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ISOBARIC VLE DATA FOR THE BINARY SYSTEMS 1,3-DICHLOROPROPANE WITH ISOMERIC BUTANOLS

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Isobaric vapour-liquid equilibrium data at 40.0 and 101.3 kPa for the binary systems 1,3-dichloropropane with 1-butanol, 2-butanol, 2-methyl-1-propanol and 2-methyl-2-propanol are reported. Some of the studied systems present azeotropes. The activity coefficients were found to be thermodynamically consistent, and they were satisfactorily correlated with the Margules, Van Laar, Wilson, NRTL and UNIQUAC equations.

KEY WORDS: Isobaric vapour-liquid equilibrium, 1,3-dichloropropane, butanols

INTRODUCTION

The knowledge of vapour-liquid equilibrium provides information about thermodynamic behaviour of the liquid mixtures. With this objective and following a research program involving haloalkanes and butanols^{1–11} we report here new isobaric vapour-liquid equilibrium measurements for the binary systems 1,3-dichloropropane with isomeric butanols at 40.0 and 101.3 kPa.

EXPERIMENTAL

The apparatus used for the present study was a still (Labodest model) manufactured by Fischer and Verfahrenstechnik (Germany). The equilibrium temperatures were measured to an accuracy of ± 0.01 K by means of a thermometer (model F25) from Automatic Systems Laboratories, and the pressure in the still was measured with a pressure transducer Druck PDCR 110/W (pressure indicator DPI201) with an accuracy of ± 0.1 kPa. The composition of the liquid and vapour samples were determined by measuring their densities at 298.15 K. The experimental procedure was checked and described in an earlier paper¹².

The liquids used, 1-butanol (better than 99.8 mol%), 2-methyl-1-propanol and 2-methyl-2-propanol (better than 99.5 mol%), and 2-butanol and 1,3-dichloropropane (better than 99 mol%) were obtained from Aldrich. The purity of the materials was checked by GLC chromatography and by measuring their physical properties. The

Table 1 Physical properties (densities at 298.15 K and normal boiling points) of the pure compounds.

Compound	$\rho/\text{kg m}^{-3}$		T_b/K	
	exptl.	lit. ¹⁴	exptl.	lit. ¹⁴
1,3-dichloropropane ^a	1180.25	1180.0	394.10	393.95
1-butanol	805.85	806.0	390.84	390.81
2-butanol	802.40	802.6	372.65	372.70
2-methyl-1-propanol	797.98	797.8	380.72	380.81
2-methyl-2-propanol	781.00	781.2	355.50	355.57

^a Reference 13

comparison of measured physical properties, density and normal boiling point, with literature values^{13,14} are shown in Table 1.

RESULTS AND DISCUSSION

Table 2 shows the vapour-liquid equilibrium data (T, x_1, y_1), the activity coefficients and excess free energies at 40.0 and 101.3 kPa. In Figures 1–4 the $T - x_1 - y_1$ diagrams

Table 2 Experimental vapour-liquid equilibrium data for 1,3-dichloropropane(1) with isomeric butanols (2) at indicated pressure.

T/K	x_1	y_1	γ_1	γ_2	$G^E/\text{J mol}^{-1}$
1,3-dichloropropane(1) + 1-butanol(2) at 40.0 kPa					
364.76	0.0526	0.1320	2.427	0.998	136.2
362.57	0.1144	0.2542	2.313	1.006	306.0
360.84	0.1971	0.3555	1.992	1.033	485.7
359.73	0.2789	0.4298	1.768	1.068	616.9
358.86	0.3613	0.4864	1.592	1.128	730.8
358.27	0.4490	0.5296	1.424	1.229	811.2
358.16	0.4971	0.5529	1.348	1.286	818.8
358.05	0.6090	0.6077	1.214	1.459	790.9
358.48	0.7590	0.6849	1.081	1.866	624.4
359.25	0.8497	0.7495	1.029	2.300	445.6
359.95	0.8927	0.7944	1.013	2.565	336.3
361.04	0.9386	0.8620	1.007	2.870	212.7
1,3-dichloropropane(1) + 1-butanol(2) at 101.3 kPa					
389.26	0.0504	0.0961	2.181	1.006	146.6
388.07	0.1028	0.1721	1.979	1.017	274.3
386.97	0.1491	0.2368	1.936	1.027	389.9
385.75	0.2385	0.3262	1.726	1.058	554.5
385.16	0.3195	0.3889	1.562	1.096	656.3
384.74	0.4205	0.4513	1.393	1.173	742.6
384.65	0.5399	0.5171	1.246	1.305	772.1
385.16	0.6290	0.5748	1.172	1.400	719.0
385.57	0.7060	0.6147	1.104	1.577	652.7
385.93	0.7356	0.6333	1.080	1.648	605.9
386.53	0.8030	0.6884	1.057	1.840	530.2
389.55	0.9041	0.8211	1.030	1.954	293.3

Table 2 (Continued.)

T/K	x_1	y_1	γ_1	γ_2	$G^E/\text{J mol}^{-1}$
1,3-dichloropropane(1) + 2-butanol(2) at 40.0 kPa					
348.96	0.0853	0.1183	2.354	0.999	209.5
348.37	0.1585	0.1896	2.076	1.024	393.9
348.16	0.2323	0.2451	1.846	1.056	532.4
347.95	0.2898	0.2789	1.697	1.100	639.4
347.95	0.4082	0.3447	1.489	1.200	781.9
348.16	0.5004	0.3852	1.346	1.321	833.6
348.88	0.6087	0.4356	1.218	1.501	808.4
350.35	0.7054	0.4870	1.111	1.700	672.0
351.53	0.7706	0.5384	1.076	1.867	583.8
353.77	0.8512	0.6184	1.030	2.165	412.7
359.76	0.9600	0.8437	1.007	2.583	133.0
1,3-dichloropropane(1) + 2-butanol(2) at 101.3 kPa					
372.36	0.0620	0.0691	2.099	1.003	151.9
372.15	0.1639	0.1565	1.809	1.028	371.2
372.15	0.2200	0.1954	1.683	1.051	473.8
372.25	0.3046	0.2463	1.527	1.100	604.5
372.47	0.3578	0.2780	1.457	1.132	664.2
373.15	0.4966	0.3500	1.294	1.269	769.5
374.16	0.5874	0.3970	1.202	1.387	756.1
376.70	0.6935	0.4627	1.098	1.523	606.1
379.76	0.7798	0.5345	1.029	1.654	419.5
383.82	0.8844	0.6830	1.029	1.876	313.0
387.76	0.9450	0.7920	0.999	2.280	141.6
1,3-dichloropropane(1) + 2-methyl-1-propanol(2) at 40.0 kPa					
355.75	0.0697	0.1410	2.673	1.002	209.3
355.00	0.1069	0.1911	2.426	1.016	320.5
354.15	0.1636	0.2544	2.176	1.037	464.5
353.63	0.2233	0.3093	1.975	1.058	576.8
353.27	0.3034	0.3636	1.732	1.105	693.1
353.05	0.3674	0.3994	1.583	1.159	770.0
352.90	0.4667	0.4441	1.394	1.281	842.2
352.95	0.5310	0.4719	1.299	1.381	852.0
353.33	0.6257	0.5148	1.186	1.564	804.9
353.67	0.6656	0.5352	1.145	1.652	757.9
355.15	0.8034	0.6245	1.049	2.129	551.0
357.14	0.8768	0.7066	1.012	2.438	357.7
358.86	0.9163	0.7729	0.997	2.583	229.1
1,3-dichloropropane(1) + 2-methyl-1-propanol(2) at 101.3 kPa					
380.46	0.0340	0.0538	2.328	1.000	90.1
380.15	0.0562	0.0849	2.243	1.001	145.0
379.32	0.1194	0.1585	2.020	1.015	307.3
378.67	0.2056	0.2358	1.779	1.046	485.8
378.55	0.2991	0.2985	1.553	1.093	611.1
378.66	0.3675	0.3397	1.434	1.136	670.6
378.86	0.4693	0.3925	1.290	1.237	731.0
378.94	0.5351	0.4278	1.230	1.326	761.8
379.85	0.6285	0.4790	1.141	1.463	707.8
380.86	0.7116	0.5312	1.084	1.637	632.5
382.27	0.8023	0.6098	1.059	1.892	547.7
384.00	0.8650	0.6900	1.057	2.074	468.6
387.07	0.9118	0.7485	0.997	2.321	230.7

Table 2 (Continued.)

<i>T</i> /K	x_1	y_1	γ_1	γ_2	$G^E/\text{J mol}^{-1}$
1,3-dichloropropane(1) + 2-methyl-2-propanol(2) at 40.0 kPa					
334.05	0.0954	0.0765	2.488	1.014	276.5
334.37	0.1434	0.1044	2.228	1.023	374.6
334.73	0.2288	0.1469	1.934	1.065	555.9
335.45	0.3237	0.1845	1.665	1.124	680.9
336.16	0.4130	0.2109	1.447	1.214	744.7
337.60	0.5362	0.2564	1.275	1.358	764.5
339.25	0.6413	0.2961	1.150	1.546	693.5
342.34	0.7686	0.3628	1.037	1.900	501.4
345.65	0.8306	0.4381	1.016	1.992	373.7
352.47	0.9322	0.6323	1.009	2.479	204.2
357.65	0.9700	0.7910	1.006	2.614	102.6
1,3-dichloropropane(1) + 2-methyl-2-propanol(2) at 101.3 kPa					
355.86	0.0616	0.0422	2.242	1.007	166.6
356.48	0.1266	0.0764	1.932	1.020	297.1
347.35	0.2045	0.1158	1.757	1.038	429.9
358.07	0.2749	0.1429	1.573	1.075	526.0
359.16	0.3526	0.1748	1.444	1.114	594.6
360.37	0.4451	0.2082	1.306	1.193	649.9
362.39	0.5461	0.2508	1.197	1.284	637.4
364.18	0.6114	0.2797	1.122	1.345	570.0
366.63	0.7080	0.3366	1.075	1.525	531.8
369.97	0.7832	0.4033	1.045	1.650	439.8
374.65	0.8493	0.4929	1.016	1.731	300.9
379.36	0.9063	0.6054	1.014	1.867	225.0
383.19	0.9383	0.6942	1.004	1.955	143.4

are represented. Mixtures containing 2-methyl-2-propanol at 40.0 kPa and 1-butanol, 2-butanol and 2-methyl-1-propanol at both pressures 40.0 and 101.3 kPa present minimum temperature azeotropes, furthermore when pressure increases the composition of the azeotropic mixture becomes richer in isomeric butanol, the composition and the boiling temperature of the azeotropes are gathered in Table 3.

The activity coefficients γ_i were calculated, taking into account the non-ideality of the vapour phase, from following equations:

$$\gamma_1 = \frac{y_1 P}{x_1 p_i^0} \exp \left[\frac{(B_{ii} - V_i^0)(p - p_i^0) + (1 - y_1)^2 P \delta_{ij}}{RT} \right] \quad (1)$$

$$\delta_{ij} = 2B_{ij} - B_{ii} - B_{jj} \quad (2)$$

here x_i and y_i are the liquid and vapour phase compositions, P is the total pressure, p_i^0 are the vapour-pressures of the pure compounds calculated by using the Antoine equation, where the constants appear in Table 4, B_{ij} are the second virial coefficients and B_{ij} are the cross second virial coefficients calculated using the Amdur-Mason equation¹⁵, and V_i^0 are the molar volumes of the saturated liquids estimated by the

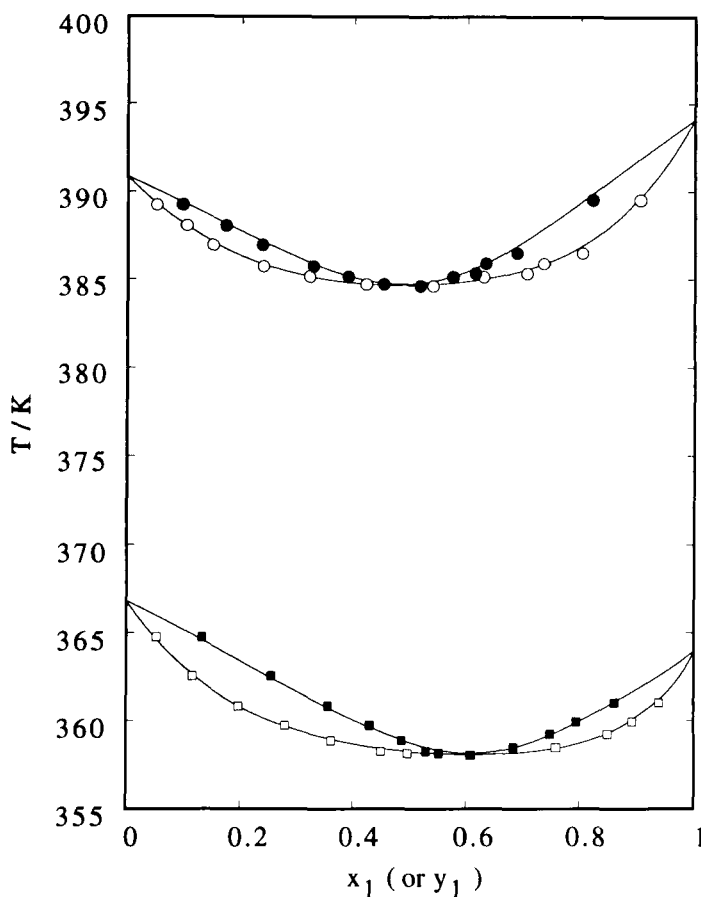


Figure 1 $T-x_1-y_1$ diagrams for 1,3-dichloropropane(1) + 1-butanol(2): (\square , \blacksquare) exptl. data at 40.0 kPa; (\circ , \bullet) exptl. data at 101.3 kPa; (—) Wilson equation.

Table 3 Composition ($x_{1(a2)}$) and boiling temperature ($T_{(a2)}$) of the azeotropic mixtures.

System	P/kPa	$x_{1(a2)}$	$T_{(a2)}/\text{K}$
1,3-dichloropropane + 1-butanol	40.0	0.6077	358.01
	101.3	0.4887	384.65
2-butanol	40.0	0.2826	347.93
	101.3	0.1652	371.89
2-methyl-1-propanol	40.0	0.4246	352.96
	101.3	0.3297	378.59
2-methyl-2-propanol	40.0	0.0248	333.87

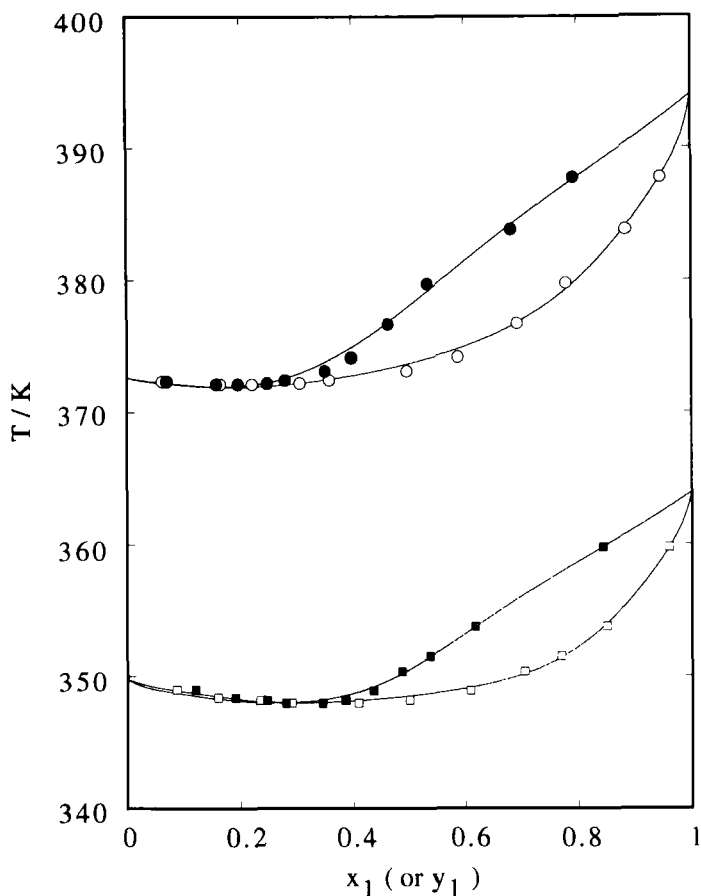


Figure 2 $T-x_1-y_1$ diagrams for 1,3-dichloropropane(1) + 2-butanol(2); (\square , \blacksquare) exptl. data at 40.0 kPa; (\circ , \bullet) exptl. data at 101.3 kPa; (—) Wilson equation.

Table 4 Constants of Antoine's equation for vapour pressures of the compounds, (pressure in kPa).

Compound	A	B	C
1,3-dichloropropane ^a	5.48790	1020.494	172.038
1-butanol	6.54743	1338.769	177.042
1-butanol	6.35457	1171.981	169.955
2-methyl-1-propanol	6.50091	1275.197	175.187
2-methyl-2-propanol	6.35648	1107.060	172.102

^aOur own vapour pressure measurements

Yen and Woods method¹⁶. The Antoine's constants for the 1,3-dichloropropane were obtained from our own vapour-pressure measurements and for the isomeric butanols were taken from Riddick *et al.*¹⁷. The second virial coefficients for the 1,3-dichloropropane were estimated by the Redlich-Kwong equation¹⁸, and for the

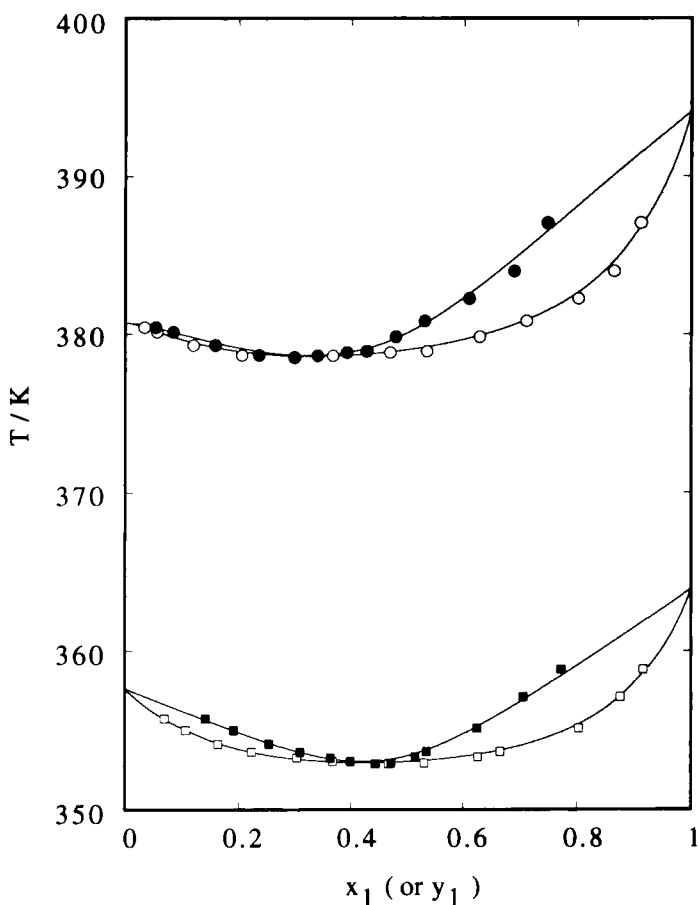


Figure 3 $T-x_1-y_1$ diagrams for 1,3-dichloropropane(1) + 2-methyl-1-propanol(2): (\square , \blacksquare) exptl. data at 40.0 kPa; (\circ , \bullet) exptl. data at 101.3 kPa; (—) Wilson equation.

isomeric butanols were obtained from TRC-tables¹⁴. The correction for the non-ideality of the vapour phase, represented by the exponential term in Eq. 1, was important only at very dilute concentrations.

The thermodynamic consistency of the activity coefficients was tested using the Van Ness method¹⁹, described by Fredeslund *et al.*²⁰ using a third order Legendre polynomial. According to this test, the experimental activity coefficients are considered consistent if the average deviation in $y(\Delta y)$ is smaller than 0.01, all the present systems satisfy this condition.

The activity coefficients were correlated with the Margules, Van Laar, Wilson, NRTL and UNIQUAC equation. The parameters have been obtained by fitting the reduced excess free energies G^E/RT using the simplex method. These parameters along with the average deviation in T (ΔT), average deviation by y (Δy) and activity coefficients at infinite dilution are listed in Table 5. All the equations correlated the activity coefficients quite well.

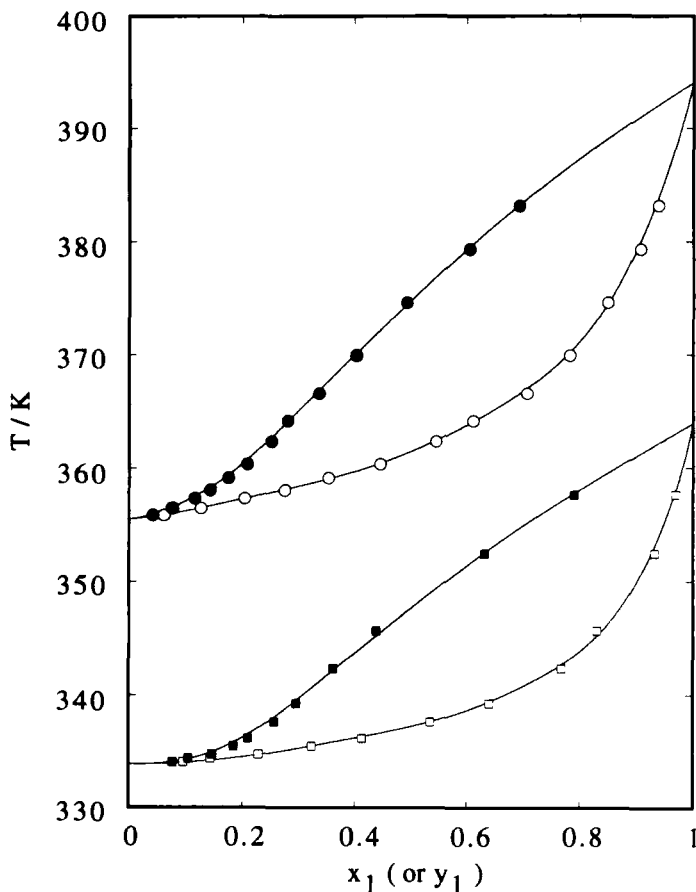


Figure 4 $T-x_1-y_1$ diagrams for 1,3-dichloropropane(1) + 2-methyl-2-propanol(2); (\square , \blacksquare) exptl. data at 40.0 kPa; (\circ , \bullet) exptl. data at 101.3 kPa; (—) Wilson equation.

The systems deviate positively from ideality and the activity coefficients decrease with increasing pressure, the greatest activity coefficients correspond to the mixture containing 2-methyl-2-propanol. The molecular interactions that govern the thermodynamic behaviour has been previously described in a previous paper⁸, and the most important effect in the mixture process are the breaking of the self-association in the butanols and the specific interaction between the Cl-group of the dichloroalkane and the OH-group of the butanol. The excess free energies has been calculated from activity coefficients and they are positive over the entire range of composition and also decrease with increasing pressure. At 40.0 kPa the values of G^E fall in the order 2-methyl-1-propanol > 2-butanol > 1-butanol > 2-methyl-2-propanol while at 101.3 kPa the order is 1-butanol > 2-butanol > 2-methyl-1-propanol > 2-methyl-2-propanol, although the G^E values for the mixtures containing 1-butanol, 2-butanol and 2-methyl-1-propanol are very similar. Furthermore the G^E values at 101.3 kPa

Table 5 Correlation parameters, average derivative in $T(\Delta T)$, average deviation in $y(\Delta y)$, and activity coefficients at infinite dilution γ_i^∞ .

Equation	A_{12}	A_{21}	$\Delta T/K$	Δy	γ_1^∞	γ_2^∞
1,3-dichloropropane(1) + 1-butanol(2) at 40.0 kPa						
Margules	0.9752	1.2129	0.04	0.0022	2.65	3.36
Van Laar	0.9875	1.2213	0.05	0.0019	2.68	3.39
Wilson	0.6604	0.3996	0.07	0.0024	2.76	3.51
NRTL($\alpha_{12} = 0.3$) ^b	0.9899	0.2569	0.05	0.0019	2.70	3.41
UNIQUAC ^b	0.6534	1.0275	0.05	0.0019	2.69	3.40
1,3-dichloropropane(1) + 1-butanol(2) at 101.3 kPa						
Margules	0.9174	1.0144	0.08	0.0067	2.50	2.76
Van Laar	0.9183	1.0177	0.08	0.0066	2.51	2.77
Wilson	0.6454	0.5029	0.07	0.0057	2.55	2.83
NRTL($\alpha_{12} = 0.3$)	0.7072	0.3505	0.07	0.0064	2.52	2.78
UNIQUAC	0.7557	0.9490	0.08	0.0065	2.51	2.77
1,3-dichloropropane(1) + 2-butanol(2) at 40.0 kPa						
Margules	0.0268	1.1972	0.15	0.0071	2.79	3.31
Van Laar	1.0362	1.1974	0.15	0.0068	2.82	3.31
Wilson	0.6118	0.4286	0.18	0.0059	2.89	3.44
NRTL($\alpha_{12} = 0.3$)	0.8775	0.3670	0.16	0.0066	2.84	3.34
UNIQUAC	0.7014	0.9637	0.16	0.0067	2.83	3.32
1,3-dichloropropane(1) + 2-butanol(2) at 101.3 kPa						
Margules	0.9390	0.9213	0.26	0.0075	2.56	2.51
Van Laar	0.9384	0.9222	0.26	0.0075	2.56	2.51
Wilson	0.5750	0.5980	0.27	0.0067	2.60	2.56
NRTL($\alpha_{12} = 0.3$)	0.4717	0.5334	0.26	0.0073	2.57	2.53
UNIQUAC	0.8645	0.8455	0.26	0.0074	2.56	2.52
1,3-dichloropropane(1) + 2-methyl-1-propanol(2) at 40.0 kPa						
Margules	1.1275	1.1676	0.09	0.0018	3.09	3.21
Van Laar	1.1282	1.1675	0.09	0.0018	3.09	3.21
Wilson	0.5255	0.4821	0.12	0.0020	3.19	3.33
NRTL($\alpha_{12} = 0.3$)	0.6947	0.5723	0.10	0.0016	3.12	3.24
UNIQUAC	0.7870	0.8584	0.09	0.0017	3.10	3.22
1,3-dichloropropane(1) + 2-methyl-1-propanol(2) at 101.3 kPa						
Margules	0.8504	1.0744	0.15	0.0083	2.34	2.93
Van Laar	0.8557	1.0936	0.15	0.0084	2.35	2.98
Wilson	0.7465	0.4196	0.15	0.0082	2.39	3.07
NRTL($\alpha_{12} = 0.3$)	0.9628	0.1378	0.15	0.0083	2.36	2.99
UNIQUAC	0.6448	1.0819	0.15	0.0084	2.36	2.99
1,3-dichloropropane(1) + 2-methyl-2-propanol(2) at 40.0 kPa						
Margules	1.1816	0.9731	0.11	0.0069	3.26	2.65
Van Laar	1.1866	0.9841	0.12	0.0067	3.28	2.68
Wilson	0.4176	0.6525	0.13	0.0064	3.39	2.74
NRTL($\alpha_{12} = 0.3$)	0.2835	0.9329	0.12	0.0066	3.30	2.69
uniquac	1.0281	0.6662	0.12	0.0066	3.29	2.68
1,3-dichloropropane(1) + 2-methyl-2-propanol(2) at 101.3 kPa						
Margules	0.9278	0.7792	0.10	0.0065	2.53	2.18
Van Laar	0.9342	0.7838	0.10	0.0065	2.55	2.19
Wilson	0.4980	0.7459	0.10	0.0065	2.59	2.21
NRTL($\alpha_{12} = 0.3$)	0.1735	0.7720	0.10	0.0065	2.55	2.19
UNIQUAC	1.0564	0.7035	0.10	0.0065	2.55	2.19

^a $\Lambda_{12}, \Lambda_{21}$

^b τ_{12}, τ_{21}

for the studied mixtures are greater than for the mixtures of 1,2-dichloroethane with isomeric butanols²¹.

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