

LIQUID-VAPOUR EQUILIBRIUM AND HEATS OF MIXING IN THE ETHANOL-ACETONITRILE SYSTEM*

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Vapour-liquid equilibrium and heats of mixing were measured in the ethanol-acetonitrile system. The isobaric vapour-liquid equilibrium measurements were carried out at the pressures of 43.35, 56.44, 69.48 and 86.05 kPa and the heats of mixing were measured at the temperatures of 298.15, 308.15 and 318.15 K. The individual vapour-liquid equilibrium data sets were correlated by various two-parameter equations and the data on heats of mixing by the Redlich-Kister polynomial. The extensive data obtained for both excess functions, covering the temperature range of 50 K, were correlated successfully by the Wilson equation with the energy parameters linearly dependent on temperature.

Experimental data on the properties of solutions of *n*-alcohols with acetonitrile are relatively rare. In one of our foregoing papers¹, we have reported the vapour-liquid equilibrium data in the methanol-acetonitrile system at some conditions. Analogously to this system, practically no experimental data on vapour-liquid equilibrium and only limited ones on excess enthalpy² were available until lately for the ethanol-acetonitrile system as well. This situation stimulated experimentalists so that in recent years, the isothermal static measurements³ of vapour-liquid equilibrium at the temperatures 293.15, 343.15 and 395.15 K and the circulation measurements⁴ at the temperature 313.15 K have been published. In this work we report the results of isobaric vapour-liquid equilibrium measurements at four subatmospheric pressures and of heat of mixing measurements at three temperatures.

EXPERIMENTAL

Substances Used

A superfine absolute ethanol, produced by Lachema, was dried by copper sulphate. After shaking always with freshly calcined sulphate several times, the salt traces were removed by distillation. The final drying of the product was carried out by molecular sieves 3A. The water content in the final product as found by the Fischer analysis was 0.01 mass%.

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Acetonitrile, A.R., produced by Avondale Laboratories, was distilled on an 1.5 m long column filled with stainless steel helices. The product was dried up with molecular sieves 3A. The water content found by the Fisher analysis was 0.007 mass%.

The measured values of density and refractive index of both the substances are compared with the literature ones in Table I.

Measurement of Vapour-Liquid Equilibrium and Heat of Mixing

The vapour-liquid equilibrium was measured using a modified circulation still of the Gillespie type⁵. In isobaric arrangement, the oil pump is connected *via* a damping volume capacity to the equilibrium still and controlled by a mercury manostat. Temperature is measured by a platinum resistance thermometer calibrated *in situ* by several ebulliometric standards (water, benzene). To analyze the equilibrium mixtures, we used interferometry¹. The measurement of heats of mixing was carried out in an isothermal calorimeter described⁶ in detail before.

RESULTS

The vapour-liquid equilibrium was determined at four pressures: 45.35, 56.44, 69.48 and 86.05 kPa. The activity coefficients γ_i and the excess Gibbs energy G^E were calculated from the experimental T , P , x , y values in terms of the following relations

$$\ln \gamma_i = \ln \frac{y_i P}{x_i P_i^s} + \frac{(B_{ii} - v_i^L)(P - P_i^s)}{RT} + \frac{P D_{ij}(1 - y_i)^2}{RT}, \quad (1)$$

$$D_{ij} = 2B_{ij} - B_{ii} - B_{jj}$$

and

$$G^E \equiv \frac{g^E}{RT} = x_1 \ln \gamma_1 + x_2 \ln \gamma_2, \quad (2)$$

where x_i , y_i are the mole fractions of component i in the liquid and vapour phases, respectively, v_i^L is the molar volume of pure liquid component i , P the total pressure and P_i^s the saturated vapour pressure of pure component i at the temperature T . The second virial coefficients B_{ii} , B_{ij} were estimated by means of the correlation by O'Connell and Prausnitz⁷. The saturated vapour pressures were calculated from the Antoine equation whose constants are given in Table I.

The inaccuracies of the measured and further input quantities were estimated as follows: $\sigma_{(x)} = \sigma_{(y)} = 0.0005$, $\sigma_{(T)} = 0.02$ K, $\sigma_{(P)} = 40$ Pa, $\sigma_{(P_i^s)} = 170$ Pa, $\sigma_{(B)} = 300$ cm³ mol⁻¹. According to the error propagation formula, the variances of calculated activity coefficients were determined⁸ from these inaccuracies. The found variances were used partly in the consistence test, partly as the weighting factors in the objective function minimized with the aim to find the best values of parameters of correlation equation. The experimental data, the derived values of activity coefficients and their estimated errors are summarized in Table II.

The consistence of the measured equilibrium data was checked by an integral test. For isobaric data subject to no experimental errors, should hold

$$D \equiv \int_{x_1^{(1)}}^{x_1^{(n)}} \left(\ln \frac{\gamma_1}{\gamma_2} - \frac{h^E}{RT^2} \frac{\partial T}{\partial x_1} \right) dx_1 + G^E(x_1^{(1)}) - G^E(x_1^{(n)}) = 0, \quad (3)$$

where $x_1^{(1)}$, $x_1^{(n)}$ is the lowest and highest, respectively, experimental value of mole fraction x_1 . The value of the integral was determined by the trapezoidal rule. The calculated values of D for single sets are given in Table III. Considering that the experimental data are always subject to random errors, the value of D is not generally equal zero. The permitted tolerance is often chosen empirically. However, it can be determined as a confidence interval by calculation from inaccuracies of input quantities in terms of the error propagation formula. Comparing the values D with their 95% confidence intervals in Table III, we can see that for the first three data sets, the absolute value of D is lower than the value of $2\sigma(D)$ or comparable. These data can be considered consequently as consistent in the framework of the above-mentioned errors of input quantities. For the set at the highest pressure (86.05 kPa), $|D|$ is fairly small, nevertheless significantly larger than the value of $2\sigma(D)$, and therefore the experimental errors and/or the errors in further input quantities are here apparently higher than the values given above. Let us remark that possible discre-

TABLE I

Density and refractive index of pure substances at 298.15 K. The temperature dependence of saturated vapour pressures and of liquid molar volumes

	Ethanol	Acetonitrile
Density, g cm ⁻³	0.7852 0.78506 ^a	0.7766 0.77656 ^b
Refractive index	1.35945 1.35955 ^a	1.34150 1.34154 ^b
Constants of the Antoine equation ^c	7.23710 ^d 1 592.864 -46.966	6.46476 ^e 1 482.29 -22.627
Constants of the temperature dependence of liquid molar volume ^f	34.52 ^a 0.07996	31.31 ^a 0.07227

^a Ref.¹⁷; ^b ref.¹⁸; ^c $\log P^s$ [kPa] = $A - B/(T$ [K] + $C)$; ^d ref.¹⁹; ^e ref.²⁰; ^f v^L [cm³/mol] = $= v_A + v_B T$ [K].

TABLE II
Experimental vapour-liquid equilibrium data

x_1	y_1	T, K	$\ln \gamma_1$	$\ln \gamma_2$	$\sigma(\ln \gamma_1)$	$\sigma(\ln \gamma_2)$
$P = 45.35 \text{ kPa}$						
0.0274	0.0700	330.00	1.057	-0.013	0.023	0.004
0.0579	0.1198	328.91	0.894	0.001	0.014	0.004
0.0956	0.1782	327.75	0.841	0.014	0.011	0.005
0.1551	0.2508	326.64	0.748	0.029	0.009	0.005
0.1832	0.2799	326.22	0.710	0.038	0.009	0.005
0.2341	0.3262	325.73	0.639	0.054	0.008	0.005
0.2873	0.3643	325.37	0.561	0.081	0.007	0.005
0.3183	0.3832	325.19	0.517	0.102	0.007	0.005
0.3886	0.4205	324.98	0.419	0.156	0.007	0.005
0.4351	0.4429	324.96	0.359	0.197	0.007	0.006
0.4846	0.4669	324.94	0.304	0.246	0.007	0.006
0.5309	0.4884	324.96	0.257	0.298	0.006	0.006
0.5885	0.5211	325.02	0.215	0.362	0.006	0.006
0.6814	0.5693	325.42	0.138	0.498	0.006	0.006
0.7192	0.5937	325.65	0.115	0.558	0.006	0.007
0.8051	0.6588	326.66	0.059	0.714	0.005	0.007
0.8697	0.7327	327.90	0.031	0.831	0.005	0.009
0.8935	0.7707	328.63	0.021	0.854	0.005	0.010
0.9325	0.8295	329.64	0.006	0.980	0.005	0.012
0.9726	0.9163	331.03	0.001	1.125	0.004	0.022
$P = 56.44 \text{ kPa}$						
0.0412	0.0910	335.47	0.886	0.001	0.018	0.003
0.0676	0.1419	334.53	0.874	0.003	0.014	0.004
0.0873	0.1755	333.86	0.859	0.007	0.012	0.004
0.1212	0.2210	333.08	0.795	0.014	0.011	0.004
0.1831	0.2897	332.00	0.698	0.032	0.009	0.004
0.2325	0.3290	331.42	0.611	0.057	0.008	0.004
0.2615	0.3514	331.17	0.570	0.071	0.008	0.004
0.3319	0.3978	330.70	0.476	0.113	0.007	0.005
0.3943	0.4342	330.47	0.401	0.157	0.006	0.005
0.4941	0.4838	330.41	0.285	0.249	0.006	0.005
0.5480	0.5106	330.44	0.233	0.307	0.006	0.006
0.5887	0.5334	330.57	0.199	0.350	0.005	0.006
0.6427	0.5584	330.77	0.148	0.429	0.005	0.006
0.7073	0.5976	331.14	0.103	0.524	0.005	0.007
0.7598	0.6361	331.61	0.073	0.606	0.005	0.007
0.8152	0.6779	332.23	0.038	0.726	0.004	0.008
0.8607	0.7237	332.90	0.019	0.834	0.004	0.009
0.9007	0.7714	333.70	0.002	0.957	0.004	0.011
0.9290	0.8256	334.56	0.002	0.995	0.004	0.013
0.9451	0.8612	335.08	0.004	1.007	0.004	0.015

TABLE II
 (Continued)

x_1	y_1	T, K	$\ln \gamma_1$	$\ln \gamma_2$	$\sigma(\ln \gamma_1)$	$\sigma(\ln \gamma_2)$
$P = 69.48 \text{ kPa}$						
0.0576	0.1264	340.81	0.858	0.002	0.017	0.003
0.0911	0.1817	339.70	0.807	0.008	0.014	0.003
0.1383	0.2411	338.55	0.718	0.023	0.011	0.003
0.2115	0.3188	337.33	0.622	0.044	0.009	0.004
0.2423	0.3451	336.97	0.580	0.057	0.008	0.004
0.2824	0.3763	336.63	0.527	0.074	0.008	0.004
0.3383	0.4072	336.27	0.440	0.116	0.007	0.004
0.3655	0.4247	336.13	0.410	0.133	0.007	0.005
0.4415	0.4654	335.91	0.321	0.195	0.006	0.005
0.4622	0.4746	335.88	0.296	0.217	0.006	0.005
0.5085	0.5004	335.87	0.253	0.258	0.006	0.006
0.5460	0.5214	335.87	0.223	0.294	0.005	0.006
0.6406	0.5766	336.11	0.152	0.399	0.005	0.007
0.6772	0.5973	336.27	0.125	0.451	0.004	0.007
0.7557	0.6483	336.79	0.074	0.579	0.004	0.008
0.8141	0.6907	337.33	0.040	0.707	0.004	0.009
0.8584	0.7341	338.01	0.019	0.808	0.004	0.011
0.9004	0.7877	338.86	0.005	0.909	0.003	0.013
0.9325	0.8442	339.67	0.005	0.964	0.003	0.015
0.9496	0.8757	340.20	0.002	1.014	0.003	0.018
$P = 86.05 \text{ kPa}$						
0.0549	0.1273	347.26	0.863	-0.002	0.020	0.003
0.0813	0.1622	346.33	0.748	0.013	0.017	0.003
0.1448	0.2506	344.52	0.675	0.029	0.013	0.003
0.2153	0.3210	343.23	0.575	0.058	0.010	0.004
0.2412	0.3453	342.89	0.548	0.066	0.010	0.004
0.2946	0.3815	342.32	0.469	0.100	0.009	0.004
0.3422	0.4183	341.93	0.427	0.121	0.008	0.005
0.4251	0.4670	341.59	0.332	0.180	0.007	0.005
0.4397	0.4734	341.54	0.314	0.196	0.006	0.005
0.5029	0.4997	341.51	0.234	0.266	0.006	0.006
0.5085	0.5032	341.51	0.230	0.270	0.006	0.006
0.5844	0.5462	341.59	0.169	0.346	0.005	0.007
0.6097	0.5605	341.64	0.150	0.375	0.005	0.007
0.6810	0.6027	341.93	0.099	0.468	0.004	0.008
0.7230	0.6320	342.14	0.077	0.527	0.004	0.009
0.7972	0.6905	342.81	0.040	0.647	0.003	0.011
0.8561	0.7480	343.51	0.020	0.765	0.003	0.013
0.8910	0.7893	344.12	0.009	0.846	0.003	0.014
0.9158	0.8228	344.63	0.002	0.917	0.003	0.016
0.9545	0.8895	345.72	-0.005	1.029	0.003	0.021

pancies in the estimate of virial coefficients manifest themselves most strongly in case of this data set; similarly, the uncertainty of the temperature extrapolation of heat of mixing (could not be included into $\sigma(D)$) is here largest.

The measured vapour-liquid equilibrium data were correlated by several two-parameter equations: Margules, van Laar, Wilson⁹, NRTL (ref.¹⁰), enthalpy Wilson (HMW) (ref.¹¹), UNIQUAC (ref.¹²), and LCG (ref.¹³). To evaluate their adjustable parameters, the following objective function was minimized

$$S = \sum_{j=1}^m \sum_{i=1}^2 (\ln \gamma_{ij}^{\text{exp}} - \ln \gamma_{ij}^{\text{calc}})^2 / \sigma^2 (\ln \gamma_{ij}^{\text{exp}}), \quad (4)$$

where m is the number of experimental points.

The equations based on the local composition concept describe the measured data altogether better than the classical ones. The constants and correlation deviations are given in Table IV only for two most successful equations, *viz.* the Wilson one

$$\ln \gamma_i = -\ln(x_i + A_{ij}x_j) + x_j[A_{ij}/(x_i + A_{ij}x_j) - A_{ji}/(x_j + A_{ji}x_i)] \quad (5)$$

and HMW

$$\ln \gamma_i = -\frac{x_j \ln(A_{ij}/A_{ji})}{(x_i + A_{ij}x_j)(x_j + A_{ji}x_i)} \left[1 + x_i \left(1 - \frac{1}{x_i + A_{ij}x_j} - \frac{A_{ji}}{x_j + A_{ji}x_i} \right) \right], \quad (6)$$

where

$$A_{ij} = (v_j^L/v_i^L) \exp(-a_{ij}/T). \quad (7)$$

The heats of mixing were measured at the temperatures of 298.15, 308.15 and 318.15 K in the entire concentration range. The absolute accuracy of measured

TABLE III

Consistency test of the measured vapour-liquid equilibrium data

Set, kPa	$ D \cdot 10^3$	$2\sigma(D) \cdot 10^3$
45.35	11.1	13.0
56.44	12.7	11.4
69.48	14.2	10.0
86.05	31.6	9.1

values is better than 1%. The temperature dependence of h^E is increasing and the maximum values of h^E at the temperatures mentioned are 1 503, 1 576 and 1 654 J. mol⁻¹. The data reported by Mato and Coca² are in good agreement with our measurements. Shape of the $h^E(x_1)$ curves is the same, only at the maximum a difference of 1.5% (temperature 298.15 K) was found. No other measurements of heats of mixing in this system were found in the literature.

The concentration dependence of enthalpy of mixing was represented by the Redlich-Kister polynomial

$$H^E \equiv \frac{h^E}{RT} = x_1 x_2 \sum_{k=0}^p A_k (x_1 - x_2)^k, \quad (8)$$

where A_k are adjustable parameters. Number of these parameters p was controlled by the statistical criterion of significance of difference of two variances (F -test). The same weight was prescribed to all points. The experimental data, calculated constants of the Redlich-Kister expansion and standard deviation of correlation are given in Table V.

The numerous experimental data for both excess functions in the temperature range of approximately 50 K yield the possibility of their simultaneous treatment.

TABLE IV

Constants of the Wilson (upper data) and HMW (lower data) equations, standard deviations σ_{cor} and variance of fit for the representation of single vapour-liquid equilibrium data sets

Set, kPa	a_{12} , K	a_{21} , K	σ_{cor}^a	σ^b
45.35	133.0	292.1	0.017	1.57
	73.0	177.6	0.017	1.54
56.44	124.7	301.1	0.014	1.39
	72.1	179.5	0.015	1.43
69.48	125.7	284.7	0.015	1.55
	73.4	174.5	0.016	1.57
86.05	166.2	233.4	0.032	2.40
	93.5	153.1	0.034	2.41

$$^a \sigma_{\text{cor}} = [1/(2m - 2) \sum_{j=1}^m \sum_{i=1}^2 (\ln \gamma_{ij}^{\text{exp}} - \ln \gamma_{ij}^{\text{calc}})^2]^{1/2}; \quad ^b \sigma = [S/(2m - 2)]^{1/2}.$$

On the basis of our previous experience⁸ and that of other authors^{14,15}, the Wilson equation with the energy parameters linearly dependent on temperature was used for this purpose:

$$G^E = -x_1 \ln(x_1 + A_{12}x_2) - x_2 \ln(x_2 + A_{21}x_1), \quad (9)$$

$$H^E = \frac{x_1x_2}{T} \left(\frac{A_{12}a_1}{x_1 + A_{12}x_2} + \frac{A_{21}a_2}{x_2 + A_{21}x_1} \right), \quad (10)$$

TABLE V

Experimental data on heat. of mixing, constants of the Redlich-Kister expansion and standard deviations of correlation

298.15 K		308.15 K		318.15 K ^a	
x_1	H^E	x_1	H^E	x_1	H^E
0.1611	0.378	0.1769	0.409	0.1485	0.356
0.1884	0.423	0.2128	0.455	0.1754	0.405
0.2125	0.454	0.2459	0.495	0.2082	0.452
0.2319	0.479	0.2754	0.528	0.2403	0.491
0.2640	0.511	0.3362	0.575	0.2643	0.519
0.2918	0.540	0.3627	0.594	0.2847	0.540
0.3180	0.557	0.4022	0.603	0.3252	0.572
0.3388	0.571	0.4206	0.609	0.3629	0.594
0.3692	0.586	0.4546	0.614	0.3976	0.607
0.4487	0.603	0.4683	0.615	0.4480	0.617
0.4688	0.605	0.4950	0.615	0.4983	0.623
0.4948	0.606	0.5403	0.606	0.5660	0.616
0.5798	0.579	0.5886	0.588	0.5733	0.601
0.6208	0.561	0.6198	0.571	0.6207	0.580
0.6633	0.529	0.6606	0.538	0.6609	0.549
0.6928	0.503	0.6962	0.508	0.6904	0.524
0.7188	0.477	0.7275	0.480	0.7159	0.499
0.7439	0.452	0.7939	0.398	0.7474	0.467
0.7719	0.422	0.8282	0.361	0.7773	0.428
0.8149	0.360	0.8442	0.322	0.8106	0.383
A_0	2.4138		2.4520		2.4876
A_1	-0.2843		-0.2483		-0.2149
A_2	0.4178		0.4138		0.3621
σ^a	0.0017		0.0035		0.0031

$$^a \sigma = \left[\sum_{j=1}^m (H_{\text{exp},j}^E - H_{\text{calc},j}^E)^2 / (m - 3) \right]^{1/2}.$$

TABLE VI

Summary of results of simultaneous correlation. Constants of the Wilson equation, mean absolute deviations δ and deviations S_v for single data sets, variance of fit σ

	a_1 , K	b_1	a_2 , K	b_2
	776.6	-1.9214	404.8	-0.3715
Quantity	Set	δ	S_v^a	
G^E	45.35 kPa	0.004	1.24	
G^E	56.44 kPa	0.001	0.42	
G^E	69.48 kPa	0.002	0.49	
G^E	86.05 kPa	0.003	1.14	
H^E	298.15 K	0.002	0.44	
H^E	308.15 K	0.003	0.98	
H^E	318.15 K	0.003	0.72	
Overall		0.003	1.19 ^b	

$$^a S_v = [(1/m) \sum_{j=1}^m (F_{\text{exp},j}^E - F_{\text{calc},j}^E)^2 / \sigma^2 (F_{\text{exp},j}^E)]^{1/2}, m \text{ is number of points in the set,}$$

$$^b \sigma = [S / (n_G + n_H - 4)]^{1/2}.$$

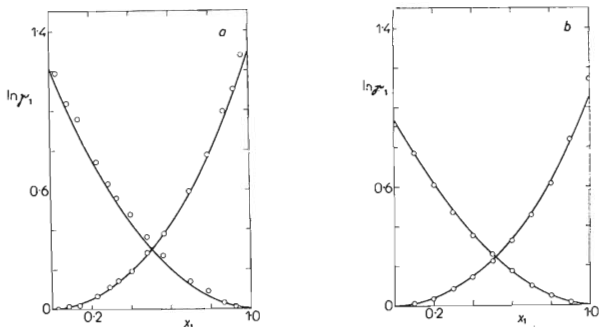


FIG. 1

Ethanol(1)-acetonitrile(2) system. Comparison of our experimental data with those of other authors. Solid line calculated from our data in terms of the Wilson equation (the constants obtained by simultaneous correlation of G^E and H^E); experimental points: *a* 313.15 K, data by Sugi and Katayama⁴, *b* 343.15 K, data by Muthu and coworkers³.

where

$$A_{ij} = (v_j^L/v_i^L) \exp [-(\lambda_{ij} - \lambda_{ii})/RT], \quad (11)$$

$$(\lambda_{ij} - \lambda_{ii})/R = a_i + b_i T. \quad (12)$$

Four adjustable constants a_1 , b_1 , a_2 , b_2 were evaluated by the weighted least-squares method on using the following objective function

$$S = \frac{n_G + n_H}{n_G} \sum_{j=1}^{n_G} \frac{(G_{\text{exp},j}^E - G_{\text{calc},j}^E)^2}{\sigma^2(G_{\text{exp},j}^E)} + \frac{n_G + n_H}{n_H} \sum_{j=1}^{n_H} \frac{(H_{\text{exp},j}^E - H_{\text{calc},j}^E)^2}{\sigma^2(H_{\text{exp},j}^E)}, \quad (13)$$

where n_G , n_H is the number of experimental values of excess Gibbs energy or excess enthalpy, respectively. The variances $\sigma^2(G_{\text{exp},j}^E)$ were estimated from the knowledge of variances of activity coefficients $\sigma^2(\ln \gamma_{ij}^{\text{exp}})$ and of composition $\sigma^2(x)$. To estimate the variances $\sigma^2(H_{\text{exp},j}^E)$, the 1% inaccuracy of experimental H^E data, *i.e.* $\sigma^2(H_{\text{exp},j}^E) = (0.01 H_{\text{exp},j}^E)^2$ was considered. To minimize the objective function, we used the Marquardt algorithm¹⁶. The calculated values of parameters and mean deviations of calculated and experimental values are given in Table VI. The quantities σ and S_v in this table estimate the ratio of correlation deviations and experimental errors for the entire set and single subsets, respectively. For well-represented data, their value should be close to unity. It can be seen in Table VI that the Wilson equation represents our experimental data altogether in the framework of estimated inaccuracies.

On using the given representation, we can easily compare our vapour-liquid equilibrium data with the data which have appeared recently in the literature. Fig. 1 shows very good agreement with the data of Sugi and Katayama⁴ and excellent agreement with those of Muthu and coworkers³.

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