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SYNTHESIS AND SOLUBILITY OF 1-[2-METHYL-1-(4-METHYLPHENYL)-5-PHENYL-1*H*-PYRROL-3-YL]ETHANONE IN ORGANIC SOLVENTS

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The temperature dependence of the solubility of 1-[2-methyl-1-(4-methylphenyl)-5-phenyl-1H-pyrrol-3-yl]ethanone in methyl acetate, ethyl acetate, acetonitrile, propan-1-ol and propan-2-ol has been investigated. The enthalpy and entropy of dissolution were calculated. The thermodynamic parameters of the melting process and the melting point were determined based on the results of differential thermal analysis. The enthalpy and entropy of melting were recalculated to 298.15 K. The enthalpy and entropy of mixing 1-[2-methyl-1-(4-methylphenyl)-5-phenyl-1H-pyrrol-3-yl]ethanone with methyl acetate, ethyl acetate, acetonitrile, propan-1-ol, and propan-2-ol were calculated.

Keywords: solubility, enthalpy of dissolution, enthalpy of mixing, enthalpy of melting, enthalpy of evaporation, polysubstituted pyrrole derivatives.

Introduction

It is a well-known fact that heterocyclic nitrogen-containing compounds have biological activity. Pyrrolic structural fragments are present in molecules C and B hemes, which is a part of hemoglobin; cytochrome – a protein molecule; chlorophyll – a natural green pigment [1]. Pyrroles, as nitrogen-containing five-membered heterocyclic compounds, are used as components of pharmaceutical agents with antibacterial [2] and anti-inflammatory [3] properties. Pyrrole derivatives are also employed in the synthesis of semiconductors, catalysts, and accumulators [4], for the production of cleaner energy.

Such a wide range of applications encourages pharmacists and organic chemists to synthesize new compounds with specific properties. It is known that processes of synthesis, purification, and processing of substances are closely related to solvents. The solvent is most often present during the synthesis of compounds, where it is primarily used as the reaction medium. The solvent is also an essential component in the purification of newly synthesized substances by recrystallization [5].

Therefore, studying the interactions that occur between the dissolved substance and the solvent, considering the thermodynamic processes, is a relevant task for research.

Purpose of the study to determine the thermodynamic parameters of the interaction of 1-[2-methyl-1-(4-methylphenyl)-5-phenyl-1H-pyrrol-3-yl]ethanone with methyl acetate, ethyl acetate, acetonitrile, propan-1-ol, and propan-2-ol.

Materials and research methods

The IR spectrum of the synthesized compound was obtained using the Shimadzu IRSpirit-T instrument in the range from $400 \text{ to } 4000 \text{ cm}^{-1}$.

The synthesis of 1-[2-methyl-1-(4-methylphenyl)-5-phenyl-1H-pyrrol-3-yl]ethanone was carried out according to the scheme shown in Fig. 1 in two stages.

In the first stage, a suspension of sodium acetylacetonate 1 (5.4 g, 0.044 mol) in 30 mL of ethanol was cooled, followed by the addition of phenacyl bromide 2 (6 g., 0.03 mol). The mixture was then stirred at room temperature for 24 hours.

The reaction mixture was filtered to remove sodium bromide, and ethanol was evaporated using a waterjet pump at 30 mmHg. The residue was then distilled under vacuum and 3-acetyl-1-phenylpentane-1,4-dione 3 was obtained, b.p. 170-175 °C /3 mm Hg. Yield: 5.76 g (88 %).

Fig. 1. Reaction scheme for the synthesis of 1-[2-methyl-1-(4-methylphenyl)-5-phenyl-1H-pyrrol-3-yl]ethanone 4

In the second stage, a mixture of 3-acetyl-1-phenylpentane-1,4-dione **3** (4.36 g., 0.02 mol) in 20 mL of absolute ethanol was combined with 4-toluidine (2.14 g., 0.02 mol). The mixture was refluxed for 16 hours. After cooling, ethanol was evaporated using a rotary evaporator, and the residue was recrystallized from hexane. A light yellow precipitate of compound **4** was obtained. Yield: 77 % (4.45 g), m. p.: 151–152 °C.

IR Spectrum (ATR, cm $^{-1}$): 1654.41, 1598.78, 1580.24, 1548.87, 1508.93, 1480.41, 1449.03, 1404.82, 1326.38, 1266.48, 1225.12, 1176.62, 1160.94, 1106.74, 1079.64, 1021.17, 952.71, 934.17, 914.20, 822.92, 761.60, 713.11, 695.99, 676.02, 657.48, 643.22, 616.12, 583.32, 513.44, 503.45, 459.24, 432.14, 406.47. MS (m/z): 290 (M $^{+}$ +1). Calcd. For C₂₀H₁₉NO: C 83.01; H 6.62; N 4.84; found: C 82.87; H 6.50; N 4.91.

The structural formula of the obtained compound resulting from the synthesis according to the proposed scheme is shown in Fig. 2.

For the study, solvents from different compound classes with relatively low boiling points were selected, making them convenient for use. Methyl acetate and ethyl acetate are among the most common and accessible solvents. They are low-toxic, highly volatile, and widely used in the chemical and pharmaceutical industries as well as in laboratory practice.

Acetonitrile, which contains a nitrile group (-CN), provides an environment free of hydrogen bonding. In this solvent, all molecular interactions are governed by dipole-dipole and dispersion forces. It is often used to study the behavior of investigated substances under conditions where hydrogen bonding

does not play a role, which is essential for understanding polar and dipole interactions.

Lower alcohols, such as propan-1-ol and propan-2-ol, are less toxic compared to methanol and exhibit properties similar to ethanol. They are readily available, cost-effective, and have lower boiling points than ethanol, making them attractive for laboratory use, especially in processes requiring easy solvent removal.

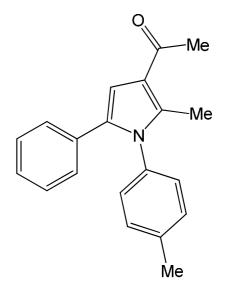


Fig. 2. Structural formula.
1-[2-methyl-1-(4-methylphenyl)5-phenyl-1H-pyrrol-3-yl]ethanone 4

Thus, the selected solvents not only represent different classes of compounds but also possess a range of properties that optimize research processes and ensure reliable results. The solvents used in the study were supplied by Merck: acetonitrile (CAS 75-05-8), methyl

acetate (CAS 79-20-9), ethyl acetate (CAS 71-43-2), n-propanol (CAS 71-23-8), and isopropanol (CAS 67-63-0), with a main component content of \geq 99.9 %, suitable for chromatographic studies.

The temperature dependence of the solubility of 1-[2-methyl-1-(4-methylphenyl)-5-phenyl-1H-pyrrol-3-yl]ethanone was determined using the gravimetric method [6–10].

1-[2-Methyl-1-(4-methylphenyl)-5-phenyl-1H-pyrrol-3-yl]ethanone was dissolved in sealed glass round-bottom flasks equipped with a Teflon stirrer, thermometer, and sampling port. These flasks were immersed in a thermostat that maintained the temperature with an accuracy of ± 0.1 K. The stirrer speed was set at 30–40 revolutions per minute. Saturation of the solutions was carried out in two stages: in the first stage, the mixture was allowed to stand for 48 hours without stirring; in the second stage, it was stirred for 2 hours.

During the experiments, the temperature regime was varied by alternating between heating and cooling to minimize the influence of external factors. The achievement of a state close to equilibrium was confirmed by the absence of a hysteresis loop on the temperature-solubility curve.

Samples were taken in sets of three, which were transferred to pre-weighed sealed heat-resistant glass vials with lids. The solvent was removed in a drying

oven at a temperature of 363–373 K. After the solvent removal, the vials were sealed, cooled in a desiccator to prevent moisture absorption, and weighed. Weighing was performed at room temperature $(296 \pm 2 \text{ K})$ on an analytical balance with an accuracy of ± 0.0002 g. This procedure ensured the highest accuracy of results by excluding the influence of external factors.

This methodology ensures high accuracy in determining solubility and reproducibility of results under various temperature conditions.

Results and discussion

The results of the experimental study on the dissolution of 1-[2-methyl-1-(4-methylphenyl)-5-phenyl-1H-pyrrol-3-yl]ethanone in organic solvents, including the mass of the dissolved substance (m_2) , the mass of the solvent (m_1) , the mole fraction of the dissolved substance (X_2) , and the temperature (T) at which solubility was determined, are presented in Table 1. The linear equations, calculated using the least squares method, are also provided in Table 1 in the form of the Schröder equation (1).

 $lnX2 = -\Delta solH/RT + \Delta solS/R$, (1) where: $\Delta solH$ and $\Delta solS$ represent the enthalpy and entropy of solubility, respectively.

Here and further, the errors of all values are given for a significance level of 0.95

Table 1

Temperature dependence of solubility

| <i>T</i> , K | m_I , g | m_2 , g | $X_2 \cdot 10^3$ | Т, К | m_1 , g | m_2 , g | $X_2 \cdot 10^3$ | <i>T</i> , K | m_I , g | m_2 , g | $X_2 \cdot 10^3$ |
|----------------|----------------|--------------|------------------|--------|-----------|-----------|------------------|--------------|-----------|-----------|------------------|
| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
| Methyl acetate | | | | | | | | | | | |
| 276.45 | 0.9352 | 0.0195 | 5.30 | 284.25 | 1.0280 | 0.0275 | 6.79 | 287.75 | 0.8261 | 0.0248 | 7.63 |
| 276.45 | 0.7204 | 0.0147 | 5.20 | 284.25 | 0.8005 | 0.0216 | 6.84 | 287.75 | 1.1244 | 0.0336 | 7.58 |
| 276.45 | 0.5933 | 0.0122 | 5.22 | 284.25 | 0.6252 | 0.0167 | 6.79 | 287.75 | 0.7888 | 0.0237 | 7.62 |
| 278.55 | 1.2377 | 0.0273 | 5.60 | 285.85 | 1.2208 | 0.0354 | 7.37 | 289.15 | 1.5779 | 0.0510 | 8.20 |
| 278.55 | 1.0656 | 0.0236 | 5.63 | 285.85 | 1.0150 | 0.0293 | 7.34 | 289.15 | 0.6041 | 0.0195 | 8.20 |
| 278.55 | 1.0879 | 0.0241 | 5.64 | 285.85 | 1.1661 | 0.0336 | 7.31 | 289.15 | 1.0706 | 0.0344 | 8.16 |
| 280.75 | 1.1602 | 0.0276 | 6.05 | 286.25 | 0.8721 | 0.0249 | 7.24 | 291.55 | 1.0226 | 0.0356 | 8.83 |
| 280.75 | 1.1652 | 0.0276 | 6.03 | 286.25 | 0.9811 | 0.0277 | 7.18 | 291.55 | 1.0206 | 0.0356 | 8.84 |
| 280.75 | 1.1575 | 0.0271 | 5.96 | 286.25 | 0.7244 | 0.0202 | 7.09 | 291.55 | 0.7587 | 0.0269 | 8.98 |
| $\ln X_2 = (4$ | $.87 \pm 0.34$ | $-(2806 \pm$ | 96)·1/T | | | | | | | | |
| | | | | | Ethyl a | cetate | | | | | |
| 276.45 | 1.3209 | 0.0312 | 7.14 | 281.95 | 1.4111 | 0.0396 | 8.46 | 286.75 | 0.6706 | 0.0222 | 9.98 |
| 276.45 | 1.4142 | 0.0329 | 7.03 | 281.95 | 1.2852 | 0.0359 | 8.43 | 286.75 | 0.8076 | 0.0267 | 9.95 |
| 276.45 | 1.2872 | 0.0304 | 7.13 | 281.95 | 1.3578 | 0.0381 | 8.47 | 286.75 | 0.7410 | 0.0246 | 9.99 |
| 279.45 | 1.3034 | 0.0336 | 7.79 | 285.35 | 0.8894 | 0.0279 | 9.45 | 288.05 | 1.1802 | 0.0412 | 10.51 |
| 279.45 | 1.5266 | 0.0399 | 7.89 | 285.35 | 0.5983 | 0.0189 | 9.50 | 288.05 | 1.2216 | 0.0429 | 10.57 |
| 279.45 | 1.4183 | 0.0367 | 7.82 | 285.35 | 0.7709 | 0.0243 | 9.51 | 288.05 | 1.3923 | 0.0490 | 10.59 |

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Continuation Table 1

| 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 1.1 | 12 | |
|--|--|------------------|------|------------------|-------------------------|------------------|---------|------------------|------------------|------------------|-------|--|
| | | | | | | | | | | 11 | | |
| 280.15 | 1.1168 | 0.0298 | 8.05 | 286.15 | 0.6965 | 0.0231 | 9.98 | 290.45 | 0.7402 | 0.0286 | 11.63 | |
| 280.15 | 1.1003 | 0.0291 | 7.99 | 286.15 | 0.9222 | 0.0304 | 9.92 | 290.45 | 0.6107 | 0.0237 | 11.66 | |
| 280.15 1.3275 0.0352 8.01 286.15 1.1941 0.0394 9.93 290.45 0.8516 0.0332 11.73 | | | | | | | | | | | 11.73 | |
| $\ln X_2 = (5.20 \pm 0.31) - (2806 \pm 87) \cdot 1/T$ | | | | | | | | | | | | |
| 270.07 | 0.5040 | 0.000# | 1.02 | 20127 | Aceton | | 0.71 | 200.05 | 1.1500 | 0.0240 | 205 | |
| 279.95 | 0.6940 | 0.0095 | 1.93 | 286.25 | 1.0188 | 0.0181 | 2.51 | 290.95 | 1.1532 | 0.0249 | 3.05 | |
| 279.95 | 0.8342 | 0.0114 | 1.94 | 286.25 | 0.8464 | 0.0150 | 2.51 | 293.45 | 1.1964 | 0.0284 | 3.36 | |
| 279.95 | 0.6730 | 0.0093 | 1.95 | 288.25 | 0.9984 | 0.0196 | 2.77 | 293.45 | 1.3259 | 0.0315 | 3.36 | |
| 281.45 | 1.1843 | 0.0166 | 1.99 | 288.25 | 0.9043 | 0.0177 | 2.76 | 293.45 | 1.3882 | 0.0332 | 3.38 | |
| 281.45 | 1.5422 | 0.0218 | 2.00 | 288.25 | 0.9142 | 0.0178 | 2.75 | 295.05 | 1.0723 | 0.0267 | 3.52 | |
| 281.45 | 1.6711 | 0.0238 | 2.02 | 290.05 | 1.0991 | 0.0230 | 2.95 | 295.05 | 1.1297 | 0.0283 | 3.54 | |
| 283.45 | 1.2456 | 0.0195 | 2.22 | 290.05 | 1.0763 | 0.0226 | 2.96 | 295.05 | 1.0435 | 0.0263 | 3.56 | |
| 283.45 | 1.5248 | 0.0237 | 2.20 | 290.05 | 1.5166 | 0.0316 | 2.95 | 297.65 | 1.3584 | 0.0374 | 3.89 | |
| 283.45 | 1.2437 | 0.0197 | 2.24 | 290.95 | 1.1992 | 0.0261 | 3.07 | 297.65 | 1.3658 | 0.0381 | 3.94 | |
| 286.25 | 1.0259 | 0.0183 | 2.52 | 290.95 | 1.0729 | 0.0232 | 3.05 | 297.65 | 1.3061 | 0.0362 | 3.92 | |
| $\ln X_2 = (5.89 \pm 0.23) - (3398 \pm 65) \cdot 1/T$ Propane-1-ol | | | | | | | | | | | | |
| 275.05 | 1 1460 | 0.0102 | 1.00 | 202.25 | | | 2.16 | 200.05 | 1.0154 | 0.0120 | 2.60 | |
| 275.85 | 1.1462 | 0.0103 | 1.86 | 282.35 | 1.0377 | 0.0108 | 2.16 | 290.95 | 1.0154 | 0.0128 | 2.60 | |
| 275.85 | 1.1720 | 0.0106 | 1.87 | 282.35 | 0.8396 | 0.0087 | 2.15 | 290.95 | 0.6798 | 0.0086 | 2.62 | |
| 275.85 | 1.1366 | 0.0101 | 1.83 | 282.35 | 1.0107 | 0.0105 | 2.14 | 290.95 | 0.5552 | 0.0071 | 2.63 | |
| 278.55 278.55 | 0.7716 1.2062 | 0.0072 0.0116 | 1.93 | 284.95 | 1.2892 | 0.0141 | 2.26 | 294.75 | 0.7334 0.9057 | 0.0100 0.0122 | 2.82 | |
| | | | | 284.95 | 1.3729 | | | 294.75 | | | | |
| 278.55 | 0.8067 | 0.0077 | 1.97 | 284.95 | 0.9583 | 0.0105 | 2.27 | 294.75 | 0.7243 | 0.0099 | 2.82 | |
| 279.35 279.35 | 0.4811 1.2601 | 0.0047 0.0122 | 2.02 | 286.25 286.25 | 0.7568 0.3726 | 0.0085 0.0042 | 2.31 | 299.25 299.25 | 0.9289 0.8689 | 0.0138 | 3.06 | |
| 279.35 | 1.1665 | 0.0122 | 2.02 | 286.25 | 0.5720 | 0.0042 | 2.34 | 299.25 | 0.8689 | 0.0129 | 3.04 | |
| 219.33 | 1.1003 | 0.0114 | 2.02 | | 0.3384 19 ± 0.12 | | l | 299.23 | 0.7363 | 0.0112 | 3.04 | |
| | | | | $m_{A2} = (0.$ | Propan | | 33) 1/1 | | | | | |
| 277.55 | 0.5762 | 0.0037 | 1.33 | 282.05 | 1.1686 | 0.0083 | 1.46 | 288.45 | 1.1364 | 0.0092 | 1.68 | |
| 277.55 | 0.5368 | 0.0036 | 1.37 | 283.95 | 1.0474 | 0.0077 | 1.52 | 288.45 | 1.5983 | 0.0129 | 1.67 | |
| 277.55 | 0.8157 | 0.0054 | 1.36 | 283.95 | 0.7912 | 0.0058 | 1.51 | 289.45 | 0.8339 | 0.0070 | 1.74 | |
| 280.75 | 0.6399 | 0.0044 | 1.43 | 283.95 | 0.9216 | 0.0069 | 1.54 | 289.45 | 0.5606 | 0.0047 | 1.72 | |
| 280.75 | 0.5482 | 0.0039 | 1.46 | 285.45 | 0.6861 | 0.0053 | 1.59 | 289.45 | 0.7620 | 0.0064 | 1.73 | |
| 280.75 | 0.7875 | 0.0055 | 1.45 | 285.45 | 0.6933 | 0.0053 | 1.57 | 291.45 | 0.7920 | 0.0053 | 1.83 | |
| 282.05 | 0.7516 | 0.0054 | 1.49 | 285.45 | 0.8656 | 0.0055 | 1.58 | 291.45 | 0.5201 | 0.0033 | 1.81 | |
| | 0.7516 | | | 288.45 | | | 1.58 | | | | | |
| 282.05 | | | | | | | | | | | | |
| | $\ln X_2 = (-0.44 \pm 0.33) - (1713 \pm 94) \cdot 1/T$ | | | | | | | | | | | |

The thermodynamic parameters of solubility, $\Delta_{sol}H$ and $\Delta_{sol}S$, presented in Table 2, account for the phase transition of solid substances into the liquid phase of the solution and the solution formation process. Therefore, to calculate the enthalpy change $(\Delta_{mix}H)$ and entropy change $(\Delta_{mix}S)$ of mixing (solvation), which reflect the interaction of

components in the solution, it is necessary to consider the enthalpy ($\Delta_{fus}H$) and entropy ($\Delta_{fus}S$) of fusion of the studied substance at the average dissolution temperature, as given by equations (2–3).

$$\Delta_{sol}H = \Delta_{fus}H + \Delta_{mix}H, \qquad (2)$$

$$\Delta_{sol}S = \Delta_{fus}S + \Delta_{mix}S \tag{3}$$

Thermodynamic functions of solubility of 1-[2-methyl-1-(4-methylphenyl) -5-phenyl-1H-pyrrol-3-yl]ethanone n solvents at 298.15 K

| Solvent | X_2 | $\Delta_{sol}H$, | $\Delta_{mix}H$, | $\Delta_{sol}S$, | $\Delta_{mix}S$, |
|----------------|----------|-------------------|-------------------|-------------------|-------------------|
| Solveill | (298.15) | kJ/mol | kJ/mol | $J/(mol \cdot K)$ | J/(mol·K) |
| Methyl acetate | 0.0109 | 23.28±0.80 | -3.5±1.6 | 40.4±2.8 | -20.8±3.4 |
| Ethyl acetate | 0.0148 | 23.22±0.37 | -3.6±1.4 | 43.2±2.6 | -18.0±3.3 |
| Acetonitrile | 0.0040 | 28.25±0.54 | 1.5±1.5 | 48.9±1.9 | -12.3±2.8 |
| Propane-1-ol | 0.0030 | 14.87±0.38 | -11.9±1.5 | 1.6±1.0 | -59.6±2.2 |
| Propane-2-ol | 0.0021 | 14.24±0.78 | -12.6±1.6 | -3.65±2.7 | -64.9±3.4 |

The values of temperature and enthalpy of melting were determined based on the results of differential thermal analysis (DTA) performed using a Q-1500 D derivatograph of the Paulik–Paulik–Erdey system in a dynamic mode under an air atmosphere with a heating rate of 5 °C/min and sensitivities set as follows: TG – 100 mg; DTA up to 773 K in a platinum crucible.

The calculation of the melting enthalpy ($\Delta_{fits}H$) was performed using equation (4), taking into account the loss of heat due to the evaporation of the sample [11]:

$$\begin{split} K \cdot S &= Q_{fus} + Q_{vap} = m_0 \cdot \Delta_{fus} H + \\ &+ \Delta m_{vap} \times \Delta_{vap} H, \end{split} \tag{4} \\ K \cdot S &= Q_{fus} + Q_{vap} = m_0 \cdot \Delta_{fus} H + \\ &+ \Delta m_{vap} \times \Delta_{vap} H, \end{split} \tag{4}$$

where K – the heat transfer coefficient of the derivatograph is equal to $8.2023 \cdot 10^{-5} \cdot T_{fus}$, $J/(K \cdot s)$; Q_{fus} and Q_{vap} – the amount of heat absorbed during melting and evaporation of the sample, respectively, J; $\Delta_{fus}H$ and $\Delta_{vap}H$ – specific enthalpies of melting and vaporization of the substances, respectively m_0 , J/g; – the sample mass corresponding to the temperature at the start of its melting T_{fus} , g; Δm_{vap} – The mass loss of the sample (mass of the vapor) during the period considered when determining the peak area $S(K \cdot s)$ on the DTA curve, g.

The necessary values for calculating the enthalpy of vaporization $(\Delta_{vap}H)$ were determined

thermogravimetrically from the temperature dependence of the evaporation rate $V=\Delta m'/\Delta \tau$ in the temperature range where the substance was in the liquid phase, and thermoxidative decomposition processes were absent. The evaporation rate was determined by differentiating the mass loss curve of the sample every 30 seconds. The temperature dependence of the evaporation rate was approximated by a linear form of the Arrhenius equation: lnV = A - B/T, where: $B = E_{axm}/R$. Assuming that the condensation of vapor in the presence of the liquid phase is nearly a non-activation process, the activation enthalpy is equal to $\Delta vapH$ of the substance, considering the correction for the expansion work, as shown in equation 5.

$$E_{\rm akt} + RT_{fus} = \Delta_{vap}H, \tag{5}$$

Table 3

Table 3 presents the main values necessary for the calculation of $\Delta_{fus}H$ and $\Delta_{vap}H$ using equations 4 and 5, as well as: $T_1 - T_2$ – the temperature range during which the mass loss rate of the sample was analyzed.

Since the experimentally determined values of $\Delta_{fus}H$ correspond to T_{fus} , and the determined thermodynamic parameters of the dissolution process, $\Delta_{sol}H$ and $\Delta_{sol}S$, correspond to the temperature intervals in which solubility was determined and are close to the generally accepted temperature of 298.15 K, it was decided to analyze the thermodynamic parameters of the dissolution process at 298.15 K in order to generalize the obtained results.

Thermodynamic parameters of phase transitions of 1-[2-methyl-1-(4-methylphenyl)-5-phenyl-1H-pyrrol-3-yl]acetone

| No. | mo, g | $Dm_{vap},*$ $10^4 { m g}$ | S, K≽s | q_{vap},J | $\Delta_{	extit{fus}}H, \ 	ext{kJ/mol}$ | T_1 - T_2 , K | A | -В, K | $\Delta_{vap}H, \ 	ext{kJ/mol}$ | | |
|----------------|---|-----------------------------|--------|----------------------|---|-------------------|-------|-----------|---------------------------------|--|--|
| | $T_{fus} = 416,45\pm1,20 \text{ K}; \text{ K}=0,03416 \text{ J/(K}\cdot\text{s})$ | | | | | | | | | | |
| 1 | 0.0984 | 3.26 | 334.3 | 0.0822 | 33.34 | 496.0-539.6 | 7.56 | 8336 | 72.77 | | |
| 2 | 0.1105 | 6.20 | 388.3 | 0.1562 | 34.33 | 490.6-525.3 | 7.42 | 8379 | 73.12 | | |
| 3 | 0.1017 | 4.92 | 352.3 | 0.1239 | 33.89 | 494.1-548.2 | 7.63 | 8357 | 72.94 | | |
| Average value: | | | | | 33.9±1.2 | | Avera | ge value: | 72.9±1.0 | | |

The recalculation of $\Delta_{fits}H$ and $\Delta_{fits}S$ to 298.15 K was carried out using equations 6 and 7, which are presented in [12].

$$\Delta_{fus}H_T = \Delta_{fus}H_{T_{fus}}\left[1 + \frac{T - T_{fus}}{1.35 \cdot T_{fus}}\right],\tag{6}$$

$$\Delta_{fus}S_T = \Delta_{fus}S_{T_{fus}} \left[\mathbf{1} + \mathbf{0.74} \cdot \ln \frac{T}{T_{fus}} \right]. \quad (7)$$

The value of the enthalpy of fusion was calculated using the equation.

$$\Delta_{fus}S_{Tfus} = D_{fus}H_{Tfus}/T_{fus} = 33900/416,45 = 81,4\pm1,8 \text{ (J/(mol \cdot K))},$$
 (8)

The values calculated using equations 6 and 7, $\Delta_{fus}S_{298,15} = 61,2\pm2,0$ (J/(mol·K));

та $\Delta_{fits}H_{298,15} = 26,8\pm1,4$ kJ/mol were used to calculate $\Delta_{mix}H$ and $\Delta_{mix}S$.

The calculated values for the mixing process are presented in Table 2.

Judging by the obtained experimental results for $\Delta_{mix}H$ and $\Delta_{mix}S$ of 1-[2-methyl-1-(4-methyl-phenyl)-5-phenyl-1H-pyrrol-3-yl]ethanone in esters and alcohols, their values are negative. This indicates that the energy spent on breaking the bonds in the initial molecules is fully, with some excess, compensated by the energy released when new bonds are formed between the solvent and the dissolved substance. As for the solution of 1-[2-methyl-1-(4-methylphenyl)-5-phenyl-1H-pyrrol-3-yl]ethanone in acetonitrile, the value of $\Delta_{mix}H$ is within the experimental error and calculation margin, close to zero, meaning that the energy balance is maintained uniformly.

Conclusion

Based on the results of the conducted studies, the temperature dependencies of 1-[2-methyl-1-(4-methylphenyl)-5-phenyl-1H-pyrrol-3-yl]ethanone in methyl acetate, ethyl acetate, acetonitrile, propan-1-ol, and propan-2-ol were determined. The thermodynamic parameters of their solubility and mixing (solvation) were calculated.

These findings provide insights into the dissolution mechanism of the compound and highlight the influence of solvent properties on the dissolution thermodynamics, which is essential for optimizing experimental conditions in chemical and pharmaceutical applications.

The obtained data are consistent with previously conducted studies and do not contradict the nature of the interaction between the hydroxyl and carbonyl groups.

References

- 1. Hunjan, M. K., Panday, S., Gupta, A., Bhaumik, J., Das, P., Laha, J. K. (2021). Recent Advances in Functionalization of Pyrroles and their Translational Potential. *Chem. Rec.* Vol. 21. P. 715–780. https://doi.org/10.1002/tcr.202100010.
- 2. Buʻrli, R. W., McMinn, D., Kaizerman, J. A., Hu, W., Ge, Y., Pack, Q., Jiang, V., Gross, M., Garcia, M., Tanaka, R., Moser, H. E. Bioorg. (2004). *Med. Chem. Lett.*, 14, 1253–1257. https://doi.org/10.1007/s11030-020-10078-2
- 3. Demopoulosn V. J., Rekka E. J. (1995). *PharmSci.* 84, 79–82 https://doi.org/10.3390/ molecules 200916354
- 4. Street, G. B., Skotheim, T. A. (1986). *Handbook of Conducting Polymers*, vol. 1, Marcel Dekker, New York, (Chapter 8) https://doi.org/10.1007/978-3-319-95987-0_17
- 5. Ridka, O. R., Matiychuk, V. S., Sobechko, I. B., Serheyev, V. V., Tishchenko, N. I. (2019). Thermodynamic properties of methyl 6-methyl-2-oxo-4-phenyl-1,2,3,4-tetrahydropyrimidine-5-carboxylate and its solutions in ethyl acetate and benzene and their mix tures. *Chemistry, Technology and Application of Substances*, 2 (2), 12–17. https://doi.org/ 10.23939/ctas2019.02.012.
- 6. Du, C. (2022). The solubility of ethyl candesartan in mono solvents and investigation of intermolecular interactions. *Liquids*, 2(4), 404–412. https://doi.org/10.3390/liquids2040023
- 7. Li, Z., Guo, J., Hu, B., Zhou, C., Zheng, Y., Zhao, H., Li, Q. (2022). Solubility measurement, modeling, and solvent effect of M-hydro xyacetophenone in ten pure and binary mixed solvents from T = (289.15–325.15) K. *Journal of Molecular Liquids*, 353, 118798. https://doi.org/10.1016/j.molliq.2022.118798.
- 8. Maharana, A., Sarkar, D. (2019). Solubility measurements and thermodynamic modeling of pyrazinamide in five different solvent-antisolvent mixtures. *Fluid Phase Equilibria*, 497, 33–54. https://doi.org/10.1016/j.fluid.2019.06.004
- 9. Huang, W., Wang, H., Li, C., Wen, T., Xu, J., Ouyang, J., Zhang, C. (2021). Measurement and correlation of solubility, Hansen solubility parameters and thermodynamic behavior of clozapine in eleven mono-solvents. *Journal of Molecular Liquids*, 333, 115894. https://doi.org/10.1016/j.molliq.2021.115894
- 10. Wu, Y., Zhang, X., Di, Y., Zhang, Y. (2017). Solubility determination and modelling of

- 4-Nitro-1,2-phenylenediamine in eleven organic solvents from T=(283.15 to 318.15) K and thermodynamic properties of solutions. *The Journal of Chemical Thermodynamics*, 106, 22–35. https://doi.org/10.1016/j.jct.2016.11.014
- 11. Sobechko, I., Dibrivnyi, V., Horak, Y., Velychkivska, N., Kochubei, V., Obushak, M. (2017). Thermodynamic properties of solubility of 2-methyl-5-arylfuran-3-carboxylic acids in organic
- solvents, *Chemistry & Chemical Technology*, Vol. 11. No. 4. P. 397–404. https://doi.org/10.23939/ chcht11. 04.397
- 12. Horak, Y. I., Shevchenko, D. S., Sobechko, I. B. (2023) Thermodynamic Parameters of 5-(Nitrophenyl)-Furan-2-Carboxylic Acids Solutions in Propan-2-Ol. *Chemistry, Technology and Application of Substances*, 6 (1), 15–21. https://doi.org/10.23939/ctas2023.01.015.

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СИНТЕЗ ТА РОЗЧИННІСТЬ 1-[2-МЕТИЛ-1-(4-МЕТИЛФЕНІЛ)-5-ФЕНІЛ-1H-ПІРОЛ-3-ІЛ]ЕТАНОНУ В ОРГАНІЧНИХ РОЗЧИННИКАХ

Досліджено температурну залежність розчинності 1-[2-метил-1-(4-метилфеніл)-5-феніл-1H-пірол-3-іл]етанону в метилацетаті, етилацетаті, ацетонітрилі, н-пропанолі та ізо-пропанолі. Розраховано ентальпію та ентропію розчинення. За результатами диференційно-термічного аналізу визначено термодинамічні параметри процесу плавлення та температуру плавлення. Проведено перерахунок ентальпії та ентропії плавлення до 298,15 К. Розраховано величини ентальпії та ентропії змішування 1-[2-метил-1-(4-метилфеніл)-5-феніл-1H-пірол-3-іл]етанону з метилацетатом, етилацетатом, ацетонітрилом, н-пропанолом та ізо-пропанолом.

Ключові слова: розчинність, ентальпія розчинення, ентальпія змішування, ентальпія плавлення, ентальпія випаровування, полізаміщені похідні піролу.