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Vapor−Liquid Equilibrium (VLE) Properties for the Binary Systems Propane (1) + *n*-Butane (2) and Propane (1) + Isobutane (3)

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PTxy data (bubble-point and dew-point pressures at a specific composition) for two binary systems, propane (1) + *n*-butane (2) and propane (1) + isobutane (3), are presented in this paper. These binary systems are under consideration for use as a refrigerant for a Lorentz cycle. A recirculation method along with the aid of a new composition measurement system was employed. The experimental uncertainty is 3 mK for temperature, 0.027% + 1.04 kPa for pressure, and 0.11 mol % for composition. A total of 60 data points were obtained in the temperature range of (270 to 310) K for propane (1) + *n*-butane (2) and (260 to 320) K for propane (1) + isobutane (3). Comparisons of the present data with available mixture thermodynamic models and other experimental data are discussed in this paper.

Introduction

Light hydrocarbon (HC) blends are receiving attention for use in refrigeration engineering because of their potential as a mixture refrigerant that realizes the Lorentz cycle with high performance. There are some experimental studies conducted via a drop-in test for the propane (1) + *n*-butane (2) mixture or the propane (1) + isobutane (3) mixture reporting that the COPc (coefficient of performance for cooling) improves by up to 11%[1,2] compared with those with R 22.

VLE (vapor−liquid equilibrium) properties such as P, T, ρ', ρ'', and xy (pressure, temperature, densities, and compositions at the bubble point or dew point) are some of the most essential thermodynamic properties needed to examine the cycle characteristics.

Recently, Miyamoto and Watanabe developed Helmholtz-type equations of state (EoS) for propane (1),[3] *n*-butane (2),[4] and isobutane (3).[5] Their EoSs are included in thermodynamic-property calculation software, REFPROP (version 7.0)[6] released by NIST. In this software, mixture thermodynamic properties are calculated by a mixing rule proposed by Lemmon and Jacobsen.[7] Their mixing rule is a universal one, applicable for most mixture systems. Miyamoto and Watanabe[8] also formulated a mixture model for the binary and ternary mixtures of propane (1), *n*-butane (2), and isobutane (3). They determined the fitting parameters using the available mixture data for each binary system. To investigate the performance of these mixture models, they must be compared with available mixture data.

Concerning the VLE properties for HC refrigerants, most of the data were measured several decades ago because of other demands for thermodynamic properties of natural gas components. Therefore, in the present study we aimed to measure VLE properties for HC refrigerant mixtures by means of a recirculation method.[9,10] The uncertainty in the composition measurement was successfully improved by employing a new gas chromatograph and a sampling system. A total of 60 *PTxy* data points were obtained for the binary systems propane (1) + *n*-butane (2) and propane (1) + isobutane (3). The data were compared with the two mixture models.

Experimental Section

Overview of the Apparatus. In the present study, a VLE property measurement apparatus originally developed by Shimawaki et al.[9] was employed after minor modifications. A schematic diagram of the apparatus is illustrated in Figure 1. The apparatus consists of a liquid bath (LB) and equilibrium cell (EC), an air bath (AB) for the sampling loops, and some measuring instruments.

The equilibrium cell is located inside the thermostated liquid bath. Its temperature was precisely controlled by means of an electric subheater (H1) and a circulation bath (CB) within a fluctuation of ±1 mK. Ethylene glycol or water was used as a heat-transfer medium. The sample temperature was detected with a platinum resistance thermometer placed on the equilibrium cell wall and automatically recorded by a computer (PC1). The sample pressure was measured by a quartz pressure transducer PT (Paroscientific, 2900AT) and also recorded by the computer (PC1). A new gas chromatograph (GC, Agilent Technologies, 6890N) was used for measurements of the mole fraction. The sampling system is discussed below.

Sampling System. In the present study, we made a minor modification to the sampling system. In Figure 1, the saturated vapor is circulated from the top of the equilibrium cell by a small magnetic pump (CP1) through a hexagon valve (VV1) to the bottom of the cell. The saturated liquid circulates in the opposite direction via another hexagon valve (VV2). By switching the hexagon valve, a certain amount of refrigerant is sampled and fed to a secondary circulation loop that was evacuated in advance. The secondary loop for the saturated liquid has enough volume to let the refrigerant vaporize by adding an expansion vessel (EV). Thus, the sampled refrigerant

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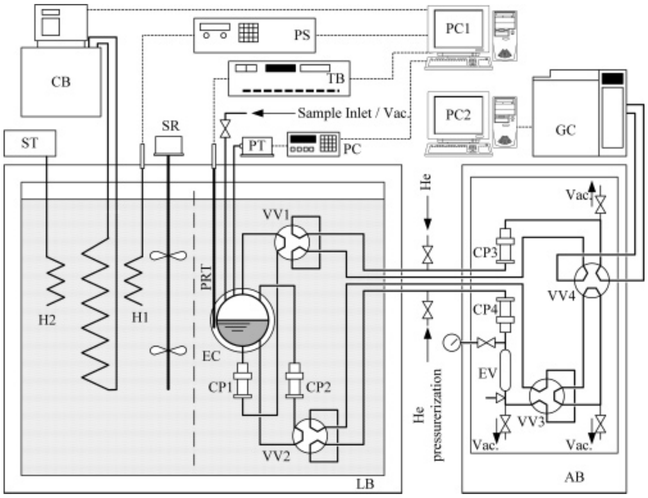

Figure 1. Schematic diagram of the VLE property measurement system: AB, air thermostatic bath; CB, circulation bath; CP1-4, circulation pumps; EC, equilibrium cell; EV, expansion vessel; GC, gas chromatograph; H1, 2, electric heaters; LB, liquid bath; PC, pressure computer; PC1, 2, personal computers; PRT, platinum resistance thermometer; PS, power supply; PT, pressure transducer; SR, stirrer; ST, slide transformer; TB, thermometer bridge; VV1-4, hexagon valves.

Table 1. Measurement Uncertainty for VLE Property Apparatus

property	source	uncertainty
T	PRT	1 mK
	fluctuation	1 mK
→ total ($k = 2$):		3 mK
P	repeatability	0.01%
	calibration	0.0090% + 0.16 kPa
	fluctuation	0.5 kPa
→ total ($k = 2$):		0.027% + 1.04 kPa
x, y	GC calibration	0.02 mol %
	repeatability	0.05 mol %
→ total ($k = 2$):		0.11 mol %

inside the secondary loop is pressurized by introducing high-purity (99.999%) helium gas and is then circulated by another magnetic pump (CP3 or 4). A VV4 hexagon valve is used for switching the sampling loop of the gas chromatograph to the secondary loop for the saturated vapor or liquid. As for the saturated-liquid secondary loop, the refrigerant is once again diluted through a hexagon valve (VV3).

Composition Measurement. The composition of the diluted refrigerant sample circulating through the secondary loop is measured by the gas chromatograph. As a result, one can obtain area fractions calculated from the peaks of the gas chromatogram. The calculated area fraction data is converted to mole fraction by applying conversion factors that were determined as described below.

The ternary gas mixture propane (1) + n-butane (2) + isobutane (3) of known composition ($x_1, x_2 = 0.3728, 0.3140$) is prepared in a large bottle (10 L) by mixing pure refrigerants that were precisely weighed to 0.1 mg. This reference sample was measured by the gas chromatograph several times, and then the conversion factors for propane (1), n-butane (2), and isobutane (3) were calculated from each peak area and the mole fraction.

Uncertainty. In the present study, the experimental uncertainty was evaluated using the ISO guideline[11] with the coverage factor $k = 2$.

Table 1 summarizes the uncertainty budget of the present study. The PRT and the pressure gauge were calibrated at the National Metrology Institute of Japan (NMIJ). By adding the temperature fluctuation of the PID control (1 mK of the standard deviation), we evaluated the temperature measurement uncertainty to be 3 mK. However, that for the pressure measurement becomes 0.027% + 1.04 kPa because of the repeatability of the pressure gauge, calibration, and pressure fluctuation during the measurement caused by the pulsation of the magnetic pumps.

Table 2. Present Experimental VLE Property Data

T/K	P/kPa	x_1	y_1	T/K	P/kPa	x_1	y_1
			Propane (1) + n-Butane (2)				
270.000	142.6	0.1744	0.4616	290.000	269.8		0.4653
270.000	231.8	0.4226	0.7558	290.000	371.7	0.3294	0.6425
270.000	317.0	0.6662	0.8935	290.000	525.5	0.5947	0.8360
270.000	388.2	0.8702	0.9658	290.000	586.2	0.6934	0.8848
280.000	197.9	0.1734	0.4585	290.000	675.1	0.8039	
280.000	319.7	0.4327	0.7427	300.000	355.0		0.2928
280.000	394.4	0.5906	0.8484	300.000	449.5	0.3090	0.5339
280.000	521.8	0.8704	0.9603	300.000	532.8	0.3878	0.6703
				300.000	593.2	0.4804	0.7433
				300.000	720.6	0.6488	0.8492
				300.000	906.5		0.9467
				310.000	471.1	0.1692	0.3550
			Propane (1) + Isobutane (3)				
260.000	128.9		0.4165	280.000	252.3	0.1834	0.3508
260.000	161.2	0.3410	0.6172	280.000	315.5	0.3441	0.5720
260.000	190.6	0.4770	0.7440	280.000	350.9	0.4405	0.6659
260.000	218.1	0.6078	0.8299	280.000	398.7	0.5686	0.7692
260.000	241.2		0.8862	280.000	440.7	0.6749	0.8295
260.000	259.6	0.8030	0.9175	280.000	492.5	0.8095	
260.000	276.3	0.8778	0.9555	290.000	462.7	0.4249	0.6484
270.000	195.1	0.2169	0.4431	290.000	499.8	0.4900	0.6796
270.000	245.5	0.3947		290.000	546.6	0.5838	0.7715
270.000	305.2	0.6035		300.000	520.4	0.2833	0.4633
270.000	357.4	0.7941	0.8974	300.000	589.8	0.3989	0.6101
270.000	200.1	0.2383	0.4779	300.000	671.8	0.5310	0.7252
270.000	227.4	0.3357	0.5957	300.000	737.8	0.6369	0.8027
270.000	280.8	0.5311	0.7600	300.000	813.3	0.7500	0.8732
270.000	306.5	0.6131	0.8239	300.000	857.0	0.8186	0.9091
270.000	333.5	0.7047	0.8748	300.000	890.4	0.8776	0.9366
270.000	356.9	0.7878	0.9143	320.000	855.8	0.2727	0.4050
270.000	370.8	0.8380	0.9333	320.000	930.9	0.3665	0.5120
				320.000	1002.4		0.6009
				320.000	1103.1	0.5506	0.7102
				320.000	1194.7	0.6407	0.7856
				320.000	1318.3	0.7655	0.8643

One cannot estimate the random effect of composition measurement uncertainty during the experiment because the sample state will change after the sampling of a certain amount of gas. In other words, it is categorized as type-B uncertainty.[11] Hence, in the present study it is estimated from the calibration results described before to be 0.05 mol %. The total uncertainty in the composition measurement was evaluated as 0.11 mol %.

Materials. Research-grade samples for propane (1), n-butane (2), and isobutane (3) of 99.99 mol % purity were supplied by Takachio Co., Ltd. Although further purification was not conducted in the present study, the refrigerant sample was drawn from the liquid phase to ensure that the equilibrium cell was charged with a noncondensable gas.

Results and Discussion

Introductory Remarks. In the present study, 20 $PTxy$ property data points were obtained for the binary system propane (1) + n-butane (2). Forty $PTxy$ property data points were also measured for the binary system propane (1) + isobutane (3). All of the measured data are tabulated in Table 2. Note that there is some lack of data due to experimental problems. Sometimes saturated liquid gets mixed into the saturated-vapor sampling loop and vice

Table 3. Relative Pressure Deviation of the Bubble-Point and Dew-Point Pressures for the Binary System Propane (1) + *n*-Butane (2) from the Lemmon and Jacobsen (LJ) Model[7] and the Miyamoto and Watanabe (MW) Model[8]

		experimental range		LJ model			MW model		
first author	N^a	T/K	x_1, y_1	bias	std	rms	bias	std	rms
			Bubble-Point Pressure Data						
this work	17	270 to 310	0.17 to 0.87	−0.16	2.40	2.33	0.26	2.31	2.25
Barber[15]	83	340 to 419	0.15 to 0.93	−0.13	1.79	1.79	0.72	0.71	1.01
Beranek[16]	29	303 to 363	0.06 to 0.91	−1.03	0.82	1.31	−0.85	0.83	1.17
Clark[17]	30	260 to 280	0.00 to 1.00	−1.23	1.57	1.97	−1.01	1.46	1.76
Hirata[14]	35	237 to 249	0.00 to 1.00	2.21	1.64	2.73	2.73	1.80	3.26
Holcomb[13]	69	237 to 414	0.15 to 0.83	2.54	5.23	5.79	2.52	4.73	5.33
Kaminishi[12]	17	273 to 323	0.20 to 0.80	0.01	0.17	0.17	0.32	0.18	0.36
Kay[18]	67	343 to 419	0.15 to 0.93	0.16	1.70	1.70	0.81	2.21	2.35
Kayukawa[19]	21	240 to 380	0.25 to 0.75	−0.69	0.24	0.73	−0.31	0.31	0.43
Nysewander[21]	32	311 to 411	0.20 to 0.85	2.43	2.01	3.13	2.40	2.01	3.11
Skripka[21]	24	253 to 273	0.00 to 1.00	−2.01	1.50	2.49	−1.81	1.31	2.22
			Dew-Point Pressure Data						
this work	20	270 to 310	0.29 to 0.97	0.15	3.09	3.01	0.59	3.02	3.00
Barber[15]	87	340 to 419	0.15 to 0.93	−0.40	0.70	0.80	0.08	0.70	0.70
Beranek[16]	29	303 to 363	0.12 to 0.96	−0.56	1.05	1.18	−0.32	1.02	1.05
Clark[17]	30	260 to 280	0.00 to 1.00	−0.79	1.70	1.85	−0.51	1.50	1.56
Hirata[14]	35	237 to 249	0.00 to 1.00	6.83	3.60	7.69	7.13	3.78	8.05
Holcomb[13]	69	237 to 414	0.18 to 0.94	1.47	4.46	4.66	1.84	4.52	4.85
Kay[18]	67	343 to 419	0.15 to 0.93	−0.39	0.47	0.61	0.06	0.51	0.51
Nysewander[20]	16	344 to 411	0.20 to 0.85	−0.11	1.63	1.58	0.35	1.47	1.46
Skripka[21]	24	253 to 273	0.00 to 1.00	−1.39	4.10	4.25	−1.25	3.96	4.08

[a] Number of measurements.

Table 4. Relative Pressure Deviation of the Bubble-Point and Dew-Point Pressures for the Binary System Propane (1) + Isobutane (3) from the Lemmon and Jacobsen (LJ) Model[7] and the Miyamoto and Watanabe (MW) Model[8]

		experimental range		LJ model			MW model		
first author	N^a	T/K	x_1, y_1	bias	std	rms	bias	std	rms
			Bubble-Point Pressure Data						
this work	37	260 to 320	0.18 to 0.88	−0.79	0.80	1.12	−2.73	0.70	2.81
Higashi[22]	18	283 to 313	0.17 to 0.80	0.22	2.58	2.51	−1.79	2.70	3.17
Hipkin[23]	86	267 to 394	0.00 to 1.00	0.81	2.44	2.55	−0.12	2.45	2.44
Hirata[14]	37	237 to 249	0.00 to 1.00	5.00	2.01	5.37	2.00	1.17	2.31
Hirata[24]	14	340 to 340	0.05 to 0.99	−0.04	0.70	0.67	−1.12	0.93	1.44
Kaminishi[12]	23	273 to 323	0.17 to 0.80	1.91	0.72	2.04	0.04	0.44	0.43
Kayukawa[19]	23	240 to 380	0.25 to 0.75	1.11	1.40	1.76	−0.67	1.07	1.24
Lim[25]	44	273 to 303	0.00 to 1.00	1.20	0.70	1.39	−0.30	0.60	0.66
Skripka[21]	24	253 to 273	0.00 to 1.00	−0.16	0.66	0.66	−1.75	1.10	2.05
			Dew-Point Pressure Data						
this work	38	260 to 320	0.35 to 0.96	−0.57	1.36	1.46	−2.55	1.45	2.92
Higashi[22]	18	283 to 313	0.29 to 0.89	1.56	1.13	1.91	−0.25	1.20	1.19
Hipkin[23]	86	267 to 394	0.00 to 1.00	0.77	2.31	2.42	−0.03	2.13	2.12
Hirata[14]	37	237 to 249	0.00 to 1.00	6.08	2.72	6.65	3.77	1.80	4.16
Hirata[24]	14	340 to 340	0.09 to 0.99	0.18	1.03	1.01	−0.78	0.95	1.20
Lim[25]	44	273 to 303	0.00 to 1.00	2.22	1.22	2.53	0.76	0.50	0.90
Skripka[21]	24	253 to 273	0.00 to 1.00	−0.49	2.02	2.03	−1.67	2.40	2.89

[a] Number of measurements.

versa so as to cause an incorrect measurement of the composition.

Comparison with Equations of State. The measured data are plotted in a $P-x$ diagram as shown in Figure 2. Calculated isotherms of the bubble-point and dew-point pressure predicted by a couple of mixture models, the Lemmon and Jacobsen[7] (LJ) model and the Miyamoto and Watanabe[8] (MW) model, are also included.

In Figure 2, no pressure difference is observed between the predictions of both models for the binary system propane (1) + *n*-butane (2). The present measured data for the system also agree with the predictions.

However, there exists some discrepancy between calculated isotherms for the binary system propane (1) + isobutane (3). The LJ model gives slightly higher bubble-point and dew-point pressures than those computed with the MW model. The present data are better represented by the LJ model.

Comparison with Literature Data. In this study, experimental VLE data were compared with the LJ model and the MW model in additional detail. Table 3 summarizes the statistical characteristics (bias, std, and rms) of the percent deviation of available data for the binary system propane (1) + *n*-butane (2) from those calculated by the two models. The number of measurements and experimental temperature ranges for the literature data are also included.

As described above, it is recognized that both models give almost the same results. The bias from the LJ model is a little smaller than that from the MW model, but the difference is not larger than 0.85%. The data set by Kaminishi et al.[12] has a very small std and rms for both the LJ and MW models. On the contrary, those for the data set by Holcomb et al.[13] are as large as 5%.

Concerning the dew-point data, there is a slightly larger (about 3.0%) std and rms for the present data. The dew-point data by Hirata et al.[14] are found to deviate by about 8% of the rms. This may be due to very small absolute pressures at such low temperatures below 250 K. The std and rms for the data by Holcomb et al. are slightly larger

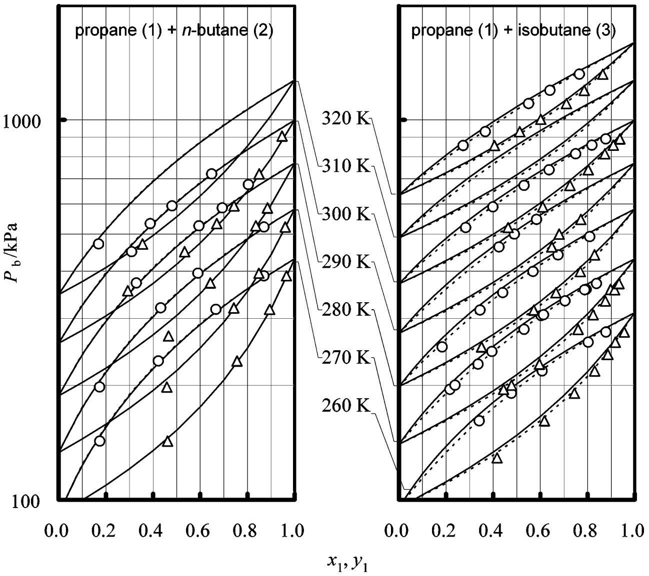

Figure 2. $P-x$ diagram of the binary systems propane (1) + *n*-butane (2) and propane (1) + isobutane (3): ○, bubble point, this work; △, dew point, this work; –, Miyamoto and Watanabe model;[8] - - -, Lemmon and Jacobsen model.[7]

for both mixture models. It is also recognized that the bias from the MW model is larger (by about 0.14 to 0.48%) than those from the LJ model for all of the data.

On the contrary, regarding the binary system propane (1) + isobutane (3), the statistical results are rather different from the two models, as summarized in Table 4. The bubble-point and dew-point pressures calculated by the MW model are slightly higher (by 1 to 3%) than those calculated by the LJ model.

As for the dew-point pressure, the data by Hirata et al.[14] have a larger discrepancy from both models. The same conclusion is reached that the percent deviation increases at very low temperatures.

Conclusions

A total of 60 data points of VLE (*PTxy*) properties for two binary hydrocarbon systems, propane (1) + *n*-butane (2) and propane (1) + isobutane (3), were obtained in the present study. We have modified the sampling system and the gas chromatograph as well as the calibration procedures of the gas chromatograph to improve the reliability of the composition measurement. The experimental uncertainty is 3 mK for temperature, 0.027% + 1.04 kPa for pressure, and 0.11 mol % for composition. The experimental data were compared with two mixture Helmholtz equations of state by Lemmon and Jacobsen and Miyamoto and Watanabe, together with available literature data.

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