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Volumetric Behavior of Carbon Dioxide + Butan-1-ol Mixtures at 313.15 K

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Supporting Information

ABSTRACT: Volumetric behavior was measured of carbon dioxide + butan-1-ol mixtures at 313.15 K using a vibrating tube density meter. The saturated pressures were also measured of carbon dioxide + butan-1-ol mixtures at 313.15 K by the conventional synthetic method. The equilibrium vapor compositions were newly evaluated by combining the molar volume equation, the experimental values of the equilibrium vapor density, vapor density of pure carbon dioxide, and pressure.

INTRODUCTION

The phase equilibrium properties of mixtures containing supercritical fluid (SCF) at elevated pressures continue to be of importance in process development and research. The growing motivation for studying equilibrium of several coexisting phases arises from the need to describe such phase behavior for fluid mixtures of interest in SCF extraction. A large number of applications of SCF technology have been proposed covering the food, pharmaceutical, chemical reaction, coal and oil processing, waste treatment, plastic recycling, and producing industries. Carbon dioxide is widely used in SCF applications because it has moderate critical temperature, nontoxicity, nonflammability, safety, and a small impact on the earth's environment. Supercritical carbon dioxide and alcohol are excellent candidates for industrial use because of their unique and suitable solvent properties. The volumetric properties and vapor−liquid equilibria (VLE) are required for the design and operation of the separation equipment with SCF. Butan-1-ol is widely used as a raw material of the production of other compounds, the solvent during organic chemistry and fiber production, and is also an important alcohol for color paints as a thinner of paints; therefore, the volumetric behavior including butan-1-ol are necessary to the development of the painting technology with supercritical carbon dioxide.

For carbon dioxide + butan-1-ol mixtures, VLE were reported by some researchers[1−6] without the volumetric properties data. On the other hand, Ishihara et al.[7] had reported VLE data with saturated density behavior; however, there were not any volumetric properties data at constant composition. Recently, Elizalde-Solis and Galicia-Luna[8] had reported VLE and phase densities at saturation using a static-analytic type apparatus, but there is no data included from the homogeneous region.

In the present study, the volumetric behavior was measured of carbon dioxide + butan-1-ol mixtures at 313.15 K using a vibrating tube density meter without any analytical instrument. The saturated pressures were also measured of carbon dioxide + butan-1-ol mixtures at 313.15 K by the conventional synthetic method.[9−15] The equilibrium vapor compositions were newly evaluated by combining the molar volume equation, experimental values of the equilibrium vapor density, vapor density of pure carbon dioxide, and pressure.

Table 1. Normal Boiling Point, T_b, Density, ρ, and Refractive Index, n_D, of Butan-1-ol Used

T_b/K		ρ(298.15 K)/kg·m^{-3}		n_D(298.15 K)	
exptl	literature[a]	exptl	literature[a]	exptl	lterature[a]
390.84	390.81	805.8	806.0	1.3973	1.3973

[a] TRC Thermodynamic Table Non-Hydrocarbons.[20]

EXPERIMENTAL SECTION

Materials. In the present experiments, carbon dioxide was supplied by Showa Tansan Co. Ltd., with the guarantee of 0.999 99 mol fraction purity. Butan-1-ol was a special grade reagent of Wako Pure Chemical Industries, Ltd. and was used without further purification. The purity of butan-1-ol was more than 0.999 mol fraction by gas chromatographic area analysis. The refractive index of butan-1-ol was measured by using the Atago model3 abbe refractometer within ± 0.1 % accuracy. The physical properties of butan-1-ol used in this work are listed in Table 1.

Apparatus and Procedure. The apparatus and experimental procedure are almost same as that described previously study.[9−15] The experimental density data at high pressures were obtained with the variable volume apparatus equipped with the vibrating tube density meters. The maximum operating temperature and pressure of the apparatus are 400 K and 20 MPa, respectively. The main parts of the apparatus are a variable volume cell, piston, Ruska 2465-752 and Ruska 2470-703 air lubricated dead weight gauge, Ruska 2480-700 oil dead weight gauge, hand pump syringe, Anton Paar DMA 512P density meters, circulation pumps, gas reservoir, and Ruska 2439-702 pressure transducers. The density meters were calibrated by dry air and pure water at the experimental temperature.

The apparatus is housed in a constant temperature liquid bath controlled to a temperature to about ± 0.01 K. Temperatures

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Table 2. Experimental Density ρ, Mole Fraction x, and Pressure P of Carbon Dioxide (1) + Butan-1-ol (2) at 313.15 K

	ρ/kg·m^{-3} at P/MPa									
x_1	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0	9.0	10.0
0.000	794.9	795.6	796.3	797.0	797.6	798.3	798.9	799.6	800.2	800.9
0.103		802.2	803.0	803.8	804.6	805.4	806.2	806.9	807.7	808.4
0.204				810.6	811.6	812.4	813.4	814.3	815.2	816.1
0.300					817.0	818.1	819.1	820.2	821.2	822.3
0.404							825.1	826.5	827.8	829.1
0.503							829.1	830.9	832.6	834.3
0.600								830.9	833.4	835.8
0.703								824.9	829.1	833.1
0.802									810.5	817.8
0.900									762.6	779.1
0.950									704.5	735.5
1.000	17.9	37.1	58.7	83.5	112.7	148.8	197.3	276.7	483.4	627.4

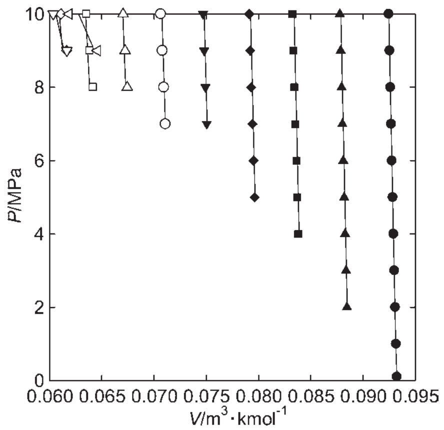

Figure 1. Volumetric behavior of carbon dioxide (1) + butan-1-ol (2) at 313.15 K; ●, x = 0.0; ▲, 0.1; ■, 0.2; ♦, 0.3; ▼, 0.4; ○, 0.5; △, 0.6; □, 0.7; ◇, 0.8; ▽, 0.9; left pointing open triangle, 0.95; —, calculated by eq 1.

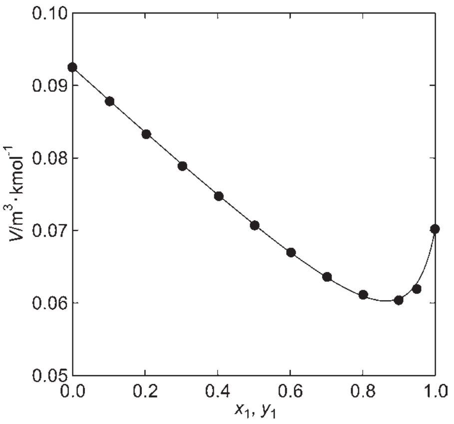

Figure 2. Molar volume of carbon dioxide (1) + butan-1-ol (2) at 313.15 K; ●, this work at 10.000 MPa; —, calculated by eq 1.

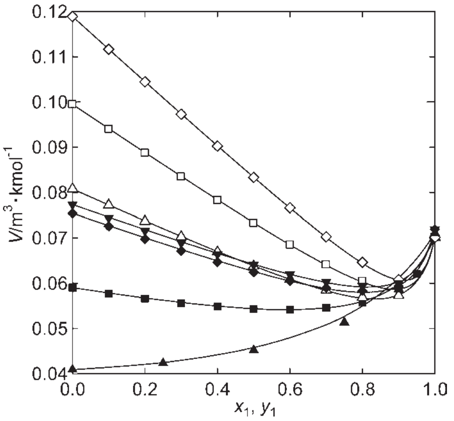

Figure 3. Molar volume of carbon dioxide (1) + solvent (2) at 313.15 K. ▲, carbon dioxide (1) + methanol (2) at 9.887 MPa;[9] ■, carbon dioxide (1) + ethanol (2) at 10.000 MPa;[10] ◆, carbon dioxide (1) + propan-1-ol (2) at 9.800 MPa;[11] ▼, carbon dioxide (1) + propan-2-ol (2) at 9.800 MPa;[12] △, carbon dioxide (1) + methyl acetate (2) at 10.000 MPa; [13] □, carbon dioxide (1) + ethyl acetate (2) at 10.000 MPa;[14] ◇, carbon dioxide (1) + 1-methylethyl acetate (2) at 10.000 MPa;[15] —, calculated by eq 1.

were measured with the Hewlett-Packard 2804A quartz thermometer calibrated by the triple point of water. The volume of the lower portion of the cell could be varied between 234 and 299 cm^3 by moving a piston, which gave a volume uncertainty of ± 0.01 cm^3. The accuracy of the total volume is known about ± 1 cm^3.

First, the cell was evacuated and then filled with carbon dioxide. The pressure difference between the upper and lower spaces was kept close to zero, to minimize the leakage between the upper and lower rooms in the cell. The volume was determined by the motion of the piston. The amount of carbon dioxide was determined from the volume and experimental density at the experimental pressure and temperature. The volume of butan-1-ol was then charged into the cell with the syringe pump with an uncertainty of ± 0.01 cm^3 after pressurizing the syringe pump as quickly as possible, to eliminate the dissolution effect of carbon dioxide into butan-1-ol in the syringe pump. The amount of butan-1-ol could be determined by the volume and density. The composition of the mixture was evaluated by the amount of carbon dioxide and the one of butan-1-ol injected into the cell. As the volume of cell was changed, the volumetric properties of the mixture could be measured at a fixed and known composition. The discontinuity of the pressure as a function of cell volume was used as the determinant of the saturated pressure. The saturated densities of the vapor and liquid were measured at the fixed pressure while maintaining the vapor−liquid equilibrium separation in the cell. The experimental uncertainties of the pressure, density, temperature, and composition, respectively, are 0.001 MPa, 0.1 kg·m^{-3}, 0.01 K, and 0.001 mol fraction.

■ CORRELATION

Molar Volume Equation. In the present study, the previous density equation[16,17] was simplified to reduce the number of parameters from eight or six to three, introducing the molar

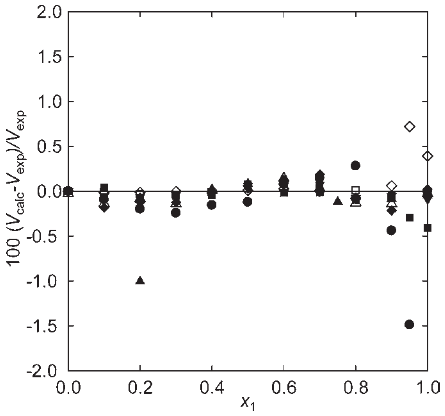

Figure 4. Residuals (experimental − calculated values) for the experimental molar volume of carbon dioxide (1) + solvent (2) to eq 1. ●, carbon dioxide (1) + butan-1-ol (2); ▲, carbon dioxide (1) + methanol (2);[9] ■, carbon dioxide (1) + ethanol (2);[10] ◆, carbon dioxide (1) + propan-1-ol (2);[11] ▼, carbon dioxide (1)+propan-2-ol (2);[12] △, carbon dioxide (1) + methyl acetate (2);[13] □, carbon dioxide (1) + ethyl acetate (2);[14] ◇, carbon dioxide (1) + 1-methylethyl acetate (2).[15]

volume equation as follows:

$$V = \left[x_1 V_1^{0*} + x_2 V_2^{0*} - x_1 x_2 \frac{A}{x_1 + B x_2}\right] \left[1 - \ln\left\{1 - \frac{1-(P/P^*)}{1+\exp[C x_2/(x_1+6x_2)]}\right\}\right] \qquad (1)$$

where V, x, and P are molar volume, composition of mole fraction, and pressure, respectively. The superscript 0 denotes pure. P^* means the reference pressure at high pressure. The subscripts, 1 and 2, denote carbon dioxide and solvent, respectively. Equation 1 has three parameters, A, B, and C. Those parameters are independent of composition. The parameter A is null in ideal solution. The parameters in eq 1 and the standard deviations are shown in Table 4, giving fine results. The applicable composition range was from 0.00 to 0.95 in the mole fraction of carbon dioxide.

The high pressure volumetric data previously measured by the authors[9−15] and the present experimental ones for the eight fluid mixtures made of carbon dioxide with methanol, ethanol, propan-1-ol, propan-2-ol, methyl acetate, ethyl acetate, or 1-methylethyl acetate or butan-1-ol at 313.15 K have been correlated by eq 1.

Equilibrium Vapor Composition. With the combination of the molar volume equation and the experimental values of the equilibrium vapor density, vapor density of pure carbon dioxide, and pressure, the equilibrium vapor compositions were newly evaluated as follows:

$$y_2 = \frac{(\rho - \rho_1^0)V_1^0}{(\rho - \rho_1^0)V_1^0 + (M_2 - \rho\overline{V}_2^{\infty})} \qquad (2)$$

where

$$\overline{V}_2^{\infty} = \left(\frac{P^*}{P}\right)\overline{V}_2^{\infty*}, \quad \overline{V}_2^{\infty*} = V_2^{0*} - A \qquad (3)$$

in which, A, M, y, V, $\overline{V}$, and ρ, respectively, denote parameters in eq 1, molecular weight, vapor mole fraction, molar volume, partial molar volume, and density. The subscripts, 1 and 2, mean the light and heavy components, respectively. The superscripts, 0, ∗, and ∞, respectively, denote the pure component, reference pressure, and infinite dilution. Equation 2 can be derived in the dilution range of the solvent. In the present study, the partial molar volume of solvent is given as the one at the reference pressure for simplicity. The partial molar volume of solvent at infinite dilution $\overline{V}_2^{\infty}$ is given by eq 3, based on eq 1. Equations 2 and 3 can be derived in the dilution composition range of the solvent, as shown in the Appendices in the Supporting Information.

Table 3. Saturation Pressure P, Liquid Mole Fraction x, Vapor Mole Fraction y, Liquid Density ρ_L, and Vapor Density ρ_V, of Carbon Dioxide (1) + Butan-1-ol (2) at 313.15 K

P/MPa	x_1	y_1	ρ_L/kg·m^{-3}	ρ_V/kg·m^{-3}
1.692[a]	0.103[a]	0.964[b]	803.1	34.0
2.690[c]			807.6[c]	54.9[c]
3.197[c]			810.1[c]	67.1[c]
3.284[a]	0.204[a]	0.976[b]	811.0	69.6
4.164[c]			815.0[c]	92.6[c]
4.841[a]	0.300[a]	0.979[b]	818.9	114.4
4.872[c]			818.9[c]	115.2[c]
5.553[c]			822.8[c]	138.3[c]
6.124[a]	0.404[a]	0.980[b]	825.5	163.1
6.618[c]			827.7[c]	185.5[c]
6.702[d]	0.489[d]	0.995[d]	828.8[d]	187.5[d]
6.913[a]	0.503[a]	0.987[b]	829.4	200.1
7.012[d]	0.525[d]	0.997[d]	830.2[d]	205.6[d]
7.405[d]	0.594[d]	0.997[d]	831.0[d]	234.9[d]
7.557[a]	0.600[a]	0.976[b]	828.8	250.8
7.740[d]	0.681[d]	0.996[d]	827.1[d]	269.3[d]
7.794[a]	0.703[a]	0.974[b]	824.8	275.2
7.869[d]	0.731[d]	0.995[d]	821.1[d]	286.3[d]
8.092[a]	0.802[a]			
8.226[a]	0.900[a]			
8.309[a]	0.950[a]			

[a] Conventional synthetic. [b] Present synthetic. [c] VLE separation. [d] Ishihara et al.[7]

RESULTS AND DISCUSSION

Table 2 and Figure 1 give the volumetric behavior of carbon dioxide + butan-1-ol mixture at 313.15 K. As shown in Tables 2 and 4 and Figure 1, the experimental results are correlated by eq 1, and the standard deviation is 0.000 18. Figure 2 gives the molar volume for fluid mixtures made of carbon dioxide with butan-1-ol at 10.000 MPa. The experimental molar volume decreased until 0.8 mol fraction of carbon dioxide and increased after 0.8 mol fraction of carbon dioxide. The correlated result by eq 1 agreed very well with the experimental value, and the standard deviation is 0.000 17. Figure 3 shows the molar volume at the reference high pressures for seven fluid mixtures made of carbon dioxide with methanol, ethanol, propan-1-ol, propan-2-ol, methyl acetate, ethyl acetate, or 1-methylethyl acetate at 313.15 K. Figure 4 shows the deviations of the experimental or literature[9−15] molar volume data from the correlated results by eq 1. There was good agreement for the molar volumes of carbon dioxide + solvent mixtures between the experimental or literature[9−15] molar volume data and the correlated results by eq 1, in which the average standard deviation is 0.000 25. Table 3

Table 4. Parameters in Molar Volume Equation 1 for Fluid Mixtures at 313.15 K[a]

system	N	$V_{1,\mathrm{expl}}{}^{*}$/m^3·kmol^{-1}	$V_{2,\mathrm{expl}}{}^{0*}$/m^3·kmol^{-1}	$P_{\mathrm{expl}}{}^{*}$/MPa	A	B	C	σ
CO_2 (1) + methanol (2)[9]	26	0.071 10	0.040 91	9.887	0.0976	3.74	22.8	0.000 58
CO_2 (1) + ethanol (2)[10]	53	0.070 15	0.059 03	10.000	0.1665	7.16	26.5	0.000 33
CO_2 (1) + propan-1-ol (2)[11]	63	0.071 91	0.075 42	9.800	0.3167	13.33	28.0	0.000 30
CO_2 (1) + propan-2-ol (2)[12]	67	0.071 91	0.077 35	9.800	0.2994	13.34	26.5	0.000 30
CO_2 (1) + methyl acetate (2)[13]	68	0.070 15	0.080 78	10.000	0.3595	14.47	27.0	0.000 12
CO_2 (1) + ethyl acetate (2)[14]	72	0.070 15	0.099 46	10.000	0.4198	17.34	27.4	0.000 10
CO_2 (1) + 1-methylethyl acetate (2)[15]	69	0.070 15	0.118 88	10.000	0.4373	18.60	26.8	0.000 06
CO_2 (1) + butan-1-ol (2)	52	0.070 15	0.092 46	10.000	0.2804	12.43	29.0	0.000 18
							average	0.000 25

[a] N: number of data points. σ: standard deviation. $\sigma = \sqrt{\frac{\sum (V_{\mathrm{calc}} - V_{\mathrm{expl}})^2}{N-1}}$/m^3·kmol^{-1}

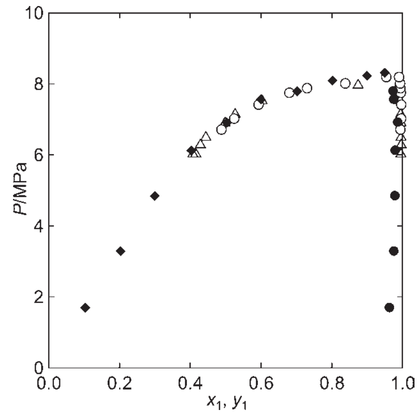

Figure 5. Vapor−liquid equilibrium of carbon dioxide (1) + butan-1-ol (2) at 313.15 K; ◆, this work (conventional synthetic by saturation pressure); ●, this work (present synthetic by eqs 2 and 3); △, Hiaki et al.;[6] ○, Ishihara et al.[7]

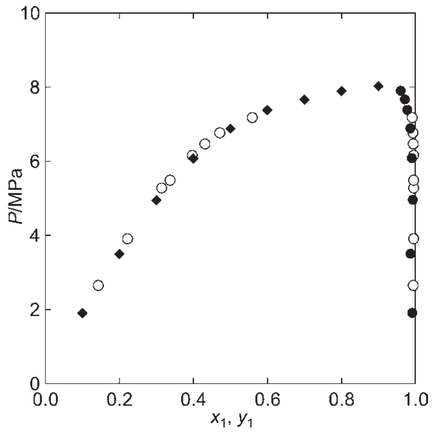

Figure 6. Vapor−liquid equilibrium of carbon dioxide (1) + propan-1-ol (2) at 313.15 K; ●, this work (present synthetic by eqs 2 and 3); ◆, Yaginuma et al.[11] (conventional synthetic by saturation pressure); ○, Vandana et al.[18] at 315.0 K.

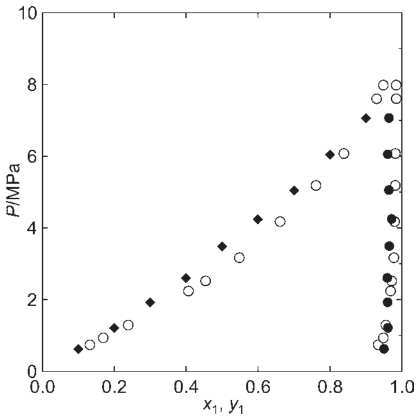

Figure 7. Vapor−liquid equilibrium of carbon dioxide (1) + methyl acetate (2) at 313.15 K; ●, this work (present synthetic by eqs 2 and 3); ◆, Kato et al.[13] (conventional synthetic by saturation pressure); ○, Ohgaki and Katayama.[19]

and Figure 5 show the phase equilibrium results of carbon dioxide + butan-1-ol at 313.15 K. As shown in Figure 5, the equilibrium liquid composition agreed fairly well with the literature data[6,7] by the analytical method, in which the absolute average deviation ($\sum|\Delta x|/N$) between the experimental data and the literature data by Hiaki et al.[6] or Ishihara et al.[7] are 0.027 and 0.033, respectively. On the equilibrium vapor composition, there was a little difference between the evaluated value by eqs 2 and 3 and the literature data, in which the absolute average deviation ($\sum|\Delta y|/N$) between the experimental data and the literature data by Hiaki et al.[6] or Ishihara et al.[7] are 0.013 and 0.016, respectively. Therefore, the saturated vapor points given by eqs 2 and 3 seem reliable over the composition 0.97 mol fraction of the light component. Less than 0.97 mol fraction of the light component, the saturated vapor points given by eqs 2 and 3 seem doubtful because these equations are composed in the dilution composition range of the heavy component. Figures 6 and 7, respectively, show the phase equilibrium results of carbon dioxide + propan-1-ol and carbon dioxide + methyl acetate at 313.15 K. The saturated vapor points were determined by the present synthetic technique with eqs 1 to 3. The absolute average deviation ($\sum|\Delta y|/N$) between evaluated values by the present synthetic technique and literature data[18,19] are 0.005 for carbon dioxide + propan-1-ol and 0.012 for carbon dioxide + methyl acetate, respectively. In the determination of the saturated vapor points, all terms in the right-hand side in eqs 2 and 3 were given by the experimental values. Therefore, the reliability of the saturated vapor point seems reasonable as shown in Figures 6 and 7.

■ CONCLUSIONS

The volumetric properties and saturated pressures of carbon dioxide + butan-1-ol were newly measured in the present study.

The number of parameters in the density equation at the previous studies[16,17] was reduced from eight or six to three. An equation for the correlation of high pressure molar volume behavior of fluid mixtures made of carbon dioxide with methanol, ethanol, propan-1-ol, propan-2-ol, methyl acetate, ethyl acetate, 1-methylethyl acetate, or butan-1-ol at 313.15 K was presented in the composition range from 0.00 to 0.95 mol fraction of carbon dioxide. With the combination of the molar volume equation and the experimental values of the equilibrium vapor density, vapor density of pure carbon dioxide, and pressure, the equilibrium vapor compositions were evaluated.

■ ASSOCIATED CONTENT

Ⓢ **Supporting Information.** Derivation of eqs 2 (Appendix A) and 3 (Appendix B). This material is available free of charge via the Internet at http://pubs.acs.org.

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