Solubilities of Organoboron Compounds in Organic Solvents. 5. Solid−Liquid Equilibria of Some Pyrazaboles in Hexanols

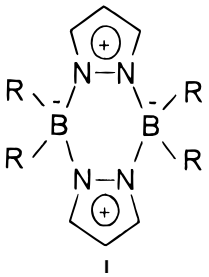
M. Dąbrowski,* J. Serwatowski, and A. Sporzyński

Faculty of Chemistry, Warsaw University of Technology (Politechnika), Noakowskiego 3, 00-664 Warsaw, Poland

The solubilities of some pyrazaboles (pyrazabole, 4,4,8,8-tetraethylpyrazabole, and 4,4,8,8-tetrapropylpyrazabole) in hexanols (1-hexanol, 2-methyl-1-pentanol, 2-ethyl-1-butanol, 4-methyl-2-pentanol, 3-methyl-3-pentanol) have been determined experimentally by a dynamic method at temperatures ranging from the boiling point of the solvent to 273 K. The results have been correlated by the Wilson and UNIQUAC ASM equations. The existence of solid−solid first-order phase transition in tetraethylpyrazabole has been taken into consideration in the solubility curve calculations. The average root mean square deviations of the solubility temperatures for all the measured data vary from (0.52 to 3.54) K and depend on the particular equation used.

Introduction

The present work is a continuation of systematic studies on solid−liquid equilibria of the systems containing organoboron compounds. Pyrazaboles (**I**), the compounds possessing four-coordinated boron atom in a heteroaromatic system that causes high chemical stability (Niedenzu and Trofimenko, 1986), were chosen for investigation regarding their interesting properties. Selected pyrazaboles were found to form liquid crystals (Barberá *et al.*, 1994); the others can be fragments of the molecules used in supramolecular chemistry (Jäkle *et al.*, 1996).



I

The aim of the present work was to study the solubility of three selected pyrazaboles in a series of isomeric hexanols to investigate the influence of the steric hindrance on solubility and attempt a correlation of the experimental results with established theories of solutions. The phase transition, with high enthalpy of transformation, ΔH_{trs1}, 28.61 kJ mol^{-1} at the temperature $T_{trs1} = (342.30 \pm 0.15)$ K, for 4,4,8,8-tetraethylpyrazabole (TEP) was observed. Recently, a polymorphic transition in the solid state has been discovered as a solid−solid phase transition below the melting point.

Experimental Section

Materials. Pyrazabole (PYR), tetraethylpyrazabole (4,4,8,8-tetraethylpyrazabole) (TEP), and tetrapropylpyrazabole (4,4,8,8-tetrapropylpyrazabole) (TPP) were synthesized by Niedenzu. The solvents were dried over 4A molecular sieves and were fractionally distilled using a 35-plate laboratory column. The characteristics of the solutes and solvents are listed in Table 1. Solubilities were determined by a dynamic (synthetic) method, described in the first paper in this series (Domańska *et al.*, 1993). The reproducibility of the measurements was 0.1 K, which corresponded to a standard error in mole fraction δx_1 of 0.0005. All the experimental data (except in hexanol, published in a previous paper, Dąbrowski *et al.*, 1996) are shown in Tables 2−4.

* To whom correspondence should be addressed. E-mail: marek@ch.pw.edu.pl.

Results and Discussion

The solubility of PYR, TEP and TPP in each solvent was lower than expected from ideal solution behavior, and the solution showed positive deviations from ideality ($\gamma_1 \gg 1$). The solubilities of the solute were close to ideal values in the order PYR < TEP < TPP.

All experimental activity coefficients are listed in Tables 2−4, and the solid−liquid equilibrium data are shown in comparison with the ideal solubility in Figures 1, 2, and 3.

For all investigated pyrazaboles, solubility decreases in order ternary alcohol > secondary alcohol ≥ primary alcohol. The best solubility is observed in the alcohol with a hindered hydroxyl group (3-methyl-3-pentanol). However, differences are small, and solubilities in investigated alcohols are close to each other. These results are in agreement with previous observations and lead to the conclusion that pyrazaboles are not able to form intermolecular hydrogen bonds or stable complexes with polar solvents.

The solubility of a solid nonelectrolyte 1 in a liquid solvent can be expressed as

$$-\ln x_1 = \frac{\Delta_{fus}H_1}{R}\left(\frac{1}{T} - \frac{1}{T_{fus1}}\right) + \ln \gamma_1 \qquad (1)$$

where x_1 is the mole fraction, γ_1 is the activity coefficient, $\Delta_{fus}H_1$ is the enthalpy of fusion, T_{fus1} is the melting temperature, and T is the equilibrium temperature of the solute. If the solid−solid transition occurs before fusion, an additional term must be added to the right-hand side of eq 1

$$-\ln x_1 = \frac{\Delta_{fus}H_1}{R}\left(\frac{1}{T} - \frac{1}{T_{fus1}}\right) + \frac{\Delta_{trs}H_1}{R}\left(\frac{1}{T} - \frac{1}{T_{trs1}}\right) + \ln \gamma_1 \qquad (2)$$

where $\Delta_{trs}H_1$ and T_{trs1} are the enthalpy and temperature of the solid−solid transition of the solute. Equations 1 and

S0021-9568(97)00090-3 CCC: $14.00

Table 1. Physical Properties of the Pure Components at 298.15 K

		n_D[b]				
	V_m°[a,c]/cm^3 mol^{-1})	exptl	lit.[c]	T_{m1}[d]/K	ΔH_{tr1}[d]/kJ mol^{-1}	ΔH_{m1}[d]/kJ mol^{-1}
PYR	170.0[d]			354.25		11.83
TEP	287.0[d]			379.15	28.61	3.22
TPP	345.0[d]			382.15		33.00
hexanol	125.23	1.415 94	1.4161			
2-ethyl-butan-1-ol	123.18	1.419 75	1.4205			
2-methyl-pentan-1-ol	124.51	1.416 77	1.4172			
4-methyl-pentan-2-ol	127.15	1.408 99	1.4090			
3-methyl-pentan-3-ol	124.70	1.416 41	1.418[e]			

[a] V_m° is the molar volume. [b] n_D is the refractive index. [c] Riddick and Bunger (1986). [d] Domańska *et al.* (1993). [e] At 294.15 K; Pollock and Stevens (1965).

Table 2. Experimental Mole Fraction Solubilities and Activity Coefficients of PYR

x_1	$T_{1/K}$	γ_1	x_1	$T_{1/K}$	γ_1
		2-Ethyl-butan-1-ol			
0.0138	277.85	24.015	0.3569	328.15	2.036
0.0332	289.05	12.173	0.3862	329.55	1.916
0.0517	298.05	9.070	0.4322	332.05	1.769
0.0728	304.05	7.077	0.5012	333.45	1.553
0.0909	308.25	6.041	0.5606	336.15	1.437
0.1074	311.25	5.346	0.6001	337.25	1.361
0.1349	315.05	4.497	0.6890	340.75	1.238
0.1597	317.25	3.920	0.7483	343.15	1.174
0.1842	319.85	3.525	0.8214	345.85	1.104
0.2101	320.95	3.138	0.8665	348.15	1.076
0.2416	322.95	2.804	0.9285	350.55	1.032
0.2722	324.35	2.537	1.0000	354.25	1.000
0.3129	326.75	2.279			
		2-Methyl-pentan-1-ol			
0.0203	275.55	15.642	0.4854	335.25	1.641
0.0231	280.05	14.936	0.5265	336.35	1.534
0.0379	290.15	10.864	0.5780	337.15	1.411
0.0564	299.05	8.448	0.6333	339.45	1.325
0.0836	308.05	6.549	0.6935	343.35	1.269
0.1139	311.75	5.078	0.7303	344.15	1.217
0.1583	317.55	3.971	0.7875	345.35	1.145
0.1949	320.05	3.340	0.8482	347.55	1.091
0.2426	323.75	2.823	0.9005	349.75	1.055
0.2922	325.65	2.405	0.9496	351.95	1.026
0.3233	327.35	2.224	1.0000	354.25	1.000
0.3747	330.85	2.009			
		4-Methyl-pentan-2-ol			
0.0136	277.45	24.189	0.3690	330.85	2.040
0.0161	283.85	22.937	0.4430	332.95	1.746
0.0344	298.55	13.741	0.5207	335.35	1.531
0.0782	308.15	7.012	0.5963	337.65	1.377
0.1173	314.25	5.113	0.6701	340.45	1.268
0.1453	317.35	4.314	0.7380	342.95	1.187
0.1856	321.55	3.581	0.8211	346.65	1.115
0.2290	324.95	3.040	0.8581	348.65	1.093
0.2673	326.85	2.672	0.9231	350.85	1.042
0.3125	328.45	2.334	1.0000	354.25	1.000
		3-Methyl-pentan-3-ol			
0.0207	279.35	16.456	0.4991	329.35	1.479
0.0310	284.85	12.124	0.5451	331.95	1.401
0.0563	294.05	7.805	0.6039	334.35	1.304
0.0941	301.35	5.251	0.6675	336.25	1.208
0.1301	305.95	4.077	0.7263	340.15	1.166
0.1675	311.05	3.418	0.7583	340.35	1.119
0.2309	315.05	2.627	0.7842	342.65	1.113
0.2942	318.75	2.173	0.8185	345.05	1.098
0.3240	320.95	2.035	0.8344	345.85	1.087
0.3580	322.25	1.875	0.9052	349.95	1.052
0.3967	324.35	1.741	0.9253	350.65	1.037
0.4402	326.45	1.614	1.0000	354.25	1.000

2 are the simplified versions of the solubility equations, missing the terms containing ΔC_p, because sufficiently accurate thermodynamic data are not available for pyrazaboles. For the systems investigated, eq 1 was applied for temperatures above the temperature of transition and eq 2 at lower temperatures. Experimental values

Table 3. Experimental Mole Fraction Solubilities and Activity Coefficients of TEP

x_1	$T_{\beta 1}$/K	γ_1	x_1	$T_{\beta 1}$/K	$T_{\alpha 1}$/K	γ_1
		2-Ethyl-butan-1-ol				
0.0167	275.85	3.642	0.4801	332.35		1.341
0.0183	278.85	3.859	0.5086	332.65		1.279
0.0339	290.75	3.654	0.5394	333.65		1.248
0.0522	298.75	3.376	0.5776	334.35		1.194
0.0714	304.45	3.138	0.6295	335.45		1.138
0.0899	309.85	3.103	0.6756	336.45		1.097
0.1182	313.15	2.688	0.7356	337.55		1.045
0.1452	315.85	2.429	0.7487	337.75		1.034
0.1735	318.55	2.253	0.7916	339.15		1.025
0.2053	321.25	2.106	0.8375	340.35		1.008
0.2463	323.75	1.925	0.8713	341.75		1.014
0.2848	325.45	1.771	0.8815	341.95		1.009
0.3293	327.45	1.645	0.8962		347.95	1.018
0.3669	328.45	1.530	0.9030		351.75	1.023
0.3880	328.95	1.473	0.9255		359.15	1.021
0.4266	331.05	1.442	1.0000		379.15	1.000
		2-Methyl-pentan-1-ol				
0.0169	275.15	3.474	0.4695	332.25		1.366
0.0336	290.35	3.620	0.4970	332.95		1.323
0.0566	301.65	3.522	0.5432	334.05		1.257
0.0760	305.35	3.059	0.5944	334.85		1.180
0.0927	311.35	3.193	0.6304	335.95		1.155
0.1190	313.45	2.701	0.6829	337.15		1.111
0.1488	317.05	2.482	0.7450	338.45		1.064
0.1755	320.25	2.374	0.8194	339.75		1.010
0.2011	321.35	2.158	0.8390	339.95		0.993
0.2396	323.75	1.979	0.8898		347.45	1.024
0.2832	325.95	1.813	0.9125		352.75	1.015
0.3323	328.85	1.714	0.9460		364.45	1.014
0.3792	329.65	1.545	1.0000		379.15	1.000
0.4194	330.65	1.447				
		4-Methyl-pentan-2-ol				
0.0151	278.35	4.563	0.3783	328.25		1.474
0.0235	288.05	4.659	0.4426	330.35		1.357
0.0353	296.05	4.442	0.5189	332.55		1.249
0.0550	301.95	3.670	0.6041	334.25		1.138
0.0804	306.75	3.062	0.6701	335.75		1.080
0.1119	310.75	2.583	0.7357	337.55		1.045
0.1441	314.45	2.319	0.7689	338.35		1.027
0.1803	318.05	2.127	0.8234	340.05		1.015
0.2225	321.25	1.943	0.8672		344.25	1.040
0.2697	323.75	1.758	0.9209		361.95	1.034
0.3275	326.45	1.596	1.0000		379.15	1.000
		3-Methyl-pentan-3-ol				
0.0283	278.45	2.447	0.6437	332.65		1.011
0.0477	288.95	2.392	0.6815	333.85		0.995
0.0775	296.25	2.041	0.7258	335.35		0.983
0.1175	304.55	1.914	0.7822	337.25		0.973
0.1556	309.05	1.736	0.8288	339.05		0.975
0.1920	313.15	1.655	0.8341	339.45		0.982
0.2346	315.95	1.509	0.8693	339.75		0.952
0.2567	316.95	1.433	0.9082		349.35	1.009
0.2935	319.35	1.372	0.9096		351.35	1.014
0.3981	323.95	1.200	0.9305		355.45	1.004
0.4610	326.85	1.150	0.9531		367.05	1.014
0.5309	329.35	1.092	1.0000		379.15	1.000
0.5733	330.45	1.051				

Table 4. Experimental Mole Fraction Solubilities and Activity Coefficients of TPP

x_1	T_1/K	γ_1	x_1	T_1/K	γ_1
		2-Ethyl-butan-1-ol			
0.0085	277.55	2.348	0.2362	348.75	1.566
0.0103	280.55	2.258	0.2763	352.25	1.499
0.0176	290.95	2.191	0.3190	354.95	1.414
0.0257	299.45	2.210	0.3515	357.45	1.388
0.0381	310.15	2.355	0.4086	361.45	1.350
0.0523	317.65	2.321	0.4689	364.85	1.303
0.0724	324.35	2.170	0.5050	366.65	1.277
0.0947	329.25	1.990	0.5456	367.95	1.228
0.1148	333.25	1.898	0.5960	369.35	1.171
0.1569	340.05	1.762	0.6420	371.05	1.142
0.1791	341.95	1.647	1.0000	382.15	1.000
0.2070	345.95	1.629			
		2-Methyl-pentan-1-ol			
0.0123	280.05	1.843	0.2833	354.05	1.548
0.0153	288.65	2.261	0.3097	355.65	1.489
0.0275	302.65	2.376	0.3382	357.15	1.429
0.0372	309.05	2.304	0.3809	359.55	1.367
0.0544	318.35	2.293	0.4265	361.55	1.297
0.0998	331.55	2.053	0.4939	364.35	1.219
0.1340	338.75	1.972	0.5590	367.25	1.174
0.1769	345.35	1.869	0.6119	369.85	1.157
0.2110	348.35	1.730	1.0000	382.15	1.000
0.2478	351.05	1.608			
		4-Methyl-pentan-2-ol			
0.0068	274.95	2.564	0.1957	343.35	1.580
0.0096	282.05	2.612	0.2419	347.25	1.456
0.0148	290.95	2.605	0.2936	351.35	1.370
0.0224	298.85	2.469	0.3535	356.35	1.334
0.0330	306.45	2.329	0.4193	360.45	1.276
0.0474	313.85	2.201	0.4658	362.35	1.217
0.0668	320.85	2.058	0.5512	366.55	1.166
0.0896	327.35	1.962	0.6023	368.55	1.132
0.1128	331.95	1.843	0.6474	370.45	1.113
0.1351	336.05	1.781	1.0000	382.15	1.000
0.1640	339.45	1.651			
		3-Methyl-pentan-3-ol			
0.0154	279.05	1.399	0.2554	341.65	1.143
0.0282	289.95	1.304	0.2924	345.75	1.146
0.0425	299.15	1.319	0.3442	350.75	1.147
0.0583	305.05	1.242	0.3826	354.35	1.157
0.0757	311.55	1.255	0.4455	358.95	1.147
0.0958	317.35	1.252	0.5156	362.95	1.120
0.1198	322.45	1.220	0.5581	363.85	1.063
0.1406	326.55	1.213	0.5965	366.45	1.074
0.1746	332.05	1.195	0.6697	370.25	1.069
0.1979	334.85	1.165	1.0000	382.15	1.000
0.2282	338.65	1.154			

of the temperatures of the solid−solid phase transition were determined from solubility curves and DSC measurements (Table 1). Two methods were chosen on the basis of earlier results for pyrazaboles (Domańska *et al.*, 1993) to represent the solute activity coefficients (γ_1) from the so-called correlation equations describing the excess Gibbs free energy of mixing (G^E): the Wilson equation and the UNIQUAC associated-solution model. The exact mathematical forms of the equations are reported by Domańska *et al.*, 1989. The calculations with the UNIQUAC associated-solution model (UNIQUAC ASM) were carried out using a K_2 value as a third adjustable parameter with the hydrogen-bond formation enthalpy $\Delta H°$, −22.4 kJ mol^{-1} according to Nagata (1986). The pure component structural parameters r (volume parameter) and q (surface parameter) were obtained according to Domańska (1990). The temperature dependence of the association constant was calculated from the van't Hoff relation assuming the enthalpy of hydrogen-bond formation to be temperature independent. The parameters of the equations were found by an optimization technique using a maximum neighborhood method for minimization

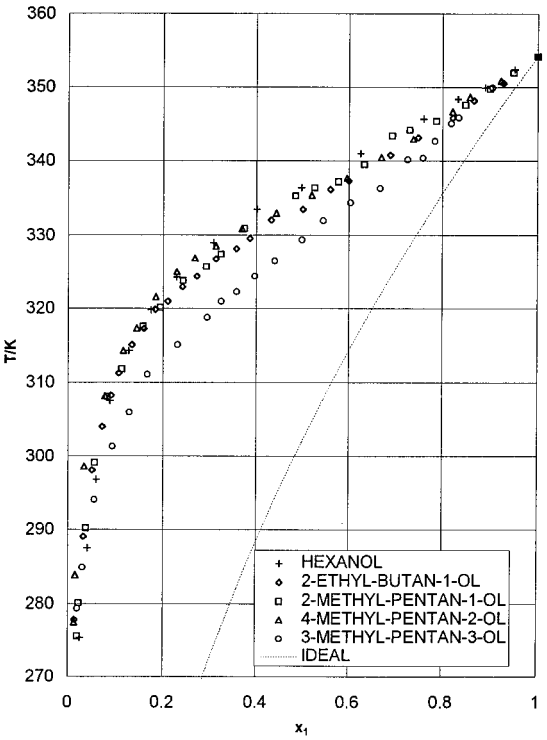


Figure 1. Solubility of PYR in hexanol, 2-methyl-propan-1-ol, 2-ethyl-butan-1-ol, 4-methyl-propan-2-ol, and 3-methyl-propan-3-ol. The dotted line represents the ideal solubility.

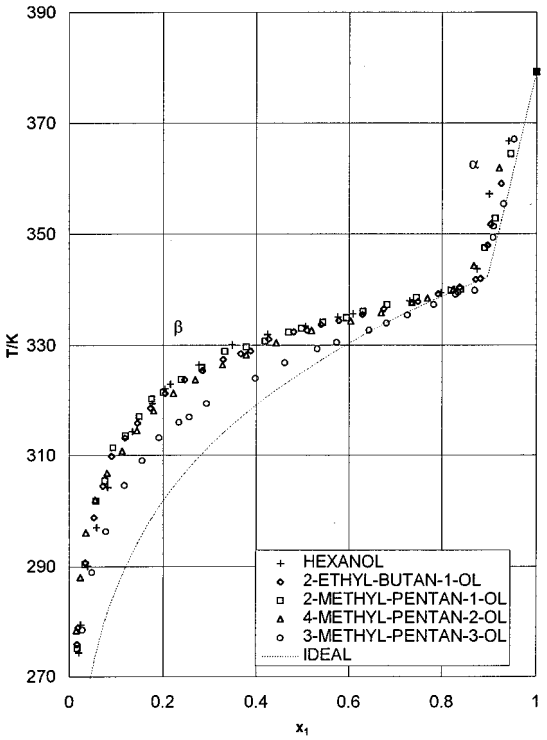


Figure 2. Solubility of TEP in hexanol, 2-methyl-propan-2-ol, 2-ethyl-butan-1-ol, 4-methyl-propan-2-ol, and 3-methyl-propan-3-ol. The dotted line represents the ideal solubility.

$$\Omega = \sum_{i=1}^{n} [T_i^{\text{exp}} - T_i^{\text{cal}}(x_1, P_1, P_2)]^2 \qquad (3)$$

where T_i^{exp} denotes an experimental value of the temperature for a given concentration x_{1i} and T_i^{cal} is the temperature calculated for a given concentration x_{1i} and parameters P_1 and P_2 were obtained by solving the nonlinear equation (eq 1 or 2), depending on the temperature) and

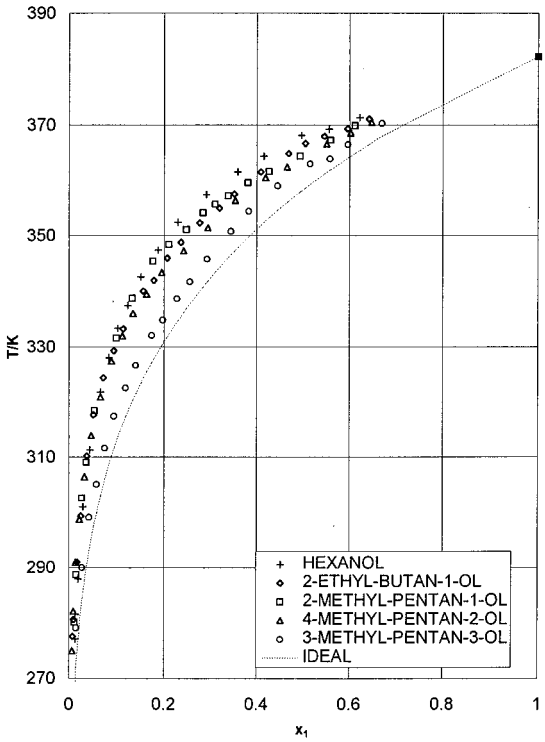


Figure 3. Solubility of TPP in hexanol, 2-methyl-propan-1-ol, 2-ethyl-butan-1-ol, 4-methyl-propan-2-ol, and 3-methyl-propan-3-ol. The dotted line represents the ideal solubility.

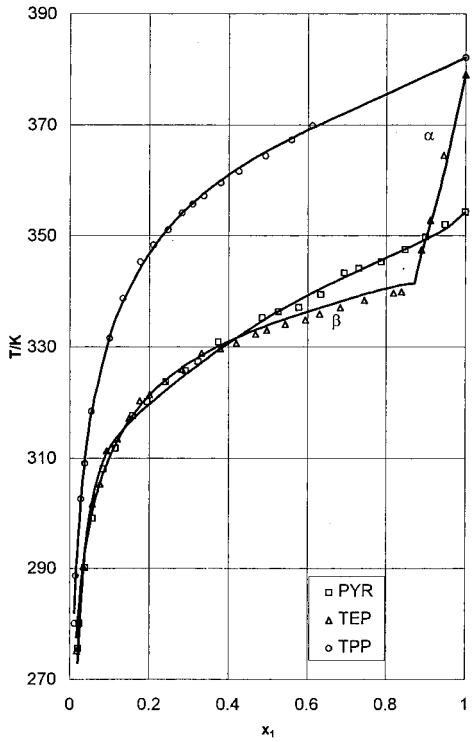


Figure 4. Solubility of PYR , TEP, and TPP in 2-methyl-propan-1-ol. Experimental points are matched by curves calculated by the UNIQUAC ASM KW equation.

Table 5. Analyses of Solubility Data of Pyrazaboles in Five Alcohols by the WILSON Equation, Values of the Parameters and Measures of Deviations (a in kJ mol^{-1}; σ in K)

solvent	PYR	TEP	TPP
hexanol			
$a_{12} \times 10^{-3}$	1.0041	−0.3384	−0.9311
$a_{21} \times 10^{-3}$	1.5987	1.6965	2.4703
σ	2.05	2.69	1.00
2-ethyl-butan-1-ol			
$a_{12} \times 10^{-3}$	1.4505	−0.0810	−0.6143
$a_{21} \times 10^{-3}$	1.1360	1.3149	1.5531
σ	2.95	1.42	1.18
2-methyl-pentan-1-ol			
$a_{12} \times 10^{-3}$	1.2061	−0.1221	−0.6984
$a_{21} \times 10^{-3}$	1.3651	1.4333	1.7586
σ	2.57	1.39	1.09
4-methyl-pentan-2-ol			
$a_{12} \times 10^{-3}$	1.6029	0.2371	−0.3881
$a_{21} \times 10^{-3}$	1.1980	0.9748	1.1909
σ	2.98	2.44	0.87
3-methyl-pentan-3-ol			
$a_{12} \times 10^{-3}$	1.4049	−0.0589	−0.7762
$a_{21} \times 10^{-3}$	0.8732	0.7392	1.0409
σ	3.10	1.57	1.17

the expression for the logarithm of the activity according to the assumed model. The nonlinear equations were solved using the secant method. The root mean square (rms) deviation of the temperatures defined by eq 4 was used as a measure of the goodness of fit

$$\sigma = \left[\sum_{i=1}^{n} \frac{(T_i^{\exp} - T_i^{\mathrm{cal}})^2}{(n-l)}\right]^{1/2} \qquad (4)$$

where n is the number of experimental points (including the melting point) and l is the number of adjustable parameters.

The Wilson equation has been tested with a parameter

Table 6. Analyses of Solubility Data of Pyrazaboles in Five Alcohols by the UNIQUAC ASM KW Equation, Values of the Parameters and Measures of Deviations (Δg in J mol^{-1}; σ in K)

solvent	PYR	TEP	TPP
hexanol			
Δg_{12}	−1707.28	−1753.94	−1720.51
Δg_{21}	3494.46	2472.28	2143.58
k_2	55.3	20.6	25.0
σ	1.21	2.32	0.59
2-ethyl-butan-1-ol			
Δg_{12}	−1815.27	−1333.12	1253.96
Δg_{21}	3967.09	1770.38	−1241.53
k_2	27.9	8.8	18.2
σ	3.54	1.32	0.93
2-methyl-pentan-1-ol			
Δg_{12}	−1808.30	−1354.21	−1929.48
Δg_{21}	3860.91	1794.19	2717.13
k_2	42.4	10.9	14.5
σ	1.50	1.30	0.75
4-methyl-pentan-2-ol			
Δg_{12}	−1901.44	−1921.49	770.38
Δg_{21}	4296.17	3106.72	−857.27
k_2	44.3	10.5	10.7
σ	3.14	2.17	0.52
3-methyl-pentan-3-ol			
Δg_{12}	−1915.60	−1186.01	527.51
Δg_{21}	4202.46	1471.67	−873.58
K_2	17.3	2.2	8.4
σ	1.86	1.64	0.70

Λ_{12} in the form

$$\Lambda_{12} = (V_2/V_1)\exp[-(g_{12} - g_{11})/RT] \qquad (5)$$

where

$$(g_{12} - g_{11}) = \frac{a_{12}}{T}; \quad a_{12} \neq f(T) \qquad (6)$$

V_2, V_1 are the molar volumes of pure solute and solvent in the liquid phase, g_{12} is the molar energy of interaction

between the 1 and 2 components, and a_{12} is the binary interaction parameter.

Table 5 lists the results of fitting the solubility curves by the Wilson equation and Table 6 by the UNIQUAC ASM KW equation. As an example, the results for the systems containing 2-methyl-1-pentanol are shown additionally in Figure 4. For 15 systems the description of the solid−liquid equilibria given by the Wilson equation is with the average r.m.s. deviation 1.9 K.

Better solubility correlation was obtained with UNIQUAC ASM KW. In this case the average rms deviation is reduced to $\sigma = 1.6$ K.

Acknowledgment

The authors thank Prof. K. Niedenzu (University of Kentucky, Dept. of Chemistry, Lexington, KY) for pyrazaboles used in this work.

Literature Cited

Barberá, J.; Giménez, R.; Serrano, J. L. Pyrazaboles: New room temperature columnar liquid crystals. *Adv. Mater.* **1994,** *6*, 470−472.

Domańska, U. Solubility of benzoyl-substituted naphthols in mixtures of hexane and t-butanol. *Ind. Eng. Chem. Res.* **1990,** *29*, 470−475.

Domańska, U.; Domański, K.; Klofutar, K.; Paljk, S. Solubility of tetracosane in aliphatic alcohols. *Fluid Phase Equilib.* **1989,** *46*, 25−39.

Domańska, U.; Serwatowski, J.; Sporzyński, A.; Dąbrowski, M. Solubilities of organoboron compounds in organic solvents. 1. Solid-liquid equilibria of some pyrazaboles + heptane or + 2-propanol. *Thermochim. Acta* **1993**, *222*, 279−290.

Dąbrowski, M.; Serwatowski, J.; Sporzyński, A. Solubilities of organoboron compounds in organic solvents. 4. Solid-liquid equilibria of some pyrazaboles + methanol, ethanol, 2-propanol or t-butanol. *Thermochim. Acta* **1996**, *288*, 191−202.

Jäkle, F.; Peters, F.; Priermeier, T.; Wagner, M. Structural aspects, dynamic behaviour and reactivity of ferrocene-bridged boron-phosphorous adducts. Ninth International Meeting on Boron Chemistry IMEBORON IX, Heidelberg, 1996; Ruprecht-Karls-Universität: Heidelberg, 1996; p 37.

Nagata, I. Thermodynamics of alcohol solutions. Phase equilibria of binary and ternary mixtures containing any number of alcohols. *Thermochim. Acta* **1986**, *107*, 199−217.

Niedenzu, K.; Trofimenko, S. Pyrazabole derivatives of boron. *Top. Curr. Chem.* **1986,** *131*, 1−37.

Pollock, J. R. A.; Stevens, R. *Dictionary of Organic Compounds*, 4th ed.; Eyre and Spottiswoode Publishers Ltd.: London, 1965.

Riddick J. A.; Bunger W. B. *Organic Solvents. Physical Properties and Methods of Purification*, 3rd ed.; John Wiley & Sons: New York, 1970.

Received for review April 17, 1997. Accepted July 8, 1997.[⊗]

JE970090W

[⊗] Abstract published in *Advance ACS Abstracts*, September 1, 1997.