

accuracy, 5-10%, given by Reid et al. (12) for this correlation.

Conclusion

New binary diffusion coefficients for the halocarbons CCl_2F_2 and $\text{C}_2\text{Cl}_2\text{F}_4$, with H_2 , N_2 , and He, are presented together with the corresponding temperature dependence coefficients in the 298-343 K region. The arrested-flow method is easy to implement with a reasonable accuracy using simple equipment. The discrepancy observed between experimental and predicted values shows the necessity of experimental measurements to obtain reliable data.

Glossary

D_{AB}	binary diffusion coefficient, cm^2/s
D_{axial}	axial diffusion coefficient, cm^2/s
F_e, F_c	experimental flow rate and flow rate corrected to column conditions, cm^3/min
n	exponent of the temperature-dependent equation for D_{AB}
$P_{\text{H}_2\text{O}}$	vapor pressure of water at ambient temperature, mm Hg
s_t^2	spatial variance, m^2
S_a	cross-sectional area of the chromatographic column, m^2
t_{arrested}	time period corresponding to the flow arrest time, s
t_A	time period during convection of solute pulse to column midregion, s
t_B	time period during convection of solute pulse out of the column, s

T_a, T_c	ambient and column temperature, K
v_1	linear velocity of the carrier gas, cm/min
σ_t^2	standard deviation, s^2
Δ	width at half-height of a chromatographic peak, s

Registry No. R12, 75-71-8; R114, 76-14-2; H_2 , 1333-74-0; N_2 , 7727-37-9; He, 7440-59-7.

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Vapor-Liquid Equilibrium for the 2-Propanol-Methyl Acetate-Dichloromethane System at 298.15 K[†]

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Isothermal vapor-liquid equilibrium data were determined for the 2-propanol-methyl acetate-dichloromethane system at 298.15 K, by using a modified version of a Boublik-Benson still. The data proved to be thermodynamically consistent. Excess molar Gibbs energies G^E were calculated over the entire range of composition. Different expressions existing in the literature were used to predict G^E from the corresponding binary data. The empirical correlation of Cibulka resulted in being the best one for this system.

Introduction

Vapor-liquid equilibrium (VLE) data are necessary for the design of distillation processes. No experimental data have been reported in the available literature for the ternary 2-propanol + methyl acetate + dichloromethane system at 298.15 K. In previous papers we have published densities and viscosities (1), as well as molar excess Gibbs energies G^E for

the corresponding binary mixtures (2-4). From the experimental VLE data, for this ternary system, activity coefficients γ_i and G^E can be calculated and compared with values predicted from different expressions in terms of the binary data (5-11).

Experimental Section

The methods used in our laboratory have already been described (1-4). Densities were determined with a digital densimeter AP, Model DMA 45. All weighings were made on an H315 Mettler balance. A thermostatically controlled bath (constant to 0.01 K) was used. Temperatures T were read from calibrated thermometers. Calibration was done with air and doubly distilled water. The accuracy in density ρ was 0.01%.

Equilibrium data, pressure P , and mole fractions x_i in the liquid phase and y_i in the vapor phase were determined by using a modified version of the still described by Boublik and Benson (12). The still and a water ebullometer were attached to a large vessel that could be maintained at the required constant pressure. P was calculated from the boiling point of water in the ebullometer. The latter could be determined with an accuracy of 0.01 K with a Digitec digital thermometer. The temperature in the still was also measured to 0.01 K with a cal-

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Table I. Densities ρ_i and Vapor Pressure P_i of Pure Components at 298.15 K

compound	$\rho_i/(10^{-3} \text{ kg m}^{-3})$		P_i/kPa	
	this work	lit.	this work	lit.
2-propanol (1)	0.7800	0.7807 (17)	5.81	5.81 (18)
methyl acetate (2)	0.9272	0.9274 (19)	28.86	28.86 (19)
dichloromethane (3)	1.3151	1.3168 (19)	57.85	58.29 (20)

brated thermometer. The analysis of the liquid and condensed vapor were carried out by using a Perkin Elmer-type Sigma 300 gas chromatograph, equipped with a thermal conductivity detector. The current supplied to the detector was 150 mA. The copper column was 180 cm long, supplied ready-packed with 1.5% Carbowax 20M over graphitized carbon black 70/100. The injector and detector temperatures were 120 and 150 °C, respectively. Hydrogen was used as the carrier gas. Calibration analyses were carried out with 15 samples of known compositions, three injections for each sample were made, and an LC-1-100 integrator was used to calculate areas. The sample concentrations were accurate to better than 0.5 mol %.

The materials used for the experiments were purified as described previously (1-4). Mixtures were prepared by mixing weighted amounts of the pure liquids. Caution was taken to prevent evaporation.

Results

The experimental densities ρ_i and vapor pressures P_i for the pure liquids are reported in Table I, together with literature values for comparison.

The experimental values of x_i , y_i , and P are listed in Table II. The γ_i values (Table II) were calculated from the experimental data, taking into account the nonideality of the vapor phase, by employing the following equation (13):

$$\ln \gamma_i = \ln \left(\frac{y_i P}{x_i P_i} \right) + \frac{(B_{ii} - V_i)(P - P_i)}{RT} + \frac{P}{2RT} \sum_{j=1}^n \sum_{k=1}^n y_j y_k (2\delta_{jk} - \delta_{jk}) \quad (1)$$

where

$$\delta_{jk} = 2B_{jk} - B_j - B_k \quad (2)$$

The values of the molar virial coefficients B_{ij} of the pure components and the cross virial coefficients for the binary mixtures are as follows: $B_{11} = -2408 \times 10^{-6} \text{ m}^3 \text{ mol}^{-1}$; $B_{22} = -1626 \times 10^{-6} \text{ m}^3 \text{ mol}^{-1}$; $B_{33} = -862 \times 10^{-6} \text{ m}^3 \text{ mol}^{-1}$; $B_{12} = -2381 \times 10^{-6} \text{ m}^3 \text{ mol}^{-1}$; $B_{13} = -1638 \times 10^{-6} \text{ m}^3 \text{ mol}^{-1}$; $B_{23} = -1239 \times 10^{-6} \text{ m}^3 \text{ mol}^{-1}$. These values were estimated from the Hayden and O'Connell method (14). The molar volumes V_i were calculated from the densities ρ_i (Table I).

The ternary data reported in Table II were found to be thermodynamically consistent, as tested by the McDermot-Ellis method (15), following Wisniak and Tamir (16). Two experimental points are considered thermodynamically consistent if the following condition are fulfilled:

$$D < D_{\max} \quad (3)$$

where

$$D = \sum_{i=1}^n (x_{ia} + x_{ib}) (\ln \gamma_{ib} - \ln \gamma_{ia}) \quad (4)$$

and

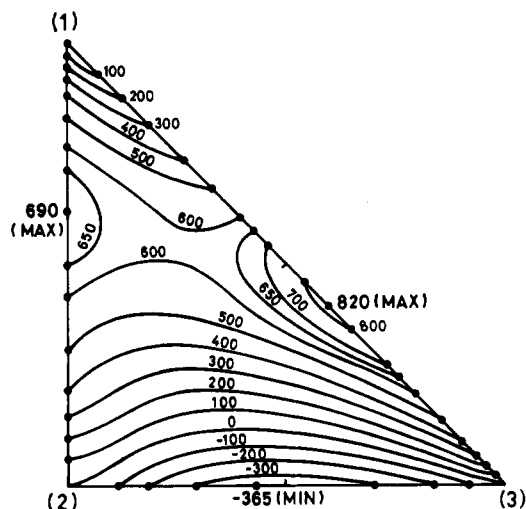
$$D_{\max} = \sum_{i=1}^n (x_{ia} + x_{ib}) \left(\frac{1}{x_{ib}} + \frac{1}{y_{ib}} + \frac{1}{x_{ia}} + \frac{1}{y_{ia}} \right) \Delta x + 2 \sum_{i=1}^n |(\ln \gamma_{ib} - \ln \gamma_{ia})| \Delta x + \sum_{i=1}^n (x_{ia} + x_{ib}) \frac{\Delta P}{P} \quad (5)$$

Table II. Experimental Vapor-Liquid Equilibria Data, Liquid Mole Fraction x_i , Vapor Mole Fraction y_i , and Pressure P for the 2-Propanol (1) + Methyl Acetate (2) + Dichloromethane (3) System at 298.15 K

x_1	x_2	y_1	y_2	P/Pa	γ_1	γ_2	γ_3
0.179	0.697	0.061	0.773	28290	1.613	1.087	0.662
0.185	0.386	0.072	0.259	36020	2.335	0.833	0.979
0.200	0.661	0.069	0.732	28380	1.638	1.089	0.170
0.204	0.272	0.077	0.134	40350	2.528	0.683	1.057
0.228	0.143	0.072	0.048	46000	2.400	0.528	1.117
0.246	0.370	0.075	0.287	34900	1.774	0.934	1.011
0.272	0.190	0.085	0.077	42080	2.180	0.585	1.140
0.298	0.193	0.077	0.108	41010	1.758	0.788	1.142
0.325	0.219	0.084	0.140	38700	1.663	0.851	1.147
0.329	0.220	0.083	0.147	38530	1.616	0.886	1.146
0.330	0.564	0.126	0.728	25560	1.639	1.145	0.610
0.337	0.268	0.094	0.178	36150	1.681	0.833	1.155
0.396	0.212	0.096	0.149	37140	1.499	0.899	1.246
0.400	0.298	0.104	0.283	32510	1.413	1.066	1.152
0.401	0.394	0.120	0.479	28200	1.415	1.188	0.965
0.424	0.318	0.115	0.320	30980	1.408	1.078	1.185
0.425	0.367	0.128	0.435	28360	1.433	1.164	1.042
0.456	0.285	0.119	0.316	31020	1.355	1.189	1.182
0.466	0.032	0.085	0.026	44750	1.251	1.245	1.377
0.469	0.339	0.149	0.433	26830	1.432	1.188	1.022
0.491	0.281	0.126	0.387	29410	1.265	1.402	1.098
0.498	0.471	0.185	0.755	22200	1.391	1.238	0.753
0.507	0.070	0.105	0.027	40260	1.399	0.533	1.436
0.516	0.151	0.119	0.135	34890	1.343	1.075	1.363
0.544	0.168	0.123	0.178	33170	1.254	1.213	1.405
0.552	0.037	0.099	0.040	40290	1.201	1.496	1.469
0.565	0.132	0.127	0.130	33370	1.254	1.134	1.428
0.569	0.349	0.219	0.655	21220	1.380	1.387	0.572
0.654	0.214	0.219	0.346	22700	1.384	1.276	1.311
0.713	0.040	0.153	0.032	30410	1.095	0.841	1.752
0.717	0.096	0.188	0.159	25570	1.130	1.469	1.563
0.826	0.065	0.265	0.181	19730	1.074	1.913	1.759
0.835	0.125	0.333	0.429	16160	1.096	1.937	1.689
0.936	0.039	0.580	0.190	9998	1.602	1.707	1.619

Table III. Coefficients a_k , Equation 7, and Standard Deviations σ , Equation 8, Determined by the Least-Squares Method for the 2-Propanol (1) + Methyl Acetate (2) + Dichloromethane (3) System at 298.15 K

system	a_0	a_1	a_2	a_3	a_4	a_5	σ	ref
1 + 2	2590	1221	1350	-2145	-1070	3523	8	3
1 + 3	3048	-2326	-1607	4607	2998	-5658	18	2
2 + 3	-1441	-291	-582	1400	1365		7	4

**Figure 1.** Lines of constant excess molar Gibbs energies G^E , in J mol^{-1} , for the 2-propanol (1) + methyl acetate (2) + dichloromethane (3) system at 298.15 K.

The errors in the measurements are $\Delta x = 0.005$ and $\Delta P = 53 \text{ Pa}$. Every pair of points must obey the condition where $x \leq 0.1$.

The excess molar Gibbs energy G_{123}^E of the ternary system is given by

$$G_{123}^E = RT \sum_{i=1}^3 x_i \ln \gamma_i \quad (6)$$

Figure 1 shows the lines of constant G_{123}^E .

The G_{ij}^E of the binary systems were represented by

$$G_{ij}^E = x_i x_j \sum_{k=0}^n a_k (x_i - x_j)^k \quad (7)$$

The least-squares method was used to determine the values of the coefficients a_k . In each case, the optimum number n of coefficients was ascertained from an examination of the variation of the standard error of estimate σ with n :

$$\sigma = [\sum (G_{\text{obs}}^E - G_{\text{cal}}^E)^2 / (N - n - 1)]^{1/2} \quad (8)$$

where N is the number of experimental data. The a_k and σ values are summarized in Table III for the three binary systems.

The best agreement with experimental data was obtained with the equations of Cibulka (10) and Singh et al. (11), given below:

$$G_{123}^E = G_{12}^E + G_{13}^E + G_{23}^E + x_1 x_2 x_3 (A + Bx_1 + Cx_2) \quad (9)$$

$$G_{123}^E = G_{12}^E + G_{13}^E + G_{23}^E + x_1 x_2 x_3 [A + Bx_1(x_2 - x_3) + Cx_1^2(x_2 - x_3)^2] \quad (10)$$

Table IV. Standard Deviations in G^E for the 2-Propanol (1) + Methyl Acetate (2) + Dichloromethane (3) System at 298.15 K

equation	$\sigma / (\text{J mol}^{-1})$	equation	$\sigma / (\text{J mol}^{-1})$
Radojkovič et al. (6)	32	Tsao and Smith (9)	53
Kohler (7)	36	Cibulka (10), eq 9	19
Jacob and Fitzner (8)	32	Singh et al. (11), eq 10	27

where A , B , and C are parameters characteristic of the mixture evaluated by fitting the equation by the least-squares method with a standard deviation defined as in eq 8.

The parameters obtained for this system are

for eq 9

$$A = 4273; \quad B = 8353; \quad C = 3643$$

for eq 10

$$A = -636; \quad B = 5311; \quad C = 39034$$

Table IV shows the standard deviations calculated by applying the equations proposed in refs 5–11 for the ternary system. Obviously, the models requiring ternary parameters are superior to the predictive ones (not containing ternary parameters).

Acknowledgment

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Glossary

a_k	coefficients of eq 7
A, B, C	adjustable parameters in eqs 9 and 10
B_{ij}, B_{ij}^E	B_{ij}, B_{ij}^E
G^E	Excess molar Gibbs energy, J mol^{-1}
P	total pressure, Pa
P_i	pure-component vapor pressure, Pa
R	molar gas constant, $8.314 \text{ J K}^{-1} \text{ mol}^{-1}$
T	temperature, K
V_i	molar volume of component i , $\text{m}^3 \text{ mol}^{-1}$
x_i^0	mole fraction of component i in the binary system
x_i, y_i	mole fraction of component i in the liquid and vapor phase

Greek Letters

γ_i	activity coefficient of component i
ρ	density, kg m^{-3}
σ	standard deviation, eq 8

Registry No. *iso*-PrOH, 67-63-0; CH_2Cl_2 , 75-09-2; MeOAc, 79-20-9.

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Excess Viscosities of Binary Mixtures of Chloroform and Alcohols

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Viscosities of mixtures of chloroform with methanol, ethanol, and 1-propanol were measured at 30, 38, and 46 °C. At least 5 mole fractions, covering the range, were studied for each liquid pair. Excess viscosities show the interesting result of being positive at some mole fractions and negative at other mole fractions.

Introduction

The heat of mixing of most binary pairs of liquids either is exothermic throughout the entire range of mole fractions or is endothermic for all mole fractions. Mixtures of chloroform and alcohols are unusual, in that ΔH_{mix} is negative when the mole fraction of chloroform is small and positive when the mole fraction of chloroform is large (1-4).

Other properties of these systems also exhibit interesting behavior. The volumes of mixing of solutions of chloroform with either ethanol or 1-propanol also undergo a sign change as mole fraction is varied (5). This behavior is consistent with the enthalpy measurements, in that ΔV_{mix} is positive over approximately the same mole fraction range that ΔH_{mix} is positive. Both of these quantities have been interpreted in terms of the relative magnitude of the interactions between molecules in the two pure liquids compared with the interactions in the mixture (6). On the other hand, the $\text{CHCl}_3\text{-CH}_3\text{OH}$ system exhibits a volume of mixing that is negative for all mole fractions (5).

Both methanol and ethanol have a minimum boiling azeotrope with chloroform (7). Such azeotropes generally mean that the forces between unlike molecules in the binary pair are weak compared with forces between like molecules (8). This is consistent with endothermic mixing, but not with exothermic mixing.

Viscosity also depends on the magnitude of interactions in a fluid. Thus, as part of our research program in viscosity measurements (9), we have undertaken a study of the viscosities of these interesting systems in an attempt to better understand the reasons for their unusual behavior.

Experimental Section

Reagent-grade chloroform, methanol, and 1-propanol were purified by simple distillation. Anhydrous ethanol was kept dry by storage over no. 4 molecular sieves.

Flow times were measured to 100th of a second by using a Schott Gerate AVS 300 viscosity measuring unit. The Ubbelohde viscometer tubes were calibrated with use of doubly

Table I. Viscosities and Densities of Mixtures of Chloroform and Alcohols at 303, 311, and 319 K

X_{CHCl_3}	303 K		311 K		319 K	
	d , g/mL	η , cP	d , g/mL	η , cP	d , g/mL	η , cP
Methanol						
0.0000	0.7835	0.5126	0.7763	0.4603	0.7608	0.4116
0.0960	0.9018	0.5644	0.8925	0.5043	0.8836	0.4586
0.1963	1.0177	0.6289	1.0060	0.5549	0.9949	0.4932
0.2971	1.1018	0.6315	1.0883	0.5593	1.0783	0.5022
0.4963	1.2380	0.5872	1.2247	0.5271	1.2120	0.4779
0.6952	1.3441	0.5447	1.3296	0.4971	1.3151	0.4574
0.9092	1.4321	0.5211	1.4168	0.4822	1.4010	0.4489
1.0000	1.4628	0.5249	1.4472	0.4893	1.4315	0.4571
Ethanol						
0.0000	0.7813	0.9817	0.7743	0.8481	0.7672	0.7348
0.1074	0.8718	0.9539	0.8626	0.8217	0.8536	0.7107
0.2892					1.0034	0.6097
0.2992	1.0352	0.8095	1.0209	0.7021		
0.5029	1.1766	0.6591	1.1593	0.5849	1.1457	0.5234
0.6978	1.3016	0.5679	1.2875	0.5169	1.2715	0.4922
0.9003	1.4120	0.5256	1.3969	0.4864	1.3817	0.4517
1.0000	1.4638	0.5254	1.4485	0.4893	1.4330	0.4566
1-Propanol						
0.0000	0.7979	1.7229	0.7913	1.4295	0.7851	1.1971
0.0987	0.8676	1.5382	0.8600	1.2831	0.8518	1.0802
0.2941					0.9818	0.8178
0.2987	1.0079	1.0650	0.9957	0.9064		
0.4970					1.1175	0.6404
0.4992	1.1400	0.8162	1.1295	0.7227		
0.6990	1.2707	0.6346	1.2567	0.5734	1.2406	0.5202
0.9085	1.4035	0.5383	1.3889	0.4977	1.3730	0.4655
1.0000	1.4628	0.5249	1.4472	0.4893	1.4315	0.4571

Table II. Literature Values of Viscosities (cP) of Pure Alcohols at 30 °C

ref	CH_3OH	$\text{C}_2\text{H}_5\text{OH}$	$\text{C}_3\text{H}_7\text{OH}$
10	0.513	0.994	1.728
this work	0.5126	0.9817	1.7229
11 ^a	0.5091	0.9628	1.6517

^aData from ref 11 fit by least-squares to give the following equations: CH_3OH , $\ln \eta = 1247.6(1/T) - 4.7928$ ($r = 0.9999$); $\text{C}_2\text{H}_5\text{OH}$, $\ln \eta = 1667.3(1/T) - 5.5406$ ($r = 0.9999$); $\text{C}_3\text{H}_7\text{OH}$, $\ln \eta = 2022.1(1/T) - 6.1721$ ($r = 0.9978$).

deionized water. Temperatures were maintained to ± 0.02 °C with a Schott Gerate CT050 temperature controller.

Densities were determined to ± 0.0002 g/cm³ by using a Mettler Parr DMA 45 digital density meter, calibrated with water at each temperature. Temperature was controlled to ± 0.01