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Isobaric Vapor−Liquid Equilibrium of Binary Mixtures of 1-Butanol + Chlorobenzene and 2-Butanol + Chlorobenzene at 20 and 100 kPa

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Isobaric vapor−liquid equilibria were obtained for 1-butanol + chlorobenzene and for 2-butanol + chlorobenzene systems at 20 and 100 kPa using a dynamic still. The experimental error in temperature was ±0.1 K, in pressure ±0.01 kPa and ±0.1 kPa for the experiments carried out at 20 and 100 kPa, respectively, and in liquid and vapor composition ±0.001. The two systems satisfy the point-to-point thermodynamic consistency test. Both systems show a positive deviation from ideality. The data were correlated with the Wilson equation.

Introduction

Vapor−liquid equilibrium (VLE) data are indispensable in the design of separation processes such as distillation and extractive distillation. Values can be obtained either by experiment or by predictive methods. Among the estimation methods, the most noteworthy are those of group contribution, mainly the UNIFAC method (Fredenslund et al., 1977). This model requires a complete and updated experimental VLE data bank in order to fit the group interaction parameters. There is a lack of VLE measurements for some groups (Gmehling et al., 1993). The present work is part of a project for determining vapor−liquid equilibrium in mixtures in which one component, at least, is an alcohol. On the other hand, another functional group for which more experimental data are desirable is the ACCl group.

Chlorobenzene forms azeotropic mixture with 1-butanol (Arzhanov et al., 1976; Venkateswara Rao et al., 1977; Kormina et al., 1979). Venkateswara Rao et al. (1980) have also studied the 2-butanol + chlorobenzene system, but in this case an azeotropic mixture has not been clearly obtained even though a minimum in temperature has been observed. In this article we report the equilibrium data at 20 and 100 kPa for both systems to study the influence of the pressure on the azeotropic composition.

Experimental Section

Chemicals. All components used were purchased from Aldrich Chemie Co. The purity of all chemicals was checked by gas chromatography (GC) and found to be 1-butanol (99.94 mass %), 2-butanol (99.92 mass %), and chlorobenzene (99.99 mass %). They were used without further purification. The water content was small in all chemicals (<0.05 mass %, checked by GC). The densities of the pure liquids were measured at 298.15 K using an Anton Paar DMA 55 densimeter. The refractive indexes of the pure liquids were measured at 298.15 K in an Abbe refractometer, Atago 3T. The temperature was controlled to ±0.01 K with a thermostated bath. The accuracies in density and refractive index measurements are ±0.01 $\mathrm{kg{\cdot}m^{-3}}$ and ±0.0002, respectively. The experimental values of these properties and the boiling points are given in Table 1 together with those of the literature.

Table 1. Densities *d*, Refractive Indexes *n*, and Boiling Points T_b of the Chemicals

	d(298.15 K)/ $\mathrm{kg{\cdot}m^{-3}}$		n (D, 298.15 K)		T_b (100 kPa)/K	
component	exptl	lit.	exptl	lit.	exptl	lit.
1-butanol	805.70	806.0[a]	1.3972	1.3971[a]	390.25	390.51[a]
2-butanol	802.59	802.6[b]	1.3952	1.3949[b]	371.85	372.34[b]
chlorobenzene	1101.01	1101.1[c]	1.5218	1.52138[c]	404.05	404.42[c]

[a] TRC, 1976, 1966, 1970. [b] TRC, 1965, 1966, 1970. [c] TRC, 1989, 1990, 1990.

Apparatus and Procedure. The equilibrium vessel used in this work was an all-glass, dynamic recirculating still described by Walas (1985), equipped with a Cottrell pump. The still (Labodest model) manufactured by Fischer Labor und Verfahrenstechnik (Germany) is capable of handling pressures P from 0.25 to 400 kPa and temperatures T up to 523.15 K. The Cottrell pump ensures that both liquid and vapor phases are in intimate contact and also in contact with the temperature sensing element. The equilibrium temperature was measured with a digital Fisher thermometer with an accuracy of ±0.1 K, and the pressure with a digital manometer with an accuracy of ±0.01 kPa. The temperature probe was calibrated against the ice and steam points of distilled water. The manometer was calibrated using the vapor pressure data of high-purity hexane (Daubert and Danner, 1995).

In each experiment, the pressure was fixed and the heating and shaking system of the liquid mixture was connected. The still was operated until equilibrium was reached. Equilibrium conditions were assumed when constant temperature and pressure were obtained for 15 min or longer. The experimental error in temperature was ±0.1 K and in pressure was ±0.01 kPa and ±0.1 kPa for the experiments carried out at 20 and 100 kPa, respectively. At this time, samples of liquid and condensate were taken for analysis. The extractions were carried out with special syringes that allowed us to take small volume samples in a system under partial vacuum.

Analysis. Samples of the liquid and condensed vapor phases were analyzed by using a Hewlett-Packard 5890 S-II gas chromatograph (GC), after calibration with gravimetrically prepared standard solutions. A flame ionization detector was used together with a 60-m, 0.2 mm i.d. fused silica capillary column, SUPELCOWAX 10. The GC response peaks were integrated by using a Hewlett-Packard 3396 integrator. At least two analyses were made of each

Table 2. Vapor Pressure P_i°, Antoine Coefficients A, B, and C, and Standard Deviations (σ),[a] of the Pure Components

1-butanol		2-butanol		chlorobenzene	
T/K	P_i°/kPa	T/K	P_i°/kPa	T/K	P_i°/kPa
315.35	2.86	305.65	3.92	320.55	4.92
319.55	3.76	309.65	5.04	324.65	5.96
323.85	4.85	313.25	6.25	328.35	7.07
326.45	5.62	316.25	7.43	331.35	8.08
329.45	6.65	318.55	8.51	334.15	9.14
332.25	7.77	320.85	9.72	338.45	10.96
334.65	8.83	323.15	11.00	342.55	12.98
336.75	9.86	324.95	12.11	346.35	15.11
338.85	11.01	326.75	13.37	349.35	17.00
340.65	12.08	328.35	14.50	353.55	20.00
342.25	13.10	329.75	15.61	354.05	20.34
343.85	14.18	331.25	16.85	357.95	23.50
345.25	15.20	332.55	18.03	361.05	26.27
346.95	16.52	333.85	19.25	363.75	28.88
348.35	17.68	335.45	20.82	366.85	32.19
349.75	18.89	336.65	22.08	370.15	35.95
350.75	19.82	337.75	23.35	372.65	38.99
352.45	21.43	340.65	26.88	376.55	44.29
356.25	25.48	344.05	31.56	380.05	49.51
359.05	28.88	347.25	36.50	382.95	54.21
363.55	35.15	350.55	42.22	385.95	59.43
366.65	40.12	353.25	47.62	389.25	65.64
369.35	44.91	355.55	52.47	391.25	69.58
372.85	51.75	358.05	58.27	393.85	75.10
374.95	56.35	360.05	63.30	396.25	80.57
377.25	61.68	361.95	68.46	398.35	85.48
380.25	69.27	363.95	74.13	400.15	89.94
382.35	74.81	365.95	80.07	401.35	92.95
384.15	80.01	367.45	84.85	402.65	96.39
385.85	85.42	368.95	89.84	404.05	100.00
387.25	90.09	370.35	94.64	404.55	101.32
388.55	94.27	371.65	99.52	404.75	101.87
390.25	100.04	372.95	104.61		

component	A	B	C	σ/kPa
1-butanol	15.8219	3499.92	−78.111	0.040
2-butanol	15.4438	3110.70	−84.708	0.065
chlorobenzene	13.8897	3168.06	−62.819	0.052

[a] $\sigma = f[\sum(P^\circ - P^\circ(\text{calc}))^2/(N - p)]^{1/2}$; N = no. of data points; p = no. of parameters.

liquid and each vapor sample. The experimental error in these variables was less than ±0.001 mole fraction.

Results and Discussion

The vapor pressures of the pure components P_i° were measured with the same recirculating still. The experimental values, in the range of work temperature, together with the parameters of the Antoine equation

$$\ln(P_i^\circ/\text{kPa}) = A_i - \frac{B_i}{(T/\text{K}) + C_i} \qquad (1)$$

and the standard deviation (σ) are given in Table 2.

The VLE measurements were made at 20 and 100 kPa for both systems, and the results are presented in Tables 3−6. The $T-x-y$ diagrams for the two systems are shown in Figures 1 and 2. Figure 3 shows a comparison among the experimental values obtained in this work at 100 kPa and those of the literature at 760 mmHg for the 1-butanol + chlorobenzene (Venkateswara Rao et al., 1977; Kormina et al., 1979) and the 2-butanol + chlorobenzene (Venkateswara Rao et al., 1980) systems. This figure shows that the results obtained in this work for the 1-butanol + chlorobenzene system agree well with those of the literature, and so they are interesting because they provide more data, obtained with high precision, in order to improve the existing information about this system. In that way, Venkateswara Rao et al. (1977) pointed out in their work that the accuracy of their points, mainly those around the azeotropic point, were not too good. Otherwise, the results obtained in this work for the 2-butanol + chlorobenzene

Table 3. Vapor−Liquid Equilibrium Data, Liquid-Phase Mole Fraction x_1, Vapor−Phase Mole Fraction y_1, Temperature T, and Activity Coefficients γ_i for 1-Butanol (1) + Chlorobenzene (2) at 20 kPa

x_1	y_1	T/K	γ_1	γ_2
0.000	0.000	353.55		
0.017	0.088	351.45	5.0514	1.0029
0.029	0.132	350.65	4.6630	0.9958
0.042	0.174	349.55	4.4074	1.0026
0.064	0.222	348.35	3.8911	1.0117
0.089	0.270	347.45	3.5740	1.0096
0.120	0.300	346.65	3.0644	1.0342
0.149	0.328	346.15	2.7537	1.0480
0.183	0.352	345.65	2.4627	1.0732
0.222	0.369	345.35	2.1651	1.1101
0.261	0.387	345.05	1.9566	1.1496
0.306	0.404	344.85	1.7598	1.1990
0.351	0.433	344.65	1.6623	1.2289
0.396	0.440	344.55	1.5044	1.3094
0.443	0.456	344.45	1.3988	1.3862
0.488	0.473	344.35	1.3243	1.4662
0.538	0.491	344.45	1.2413	1.5624
0.587	0.515	344.55	1.1851	1.6627
0.636	0.539	344.65	1.1418	1.7814
0.687	0.567	345.05	1.0901	1.9152
0.734	0.601	345.45	1.0616	2.0400
0.779	0.639	345.85	1.0412	2.1954
0.823	0.682	346.45	1.0215	2.3596
0.862	0.729	347.05	1.0136	2.5127
0.898	0.781	347.75	1.0071	2.6901
0.930	0.833	348.45	1.0045	2.8739
0.955	0.886	349.25	1.0018	2.9574
0.974	0.930	349.85	1.0019	3.0769
0.988	0.965	350.35	1.0012	3.1934
0.995	0.986	350.65	1.0013	3.3152
1.000	1.000	350.95		

Table 4. Vapor−Liquid Equilibrium Data, Liquid-Phase Mole Fraction x_1, Vapor−Phase Mole Fraction y_1, Temperature T, and Activity Coefficients γ_i for 1-Butanol (1) + Chlorobenzene (2) at 100 kPa

x_1	y_1	T/K	γ_1	γ_2
0.000	0.000	404.05		
0.023	0.103	401.65	3.0251	0.9734
0.034	0.140	400.05	2.9080	0.9867
0.063	0.215	398.05	2.6052	0.9804
0.088	0.265	396.25	2.4464	0.9914
0.127	0.338	394.15	2.3148	0.9903
0.167	0.391	392.45	2.1466	1.0038
0.217	0.442	391.25	1.9584	1.0113
0.266	0.477	390.25	1.7810	1.0422
0.340	0.513	389.45	1.5453	1.1026
0.366	0.526	388.85	1.5014	1.1381
0.417	0.545	388.45	1.3823	1.2043
0.465	0.573	388.05	1.3258	1.2428
0.515	0.594	387.85	1.2471	1.3143
0.562	0.616	387.65	1.1949	1.3832
0.605	0.639	387.55	1.1555	1.4463
0.647	0.661	387.45	1.1224	1.5221
0.688	0.683	387.45	1.0907	1.6100
0.732	0.711	387.55	1.0625	1.7061
0.768	0.739	387.65	1.0486	1.7759
0.804	0.764	387.75	1.0320	1.8952
0.819	0.779	387.95	1.0249	1.9150
0.869	0.826	388.15	1.0182	2.0569
0.899	0.858	388.45	1.0113	2.1571
0.916	0.880	388.75	1.0067	2.1834
0.944	0.916	389.05	1.0061	2.2640
0.960	0.938	389.35	1.0029	2.2972
0.979	0.968	389.55	1.0062	2.3461
0.990	0.984	389.75	1.0048	2.4175
1.000	1.000	390.25		

Table 5. Vapor–Liquid Equilibrium Data, Liquid-Phase Mole Fraction x_1, Vapor–Phase Mole Fraction y_1, Temperature T, and Activity Coefficients γ_i for 2-Butanol (1) + Chlorobenzene (2) at 20 kPa

x_1	y_1	T/K	γ_1	γ_2
0.000	0.000	353.55		
0.013	0.098	351.05	3.6540	1.0025
0.025	0.172	349.15	3.5132	1.0020
0.048	0.284	346.55	3.3765	0.9823
0.071	0.352	343.65	3.2387	1.0217
0.106	0.431	341.25	2.9515	1.0275
0.145	0.486	339.65	2.6369	1.0361
0.188	0.533	338.25	2.3761	1.0496
0.253	0.572	337.35	1.9841	1.0857
0.294	0.597	336.45	1.8608	1.1236
0.345	0.609	335.85	1.6644	1.2061
0.401	0.631	335.45	1.5131	1.2653
0.441	0.648	334.95	1.4480	1.3222
0.493	0.666	334.65	1.3511	1.4012
0.541	0.672	334.35	1.2628	1.5372
0.589	0.699	334.05	1.2231	1.5982
0.634	0.713	333.85	1.1705	1.7271
0.673	0.732	333.65	1.1442	1.8190
0.712	0.759	333.55	1.1264	1.8674
0.751	0.770	333.55	1.0835	2.0613
0.787	0.791	333.45	1.0681	2.1949
0.821	0.812	333.45	1.0511	2.3482
0.852	0.835	333.55	1.0350	2.4985
0.879	0.856	333.55	1.0298	2.6473
0.905	0.881	333.65	1.0233	2.7968
0.928	0.905	333.75	1.0205	2.9249
0.961	0.944	334.05	1.0125	3.1476
0.980	0.971	334.15	1.0160	3.2259
0.990	0.984	334.25	1.0149	3.3619
1.000	1.000	334.65		

Table 6. Vapor–Liquid Equilibrium Data, Liquid-Phase Mole Fraction x_1, Vapor–Phase Mole Fraction y_1, Temperature T, and Activity Coefficients γ_i for 2-Butanol (1) + Chlorobenzene (2) at 100 kPa

x_1	y_1	T/K	γ_1	γ_2
0.000	0.000	404.05		
0.012	0.081	401.65	2.4091	0.9872
0.023	0.141	399.65	2.3766	0.9851
0.037	0.203	397.45	2.2932	0.9859
0.066	0.311	393.65	2.2290	0.9790
0.139	0.479	386.55	2.0695	0.9884
0.173	0.526	383.85	2.0037	1.0143
0.212	0.579	381.65	1.9376	1.0128
0.257	0.619	379.75	1.8276	1.0311
0.309	0.648	378.25	1.6842	1.0709
0.357	0.671	377.05	1.5723	1.1188
0.410	0.701	376.05	1.4822	1.1467
0.458	0.722	375.25	1.4077	1.1883
0.511	0.731	374.65	1.3077	1.2974
0.561	0.748	374.05	1.2435	1.3857
0.603	0.768	373.55	1.2115	1.4295
0.645	0.787	373.15	1.1776	1.4893
0.690	0.804	372.75	1.1421	1.5860
0.732	0.823	372.45	1.1143	1.6734
0.768	0.842	372.15	1.0976	1.7500
0.805	0.861	371.95	1.0793	1.8425
0.837	0.879	371.75	1.0677	1.9296
0.868	0.897	371.65	1.0550	2.0302
0.894	0.914	371.55	1.0468	2.1320
0.921	0.933	371.45	1.0417	2.2213
0.941	0.948	371.45	1.0355	2.3233
0.963	0.965	371.35	1.0345	2.4672
0.982	0.982	371.35	1.0323	2.5929
0.991	0.990	371.65	1.0199	2.7501
1.000	1.000	371.85		

system are quite different from those reported by Venkateswara Rao et al. (1980). They clearly show the existence of an azeotropic point, so they provide estimable additional information in relation to the previous work done with this system.

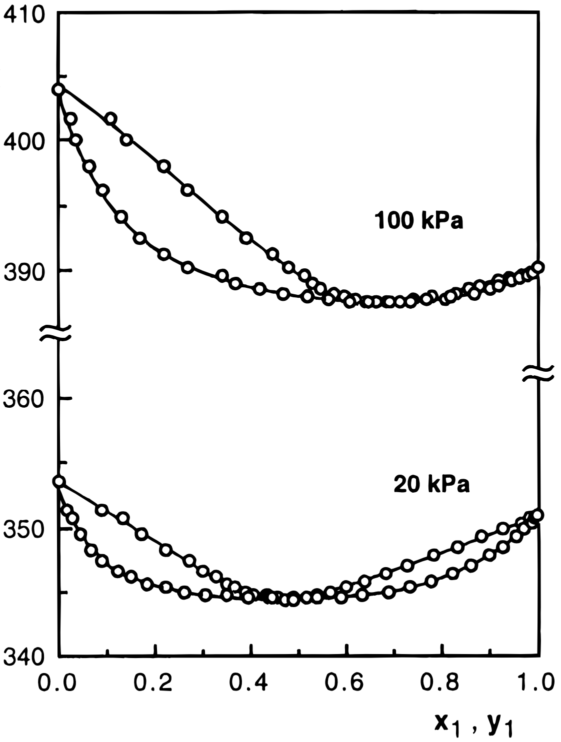

Figure 1. Vapor–liquid equilibrium of the system 1-butanol (1) + chlorobenzene (2) at 20 and 100 kPa as a function of the mole fraction of component 1: (○) experimental points; (—) splined curves.

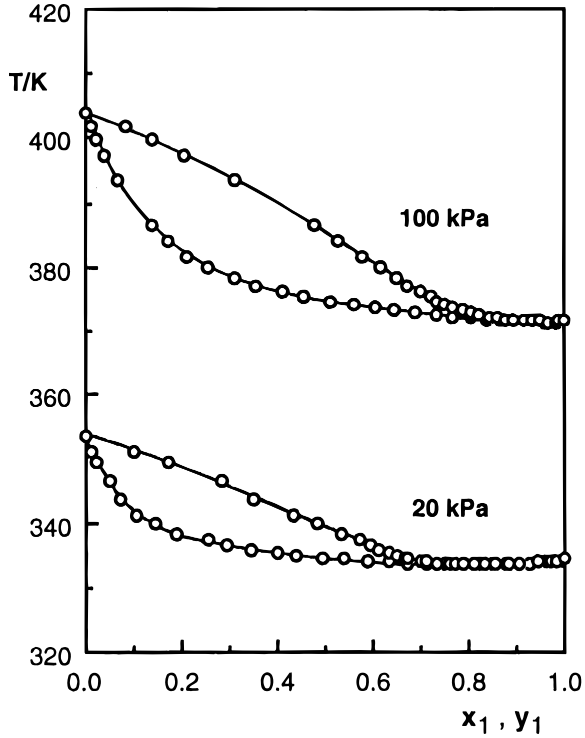

Figure 2. Vapor–liquid equilibrium of the system 2-butanol (1) + chlorobenzene (2) at 20 and 100 kPa as a function of the mole fraction of component 1: (○) experimental points; (—) splined curves.

Figures 1 and 2 show that both systems present a minimum boiling azeotrope and the mole fraction of 1-butanol or 2-butanol at this point increases with pressure. For the 2-butanol + chlorobenzene system the azeotropic point is clearly observed at 20 kPa but at 100 kPa it has almost disappeared. The results of Venkateswara Rao et

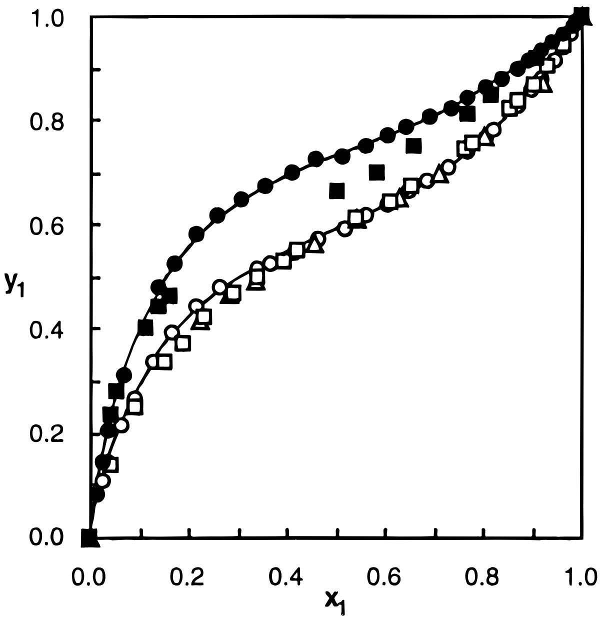

Figure 3. Comparison among the experimental values obtained in this work at 100 kPa and those from literature: 1-butanol (1) + chlorobenzene (2) ((○) experimental, (□) Venkateswara Rao et al. (1977), (△) Kormina et al. (1979)) and 2-butanol (1) + chlorobenzene (2) ((●) experimental, (■) Venkateswara Rao et al. (1980)).

Table 7. Variation of the Azeotropic Point with Pressure

P/kPa	y_1	*T*/K	reference
	1-Butanol (1) + Chlorobenzene (2)		
8	0.4050	325.35	Kormina et al. (1979)
13.33	0.4450	336.25	Kormina et al. (1979)
20	0.464	344.35	this work
100	0.677	387.45	this work
101.32	0.6900	388.25	Kormina et al. (1979)
101.32	0.6950	388.25	Venkateswara Rao et al. (1977)
	2-Butanol (1) + Chlorobenzene (2)		
20	0.797	333.45	this work
100	0.982	371.35	this work
101.32	none	none	Venkateswara Rao et al. (1980)

al. (1980) do not show an azeotrope at 760 mmHg. In Table 7 the azeotropic data obtained in this work at 20 and 100 kPa for both systems are given together with those of the literature. From this table it can also be said that the azeotropic composition and temperature obtained in this work for the 1-butanol + chlorobenzene system at 20 and 100 kPa agree well with those of the literature. For the 2-butanol + chlorobenzene system the results obtained in this work clearly show the existence of an azeotrope for this system and differ from those of Venkateswara Rao et al. (1980).

The liquid-phase activity coefficients of the components were calculated by the equation

$$y_i\phi_i P = x_i\gamma_i\phi_i^{\,s}P_i^{\circ}\exp[v_i(P - P_i^{\circ})/RT] \qquad (2)$$

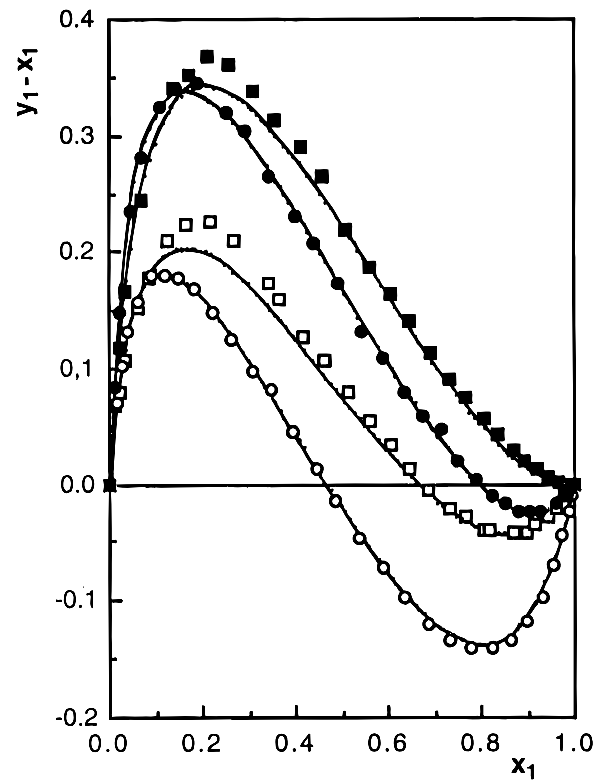

Figure 4. Comparison among the experimental values obtained in this work (1-butanol (1) + chlorobenzene (2) at 20 kPa (○) and at 100 kPa (□) and 2-butanol (1) + chlorobenzene (2) at 20 kPa (●) and at 100 kPa (■)) and those calculated using the Wilson equation (—).

where x_i and y_i are the liquid and vapor mole fractions in equilibrium, ϕ_i is the fugacity coefficient, *P* is the total pressure, γ_i is the activity coefficient, $\phi_i^{\,s}$ is the pure component fugacity coefficient at saturation, P_i° is the pure component vapor pressure, v_i is the liquid molar volume, *R* is the universal gas constant, and *T* is the absolute temperature.

Fugacity coefficients ϕ_i and $\phi_i^{\,s}$ were calculated by means of the virial equation of state. The liquid molar volumes as well as the equation and the parameters to calculate the second virial coefficients were taken from the literature (Daubert and Danner, 1995). The values of the activity coefficients calculated using eq 2 are listed in Tables 3−6. Both systems show positive deviations from ideality.

The results were tested for thermodynamic consistency using the point-to-point method of Van Ness et al. (1973), modified by Fredenslund et al. (1977). A four-parameter Legendre polynomial was used for the excess Gibbs free energy. According to Fredenslund et al., the *P, T, x, y* values are consistent if the mean absolute deviation between calculated and measured mole fractions of component 1 in the vapor phase, $\delta(y)$, is less than 0.01. The results of this test for the binary systems in consideration are $\delta(y) = 0.0040$ and $\delta(y) = 0.0050$ for the 1-butanol + chlorobenzene system at 20 and 100 kPa, respectively, and $\delta(y) = 0.0066$ and $\delta(y) = 0.0054$ for the 2-butanol +

Table 8. Parameters and Deviations between Calculated and Experimental Vapor-Phase Mole Fractions and Temperatures Obtained Using the Wilson Equation

system	*P*/kPa	A_{12}/J·mol^{-1}	A_{21}/J·mol^{-1}	$\delta(y)^a$	$\delta(T)^b$/K
1-butanol + chlorobenzene	20	4378.748	881.221	0.0030	0.089
	100	3332.257	667.891	0.0073	0.172
2-butanol + chlorobenzene	20	3004.875	1444.396	0.0033	0.187
	100	1912.304	1600.647	0.0077	0.282

[a] $\delta(y) = \sum|y - y(\text{calc})|/N$. [b] $\delta(T) = \sum|T - T(\text{calc})|/N$; N = no. of data points.

chlorobenzene system at 20 and 100 kPa, respectively. These results indicate that the experimental data for the two systems are thermodynamically consistent.

The activity coefficients were correlated with the Wilson equation (Gmehling and Onken, 1977). For fitting the binary parameters the following objective function was used:

$$F = \sum\left(\frac{\gamma_1 - \gamma_{1(\mathrm{calc})}}{\gamma_1}\right)^2 + \sum\left(\frac{\gamma_2 - \gamma_{2(\mathrm{calc})}}{\gamma_2}\right)^2 \qquad (3)$$

Figure 4 shows a comparison among the experimental values and those calculated using this model. The parameters and average deviations obtained for this equation are reported in Table 8. The molar volumes used for fitting the Wilson parameters were 0.091 943 m^3/kmol for 1-butanol, 0.092 118 m^3/kmol for 2-butanol, and 0.102 264 m^3/kmol for chlorobenzene (Daubert and Danner, 1995).

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