

# Phase Equilibrium and Excess Enthalpy Data for the System Methanol + 2,2'-Diethanolamine + Water

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Isothermal vapor–liquid equilibrium (VLE) data are reported for the binary systems methanol + 2,2'-diethanolamine and water + 2,2'-diethanolamine and for the ternary system methanol + 2,2'-diethanolamine + water. Moreover, excess enthalpies ( $H^E$ ) were measured for both binary systems with 2,2'-diethanolamine. The data were measured by means of a computer operated static apparatus and isothermal flow calorimetry, respectively. For the pure component 2,2'-diethanolamine, also vapor pressure data were measured using the dynamic ebulliometric method. The experimental binary data were correlated using linear or even quadratic temperature-dependent NRTL interaction parameters. The ternary data were used to validate the capability of  $g^E$  models such as NRTL for the prediction of multicomponent mixtures on the basis of binary information.

## Introduction

Aqueous alkanolamine solutions are popular chemical solvents for acid gas removal from natural gas. The properties of these solvents, such as their capacity, selectivity, melting points, viscosities, and so forth, can be modified by variation of the composition of the solvent blends. The aims of this study are to investigate the real phase equilibrium behavior of the ternary system methanol + 2,2'-diethanolamine + water and to test if it can be predicted with only the information on the binary subsystems.

Whereas the vapor pressure curves for methanol and water are well-known, vapor pressures for 2,2'-diethanolamine were measured using the dynamic method.

For the binary systems methanol + 2,2'-diethanolamine and water + 2,2'-diethanolamine, where only a few experimental data points are available in the literature, isothermal  $P$ - $x$  data at 365.15 K were measured with a computer controlled static apparatus. In addition to VLE data, heats of mixing data for these systems were measured by means of an isothermal flow calorimeter. The  $H^E$  data are important to describe the temperature dependence of the activity coefficients following the Gibbs–Helmholtz equation

$$\left(\frac{\partial \ln \gamma_i}{\partial 1/T}\right)_{P,x} = \frac{H_i^E}{R} \quad (1)$$

where  $H_i^E$  is the partial molar value of the excess enthalpy.

The binary VLE and  $H^E$  data from this work are compared to values of other authors and calculated results using temperature-dependent NRTL interaction parameters, which were fitted simultaneously to the experimental data.

$g^E$  models such as NRTL are able to predict the multicomponent mixture behavior using binary information only. To validate the applicability of the model, ternary VLE measurements were also performed with the static apparatus. The required binary interaction parameters for the methanol + water system were fitted to literature data.

## Experimental Section

**Materials.** Water was distilled twice. Methanol was purchased from Scharlau. Its final purity after drying over molecular sieves, degassing, and distillation was 99.99 wt %. 2,2'-Diethanolamine was purchased from Acros and dried, degassed, and distilled like methanol, as described by Fischer and Gmehling.<sup>1</sup> Its final purity was 99.97 wt %. For the  $H^E$  and vapor pressure measurements the chemicals were used without degassing. The purity and water contents were checked by gas chromatography and Karl Fischer titration, respectively.

**Apparatus and Procedures.** The pure component vapor pressures were measured by means of the dynamic method with an ebulliometer (Figure 1). With this equipment, vapor pressures of pure components can be measured in a temperature range from about 270 to 620 K and at pressures from about 0.2 kPa up to 100 kPa. At a given pressure (regulated by an electronic pressure control, MKS Instruments) the boiling temperatures of the component were measured. Because of the volume of the equipment, only small amounts of the substances (about 20 mL) are needed.

The isothermal  $P$ - $x$  data were measured with a computer driven static apparatus. The experimental procedure for the determination of the VLE data is based on that proposed by Gibbs and Van Ness,<sup>2</sup> where the total pressure  $P$  is measured for different overall compositions at constant temperature. The apparatus that has been described previously<sup>3,4</sup> can be used at temperatures between 278 and 368 K and pressures up to 0.3 MPa.

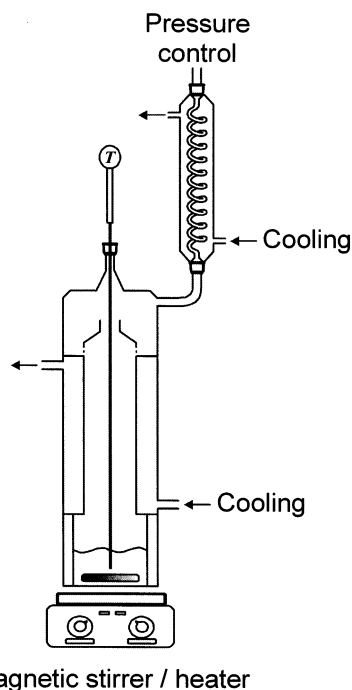
The thermostated, purified, and degassed compounds are charged into the VLE cell, which is evacuated and im-

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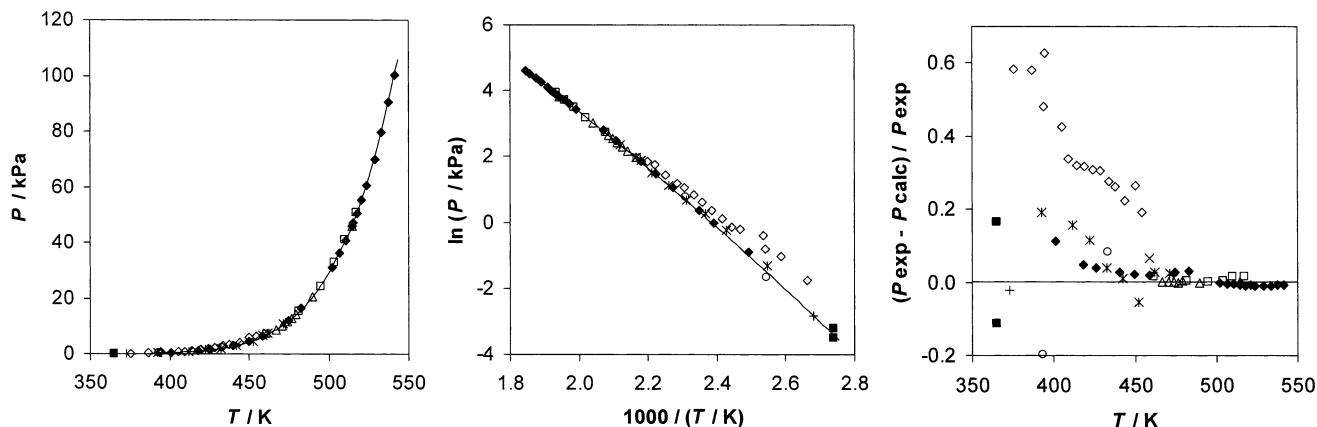


Magnetic stirrer / heater

**Figure 1.** Schematic diagram of the ebullimeter.

mersed in a thermostatic oil bath. The pressure inside the cell is monitored with a Digiquartz pressure sensor (Model 245A, Paroscientific), and the temperature is measured with a Pt100 resistance thermometer (Model 1506, Hart Scientific). The overall compositions are determined from the known quantities of liquids injected into the equilibrium cell by stepping motor driven injection pumps and automatic valves. The liquid-phase compositions are obtained by solving mass and volume balance equations taking into account the vapor–liquid equilibrium. The ternary measurements were performed by starting with binary mixtures of 2,2'-diethanolamine + water. In the evaluation of the liquid-phase composition, this mixture was also treated as a pseudocomponent. The estimated experimental uncertainties of this apparatus are as follows:  $\sigma(T) = 0.03$  K;  $\sigma(P) = 20$  Pa +  $0.0001(P/\text{Pa})$ ;  $\sigma(x_i) = 0.0001$ .

The commercial isothermal flow calorimeter (Model 7501, Hart Scientific) used for the determination of the excess enthalpy data has been described previously.<sup>5</sup> In this apparatus, two syringe pumps (Model LC-2600, ISCO) provide a flow of constant composition through a thermo-



**Figure 2.** Experimental and calculated vapor pressure data for 2,2'-diethanolamine (normal and logarithmic scale, relative deviations from Antoine): experimental data from this work from the (◆) dynamic method and from (■) static VLE measurements; experimental data of Danov et al.<sup>6</sup> (◇) and other authors<sup>7–12</sup> (□, △, ○, +, ×, \*); (—) Antoine.

**Table 1.** Experimental Vapor Pressures for 2,2'-Diethanolamine

<i>T</i> /K	<i>P</i> /kPa	<i>T</i> /K	<i>P</i> /kPa	<i>T</i> /K	<i>P</i> /kPa
401.13	0.41	482.83	16.41	520.84	55.44
418.21	0.99	502.22	31.07	523.82	60.43
425.96	1.46	506.97	36.18	528.77	69.90
440.27	2.88	510.77	40.77	533.33	79.65
449.80	4.38	514.59	45.83	537.82	90.57
458.60	6.34	515.48	47.27	541.44	100.28
474.59	12.06	517.83	50.56		

**Table 2.** Pure Component Antoine Coefficients  $A_i$ ,  $B_i$ , and  $C_i$ 

component	$A_i$	$B_i/\text{K}$	$C_i/\text{K}$
methanol	7.205 87	1582.27	−33.450
2,2'-diethanolamine	7.260 44	2326.23	−98.907
water	7.196 21	1730.63	−39.724

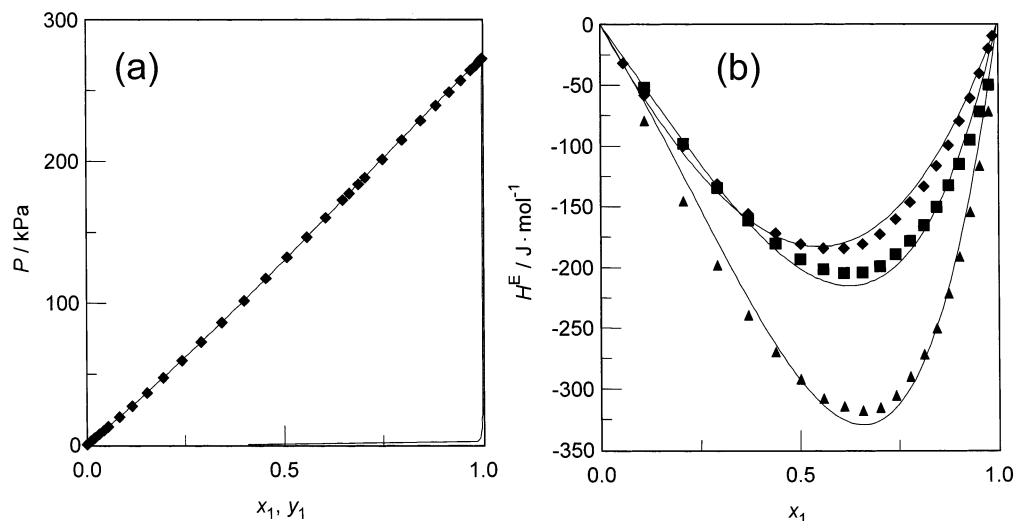
stated calorimeter cell equipped with a pulsed heater and a Peltier cooler. The Peltier cooler is working at constant power, producing a constant heat loss from the calorimeter cell, which is compensated by the pulsed heater. The required frequency is influenced by endothermal or exothermal heat effects, so that heats of mixing can be determined from the observed frequency change between the baseline and the actual measurement. A back-pressure regulator serves to keep the pressure at the desired level for which evaporation and degassing effects can be prevented. The pressure dependence of the  $H^E$  values in the investigated pressure range is not significant for liquid systems. The experimental uncertainties of this device are as follows:  $\sigma(T) = 0.03$  K;  $\sigma(H^E) = 2$  J·mol<sup>−1</sup> +  $0.01(H^E/\text{J}·\text{mol}^{-1})$ ;  $\sigma(x_i) = 0.0001$ .

## Results

For 2,2'-diethanolamine only a few experimental pure component vapor pressures are available in the literature.<sup>6–12</sup> The experimental vapor pressures from this work are listed in Table 1. Using these data and the data of other authors, coefficients for the Antoine equation for vapor pressures

$$\log(P_i^s/\text{kPa}) = A_i - \frac{B_i}{C_i + T/\text{K}} \quad (2)$$

were fitted for all three components. The parameters are given in Table 2. In Figure 2, a comparison between the new data, the data from literature, and the Antoine calculations is shown for 2,2'-diethanolamine. Except the



**Figure 3.** Experimental and predicted mixture behavior of the binary system methanol (1) + 2,2'-diethanolamine (2): (a) vapor-liquid equilibrium with experimental data from this work at (◆) 365.15 K; (b) excess enthalpies with experimental data from this work at (◆) 323.15 K, (■) 363.15 K, and (▲) 413.15 K; (---) NRTL.

**Table 3. Experimental Vapor-Liquid Equilibrium Data for the Binary System Methanol (1) + 2,2'-Diethanolamine (2) at 365.15 K**

$x_1$	$P/\text{kPa}$	$x_1$	$P/\text{kPa}$	$x_1$	$P/\text{kPa}$
0.00000	0.03	0.34321	86.33	0.84688	228.65
0.01012	2.37	0.39989	101.81	0.88434	239.26
0.01939	4.53	0.45526	117.28	0.91823	248.79
0.03144	7.34	0.50889	132.42	0.94728	257.05
0.04311	10.11	0.55927	146.78	0.97188	264.11
0.05389	12.69	0.60616	160.25	0.98498	267.88
0.08325	19.61	0.64920	172.65	0.99246	270.16
0.11471	27.24	0.66665	177.47	0.99629	271.22
0.15293	36.76	0.68831	183.90	0.99803	271.75
0.19517	47.37	0.70568	188.67	0.99912	272.04
0.24181	59.37	0.74963	201.24	0.99965	272.21
0.29159	72.47	0.79949	215.25	1.00000	272.32

**Table 4. Experimental Vapor-Liquid Equilibrium Data for the Binary System Water (1) + 2,2'-Diethanolamine (2) at 365.15 K**

$x_1$	$P/\text{kPa}$	$x_1$	$P/\text{kPa}$	$x_1$	$P/\text{kPa}$
0.000 00	0.04	0.685 41	48.04	0.962 02	72.58
0.013 05	0.68	0.728 82	52.15	0.971 45	73.29
0.023 59	1.20	0.766 28	55.70	0.978 54	73.82
0.038 73	1.96	0.801 76	59.03	0.984 33	74.18
0.056 19	2.86	0.819 65	60.80	0.988 87	74.53
0.083 22	4.29	0.828 66	61.51	0.992 32	74.80
0.130 76	6.91	0.843 95	63.00	0.994 91	75.00
0.185 69	10.05	0.851 78	63.61	0.996 52	75.14
0.249 21	13.84	0.866 43	64.98	0.997 69	75.28
0.315 12	18.13	0.871 31	65.35	0.998 48	75.36
0.381 67	22.71	0.887 26	66.75	0.999 02	75.47
0.448 33	27.63	0.906 10	68.31	0.999 54	75.46
0.512 29	32.71	0.922 94	69.66	0.999 77	75.47
0.577 24	38.21	0.937 84	70.80	1.000 00	75.52
0.634 81	43.35	0.950 90	71.79		

data of Danov et al.,<sup>6</sup> which show deviations at low pressures, good agreement between the experiments and calculations is observed. Also, the data from the static VLE measurements are included in the diagram.

The experimental binary VLE and  $H^E$  data for the systems methanol + 2,2'-diethanolamine and water + 2,2'-diethanolamine are listed in Tables 3–6. In Figure 3, the experimental results for the system methanol + 2,2'-diethanolamine are compared to calculations using quadratic temperature-dependent NRTL parameters, which were fitted to the VLE and  $H^E$  data. The temperature

**Table 5. Experimental Excess Enthalpy Data for the Binary System Methanol (1) + 2,2'-Diethanolamine (2)**

323.15 K; 1.82 MPa		363.15 K; 1.75 MPa		413.15 K; 1.75 MPa	
$x_1$	$H^E/\text{J}\cdot\text{mol}^{-1}$	$x_1$	$H^E/\text{J}\cdot\text{mol}^{-1}$	$x_1$	$H^E/\text{J}\cdot\text{mol}^{-1}$
0.0569	-32	0.1102	-52	0.1102	-80
0.1102	-58	0.2072	-98	0.2072	-146
0.2072	-100	0.2933	-135	0.2933	-199
0.2933	-131	0.3703	-161	0.3703	-240
0.3703	-156	0.4395	-180	0.4395	-270
0.4395	-172	0.5020	-193	0.5020	-292
0.5020	-181	0.5588	-202	0.5588	-308
0.5588	-184	0.6106	-205	0.6106	-315
0.6106	-184	0.6580	-204	0.6580	-318
0.6581	-181	0.7017	-199	0.7017	-316
0.7017	-173	0.7419	-189	0.7419	-306
0.7419	-160	0.7791	-178	0.7791	-291
0.7792	-147	0.8137	-165	0.8137	-272
0.8137	-134	0.8459	-151	0.8459	-251
0.8459	-116	0.8759	-133	0.8759	-222
0.8759	-100	0.9039	-115	0.9039	-192
0.9039	-80	0.9302	-95	0.9302	-155
0.9302	-61	0.9549	-72	0.9549	-117
0.9549	-41	0.9781	-50	0.9781	-72
0.9781	-20				
0.9892	-10				

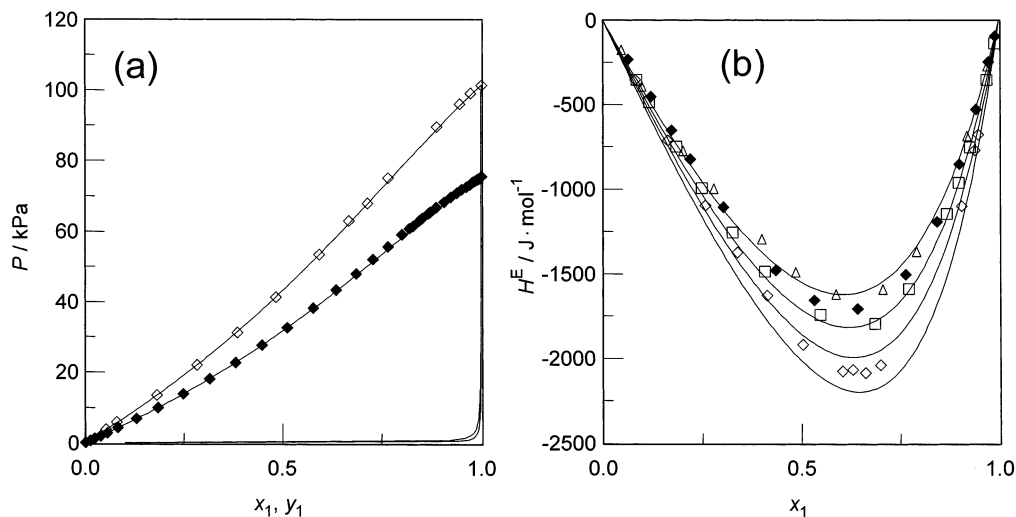
**Table 6. Experimental Excess Enthalpy Data for the Binary System Water (1) + 2,2'-Diethanolamine (2) at 363.15 K and 1.75 MPa**

$x_1$	$H^E/\text{J}\cdot\text{mol}^{-1}$	$x_1$	$H^E/\text{J}\cdot\text{mol}^{-1}$
0.0636	-232	0.6414	-1707
0.1209	-453	0.7630	-1504
0.1729	-650	0.8429	-1190
0.2202	-823	0.8994	-850
0.3032	-1106	0.9415	-529
0.4339	-1478	0.9741	-247
0.5323	-1655	0.9903	-93

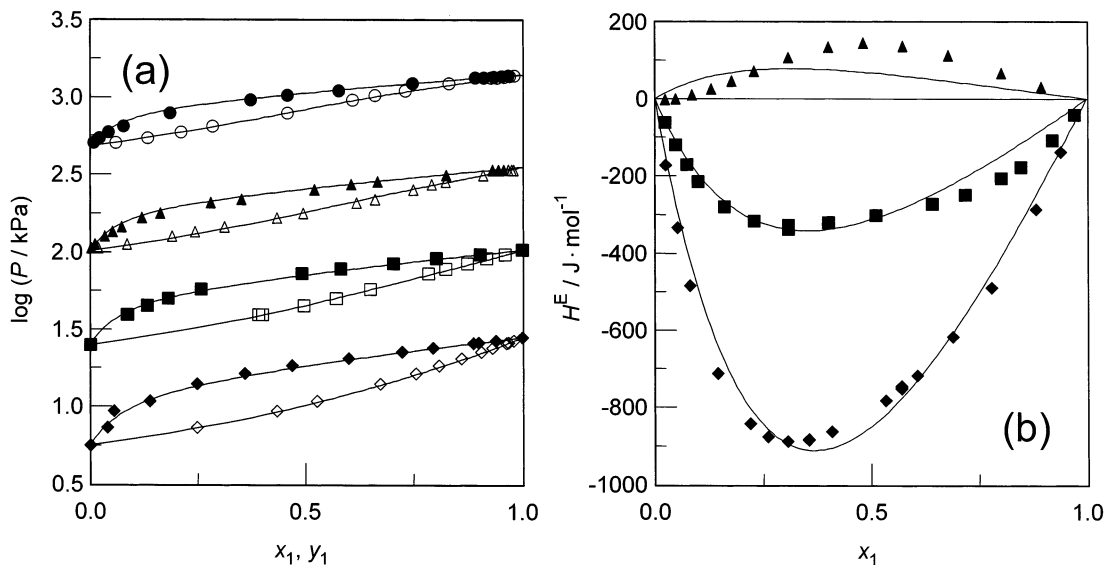
dependence of the parameters is described by the following expression:

$$\Delta g_{ij}/\text{J}\cdot\text{mol}^{-1} = a_{ij} + b_{ij}(TK) + c_{ij}(TK)^2 \quad (3)$$

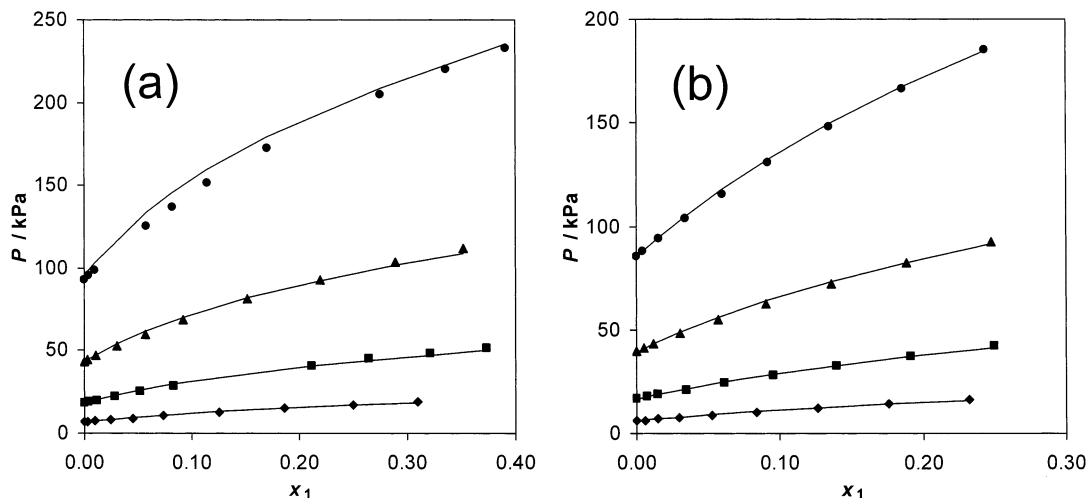
As can be seen from Figure 3, the temperature dependence of the phase equilibrium behavior (excess enthalpy data) can also be described reliably. For the system water + 2,2'-diethanolamine, experimental data of other authors<sup>10–11,13–15</sup> were also available for fitting the parameters. The experi-



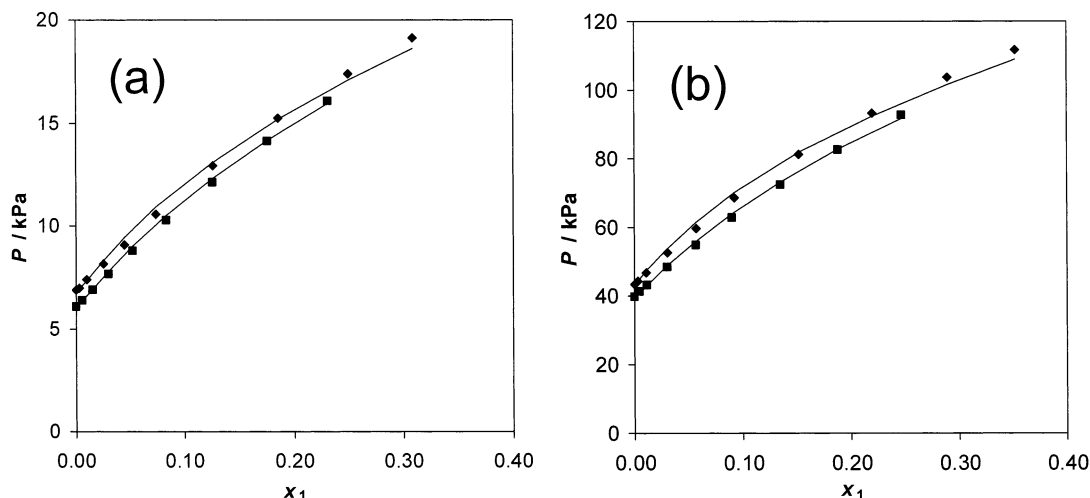
**Figure 4.** Experimental and predicted mixture behavior of the binary system water (1) + 2,2'-diethanolamine (2): (a) vapor-liquid equilibrium with experimental data from this work at (◆) 365.15 K, from Wilding et al. (1991)<sup>10</sup> at (◇) 373.15 K; (b) excess enthalpies with experimental data from this work at (◆) 363.15 K, from Maham et al. (1997)<sup>14</sup> at (◇) 298.15 K, from Maham et al. (2000)<sup>15</sup> at (□) 318.15 K and (△) 338.15 K; (—) NRTL.



**Figure 5.** Experimental and predicted mixture behavior of the binary system methanol (1) + water (2): (a) vapor-liquid equilibrium with experimental data from McGlashan and Williamson<sup>16</sup> at (◆, ◇) 308.15 K and (■, □) 338.15 K, from Griswold and Wong<sup>17</sup> at (▲, △) 373.15 K and (●, ○) 423.15 K; (b) excess enthalpies with experimental data from Gmehling<sup>5</sup> at (◆) 298.15 K, from Horstmann et al.<sup>18</sup> at (■) 363.15 K and (▲) 413.15 K; (—) NRTL.



**Figure 6.** Experimental and predicted VLE behavior of the ternary system methanol (1) + 2,2'-diethanolamine (2) + water (3): (a) starting from the binary mixture  $x(2,2'\text{-diethanolamine}) = 0.078\ 69$ ,  $x(\text{water}) = 0.921\ 31$  at (◆) 313.13 K, (■) 333.18 K, (▲) 353.18 K, and (●) 374.07 K; (b) starting from the binary mixture  $x(2,2'\text{-diethanolamine}) = 0.145\ 53$ ,  $x(\text{water}) = 0.854\ 47$  at (◆) 313.14 K, (■) 333.20 K, (▲) 353.17 K, and (●) 373.15 K; (—) NRTL.



**Figure 7.** Experimental and predicted VLE behavior of the ternary system methanol (1) + 2,2'-diethanolamine (2) + water (3): (a) at about 313 K (◆) starting from the binary mixture  $x(2,2'$ -diethanolamine) = 0.078 69,  $x(\text{water}) = 0.921$  31 and (■) starting from the binary mixture  $x(2,2'$ -diethanolamine) = 0.145 53,  $x(\text{water}) = 0.854$  47; (b) at about 353 K (◆) starting from the binary mixture  $x(2,2'$ -diethanolamine) = 0.078 69,  $x(\text{water}) = 0.921$  31 and (■) starting from the binary mixture  $x(2,2'$ -diethanolamine) = 0.145 53,  $x(\text{water}) = 0.854$  47; (—) NRTL.

**Table 7.** NRTL Interaction Parameters Fitted to Binary Experimental Data

component 1	component 2	$i$	$j$	$a_{ij}/\text{J}\cdot\text{mol}^{-1}$	$b_{ij}/\text{J}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$	$c_{ij}/\text{J}\cdot\text{mol}^{-1}\cdot\text{K}^{-2}$
methanol	2,2'-diethanolamine	1	2	-5566.6	19.183	0.012 601
		2	1	5548.4	-28.196	0.013 741
				$\alpha = 0.2495$		
water	2,2'-diethanolamine	1	2	4304.6	-6.7550	
		2	1	-6582.3	10.625	
				$\alpha = 0.4700$		
methanol	water	1	2	-2574.3	9.7082	-0.015 479
		2	1	-10230	63.963	-0.063 367
				$\alpha = 0.4700$		

**Table 8.** Experimental Vapor-Liquid Equilibrium Data for the Ternary System Methanol (1) + 2,2'-Diethanolamine (2) + Water (3) Starting with the Binary Mixture:  $x_2 = 0.078$  69,  $x_3 = 0.921$  31

313.13 K			333.18 K		
$x_1$	$x_2$	$P/\text{kPa}$	$x_1$	$x_2$	$P/\text{kPa}$
0.000 00	0.078 69	6.86	0.000 00	0.078 69	18.39
0.003 31	0.078 43	6.98	0.003 73	0.078 40	18.86
0.009 76	0.077 92	7.38	0.011 65	0.077 77	19.94
0.024 78	0.076 74	8.16	0.028 76	0.076 43	22.24
0.044 93	0.075 16	9.10	0.051 88	0.074 61	25.17
0.073 94	0.072 87	10.56	0.083 53	0.072 12	28.85
0.125 44	0.068 82	12.90	0.211 53	0.062 05	40.88
0.186 12	0.064 05	15.26	0.264 93	0.057 84	44.87
0.250 30	0.059 00	17.40	0.321 38	0.053 40	48.60
0.309 38	0.054 35	19.13	0.373 40	0.049 31	51.72
353.18 K			374.07 K		
$x_1$	$x_2$	$P/\text{kPa}$	$x_1$	$x_2$	$P/\text{kPa}$
0.000 00	0.078 69	43.45	0.000 00	0.078 69	93.12
0.002 74	0.078 47	44.37	0.003 44	0.078 42	95.29
0.010 82	0.077 84	46.81	0.009 35	0.077 96	98.62
0.030 68	0.076 28	52.74	0.058 25	0.074 11	125.39
0.057 31	0.074 18	59.82	0.082 79	0.072 18	137.04
0.092 62	0.071 40	68.56	0.115 18	0.069 63	151.21
0.151 95	0.066 73	81.13	0.170 48	0.065 28	172.24
0.219 62	0.061 41	93.19	0.275 79	0.056 99	204.68
0.289 27	0.055 93	103.59	0.335 79	0.052 27	220.00
0.352 12	0.050 98	111.80	0.390 71	0.047 95	232.68

mental and calculated results, using linear temperature-dependent parameters, are shown in Figure 4. It can be seen that the agreement between our VLE data and the data of Wilding et al.<sup>10</sup> is very good. Small deviations are observed only for the excess enthalpies reported by Maham et al.<sup>14,15</sup> For the binary system methanol + water, a large

**Table 9.** Experimental Vapor-Liquid Equilibrium Data for the Ternary System Methanol (1) + 2,2'-Diethanolamine (2) + Water (3) Starting with the Binary Mixture:  $x_2 = 0.145$  53,  $x_3 = 0.854$  47

313.14 K			333.20 K		
$x_1$	$x_2$	$P/\text{kPa}$	$x_1$	$x_2$	$P/\text{kPa}$
0.000 00	0.145 53	6.04	0.000 00	0.145 53	16.66
0.005 92	0.144 67	6.36	0.007 46	0.144 45	17.66
0.015 05	0.143 34	6.87	0.015 12	0.143 33	18.65
0.029 66	0.141 22	7.63	0.034 72	0.140 48	21.11
0.052 33	0.137 92	8.75	0.061 15	0.136 63	24.24
0.083 38	0.133 40	10.24	0.095 08	0.131 69	28.10
0.126 07	0.127 18	12.11	0.139 52	0.125 23	32.60
0.176 02	0.119 91	14.09	0.191 49	0.117 66	37.33
0.231 84	0.111 79	16.06	0.249 62	0.109 20	41.99
353.17 K			373.15 K		
$x_1$	$x_2$	$P/\text{kPa}$	$x_1$	$x_2$	$P/\text{kPa}$
0.000 00	0.145 53	39.69	0.000 00	0.145 53	85.39
0.004 81	0.144 83	41.15	0.004 74	0.144 84	87.89
0.011 91	0.143 80	43.22	0.015 82	0.143 23	94.09
0.030 65	0.141 07	48.25	0.033 99	0.140 59	103.60
0.056 75	0.137 28	54.77	0.059 54	0.136 87	115.71
0.090 36	0.132 38	62.77	0.091 64	0.132 20	130.57
0.135 60	0.125 80	72.42	0.134 46	0.125 96	147.97
0.188 56	0.118 09	82.60	0.185 66	0.118 51	166.50
0.247 40	0.109 53	92.48	0.243 43	0.110 11	185.29

number of data sets is available in the literature. Figure 5 shows the calculated results of quadratic temperature-dependent NRTL parameters fitted to those data in comparison with selected experimental data.

The three binary systems are miscible over the entire composition range. The mixture behavior of methanol + 2,2'-diethanolamine and water + 2,2'-diethanolamine is

almost ideal. Small positive deviations from Raoult's law are observed for methanol + water. The NRTL model is able to predict the mixture behavior of all binary systems using the interaction parameters given in Table 7.

Starting with two binary mixtures of 2,2'-diethanolamine and water, VLE data for the ternary system methanol + 2,2'-diethanolamine + water were also measured with the static apparatus at 313, 333, 353, and 373 K. The experimental data are listed in Tables 8 and 9. In Figures 6 and 7, they are compared graphically to the NRTL calculations using the interaction parameters from Table 7. In Figure 6, a correct description of the temperature dependence of the VLE behavior was demonstrated. The influence of increasing the concentration of 2,2'-diethanolamine in the mixture can be seen in Figure 7.

## Conclusions

In this project, reliable phase equilibrium and excess properties were measured for the ternary system methanol + 2,2'-diethanolamine + water, the binary subsystems, and the pure compounds. For this purpose, three experimental methods were used including ebulliometry for the vapor pressures, a static apparatus for the synthetic VLE data, and isothermal flow calorimetry for the excess enthalpies. The binary experimental data of this work and of other authors were used to fit temperature-dependent NRTL interaction parameters. The NRTL model is able to describe the binary VLE and  $H^E$  behavior of all the three subsystems using linear or quadratic temperature-dependent interaction parameters. With these parameters, the ternary system can be predicted reliably, as validated in the temperature range from 40 to 100 °C.

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