# Solubilities of 2-(6-Oxido-6*H*-dibenz[*c*,*e*][1,2]oxaphosphorin-6-yl)-1,4-dihydroxy Phenylene in the Selected Solvents

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The 2-(6-oxido-6*H*-dibenz[*c*,*e*][1,2]oxaphosphorin-6-yl)-1,4-dihydroxy phenylene (ODOPB) was prepared by the addition reaction between 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO) and *p*-benzoquinone. The DOPO was synthesized through a multistep reaction from *o*-phenylphenol and phosphorus trichloride. The structures and the thermal stability of the compound were characterized by infrared spectroscopy (IR), nuclear magnetic resonance ($^1$H NMR and $^{31}$P NMR), mass spectroscopy (MS), elemental analysis, and thermogravimetric analysis (TGA). Using a static analytical method, the solubilities of ODOPB in 2-ethoxyethanol in the temperature range from (293.15 to 367.65) K and in methanol in the temperature range from (293.15 to 322.63) K were determined. The solubilities of ODOPB in acetone, ethanol, and 2-ethoxyethanol + water binary mixtures were also measured for comparison. The solubility data were correlated with an empirical equation.

## Introduction

Polymers with improved fire retardancy are needed for a number of future industrial and commercial applications. Commercial fire retardant polymers generally contain elements such as phosphorus, nitrogen, and halogen. To avoid the problems of smoke, toxicity, and corrosion that were caused by organic bromide, more and more organic phosphorus compounds are being used as flame retardants in polymers owing to their excellent char-forming property and because they generate less toxic combustion products in the case of fire.[1–5] Phosphorus-containing polymers are increasingly gaining popularity over their halogen counterparts in the aspects of environmental and health considerations.

An aromatic phosphorus-containing diol 2-(6-oxido-6*H*-dibenz[*c*,*e*][1,2]oxaphosphorin-6-yl)-1,4-dihydroxy phenylene (ODOPB; its formula is shown in Figure 1) (CASRN 99208-50-1) has been used as a reactive flame retardant.[6] Owing to the introduction of the rigid structure of ODOPB and the pendant phosphorus-containing group, the resultant resins provided not only better flame retardant properties[7] but also higher thermal stability and glass transition temperature.[8] One important application of ODOPB is its use in epoxy resin based laminates for printed circuit boards. ODOPB has also been incorporated with phenyl phosphonic dichloride to obtain polyesters with an enhanced content of phosphorus and of an aryl group.[9–11]

To be a monomer of a polymer, high purity is needed. ODOPB was prepared by the reaction of 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO) and excessive *p*-benzoquinone in 2-ethoxyethanol. The product was obtained by a subsequent crystallization upon cooling the reaction mixture and washing the filter cake with methanol.[6] Knowledge of solubilities of ODOPB in 2-ethoxyethanol and methanol (as well as other common solvents such as ethanol and acetone) as a function of temperature is necessary for subsequent purification.

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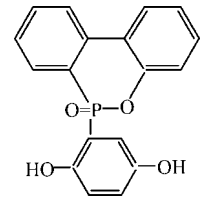


**Figure 1.** Structure of 2-(6-oxido-6*H*-dibenz[*c*,*e*][1,2]oxaphosphorin-6-yl)-1,4-dihydroxy phenylene.

**Table 1. Mass Fraction Purity *w*, Density $\rho$, and Refractive Index $n_D$ for the Organic Solvents Used in This Work at $T = 293.15$ K**

| solvent | *w*/% | $\rho$/g·cm$^{-3}$ | $n_D$ |
|---|---|---|---|
| 2-ethoxyethanol | 99.5 | 0.929 | 1.4065 |
| acetone | 99.5 | 0.790 | 1.3590 |
| ethanol | 99.7 | 0.789 | 1.3619 |
| methanol | 99.5 | 0.791 | 1.3287 |

As our continuous efforts to search for high thermally stable flame-retardant polyesters, ODOPB was synthesized and characterized. The solubility data of ODOPB in 2-ethoxyethanol, methanol, ethanol, and acetone were measured. In the purification of ODOPB, it was found that the solid can be washed by water instead of methanol to remove the unreacted *p*-benzoquinone. The solubilities of ODOPB in 2-ethoxyethanol + water binary mixtures were also measured. To the best of our knowledge, no such data have been reported in the literature.

## Experimental Section

***Materials.*** All the chemicals in the synthesis and measurement were analytical grade reagents, which were purchased from Beijing Chemical Factory. They were used without further purification. Table 1 presents some physical properties of solvents such as density, refractive index, and purity. Their mass fraction purities were all higher than 99 %.

***Apparatus and Procedure.*** The melting points and enthalpy of fusion were determined with a DSC Q100 differential scanning calorimeter (DSC) in flowing nitrogen at a heating rate of 10 K·min$^{-1}$. The elemental analysis was performed on

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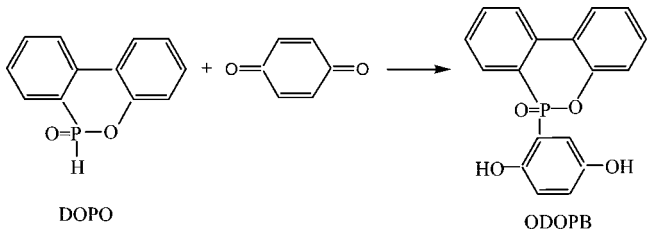
**Scheme 1**




an Elementar Vario EL element analyzer. IR spectra (Fourier transform infrared (FTIR)) were recorded on a Magna-IR 750 using KBr pellets. Mass spectra were recorded by a VG-ZAB-HS. $^{1}$H NMR and $^{31}$P NMR spectra were obtained with a BrukerARX-400 and JEOL ECA-600, respectively. Thermogravimetric analysis (TGA) was carried out with an SDT Q600 thermogravimetric analyzer at a heating rate of 10 K·min$^{-1}$ under nitrogen from (298.15 to 1073.15) K.

A jacketed equilibrium cell was used for the solubility measurement with a working volume of 120 mL and a magnetic stirrer, as described by Wang et al.[12,13] A circulating water bath was used with a thermostat (type 50 L, made from Shanghai Laboratory Instrument Works Co., Ltd.), which is capable of maintaining the temperature within ± 0.05 K. An analytical balance (type TG328B, Shanghai Balance Instrument Works Co.) with an uncertainty of ± 0.1 mg was used during the mass measurements.

***Synthesis of ODOPB.*** ODOPB was prepared by the reaction of 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO) and *p*-benzoquinone, according to the published procedure (Scheme 1).[5] The yield of ODOPB was 92 %, and the melting point was 524.07 K (lit. (528.15 to 529.15) K,[5] (518.15 to 519.15) K[14]). IR (KBr): 3178 (Ph−OH); 1182 (*P*=O); 932 (P−O−Ph); 1475 (P−Ph); 1503, 1594, 756 cm$^{-1}$ (Ph). MS (EI) *m*/*z*: 324(M)$^{+}$. $^{1}$H NMR (400 MHz, DMSO) ppm: $\delta$ = 6.62 to 6.66 (t, 1H), 6.88 to 6.91 (d, 1H), 7.18 to 7.32 (m, 3H), 7.43 to 7.59 (m, 3H), 7.61 to 7.74 (t, 1H), 8.23 to 8.24 (d, 2H), 9.18 (s, 1H, OH), 9.47 (s, 1H, OH).[11] $^{31}$P NMR (600 MHz, DMSO-$d_6$) ppm: $\delta$ = 22.74 (s) (lit. 21.52[4]). Elemental analysis (%, calcd): *C* = 66.43 % (66.67 %); *H* = 4.43 % (4.04 %). Based on the above analysis, the purity of ODOPB used in this work was higher than 99.0 %.

***Thermogravimetric Analysis.*** An SDT Q600 Simultaneous DTA-TGA thermogravimetric analyzer was employed for thermogravimetric analysis at a heating rate of 10 K·min$^{-1}$ under nitrogen from (298.2 to 1073.2) K. The thermogravimetric curve of ODOPB is shown in Figure 2. The initial decomposition temperature of ODOPB was around 605.9 K. The temperature at 93.42 % mass loss was 658.2 K, and the char yield at 973.2 K was 6.58 %. Figure 3 shows the results of the differential scanning calorimeter (DSC) measurement of ODOPB. The enthalpy of fusion of ODOPB was 128.6 J·g$^{-1}$.

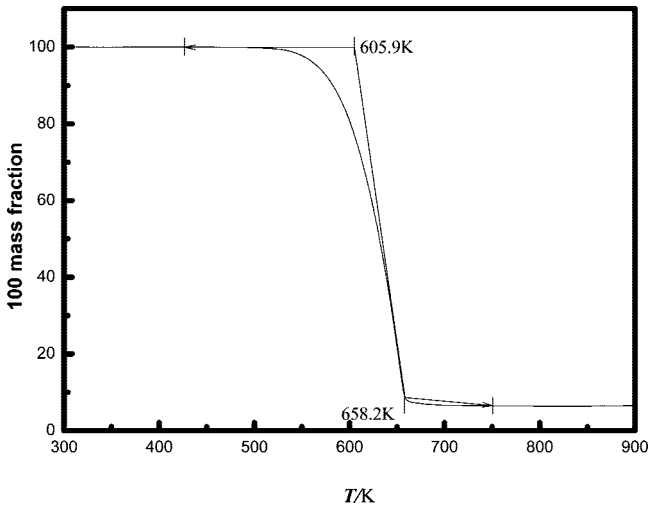



**Figure 2.** Thermogravimetric analysis (TGA) curves of ODOPB.

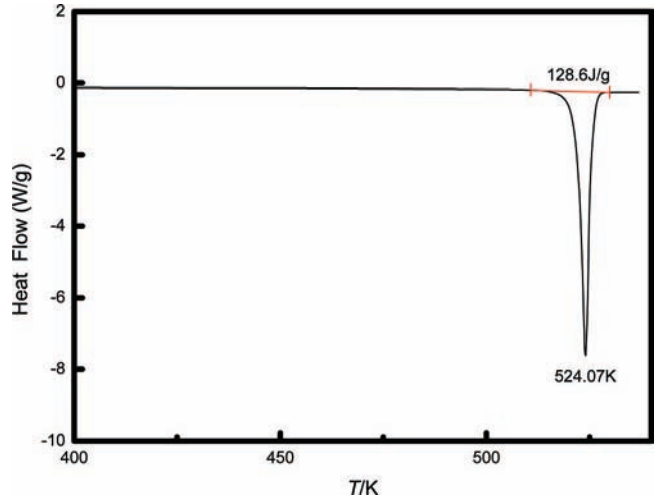



**Figure 3.** Results of the differential scanning calorimeter (DSC) measurement of ODOPB.

***Solubility Measurement.*** The solubilities were measured by a gravimetric method.[12] For each measurement, an excess mass of ODOPB was added to a known mass of solvent. Then the equilibrium cell was heated to a constant temperature with continuous stirring. After at least 2 h (the temperature of the water bath approached constant value, then the actual value of the temperature was recorded), the stirring was stopped and the solution was kept still for 2 h. A preheated injector withdrew 2 mL of the clear upper portion of the solution to another previously weighed measuring vial ($m_0$). The vial was quickly and tightly closed and weighed ($m_1$) to determine the mass of the sample ($m_1 - m_0$). Then the vial was covered with a piece of filter paper to prevent dust contamination. After the solvent in the vial had completely evaporated, the vial was dried and reweighed ($m_2$) to determine the mass of the constant residue solid ($m_2 - m_0$). Thus, the solid concentration of the sample solution in mole fraction, *x*, could be determined from eq 1 or eq 2[15]

$$x = \frac{(m_2 - m_0)/M_1}{(m_2 - m_0)/M_1 + (m_1 - m_2)/M_2} \quad (1)$$

where the $M_1$ is the molar mass of ODOPB and $M_2$ is the molar mass of the solvent.

$$x = \frac{(m_2 - m_0)/M_1}{(m_2 - m_0)/M_1 + (m_1 - m_2)w_2/M_2 + (m_1 - m_2)(1 - w_2)/M_3} \quad (2)$$

Equation 2 is for the mixed solvent, where $M_1$, $M_2$, and $M_3$ are the molar masses of ODOPB, 2-ethoxyethanol, and water, where $w_2$ is the mass fraction of 2-ethoxyethanol in the solvents.

Different dissolution times were tested to determine a suitable equilibrium time. It was found that 2 h was enough for ODOPB in all solvents to reach equilibrium. During our experiments,

**Table 2. Mole Fraction Solubilities $x$ of ODOPB in the Selected Solvents**

| solvent | $T$/K | $10^3x$ | $\gamma$ | $(x - x^{calcd})/x$ |
|---|---|---|---|---|
| methanol | 293.15 | 1.07 | 0.499 | 0.004 |
| | 298.00 | 1.28 | 0.553 | 0.012 |
| | 302.90 | 1.50 | 0.618 | −0.028 |
| | 307.85 | 1.82 | 0.664 | 0.000 |
| | 312.84 | 2.27 | 0.692 | 0.051 |
| | 317.60 | 2.60 | 0.768 | 0.021 |
| | 322.63 | 2.92 | 0.875 | −0.030 |
| 2-ethoxyethanol | 293.15 | 0.896 | 0.598 | 0.011 |
| | 298.15 | 1.20 | 0.596 | 0.009 |
| | 302.98 | 1.57 | 0.593 | 0.008 |
| | 308.00 | 2.07 | 0.591 | 0.007 |
| | 313.00 | 2.48 | 0.639 | −0.079 |
| | 317.80 | 3.47 | 0.581 | 0.015 |
| | 322.70 | 4.39 | 0.585 | 0.004 |
| | 327.65 | 5.56 | 0.583 | 0.003 |
| | 332.65 | 7.02 | 0.581 | 0.002 |
| | 337.60 | 8.96 | 0.568 | 0.020 |
| | 342.58 | 10.9 | 0.577 | 0.000 |
| | 347.40 | 13.4 | 0.576 | −0.001 |
| | 352.83 | 16.8 | 0.574 | −0.002 |
| | 357.70 | 20.7 | 0.567 | 0.006 |
| | 362.67 | 24.9 | 0.571 | −0.004 |
| | 367.65 | 30.1 | 0.569 | −0.004 |
| acetone | 292.15 | 1.53 | 0.330 | 0.030 |
| | 297.15 | 1.82 | 0.370 | −0.023 |
| | 302.15 | 2.26 | 0.395 | −0.032 |
| | 307.85 | 3.04 | 0.399 | 0.022 |
| | 312.90 | 3.70 | 0.426 | 0.009 |
| | 317.65 | 4.27 | 0.469 | −0.037 |
| | 322.60 | 5.51 | 0.463 | 0.026 |
| ethanol | 293.15 | 2.05 | 0.261 | −0.002 |
| | 298.10 | 2.24 | 0.318 | −0.002 |
| | 303.05 | 2.41 | 0.389 | −0.011 |
| | 307.95 | 2.70 | 0.451 | 0.023 |
| | 312.95 | 2.82 | 0.560 | −0.011 |
| | 317.80 | 3.09 | 0.653 | 0.006 |
| | 322.80 | 3.32 | 0.775 | 0.005 |
| | 327.85 | 3.52 | 0.929 | −0.009 |
| water | 303.00 | 0.139 | 6.719 | 0.007 |
| | 307.95 | 0.143 | 8.520 | −0.010 |
| | 312.85 | 0.151 | 10.41 | 0.007 |
| | 317.60 | 0.155 | 12.89 | −0.007 |
| | 322.45 | 0.165 | 15.35 | 0.012 |
| | 327.65 | 0.168 | 19.29 | −0.012 |
| | 332.50 | 0.175 | 23.15 | −0.006 |
| | 337.55 | 0.184 | 27.59 | 0.008 |
| | 342.35 | 0.189 | 33.07 | 0.002 |

three parallel measurements were performed at the same composition of solvent for each temperature, and an average value is given. The maximum standard deviation of each triplicate data is 0.25 %, and the minimum is 0.15 %. The estimated relative uncertainty of the solubility values based on error analysis and repeated observations was within 0.02.

## Results and Discussion

The mole fraction solubility data of ODOPB, $x$, in selected solvents are summarized in Table 2 and Table 3, and plotted as ln $x$ vs temperature in Figures 4 and 5. From these figures, it can be seen that a trend of increasing solubility with temperature is observed. The solubilities were correlated as a function of temperature by

$$\ln x = A + B/(T/\mathrm{K}) \tag{3}$$

Parameters $A$ and $B$ for each solvent are listed in Table 4. The smoothed data calculated from eq 3 are listed in Table 2 and Table 3. The relative standard deviations (RSD), defined by eq 4, are also presented in Table 4

**Table 3. Mole Fraction Solubilities $x$ of ODOBP in $w$ 2-Ethoxyethanol + (1−$w$) Water**

| $w$ | $T$/K | $10^3x$ | $\gamma$ | $(x - x^{calcd})/x$ |
|---|---|---|---|---|
| 0.1983 | 298.25 | 0.136 | 5.277 | 0.090 |
| | 303.20 | 0.151 | 6.252 | 0.011 |
| | 308.05 | 0.172 | 7.121 | −0.040 |
| | 313.00 | 0.202 | 7.842 | −0.056 |
| | 317.85 | 0.229 | 8.832 | −0.099 |
| | 322.75 | 0.297 | 8.652 | 0.004 |
| | 327.65 | 0.340 | 9.533 | −0.022 |
| | 332.65 | 0.430 | 9.486 | 0.054 |
| | 337.60 | 0.492 | 10.34 | 0.039 |
| | 342.55 | 0.551 | 11.44 | 0.006 |
| 0.4012 | 298.20 | 0.149 | 4.803 | −0.035 |
| | 303.10 | 0.200 | 4.695 | 0.049 |
| | 308.05 | 0.242 | 5.061 | 0.031 |
| | 312.90 | 0.293 | 5.379 | 0.021 |
| | 317.85 | 0.330 | 6.129 | −0.058 |
| | 322.75 | 0.405 | 6.345 | −0.040 |
| | 327.90 | 0.504 | 6.506 | −0.013 |
| | 332.85 | 0.619 | 6.650 | 0.014 |
| | 337.70 | 0.726 | 7.038 | 0.002 |
| | 342.60 | 0.878 | 7.196 | 0.024 |
| 0.6032 | 293.25 | 0.496 | 1.086 | 0.043 |
| | 298.20 | 0.593 | 1.207 | 0.021 |
| | 304.40 | 0.750 | 1.344 | 0.010 |
| | 308.05 | 0.830 | 1.476 | −0.029 |
| | 313.00 | 0.981 | 1.615 | −0.046 |
| | 317.95 | 1.19 | 1.708 | −0.031 |
| | 322.95 | 1.43 | 1.817 | −0.023 |
| | 327.90 | 1.69 | 1.944 | −0.024 |
| | 332.85 | 2.05 | 2.012 | 0.007 |
| | 337.85 | 2.43 | 2.120 | 0.018 |
| | 342.80 | 2.92 | 2.181 | 0.049 |
| 0.8043 | 293.30 | 0.759 | 0.712 | 0.021 |
| | 298.30 | 0.982 | 0.733 | −0.001 |
| | 303.20 | 1.29 | 0.732 | 0.007 |
| | 308.35 | 1.71 | 0.729 | 0.019 |
| | 313.05 | 2.11 | 0.752 | −0.007 |
| | 317.95 | 2.70 | 0.752 | −0.000 |
| | 322.95 | 3.16 | 0.822 | −0.086 |
| | 327.85 | 4.16 | 0.787 | −0.033 |
| | 332.90 | 5.47 | 0.754 | 0.015 |
| | 337.90 | 6.94 | 0.743 | 0.036 |
| | 342.85 | 8.46 | 0.755 | 0.026 |

$$\mathrm{RSD} = \left[\frac{1}{N}\sum_{i=1}^{N}\left(\frac{x_i - x_i^{\mathrm{calcd}}}{x_i}\right)^2\right]^{1/2} \tag{4}$$

where calcd stands for the calculated values and $N$ is the number of experimental points. The results show that eq 3 can be used

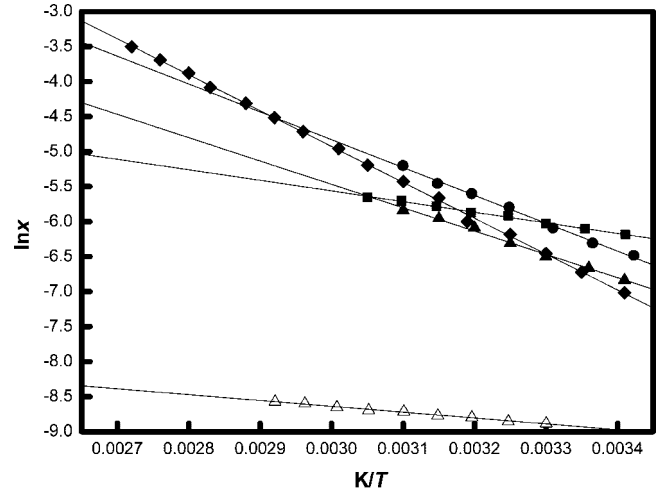



**Figure 4.** Mole fraction solubilities of ODOPB in: ♦, 2-ethoxyethanol; ●, acetone; ■, ethanol; ▲, methanol; Δ, water.

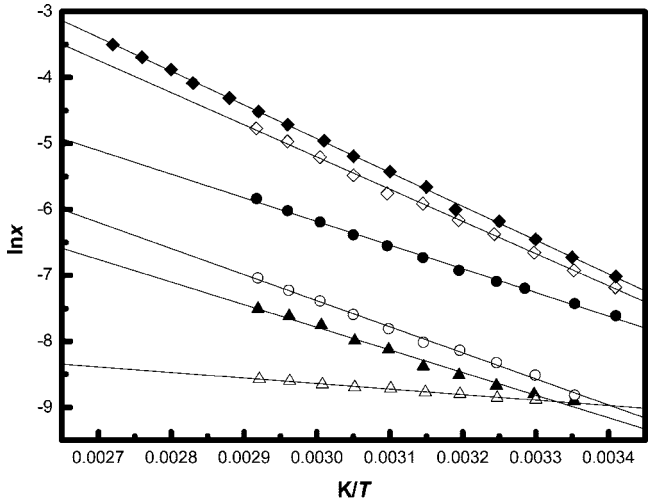



**Figure 5.** Mole fraction solubilities of ODPB in $w$ 2-ethoxyethanol + (1−$w$) water: Δ, $w = 0$; ▲, $w = 0.1983$; ○, $w = 0.4012$; ●, $w = 0.6032$; ◇, $w = 0.8043$; ♦, $w = 1$; -, solubility curve calculated from eq 3.

**Table 4. Parameters of Equation 3 and Root-Mean-Square Deviations of the Measured Solubility Calculated from Equation 4 for $w$ 2-Ethoxyethanol + (1−$w$) Water, Acetone, Methanol, and Ethanol**

| solvent | $A$ | $B$ | RSD |
|---|---|---|---|
| $w = 0$ | −6.1383 | −833.73 | 0.008 |
| $w = 0.1983$ | 2.5027 | −3429.7 | 0.053 |
| $w = 0.4012$ | 4.4668 | −3950.0 | 0.033 |
| $w = 0.6032$ | 4.5755 | −3586.2 | 0.030 |
| $w = 0.8043$ | 9.4403 | −4881.7 | 0.032 |
| $w = 1$ | 10.443 | −5123.6 | 0.021 |
| acetone | 7.1031 | −3977.8 | 0.027 |
| methanol | 4.5352 | −3334.6 | 0.027 |
| ethanol | −1.0203 | −1514.4 | 0.011 |

to correlate the solubility data. Within the temperature range of the measurements, the solubilities of ODOPB in all of the investigated solvents increased with an increase in temperature. The solubility of ODOPB in water shows the lowest value and in acetone shows the highest value from (307.85 to 322.6) K. This result is similar to that for the solubilities of 2-(6-oxido-6*H*-dibenz[*c*,*e*][1,2]oxaphosphorin-6-yl)-methanol (ODOPM) obtained by Wang et al.[16]

In the preparation of ODOPB, an excess amount of *p*-benzoquinone (as compared with the stoichiometric amount) is needed. The *p*-benzoquinone is the most likely impurity in the reaction mixture. The separation of the *p*-benzoquinone from ODOPB can be achieved by washing the reaction mixture with warm water. The solubility data of ODOPB and *p*-benzoquinone (the mass in 100 g of solvent), $c$, in the water are plotted vs temperature in Figure 6. From Figure 6, it can be shown that the evidence of the first stage of purification by using water is obvious. However, because of its higher boiling point and good dissolubility with ODOPB, 2-ethoxyethanol is recommended as the best solvent for the recrystallization of ODOPB, as the second stage of purification. For the final stage of purification, water is recommended as the solvent to remove the 2-ethoxyethanol from the slurry by quickly filtrating and drying.

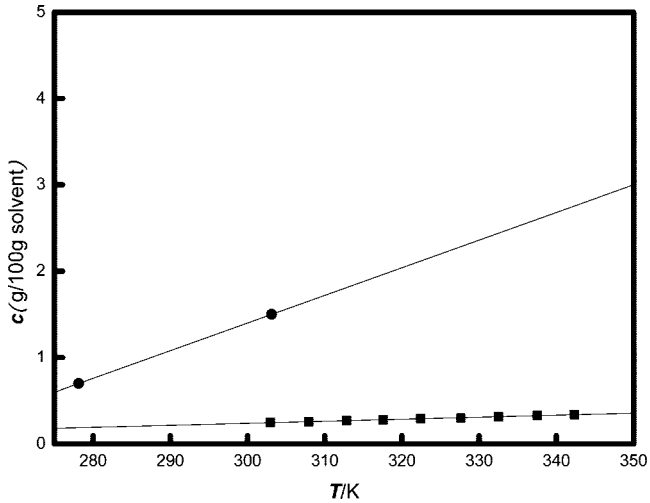



**Figure 6.** Solubility data of ODOPB ■, and *p*-benzoquinone ●, in water. ●, data from ref 17; ■, experimental data.

To obtain the activity coefficients of ODOPB in the solvents from the experimental data, the following equilibrium equation for solute 1 was derived as a fair approximation[18]

$$\ln \frac{1}{x_1 \gamma_1} = \frac{\Delta H_f}{RT_m}\left(\frac{T_m}{T} - 1\right) \quad (5)$$

where $\Delta H_f$ refers to the enthalpy of fusion; $T_m$ is the melting temperature; $R$ is the gas constant; and $x_1$ and $\gamma_1$ refer to the mole fraction and activity coefficient of solute in the solution, respectively. With the experimental $x_1$, $T$, $\Delta H_f$, and $T_m$ values known, the activity coefficients of ODOPB in different solvents were obtained. The results are listed in Tables 2 and 3. From Tables 2 and 3, it can be seen that the activity coefficients of ODOPB in methanol, ethanol, acetone, and 2-ethoxyethanol are all less than unity. A relatively higher solubility in those solvents than the ideal behavior which corresponds to a polar or specific chemical force is important.

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