Isobaric Vapor–Liquid Equilibrium for Ethanol + Water + Copper(II) Chloride

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Isobaric vapor–liquid equilibrium for ethanol(1) + water(2) + copper(II) chloride (3) at different mole fractions of copper(II) chloride has been measured at 100.0 kPa. The results in the range where the mole fraction of salt in the liquid phase was less than 0.146 times the mole fraction of water were correlated by assuming that the salt was in ionic form and it was associated only with the water. Thermodynamic consistency was checked according to Herington's method with satisfactory results.

Introduction

The extractive distillation in which an electrolyte is employed as the extractive agent is an important alternative to the conventional methods for separating mixtures involving azeotropes. Many papers dealing with the salt effect on vapor–liquid equilibrium have been published, as such systems have important industrial applications. However, these systems are still poorly understood because of the complex interactions of electrolytes with the solvents.

Some methods for the correlation of the activity coefficients of the solvents in these ternary mixtures consider that the mixtures can be treated as pseudobinary systems (Natarajan, 1980; Schmitt and Vogelpohl, 1983; Vercher *et al.*, 1991, 1994; Peña *et al.*, 1994). Another approach uses correlations based on modifications of the vapor–liquid equilibrium local composition model (Chen *et al.*, 1982; Chen and Evans, 1986; Sander *et al.*, 1986; Tan, 1987; Kikic *et al.*, 1991).

The present work studies the vapor–liquid equilibrium of ethanol + water + copper(II) chloride with varying concentrations of salt. The addition of copper(II) chloride to this solvent mixture increases the amount of alcohol present in the vapor phase at equilibrium, eventually eliminating the azeotrope. This indicates a preferencial association of the salt with the less volatile component of mixed solvent.

The vapor–liquid equilibrium of the ethanol + water system saturated with copper(II) chloride has been determined by Costa and Moragues (1952), Galán *et al.* (1975), Meranda and Furter (1974), and Martinez de la Ossa and Galán (1986, 1991). Furthermore, this system is quoted in the reviews by Ciparis (1966, 1973), but we have not found any reported vapor–liquid equilibrium for (ethanol + water) with various concentrations of copper(II) chloride below saturation.

Experimental Section

The chemicals were absolute ethanol (Baker-analyzed reagent, >99.5 mass %), distilled water, and copper(II) chloride (Probus, >99 mass %). They were used without further purification.

The equilibrium apparatus was a recirculating still of the Labodest model, manufactured by Fischer. The vapor–liquid equilibrium data were obtained at (100.00 ± 0.04) kPa. The vapor pressure of water with various concentrations of salt was also measured with the same apparatus.

Every experimental point was obtained from an initial sample prepared gravimetrically by using a Sartorius analytical balance with a precision of ±0.0001 g and, afterward, by adding different quantities of ethanol, water, ethanol–salt solution, or water–salt solution. Each experiment was kept at the boiling point for 15 min or more to ensure the stationary state. The accuracy of the temperature measurement was ±0.1 K.

Compositions of the condensed vapor phase were analyzed by using a Hewlett-Packard 5700 A gas chromatograph with a thermal conductivity detector, connected to a Hewlett-Packard 3394 A integrator. The chromatographic column (2 m × $^1/_8$ in.) was packed with Porapak P. The gas carrier was helium flowing at 50 $cm^3 \cdot min^{-1}$, and the column temperature was 383 K. The calibration was carried out with gravimetrically prepared standard solutions. The accuracy of the measured vapor-phase mole fraction was ±0.002.

The liquid phase was composed of ethanol, water, and copper(II) chloride. The salt mass fraction in the liquid phase was gravimetrically determined after the volatile components were separated from a known mass of sample by evaporation to dryness. Also, the density of the liquid phase was determined with an Anton Paar DMA 55 densimeter matched to a Julabo circulator with proportional temperature control and an automatic drift correction system that kept the samples at (298.15 ± 0.01) K. Previously, the density of a set of standard solutions with known amounts of ethanol, water, and salt in the ternary mixtures had been measured, and a correlation that allowed determination of the mass fraction of ethanol in the sample, given the density and the mass fraction of salt in the solution, had been obtained. The mass fractions were then translated into mole fractions. The accuracy in the measurement of the ethanol, water, and copper(II) chloride mole fractions in the liquid phase was ±0.005. This method of analyzing the composition of salt-containing mixtures was found to be reproducible and of consistent accuracy, as described in a previous work (Vercher *et al.*, 1994).

Results and Discussion

In Tables 1 and 2, the vapor–liquid equilibrium for the ethanol (1) + water (2) + copper(II) chloride (3) system at a pressure of 100.0 kPa is reported. Included are the

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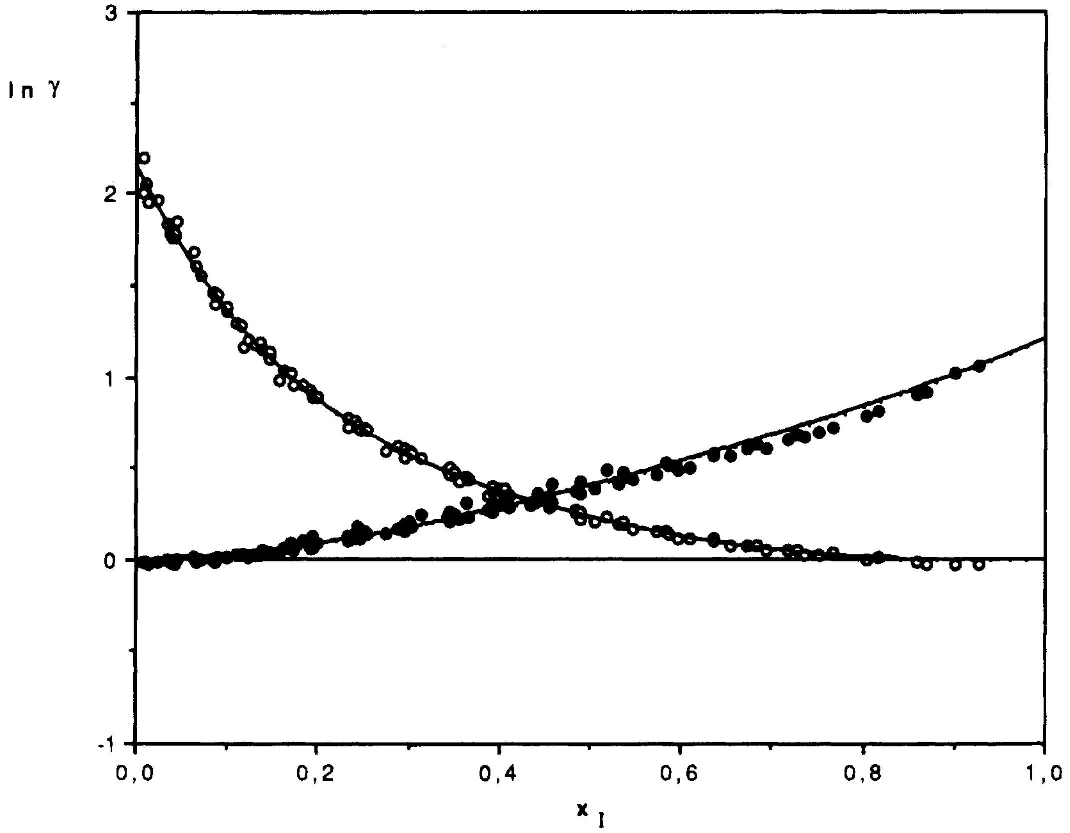

Figure 1. Experimental solvent activity coefficients for the system ethanol (1) + water (2) + copper(II) chloride (3) at 100.0 kPa in the range $x_3 < 0.146x_2$: (○) ln γ_I; (●) ln γ_{II}; (—) calculated results by eqs 4 and 5.

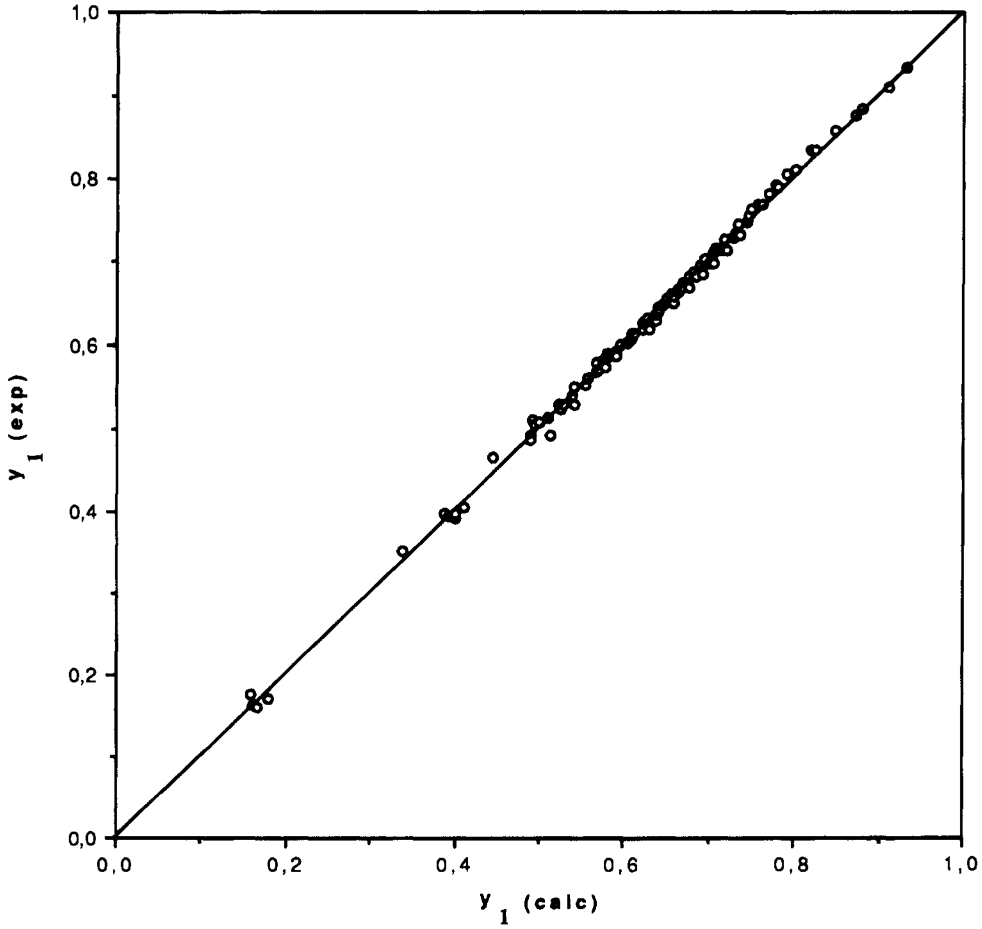

Figure 2. Comparison of calculated ethanol vapor composition, y_1(calcd), with experimental values, y_1(exptl).

Table 1. Vapor–Liquid Equilibrium for Ethanol (1) + Water (2) + Copper(II) Chloride (3) at 100.0 kPa in the Range of $x_3 < 0.146\, x_2$

T/K	x_1	x_2	x_3	y_1	γ_I	γ_{II}
375.7	0.008	0.895	0.097	0.178	9.050	0.981
375.1	0.009	0.904	0.087	0.159	7.457	0.985
371.0	0.010	0.947	0.044	0.164	7.853	0.978
369.4	0.012	0.967	0.021	0.170	7.031	0.974
369.7	0.024	0.879	0.097	0.352	7.157	0.982
366.5	0.035	0.881	0.084	0.394	6.247	0.992
366.6	0.037	0.880	0.083	0.396	5.934	0.982
366.6	0.038	0.875	0.087	0.406	5.840	0.985
365.5	0.039	0.890	0.072	0.391	5.760	0.989
363.8	0.041	0.909	0.049	0.394	5.790	0.977
363.0	0.042	0.920	0.038	0.396	5.952	0.971
364.6	0.043	0.861	0.096	0.467	6.336	0.999
361.5	0.063	0.854	0.084	0.509	5.354	1.007
361.7	0.066	0.857	0.076	0.504	4.945	0.984
361.6	0.072	0.848	0.080	0.514	4.694	0.993
359.0	0.083	0.883	0.034	0.491	4.289	0.991
359.0	0.085	0.870	0.045	0.508	4.297	0.992
358.8	0.088	0.888	0.024	0.487	4.038	0.985
359.5	0.088	0.846	0.065	0.529	4.245	1.005
358.9	0.099	0.845	0.056	0.529	3.865	1.008
359.0	0.099	0.832	0.069	0.551	4.003	1.006
359.0	0.111	0.816	0.073	0.561	3.645	1.016
359.0	0.116	0.801	0.083	0.580	3.600	1.028
357.6	0.118	0.873	0.009	0.493	3.181	1.017
357.1	0.123	0.857	0.020	0.525	3.312	1.006
356.8	0.130	0.840	0.029	0.540	3.239	1.020
357.4	0.135	0.803	0.061	0.579	3.266	1.027
359.1	0.138	0.768	0.094	0.607	3.146	1.055
356.6	0.145	0.815	0.040	0.560	3.033	1.037
357.3	0.146	0.789	0.065	0.590	3.107	1.033
357.0	0.148	0.802	0.050	0.568	2.986	1.042
356.4	0.157	0.834	0.009	0.529	2.673	1.037
356.7	0.163	0.781	0.056	0.586	2.824	1.058
357.3	0.169	0.753	0.078	0.610	2.764	1.087
355.8	0.172	0.812	0.015	0.553	2.603	1.047
356.7	0.184	0.748	0.068	0.606	2.584	1.099
355.6	0.190	0.775	0.035	0.585	2.519	1.064
355.5	0.193	0.783	0.024	0.572	2.436	1.069
357.7	0.193	0.719	0.089	0.628	2.458	1.128
355.8	0.198	0.758	0.044	0.592	2.428	1.086
355.0	0.232	0.757	0.011	0.573	2.069	1.102
355.4	0.233	0.717	0.050	0.613	2.161	1.132
355.7	0.240	0.699	0.061	0.626	2.126	1.147
354.8	0.244	0.727	0.029	0.600	2.070	1.119
356.6	0.245	0.672	0.084	0.650	2.087	1.191
354.8	0.246	0.734	0.019	0.590	2.020	1.116
355.9	0.251	0.680	0.069	0.639	2.055	1.170
354.9	0.253	0.708	0.038	0.609	2.019	1.144
354.5	0.276	0.716	0.008	0.586	1.811	1.148
354.7	0.288	0.667	0.045	0.631	1.851	1.185
354.2	0.295	0.682	0.023	0.614	1.794	1.170
354.9	0.295	0.651	0.054	0.642	1.823	1.208
354.3	0.297	0.688	0.015	0.601	1.741	1.174
355.2	0.300	0.635	0.064	0.656	1.809	1.228
354.3	0.303	0.664	0.033	0.626	1.774	1.189
355.5	0.313	0.613	0.074	0.668	1.748	1.281
354.1	0.344	0.616	0.040	0.646	1.624	1.254
354.7	0.344	0.595	0.060	0.670	1.642	1.283
353.8	0.346	0.635	0.019	0.629	1.591	1.221
354.3	0.350	0.601	0.049	0.659	1.615	1.274
353.7	0.355	0.633	0.011	0.619	1.531	1.243
354.9	0.364	0.567	0.069	0.684	1.573	1.352
353.7	0.366	0.606	0.027	0.644	1.542	1.263
353.5	0.386	0.606	0.008	0.619	1.419	1.303
353.4	0.392	0.593	0.015	0.638	1.447	1.290
353.6	0.392	0.573	0.035	0.661	1.485	1.312
354.2	0.393	0.551	0.056	0.683	1.495	1.359
353.8	0.401	0.554	0.045	0.676	1.473	1.341
354.4	0.406	0.529	0.065	0.697	1.468	1.418
353.3	0.412	0.565	0.023	0.656	1.421	1.320
353.0	0.435	0.553	0.012	0.650	1.347	1.354
353.2	0.440	0.530	0.030	0.673	1.368	1.381
353.7	0.442	0.507	0.051	0.698	1.385	1.437
353.3	0.454	0.505	0.041	0.694	1.362	1.415
353.0	0.457	0.524	0.019	0.667	1.317	1.387
353.9	0.459	0.482	0.059	0.712	1.352	1.517
353.1	0.483	0.480	0.037	0.700	1.300	1.458
352.8	0.484	0.489	0.027	0.687	1.290	1.448
352.7	0.488	0.503	0.008	0.663	1.239	1.441
353.3	0.490	0.462	0.047	0.713	1.296	1.525
352.6	0.506	0.479	0.015	0.681	1.234	1.468
353.3	0.519	0.429	0.052	0.731	1.254	1.621
352.5	0.531	0.447	0.022	0.703	1.216	1.514
352.7	0.536	0.430	0.033	0.716	1.217	1.569
353.0	0.537	0.420	0.043	0.729	1.224	1.607
352.3	0.548	0.440	0.012	0.694	1.173	1.544
352.2	0.572	0.409	0.019	0.716	1.164	1.588
352.6	0.585	0.377	0.038	0.748	1.170	1.689
352.4	0.588	0.383	0.030	0.733	1.151	1.679
352.0	0.596	0.395	0.009	0.711	1.117	1.638
352.0	0.609	0.376	0.015	0.727	1.118	1.659
352.3	0.635	0.332	0.033	0.769	1.120	1.780
352.1	0.637	0.338	0.026	0.756	1.107	1.761
351.8	0.655	0.334	0.012	0.745	1.074	1.751
351.8	0.673	0.306	0.021	0.769	1.077	1.831
352.0	0.683	0.288	0.029	0.790	1.082	1.887
351.6	0.694	0.297	0.009	0.763	1.044	1.839
351.6	0.717	0.265	0.018	0.791	1.048	1.936
351.8	0.728	0.247	0.025	0.812	1.051	1.988
351.4	0.735	0.258	0.007	0.781	1.019	1.954
351.5	0.750	0.236	0.014	0.804	1.023	2.004
351.6	0.767	0.212	0.021	0.834	1.032	2.058
351.3	0.805	0.184	0.011	0.835	0.997	2.187
351.4	0.815	0.167	0.017	0.859	1.008	2.257
351.3	0.860	0.127	0.013	0.885	0.989	2.465
351.1	0.869	0.123	0.008	0.877	0.976	2.484
351.2	0.901	0.089	0.010	0.911	0.975	2.775
351.1	0.928	0.065	0.007	0.933	0.973	2.880

equilibrium temperature (T/K), the mole fractions of ethanol (x_1), water (x_2), and salt (x_3) in the ternary liquid phase, and the mole fraction of ethanol in the vapor phase (y_1). In Table 1, we give the results in the range of $x_3 < 0.146x_2$, and in Table 2, we show the results in the range of $x_3 > 0.146x_2$.

To make possible the thermodynamic treatment of vapor–liquid equilibrium, we postulate that, in the range of liquid-phase salt compositions studied, the salt is in ionic form and it is associated only with the water (Vercher *et al.*, 1991). Therefore, the ternary system can be treated as a pseudobinary system composed of pure ethanol (I) and water + salt (II) components. This assumption is only acceptable when there is enough water to dissolve all of the salt present in the liquid phase, as described in a previous work (Peña *et al.*, 1994). In the present system, all of the salt can be dissolved in water when $x_3 < 0.146x_2$. When equilibrium is established between vapor and liquid phases, the activity coefficients of both pseudocomponents will be given by the expressions

$$\gamma_I = \frac{y_I \phi_I P}{x_I P_I^0} \qquad \gamma_{II} = \frac{y_{II} \phi_{II} P}{x_{II} P_{II}^0} \tag{1}$$

where x_I is the mole fraction of pseudocomponent I in the liquid phase ($=x_1$), x_{II} is the mole fraction of pseudocomponent II in the liquid phase ($=x_2 + x_3$), y_I is the mole fraction of ethanol in the vapor phase ($=y_1$), y_{II} is the mole fraction of water in the vapor phase ($=1 - y_1$), ϕ_I is the fugacity coefficient of pseudocomponent I in the vapor phase, ϕ_{II} is the fugacity coefficient of pseudocomponent II in the vapor phase, P is the system pressure (kPa), P_I^0 is the vapor pressure of pure ethanol ($=P_1^0$), and P_{II}^0 is the vapor pressure of pseudocomponent II, both calculated at the equilibrium temperature (kPa). The value of P_{II}^0 depends on the temperature and on the salt concentration. Jaques and Furter (1972) propose that this dependency can

Table 2. Vapor–Liquid Equilibrium for Ethanol (1) + Water (2) + Copper(II) Chloride (3) at 100.0 kPa in the Range of $x_3 > 0.146\ x_2$

T/K	x_1	x_2	x_3	y_1	*T*/K	x_1	x_2	x_3	y_1
356.2	0.320	0.593	0.087	0.685	352.5	0.732	0.213	0.055	0.839
355.3	0.379	0.541	0.080	0.699	353.6	0.739	0.176	0.086	0.875
355.2	0.426	0.488	0.087	0.727	355.1	0.740	0.140	0.120	0.906
354.6	0.435	0.492	0.073	0.715	355.6	0.746	0.123	0.130	0.916
354.7	0.467	0.452	0.081	0.737	352.7	0.749	0.189	0.061	0.867
354.0	0.485	0.448	0.067	0.731	351.9	0.750	0.218	0.032	0.835
354.2	0.510	0.414	0.076	0.750	354.0	0.752	0.152	0.096	0.895
353.5	0.537	0.403	0.060	0.748	352.3	0.756	0.197	0.047	0.855
353.8	0.541	0.388	0.071	0.761	352.9	0.760	0.173	0.067	0.877
354.5	0.546	0.368	0.085	0.778	353.1	0.763	0.163	0.075	0.884
353.5	0.574	0.360	0.066	0.772	353.0	0.764	0.168	0.068	0.884
353.1	0.582	0.364	0.054	0.763	356.2	0.770	0.082	0.148	0.938
353.8	0.600	0.324	0.077	0.794	354.4	0.771	0.121	0.108	0.914
353.1	0.611	0.328	0.060	0.785	356.7	0.773	0.070	0.158	0.944
352.7	0.625	0.326	0.049	0.780	352.5	0.776	0.170	0.055	0.878
357.0	0.631	0.229	0.140	0.870	353.5	0.783	0.133	0.084	0.904
356.5	0.637	0.230	0.133	0.866	352.8	0.790	0.145	0.065	0.896
354.1	0.640	0.272	0.088	0.828	352.6	0.792	0.148	0.060	0.894
352.8	0.642	0.303	0.055	0.797	355.1	0.792	0.082	0.125	0.935
353.3	0.655	0.276	0.069	0.810	352.0	0.793	0.167	0.040	0.871
352.4	0.670	0.287	0.043	0.798	351.6	0.797	0.177	0.026	0.859
353.7	0.672	0.247	0.081	0.838	355.5	0.797	0.067	0.136	0.943
355.7	0.678	0.198	0.124	0.874	354.1	0.811	0.086	0.103	0.931
355.2	0.682	0.203	0.115	0.871	354.5	0.816	0.071	0.113	0.941
352.9	0.686	0.253	0.061	0.826	352.4	0.820	0.128	0.052	0.905
354.0	0.694	0.218	0.088	0.858	351.8	0.822	0.144	0.035	0.885
353.3	0.700	0.225	0.074	0.848	353.2	0.823	0.097	0.079	0.926
356.5	0.702	0.155	0.143	0.908	353.6	0.831	0.078	0.090	0.935
354.2	0.704	0.201	0.095	0.866	352.6	0.838	0.099	0.063	0.924
357.0	0.705	0.142	0.153	0.917	352.1	0.839	0.116	0.045	0.915
352.1	0.714	0.249	0.037	0.818	352.9	0.841	0.087	0.072	0.929
354.6	0.718	0.177	0.105	0.881	351.7	0.846	0.125	0.029	0.897
353.3	0.725	0.198	0.076	0.863	352.0	0.861	0.099	0.039	0.924
353.0	0.726	0.207	0.067	0.858	351.5	0.874	0.102	0.025	0.912
353.4	0.730	0.192	0.078	0.871					

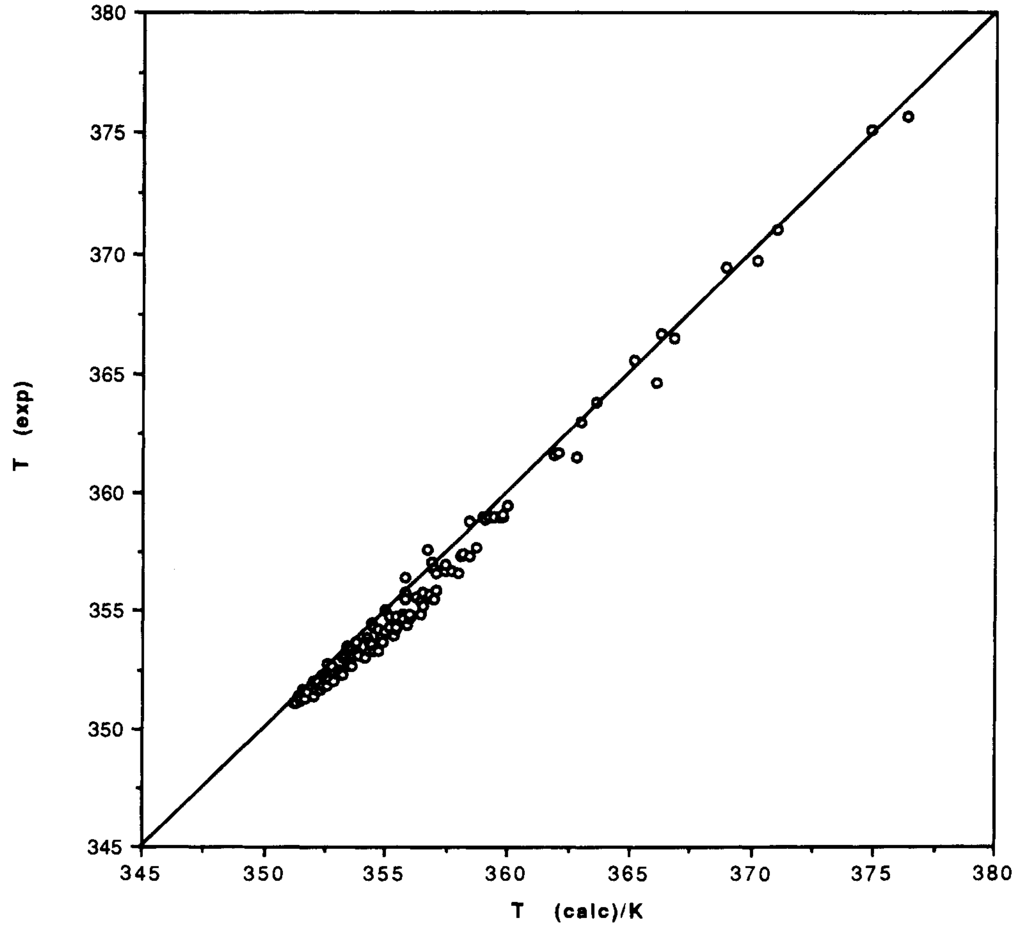

Figure 3. Comparison of calculated bubble points with experimental values.

Table 3. Boiling Points of Water + Copper(II) Chloride Mixtures at 100.0 kPa

x_3*	*T*/K	x_3*	*T*/K	x_3*	*T*/K
0.0969	380.9	0.0609	377.2	0.0215	374.0
0.0936	380.6	0.0536	376.5	0.0179	373.8
0.0886	380.0	0.0458	375.9	0.0151	373.6
0.0844	379.5	0.0384	375.3	0.0124	373.5
0.0773	378.8	0.0311	374.7	0.0103	373.3
0.0692	377.9	0.0258	374.4		

be expressed as

$$P_{II}{}^{0} = P_2{}^{0}(T)\epsilon(x_3{}^{*}) \qquad (2)$$

where $P_2{}^0$ is the vapor pressure of pure water (kPa) and ϵ is the vapor pressure correction factor, which depends only on x_3*, the mole fraction of the salt component in the pseudocomponent II [$=x_3/(x_2 + x_3)$].

The vapor pressure correction factor was computed according to the method of Jaques and Furter (1972) from results of the vapor pressure of water with different amounts of salt that we have obtained, given in Table 3, and the vapor pressure of pure water obtained from the Antoine equation with the parameters given in the literature (Gmehling *et al.*, 1981), both at the same temperature.

The correlation equation obtained from experimental results for the vapor pressure correction factor (ϵ) with the salt mole fraction in pseudocomponent II (x_3*), for the entire range of salt concentrations studied, is

$$\epsilon = 1 - 1.8956x_3{}^{*} - 6.9609(x_3{}^{*})^2 \qquad (3)$$

The fugacity coefficients ϕ_I and ϕ_{II} were calculated by means of the virial equation of state, and the second virial coefficients were calculated from the Pitzer and Curl (1957) equations and the Tsonopoulos (1974) correlation correction for polar compounds.

For each experimental value, the activity coefficients of the pseudocomponents I and II in the liquid phase have been calculated following the above procedure. The obtained results of γ_I and γ_{II} are shown in columns 6 and 7 of Table 1.

In Figure 1, the logarithms of γ_I and γ_{II} are plotted against x_I. In this figure, the logarithms of activity coefficients for both pseudocomponents can be adjusted to one curve whatever the salt concentration may be. This fact implies that the pseudobinary model proposed explains the behavior of the system in the range of compositions established.

The thermodynamic consistency of the results was tested by the integral method described by Herington (1947, 1951) and Redlich and Kister (1948). The area test parameters, *D* and *J*, obtained were $D = 1.95\%$, $J = 10.51\%$, and $D - J = -8.56\%$. According to Herington's interpretation of the area test results, the results were probably consistent.

For the purpose of reproducing experimental data, the activity coefficients obtained for every pseudocomponent were adjusted against their composition in the liquid phase, and the equations found were

$$\ln \gamma_I = (2.1561 - 5.7085x_I + 10.355x_I{}^2 - 7.6752x_I{}^3)(1 - x_I)^2 \qquad (4)$$

$$\ln \gamma_{II} = (1.2 + 0.3642x_{II} + 0.8455x_{II}^2)(1 - x_{II})^2 \qquad (5)$$

These equations were used to recalculate the vapor composition and temperature results in equilibrium from the liquid-phase composition. From comparison of experimental and calculated results, we found the following: $y_{exptl} - y_{calcd}$, mean, 0.006, standard deviation, 0.007; $T_{exptl} - T_{calcd}$, mean, 0.64 K, standard deviation, 0.51 K.

The experimental results are plotted against the corresponding calculated ones in Figure 2 for the vapor composition and in Figure 3 for the temperature. In these figures can be noted the agreement between both experimental and calculated results.

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