**EXERCISE 5**

**PHASE RULE**

**Theoretical topics**

law of mass action, extraction process, partition coefficient, extraction coefficient, phase rule, Nernst distribution law, independent component, logP, degrees of freedom

**Experimental Part**

**Topic:**  
Partition coefficient.

**Objective:**  
Determining the partition coefficient of acetic acid between water and an organic solvent.

**Apparatus:**  
250 cm³ bottles, beakers, 50 cm³ burette, 25 cm³ pipettes, 100 cm³ separators, 100 cm³ volumetric flasks, conical flasks

**Reagents:**  
phenolphthalein, 2M CH3COOH, 0.2M NaOH, hexane or toluene (solvent no. 1), butanol (solvent no. 2)

**Procedure:**

1. Prepare 100 cm³ of acetic acid solutions at the concentrations indicated in Table A. To do this, measure the appropriate amounts of 2M acetic acid solution into 100 cm³ volumetric flasks using a pipette, top up with water to the mark, and mix.
2. In 250 cm³ glass vessels, place 25 cm³ of the prepared acid solutions. Add 1M acid to vessels 1 and 2, 0.75M acid to vessels 3 and 4, and 0.5M acid to vessels 5 and 6.
3. Add 25 cm³ of organic solvent no. 1 to vessels 1, 3, and 5, and 25 cm³ of solvent no. 2 to vessels 2, 4, and 6. Organic solvents should be added under a fume hood using appropriate pipettes.
4. Seal the vessels tightly, place them on a shaker, and shake for 30 minutes (speed 5).
5. During shaking, determine the exact concentration of the acid in the remaining portion of the prepared solutions (from step 1) by titrating samples with NaOH. Perform titration twice for each of the three prepared acids. For each titration, pipette 2 cm³ of the starting acid solution into two conical flasks, add 3 drops of phenolphthalein, and titrate with 0.2M NaOH solution. Record the results in the table.
6. After shaking, determine the acetic acid content in the aqueous phase of the water-organic mixtures. Transfer the contents of the vessels to separators and separate the aqueous layer from the organic. In the separated aqueous phase, determine the concentration of acetic acid by taking 2 cm³ of the solution into a conical flask, adding 3 drops of phenolphthalein, and titrating with 0.2M NaOH, as done previously. Perform two repetitions for each of the six post-extraction aqueous solutions. Record the results in the table.

**Table A.** Volumes of 2M acetic acid needed to prepare 100 cm³ solutions of specified concentrations.

|  |  |  |  |
| --- | --- | --- | --- |
|  | Solution 1 | Solution 2 | Solution 3 |
| Concentration [mol/dm3] | 1.0 | 0.75 | 0.5 |
| Volume to be taken [cm3] | 50 | 37.5 | 25 |

**Report:**

1. Present the results in Table 1.
2. Calculate the following:
   * Initial concentration of acetic acid in the prepared solutions (*cp*)
   * Acetic acid concentration in the aqueous phase after shaking with the organic solvent (*cw*)
   * Acetic acid concentration in the organic layer (*cp - cw*)
   * Percentage of acetic acid extracted into the organic phase
   * Partition coefficient *K* as the ratio of concentration in the aqueous phase to the concentration in the organic phase

Summarize the calculation results in Table 2.

1. Provide conclusions for the experiment.