Vapor−Liquid Equilibrium Data for the Six Binaries Heptafluoropropyl Methyl Ether (HFE-347mcc) or 1,1,1,2,3,3-Hexafluoro-3-propane (HFE-449mec-f) with 1-Propanol, 2-Butanone, or Ethyl Acetate

Katsumi Tochigi,* Chie Kikuchi, Kiyofumi Kurihara, and Kenji Ochi

Department of Materials and Applied Chemistry, Nihon University, 1-8 Kanda Surugadai, Chiyoda-ku, Tokyo 101-8308, Japan

Junji Murata

Research Institute of Innovative Technology for the Earth (RITE), 1-1-1 Higashi, Tsukuba, Ibaraki 305-8565, Japan

Katuto Otake

National Institute of Advanced Industrial Science and Technology (AIST), 1-1-1 Higashi, Tsukuba, Ibaraki 305-8565, Japan

Isobaric vapor−liquid equilibria were measured for the six binary systems heptafluoropropyl methyl ether (HFE-347mcc) or 1,1,1,2,3,3-hexafluoro-3-propane (HFE-449mec-f) with 1-propanol, 2-butanone, or ethyl acetate at atmospheric pressure. The mixtures HFE-347mcc and HFE-449mec-f with 1-propanol both formed a minimum boiling azeotrope. The mixtures HFE-449mec-f with 2-butanone and with ethyl acetate formed maximum boiling azeotropes. The experimental data were correlated by the Wilson and NRTL activity coefficient models. The Wilson and NRTL equations gave similar results.

Introduction

The hydrochlorofluorocarbons (HCFCs) have been widely used in the fields of cleaning solvents. However, because of (COP-3) and their environmental initiatives, the development of the new pure and mixed cleaning solvents in place of HCFCs has been pursued. To further investigate new mixed cleaning solvents, it is essential to have vapor−liquid equilibrium (VLE) data.

In our recent studies, we reported the experimental isobaric VLE data for four binary systems: perfluoro-2-methylpentane ($(CF_3)_2CFCF_2CF_2CF_3$, FC-5114 mmyc2) + perfluorooctane ($CF_3(CF_2)_6CF_3$, FC-7118mc6); 1,1,1,2,2,3,3,4,4-nonafluorohexane ($CF_3(CF_2)_3CH_2CH_3$, HFC-569mccf) + octane; 2,2,2-trifluorodiethyl ether ($CF_3CH_2OCH_2CF_3$, HFE-356mf-f) + butyl ethyl ether; and methyl perfluoroisopropyl ether ($CF_3CF(CF_3)OCH_3$, HFE-347 mmy) + heptane at 101.3 kPa.[1]

In continuation of this experimental work, this paper reports the experimental VLE data for the six binary systems heptafluoropropyl methyl ether ($CF_3CF_2CF_2OCH_3$, HFE-347mcc) or 1,1,1,2,3,3-hexafluoro-3-propane (CF_3-$CHFCF_2OCH_2CF_3$, HFE-449mec-f) with 1-propanol, 2-butanone, or ethyl acetate at 101.3 kPa. VLE data for these systems are not available in the literature.

Experimental Section

Apparatus and Procedure. For our VLE measurements, we used a modified Rogalski−Malanowski equilibrium still that was installed in a constant-temperature air bath that provided a stable environmental temperature. The temperature stability of the air bath was ±0.1 K in the measured temperature range of this work. This still has been described in detail by Hiaki et al.[2] and Kurihara et al.[3] and is a liquid−vapor ebullition-type.

* Corresponding author. E-mail: tochigi@chem.cst.nihon-u.ac.jp.

Table 1. Purities, Densities, ρ, and Normal Boiling Points, T_b, of the Components

		ρ(298.15 K)/(kg·m^{-3})		T_b/K	
component	purity/%	exptl	lit.	exptl	lit.[a]
HFE-347mcc	99.9	1409.2[a]	1409.4[a,b]	307.31	307.33[b]
HFE-449mec-f	99.9	1531.2[a]		345.03	
2-butanone	99.9	800.2	799.7[c]	352.93	352.733[c]
1-propanol	99.9	799.6	799.6[c]	370.4	370.301[c]
ethyl acetate	99.9	894.4	894.55[c]	350.2	350.261[c]

[a] This value was measured at 293.15 K. [b] The value is measured by RITE. [c] Riddick et al.[5]

Table 2. Antoine Constants[a] and Liquid Molar Volumes of the Components

component	*A*	*B*	*C*	$10^6 v_i^L$/m^3·mol^{-1}
HFE-347mcc[b]	5.605 63	807.079	−83.116	141.94
HFE-449mec-f[b]	6.089 92	1126.735	−69.161	158.90
1-propanol[c]	6.874 52	1441.705	−74.291	75.15
2-butanone[c]	6.181 72	1259.223	−51.392	90.17
ethyl acetate[c]	6.188 89	1224.673	−57.438	98.49

[a] $\log(P/\text{kPa}) = A - B/[(T/\text{K}) + C]$. [b] The values were determined by RITE. [c] The values were published by Gmehling and Onken.[6]

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Table 3. Isobaric Vapor−Liquid Equilibrium Data, Liquid Phase, x_1, and Vapor Phase, y_1, Mole Fractions, Temperature, T, and Activity Coefficients, γ_i, for the Six Binary Systems at 101.3 kPa

x_1	y_1	T/K	γ_1	γ_2	x_1	y_1	T/K	γ_1	γ_2	x_1	y_1	T/K	γ_1	γ_2
HFE-347mcc (1) + 1-Propanol (2)														
0.000	0.000	370.40		1.000	0.397	0.954	309.18	2.241	1.425	0.815	0.963	307.91	1.156	3.976
0.082	0.880	324.20	6.022	1.041	0.441	0.956	309.08	2.032	1.466	0.826	0.964	307.75	1.148	4.182
0.113	0.917	317.71	5.624	1.061	0.493	0.958	308.63	1.851	1.586	0.889	0.966	307.57	1.076	6.216
0.134	0.928	315.79	5.129	1.056	0.519	0.958	308.74	1.751	1.650	0.914	0.970	307.52	1.053	7.230
0.159	0.934	314.16	4.604	1.079	0.592	0.956	308.47	1.547	2.085	0.944	0.975	307.33	1.032	9.219
0.178	0.939	313.10	4.277	1.102	0.647	0.961	308.31	1.430	2.188	0.961	0.977	307.28	1.018	12.246
0.187	0.940	312.51	4.158	1.130	0.676	0.960	308.20	1.375	2.425	0.980	0.982	307.25	1.004	18.926
0.239	0.947	311.34	3.421	1.135	0.721	0.960	308.22	1.288	2.806	0.990	0.989	307.27	1.000	23.915
0.262	0.949	310.54	3.227	1.177	0.739	0.963	308.06	1.268	2.850	0.995	0.993	307.29	1.000	26.206
0.312	0.952	309.85	2.778	1.241	0.775	0.963	307.93	1.214	3.332	1.000	1.000	307.31	1.000	
0.350	0.956	309.37	2.536	1.238										
HFE-347mcc (1) + 2-Butanone (2)														
0.000	0.000	352.93		1.000	0.358	0.748	322.80	1.221	1.143	0.721	0.900	313.24	1.007	1.547
0.071	0.379	340.05	1.844	1.027	0.422	0.783	320.62	1.167	1.191	0.799	0.930	311.53	1.000	1.616
0.086	0.425	338.39	1.798	1.024	0.469	0.804	319.31	1.124	1.234	0.823	0.938	311.03	1.000	1.658
0.142	0.538	333.73	1.586	1.034	0.503	0.820	318.73	1.090	1.240	0.851	0.948	310.36	1.000	1.694
0.175	0.592	331.22	1.525	1.043	0.517	0.825	318.05	1.092	1.274	0.944	0.980	308.45	1.000	1.909
0.249	0.666	327.21	1.360	1.090	0.577	0.850	316.39	1.068	1.335	0.973	0.990	307.85	1.000	1.975
0.265	0.678	326.69	1.324	1.095	0.605	0.860	315.77	1.051	1.368	1.000	1.000	307.31	1.000	
0.337	0.734	323.60	1.243	1.130	0.671	0.885	314.18	1.030	1.444					
HFE-347mcc (1) + Ethyl Acetate (2)														
0.000	0.000	350.20		1.000	0.137	0.490	334.21	1.466	1.032	0.643	0.870	314.85	1.034	1.363
0.020	0.120	346.84	1.717	1.004	0.168	0.540	331.90	1.416	1.051	0.710	0.894	313.41	1.011	1.462
0.038	0.208	344.27	1.688	1.004	0.254	0.645	327.12	1.294	1.085	0.746	0.909	312.57	1.008	1.482
0.062	0.299	341.28	1.624	1.011	0.330	0.710	323.84	1.216	1.122	0.789	0.924	311.64	1.001	1.551
0.064	0.299	341.32	1.575	1.011	0.403	0.758	321.30	1.155	1.166	0.815	0.934	311.02	1.001	1.579
0.070	0.319	340.61	1.570	1.015	0.458	0.788	319.61	1.116	1.207	0.846	0.946	310.39	1.000	1.609
0.101	0.408	337.47	1.518	1.020	0.560	0.835	316.91	1.060	1.292	0.899	0.964	309.24	1.000	1.693
0.115	0.441	336.26	1.483	1.023	0.605	0.855	315.77	1.045	1.328	1.000	1.000	307.31	1.000	
HFE-449mec-f (1) + 1-Propanol (2)														
0.000	0.000	370.40		1.000	0.193	0.630	349.16	2.851	1.089	0.735	0.782	343.85	1.108	2.481
0.008	0.084	368.31	5.092	1.000	0.315	0.680	346.78	2.035	1.234	0.781	0.799	343.72	1.071	2.780
0.044	0.336	360.91	4.581	1.006	0.380	0.697	346.07	1.770	1.334	0.828	0.819	343.74	1.035	3.174
0.057	0.391	359.08	4.319	1.009	0.430	0.711	345.52	1.629	1.417	0.849	0.834	343.69	1.028	3.350
0.067	0.434	357.52	4.289	1.011	0.517	0.729	344.98	1.411	1.611	0.883	0.856	343.86	1.009	3.713
0.078	0.464	356.41	4.109	1.013	0.541	0.734	344.75	1.369	1.681	0.909	0.879	343.92	1.006	3.977
0.105	0.521	354.13	3.665	1.028	0.581	0.741	344.56	1.295	1.809	0.936	0.908	344.25	1.000	4.288
0.131	0.578	351.73	3.520	1.034	0.615	0.751	344.34	1.250	1.911	1.000	1.000	345.03	1.000	
0.159	0.605	350.43	3.171	1.057	0.693	0.770	343.98	1.152	2.245					
HFE-449mec-f (1) + 2-Butanone (2)														
0.000	0.000	352.93		1.000	0.281	0.279	354.85	0.719	0.943	0.638	0.725	351.79	0.907	0.790
0.018	0.014	353.35	0.572	0.991	0.326	0.335	354.86	0.742	0.929	0.678	0.766	351.19	0.920	0.769
0.032	0.024	353.48	0.576	0.990	0.360	0.379	354.79	0.764	0.914	0.700	0.786	350.86	0.925	0.762
0.049	0.038	353.64	0.583	0.989	0.393	0.423	354.64	0.785	0.900	0.720	0.807	350.47	0.934	0.749
0.072	0.057	353.83	0.592	0.988	0.425	0.466	354.44	0.804	0.886	0.739	0.825	350.10	0.942	0.737
0.091	0.074	353.97	0.605	0.986	0.444	0.489	354.29	0.812	0.880	0.761	0.842	349.76	0.944	0.733
0.112	0.093	354.15	0.614	0.983	0.456	0.506	354.05	0.825	0.875	0.776	0.856	349.46	0.951	0.721
0.131	0.112	354.27	0.629	0.980	0.489	0.550	353.73	0.845	0.857	0.793	0.870	349.14	0.956	0.710
0.154	0.134	354.43	0.638	0.976	0.572	0.653	352.73	0.885	0.815	0.828	0.896	348.54	0.962	0.694
0.180	0.161	354.58	0.655	0.971	0.585	0.668	352.54	0.890	0.810	0.912	0.950	347.01	0.975	0.682
0.205	0.188	354.68	0.668	0.966	0.601	0.687	352.27	0.898	0.803	0.957	0.977	346.26	0.980	0.656
0.275	0.271	354.84	0.714	0.946	0.612	0.699	352.12	0.902	0.796	1.000	1.000	345.03	1.000	
HFE-449mec-f (1) + Ethyl Acetate (2)														
0.000	0.000	350.20		1.000	0.263	0.237	352.75	0.696	0.953	0.494	0.535	352.57	0.843	0.851
0.024	0.017	350.60	0.577	0.994	0.277	0.253	352.80	0.707	0.949	0.521	0.571	352.33	0.859	0.836
0.048	0.033	350.83	0.574	0.995	0.293	0.272	352.89	0.717	0.943	0.559	0.614	352.02	0.871	0.825
0.069	0.049	351.11	0.577	0.991	0.308	0.291	352.95	0.727	0.937	0.591	0.656	351.64	0.891	0.802
0.077	0.055	351.13	0.587	0.993	0.323	0.309	352.98	0.737	0.931	0.636	0.702	351.16	0.901	0.792
0.092	0.067	351.36	0.589	0.989	0.340	0.332	353.03	0.748	0.924	0.677	0.751	350.56	0.923	0.761
0.106	0.078	351.42	0.600	0.990	0.360	0.357	353.05	0.760	0.916	0.728	0.798	349.89	0.932	0.750
0.121	0.091	351.63	0.604	0.987	0.381	0.384	353.04	0.774	0.907	0.764	0.834	349.25	0.948	0.724
0.136	0.105	351.72	0.616	0.986	0.402	0.412	353.04	0.786	0.897	0.825	0.887	348.10	0.970	0.691
0.152	0.119	351.95	0.624	0.981	0.426	0.444	352.96	0.801	0.886	0.888	0.931	347.18	0.975	0.681
0.163	0.130	351.96	0.635	0.981	0.445	0.470	352.84	0.815	0.877	0.939	0.965	346.40	0.982	0.647
0.190	0.156	352.26	0.648	0.973	0.468	0.501	352.74	0.829	0.864	1.000	1.000	345.03	1.000	
0.234	0.204	352.58	0.678	0.962										

The pressure in the still was measured using a Fortin-type mercury barometer. Since the barometric pressure changed slightly, the experimental temperatures were corrected to 101.3 kPa.[4] The equilibrium temperature was measured with a calibrated platinum resistance thermometer with an accuracy of ±0.01 K.

Analysis. Vapor and liquid samples were analyzed with a gas chromatograph (HP-GC6896 series) equipped with a

Table 4. Results[a] of Thermodynamic Consistency Tests of VLE Data for Six Binary Systems Containing HFE-347mcc or HFE-449mec-f at 101.3 kPa

	HFE-347-mcc(1) + 1-propanol(2)	HFE-347-mcc(1) + 2-butanone(2)	HFE-347 cc (1) + ethyl acetate(2)	HFE-449 ec-f (1) + 1-propanol(2)	HFE-449-mec-f(1) + 2-butanone(2)	HFE-449-mec-f(1) + ethyl acetate(2)
point test[b]	+	+	+	−	−	−
Δy_1	0.007	0.007	0.008	0.005	0.005	0.004
area test[c]	+	+	+	+	+	+
$(D - J)$/%	−28.41	0.409	9.838	−16.97	−19.43	−17.19

[a] Results of the tests are characterized by the signs + (pass) and − (not pass). [b] The criterion for passing the test is $\Delta y_1 \leq 0.010$ absolute in mole fraction.[6,7] [c] The criterion for passing the test is $D - J \leq 10\%$.[6,8]

Table 5. Azeotropic Composition $x_{1(az)}$ in Mole Fraction and Temperature $T_{(az)}$ for the Four Binary Systems at 101.3 kPa

system	$x_{1(az)}$	$T_{(az)}$/K
HFE-347mcc (1) + 1-propanol (2)	0.984	307.25
HFE-449mec-f (1) + 1-propanol (2)	0.812	343.70
HFE-449mec-f (1) + 2-butanone (2)	0.290	354.92
HFE-449mec-f (1) + ethyl acetate (2)	0.369	353.08

Table 6. Parameters[a] for the Six Binary Systems Using the Wilson and NRTL Equations

	Wilson eq		NRTL eq		
system	$\lambda_{12} - \lambda_{11}$	$\lambda_{12} - \lambda_{22}$	$g_{12} - g_{22}$	$g_{21} - g_{11}$	α_{12}
HFE-347mcc (1) + 1-propanol (2)	17 369.36	82 576.75	6021.01	2892.84	0.40
HFE-347mcc (1) + 2-butanone (2)	8 627.20	13 411.27	−176.37	2309.84	0.30
HFE-347mcc (1) + ethyl acetate (2)	3 358.95	13 447.31	292.53	1349.94	0.30
HFE-449mec-f (1) + 1-propanol (2)	11 072.97	51 327.79	2342.59	329.88	0.30
HFE-449mec-f (1) + 2-butanone (2)	−18 204.82	3 447.14	−1731.97	183.70	0.30
HFE-449mec-f (1) + ethyl acetate (2)	−19 027.71	3 295.55	8150.52	−5782.79	0.25

[a] $J \cdot mol^{-1}$.

Table 7. Deviations between the Calculated and Experimental Vapor-Phase Mole Fractions, Δy_1, and Temperature, ΔT, for the Six Binary Systems Using the Wilson and NRTL Equations[a]

		Wilson eq		NRTL eq	
system		Δy_1	ΔT/K	Δy_1	ΔT/K
HFE-347mcc (1) + 1-propanol (2)	avg	0.002	0.50	0.005	0.88
	max.	0.009	2.39	0.018	3.78
HFE-347mcc (1) + 2-butanone (2)	avg	0.011	0.72	0.011	0.76
	max.	0.024	1.36	0.026	1.42
HFE-347mcc (1) + ethyl acetate (2)	avg	0.012	0.43	0.012	0.44
	max.	0.022	0.76	0.023	0.83
HFE-449mec-f (1) + 1-propanol (2)	avg	0.008	0.25	0.007	0.21
	max.	0.016	0.41	0.019	0.66
HFE-449mec-f (1) + 2-butanone (2)	avg	0.005	0.57	0.006	0.45
	max.	0.012	0.82	0.011	0.70
HFE-449mec-f (1) + ethyl acetate (2)	avg	0.006	0.45	0.005	0.44
	max.	0.009	0.63	0.015	0.62

[a] $\Delta y_1 = \sum_k |y_{1,exp} - y_{1,calc}|_k / N$; $\Delta T = 100 \sum_k |T_{exp} - T_{calc}|_k / N$; N = number of data points.

flame ionization detector. Pora PLOT Q (GL Science Co.) was used as the column packing and helium as the carrier gas. The relationship between peak area and composition was determined from analysis of samples of known composition. The accuracy of the equilibrium concentration was estimated to be ±0.005 in mole fraction.

Materials. HFE-347mcc and HFE-449mec-f were provided by Research Institute of Innovative Technology for the Earth (RITE). 1-Propanol, 2-butanone, and ethyl acetate were special grade pure reagents (Wako Pure Chemical Industry, Inc., Japan, and Aldrich Chemical Co., Inc., USA). These materials were used without further purification. The purity of all organic chemicals was checked by gas chromatography and was found to be better than 99.9 mol %. In Table 1, some measured properties of the samples are shown together with the literature values.

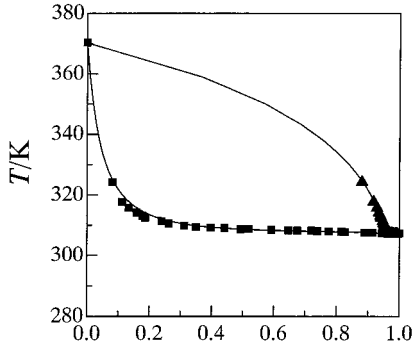

Figure 1. Temperature−composition diagram for the HFE-347mcc (1) + 1-propanol (2) system: (■) experimental liquid-phase mole fractions x_1; (▲) experimental vapor-phase mole fractions y_1; (−) Wilson equation.

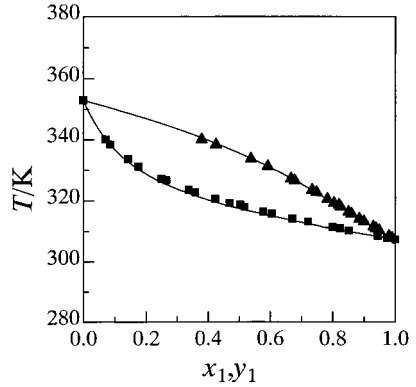

Figure 2. Temperature−composition diagram for the HFE-347mcc (1) + 2-butanone (2) system: (■) experimental liquid-phase mole fractions x_1; (▲) experimental vapor-phase mole fractions y_1; (−) Wilson equation.

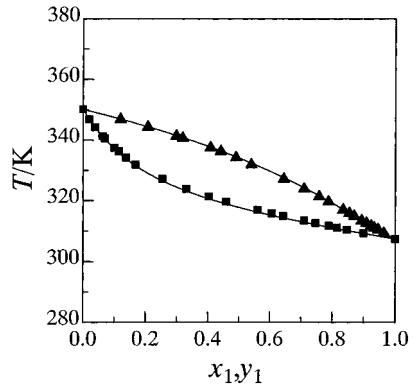

Figure 3. Temperature−composition diagram for the HFE-347mcc (1) + ethyl acetate (2) system: (■) experimental liquid-phase mole fractions x_1; (▲) experimental vapor-phase mole fractions y_1; (−) Wilson equation.

Experimental Results

The experimental results of the six binary VLE data are shown in Table 3 and Figures 1−12. The activity coefficients, γ_i, in Table 3 and the figures were calculated by

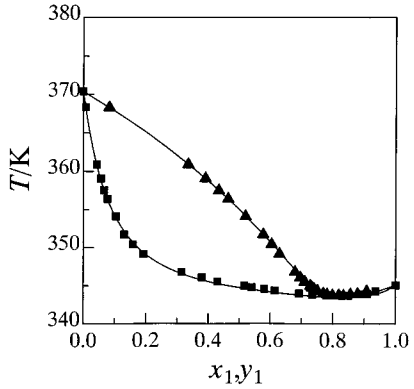

Figure 4. Temperature−composition diagram for the HFE-449mec-f (1) + 1-propanol (2) system: (■) experimental liquid-phase mole fractions x_1; (▲) experimental vapor-phase mole fractions y_1; (−) Wilson equation.

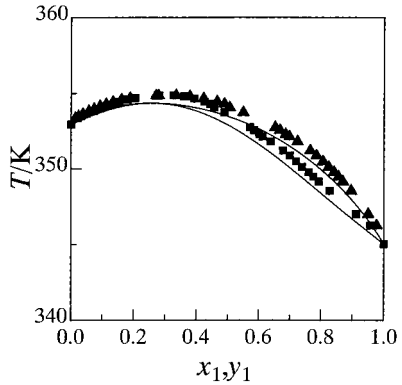

Figure 5. Temperature−composition diagram for the HFE-449mec-f (1) + 2-butanone (2) system: (■) experimental liquid-phase mole fractions x_1; (▲) experimental vapor-phase mole fractions y_1; (−) Wilson equation.

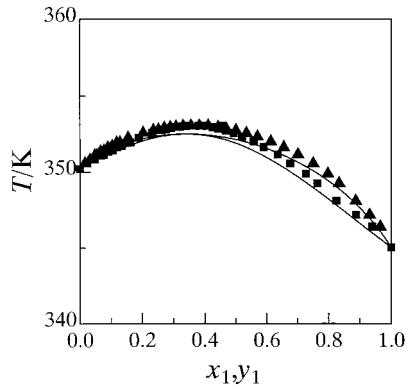

Figure 6. Temperature−composition diagram for the HFE-449mec-f (1) + ethyl acetate (2) system: (■) experimental liquid-phase mole fractions x_1; (▲) experimental vapor-phase mole fractions y_1; (−) Wilson equation.

the following equation in which the vapor phase is assumed to be an ideal gas

$$Py_i = x_i\gamma_i P_i^{S} \qquad (1)$$

because we were not able to calculate the vapor-phase fugacity coefficients of HFE-347mcc and HFE-449mec-f. In eq 1, the vapor pressures of the pure components, P_i^{S}, were calculated from the Antoine equation constants shown in Table 2.

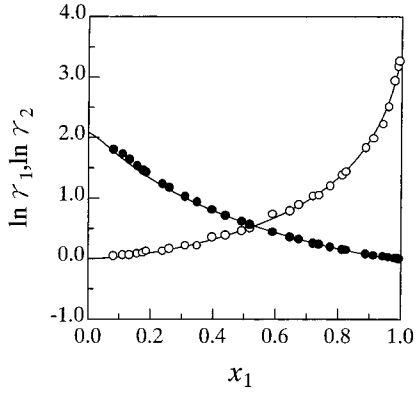

Figure 7. Activity coefficient−liquid composition for the HFE-347mcc (1) + 1-propanol (2) system: (●, ○) experimental; (−) Wilson equation.

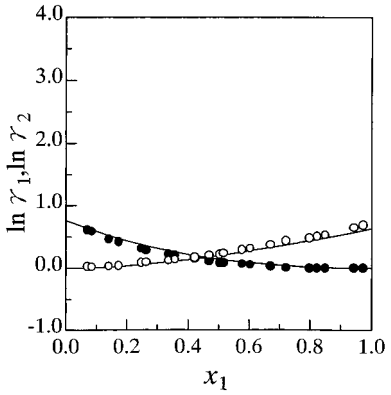

Figure 8. Activity coefficient−liquid composition for the HFE-347mcc (1) + 2-butanone (2) system: (●, ○) experimental; (−) Wilson equation.

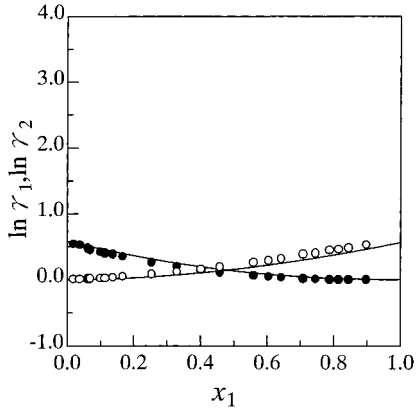

Figure 9. Activity coefficient−liquid composition for the HFE-347mcc (1) + ethyl acetate (2) system: (●, ○) experimental; (−) Wilson equation.

The experimental VLE data were examined by the thermodynamic consistency using the point test of Fredenslund et al.[7] and the area test of Herington[8] as described by Gmehling and Onken.[6] The results of these consistency tests are shown in Table 4. The reported data were found to be thermodynamically consistent according to the point and area tests.

The systems containing HFE-347mcc and HFE-449mec-f with 1-propanol form minimum boiling azeotropes. The systems containing HFE-449mec-f with 2-butanone or ethyl acetate have maximum boiling azeotropes. The binary azeotropic point was determined by a graphical method[9] on the basis of experimental VLE data. Table 5 shows the

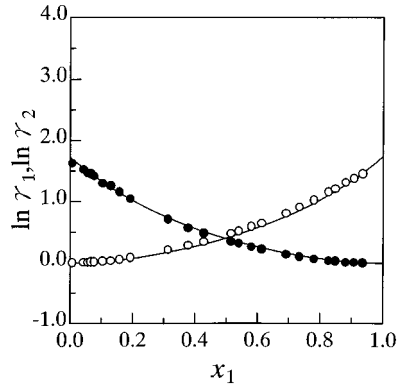

Figure 10. Activity coefficient−liquid composition for the HFE-449mec-f (1) + 1-propanol (2) system: (●, ○) experimental; (−) Wilson equation.

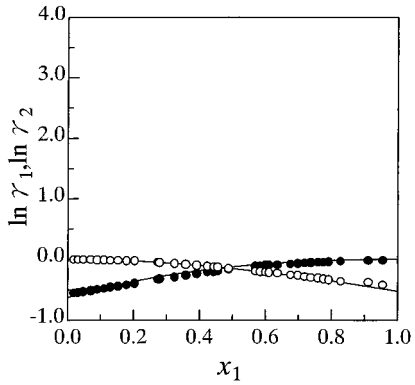

Figure 11. Activity coefficient−liquid composition for the HFE-449mec-f (1) + 2-butanone (2) system: (●, ○) experimental; (−) Wilson equation.

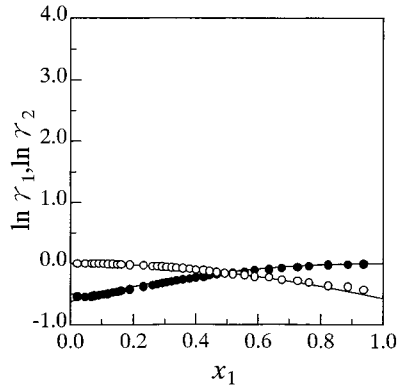

Figure 12. Activity coefficient−liquid composition for the HFE-449mec-f (1) + ethyl acetate (2) system: (●, ○) experimental; (−) Wilson equation.

azeotropic compositions and boiling points for four binary systems.

Correlation

The activity coefficients of the six binary systems were correlated by the Wilson[10] and NRTL equations.[11] The following objective function was minimized during optimization of the parameters in each of the two equations.

$$F_{\text{obj}} = \sum_{k=1}^{N}\left[\left(\frac{\gamma_{1,\text{calc}} - \gamma_{1,\text{exp}}}{\gamma_{1,\text{exp}}}\right)_k^2 + \left(\frac{\gamma_{2,\text{calc}} - \gamma_{2,\text{exp}}}{\gamma_{2,\text{exp}}}\right)_k^2\right] \quad (2)$$

Tables 6 and 7 list the estimated parameters of six binary systems and the deviations between experimental and calculated vapor-phase compositions and bubble point temperatures. The liquid molar volumes, v_i^L, used in the Wilson equation are shown in Table 2. The nonrandomness parameter α_{12} in the NRTL equation was treated as the fitting parameter for all the binary systems in this paper. The Wilson and NRTL equations yielded similar results. In Table 7, the two systems HFE-347mcc + 1-propanol and HFE-347mcc + 2-butanone show rather high deviations in bubble point temperature correlation with both equations. But for the predictions of azeotropic compositions and temperatures, the Wilson equation gave somewhat better results than those of the NRTL equation. The correlated results from the Wilson equation are illustrated in Figures 1−12.

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