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Isobaric Vapor−Liquid Equilibrium in the Systems 2,3-Dimethylpentane + Methyl 1,1-Dimethylethyl Ether, + Diisopropyl Ether and + Methyl 1,1-Dimethylpropyl Ether

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New, consistent vapor−liquid equilibrium data for the binary systems 2,3-dimethylpentane + methyl 1,1-dimethylethyl ether, + diisopropyl ether, and + methyl 1,1-dimethylpropyl ether are reported at 101.3 kPa. The measured systems deviate slightly from ideal behavior, and only the system methyl 1,1-dimethylpropyl ether + 2,3-dimethylpentane presents an azeotrope. The activity coefficients for the solutions were correlated with their compositions by the Wilson, UNIQUAC, and NRTL models. Wisniak−Tamir equations were used to correlate the boiling points of the solutions with their compositions.

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. 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 via water treatment. 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 ethers as potential additives. Diisopropyl ether (DIPE) and methyl 1,1-dimethylpropyl ether (TAME) show good characteristics as gasoline additives, including low volatility, low water solubility, high octane value, and large reductions in carbon monoxide and hydrocarbon emissions.

Phase equilibrium data of hydrocarbon and oxygenated compound mixtures are important to predict the vapor-phase composition that would be in equilibrium with reformulated gasolines, and the systems reported here constitute examples of such mixtures. The present work was undertaken to measure vapor−liquid equilibrium (VLE) data of the 2,3-dimethylpentane + methyl 1,1-dimethylethyl ether, + diisopropyl ether, and + methyl 1,1-dimethylpropyl ether binary systems at 101.3 kPa, for which no data have been previously reported.

Experimental Section

Chemicals. 2,3-Dimethylpentane (99+ mass %), MTBE (99.8+ mass %, HPLC grade), DIPE (99 mass %), and TAME (97 mass %) were purchased from Aldrich Ltd. 2,3-Dimethylpentane, MTBE, and DIPE were used without further purification after chromatography failed to show any significant impurities. TAME was purified to 99.9+ mass % by batch distillation in a Fisher SPALTROHR column HMS-500, controlled by a Fisher System D301-C. The densities and refractive indexes of the pure liquids were measured at 298.15 K using an Anton Paar DMA 55 densimeter and an Abbe refractometer Atago 3T, respectively. Temperature was controlled to ±0.01 K with a thermostated bath. The accuracies in density and refractive index measurements are $\pm 0.01\ \mathrm{kg \cdot m^{-3}}$ and ±0.0002, respectively. The experimental values of these properties and the boiling points are given in Table 1 together with those given in the literature. 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 Walas (1985). The still (Labodest model), manufactured by Fischer Labor und Verfahrenstechnik (Germany), 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. For the pressure measurement a digital manometer with an accuracy of ±0.01 kPa was used. The temperature probe was calibrated against the ice and steam points of distilled water. The manometer was 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).

Analysis. The compositions of the liquid- and condensed-phase samples were determined using a CE Instruments

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

	d(298.15 K)/ kg·m^{-3})		n_D (298.15 K)		T_b (101.3 kPa)/K	
component	exptl	lit.	exptl	lit.	exptl	lit.
MTBE	735.29	735.28[a]	1.3664	1.3663[b]	328.2	328.11[c]
DIPE	718.13	718.36[d]	1.3658	1.3655[e]	341.6	341.42[f]
TAME	765.98	765.77[g]	1.3862	1.3859[a]	359.2	359.33[c]
2,3-dimethylpentane	690.80	690.89[b]	1.3894	1.3895[a]	363.0	362.93[a]

[a] DIPPR (Daubert and Danner, 1989). [b] TRC tables, a-6040. [c] Martinez-Ageitos (1996). [d] Nakanishi and Shirai (1970). [e] Kobe et al. (1956). [f] Reid et al. (1977). [g] Linek (1987).

Table 2. Experimental Vapor Pressures Determined for DIPE and 2,3-Dimethylpentane

2,3-dimethylpentane		DIPE	
T/K	P /kPa	T/K	P /kPa
309.3	15.40	306.5	28.49
313.2	18.05	310.1	32.96
316.3	20.45	312.7	36.45
320.1	23.83	315.6	40.78
322.8	26.36	318.2	45.01
326.2	30.08	321.1	50.16
329.4	33.78	323.1	53.93
332.5	37.75	325.2	58.19
335.3	41.66	327.4	62.92
337.8	45.45	329.0	66.50
339.4	47.96	330.9	71.04
342.4	53.13	332.6	75.29
344.5	56.98	334.9	81.32
347.8	63.45	336.6	85.99
350.1	68.33	338.3	90.90
350.4	68.73	340.0	96.06
352.8	74.23	341.6	101.08
354.7	78.73	342.7	104.61
356.5	83.29	344.1	109.44
358.1	87.51	345.4	114.02
359.9	92.36	345.3	113.61
361.8	97.55	346.5	117.97
363.3	101.63	347.6	121.85
363.1	101.48	349.3	128.35
364.2	104.62		
365.4	108.39		
366.7	112.50		
368.1	117.09		
369.4	121.48		
371.3	127.95		

Table 3. Antoine Coefficients for Eq 1

compound	A_i	B_i	C_i
methyl 1,1-dimethylethyl ether (MTBE)[a]	6.34991	1312.52	26.03
diisopropyl ether (DIPE)[b]	6.05151	1185.74	48.54
methyl 1,1-dimethylproyl ether (TAME)[a]	6.23218	1351.21	39.52
2,3-dimethylpentane[b]	6.10469	1323.67	40.11

[a] Aucejo et al. (1998). [b] Antoine's parameters were calculated from the experimental data in Table 2.

GC 8000 Top gas chromatograph, after calibration with gravimetrically prepared standard solutions. A flame ionization detector was used together with a 30 m, 0.454 mm i.d., capillary column, DB-MTBE (J & W Scientific). The GC response peaks were treated with Chrom-Card for Windows, version 1.2. The column, injector, and detector temperatures were 393, 498, and 523 K for all systems. Very good separation of peaks was achieved under these conditions, and calibration analyses were carried out to convert the peak area ratios to the mass composition of the sample. At least three analyses were performed of each liquid and vapor composition. The standard deviation in the mole fraction was usually <0.001.

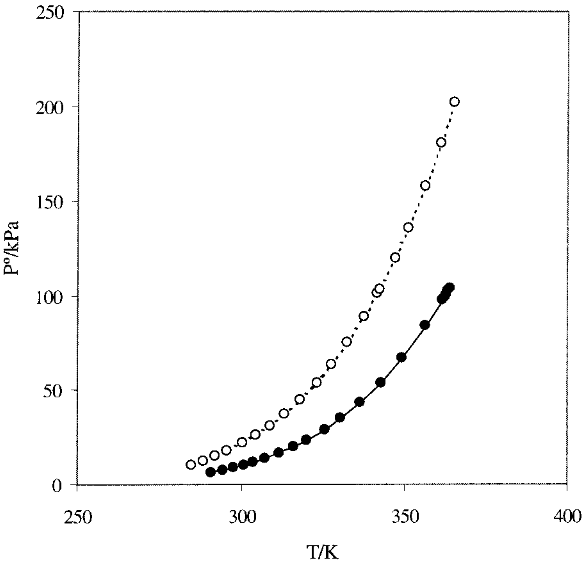

Figure 1. Comparison of correlated vapor pressures with other references: (○) experimental data reported by Ambrose et al. (1976) for DIPE; (●), experimental data of Forziati et al. (1949) for 2,3-dimethylpentane; data for DIPE (- - -) and for 2,3-dimethylpentane (—) predicted by eq 1 and the parameters in Table 3.

Results and Discussion

The pure component vapor pressures P_i° for MTBE and TAME were taken from Aucejo et al. (1998). For DIPE and 2,3-dimethylpentane, the pure component vapor pressures were determined experimentally as a function of the temperature, using the same equipment as that for obtaining the VLE data. The pertinent results appear in Table 2. The measured vapor pressures were correlated using the Antoine equation:

$$\log(P_i^\circ/\mathrm{kPa}) = A_i - \frac{B_i}{(T/K) - C_i} \qquad (1)$$

whose parameters A_i, B_i, and C_i are reported in Table 3. The vapor pressures were correlated with a mean absolute deviation (MADP) of 0.05% for DIPE and 0.10% for 2,3-dimethylpentane. In Figure 1, it can be observed that the parameters presented in Table 3 give a fair prediction of the experimental vapor pressures reported by Ambrose et al. (1976) for DIPE (MADP = 0.31%) and by Forziati et al. (1949) for 2,3-dimethylpentane (MADP = 1.49%).

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

$$\ln \gamma_i = \ln \frac{y_i P}{x_i P_i^\circ} + \frac{(B_{ii} - V_i^{L})(P - P_i^\circ)}{RT} + y_j^{2} \frac{\delta_{ij} P}{RT} \qquad (2)$$

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} is the cross second virial coefficient, and

$$\delta_{ij} = 2B_{ij} - B_{jj} - B_{ii} \qquad (3)$$

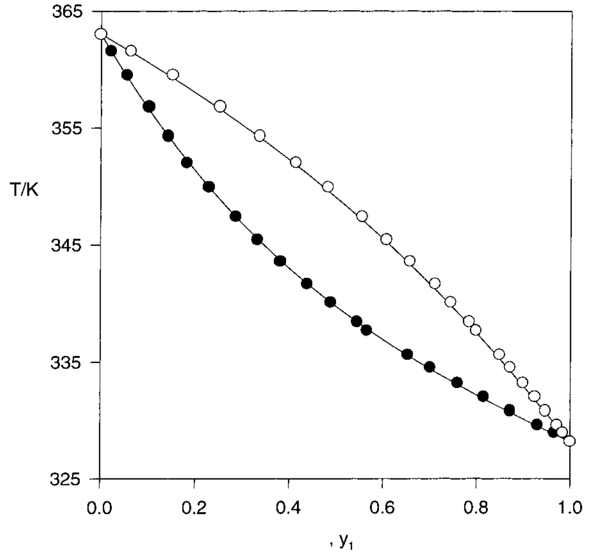

Figure 2. Data for the system MTBE (1) + 2,3-dimethylpentane (4) at 101.3 kPa: experimental data for x_1 (●); experimental data for y_1 (○); smoothed data using the Legendre polynomial used in consistency test (−).

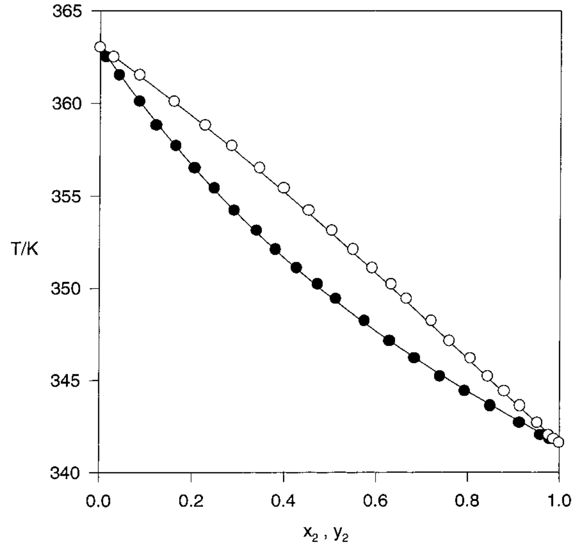

Figure 3. Data for the system DIPE (2) + 2,3-dimethylpentane (4) at 101.3 kPa: experimental data for x_2 (●); experimental data for y_2 (○); smoothed data using the Legendre polynomial used in consistency test (−).

The standard state for calculation of activity coefficients is the pure component at the pressure and temperature of the solution. Equation 2 is valid at low and moderate pressures when the virial equation of state truncated after the second coefficient is adequate to describe the vapor phase of the pure components and their mixtures, and the liquid volumes of the pure components are incompressible over the pressure range under consideration.

For the system TAME (3) + 2,3-dimethylpentane (4), activity coefficients were calculated according to the ideal relation (Van Ness and Abbott, 1982)

$$\gamma_i = \frac{y_i P}{x_i P_i^\circ} \qquad (4)$$

because, on one hand, the low pressure makes this assumption reasonable and, on the other hand, as discussed by Reich et al. (1999), the scarce physical information available for mixtures of TAME with alkanes does not allow a reliable estimation of the second virial coefficient.

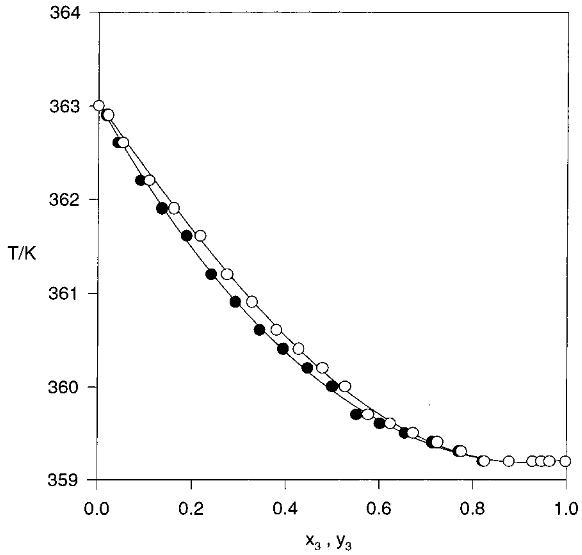

Figure 4. Data for the system TAME (3) + 2,3-dimethylpentane (4) at 101.3 kPa: experimental data for x_3 (●); experimental data for y_3 (○); smoothed data using the Legendre polynomial used in consistency test (−).

Table 4. Experimental Vapor−Liquid Equilibrium Data for MTBE (1) + 2,3-Dimethylpentane (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})
363.0	0.000	0.000		1.000			
361.6	0.022	0.063	1.121	0.998	900	1465	1119
359.5	0.056	0.152	1.122	0.993	913	1488	1135
356.8	0.102	0.252	1.102	0.994	930	1519	1157
354.3	0.142	0.337	1.122	0.993	945	1549	1178
352.0	0.182	0.414	1.142	0.986	960	1577	1198
349.9	0.229	0.482	1.119	0.985	974	1603	1217
347.4	0.286	0.555	1.104	0.987	992	1636	1240
345.4	0.332	0.608	1.101	0.988	1006	1662	1258
343.6	0.381	0.657	1.089	0.990	1019	1687	1276
341.7	0.438	0.711	1.084	0.974	1032	1713	1294
340.1	0.488	0.744	1.064	0.999	1044	1736	1310
338.4	0.544	0.784	1.056	1.002	1057	1761	1328
337.7	0.566	0.799	1.057	1.000	1063	1772	1335
335.6	0.653	0.849	1.037	1.006	1079	1803	1357
334.5	0.701	0.871	1.023	1.042	1088	1820	1369
333.2	0.759	0.898	1.014	1.064	1099	1841	1383
332.0	0.816	0.925	1.008	1.069	1109	1860	1397
330.8	0.872	0.947	1.002	1.130	1119	1880	1410
329.6	0.930	0.971	0.999	1.185	1129	1900	1424
328.9	0.965	0.984	0.997	1.378	1135	1912	1432
328.2	1.000	1.000	1.000				

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 2, particularly the second one that expresses the correction due to the nonideal behavior of the vapor phase, contributed <4.5% for the systems MTBE (1) + 2,3-dimethylpentane (4) and DIPE (2) + 2,3-dimethylpentane (4), at 101.3 kPa; in general, their influence was important only at very dilute concentrations. The calculated activity coefficients reported in Tables 4−6 indicate that the measured systems exhibit positive deviations from ideal behavior. An azeotrope composition is present in the system TAME (3) + 2,3-dimethylpentane (4) at $x_1 \approx 0.93$ and $T = 359.1$ K.

The vapor−liquid equilibria data reported in this work were found to be thermodynamically consistent by the

Table 5. Experimental Vapor−Liquid Equilibrium Data for DIPE (2) + 2,3-Dimethylpentane (4) at 101.3 kPa

T/K	x_2	y_2	γ_2	γ_4	$-B_{22}$/(cm^3 mol^{-1})	$-B_{44}$/(cm^3 mol^{-1})	$-B_{24}$/(cm^3 mol^{-1})
363.0	0.000	0.000		1.000			
362.5	0.013	0.029	1.232	1.000	1149	1455	1279
361.5	0.041	0.085	1.186	0.997	1157	1466	1289
360.1	0.086	0.161	1.123	0.997	1169	1481	1302
358.8	0.122	0.227	1.144	0.994	1180	1496	1315
357.7	0.164	0.285	1.104	0.995	1190	1509	1326
356.5	0.205	0.345	1.105	0.993	1201	1523	1338
355.4	0.248	0.399	1.085	0.995	1210	1536	1349
354.2	0.292	0.453	1.082	0.996	1221	1550	1361
353.1	0.339	0.504	1.071	0.998	1231	1563	1373
352.1	0.381	0.549	1.065	1.000	1241	1576	1383
351.1	0.427	0.591	1.053	1.008	1250	1588	1394
350.2	0.473	0.633	1.044	1.011	1259	1599	1404
349.4	0.512	0.667	1.039	1.016	1266	1610	1412
348.2	0.575	0.720	1.034	1.017	1278	1625	1426
347.1	0.630	0.760	1.029	1.033	1289	1639	1438
346.2	0.684	0.806	1.031	1.008	1298	1651	1448
345.2	0.739	0.844	1.028	1.013	1308	1665	1460
344.4	0.794	0.880	1.021	1.015	1316	1676	1469
343.6	0.849	0.914	1.015	1.021	1324	1687	1478
342.7	0.913	0.951	1.009	1.036	1334	1699	1489
342.0	0.959	0.977	1.009	1.038	1341	1709	1497
341.8	0.978	0.988	1.006	1.039	1343	1712	1500
341.4	1.000	1.000	1.000				

Table 6. Experimental Vapor−Liquid Equilibrium Data for TAME (3) + 2,3-Dimethylpentane (4) at 101.3 kPa (Vapor Phase Considered Ideal)

T/K	x_3	y_3	γ_3	γ_4
363.0	0.000	0.000		1.000
362.9	0.017	0.021	1.106	1.000
362.6	0.043	0.053	1.113	1.002
362.2	0.090	0.109	1.107	1.003
361.9	0.137	0.162	1.090	1.004
361.6	0.188	0.217	1.074	1.006
361.2	0.241	0.275	1.075	1.008
360.9	0.293	0.328	1.064	1.012
360.6	0.345	0.380	1.056	1.017
360.4	0.395	0.428	1.045	1.022
360.2	0.447	0.479	1.040	1.024
360.0	0.500	0.527	1.029	1.034
359.7	0.552	0.577	1.030	1.042
359.6	0.602	0.624	1.024	1.045
359.5	0.656	0.673	1.017	1.055
359.4	0.713	0.725	1.011	1.067
359.3	0.770	0.777	1.006	1.083
359.2	0.823	0.827	1.005	1.095
359.2	0.878	0.879	1.002	1.111
359.2	0.929	0.929	1.000	1.120
359.2	0.948	0.948	1.000	1.120
359.2	0.966	0.965	0.999	1.153
359.3	1.000	1.000	1.000	

Table 7. Consistency Test for the Various Experimental Systems Considered in VLE Measurements

system	n^a	100 × AAD $y_1{}^b$	AAD P^c/kPa
MTBE (1) + 2,3-dimethylpentane (4)	2	0.53	0.33
DIPE (2) + 2,3-dimethylpentane (4)	2	0.49	0.23
TAME (3) + 2,3-dimethylpentane (4)	2	0.08	0.08

[a] Number of parameters used in the consistency test. [b] Average absolute deviation in vapor-phase composition. [c] Average absolute deviation in pressure.

point-to-point method of Van Ness et al. (1973), as modified by Fredenslund et al. (1977). Consistency was achieved using a Legendre polynomial with two parameters for the three systems; the pertinent statistics required by the Fredenslund test are shown in Table 7 for the different systems considered in this work. The variation of activity coefficients with composition is similar for the three studied systems. Figure 5 is shown as an example of this behavior for the system TAME (3) + 2,3-dimethylpentane (4).

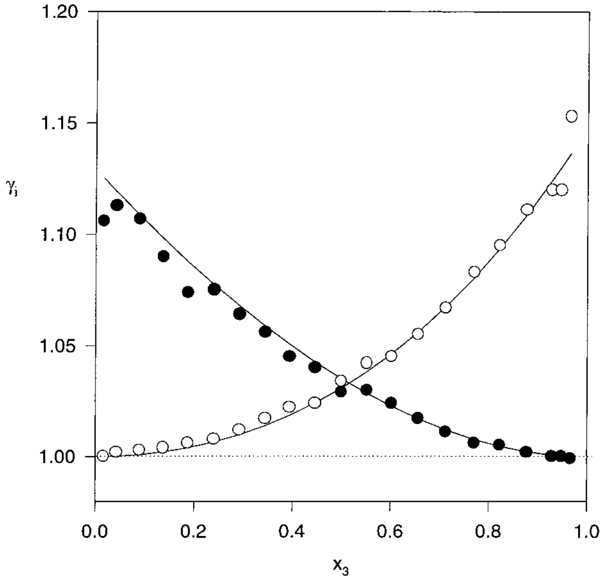

Figure 5. Activity coefficient plot of the system TAME (3) + 2,3-dimethylpentane (4) at 101.3 kPa, calculated from experimental data: γ_3 (●); γ_4 (○). Smoothed data using the Legendre polynomial used in consistency test (−).

Table 8. Parameters and Deviations between Experimental and Calculated Values for Different G^E Models

				bubble point pressures	
model	A_{ij}/(J·mol^{-1})	A_{ji}/(J·mol^{-1})	α_{ij}	ΔP^a	200 × Δy^b
	MTBE (1) + 2,3-dimethylpentane (4)				
Wilson^c	−992.62	2001.60		0.44	0.48
NRTL	1293.69	−717.96	0.3	0.38	0.61
UNIQUAC^d	664.88	−487.67		0.38	0.56
	DIPE (2) + 2,3-dimethylpentane (4)				
Wilson^c	−823.78	1336.27		0.22	0.49
NRTL	161.58	185.73	0.3	0.25	0.56
UNIQUAC^d	92.33	−670.09		0.28	0.33
	TAME (3) + 2,3-dimethylpentane (4)				
Wilson^c	31.77	369.78		0.08	0.07
NRTL	318.15	76.19	0.3	0.08	0.07
UNIQUAC^d	62.28	21.82		0.08	0.07

[a] Average percent deviation in bubble pressure $\Delta P = 100/N\sum_i^- |P_i^{\text{exp}} - P_i^{\text{calc}}|/P_i^{\text{exp}}$ (N is the 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 from DECHEMA (Gmehling and Onken, 1990).

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)

$$\mathrm{OF} = \sum_{i=1}^{N} 100\left(\left|\frac{P_i^{\text{exptl}} - P_i^{\text{calc}}}{P_i^{\text{exptl}}}\right| + \left|y_i^{\text{exptl}} - y_i^{\text{calc}}\right|\right) \tag{5}$$

and are reported in Table 8, together with the pertinent statistics of the VLE correlation. Inspection of the results given in Table 8 shows that all the models fitted well the systems.

The boiling point temperatures of each system at 101.3 kPa were well correlated with the mole fractions by the

Table 9. Coefficients in Correlation of Boiling Points, Eq 6, Average Deviation, Root Mean Square Deviation, and Maximum Deviation in Temperature

system	C_0	C_1	C_2	avg dev[a]/K	rmsd[b]/K	max dev[c]/K
1 + 4	−24.04	8.75	0.42	0.09	0.025	0.33
2 + 4	−10.34	3.60	−4.01	0.07	0.019	0.18
3 + 4	−4.64	0.62	−1.64	0.03	0.008	0.07

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

equation proposed by Wisniak and Tamir (1976):

$$T/K = x_i T_i^{\circ}/\mathrm{K} + x_j T_j^{\circ}/\mathrm{K} + x_i x_j \sum_{k=0}^{m} C_k (x_i - x_j)^k \qquad (6)$$

In this equation T_i°/K is the boiling point of pure component i and m is the number of terms used in the series expansion of $(x_i - x_j)$, usually two. The various constants of eq 6 are reported in Table 9, with information indicating the goodness of fit of the correlation.

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