Physical Pharmacy Laboratory
2nd stage
College of Pharmacy

Lab.4 : phase behavior and mutual miscibility of the two-component phenol–water

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Aim of the experiment

1. To investigate the mutual solubility of phenol and water at different temperatures.
2. To distinguish between **one-phase and two-phase** liquid systems.
3. To determine the effect of **temperature** and composition on the miscibility of phenol and water.
4. To construct a temperature–composition phase diagram and estimate the upper criticalsolution temperature (UCST).

Principle and theoretical background

- **A two-component** liquid system contains two substances, in this case phenol and water.Depending on the composition and temperature, the mixture may form either:
  - **One liquid phase:** phenol and water are completely miscible at that composition and temperature.
  - **Two liquid phases:** the mixture separates into a phenol-rich phase and a water-rich phase.

- Phenol and water exhibit **partial miscibility at lower temperatures**.
- As the temperature **increases, their mutual miscibility increases.**
- **Above the upper critical solution temperature**, approximately , **they become completely miscible in all proportions under ordinary laboratory conditions.**
- The critical temperature is the temperature above which the two liquid phases cease to exist as separate equilibrium phases.

Experimental procedure

1. Prepare 10 g of each phenol–water mixture according to the following table.
2. Place the test tubes in a **water bath** maintained at **30 C** for approximately 10 minutes.
3. Keep the tubes securely closed during equilibration, using appropriate laboratory safety procedures.
4. Gently shake each mixture to promote equilibration, then allow it to settle.
5. **Record whether each mixture forms one clear liquid phase or two separate liquid phases.**
6. Repeat the observations at **50 C ,55C , and 66.8 C**.
7. **Plot phenol concentration against temperature to construct the phase diagram.**

Experimental data and preparation of mixtures

- The following quantities are specified in your laboratory handout

Test tube	Phenol (% w/w)	Phenol (g)	Water (g)
1	2	0.2	9.8
2	7	0.7	9.3
3	11	1.1	8.9
4	13	1.3	8.7
5	34	3.4	6.6
6	60	6.0	4.0
7	63	6.3	3.7
8	70	7.0	3.0
Total	—	**Varies by mixture**	**10 g per tube**

Safety: Phenol is toxic and corrosive and can be absorbed through the skin. Work in a fume hood with suitable chemical-resistant gloves, eye protection, and a lab coat.

Do not heat tightly sealed test tubes; use an approved procedure that prevents pressure build up and phenol exposure.

The general calculation is:

$$\text{Phenol mass} = \frac{\%\ \text{phenol}}{100} \times 10$$

$$\text{Water mass} = 10 - \text{phenol mass}$$

For example, for a 13% w/w phenol mixture:

$$\text{Phenol} = \frac{13}{100} \times 10 = 1.3\ \text{g}$$

$$\text{Water} = 10 - 1.3 = 8.7\ \text{g}$$

results and observations

- approximately 7% and 70% phenol at 30 C, 11% and 63% at 50 C , and 13% and 60% at 55 C, with a critical point near 34% phenol and 66.8 C
- Using those values as approximate boundaries, the following is the observation table

Temperature	2%	7%	11%	13%	34%	60%	63%	70%
30°C	One	Boundary	Two	Two	Two	Two	Two	Boundary
50°C	One	One	Boundary	Two	Two	Two	Boundary	One
55°C	One	One	One	Boundary	Two	Boundary	One	One
65.8°C	One	One	One	One	Critical region	One	One	One

One = one liquid phase; Two = two liquid phases; Boundary = approximate phase-separation limit.

Construction of the phase diagram

- Plot the results on **rectangular coordinate paper**
- **X-axis:** Phenol concentration (% w/w), from 0 to 100%.
- **Y-axis:** Temperature (°C), from 30 to 70°C.
- Plot the approximate phase-boundary points given below.
- Connect the points with a smooth curve to obtain the miscibility-gap curve.

Temperature (°C)	Lower boundary (% phenol)	Upper boundary (% phenol)
30	7	70
50	11	63
55	13	60
65.8	Approximately 34	Approximately 34