JOURNAL OF
CHEMICAL &
ENGINEERING
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Vapor–Liquid Equilibrium Data for Methanol + *tert*-Butylamine + *N*,*N*-Dimethylformamide and Constituent Binary Systems at Atmospheric Pressure

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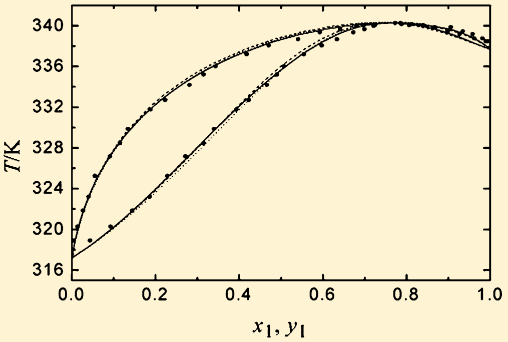
ABSTRACT: Vapor–liquid equilibrium (VLE) data for methanol + *tert*-butylamine + *N*,*N*-dimethylformamide (DMF) and constituent binary systems containing *tert*-butylamine at 101.3 kPa were measured. A maximum boiling point azeotrope was observed at the methanol mole fraction of 0.763, corresponding to the temperature of 340.28 K for the binary system methanol + *tert*-butylamine. The binary equilibrium data were correlated using Wilson, nonrandom two-liquid (NRTL), and universal quasichemical activity coefficient (UNIQUAC) models, respectively. The VLE data of the ternary system were calculated with the binary interaction parameters of the NRTL model, and the average absolute deviations of the equilibrium temperature and the vapor-phase mole fractions for methanol and *tert*-butylamine were about 0.87 K, 0.014, and 0.018, respectively. The results show that DMF can break the azeotrope of the methanol + *tert*-butylamine system as a solvent.



INTRODUCTION

Methanol and *tert*-butylamine are important organic solvents and are used commonly as mixed solvents in the pharmaceutical industry. It is of great significance to recover and purify them from process mixture. An azeotropic behavior of the binary mixture of methanol and *tert*-butylamine is observed in the process of recovery by distillation. Therefore, it is impossible to obtain high-purity methanol and *tert*-butylamine from their mixture by common distillation. Extractive distillation is an effective way for the separation of azeotropic mixtures, and *N*,*N*-dimethylformamide (DMF) has a good selectivity for the above-mentioned binary mixture. It is extremely important to determine the vapor–liquid equilibrium (VLE) data for the ternary system methanol + *tert*-butylamine + DMF and the constituent binary systems for the related process design and optimization. The VLE data for methanol + DMF at 101.3 kPa was found in the literature,[1] while VLE data of the ternary system and binary systems containing *tert*-butylamine at 101.3 kPa have not been reported.

The VLE data for the binary systems of methanol + *tert*-butylamine and *tert*-butylamine + DMF and the ternary system of methanol + *tert*-butylamine + DMF at 101.3 kPa were reported in this paper. The binary systems data were verified by the thermodynamic consistency. Binary interaction parameters adjusted on these binary systems were obtained by correlating with the Wilson, nonrandom two-liquid (NRTL), and universal quasichemical activity coefficient (UNIQUAC) equations, and the VLE data of the ternary system were predicted by the binary interaction parameters of the NRTL model.

EXPERIMENTAL SECTION

Materials. Methanol and 2-propanol were supplied by Shanghai Reagent Co. (Shanghai, China). *tert*-Butylamine and DMF were supplied by Aladdin (Shanghai, China). All liquids were subjected to no further purification. The purities of these chemicals tested by gas chromatography were methanol, 99.8 mass %, 2-propanol, 99.7 mass %, *tert*-butylamine, 99.6 mass %, DMF, 99.9 mass %. The sample table is shown as Table 1.

Table 1. Sample Table

chemical name	source	purification method	final mass fraction purity	analysis method
methanol	Shanghai Reagent Co.	none	0.998	GC
tert-butylamine	Aladdin	none	0.996	GC
2-propanol	Shanghai Reagent Co.	none	0.997	GC
DMF	Aladdin	none	0.999	GC

The mass fraction of water for all chemicals determined by Karl Kischer titration was less than 0.001. Antoine constants A_i, B_i, and C_i for the pure components, as shown in Table 2, are obtained from literature.[2,3]

Apparatus and Procedure. The VLE data were measured using a modified Rose–Williams still,[4,5] At the beginning of the experiment, the chemicals were added into the boiling chamber

Received: December 20, 2011
Accepted: March 1, 2012
Published: March 19, 2012

Table 2. Antoine Constants[a] for the Pure Components, Minimum Temperature $T_{\min}$, and Maximum Temperature $T_{\max}$

component	A_i	B_i	C_i	$T_{\min}$	$T_{\max}$
methanol[2]	7.19736	1574.99	−34.29	175.47	512.5
2-propanol[3]	6.86087	1357.514	−75.786	325	363
tert-butylamine[3]	5.90597	992.175	−62.794	292	349
DMF[3]	5.37646	1049.26	−113.84	301	426

[a] Antoine equation: $\log_{10}(p_i^s) = A_i - B_i/(T + C_i)$ with p_i^s/kPa and T/K.

and heated. The vapor rose into the equilibrium chamber through the vapor−liquid lift pipe, was condensed at the condenser, and returned to the boiling chamber through the return conduit. Both the vapor and the liquid phases were continuously recirculating to provide intimate phase contact and ensure the rapid equilibrium establishment.

The equilibrium temperature was measured with a precise mercury thermometer whose uncertainty is ± 0.1 K. In each run, equilibrium was assumed when constant equilibrium temperature had been kept for more than 30 min. The pressure was maintained at 101.3 ± 0.05 kPa by using a U-shaped differential manometer with a pressure control system. Finally, samples of vapor (cooled to liquid) and liquid phase were taken from the sampling ports and analyzed, respectively.

The compositions of vapor and liquid samples were analyzed by a gas chromatograph GC-14B (Shimadzu Co., Japan) equipped with a flame ionization detector (FID) and the calibration was carefully carried out with gravimetrically prepared standard solutions purchased from Aladdin. The GC column was a SE-30 capillary column (30 m × 0.32 mm × 1 μm) which was provided by Lanzhou Institute of Chemical Physics, Chinese Academy of Sciences. High-purity nitrogen (99.999 %) was used as the carrier gas. Each sample was analyzed at least three times to ensure the accuracy. The maximum uncertainty of mole fraction was ± 0.002.

■ RESULTS AND DISCUSSION

Experimental Reliability Test. To ensure the reliability of the experimental apparatus and the procedure, VLE data of the binary system of methanol (1) + 2-propanol (2) at 101.3 kPa were measured, as shown in Table 3. The measured VLE data were compared with the values in the literature,[6] as shown in Figure 1. As can be seen, the experimental data agree well with the literature data, indicating that the experimental apparatus and the procedure are reliable.

Table 3. Experimental VLE Data for Temperature T, Liquid Mole Fractions x_i, Vapor Mole Fractions y_i, and Activity Coefficients γ_i for Methanol (1) + 2-Propanol (2) at 101.3 kPa

no.	T/K	x_1	y_1	γ_1	γ_2
1	354.69	0.043	0.071	0.881	0.998
2	354.27	0.068	0.111	0.881	0.997
3	353.43	0.122	0.194	0.880	0.993
4	352.82	0.151	0.239	0.897	0.994
5	351.73	0.217	0.332	0.898	0.991
6	350.78	0.268	0.399	0.907	0.991
7	349.85	0.313	0.458	0.921	0.990
8	348.65	0.372	0.532	0.940	0.984
9	348.01	0.405	0.568	0.944	0.985
10	347.57	0.443	0.604	0.933	0.982
11	346.45	0.492	0.658	0.956	0.973
12	344.93	0.583	0.738	0.957	0.971
13	343.25	0.683	0.821	0.969	0.937
14	341.34	0.804	0.900	0.970	0.920
15	340.15	0.889	0.951	0.971	0.841
16	339.07	0.958	0.983	0.972	0.792

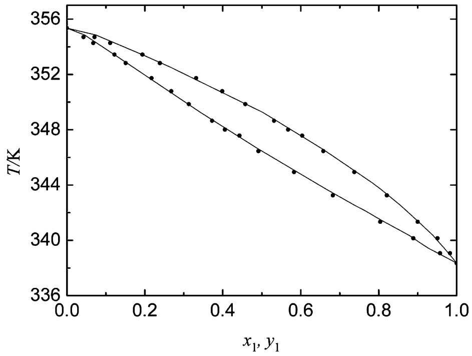


Figure 1. T−x_1−y_1 phase equilibrium for methanol (1) + 2-propanol (2) binary system at 101.3 kPa: —, literature data; ●, experimental data.

Experimental Results and Consistency Test. The VLE data for binary systems of methanol (1) + *tert*-butylamine (2) and *tert*-butylamine (1) + DMF (2) at 101.3 kPa were measured; the experimental data are shown in Tables 4 and 5. A maximum boiling point azeotrope was observed at the methanol mole fraction of 0.763, corresponding to the temperature of 340.28 K

Table 4. Experimental VLE Data for Temperature T, Liquid Mole Fractions x_i, Vapor Mole Fractions y_i, and Activity Coefficients γ_i for Methanol (1) + *tert*-Butylamine (2) at 101.3 kPa

no.	T/K	x_1	y_1	γ_1	γ_2
1	337.85	1.000	1.000	0.993	0.199
2	338.45	0.990	0.996	0.975	0.204
3	338.75	0.963	0.982	0.977	0.239
4	339.17	0.928	0.959	0.974	0.281
5	339.46	0.899	0.935	0.969	0.313
6	339.85	0.868	0.907	0.958	0.340
7	340.06	0.807	0.841	0.948	0.394
8	340.15	0.789	0.812	0.934	0.423
9	340.25	0.774	0.786	0.918	0.449
10	340.05	0.725	0.698	0.877	0.524
11	339.97	0.722	0.681	0.862	0.548
12	339.65	0.700	0.641	0.848	0.577
13	339.35	0.674	0.593	0.824	0.607
14	338.66	0.635	0.542	0.821	0.624
15	338.05	0.598	0.471	0.776	0.666
16	337.20	0.556	0.418	0.767	0.680
17	336.00	0.508	0.344	0.724	0.723
18	335.20	0.491	0.316	0.709	0.748
19	334.18	0.467	0.282	0.693	0.774
20	332.70	0.424	0.223	0.643	0.804
21	331.75	0.393	0.189	0.608	0.822
22	329.86	0.340	0.135	0.543	0.884
23	328.45	0.315	0.116	0.533	0.883
24	327.16	0.272	0.092	0.519	0.896
25	325.25	0.229	0.055	0.402	0.930
26	323.20	0.187	0.041	0.395	0.959
27	321.85	0.145	0.028	0.366	0.968
28	320.26	0.093	0.014	0.305	0.977
29	318.90	0.044	0.005	0.271	0.979
30	317.55	0.000	0.000	0.232	0.987

Table 5. Experimental VLE Data for Temperature T, Liquid Mole Fractions x_i, Vapor Mole Fractions y_i, and Activity Coefficients γ_i for *tert*-Butylamine (1) + DMF (2) at 101.3 kPa

no.	T/K	x_1	y_1	γ_1	γ_2
1	317.55	1.000	1.000	0.987	2.331
2	318.03	0.963	0.999	1.007	2.174
3	320.80	0.852	0.994	1.030	1.851
4	321.25	0.837	0.994	1.032	1.774
5	322.65	0.768	0.991	1.069	1.684
6	323.85	0.732	0.989	1.075	1.651
7	326.65	0.625	0.983	1.139	1.625
8	328.65	0.554	0.977	1.197	1.685
9	329.90	0.505	0.978	1.264	1.328
10	335.20	0.389	0.965	1.370	1.335
11	338.38	0.333	0.959	1.442	1.209
12	340.12	0.315	0.958	1.446	1.127
13	341.04	0.295	0.955	1.498	1.106
14	341.95	0.277	0.953	1.553	1.086
15	342.85	0.255	0.950	1.633	1.084
16	345.70	0.227	0.941	1.677	1.079
17	349.70	0.195	0.931	1.728	1.015
18	356.03	0.157	0.906	1.754	1.013
19	359.93	0.129	0.886	1.886	1.012
20	366.85	0.101	0.848	1.938	1.004
21	369.95	0.088	0.828	2.002	0.997
22	374.95	0.074	0.790	2.040	0.997
23	381.45	0.060	0.741	2.035	0.971
24	393.45	0.035	0.612	2.198	0.961
25	425.15	0.000	0.000	2.130	0.992

Table 6. Results of the Consistency Test for the Binary Systems[a]

	Herington test			Point test	
system	D	J	$D-J$	Δp/kPa	Δy
methanol (1) + 2-propanol (2)	0.866	7.631	−6.765	0.57	0.009
methanol (1) + *tert*-butylamine (2)	3.791	9.589	−5.798	0.93	0.009
tert-butylamine (1) + DMF (2)	13.339	35.572	−22.233	no test	no test

[a] $\Delta p = (1/n)\sum_{j=1}^{n}|p_{j,\mathrm{cal}} - p_{j,\mathrm{exp}}|$, $\Delta y = (1/n)\sum_{j=1}^{n}|y_{j,\mathrm{cal}} - y_{j,\mathrm{exp}}|$.

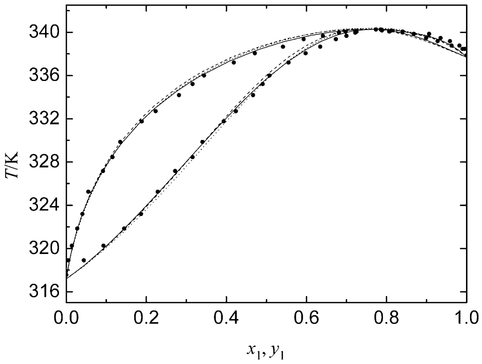


Figure 2. $T-x_1-y_1$ phase equilibrium for the system methanol (1) + *tert*-butylamine (2) at 101.3 kPa: ●, Experimental data; —, NRTL model; ---, Wilson model; ···, UNIQUAC model.

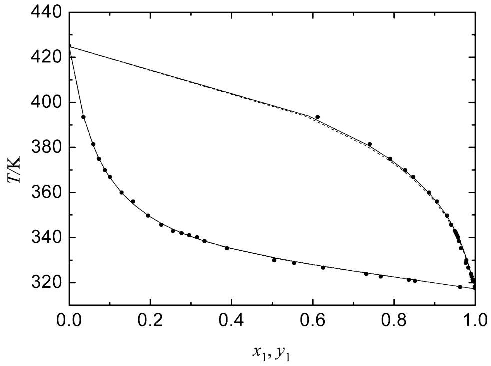


Figure 3. $T-x_1-y_1$ phase equilibrium for the system *tert*-butylamine (1) + DMF (2) at 101.3 kPa: ●, Experimental data; —, NRTL model; ---, Wilson model; ···, UNIQUAC model.

Table 7. Correlation Parameters for the Binary Systems Using the Wilson, NRTL, and UNIQUAC Equations at 101.3 kPa[a]

	A_{12}	A_{21}	
model	J·mol^{-1}	J·mol^{-1}	α
	Methanol (1) + *tert*-Butylamine (2)		
Wilson	−0.239	1.381	no parameter
NRTL	−147.488	−187.170	0.3
UNIQUAC	330.634	−348.516	no parameter
	tert-Butylamine (1) + DMF (2)		
Wilson	−0.331	−0.708	no parameter
NRTL	122.496	214.032	0.3
UNIQUAC	−52.615	−49.895	no parameter

[a] Wilson: $A_{12} = (\lambda_{12} - \lambda_{11})$, $A_{21} = (\lambda_{21} - \lambda_{22})$; NRTL: $A_{12} = (g_{12} - g_{22})$, $A_{21} = (g_{21} - g_{11})$; UNIQUAC: $A_{12} = (u_{12} - u_{22})$, $A_{21} = (u_{21} - u_{11})$.

for the binary system methanol + *tert*-butylamine. The Herington method[7] was adopted to verify the quality of all the binary experimental data. Herington suggested that:

$$D = 100\cdot\left|\frac{S_A - S_B}{S_A + S_B}\right| \tag{1}$$

$$J = 150\cdot\frac{T_{\max} - T_{\min}}{T_{\min}} \tag{2}$$

where S_A and S_B are from the area of the $\ln(\gamma_1 - \gamma_2) - x_1$ diagram, and $T_{\max}$ and $T_{\min}$ are the maximum and minimum temperatures of system, respectively. If $D - J < 10$, then the experimental data meet the thermodynamics consistency. Besides, the point test of Van Ness et al.[8] in the version of Fredeslund et al.[9] is also used to verify the thermodynamic consistency of VLE data of binary systems. Fredeslund et al. think that the excess Gibbs energy G^E/RT can be expressed as a Legendre polynomial expansion, and then the calculated value of the composition of the vapor phase and system pressure can be obtained by the vapor—liquid equilibrium equation. If the deviation of the calculated vapor-phase mole fraction to the experimental value is less than 0.01 and that of the system pressure is less than 1.01 kPa, then the experimental data pass the thermodynamics consistency test.

The results of consistency test are shown in Table 6. The results show that the experimental data meet the thermodynamics consistency test, which indicates that experimental data for all binary systems are reliable.

Correlation of the Binary Data. The activity coefficients γ_i were calculated from the vapor–liquid equilibrium equation[10]

$$py_i\hat{\varphi}_i^{\mathrm{V}} = p_i^{\mathrm{s}}\hat{\varphi}_i^{\mathrm{s}}x_i\gamma_i \exp\left[\frac{V_i^{\mathrm{L}}(p - p_i^{\mathrm{s}})}{RT}\right] \tag{3}$$

where p and T are the system pressure and equilibrium temperature, respectively; x_i and y_i are the liquid-phase and vapor-phase mole fraction of component i, respectively; γ_i is the activity coefficients of component i; p_i^{s} is the saturated vapor pressure of pure liquid i at T, which can be obtained from the Antoine equation. $\hat{\varphi}_i^{\mathrm{s}}$ is the fugacity coefficient of pure vapor component i at T and p_i^{s};$\hat{\varphi}_i^{\mathrm{V}}$ is the fugacity coefficient of component i in the vapor phase; V_i^{L} is the mole volume of pure liquid i.

At low or moderate pressure, the pointing factor term $\exp[V_i^{\mathrm{L}}(p - p_i^{\mathrm{s}})/RT]$ is close to unity. The vapor phase is considered as ideal gas, and the liquid phase is regarded as nonideal mixture; then the equilibrium equation can be expressed as

$$py_i = p_i^{\mathrm{s}}x_i\gamma_i \tag{4}$$

where p_i^{s} can be calculated using the Antoine equation. The activity coefficients γ_i can be correlated by using the Wilson,[11] NRTL,[12] and UNIQUAC[13] equations. Binary interaction parameters for the three equations can be obtained by the experimental data. The nonlinear least-squares method is used to optimize the parameters of the three equations. The objective function for the calculation is given by

$$F = \frac{1}{n}\sum_{i=1}^{n}\left[\left|\frac{y_{\mathrm{cal}} - y_{\mathrm{exp}}}{y_{\mathrm{exp}}}\right|_i + \left|\frac{T_{\mathrm{cal}} - T_{\mathrm{exp}}}{T_{\mathrm{exp}}}\right|_i\right] \tag{5}$$

where n is the number of experimental points; y is the mole fraction of the vapor phase; T is the equilibrium temperature, K; and "cal" and "exp" represent calculated and experimental values, respectively.

The calculations from the Wilson, NRTL and UNIQUAC equations are shown in Figures 2 and 3. Binary interaction parameters for methanol (1) + *tert*-butylamine (2) and *tert*-butylamine (1) + DMF (2) binary systems correlated using the Wilson, NRTL, and UNIQUAC equations are listed in Table 7. The deviations between the calculated results using three different equations and the experimental results are listed in Table 8. As can be seen, the correlated results are very satisfactory.

Table 8. Correlation Deviations in Equilibrium Temperature and Vapor-Phase Composition for the Binary Systems Using the Wilson, NRTL, and UNIQUAC Equations[a]

model	Δy_{ave}	Δy_{max}	ΔT_{ave}	ΔT_{max}
Methano l(1) + *tert*-Butylamine (2)				
Wilson	0.007	0.033	0.40	0.93
NRTL	0.005	0.018	0.24	0.59
UNIQUAC	0.007	0.019	0.32	0.70
tert-Butylamine (1) + DMF (2)				
Wilson	0.004	0.028	0.42	1.07
NRTL	0.002	0.022	0.44	1.13
UNIQUAC	0.003	0.023	0.46	1.19

[a] $\Delta y_{\mathrm{ave}} = (1/n)\sum_{j=1}^{n}|y_{j,\mathrm{cal}} - y_{j,\mathrm{exp}}|$, $\Delta y_{\mathrm{max}} = \max\{|y_{j,\mathrm{cal}} - y_{j,\mathrm{exp}}|\}$, $\Delta T_{\mathrm{ave}} = (1/n)\sum_{j=1}^{n}|T_{j,\mathrm{cal}} - T_{j,\mathrm{exp}}|$, $\Delta T_{\mathrm{max}} = \max\{|T_{j,\mathrm{cal}} - T_{j,\mathrm{exp}}|\}$.

Measured and Predicted VLE Data for Ternary Systems. The binary interaction parameters for methanol (1) + *tert*-butylamine (2) and *tert*-butylamine (1) + DMF (2) correlated with the NRTL equation, and the binary parameters for methanol (1) + DMF (2) from the literature[1] were used to predict the VLE data of the ternary system methanol (1) + *tert*-butylamine (2) + DMF (3). The predicted data for ternary system were compared with experimental data, and the results indicate that the average absolute deviations of the equilibrium

Table 9. Experimental VLE Data for Temperature T, Liquid Mole Fractions x_i, Vapor Mole Fractions y_i, Relative Volatility α_{21}, and NRTL Prediction Deviations for Methanol (1) + *tert*-Butylamine (2) + DMF (3) Ternary System at 101.3 kPa[a]

no.	T/K	x_1	x_2	y_1	y_2	ΔT	Δy_1	Δy_2	α_{21}
1	329.45	0.029	0.575	0.014	0.974	0.84	0.007	0.002	3.472
2	331.85	0.068	0.529	0.026	0.967	1.32	0.007	0.006	4.738
3	336.15	0.162	0.459	0.118	0.876	1.80	0.052	0.037	2.616
4	340.15	0.265	0.381	0.197	0.800	1.46	0.042	0.022	2.828
5	341.95	0.334	0.336	0.253	0.744	0.87	0.015	0.005	2.925
6	343.35	0.379	0.303	0.329	0.664	0.75	0.024	0.008	2.528
7	345.35	0.454	0.244	0.426	0.568	0.55	0.012	0.028	2.479
8	346.05	0.466	0.228	0.482	0.510	0.64	0.015	0.000	2.159
9	346.55	0.518	0.218	0.547	0.446	1.62	0.012	0.001	1.940
10	346.95	0.558	0.174	0.638	0.357	0.94	0.010	0.004	1.794
11	347.05	0.654	0.108	0.795	0.202	1.17	0.001	0.011	1.540
12	350.05	0.290	0.175	0.334	0.634	1.56	0.017	0.004	3.143
13	351.25	0.349	0.165	0.411	0.568	0.28	0.007	0.020	2.920
14	351.75	0.385	0.149	0.479	0.499	0.20	0.007	0.016	2.682
15	352.65	0.427	0.120	0.565	0.417	0.35	0.008	0.033	2.626
16	353.60	0.514	0.071	0.764	0.221	0.40	0.007	0.016	2.094
17	353.65	0.592	0.025	0.902	0.083	1.31	0.002	0.019	2.152
18	362.65	0.445	0.015	0.876	0.079	1.11	0.009	0.015	2.680
19	363.25	0.367	0.043	0.724	0.227	0.08	0.021	0.014	2.682
20	365.05	0.327	0.045	0.641	0.276	0.21	0.011	0.025	3.125

[a] $\Delta T_{\mathrm{ave}} = 0.87$ K, $\Delta y_{1,\mathrm{ave}} = 0.014$, $\Delta y_{2,\mathrm{ave}} = 0.018$ where $\Delta T = |T_{\mathrm{cal}} - T_{\mathrm{exp}}|$, $\Delta y_i = |y_{i,\mathrm{cal}} - y_{i,\mathrm{exp}}|$, $\Delta T_{\mathrm{ave}} = (1/n)\sum_{j=1}^{n}|T_{j,\mathrm{cal}} - T_{j,\mathrm{exp}}|$, $\Delta y_{j,\mathrm{ave}} = (1/n)\sum_{j=1}^{n}|y_{ij,\mathrm{cal}} - y_{ij,\mathrm{exp}}|$, $\alpha_{21} = [(y_2/(y_1 + y_2))/(x_2/(x_1 + x_2))]/[(y_1/(y_1 + y_2))/(x_1/(x_1 + x_2))]$.

temperature and the vapor-phase mole fractions for methanol and *tert*-butylamine were about 0.87 K, 0.014, and 0.018, respectively, as shown in Table 9. It is found that the relative volatility of *tert*-butylamine to methanol is greater than 1 in each experimental point data after the addition of DMF to methanol + *tert*-butylamine mixtures, which means that DMF can break the azeotrope of methanol + *tert*-butylamine system as a solvent.

■ CONCLUSIONS

The VLE data for binary systems of methanol + 2-propanol, methanol + *tert*-butylamine, and *tert*-butylamine + DMF at 101.3 kPa were obtained. The experimental data for methanol + 2-propanol were agreed well with the literature data, indicating that the present experimental method was reliable. All experimental data for binary systems meet the thermodynamics consistency test. From the experimental results, a maximum boiling point azeotrope was observed at the methanol mole fraction of 0.763, corresponding to the temperature of 340.28 K for the binary system methanol + *tert*-butylamine. The binary equilibrium data were correlated using Wilson, NRTL, and UNIQUAC models, respectively. The equilibrium data for methanol + *tert*-butylamine + DMF at 101.3 kPa were measured and compared with the predicted data calculated with binary interaction parameters of the NRTL equation. The results indicate that DMF can break the azeotrope of methanol + *tert*-butylamine system as a solvent.

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Funding

This work is financially supported by Science and Technology Department of Zhejiang Province, China (No. 2009C31045) and National Natural Science Foundation of China (No. 20876145).

Notes

The authors declare no competing financial interest.

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