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Solubilities of (2,5-Dihydroxyphenyl)diphenyl Phosphine Oxide in Selected Solvents

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A phosphorus-containing flame retardant (2,5-dihydroxyphenyl)diphenyl phosphine oxide (HPO) was characterized by a differential scanning calorimeter (DSC), thermogravimetric analysis (TGA), elemental analysis, and nuclear magnetic resonance (^{1}H NMR and ^{31}P NMR). Using a static analytical method, the solubilities of HPO were measured in 12 solvents and correlated with an empirical equation. The estimated uncertainty of all of the solubility values on error analysis and repeated observations was within 2.0 %.

Introduction

Epoxy resins are high-performance thermosetting resins that have excellent properties including high adhesion to many substrates, chemical and heat resistance, low shrinkage on cure, toughness, superior electrical and mechanical properties, and good dimensional stability.[1−3] Furthermore, almost any property of epoxy can be modified to meet a specific need. To extend the applications of epoxy resins as electronic materials and in the aerospace industry, it is crucial to improve their thermal and flame resistance. Several approaches have been reported in the literature for the improvement of flame resistance. With safety and environmental concerns in mind, epoxy resins that are flame-resistant and halogen-free have been the focus of attention by researchers in recent years.[4,5] Among nonhalogenated flame retardants, phosphorus-containing compounds are attractive owing to their low generation of smoke and toxic gases and the high flame-retardant efficiency.[6]

The most effective way to improve flame retardancy in epoxy resin is the reactive approach: incorporating phosphorus-containing chemical units into the polymer backbone or side chain. Thus, while flame retardancy is increased, the original physical and mechanical properties are maintained. Organophosphorus flame retardants such as 2-(6-oxido-6H-dibenz [*c*,*e*][1,2]oxaphosphorin-6-yl)-1,4-dihydroxyphenylene (ODOPB; its formula is shown in Figure 1) have been widely used for this purpose. 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 but also a higher thermal stability and glass transition temperature.[7]

An aromatic phosphorus-containing diol is (2,5-dihydroxyphenyl)diphenyl phosphine oxide (here after abbreviated as HPO,[8,9] and its formula is shown in Figure 1) has a CAS RN 13291-46-8. It is well-known that these reactive additives are useful for modifying epoxy resins. HPO was prepared by the reaction of diphenylphosphine oxide (DPPO) and excessive *p*-benzoquinone in toluene. The ODOPB was prepared via a similar process by reaction of 9,10-dihydro-9-oxa-10-phosphaphenanthrene-10-oxide (DOPO) and excessive *p*-benzoquinone. However, DOPO is more expensive than DPPO.

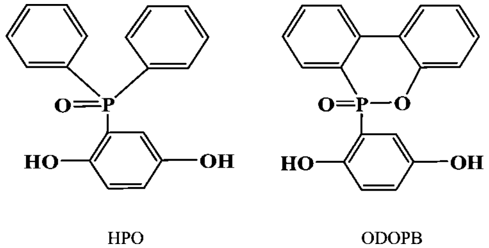

Figure 1. Structures of (2,5-dihydroxyphenyl)diphenyl phosphine oxide (HPO) and 2-(6-oxido-6*H*-dibenz[*c*,*e*][1,2]oxaphosphorin-6-yl)-1,4-dihydroxyphenylene (ODOPB).

Both ODOPB and HPO were obtained by a subsequent crystallization upon cooling the reaction mixture and washing the filter cake with ethanol.[8] The solubility data of ODOPB in selected solvents were reported by Fan et al.[10] Knowledge of solubilities of HPO in toluene and ethanol (as well as other common solvents such as methanol, 2-ethoxyethanol, and acetone) as a function of temperature is necessary for subsequent purification.

In our continuous efforts to search for high thermally stable flame-retardant polyesters, HPO was synthesized and characterized. The solubility data of HPO in 2-ethoxyethanol, methanol, ethanol, toluene, and acetone were measured. In the purification of HPO, it was found that the solid can be washed by water instead of toluene to remove the unreacted *p*-benzoquinone. The solubilities of HPO 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. HPO was synthesized and provided by Shandong Wan Zhao Co., Ltd. All of the chemicals 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 0.99. The water was deionized before use.

Apparatus and Procedure. The melting temperature 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

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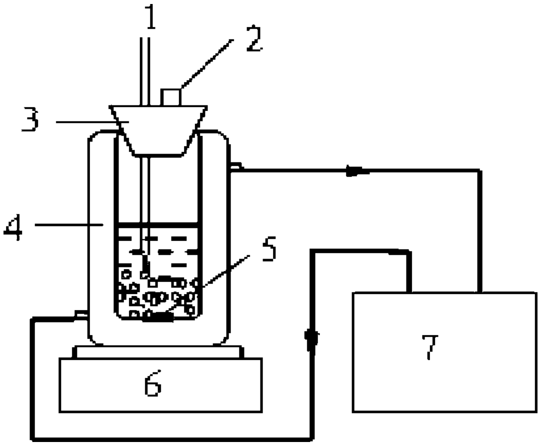

Figure 2. Schematic diagram of the experimental apparatus: 1, thermocouple; 2, sample gauge; 3, rubber plug; 4, jacket; 5, equilibrium cell; 6, magnetic stirrer; 7, water cycling bath.

Table 1. Mass Fraction Purity (ω), Density (ρ), and Refractive Index (n_D) for the Organic Solvents Used in This Work at T = 293.15 K

solvent	100 ω	ρ/g·cm^{-3}	lit.[16] ρ/g·cm^{-3}	n_D	lit.[16] n_D
methanol	99.5	0.792	0.79104	1.3301	1.32840
ethanol	99.7	0.790	0.78920	1.3660	1.36143
2-ethoxyethanol	99.5	0.929	0.92945	1.4065	1.4077
toluene	99.5	0.866	0.86683	1.4967	1.49693
acetone	99.5	0.790	0.78998	1.3590	1.35868

performed on an Elementar Vario EL element analyzer, and ^{1}H NMR and ^{31}P NMR spectra were obtained with a Bruker ARX-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.

The setup for the solubility measurement was the same as that described in the literature.[11,12] Figure 2 shows the schematic diagram of the experimental apparatus. A jacketed equilibrium cell was used for the solubility measurement with a working volume of 120 mL and a magnetic stirrer, and 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.

Characterization of HPO. ^{1}H NMR (DMSO-d_6), δ (ppm): 6.73 (d, 1H), 6.87 (d, 1H), 6.95 (d, 1H), 7.50 (m, 4H), 7.58 (m, 2H) 7.73 (m, 4H), 9.13 (s, 1H), 9.78 (s, 1H), [lit.[9] ^{1}H NMR (500 MHz, CDCl$_3$) δ (ppm): 5.05 (s 1H,), 6.43 (d, 1H), 6.85 (d, 1H), 6.91 (d, 1H), 7.44 to 7.50 (m, 4H), 7.55 to 7.60 (m, 2H), 7.63 to 7.69 (m, 4H), 10.51 (s, 1H); lit.[13] ^{1}H NMR (CD$_3$OD/TMS, δ (ppm): 5.3 (s, 2H), 6.72 (d, 1H), 6.87 (d, 1H), 6.92 (d, 1H), 7.52 (m, 4H), 7.57 (m, 2H), 7.70 (m, 4H)], ^{31}P NMR (DMSO-d_6): $\delta = 33.99$ ppm [lit.[9] ^{1}H NMR (500 MHz, CDCl$_3$) (101.3 MHz MeOH, external D$_2$O lock) $\delta = 30.1$ ppm; lit.[13] ^{31}P NMR (CD$_3$OD/H$_3$PO$_4$) $\delta = 34.09$ ppm]. Elemental analysis (%, calcd): *C*, 69.06 % (69.7 %); *H*, 5.01 % (4.9 %). On the basis of the above analysis, the mass fraction purity of HPO used in this work was higher than 0.99. The results of DSC and TGA measurements of HPO are shown in Figures 3 and 4. The melting temperature of HPO was 487.75 K (lit.[9,13] (487.15 to 489.15) K; lit.[8] (489.15 to 491.15) K). The enthalpy of fusion of HPO was 37.26 kJ·mol^{-1}. TGA results show that there is a single-step decomposition, and no or very little residue remains for HPO.

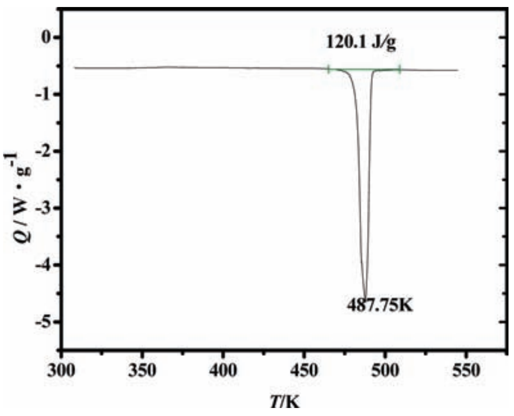

Figure 3. Experimental heat Q flow from differential scanning calorimeter (DSC) measurement of HPO.

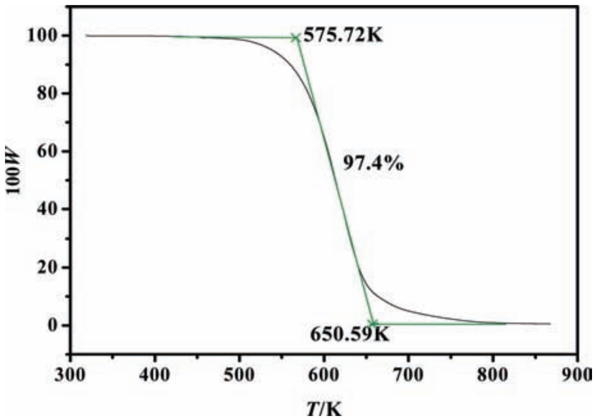

Figure 4. Experimental heat flow from thermogravimetric analysis (TGA) measurement of HPO in flowing nitrogen.

Solubility Measurement. The solubilities were measured by a gravimetric method.[11] For each measurement, an excess mass of HPO 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 a constant value, and then the actual value of temperature was recorded), the stirring was stopped, and the solution was kept still until it was clear. A preheated on−off 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 uncovered 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[11]

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

where M_1 is the molar mass of HPO and M_2 is the molar mass of solvent.

$$x = \frac{(m_2 - m_0)/M_1}{(m_2 - m_0)/M_1 + (m_1 - m_2)w_1/M_2 + (m_1 - m_2)(1 - w_2)/M_3} \tag{2}$$

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

Table 2. Mole Fraction Solubilities (x) and Activity Coefficients (γ) of HPO in the Selected Solvents

solvent	T/K	x	γ	$(x - x^{\mathrm{calcd}})/x$
toluene	293.96	0.00071	3.2968	−0.00266
	298.25	0.00075	3.8863	0.00552
	303.47	0.00078	4.8388	−0.00296
	308.24	0.00082	5.7846	0.00416
	313.45	0.00086	7.0233	−0.00201
	318.43	0.00089	8.4871	−0.00478
	323.38	0.00094	9.9675	−0.00224
	328.45	0.00099	11.721	0.00487
acetone	299.45	0.00175	1.7689	0.01143
	303.26	0.00188	1.9872	−0.00328
	308.24	0.00216	2.1961	0.00354
	313.47	0.00236	2.5617	−0.01521
	318.13	0.00262	2.8450	−0.02038
	323.26	0.00296	3.1491	−0.00798
	328.25	0.00332	3.4663	0.02099
methanol	294.16	0.00625	0.3784	0.00501
	298.35	0.00707	0.4143	0.00336
	303.87	0.00814	0.4727	−0.01369
	308.85	0.00946	0.5160	−0.00136
	313.34	0.01082	0.5554	0.01083
	318.15	0.01232	0.6056	0.01527
	323.56	0.01383	0.6827	−0.00539
ethanol	293.95	0.00571	0.4097	−0.01267
	298.26	0.00634	0.4599	−0.00522
	303.44	0.00721	0.5227	0.00802
	308.26	0.00787	0.6033	−0.00722
	313.45	0.00903	0.6689	0.02267
	318.47	0.00979	0.7729	0.00469
	323.35	0.01093	0.8561	0.01838
	328.43	0.01168	0.9927	−0.01055
	333.35	0.01267	1.11937	−0.01892
water	297.16	0.00011	25.076	−0.01138
	308.15	0.00012	39.361	−0.01069
	313.34	0.00013	46.229	0.00873
	318.15	0.00014	53.289	0.00175
	323.47	0.00014	67.183	0.00339
	328.46	0.00015	77.394	0.01142
	333.25	0.00015	94.163	0.00721
	338.17	0.00016	107.36	0.00342
	343.25	0.00017	122.94	0.00607
	348.36	0.00017	148.88	0.00031
	353.14	0.00018	167.36	−0.00381
	358.13	0.00018	199.73	−0.00906
	363.27	0.00019	225.87	−0.00039
	368.26	0.00019	266.97	−0.00734
2-ethoxyethanol	299.43	0.01076	0.2874	0.02552
	303.25	0.01131	0.3301	−0.00474
	308.24	0.01241	0.3822	−0.01355
	313.46	0.01367	0.4421	−0.01885
	318.15	0.01544	0.4832	0.01363
	323.27	0.01681	0.5547	0.00372
	328.26	0.01803	0.6385	−0.015132
	333.45	0.01983	0.7181	−0.01016
	338.75	0.02229	0.7883	0.01736
	343.15	0.02356	0.8836	0.00073
	348.15	0.02521	0.9962	−0.01144
	353.14	0.02786	1.0813	0.01126
	358.06	0.02955	1.2136	−0.00341
	363.15	0.03158	1.3534	−0.01142
	368.16	0.03482	1.4519	0.80669

Different dissolution times were tested to determine a suitable equilibrium time. It was found that 2 h was enough to reach equilibrium. During our experiments, three parallel measurements were performed at the same composition of solvent for each temperature, and an average value is given. The maximum RSD of each triplicate data is 0.24 %, and the minimum is 0.12 %. The estimated relative uncertainty of the mole fraction solubility values based on error analysis and repeated observations was within 0.02.

Results and Discussion

The mole fraction solubilities x of HPO in selected solvents are summarized in Tables 2 and 3 and plotted as ln x versus T in Figures 5 and 6. 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 relative standard deviations (RSD), defined by eq 4, are also presented in Table 4. The smoothed data calculated from eq 3 are compared with the data listed in Tables 2 and 3.

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

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

Table 3. Mole Fraction Solubilities (x) and Activity Coefficients (γ) of HPO in w 2-Ethoxyethanol + (1 − w) Water

w	T/K	x	γ	$(x - x^{\mathrm{calcd}})/x$
0.1983	299.85	0.00017	18.576	0.01035
	303.46	0.00018	20.958	−0.02511
	308.14	0.00019	24.847	−0.00269
	313.65	0.00022	27.706	0.00168
	318.37	0.00024	31.389	0.01898
	323.25	0.00027	34.508	0.02158
	328.23	0.00028	41.066	0.00376
	333.55	0.00031	46.117	−0.02053
	338.16	0.00033	52.031	−0.00055
	343.14	0.00036	57.811	−0.00237
	348.15	0.00038	66.091	−0.00489
	353.86	0.00041	75.397	−0.02089
	358.35	0.00044	82.338	0.00021
	363.24	0.00048	89.317	0.00832
	368.35	0.00051	99.756	0.01089
0.4012	298.25	0.00025	11.658	0.00368
	303.36	0.00027	13.904	0.00141
	308.07	0.00029	16.225	−0.02034
	313.44	0.00032	18.866	−0.00097
	318.56	0.00035	21.706	0.01441
	323.15	0.00037	25.074	0.00957
	328.14	0.00040	27.941	0.01965
	333.54	0.00042	34.025	−0.00999
	338.85	0.00044	40.091	−0.02139
	343.16	0.00047	44.313	0.00013
	348.15	0.00051	49.244	−0.00067
	353.34	0.00053	57.249	−0.00153
	358.06	0.00056	64.042	−0.00868
	363.35	0.00061	70.545	0.00896
	368.26	0.00063	80.515	0.00489
0.6032	299.85	0.00119	2.6537	0.01519
	303.46	0.00131	2.8797	0.00987
	308.14	0.00147	3.2116	−0.00046
	313.65	0.00165	3.6941	−0.02246
	318.37	0.00191	3.9442	0.00235
	323.25	0.00214	4.3539	−0.00104
	328.23	0.00237	4.8517	−0.01512
	333.54	0.00269	5.3125	−0.00991
	338.15	0.00298	5.7595	−0.00741
	343.16	0.00339	6.1438	0.01461
	348.15	0.00373	6.7332	0.00457
	353.84	0.00415	7.4435	−0.00239
	358.35	0.00461	7.8588	0.01329
	363.26	0.00501	8.5631	0.00178
	368.35	0.00547	9.3008	−0.00365
0.8043	298.25	0.00519	0.5615	−0.00299
	303.36	0.00571	0.6574	−0.00747
	308.05	0.00659	0.7133	0.03531
	313.44	0.00699	0.8637	−0.02484
	318.55	0.00795	0.9551	0.00520
	323.16	0.00858	1.0817	−0.01987
	328.15	0.00951	1.2051	0.00379
	333.54	0.01068	1.3381	0.01994
	338.85	0.01153	1.5299	−0.00261
	343.17	0.01251	1.6655	−0.00098
	348.13	0.01351	1.8576	−0.00319
	353.35	0.01477	2.0550	0.00616
	358.06	0.01548	2.3168	−0.02682
	363.34	0.01735	2.4794	0.00794
	368.25	0.01831	2.7694	0.00164

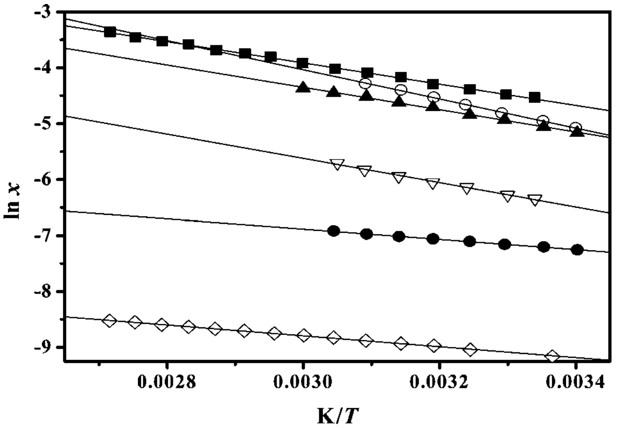

Figure 5. Mole fraction solubilities of HPO in: ■, 2-ethoxyethanol; ○, methanol; ▲, ethanol; ▽, acetone; ●, toluene; ◇, water (experimental); solid line, solubility curve calculated from eq 3.

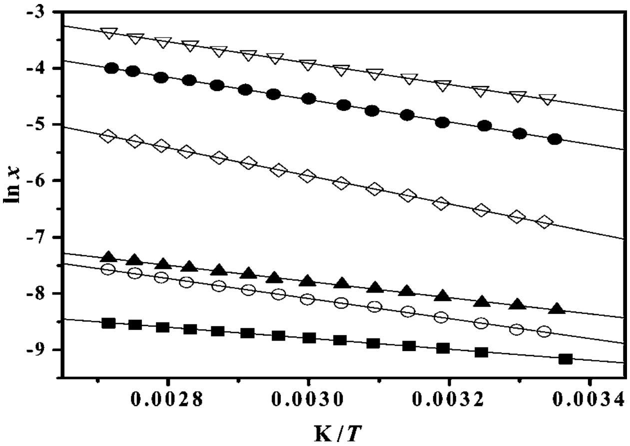

Figure 6. Mole fraction solubilities of HPO in *w* 2-ethoxyethanol + (1 − *w*) water: ▽, $w = 1$; ●, $w = 0.8043$; ◇, $w = 0.6032$; ▲, $w = 0.4012$; ○, $w = 0.1983$; ■, $w = 0$; solid line, solubility curve calculated from eq 3.

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, Toluene, and Ethanol

solvent	*A*	*B*	RSD
$w = 0$	−5.86324	−976.57	0.00773
$w = 0.1983$	−2.74466	−1780.71	0.01428
$w = 0.4012$	−3.46523	−1439.55	0.01193
$w = 0.6032$	1.5485	−2487.64	0.01126
$w = 0.8043$	1.41434	−1991.43	0.01445
$w = 1$	1.79126	−1901.11	0.01437
toluene	−4.14133	−913.61	0.00446
acetone	0.91846	−2179.25	0.01343
methanol	3.77077	−2603.37	0.00986
ethanol	1.64716	−1999.04	0.01538

to correlate the solubility data. Within the temperature range of the measurements, the solubilities of HPO in all of the investigated solvents increased with an increase in temperature. The solubility of HPO in water shows the lowest value and in 2-ethoxyethanol shows the highest value from (299.43 to 368.16) K. This result is similar to that for the solubilities of ODOPB obtained by Fan et al.[10]

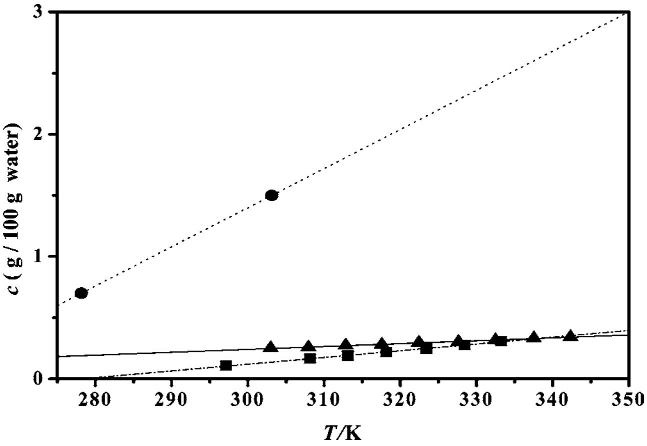

Figure 7. Solubility data of ■, HPO; ▲, ODOPB, and ●, *p*-benzoquinone; ●, dotted line, data from ref 15; ▲, data from ref 10; ■, experimental data obtained in this work; solid line, dashed line, solubility curve calculated from eq 3.

The thermogravimetric physical property of HPO and ODOPB is similar. The thermogravimetric curve of HPO is shown in Figure 4. The initial decomposition temperature of HPO was around $T = 575.72$ K, and ODOPB[10] was around $T = 605.9$ K. The temperature at 97.4 % mass loss was $T = 650.59$ K, and the temperature of ODOPB[10] at 93.4 % mass loss was $T =$ 658.2 K. Figure 3 shows the results of the DSC measurement of HPO. The enthalpy of fusion of HPO was 37.26 $\text{kJ}\cdot\text{mol}^{-1}$, and ODOPB[10] was 41.70 $\text{kJ}\cdot\text{mol}^{-1}$. The melting temperature of HPO was 487.75 K; ODOPB[10] was $T = 524.07$ K. The results indicate that the HPO has better flame retardancy for epoxy resin like the ODOPB.

In the preparation of HPO, 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 HPO can be achieved by washing the reaction mixture with warm water. The solubility data of HPO and *p*-benzoquinone (the mass in 100 g of solvent), *c*, in the water are plotted versus *T* in Figure 7. In Figure 7, it can be shown that evidence of using water in the first stage of purification for HPO is obvious. The solubility data of HPO in water from (303.00 to 342.35) K is lower than those of ODOPB derived from the literature data[10] for the sake of comparison; 2-ethoxyethanol is recommended as the best solvent for the recrystallization of HPO as the second stage of purification because of its higher boiling temperature and good dissolubility with HPO. 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.

To obtain the activity coefficients of HPO in the solvents from the experimental data, the following equilibrium equation for solute 1 was derived as a fair approximation[14]

$$\ln \frac{1}{x_1 \gamma_1} = \frac{\Delta H_{\text{fus}}}{R T_{\text{m}}} \left(\frac{T_{\text{m}}}{T} - 1 \right) \tag{5}$$

where $\Delta_{\text{fus}}H$ refers to the enthalpy of fusion, T_{m} is the melting temperature, R is the gas constant, and x_1 and γ_1 refer to the mole fraction and activity coefficient of solute in the solution, respectively. With the experimental x_1, T, $\Delta_{\text{fus}}H$, and T_{m} values known, the activity coefficients of HPO 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 HPO in 2-ethoxyethanol, methanol, ethanol, and acetone 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.

Supporting Information Available:

^{1}H NMR and ^{31}P NMR spectra of HPO information. This material is available free of charge via the Internet at http://pubs.acs.org.

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