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Vapor–Liquid Equilibria for Glycol Ether + Water Systems

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A Dvořak–Boublík recirculating still has been used to measure isothermal vapor–liquid equilibrium data for 1,2-ethanediol + water and for five different glycol ether + water systems. The glycol ethers are 2-methoxyethanol, 2-ethoxyethanol, 1-methoxy-2-propanol, 2-isopropoxyethanol, and 2-butoxyethanol. Each system has been measured at two temperatures in the range 343.15–368.15 K. The reliability of the data has been checked by using a maximum likelihood data reduction.

Introduction

Glycol ethers are high-boiling liquids which may be used as solvents in paints. They may, thereby, substitute usual organic solvents, even allowing the use of water as a major solvent.

Very little information concerning isothermal vapor–liquid equilibrium (VLE) behavior exists for aqueous glycol ether systems. We have found several data sets for 1,2-ethanediol (*1–4*) and three sets for 2-butoxyethanol (*5–7*). Isobaric data are available for 1,2-ethanediol (*8–14*), 2-ethoxyethanol (*15–17*), and 2-butoxyethanol (*18*) systems.

Experimental Section

Apparatus and Procedures. The VLE measurements were carried out in an all-glass cell, originally proposed by Dvořak and Boublík (*19*) and later modified by Boublikova and Lu (*20*). The apparatus has been used previously in this laboratory to give accurate VLE data (*21–23*). The cell provides for the recirculation of the liquid and condensed vapor phases.

The temperature of the equilibrium mixture was determined with a Systemteknik S1220 platinum resistance thermometer with a resolution of 0.01 K and an IPTS-68 calibration. The pressure was measured with a mercury-filled U-tube manometer with an inner diameter of 20 mm. The mercury levels were read by means of a Surrey cathetometer (±0.01 mm). The pressure readings were corrected from ambient (~23 °C and gravity 9.815 59 m s^{-2}) to standard conditions (0 °C and 9.806 65 m s^{-2}) (*24*). The compositions of the liquid and condensed vapor phases were determined by measuring the density at 20 °C and comparing the results with densities of mixtures of known composition. The densities were measured with an Anton Paar Model DMA-55 digital density meter (±10^{-5} g cm^{-3}). The instrument was connected to a Heto circulating thermostatic bath with a temperature control of ±0.01 K.

Density–Composition Calibration and Measurements. About 24 mixtures of known composition were prepared gravimetrically for each binary system, with a precision of 0.1 mg. The compositions covered the whole concentration range from pure water to pure organic compound. The densities were fitted to a sixth-order polynomial. Unknown compositions of liquid samples were then determined through inverse interpolation. Calibration and sample densities were measured in duplicate with an average reproducibility of 2 × 10^{-5} g cm^{-3}, so that the sample compositions are accurate within 0.0005 mole fraction. It should be noted that some of the density–mole fraction curves show a maximum. It was, however, always possible to uniquely obtain the composition of the sample in the region of this maximum by adding, after the measurement was done, a very small amount of one of the pure components and observing the change in the density.

Table I. Sources, Purities, and Densities ρ_i of the Compounds

substance	supplier	purity (%) (min)	ρ_i(293.15 K) (g cm^{-3}) exptl	ρ_i(293.15 K) (g cm^{-3}) lit. (*25*)
1,2-ethanediol	Merck	99.5	1.113 47	1.113 5
2-methoxyethanol	Merck	99.5	0.964 44	0.964 59
2-ethoxyethanol	Fluka	99.5	0.930 13[a]	0.929 45
1-methoxy-2-propanol	Aldrich	98	0.921 21	[b]
2-isopropoxyethanol	Aldrich	99	0.903 95	[b]
2-butoxyethanol	Aldrich	99	0.900 37	0.900 75

[a] At 292.88 K. [b] No available data found in the literature.

Table II. Antoine Parameters, Equation 1, and Mean Deviations AAD(P^s), Equation 2

substance	A_i	B_i	C_i	AAD(P^s) (kPa)
water	6.780 38	1481.069	−62.966	0.036
2-methoxyethanol	6.951 81	1787.028	−36.273	
2-ethoxyethanol	7.040 27	1856.286	−38.980	
1-methoxy-2-propanol	6.878 50	1705.242	−42.892	0.042
2-isopropoxyethanol	7.212 73	1998.488	−29.713	0.024
2-butoxyethanol	5.898 26	1266.270	−117.913	0.007

Table III. Experimental Vapor Pressures P^s of Glycol Ethers as a Function of Temperature T

T (K)	P^s (kPa)	T (K)	P^s (kPa)	T (K)	P^s (kPa)
		1-Methoxy-2-propanol			
331.05	9.153	357.92	29.160	375.94	57.361
339.34	13.452	363.15	35.796	382.06	70.997
353.15	24.042	369.40	45.264	389.40	90.583
		2-Isopropoxyethanol			
358.15	13.426	371.00	22.725	382.78	35.664
364.26	17.372	374.02	25.638		
368.15	20.261	377.59	29.391		
		2-Butoxyethanol			
358.17	4.241	378.45	10.930	398.38	24.181
368.15	6.883	388.79	16.720		

Materials. Table I shows the pure components used, their suppliers, and minimum guaranteed purities. In order to check for minor water impurities, the glycol ethers were contacted with 0.3-nm molecular sieves for a period of 12 h. No significant change in the density was observed. Table I also lists the experimental and literature densities of the substances. Double-distilled water was used.

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Table IV. Vapor–Liquid Equilibrium Data for Water (1) + 1,2-Ethanediol (2) at 343.15 and 363.15 K: Liquid Mole Fraction x_i, Temperature T, Vapor Mole Fraction y_i, and Pressure P

x_1	T (K)	y_1	P (kPa)	x_1	T (K)	y_1	P (kPa)	x_1	T (K)	y_1	P (kPa)
0.1986	343.10	0.9562	6.053	0.4892	343.19	0.9875	15.017	0.7682	343.17	0.9969	23.989
0.2419	343.18	0.9654	7.391	0.5385	343.20	0.9905	16.557	0.8144	343.17	0.9975	25.467
0.2784	343.12	0.9729	8.417	0.5873	343.13	0.9922	18.027	0.8484	343.18	0.9984	26.438
0.3264	343.18	0.9770	9.926	0.6300	343.20	0.9933	19.513	0.8921	343.17	0.9989	27.923
0.3670	343.15	0.9805	11.140	0.6854	343.19	0.9952	21.201	0.9349	343.18	0.9996	29.258
0.4420	343.20	0.9858	13.500	0.7316	343.18	0.9964	22.753	1.0000	343.15	1.0000	31.188
0.1395	363.18	0.9105	10.378	0.3786	363.10	0.9741	26.622	0.7765	363.15	0.9954	54.620
0.1638	363.14	0.9197	11.944	0.4520	363.16	0.9819	31.699	0.8228	363.17	0.9967	57.961
0.1866	363.13	0.9344	13.362	0.4989	363.16	0.9842	34.833	0.8491	363.10	0.9974	59.614
0.1933	363.12	0.9433	14.079	0.5437	363.18	0.9868	38.160	0.8945	363.17	0.9984	62.778
0.2204	363.10	0.9494	15.953	0.5855	363.15	0.9892	41.653	0.9364	363.15	0.9991	66.016
0.2548	363.11	0.9634	17.976	0.6332	363.20	0.9916	44.707	1.0000	363.15	1.0000	70.149
0.3057	363.20	0.9678	21.518	0.6869	363.19	0.9930	48.508				
0.3384	363.11	0.9724	23.943	0.7357	363.20	0.9945	51.948				

Table V. Vapor–Liquid Equilibrium Data for Water (1) + 2-Methoxyethanol (2) at 343.15 and 363.15 K: Liquid Mole Fraction x_i, Temperature T, Vapor Mole Fraction y_i, and Pressure P

x_1	T (K)	y_1	P (kPa)	x_1	T (K)	y_1	P (kPa)	x_1	T (K)	y_1	P (kPa)
0.0000	343.15	0.0000	13.444	0.2552	343.17	0.5043	20.988	0.6730	343.15	0.8010	28.362
0.0544	343.20	0.1649	15.175	0.2999	343.18	0.5482	22.004	0.7612	343.14	0.8511	29.398
0.1061	343.11	0.2853	16.797	0.3665	343.13	0.6046	23.471	0.8272	343.20	0.8901	30.162
0.1595	343.20	0.3820	18.416	0.4524	343.15	0.6590	25.021	0.9075	343.17	0.9330	30.828
0.2013	343.19	0.4432	19.592	0.5457	343.20	0.7181	26.682	0.9574	343.20	0.9659	31.239
0.2290	343.16	0.4796	20.289	0.5986	343.16	0.7553	27.338	1.0000	343.15	1.0000	31.188
0.0000	363.15	0.0000	30.537	0.2639	363.10	0.5085	48.263	0.6984	363.11	0.8078	65.756
0.0606	363.18	0.1691	35.108	0.3159	363.13	0.5613	51.298	0.7790	363.12	0.8449	67.678
0.1073	363.12	0.2809	38.605	0.3749	363.15	0.6103	54.308	0.8502	363.15	0.8901	68.958
0.1641	363.18	0.3774	42.306	0.4535	363.12	0.6632	57.889	0.9081	363.14	0.9245	70.197
0.2084	363.19	0.4437	45.131	0.5448	363.15	0.7196	61.434	0.9582	363.16	0.9618	70.516
0.2414	363.12	0.4841	46.931	0.6121	363.18	0.7622	63.405	1.0000	363.15	1.0000	70.149

Table VI. Vapor–Liquid Equilibrium Data for Water (1) + 2-Ethoxyethanol (2) at 343.15 and 363.15 K: Liquid Mole Fraction x_i, Temperature T, Vapor Mole Fraction y_i, and Pressure P

x_1	T (K)	y_1	P (kPa)	x_1	T (K)	y_1	P (kPa)	x_1	T (K)	y_1	P (kPa)
0.0000	343.15	0.0000	8.659	0.3035	343.20	0.7077	21.154	0.7021	343.18	0.8735	30.092
0.0459	343.15	0.2933	10.927	0.3622	343.17	0.7386	22.537	0.7635	343.17	0.8894	30.717
0.0724	343.19	0.3759	12.219	0.4143	343.20	0.7654	23.865	0.8325	343.17	0.8998	31.140
0.1016	343.18	0.4513	13.526	0.4575	343.13	0.7866	25.038	0.8909	343.20	0.9160	31.467
0.1462	343.20	0.5378	15.596	0.5007	343.14	0.8046	26.037	0.9425	343.20	0.9298	31.691
0.1800	343.18	0.5885	16.855	0.5438	343.19	0.8210	27.010	1.0000	343.15	1.0000	31.188
0.2192	343.19	0.6378	18.466	0.5913	343.20	0.8404	27.990				
0.2702	343.15	0.6788	19.953	0.6510	343.18	0.8595	29.099				
0.0000	363.15	0.0000	20.606	0.2848	363.10	0.6684	46.479	0.5882	363.13	0.8377	64.683
0.0474	363.20	0.2804	26.117	0.3242	363.16	0.7003	49.461	0.6505	363.17	0.8550	67.041
0.0751	363.19	0.3659	29.078	0.3619	363.11	0.7306	52.456	0.7070	363.14	0.8713	68.858
0.1071	363.19	0.4347	31.927	0.4053	363.10	0.7587	55.375	0.7633	363.12	0.8861	70.138
0.1625	363.10	0.5199	36.149	0.4450	363.12	0.7776	57.954	0.9410	363.17	0.9340	71.627
0.1973	363.10	0.5724	39.266	0.4983	363.15	0.8002	60.558	1.0000	363.15	1.0000	70.149
0.2505	363.11	0.6329	43.516	0.5547	363.15	0.8268	63.440				

Table VII. Vapor–Liquid Equilibrium Data for Water (1) + 1-Methoxy-2-propanol (2) at 353.15 and 363.15 K: Liquid Mole Fraction x_i, Temperature T, Vapor Mole Fraction y_i, and Pressure P

x_1	T (K)	y_1	P (kPa)	x_1	T (K)	y_1	P (kPa)	x_1	T (K)	y_1	P (kPa)
0.0000	353.15	0.0000	24.042	0.3470	353.15	0.6171	43.832	0.7112	353.15	0.7792	51.108
0.0471	353.15	0.1746	29.232	0.4150	353.15	0.6547	45.754	0.7859	353.15	0.8074	51.498
0.1285	353.15	0.3623	33.303	0.4908	353.15	0.6906	47.809	0.8564	353.15	0.8544	51.457
0.1895	353.15	0.4629	36.904	0.5580	353.15	0.7255	49.167	0.9499	353.15	0.8982	50.449
0.2617	353.15	0.5453	40.427	0.6289	353.15	0.7488	50.235	1.0000	353.15	1.0000	47.548
0.0000	363.15	0.0000	35.796	0.3564	363.15	0.6183	65.469	0.7063	363.15	0.7778	76.046
0.0844	363.15	0.2723	45.260	0.4249	363.15	0.6572	68.652	0.7861	363.15	0.8061	76.692
0.1268	363.15	0.3611	49.337	0.5013	363.15	0.6933	71.410	0.8604	363.15	0.8364	76.678
0.1982	363.15	0.4770	55.525	0.5691	363.15	0.7236	73.570	0.9524	363.15	0.8954	75.895
0.2822	363.15	0.5620	61.376	0.6375	363.15	0.7508	75.166	1.0000	363.15	1.0000	70.194

Results and Discussion

Vapor Pressures of Pure Substances. The VLE measurements were carried out as near to isothermal conditions as possible, but small differences between the selected temperature and the measured equilibrium temperature were usually observed. For the thermodynamic treatment of the data one needs the pure component vapor pressures at the

Table VIII. Vapor–Liquid Equilibrium Data for Water (1) + 2-Isopropoxyethanol (2) at 358.15 and 368.15 K: Liquid Mole Fraction x_i, Temperature T, Vapor Mole Fraction y_i, and Pressure P

x_1	T (K)	y_1	P (kPa)	x_1	T (K)	y_1	P (kPa)	x_1	T (K)	y_1	P (kPa)
0.0000	358.15	0.0000	13.426	0.2668	358.15	0.7268	37.929	0.7198	358.15	0.8926	58.976
0.0672	358.15	0.4053	20.970	0.3040	358.15	0.7557	41.026	0.8052	358.15	0.9136	60.018
0.1029	358.15	0.4677	23.153	0.4134	358.15	0.8050	47.337	0.8706	358.15	0.9133	60.416
0.1281	358.15	0.5416	26.067	0.4819	358.15	0.8339	51.417	0.9344	358.15	0.9251	60.544
0.1699	358.15	0.6112	29.295	0.5610	358.15	0.8563	54.529	0.9646	358.15	0.9337	60.355
0.2234	358.15	0.7088	35.633	0.6375	358.15	0.8749	56.979	1.0000	358.15	1.0000	57.903
0.0000	368.15	0.0000	20.261	0.2846	368.15	0.7177	56.081	0.7193	368.15	0.8889	86.718
0.0306	368.15	0.2467	25.539	0.3136	368.15	0.7400	59.496	0.8073	368.15	0.9022	88.361
0.0722	368.15	0.3886	31.189	0.4066	368.15	0.7981	69.553	0.8710	368.15	0.9107	88.885
0.1006	368.15	0.4614	34.518	0.4783	368.15	0.8270	75.378	0.9352	368.15	0.9213	89.011
0.1335	368.15	0.5297	38.520	0.5588	368.15	0.8503	80.237	0.9650	368.15	0.9320	88.757
0.2439	368.15	0.6857	51.902	0.6360	368.15	0.8718	84.029	1.0000	368.15	1.0000	84.617

Table IX. Vapor–Liquid Equilibrium Data for Water (1) + 2-Butoxyethanol (2) at 358.15 and 368.15 K: Liquid Mole Fraction x_i, Temperature T, Vapor Mole Fraction y_i, and Pressure P

x_1	T (K)	y_1	P (kPa)	x_1	T (K)	y_1	P (kPa)	x_1	T (K)	y_1	P (kPa)
0.0000	358.17	0.0000	4.241	0.0935	358.11	0.7279	14.663	0.6441	358.16	0.9564	57.235
0.0188	358.15	0.2160	5.385	0.1208	358.14	0.7729	17.465	0.7609	358.17	0.9590	59.088
0.0257	358.15	0.3416	6.415	0.1595	358.14	0.8293	21.965	0.8456	358.17	0.9613	59.386
0.0338	358.16	0.4366	7.533	0.2404	358.17	0.8855	30.354	0.8671	358.16	0.9603	59.364
0.0442	358.15	0.5233	8.811	0.3205	358.17	0.9126	38.366	0.9308	358.13	0.9604	59.343
0.0537	358.18	0.5924	10.188	0.3973	358.17	0.9339	45.460	0.9812	358.17	0.9609	59.496
0.0735	358.14	0.6730	12.356	0.5150	358.16	0.9472	52.577	1.0000	358.15	1.0000	57.903
0.0000	368.15	0.0000	6.883	0.1072	368.15	0.7352	23.847	0.7486	368.17	0.9567	86.618
0.0196	368.17	0.2165	8.713	0.1468	368.13	0.7906	29.323	0.8244	368.15	0.9575	87.038
0.0272	368.14	0.3334	10.196	0.2034	368.15	0.8431	37.944	0.8666	368.16	0.9574	87.255
0.0344	368.15	0.4329	12.010	0.2707	368.14	0.8806	47.699	0.9031	368.12	0.9592	87.090
0.0486	368.19	0.5132	13.888	0.3626	368.12	0.9194	61.619	0.9565	368.13	0.9589	87.139
0.0597	368.15	0.5993	16.467	0.4867	368.14	0.9419	75.075	0.9837	368.15	0.9590	87.201
0.0790	368.15	0.6787	19.924	0.6249	368.15	0.9503	83.321	1.0000	368.15	1.0000	84.617

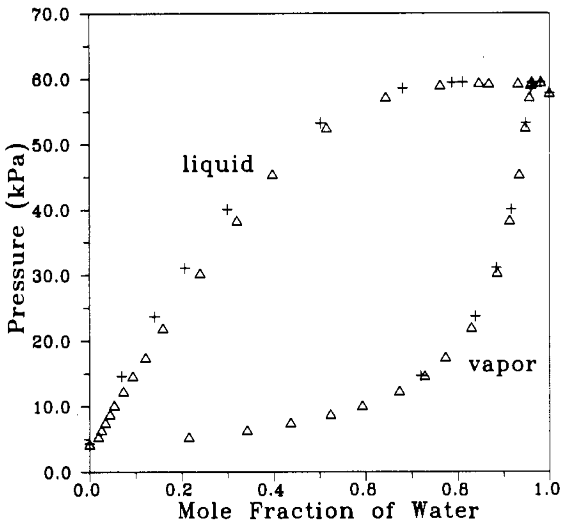

Figure 1. P–x,y diagram for water + 2-butoxyethanol at 358.15 K: (Δ) this work, (+) literature (*3*).

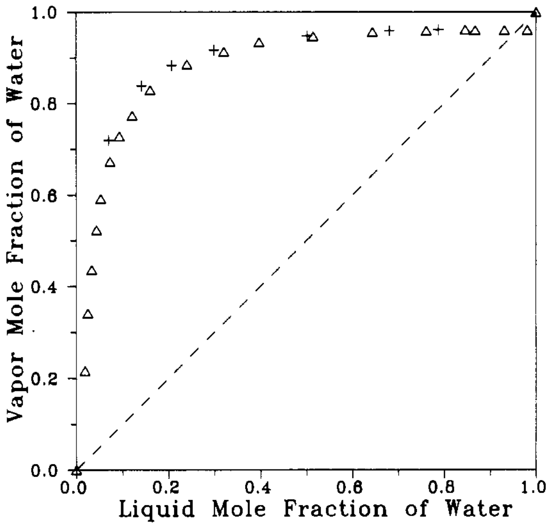

Figure 2. x–y diagram for water + 2-butoxyethanol at 358.15 K: (Δ) this work, (+) literature (*3*).

equilibrium temperatures. Parameters for the Antoine vapor pressure equation

$$\log (P^{\mathrm{s}}_i/\mathrm{kPa}) = A_i - B_i/[(T/\mathrm{K}) + C_i] \qquad (1)$$

have therefore been estimated, and they are presented in Table II along with the average absolute deviations (AAD) of the vapor pressures, which are defined in eq 2.

$$\mathrm{AAD} = \sum |(\mathrm{exptl} - \mathrm{calcd})_i| / \mathrm{no.\ of\ points} \qquad (2)$$

The Antoine parameters for water were obtained from the experimental vapor pressures measured in this work. For 2-methoxyethanol and 2-ethoxyethanol we have used literature values of C_i (*26*) while A_i and B_i were estimated from the measured values at the two selected temperatures of 343.15 and 363.15 K. For 1-methoxy-2-propanol, 2-isopropoxyethanol, and 2-butoxyethanol the vapor pressures were measured over a broader temperature range as shown in Table III, and used for the estimation of the Antoine parameters.

Vapor–Liquid Equilibrium Measurements. VLE measurements have been done for six binary systems, each at two different temperatures. The data are presented in Tables IV–IX.

The water + 1,2-ethanediol system shows an almost ideal solution behavior. It was not possible to cover the region where the concentration of ethylene glycol is high because of severe operational problems with the Dvořak–Boublík cell at pressures below approximately 4 kPa.

Table X. UNIQUAC Binary Parameters and Mean Deviations

T (K)	$A_{12}{}^a$ (K)	$A_{21}{}^a$ (K)	AAD(x)	AAD(T) (K)	AAD(y)	AAD(P) (kPa)
Water (1) (r = 0.92, q = 1.40) + 1,2-Ethanediol (2) (r = 2.4087, q = 2.2480)						
343.15	52.76	−163.6	0.0003	0.01	0.0022	0.020
363.15	54.67	−163.6	0.0015	0.04	0.0068	0.020
Water (1) + 2-Methoxyethanol (2) (r = 3.4938, q = 3.3680)						
343.15	−221.4	439.9	0.0025	0.10	0.0040	0.052
363.15	−206.9	417.0	0.0052	0.12	0.0059	0.024
Water (1) + 2-Ethoxyethanol (2) (r = 4.1682, q = 3.9080)						
343.15	−104.3	290.9	0.0050	0.16	0.0067	0.104
363.15	−42.72	207.3	0.0043	0.12	0.0049	0.043
Water (1) + 1-Methoxy-2-propanol (2) (r = 4.1674, q = 3.9040)						
353.15	−126.1	369.6	0.0024	0.17	0.0050	0.065
363.15	−117.6	361.4	0.0020	0.09	0.0029	0.016
Water (1) + 2-Isopropoxyethanol (2) (r = 4.8418, q = 4.4440)						
358.15	−38.87	256.3	0.0046	0.07	0.0059	0.017
368.15	−10.19	218.9	0.0049	0.10	0.0079	0.025
Water (1) + 2-Butoxyethanol (2) (r = 5.5170, q = 4.9880)						
358.15	43.04	196.8	0.0049	0.12	0.0058	0.047
368.15	65.90	173.0	0.0049	0.14	0.0055	0.031

[a] $A_{ij} = (u_{ij} - u_{jj})/R$ (*31*).

The water + glycol ether systems are strongly nonideal, showing azeotropic behavior in the water-rich region. For the system water + 2-butoxyethanol the P–x,y and y–x diagrams in Figures 1 and 2 indicate that there is a phase split into two liquid phases. This is in accordance with the literature (*27*), which shows that there is a lower critical solution temperature around 323 K. The liquid mole fractions reported for this system are the overall mole fractions.

The maximum likelihood method (*28*) has been used to reduce the VLE data. The vapor phase was described by means of the virial equation of state truncated after the second term. The second virial coefficients were calculated with the Hayden–O'Connell (*29*) correlation as explained in ref 30. In order to check for consistency of the data, the UNIQUAC model (*31*) was used to describe the nonideality of the liquid phase. The binary parameters of the UNIQUAC equation for all the investigated systems, together with the overall average absolute deviations between experimental and calculated values of the temperature, pressure, liquid, and vapor mole fractions, are listed in Table X. On the basis of these calculations, the data are considered to be reliable, and they have been included in the data base used for estimation of the UNIFAC group interaction parameters (*32*).

Conclusions

It has been shown that the modified Dvořak–Boublík VLE cell is applicable for water + glycol ether systems in the temperature range 343.15–368.15 K, even though there is a marked difference in the vapor pressures of the pure components.

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Glossary

AAD	average absolute deviation (eq 2)
A,B,C	Antoine vapor pressure constants
A_{12},A_{21}	UNIQUAC binary interaction parameters, K
P	pressure, kPa
q	UNIQUAC surface area parameter
r	UNIQUAC volume parameter
R	universal gas constant
T	temperature, K
u_{ij}	UNIQUAC interaction energy parameter between molecules i and j
x	liquid-phase mole fraction
y	vapor-phase mole fraction

Greek Letter

ρ	density, g cm^{-3}

Subscript

i	component i

Superscript

s	saturation

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