

# Isobaric Vapor–Liquid Equilibrium for Ethanol + Water + Sodium Nitrate

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The isobaric vapor–liquid equilibrium for ethanol (1) + water (2) + sodium nitrate (3) at various concentrations of salt and with ethanol mole fractions from 0 to 0.774 has been measured at 100.0 kPa. The results were correlated by assuming that the salt was in ionic form and it was associated only with the water.

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## Introduction

The salt effect on the vapor–liquid equilibrium of mixed solvents provides a potential technique of extractive distillation, in which a dissolved salt, rather than a liquid additive, is used as the separating agent. This salt distillation process has been used in the purification of close-boiling, azeotropic, and other systems which are difficult to separate.

Over the last decades, a considerable amount of work has been carried out on the experimental determination of the vapor–liquid equilibrium of various solvent mixtures containing a variety of dissolved salts and on the development of correlation methods for such data. However, most experimental data have been obtained at salt saturation.

Some proposed methods for the experimental data correlation consider that the mixtures can be treated as pseudobinary systems (Natarajan and Srinivasan, 1980; Schmitt and Vogelpohl, 1982; Vercher *et al.*, 1991, 1994; Peña *et al.*, 1994). Other correlations are 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 involves the measurement of the vapor–liquid equilibrium of ethanol + water + sodium nitrate at different concentrations of salt below saturation. The addition of sodium nitrate to this solvent mixture increases the amount of alcohol present in the vapor phase at equilibrium. This indicates a preferential association of the salt with the less volatile component of the mixed solvent.

The vapor–liquid equilibrium of the ethanol + water system saturated with sodium nitrate has been studied by Tursi and Thompson (1951), Yamamoto *et al.* (1952), Johnson and Furter (1965), and Michalowski and Mondeja (1977), and this system is quoted in the reviews by Ciparis (1966, 1973), but we have not found any reported vapor–liquid equilibrium for this system at salt concentrations below saturation. The effect of sodium nitrate addition in the vapor–liquid equilibrium of other binary systems has also been studied: propionic acid + water (Ramalho and Edgett, 1964); methanol + water (Johnson and Furter, 1965); 1-propanol + water (Johnson and Furter, 1965); acetone + water (Sada *et al.*, 1972).

## Experimental Section

The chemicals were absolute ethanol (Baker-analyzed reagent, >99.8 mass %, boiling point at 100.0 kPa: 351.2 K), distilled water (boiling point at 100.0 kPa: 372.8 K), and sodium nitrate (Merck GR, >99.5 mass %). They were used without further purification.

The equilibrium apparatus was a recirculating still of the Labodest model, manufactured by Fischer (Walas, 1985). The vapor–liquid equilibrium was obtained at  $(100.00 \pm 0.04)$  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 Mettler AE 200 analytical balance with a precision of  $\pm 0.0001$  g and, afterward, by adding different quantities of ethanol, water, or ethanol + water mixture. 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  $\pm 0.1$  K.

Compositions of the condensed vapor phase were analyzed by using a Varian STAR 3400 CX gas chromatograph with a thermal conductivity detector. The GC response was treated with a Star Chromatography Station. The chromatographic column (2 m  $\times$   $\frac{1}{8}$  in.) was packed with Porapak P. The gas carrier was helium flowing at 50  $\text{cm}^3\cdot\text{min}^{-1}$  and the column temperature was 383 K. The temperatures at the injector and detector were 473 and 523 K, respectively. The calibration was carried out with gravimetrically prepared standard solutions. The accuracy of the measured vapor-phase mole fraction was  $\pm 0.002$ .

The liquid phase was composed of ethanol, water, and sodium nitrate. 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. 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  $(298.15 \pm 0.01)$  K. The accuracy of the density values was  $\pm 0.00001$   $\text{g}\cdot\text{cm}^{-3}$ . 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

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**Table 1. Vapor–Liquid Equilibrium for Ethanol (1) + Water (2) + Sodium Nitrate (3) at 100.0 kPa**

$T/K$	$x_1$	$x_2$	$x_3$	$y_1$	$\gamma_1$	$\gamma_{II}$	$T/K$	$x_1$	$x_2$	$x_3$	$y_1$	$\gamma_1$	$\gamma_{II}$
373.7	0.009	0.839	0.153	0.244	11.953	0.983	353.3	0.183	0.756	0.061	0.639	3.112	1.045
371.9	0.010	0.874	0.117	0.245	11.730	0.967	355.2	0.188	0.771	0.041	0.596	2.634	1.043
370.0	0.013	0.850	0.137	0.320	11.703	0.980	354.5	0.189	0.733	0.078	0.644	2.898	1.034
368.9	0.015	0.875	0.110	0.320	10.686	0.964	355.2	0.198	0.774	0.028	0.585	2.455	1.050
368.9	0.016	0.842	0.143	0.361	11.623	0.975	352.9	0.200	0.736	0.065	0.656	2.974	1.047
366.1	0.021	0.852	0.128	0.414	11.051	0.965	353.3	0.203	0.743	0.055	0.632	2.783	1.079
365.2	0.025	0.890	0.085	0.391	8.935	0.951	355.3	0.205	0.781	0.014	0.570	2.306	1.060
364.0	0.027	0.838	0.136	0.473	10.594	0.964	354.8	0.207	0.745	0.048	0.615	2.497	1.053
363.9	0.027	0.904	0.068	0.409	8.979	0.941	354.5	0.209	0.728	0.064	0.636	2.593	1.050
364.0	0.029	0.919	0.052	0.378	7.947	0.957	354.9	0.216	0.748	0.036	0.602	2.342	1.066
362.5	0.031	0.847	0.121	0.493	10.062	0.956	354.3	0.223	0.707	0.070	0.649	2.500	1.060
363.1	0.031	0.867	0.101	0.460	9.083	0.952	354.9	0.225	0.752	0.023	0.592	2.211	1.072
362.2	0.035	0.929	0.036	0.405	7.379	0.956	354.9	0.237	0.753	0.010	0.577	2.042	1.096
363.9	0.036	0.886	0.079	0.418	7.034	0.955	354.3	0.240	0.705	0.055	0.638	2.286	1.079
359.9	0.044	0.828	0.128	0.558	8.787	0.953	354.5	0.241	0.718	0.041	0.622	2.203	1.079
360.0	0.049	0.857	0.094	0.523	7.388	0.954	354.6	0.245	0.726	0.029	0.607	2.102	1.092
359.4	0.051	0.834	0.115	0.556	7.665	0.956	354.1	0.247	0.690	0.063	0.650	2.276	1.086
361.2	0.051	0.902	0.048	0.449	5.905	0.957	354.5	0.255	0.727	0.018	0.598	1.996	1.107
360.3	0.052	0.875	0.073	0.503	6.676	0.945	354.3	0.262	0.704	0.035	0.622	2.042	1.103
358.3	0.056	0.825	0.119	0.580	7.642	0.959	354.1	0.264	0.688	0.048	0.637	2.089	1.109
360.4	0.056	0.883	0.061	0.489	5.984	0.947	354.5	0.271	0.723	0.006	0.587	1.845	1.128
359.6	0.062	0.906	0.032	0.480	5.475	0.944	354.3	0.278	0.708	0.014	0.599	1.849	1.137
358.9	0.063	0.850	0.087	0.539	6.176	0.964	353.8	0.283	0.663	0.054	0.652	2.014	1.127
357.7	0.068	0.824	0.108	0.586	6.495	0.958	354.2	0.283	0.694	0.023	0.616	1.875	1.130
358.9	0.068	0.864	0.068	0.528	5.635	0.955	353.9	0.292	0.668	0.041	0.640	1.912	1.136
359.1	0.070	0.874	0.055	0.513	5.220	0.955	354.0	0.292	0.680	0.028	0.623	1.854	1.146
359.1	0.076	0.882	0.042	0.501	4.706	0.959	354.1	0.303	0.687	0.011	0.603	1.723	1.168
359.3	0.077	0.896	0.027	0.487	4.501	0.950	353.9	0.316	0.667	0.017	0.617	1.702	1.179
357.9	0.078	0.842	0.080	0.560	5.383	0.963	353.7	0.319	0.646	0.034	0.644	1.770	1.164
358.2	0.078	0.859	0.063	0.538	5.131	0.964	353.8	0.320	0.658	0.022	0.625	1.710	1.180
358.3	0.082	0.868	0.050	0.531	4.805	0.953	353.6	0.326	0.628	0.047	0.661	1.788	1.165
356.3	0.086	0.803	0.111	0.616	5.705	0.971	353.8	0.338	0.656	0.006	0.609	1.578	1.213
356.5	0.089	0.812	0.099	0.604	5.377	0.968	353.5	0.346	0.625	0.029	0.646	1.652	1.199
358.8	0.091	0.894	0.015	0.477	3.795	0.980	353.4	0.352	0.609	0.039	0.662	1.672	1.197
357.2	0.093	0.834	0.074	0.571	4.742	0.970	353.6	0.354	0.629	0.016	0.631	1.571	1.218
357.4	0.096	0.846	0.058	0.551	4.378	0.976	353.6	0.356	0.632	0.012	0.624	1.547	1.230
357.8	0.099	0.865	0.036	0.527	4.001	0.972	353.2	0.380	0.588	0.033	0.663	1.563	1.237
356.2	0.103	0.806	0.091	0.607	4.717	0.973	353.3	0.394	0.594	0.012	0.638	1.445	1.276
356.7	0.108	0.825	0.068	0.578	4.210	0.979	353.3	0.396	0.596	0.008	0.630	1.419	1.295
357.1	0.109	0.847	0.044	0.549	3.900	0.981	353.2	0.402	0.576	0.023	0.657	1.464	1.271
357.6	0.109	0.869	0.022	0.519	3.620	0.979	352.4	0.403	0.566	0.032	0.676	1.549	1.280
357.8	0.110	0.879	0.011	0.499	3.405	0.990	353.0	0.421	0.553	0.026	0.670	1.435	1.294
355.7	0.120	0.796	0.084	0.611	4.152	0.989	353.0	0.432	0.550	0.018	0.663	1.385	1.313
356.6	0.121	0.826	0.053	0.569	3.700	0.988	353.0	0.441	0.551	0.008	0.647	1.325	1.351
357.4	0.122	0.871	0.007	0.507	3.183	0.996	352.2	0.443	0.529	0.028	0.686	1.440	1.330
356.9	0.123	0.847	0.030	0.543	3.434	0.989	352.8	0.456	0.524	0.020	0.675	1.346	1.346
355.2	0.126	0.777	0.097	0.635	4.182	0.985	352.7	0.467	0.519	0.014	0.668	1.304	1.382
356.4	0.130	0.831	0.039	0.562	3.434	0.993	352.2	0.471	0.505	0.025	0.693	1.369	1.363
356.9	0.130	0.853	0.017	0.540	3.227	0.977	352.5	0.498	0.482	0.021	0.692	1.278	1.407
356.0	0.131	0.808	0.061	0.590	3.618	0.993	352.6	0.500	0.483	0.016	0.685	1.254	1.420
355.3	0.141	0.783	0.076	0.617	3.609	1.000	352.4	0.511	0.479	0.010	0.679	1.226	1.460
354.9	0.149	0.762	0.090	0.637	3.595	1.007	352.3	0.536	0.447	0.018	0.703	1.215	1.474
356.4	0.153	0.835	0.012	0.535	2.776	1.023	352.2	0.552	0.441	0.007	0.690	1.161	1.540
356.1	0.155	0.820	0.025	0.554	2.873	1.022	352.2	0.555	0.433	0.012	0.702	1.176	1.520
355.6	0.159	0.793	0.048	0.590	3.042	1.014	352.1	0.571	0.414	0.015	0.715	1.169	1.533
354.7	0.164	0.755	0.081	0.642	3.306	1.004	351.8	0.610	0.385	0.004	0.712	1.102	1.657
355.7	0.165	0.802	0.034	0.580	2.871	1.012	351.9	0.611	0.376	0.012	0.727	1.117	1.629
353.3	0.165	0.768	0.067	0.641	3.459	1.030	351.8	0.614	0.377	0.009	0.723	1.111	1.641
355.2	0.170	0.775	0.055	0.606	2.957	1.024	351.6	0.665	0.329	0.006	0.743	1.062	1.757
355.9	0.178	0.815	0.007	0.556	2.527	1.018	351.4	0.728	0.268	0.004	0.773	1.018	1.917
355.7	0.179	0.802	0.020	0.565	2.574	1.035	351.2	0.774	0.223	0.003	0.796	0.992	2.089

into mole fractions. The accuracy in the measurement of the ethanol, water, and sodium nitrate mole fractions in the liquid phase was  $\pm 0.003$ . 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 Table 1, the vapor–liquid equilibrium for the ethanol (1) + water (2) + sodium nitrate (3) system at a pressure of 100.0 kPa is reported. Included are the equilibrium temperature ( $T$ ), 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 ( $y_1$ ).

To make possible the thermodynamic treatment of the 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 acceptable in this case in the entire range of compositions, because the solubility of sodium nitrate, which expressed as the salt mole fraction is 0.163 in water at 25 °C, decreases almost linearly when the mole fraction of ethanol

**Table 2. Boiling Points of Water + Sodium Nitrate Mixtures at 100.0 kPa**

$x_3^*$	$T/K$
0.1687	382.4
0.1607	381.9
0.1523	381.4
0.1430	380.8
0.1367	380.4
0.1301	380.0
0.1248	379.7
0.1193	379.4
0.1146	379.1
0.1090	378.8
0.1041	378.4
0.0974	378.0
0.0914	377.7
0.0866	377.4
0.0814	377.1
0.0752	376.8
0.0692	376.4
0.0641	376.1
0.0591	375.8
0.0531	375.5
0.0480	375.2
0.0425	374.9
0.0369	374.6
0.0313	374.3
0.0259	374.0
0.0211	373.7
0.0167	373.5
0.0123	373.3
0.0084	373.1
0.0037	372.9

in the mixed solvent increases, to become practically zero when the mole fraction of alcohol in the ethanol + water mixed solvent is 0.80 (Linke, 1965).

When equilibrium is established between vapor and liquid phases, the activity coefficients of both pseudocomponents will be given by the expressions

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

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 ( $=1 - y_1$ ),  $\phi_I$  is the fugacity coefficient of pseudocomponent I in the vapor phase,  $\phi_{II}$  is the fugacity coefficient of pseudocomponent II in the vapor phase,  $P$  is the system total pressure,  $P_I^0$  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. The value of  $P_{II}^0$  depends on the temperature and on the salt concentration. Jaques and Furter (1972) propose that this dependency can be expressed as

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

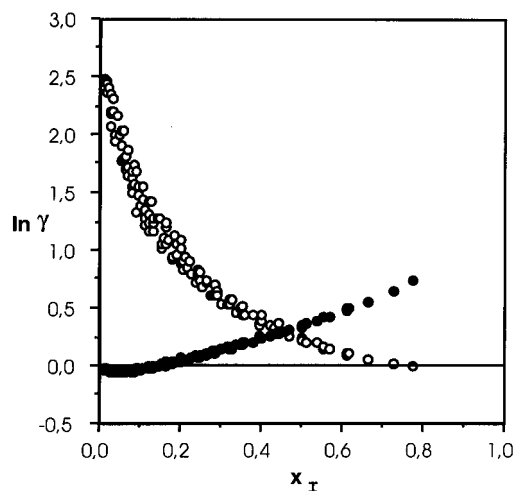
where  $P_2^0$  is the vapor pressure of pure water and  $\epsilon$  is the vapor pressure correction factor, which depends only on  $x_3^*$ , the mole fraction of the salt component in pseudocomponent II [ $=x_3/(x_2 + x_3)$ ].

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 2, and the vapor pressure of pure water obtained from the Antoine equation with the parameters given in the Table 3 (Gmehling *et al.*, 1981), both at the same temperature.

**Table 3. Antoine Coefficients<sup>a</sup> of Pure Components (Gmehling *et al.*, 1981)<sup>a</sup>**

component	A	B	C
ethanol	8.11220	1592.864	226.184
water	8.07131	1730.630	233.426

$$^a \log (P_i^0/\text{mmHg}) = A - B/((T^\circ\text{C}) + C).$$



**Figure 1.** Experimental solvent activity coefficients for the system ethanol (1) + water (2) + sodium nitrate (3) at 100.0 kPa: (O)  $\ln \gamma_I$ ; (●)  $\ln \gamma_{II}$ .

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

$$\epsilon = 1 - 1.723x_3^* + 0.11(x_3^*)^2 \quad (3)$$

The fugacity coefficients  $\phi_I$  and  $\phi_{II}$  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 Tsionopoulos (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 by following the above procedure. The obtained results of  $\gamma_I$  and  $\gamma_{II}$  are shown in columns 6 and 7 of Table 1.

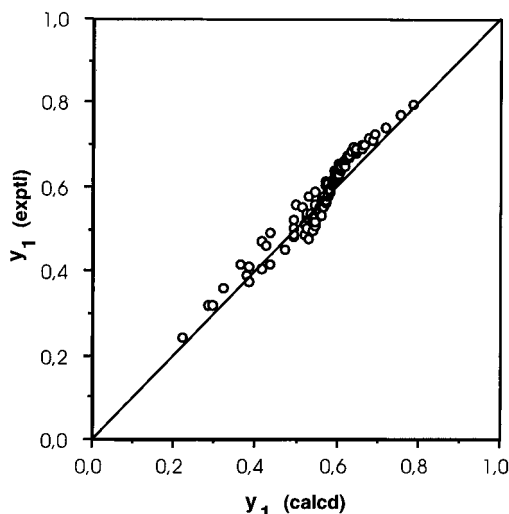
In Figure 1, the logarithms of  $\gamma_I$  and  $\gamma_{II}$  are plotted against  $x_I$ . In this figure, the logarithms of activity coefficients for both pseudocomponents can be adjusted to one curve whatever the salt concentration may be. This fact implies that the pseudobinary model proposed explains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

For the purpose of reproducing experimental data, the activity coefficients obtained for every pseudocomponent were adjusted to the Van Laar model. The obtained parameters are  $A_{12} = 2.460$  and  $A_{21} = 1.020$ .

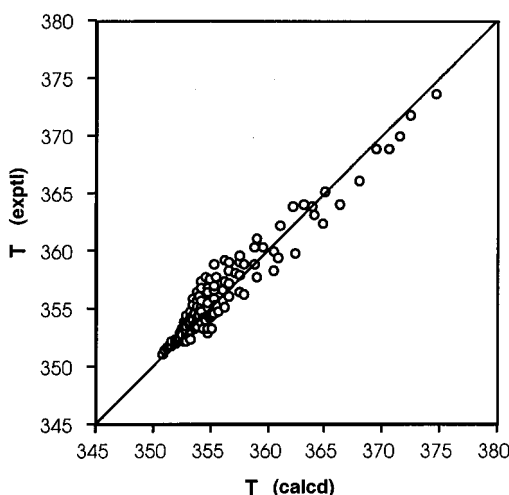
The Van Laar model and the pseudobinary approach were used to recalculate the vapor composition and temperature results in equilibrium from the liquid-phase composition. From comparison of experimental and calculated results, we found the following:

$$y_{\text{exptl}} - y_{\text{calcd}}: \text{ mean, } 0.028 \\ \text{ standard deviation, } 0.024$$

$$T_{\text{exptl}} - T_{\text{calcd}}: \text{ mean, } 1.05 \text{ K} \\ \text{ standard deviation, } 1.25 \text{ K}$$



**Figure 2.** Comparison of the calculated ethanol vapor composition,  $y_1(\text{calcd})$ , with experimental values,  $y_1(\text{exptl})$ .



**Figure 3.** Comparison of calculated bubble points,  $T(\text{calcd})$ , with experimental values,  $T(\text{exptl})$ .

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

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Received for review March 22, 1996. Accepted June 11, 1996.  
Financial support from the Generalitat Valenciana (Grant GV-1006/93) is gratefully acknowledged.

JE960115B

© Abstract published in *Advance ACS Abstracts*, August 1, 1996.