of a heterogeneous system is known as the number of phases, **P**, of the system.
Each phase is separated from other phases by a physical boundary.
When an equilibrium exists between a number of phases under external controlling conditions such as temperature, pressure, and concentration, the following relationship holds good :

$$P + F = C + 2 \qquad \text{-->} \qquad (1)$$

where **P = number of Phases in equilibrium,**
C = number of Components in the system, and
F = number of degrees of Freedom.

Equation (1) is called the ***Phase Rule***, which relates the *phases, components and degrees of freedom* of the system. The different terms used in Eq. (1) may be defined as follows :

Phase is defined as any homogeneous and physically distinct part of a system which is separated from other parts of the system by definite bounding surfaces.
The number of **Components** in a system in equilibrium is the smallest number of independently variable constituents by means of which the composition of each phase present can be expressed either directly, or in the form of a chemical equation.
The number of **degrees of freedom** or **variance** is the number of variable factors, such as temperature, pressure and concentration, which need to be fixed in order to completely define the conditions of a system in equilibrium.

When 2 partially miscible liquids are mixed and shaken together, we get 2 solutions of different compositions. For example, on shaking phenol and water, we get 2 layers : **the upper layer** is a solution of **water in phenol**, and the **lower layer** is a solution of **phenol in water**. At a fixed temperature, the composition of each solution is fixed, and both the solutions are in equilibrium.

Two solutions of different compositions existing in equilibrium with one another are known as ***conjugate solutions***. **Above a particular temperature, such solutions are completely miscible in all proportions. Such a temperature is known as the Critical Solution Temperature (CST)** or **Consolute Temperature**. As the mutual solubility increases with temperature in this particular case, it is known as **Upper Consolute Temperature**.

Mutual Solubility Curve

If we have 2 liquids **A** and **B** and we mix them, we get a mixture of composition $\mathbf{c_1}$. At any temperature $\mathbf{t_1{}^oC}$ (or below $t_1{}^oC$), the 2 liquids separate into 2 layers of different compositions.
Above $t_1{}^oC$, the 2 layers are completely miscible. Thus, the point corresponding to temperature $t_1{}^oC$ and composition c_1 is known as the **miscibility point**.

If we take another mixture of **A** and **B** of composition c_2, we can find out the temperature (say $\mathbf{t_2{}^oC}$) above which the last 2 layers become completely miscible. Similarly, we can find out corresponding temperatures for a number of mixtures of **A** and **B**.
If a curve is plotted with **temperature** (^oC) as ordinate (***y-axis***) against **concentration** (% by weight) as abscissa (***x-axis***), we get a **mutual solubility curve**.

It is found that above B, the 2 liquids will become miscible in all proportions, and hence it is known as the ***Critical Solution Temperature (CST)***.

Above this temperature, the liquid mixture is homogeneous.

Below this temperature, the mixture separates into 2 layers.

The **CST** is markedly affected by pressure and also by the presence of impurities. Hence, the **CST** may be taken as a criterion for the purity of a substance.

The solubility changes in the CST for Phenol - Water system is affected by adding an electrolyte to it on miscibility temperature.

If a graph is plotted with mean miscible temperature as ordinate (y-axis) against concentration of electrolyte (N) as ordinate (x-axis), we get the unknown concentration of electrolyte. From that, we can estimate the amount of solution supplied.

MATERIALS REQUIRED

Hard Glass Test tubes (or boiling tube as air jacket), thermometer (graduated to 0.1^{o}C), stirrer, beakers (500ml, 100 ml), volumetric flask (100 ml), pipette (1 ml, 2 ml), phenol, distilled water, sodium chloride, hot plate.

PROCEDURE

PART – A: <u>CST for Phenol - Water system</u>

1. Weigh out about 5.0 g of phenol in a dry boiling tube.
2. Add **2.0 ml of distilled water**. The solution is stirred.
3. Heat the solution in a water bath, with continuous stirring.http://start.fedoraproject.org/
4. At a certain temperature, the mixture becomes **clear**. Note this temperature (**t_1^{o}C**).
5. Remove the tube from the water bath, and allow the solution to cool down slowly. Note the temperature at which the ***turbidity*** **re-appears** (**t_2^{o}C**).
6. **Repeat Steps 2 to 6, *after each addition of 2 ml of solution, followed by heating and subsequent cooling, note the temperature of disappearance of turbidity, and the temperature of the re-appearance of turbidity. The observation is that the temperature (^{o}C) of complete miscibility rises, reaches a maximum value, and then decreases.***

PART – B: <u>CST for Phenol – NaCl system</u>

1. Weigh out about 5.0 g of phenol in a dry boiling tube.
2. Add **2.0 ml of 0.1 N NaCl** solution. The solution is stirred.
3. Repeat the steps 3 to 8 and continue the experiment by following the part – A.

PART – C: <u>Effect of Impurity (NaCl) on CST</u>

1. Following proportions of solutions are to be prepared given in the table 2.
2. The clearing and clouding temperature for the prepared solutions are noted and their mean value is taken.
3. From the mean miscibility temperature of unknown solution, the concentration (N) corresponding to them are obtained from the given values of NaCl solution and the volume of electrolyte solution supplied is calculated.

OBSERVATIONS

Room Temperature = ………. ^{o}C

$$\textbf{\% of Phenol} = \frac{\textbf{(Volume of phenol x Density of phenol)}}{\textbf{(Volume of phenol x Density of phenol) + (Volume of } H_2O \textbf{ or NaCl)}}$$

Weight of Phenol = ...

Density of Phenol = _____________ g/ml

Density of water (d) = ___________ g/ml

Density of NaCl Solution (0.1N) = ___________ g/ml

Table 1: Determination of CST for Phenol – Water system and Phenol – NaCl system.

Volume of solution added (ml) H_2O / NaCl	wt. of solution (g) (w = v * d)		% of Phenol		% wt. Of phenol [5/(5+w)]*100		Miscibility temperature (0C) H_2O			0.1 N NaCl		
	H_2O	NaCl	H_2O	NaCl	H_2O	NaCl	t_1	t_2	Mean	t_1	t_2	Mean

Table 2: Effect of Impurity (NaCl) on CST (Phenol + Water + NaCl system)

S.No.	Volume of Phenol (ml)	Volume of 0.1 N NaCl taken (ml)	Volume of H_2O (ml)	Clearing temperatue (^0C)	Clouding temperature (^0C)	Mean Temp. (^0C)	Conc. Of NaCl (N)
1.	5.0	1.0	4.0				
2.	5.0	2.0	3.0				
3.	5.0	3.0	2.0				
4.	5.0	4.0	1.0				
5.	5.0	x_1	y_1				
6.	5.0	x_2	y_2				

CALCULATIONS

1. A curve is plotted with ***miscibility temperature (^0C)*** as ordinate against ***concentration of phenol (percentage by weight)*** as abscissa.
2. This curve will be the ***mutual solubility curve*** of the ***phenol – water system***.
3. The maximum of the solubility curve will give the value of the **Critical Solution Temperature**.
4. A graph is plotted with **mean miscibility temperature (^0C)** as **ordinate** against **concentration of NaCl (N)** as **abscissa.**
5. This graph gives the unknown concentration of electrolyte solution supplied.

NOTE :

(i) The temperature of the solution should be increased or decreased ***very slowly***.
(ii) The mixture of phenol and water should be ***continuously and uniformly stirred***.
(iii) The stirrer should not touch the bottom of the boiling tube.
(iv) Care should be taken while handling phenol.

RESULTS

1. (i) The Critical Solution Temperature for the Phenol – Water system	= **…………. ^{o}C**
(ii) % of phenol at CST	= **…………. %**
2. (i) In the presence of NaCl, the CST for the Phenol - Water system	= **…………. ^{o}C**
(ii) % of phenol at CST	= **…………. %**
3. Volume of electrolyte for unknown solutions	= **............... ml**
4. Concentration of the unknown solutions	= **............... N**

5. Comment on the difference in the 2 values of the CST (if any) and nature of the graph(s).