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Isobaric Vapor−Liquid Equilibrium in the Systems 3-Methylpentane + Ethyl 1,1-Dimethylethyl Ether, + Diisopropyl Ether, and + Tetrahydrofuran

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New consistent vapor−liquid equilibrium data for the binary systems 3-methylpentane + ethyl 1,1-dimethylethyl ether, + diisopropyl ether, and + tetrahydrofuran are reported at 101.3 kPa. The measured systems, which deviate from ideal behavior, can be described as symmetric solutions, and only the system 3-methylpentane + tetrahydrofuran presents an azeotrope. The activity coefficients and boiling points of the solutions were correlated with composition by Wilson, UNIQUAC, NRTL, and Wisniak−Tamir equations.

Introduction

Environmental legislation around the world has forced the use of oxygenates for gasoline blending to phase out the lead additives and to reduce the reactive evaporative and exhaust emissions. Amendments of the U.S. Clean Air Act in 1990 have mandated that new gasoline formulations be sold in highly polluted areas of the country, with oxygenated gasolines being supplied particularly during the winter. Methyl 1,1-dimethylethyl ether (MTBE) is the primary oxygenated compound being used to reformulate gasolines to improve their octane rating and pollution-reducing capability. However potential and documented contamination of water resources by MTBE has become a major public issue over the past few years, and restrictions in its use as an additive are expected. MTBE readily dissolves in water, can move rapidly through soils and aquifers, is resistant to microbial decomposition, and is difficult to remove in water treatment. The U.S. Environmental Protection Agency has classified it as a possible human carcinogen. Finally, it is reported to have an unpleasant taste and odor in water. These factors have caused widespread concern that drinking water supplies and human health may be at risk, a situation which promotes further research with other branched or cyclic ethers as potential additives. Ethyl 1,1-dimethylethyl ether (ETBE), diisopropyl ether (DIPE), and tetrahydrofuran (THF) show good characteristics as a gasoline additive, including low volatility, low water solubility, high octane value, and large reductions in carbon monoxide and hydrocarbon emissions.

Phase equilibrium data of oxygenated mixtures are important for predicting the vapor-phase composition that would be in equilibrium with hydrocarbon mixtures, and the systems reported here constitute examples of such mixtures. The present work was undertaken to measure vapor−liquid equilibrium (VLE) data of the entitled systems at 101.3 kPa, for which no data (isobaric or isothermal) have been published in the literature.

Experimental Section

Chemicals. 3-Methylpentane (99+ mass %), DIPE (99.8+ mass %, HPLC grade) and THF (99.9 mass %) were purchased from Aldrich Ltd., and ETBE (96+ mass %) was purchased from Tokyo Chemical Industry Co. Ltd. 3-Methylpentane, DIPE, and THF were used without further purification after chromatography failed to show any significant impurities. ETBE was purified to 99.9+ mass %, in a 1 m height × 30 mm diameter Normschliffgerätebau adiabatic column (packed with 3 × 3 mm SS spirals) working at a 1:100 reflux ratio. The properties and purity (as determined by GLC) of the pure components, as used for VLE measurements, appear in Table 1. Appropriate precautions were taken when handling ethers in order to avoid peroxide formation.

Apparatus and Procedure. The equilibrium vessel was an all-glass, dynamic-recirculating still equipped with a Cottrell circulation pump. General details of the experimental equipment and procedure appear in another publication (Wisniak et al., 1997). The still (Labodest model), manufactured by Fischer Labor und Verfahrenstechnik, is capable of handling pressures from (0.25 to 400) kPa, and temperatures up to 523 K. The Cottrell pump ensures that both liquid and vapor phases are in intimate contact during boiling and also in contact with the temperature sensing element. The equilibrium temperature was measured with a digital Fischer thermometer with an accuracy of ±0.1 K. The apparatus is equipped with two digital sensors of pressure: one for the low-pressure zone with an accuracy of ±0.01 kPa and another one for the high pressures with an accuracy of ±0.1 kPa. The temperature probe was calibrated against the ice and steam points of distilled water. The manometers were calibrated using the vapor pressure of ultrapure water. The still was operated under constant pressure until equilibrium was reached. Equilibrium conditions were assumed when constant temperature and pressure were obtained for 60 min or longer. Then, samples of liquid and condensate were taken for analysis. The sample extractions were carried out with special syringes that allowed one to withdraw small volume samples (1.0 μL) in a system under partial vacuum or under overpressure conditions.

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Table 1. Densities d, Refractive Index n_D, and Normal Boiling Points T_b of Pure Components

	d(298.15K)/(kg m^{-3})		n_D(298.15K)		T_b(101.3 kPa)/K	
component	expt	lit.	expt	lit.	expt	lit.
3-methylpentane	659.51	660.04[a]	1.3738	1.3739[b]	336.3	336.43[c]
ETBE	735.62	735.09[b]	1.3730	1.3729[b]	345.8	345.86[d]
DIPE	718.14	718.36[e]	1.3656	1.3655[f]	341.4	341.42[g]
THF	881.91	882.30[h]	1.4046	1.4050[f]	339.1	339.08[i]

[a] Awwad and Pethrick (1983). [b] DIPPR (Daubert and Danner, 1989). [c] Willingham et al. (1945). [d] Krähenbühl and Gmehling (1994). [e] Nakanishi and Shirai (1970). [f] Kobe et al. (1956). [g] Reid et al. (1977). [h] Ramkumar and Kudchadker (1989). [i] Wu and Sandler (1988).

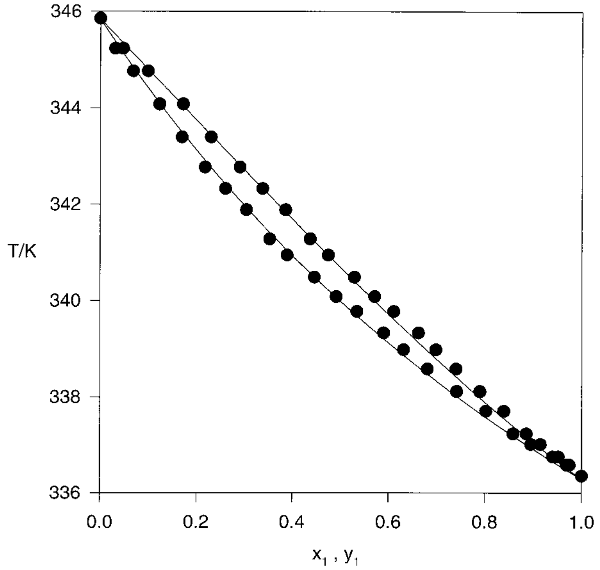

Figure 1. Experimental data for the system 3-methylpentane (1) + ETBE (2) at 101.3 kPa: experimental data (●); smoothed data using the regular model with the parameters given in Table 6, eq 5 (—).

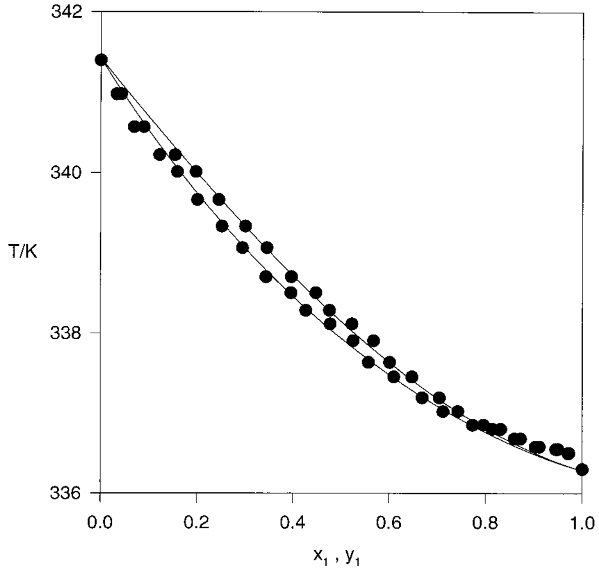

Figure 2. Experimental data for the system 3-methylpentane (1) + DIPE (3) at 101.3 kPa: experimental data (●); smoothed data using the regular model with the parameters given in Table 6, eq 5 (—).

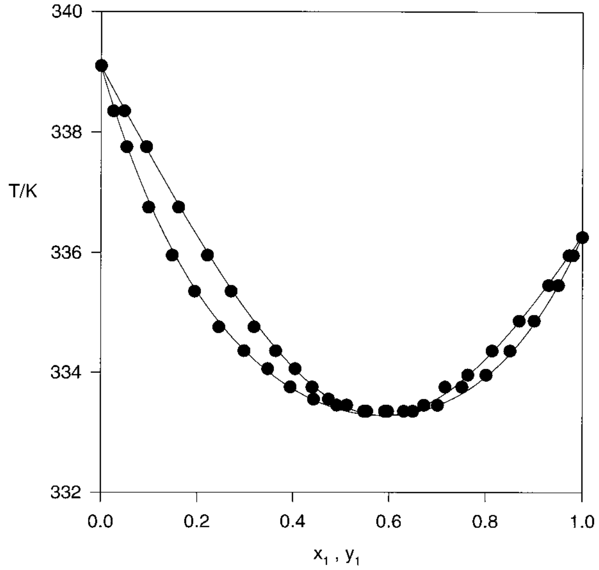

Figure 3. Experimental data for the system 3-methylpentane (1) + THF (4) at 101.3 kPa: experimental data (●); smoothed data using the regular model with the parameters given in Table 6, eq 5 (—).

Analysis. Mole fractions of the liquid-phase and condensed vapor-phase samples were determined using a Hewlett-Packard 5890 S-II gas chromatograph (GC), after calibration with gravimetrically prepared standard solutions. A flame ionization detector was used together with a 60 m, 0.2 mm i.d., fused silica capillary column, SUPELCOWAX 10. The GC response peaks were integrated with a Hewlett-Packard 3396 integrator. Column, injector, and detector temperatures were (333, 373, 413) K for all of the systems. Very good separation was achieved under these conditions, and calibration analyses were carried out to convert the peak ratio to the mass composition of the sample. At least three analyses were made of each vapor composition; the standard deviation in the mole fraction was usually less than 0.001.

Results and Discussion

The temperature T and the liquid-phase x_i and vapor-phase y_i mole fractions at 101.3 kPa are reported in Figures 1−3 and in Tables 2−4. Figures 4−6 show the activity coefficients γ_i that, for the systems 3-methylpentane (1) + DIPE (3) and 3-methylpentane (1) + THF (4), were calculated from the following equation (Van Ness and Abbott, 1982):

$$\ln\gamma_i = \ln\left(\frac{y_iP}{x_iP_i^\circ}\right) + \frac{(B_{ii} - V_i^{\mathrm{L}})(P - P_i^\circ)}{RT} + y_j^2\frac{\delta_{ij}P}{RT} \qquad (1)$$

where T and P are the boiling point and the total pressure, V_i^{L} is the molar liquid volume of component i, B_{ii} and B_{jj} are the second virial coefficients of the pure gases, P_i° is the vapor pressure, B_{ij} the cross second virial coefficient and

$$\delta_{ij} = 2B_{ij} - B_{jj} - B_{ii} \qquad (2)$$

The standard state for calculation of activity coefficients is the pure component at the pressure and temperature of

Table 2. Experimental Vapor−Liquid Equilibrium Data for 3-Methylpentane (1) + ETBE (2) at 101.3 KPa (Vapor Phase Considered Ideal)

T/K	x_1	y_1	γ_1	γ_2
336.3	1.000	1.000	1.000	
336.6	0.969	0.975	0.997	1.103
336.8	0.940	0.952	0.997	1.083
337.0	0.895	0.915	0.999	1.083
337.2	0.859	0.886	1.002	1.065
337.7	0.802	0.839	1.001	1.055
338.1	0.742	0.790	1.006	1.043
338.6	0.681	0.741	1.012	1.027
339.0	0.631	0.699	1.017	1.019
339.3	0.590	0.662	1.021	1.015
339.8	0.534	0.611	1.025	1.015
340.1	0.491	0.571	1.033	1.013
340.5	0.446	0.529	1.040	1.009
340.9	0.389	0.475	1.054	1.006
341.3	0.353	0.437	1.061	1.006
341.9	0.304	0.386	1.065	1.002
342.3	0.260	0.338	1.076	1.001
342.8	0.218	0.291	1.089	1.000
343.4	0.170	0.230	1.090	1.002
344.1	0.123	0.172	1.099	0.998
344.8	0.068	0.099	1.118	1.001
345.2	0.031	0.047	1.151	1.003
345.8	0.000	0.000		1.000

Table 3. Experimental Vapor−Liquid Equilibrium Data for 3-Methylpentane (1) + DIPE (3) at 101.3 KPa

T/K	x_1	y_1	γ_1	γ_3	$-B_{11}$/ (cm^3 mol^{-1})	$-B_{33}$/ (cm^3 mol^{-1})	$-B_{13}$/ (cm^3 mol^{-1})
336.3	1.000	1.000	1.000				
336.5	0.971	0.972	0.995	1.102	1274	1328	1288
336.6	0.946	0.950	0.995	1.076	1273	1327	1286
336.6	0.904	0.911	0.999	1.071	1273	1327	1286
336.7	0.860	0.873	1.002	1.056	1272	1326	1285
336.8	0.814	0.832	1.006	1.048	1271	1325	1284
336.9	0.774	0.796	1.010	1.040	1270	1324	1283
337.0	0.712	0.743	1.021	1.027	1269	1322	1282
337.2	0.669	0.705	1.025	1.020	1267	1320	1280
337.5	0.610	0.648	1.024	1.022	1264	1317	1277
337.6	0.557	0.601	1.038	1.016	1263	1316	1276
337.9	0.526	0.568	1.030	1.018	1260	1313	1273
338.1	0.478	0.523	1.037	1.014	1258	1311	1271
338.3	0.427	0.476	1.051	1.008	1256	1309	1269
338.5	0.396	0.448	1.059	1.002	1254	1307	1267
338.7	0.344	0.397	1.074	1.001	1252	1305	1265
339.1	0.295	0.346	1.079	0.998	1248	1300	1261
339.3	0.252	0.301	1.091	0.999	1246	1298	1259
339.7	0.201	0.246	1.103	0.997	1242	1294	1255
340.0	0.159	0.198	1.114	0.997	1240	1291	1252
340.2	0.122	0.154	1.127	1.000	1238	1289	1250
340.6	0.069	0.089	1.136	1.004	1234	1285	1246
341.0	0.033	0.043	1.129	1.003	1230	1281	1243
341.4	0.000	0.000		1.000			

the solution. Equation 1 is valid at low and moderate pressures when the virial equation of state truncated after the second coefficient is adequate for describing the vapor phase of the pure components and their mixtures, and liquid volumes of the pure components are incompressible over the pressure range under consideration.

For the system 3-methylpentane (1) + ETBE (2), activity coefficients were calculated according to the ideal relation (Van Ness and Abbott, 1982)

$$\gamma_i = y_i P / x_i P_i^\circ \tag{3}$$

because, on one hand, the low pressure makes this assumption reasonable and, on the other hand, as discussed by Reich et al. (1998), the scarce physical information available for mixtures of ETBE with alkanes does not allow a reliable estimation of the second virial coefficient.

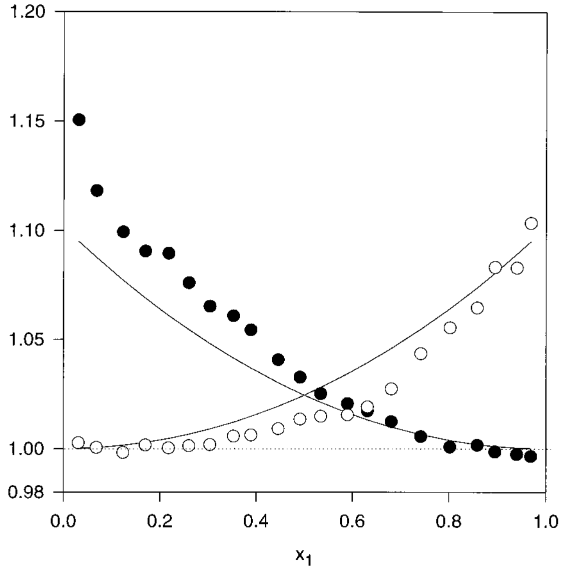

Figure 4. Activity coefficient plot of the system 3-methylpentane (1) + ETBE (2) at 101.3 kPa, calculated from experimental data: γ_1 (●); γ_2 (○); smoothed data using the regular model with the parameters given in Table 6, eq 5 (—).

Table 4. Experimental Vapor−Liquid Equilibrium Data for 3-Methylpentane (1) + THF (4) at 101.3 KPa

T/K	x_1	y_1	γ_1	γ_4	$-B_{11}$/ (cm^3 mol^{-1})	$-B_{44}$/ (cm^3 mol^{-1})	$-B_{14}$/ (cm^3 mol^{-1})
336.3	1.000	1.000	1.000				
336.0	0.981	0.972	1.002	1.598	1280	919	1020
335.5	0.950	0.931	1.004	1.580	1285	922	1024
334.9	0.901	0.870	1.009	1.512	1291	927	1029
334.4	0.851	0.814	1.015	1.459	1296	930	1033
334.0	0.802	0.764	1.023	1.410	1300	933	1036
333.8	0.752	0.717	1.031	1.359	1302	934	1038
333.5	0.701	0.672	1.047	1.317	1305	937	1040
333.4	0.650	0.630	1.063	1.272	1306	937	1041
333.4	0.597	0.591	1.084	1.223	1306	937	1041
333.4	0.548	0.554	1.108	1.189	1306	937	1041
333.5	0.491	0.512	1.139	1.152	1305	937	1040
333.6	0.443	0.474	1.165	1.130	1304	936	1039
333.8	0.395	0.440	1.207	1.100	1302	934	1038
334.0	0.348	0.405	1.252	1.078	1300	933	1036
334.4	0.298	0.364	1.299	1.056	1296	930	1033
334.8	0.246	0.319	1.363	1.039	1292	927	1030
335.4	0.195	0.271	1.436	1.021	1286	923	1025
336.0	0.148	0.222	1.517	1.011	1280	919	1020
336.8	0.099	0.161	1.613	1.003	1271	914	1014
337.8	0.053	0.094	1.702	0.998	1261	907	1007
338.4	0.026	0.048	1.782	1.000	1256	903	1002
339.1	0.000	0.000		1.000			

The vapor pressures of the pure compounds P_i° were expressed by the Antoine equation:

$$\log(P_i^\circ/\mathrm{kPa}) = A_i - \frac{B_i}{(T/K) - C_i} \tag{4}$$

The constants A_i, B_i, and C_i were taken from literature: Loras et al. (1998) for 3-methylpentane, Reich et al. (1998) for ETBE, Reid et al. (1977) for DIPE, and Wu and Sandler (1988) for THF, the values are reported in Table 5.

When required, the molar virial coefficients B_{ii} and B_{ij} were estimated by the method of Hayden and O'Connell (1975) assuming the association parameter η to be zero. Critical properties of all components were taken from DIPPR (Daubert and Danner, 1989). The last two terms in eq 1, particularly the second one that expresses the correction due to the nonideal behavior of the vapor phase,

Table 5. Antoine Coefficients, Equation 4

compound	A_i	B_i	C_i
3-methylpentane[a]	6.241 16	1302.78	28.69
ethyl 1,1-dimethylethyl ether[b]	5.966 51	1151.73	55.06
diisopropyl ether[c]	6.222 01	1257.61	43.15
tetrahydrofuran[d]	6.441 02	1384.21	27.00

[a] Loras et al. (1998). [b] Reich et al. (1998). [c] Reid et al. (1977). [d] Wu and Sandler (1988).

Table 6. Consistency Test for the Various Experimental Systems Considered in VLE Measurements

system	A[a]	100 AADy_1[b]	AADP[c]/kPa	γ_1^{∞} [d]
1 + 2	0.097	0.35	0.29	1.102
1 + 3	0.108	0.49	0.27	1.114
1 + 4	0.548	0.34	0.15	1.730

[a] Zero-order Legendre polynomial (or Porter model) parameter in eq 5. [b] Average absolute deviation in vapor-phase composition. [c] Average absolute deviation in pressure. [d] Limiting activity coefficient of 3-methylpentane, predicted by eq 5.

contributed less than 1% for the systems 3-methylpentane (1) + DIPE (3) and 3-methylpentane (1) + THF (4), at 101.3 kPa; in general, their influence was important only at very dilute concentrations. The calculated activity coefficients reported in Tables 2−4 are estimated to be accurate to within ±2%. The results reported in these tables indicate that the measured systems exhibit positive deviations from ideal behavior. An azeotrope composition is present in the system 3-methylpentane (1) + THF (4) at $x_1 \approx 0.59$ and $T = 333.3$ K.

The vapor−liquid equilibria data reported in Tables 2−4 were found to be thermodynamically consistent by the point-to-point method of Van Ness et al. (1973), as modified by Fredenslund et al. (1977). Consistency was achieved using a one parameter (zero-order) Legendre polynomial, the Porter or two-suffix Margules model, which reduces the functionality of the excess Gibbs energy G^E to the following symmetric relation

$$G^E = Ax_1x_2 \tag{5}$$

The parameter A in eq 5, together with the pertinent statistics required by the Fredenslund et al. test, are shown in Table 6 for the different systems considered in this work. From Table 6 it is concluded that eq 5 gives an excellent fit to the data.

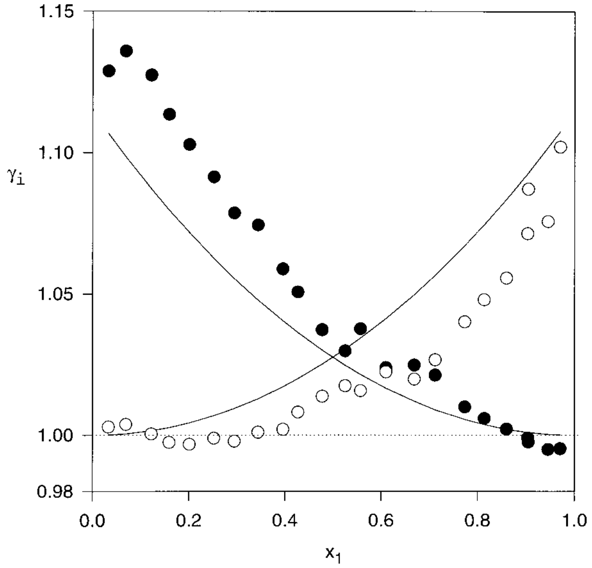

Figure 5. Activity coefficient plot of the system 3-methylpentane (1) + DIPE (3) at 101.3 kPa, calculated from experimental data: γ_1 (●); γ_3 (○); smoothed data using the regular model with the parameters given in Table 6, eq 5 (—).

The variation of activity coefficients with composition appears in Figures 4−6. These figures show a symmetric trend of experimental activity coefficients that, in high dilution ranges, are approximately equivalent for both components in each binary system. In addition, from the figures, it is clear that activity coefficients exhibit intersection about the composition $x_1 = 0.5$ and are reasonably represented by eq 5. Thus, according to the present experimental data, and the pertinent consistency test, it is concluded that the systems can be satisfactorily explained by symmetrical solution behavior.

The activity coefficients were correlated with the Wilson, NRTL, and UNIQUAC equations (Walas, 1985). The parameters of these equations were obtained by minimizing the following objective function (OF):

$$\mathrm{OF} = \sum_{i=1}^{N} 100\left(\left|\frac{P_i^{\mathrm{expt}} - P_i^{\mathrm{calc}}}{P_i^{\mathrm{expt}}}\right| + \left|y_i^{\mathrm{expt}} - y_i^{\mathrm{calc}}\right|\right) \tag{6}$$

and are reported in Table 7, together with the pertinent statistics of VLE interpolation. Inspection of the results

Table 7. Parameters and Deviations between Experimental and Calculated Values for Different G^E Models

model	A_{ij} (J/mol)	A_{ji} (J/mol)	α_{ij}	bubble-point pressures		dew-point pressures	
				ΔP [a]	100 Δy [b]	ΔP [a]	100 Δx [b]
			3-Methylpentane (1) + ETBE (2)				
[c]Wilson	−337.50	701.81		0.38	0.30	0.39	0.30
NRTL	1933.52	−1340.45	0.3	0.48	0.25	0.47	0.24
[d]UNIQUAC	−1299.15	410.93		0.37	0.14	0.36	0.13
			3-Methylpentane (1) + DIPE (3)				
[c]Wilson	−126.76	472.68		0.31	0.47	0.31	0.47
NRTL	−138.44	460.00	0.3	0.26	0.50	0.26	0.51
[e]UNIQUAC	−214.89	305.57		0.26	0.49	0.27	0.50
			3-Methylpentane (1) + THF (4)				
[c]Wilson	−189.70	1882.85		0.17	0.09	0.19	0.09
NRTL	−227.56	1870.29	0.2	0.13	0.10	0.16	0.10
[e]UNIQUAC	796.32	−230.81		0.15	0.10	0.17	0.09

[a] Average percentual deviation in bubble pressure: $\Delta P = 100/N \sum_i^N |P_i^{\mathrm{expt}} - P_i^{\mathrm{calc}}|/P_i^{\mathrm{expt}}$ (N = number of data points). [b] Average absolute deviation in vapor-phase composition. [c] Liquid volumes have been estimated from the Rackett equation (Rackett, 1970). [d] Volume and surface parameters calculated from UNIFAC (Gmehling et al., 1993) [e] Volume and surface parameters from DECHEMA (Gmehling and Oken, 1990).

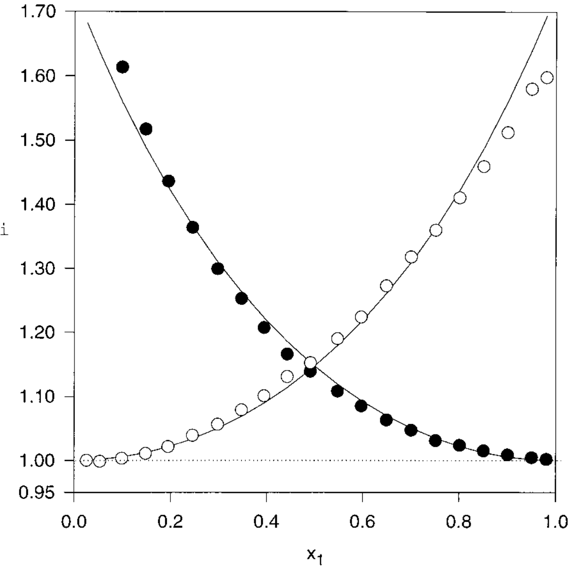

Figure 6. Activity coefficient plot of the system 3-methylpentane (1) + THF (4) at 101.3 kPa, calculated from experimental data: γ_1 (●); γ_4 (○); smoothed data using the regular model with the parameters given in Table 6, eq 5 (—).

Table 8. Coefficients in Correlation of Boiling Points, Equation 7, Average Deviation, Root Mean Square Deviation, and Maximum Deviation in Temperature

system	C_0	C_1	C_2	av dev[a]/K	rmsd[b]/K	max dev[c]/K
1 + 2	−4.11	3.58	−0.29	0.09	0.023	0.20
1 + 3	−3.74	3.01	1.05	0.13	0.029	0.27
1 + 4	−16.98	2.79	−6.29	0.04	0.012	0.11

[a] Average deviation. [b] Root-mean-square deviation. [c] Maximum deviation.

given in Table 7 shows that all the models fitted well the systems, the best fit corresponding to the UNIQUAC model for the 3-methylpentane (1) + ETBE (2) system, and in the case of the 3-methylpentane (1) + DIPE (3) and 3-methylpentane (1) + THF (4) systems all of models give a similar fit to the data.

The boiling point temperatures of each system at 101.3 kPa were well-correlated with mole fractions by the equation proposed by Wisniak and Tamir (1976):

$$T/K = x_1(T_1°/\mathrm{K}) + x_2(T_2°/\mathrm{K}) + x_1x_2\sum_{k=0}^{m} C_k(x_1 - x_2)^k \quad (7)$$

In this equation $T_i°/\mathrm{K}$ is the boiling point of the pure component i, and m is the number of terms used in the series expansion of $(x_1 - x_2)$, usually two. The various constants of eq 7 are reported in Table 8, with information indicating the goodness of fit of the correlation.

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