# Solubility of Salicylic Acid in Water, Ethanol, Carbon Tetrachloride, Ethyl Acetate, and Xylene

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The solubility of salicylic acid in water, ethanol, carbon tetrachloride, ethyl acetate, and xylene was measured by a gravimetrical method from (298 to 348) K, and the solubility data were correlated against temperature. The experimental solubility of salicylic acid in water and ethyl acetate was compared with the literature data. The solubility of salicylic acid in ethanol and ethyl acetate was high compared with other solvents.

## Introduction

Salicylic acid is widely used in the synthesis and production of aspirin and as an intermediate for production of many industrial compounds.[1]

In industry, salicylic acid is crystallized from solution as a final step. So, crystallization is a key step in many respects, and it determines the yield and quality of the target product. To determine proper solvents and to design an optimized production process, it is necessary to know the solubility of salicylic acid in different solvents. The solubility of salicylic acid in various solvents has been reported.[1–4]

In this work, the solubility of salicylic acid in water, ethanol, carbon tetrachloride, ethyl acetate, and xylene was measured by a gravimetrical method from (298 to 348) K.

## Experimental Section

***Chemicals.*** Salicylic acid was produced in our department and had a purity of 0.997 mass fraction determined by HPLC. It was dried in a vacuum at 50 °C for 8 h and stored in desiccators. Other reagents used such as ethanol, carbon tetrachloride, ethyl acetate, and xylene were of analytical purity grade (Merck, Darmstadt, Germany). Double-distilled–deionized water was used. All the chemicals were used without further purification.

***Apparatus and Procedures.*** The experiments were carried out in a magnetically stirred, jacketed equilibrium cell with a volume of 100 mL as described in the literature.[5–7] The equilibrium cell was sealed by a rubber plug to prevent evaporation of the solvent. The temperature of the equilibrium cell was controlled by circulating water from a thermostat within ± 0.05 K. The sample mass was determined by an electronic balance (2842, Sartorius GMBH, Germany) with an uncertainty of ± 0.1 mg.

***Solubility Measurements.*** The solubilities of salicylic acid in water, ethanol, carbon tetrachloride, ethyl acetate, and xylene were measured by the gravimetric method.[8] To find the suitable time for the equilibrium, the test experiments were carried out over 8 h, 6 h, 4 h, 2 h, 1 h, and 0.5 h, respectively. The solubility data that were measured over 8 h, 6 h, 4 h, and 2 h showed a better agreement, compared with those obtained over 1 h and 0.5 h. So the equilibrium time of 2 h was chosen. For each measurement, an excess amount of salicylic acid was added to a known mass of solvent. Then, the equilibrium cell was heated to the required temperature with continuous stirring. After 2 h, the stirring was stopped, and the solution was kept still for 2 h. Then, the excess solid could be observed in the lower part of the equilibrium cell. The sample of the upper part of the solution was withdrawn with a suitable warmed pipet to another weighed vial. The vial was closed tightly and weighed to determine the mass of the sample. Then, the vial was placed in an oven to evaporate the solvent. After the evaporation of the solvent, the vial was dried for another 5 h and reweighed to determine the mass of the solid. Thus, the solid concentration of the sample could be determined. An average value was taken from three measurements for each temperature.

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## Results and Discussion

The mass fraction solubilities $w$ of salicylic acid in water, ethanol, carbon tetrachloride, ethyl acetate, and xylene were measured from (298 to 348) K and are summarized in Table 1.

As shown in Figure 1, the mass fraction solubility $w$ of salicylic acid was correlated as a function of temperature as follows

$$\ln w = A + B\,(T/\mathrm{K}) \tag{1}$$

The parameters $A$ and $B$ for the solvents and root-mean-square deviations are listed in Table 2. The root-mean-square deviation ($\sigma$) is defined as

$$\sigma = \left[\frac{\sum_{i=1}^{n}(w_{ci} - w_i)^2}{n-1}\right]^{\frac{1}{2}} \tag{2}$$

where, $w_{ci}$ and $w_i$ are calculated and experimental mass fraction solubility, respectively, and $n$ is the number of experimental points. The root-mean-square deviations of calculated solubility with respect to experimental solubility are reported in Table 2.

Within the temperature range of the measurements, solubility of salicylic acid in the solvents increased with an increase in temperature. The experimental solubility of salicylic acid in water and ethyl acetate was compared with the literature data shown in Figure 2. As can be seen, there is a satisfactory agreement between our solubilities of salicylic acid in water and those reported in the literature.[1–3] But the results reported by Nordström and Rasmuson[4] are considerably lower than our

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**Table 1. Mass Fraction Solubility of Salicylic Acid in Five Solvents between (298 and 348) K**

| $T$/K | $10^3$ $w$ | $T$/K | $10^3$ $w$ |
|---|---|---|---|
| | Water | | |
| 298 | 2.20 | 328 | 7.44 |
| 303 | 2.92 | 333 | 9.55 |
| 308 | 3.50 | 338 | 11.64 |
| 313 | 4.11 | 343 | 13.92 |
| 318 | 5.38 | 348 | 17.10 |
| 323 | 6.27 | | |
| | Ethanol | | |
| 298 | 325.38 | 328 | 477.86 |
| 303 | 356.96 | 333 | 499.58 |
| 308 | 385.13 | 338 | 525.19 |
| 313 | 405.53 | 343 | 543.40 |
| 318 | 427.40 | 348 | 569.77 |
| 323 | 448.90 | | |
| | Carbon Tetrachloride | | |
| 298 | 2.99 | 328 | 12.55 |
| 303 | 3.77 | 333 | 15.12 |
| 308 | 4.89 | 338 | 18.01 |
| 313 | 6.31 | 343 | 21.27 |
| 318 | 7.69 | 348 | 25.02 |
| 323 | 10.19 | | |
| | Ethyl Acetate | | |
| 298 | 200.76 | 328 | 301.02 |
| 303 | 217.88 | 333 | 318.02 |
| 308 | 235.82 | 338 | 334.76 |
| 313 | 252.46 | 343 | 351.51 |
| 318 | 269.24 | 348 | 369.21 |
| 323 | 285.96 | | |
| | Xylene | | |
| 298 | 6.16 | 328 | 27.04 |
| 303 | 8.43 | 333 | 33.54 |
| 308 | 11.01 | 338 | 40.13 |
| 313 | 13.82 | 343 | 48.35 |
| 318 | 17.51 | 348 | 57.82 |
| 323 | 22.16 | | |

values. It is worthwhile to note that applied equipment and procedures were different in both works, although there is a satisfactory agreement between our solubilities of salicylic acid in ethyl acetate and those determined by Nordström and Rasmuson.

The solubility of salicylic acid decreases in the order of ethanol, ethyl acetate, carbon tetrachloride, xylene, and water.

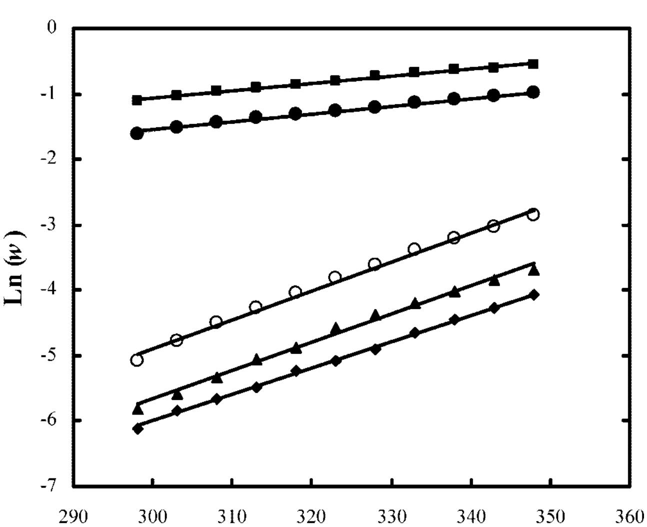


**Figure 1.** Solubility $w$ of salicylic acid as a function of temperature in: ♦, water; ■, ethanol; ▲, carbon tetrachloride; •, ethyl acetate; ○, xylene. Solid lines are values from eq 1 with coefficients from Table 2.

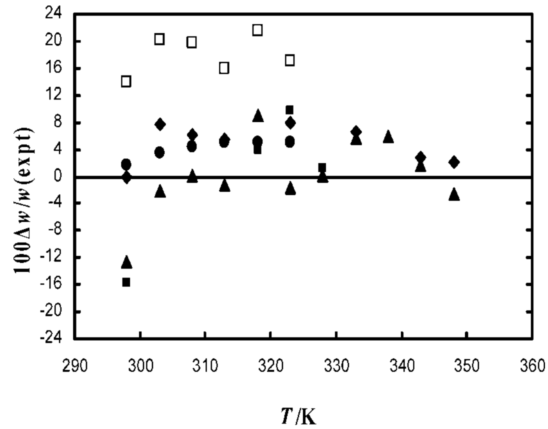


**Figure 2.** Fractional deviations $\Delta w = w(\text{exptl}) - w(\text{ref})$ of salicylic acid solubility at various temperatures obtained in this work, $w$(exptl), from literature values, $w$(ref). ♦, ref 1, water; □, ref 2, water; ▲, ref 3, water; ■, ref 4, water; •, ref 2, ethyl acetate.

**Table 2. *A* and *B* Values and the Root-Mean-Square Deviations (σ) of the Measured Solubility from the Calculated Results**

| solvent | $A$ | $B$ | $10^3$ σ |
|---|---|---|---|
| water | −18.06 | 0.04 | 0.10 |
| ethanol | −4.30 | 0.01 | 8.20 |
| carbon tetrachloride | −18.59 | 0.04 | 1.00 |
| ethyl acetate | −5.14 | 0.02 | 6.0 |
| xylene | −18.13 | 0.04 | 1.70 |

The low cohesive energy of the solvent may relate to high solubility in ethanol and ethyl acetate. In carbon tetrachloride and xylene, the affinity for salicylic acid is reduced. Salicylic acid exhibits a very low solubility in water as compared with the organic solvents. The cohesive energy of water itself is high as substantiated through the high boiling point and density.[4]

Although water is often used to recrystallize salicylic acid from solutions, the solubility of salicylic acid in ethanol was higher than in water. As listed in Table 1, the solubility of salicylic acid in ethanol showed a higher value than those in the other solvents. Thus, ethanol may be a better solvent to separate and purify salicylic acid from solutions.

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