Solubility diagram of two partially miscible liquids

(Item No.: P3030501)

Curricular Relevance

Area of Expertise: Chemistry

Education Level: University

Topic: Physical Chemistry

Subtopic: Phase Equilibrium

Experiment: Solubility diagram of two partially miscible liquids

Difficulty	Preparation Time	Execution Time	Recommended Group Size
Intermediate	10 Minutes	10 Minutes	2 Students

Additional Requirements:

- Precision balance, 620 g / 0.001 g

Experiment Variations:

Keywords:

binary system, miscibility gap, mixed phase, coexisting phase, Raoult’s law, critical dissolution temperature

Overview

Short description

Principle

A number of different mixtures of phenol and water are prepared and heated until complete miscibility is achieved. As the mixtures cool, two-phase systems form at certain temperatures which are recognisable by the appearance of turbidity. Plotting separation temperatures against compositions of the mixtures gives the separation curve.

Fig. 1: Experimental set-up

Safety instructions

Phenol, loose crystals
H301: Toxic if swallowed
H314: Causes severe skin burns and eye damage
H373: May cause damage to organs through prolonged or repeated exposure
P280: Wear protective gloves/protective clothing/eye protection/face protection.

Equipment

Position No.	Material	Order No.	Quantity
1	Immersion thermostat Alpha A, 230 V	08493-93	1
2	External circulation set for thermostat Alpha A	08493-02	1
3	Bath for thermostat, makrolon	08487-02	1
4	Rack for 20 test tubes, Makrolon	08487-03	1
5	Rubber tubing, i.d. 6 mm	39282-00	3
6	Hose clip, diam. 8-16 mm, 1 pc.	40996-02	4
7	Burette, 10 ml, grad. 0.05 ml	47152-01	1
8	Burette clamp, roller mount., 2 pl.	37720-00	1
9	Retort stand, h = 750 mm	37694-00	1
10	Funnel, glass, top dia. 55 mm	34457-00	1
11	Test tube, 160 x 16 mm, 100 pcs	37656-10	1
12	Rubber stopper,d=18/14mm,w/o hole	39254-00	7
13	Beaker, high, BORO 3.3, 150 ml	46032-00	1
14	Wash bottle, plastic, 500 ml	33931-00	1
15	Spoon, special steel	33398-00	1
16	Pasteur pipettes, 250 pcs	36590-00	1
17	Rubber caps, 10 pcs	39275-03	1
18	Laboratory pen, waterproof, black	38711-00	1
19	Phenol, loose crystals 100 g	30185-10	1
20	Water, distilled 5 l	31246-81	1
21	Tubing connector, ID 6-10mm	47516-01	2

Tasks

1. Plot the separation curve of the phenol / water binary system and prepare a temperature / mass fraction diagram.
2. Determine the critical separation point.

Set-up and Procedure

Set up the experiment as shown in Fig. 1.

Prepare the phenol / water mixtures listed in Table 1 in seven test tubes.

Table 1: Weights and mixing ratios of the samples

Weigh the respective phenol portions into appropriately numbered test tubes (use a beaker to support the test tubes) and use the microburette to add the required quantity of water. Seal the test tubes with rubber stoppers and heat them in a temperaturecontrolled bath to 75 °C. During heating remove the rubber stoppers from time to time to release excess pressure and shake the mixtures. When clear solutions have formed in all test tubes, switch off the thermostat heating and start the cooling function. Record the temperatures at which the turbidity caused by separation becomes visible. Plot the separation temperatures against the composition of the mixtures as weight percentage w / % (see Fig. 2).

Mixture	m_{phenol}/g	m_{water}/g	$w_{phenol}/\%$	x_{phenol}
1	1.00	9.00	10	0.021
2	2.00	8.00	20	0.046
3	3.00	7.00	30	0.076
4	4.00	6.00	40	0.113
5	5.00	5.00	50	0.161
6	6.00	4.00	60	0.223
7	7.00	3.00	70	0.309

Theory and Evaluation

A mixture of liquids is a homogeneous distribution of two or more substances, whereby all components have a definite vapour pressure. Two liquids may either be completely miscible or only partly miscible. When the van der Waal forces between the two components are smaller than those between molecules of the same type, then an increase in the vapour pressure results. The molecules can leave their arrangement more easily than with equally large attractive forces. With sufficiently high deviation from Raoult's law:

$$P_A = P_A^* \cdot x_A \quad (1)$$

where

P_A^* Vapour pressure of pure substance A

P_A Partial vapour pressure of substance A in solution

x_A Molar fraction of substance A

the components of a binary system no longer continuously mix, but instead have the tendency to again unmix. A miscibility gap can be observed. This is a range of concentrations in which the two liquids form two phases. The molar mixing enthalpy is positive. Unmixing means in this case the transition to a lower energy condition.

Systems with limited miscibility can be presented as isobars in temperature / mass content and temperature / quantity diagrams. In these separation curves, the compositions of the two coexisting liquid phases, which form from the homogeneous mixture when a certain temperature has been reached, are plotted as functions of temperature. The coexisting liquid phases are described as conjugated solutions. They are saturated solutions of the one component in the other. The line connecting the coexisting liquids is designated as the tie line. Normally the mutual solubility of liquid components increases with increasing temperature. The coexisting solutions are identical at a critical dissolving temperature. Above the critical dissolving temperature the components are miscible with one another in any ratio. The compositions of the coexisting solutions at certain temperature are constant and independent of the mass ratios or the two components.

In the case of a mixture of phenol and water at room temperature, up to 28% of water dissolves in phenol, and up to 8% of phenol in water. These values increase with increasing temperature until, at 68.8°C (the critical solution temperature) complete miscibility is reached (Fig. 2). The left side of the curve shows solutions of phenol in water, and the right side of water in phenol. Unmixing occurs within the area of the miscibility gap under the formation of two phases, the compositions of which correspond to the abscissa values at the temperature concerned. If we mix phenol and water at 50°C in a ratio of 1:1 (point c) then unmixing occurs. A 12% solution of phenol in water, and a 36% solution of water in phenol, are formed.

If the mass content$w$ is used as the concentration variable the mass ratio of the two liquid phases can be determined using the so-called rationality law. This states that the masses of phases a and b are inversely proportional to the distance of their composition from the composition of the original mixture c, from which it follows that:

$\frac{w_b}{w_a} = \frac{ac}{bc}$ (2)

The relationship is shown in Fig. 2.

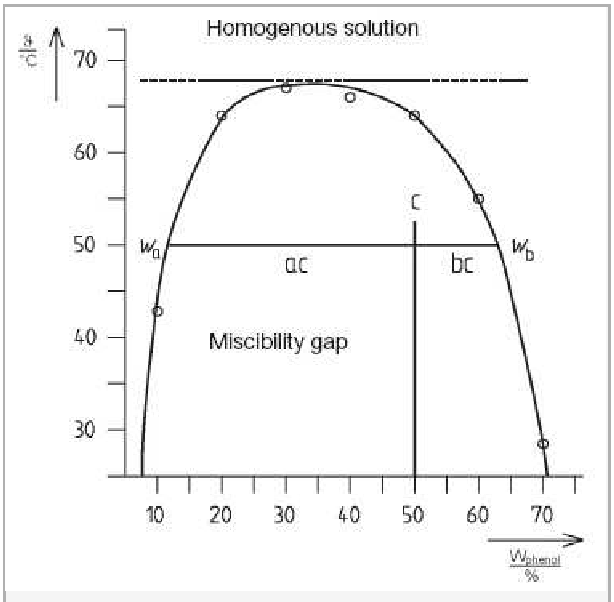

Fig. 2: Solubility diagram of the phenol/water system