Equilibrium Phase Properties of the Carbon Dioxide–*n*-Heptane System

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Vapor and liquid equilibrium phase compositions have been determined for the carbon dioxide–*n*-heptane system at temperatures of 99.5, 175, 250, and 399.3 °F at several pressures between the vapor pressure of *n*-heptane and the critical pressure of the system. Refractive index measurements were made on *n*-heptane at temperatures of 100, 175, and 250 °F and on the equilibrium vapor and liquid phases at each experimental pressure at 99.5, 175, and 250 °F. These measurements were used with the phase composition data to calculate the equilibrium phase densities using the Lorentz–Lorenz molar refractivity relationship.

Introduction

The recovery, transportation, and processing of reservoir fluids invariably requires a knowledge of the thermodynamic and other properties of complex mixtures which include nonhydrocarbons such as nitrogen, carbon dioxide, and hydrogen sulfide together with the simple light hydrocarbons up to hexane and the more complex heavier hydrocarbons containing seven or more carbon atoms. These heavier fractions usually consist of paraffinic, aromatic, and naphthenic fractions. Essentially all generalized correlations or equations of state used for calculating the properties of these mixtures require at least one fitted binary parameter which is characteristic of each possible binary pair that could be formed from the mixture. Thus experimental data are required on binary systems containing each of the nonhydrocarbon components with typical paraffinic, aromatic, and naphthenic fractions.

The importance of data on systems containing carbon dioxide has increased with the trend toward its widespread use in solvent flooding operations and the consequent processing of gases with high carbon dioxide concentrations. Carbon dioxide is also invariably found in significant quantities in coal gasification or in bitumen recovery and processing.

As the initial phase of a program to measure the properties of these nonhydrocarbon–heavier hydrocarbon binaries, it was decided to study the phase behavior of the carbon dioxide–*n*-heptane system.

Experimental Method

The experimental method used in this work at 99.5, 175, and 250 °F was basically the same as that used in several earlier studies on light hydrocarbon–nonhydrocarbon systems. This has been described in detail by Besserer and Robinson (*2*, *4*). The equilibrium cell was the variable volume type consisting of two cylinder-piston end sections and a smaller diameter central windowed section. The working volume of the cell was from about 10 to 175 mL and its working pressure was about 3000 psia at temperatures in the 0–250 °F range. The windowed section was designed so that visual observation of the cell contents was possible and so that refractive index measurements of each phase could be made at the experimental conditions.

Because of the nature of the components under investigation in this work, some changes in the equipment design and the procedures used had to be made. These are described below.

The first problem encountered during the experimental work was related to the presence of the relatively nonvolatile *n*-heptane with carbon dioxide, a much more volatile component. In these circumstances, during the process of sampling, the "flashing" phenomenon occurs whereby a liquid containing a relatively light and a heavy component undergoes a distillation process when it is throttled across a valve from a high pressure into an evacuated space. Because of this, the concentration of the light component in the material passing through the valve is higher than the concentration of the light component in the liquid upstream from the valve. Thus the sample obtained for analysis is not representative of the equilibrium liquid phase. This problem became apparent during the initial runs at 99.5 °F. Later an indirect reference to the phenomenon was found in an article by Wan and Dodge (*16*) on equilibrium in the carbon dioxide–benzene system.

In order to avoid this difficulty a complete new procedure was adopted for sampling the liquid phase. This consisted of inserting a four-port ball valve in a small line connecting the vapor space with the liquid space. The liquid level in the cell at equilibrium conditions was raised using a double acting Ruska pump to move the upper and lower pistons simultaneously at constant pressure and temperature until the liquid flooded one through-port in the ball valve. The valve was then rotated 90°, whereupon the filled port came in line with hot circulating helium. The helium was circulated until all the liquid sample was vaporized and then the flow was switched to the chromatographic sample valve for analysis. This procedure worked well and gave consistent results.

The second problem in obtaining consistent phase analyses was also related to the low volatility of *n*-heptane. In previous studies on binary systems, the heavier of the two components was always sufficiently volatile so that the samples of vapor or liquid could be maintained in the gaseous state in a simple electrical tape-heated manifold operating at pressures of 150 mm of mercury or less. However, in this study, much more elaborate precautions had to be taken to prevent partial condensation of the heptane during sampling. This was achieved by using an electrically heated and thermostated manifold enclosed in an insulated housing. The manifold was provided with a stainless steel bellows type diaphragm pump which could circulate a helium stream serving as a carrier for the sample drawn from the cell. The circulation process served as a means for homogenizing the sample which could be injected into the gas chromatograph through a GC gas sampling valve located in the circulation network of lines.

The experimental method used for studying the behavior of this system at 399.3 °F was essentially the same as that recently described in detail by Ng and Robinson (*10*). The equilibrium cell had a constant volume of about 150 mL. Temperature was controlled using pencil-type electrical elements inserted into an aluminum shroud surrounding the cell. The liquid sampling mechanism was fashioned after that developed by Fredenslund et al. (*8*). One bull's-eye observation port was located in the lower third of the cell, but no provision was made for making refractive index measurements.

The temperature of the cell contents in both equilibrium cells was measured with a calibrated iron–constantan thermocouple and is believed known to within ±0.1 °F. The pressure was

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measured using two calibrated Heise gauges having ranges of 0–2000 and 0–3000 psi. Pressures are believed known to within about 0.1% of the full range pressure.

The refractive index of either phase in the variable volume cell was determined using a special window and mirror arrangement and an autocollimating telescope. Light was provided by a helium-neon laser at a wavelength of 6328 Å. Details of this method have been given by Besserer and Robinson (*2*).

Materials and Analyses

The carbon dioxide used in this work was provided by Linde and it had a purity of 99.9+ mol %. The *n*-heptane was supplied by Aldrich Chemicals at a stated purity of 99+ mol % but a chromatographic scan indicated it to be better than 99.9+% pure. Both materials were used without further purification.

The samples of liquid and vapor from the variable volume cell were analyzed using a Hewlett-Packard Model 700 chromatograph with a thermal conductivity detector. The detector was maintained at a temperature of 200 °C with a filament current of 150 mA. A 3-ft column $^1/_8$ in. in diameter packed with Porapak QS and held at 150 °C was used for the separation.

The samples of liquid and vapor from the constant volume cell were analyzed using a Hewlett-Packard 5830A gas chromatograph supplied with a 18850A GC terminal.

The chromatographs were calibrated using pure components only. The response was found to be linear at sample pressures up to about 200 mm of mercury absolute pressure. The compositions are believed known to within ±0.005 mole fraction over the entire concentration range.

Operational Procedure

At the start of a run, the equilibrium cell was evacuated and a sufficient amount of *n*-heptane was added to provide a visible liquid layer. Following this, the carbon dioxide was added until a sufficient pressure was reached for the first equilibrium condition at the given temperature. Equilibrium was obtained in the variable volume cell by moving the pistons up and down and in the constant volume cell by rotating the externally coupled magnet. Equilibrium was judged to have been obtained when the pressure and the refractive index line had stabilized.

Adjustments in composition were necessary at each new pressure condition. A certain amount of trial-and-error was required to maintain the two-phase condition near the critical region.

During the process of mixing the liquid and vapor phases to attain equilibrium in the variable volume cell, some liquid drops frequently lodged themselves in the vapor port cavity in the cell body. This was removed by flushing out the needle valve with two or three vapor samples. This accounts for the slight drop in pressure between liquid and vapor samples in the analyses reported.

Results

The experimentally measured equilibrium phase compositions and refractive indices, together with the calculated molar volumes and equilibrium ratios are given in Table I. Isothermal pressure–composition diagrams at 99.5, 175, 250, and 399.3 °F are shown graphically in Figure 1. The calculated equilibrium ratios are shown at each temperature in Figure 2. Whenever a change in pressure occurred between liquid and vapor samples, interpolated values of the composition of the other phase were determined so that the equilibrium ratios could be calculated at constant pressure in both phases. Any error resulting from these interpolations is considered to be negligible.

The critical locus as defined by the pure component criticals and the four experimental points is shown graphically in Figure 3 and the numerical values are tabulated in Table II.

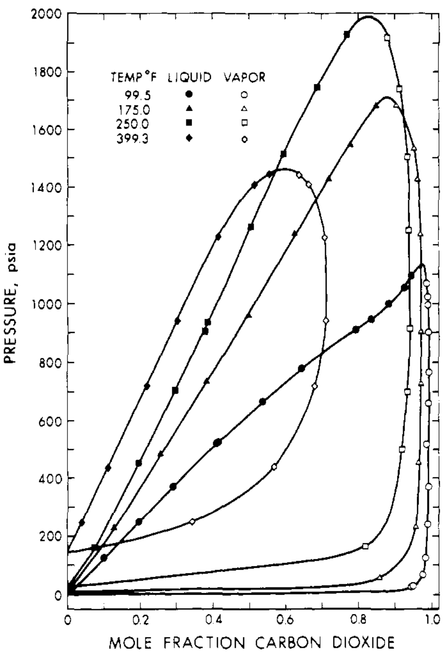

Figure 1. Equilibrium phase compositions for the carbon dioxide–*n*-heptane binary system.

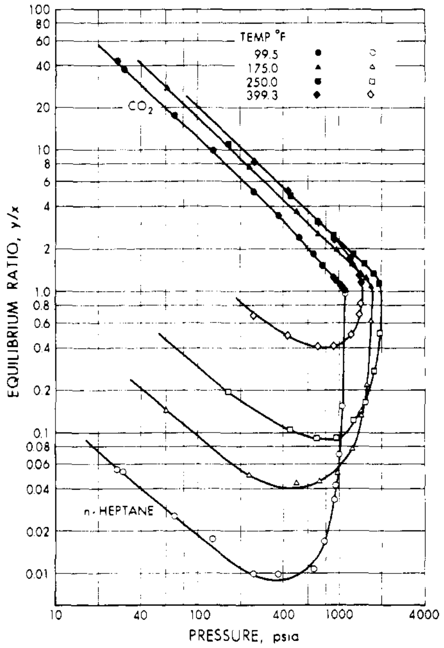

Figure 2. Equilibrium ratios for carbon dioxide and *n*-heptane in the carbon dioxide–*n*-heptane binary system.

The molar volumes of each phase at the three lower temperatures were calculated from the measured equilibrium phase compositions and refractive indices using the Lorentz–Lorenz molar refractivity relationship as explained in earlier papers by Besserer and Robinson (*2*, *4*). In making these calculations it was assumed that any possible nonadditive terms for the density of carbon dioxide–*n*-heptane mixtures could be neglected without introducing any appreciable error. The justification for this has been explained by Smyth et al. (*15*).

To calculate the refractivity of the pure components, experimental refractive index measurements were made on *n*-heptane at 100, 175, and 250 °F at pressures up to 3000 psia. These data are presented in Table III. Data obtained earlier (*3*) were used for carbon dioxide at 100, 175, and 250 °F. These data, together with densities calculated for carbon dioxide

Table I. Equilibrium Phase Properties of the Carbon Dioxide-*n*-Heptane System

pressure, psia	carbon dioxide concn[d]		refractive index[a]		molar volume[b]		equilibrium ratios	
	x	y	n_L	n_V	V_L	V_V	K_{CO_2}	K_{nC_7}
99.5 °F								
27.0	0.022$^+$	0.946	1.3760	1.0011	2.30	177	43.0	0.055
30.5	0.025$^+$	0.949	1.3759	1.0012	2.30	161	38.0	0.052
68.5	0.055$^+$	0.976	1.3741	1.0024	2.25	73.1	17.8	0.025
128	0.100	0.984	1.3710	1.0040	2.18	42.6	9.84	0.018
242	0.188$^+$	0.991	1.3639	1.0072	2.05	23.0	5.27	0.011
250	0.195	0.992	1.3634	1.0075	2.04	22.0	5.09	0.010
373	0.290	0.993	1.3549	1.0117	1.90	14.1	3.42	0.010
520	0.408	0.989	1.3417	1.0177	1.72	9.46	2.42	0.019
526	0.414	0.993$^+$	1.3410	1.0180	1.71	9.16	2.40	0.012
662	0.532$^+$	0.992	1.3244	1.0242	1.54	6.85	1.64	0.015
664	0.536	0.995$^+$	1.3239	1.0243	1.53	6.74	1.86	0.011
768	0.635$^+$	0.994	1.3056	1.0300	1.39	5.48	1.57	0.016
778	0.645	0.994$^+$	1.3036	1.0307	1.38	5.36	1.54	0.017
904	0.784$^+$	0.992	1.2657	1.0401	1.20	4.14	1.27	0.037
912	0.793	0.993$^+$	1.2628	1.0410	1.19	4.04	1.25	0.034
948	0.835	0.993$^+$	1.2483	1.0443	1.13	3.74	1.19	0.042
997	0.884$^+$	0.992	1.2261	1.0499	1.09	3.34	1.12	0.067
1002	0.885	0.992$^+$	1.2237	1.0505	1.10	3.30	1.12	0.070
1052	0.926$^+$	0.991	1.1982	1.0531	1.09	3.15	1.07	0.122
1054	0.929	0.989$^+$	1.1978	1.0587	1.08	2.87	1.06	0.155
1070	0.935$^+$	0.987	1.1887	1.0619	1.11	2.75	1.06	0.200
1095	0.947$^+$	0.954	1.1747	1.0680	1.15	2.82	1.01	0.868
1097	0.949	0.950	1.1739	1.0685	1.15	2.84	1.00	0.980
175.0 °F								
61.5	0.031	0.860	1.3533	1.0023	2.45	110	27.7	0.145
230	0.126	0.956	1.3463	1.0070	2.30	27	7.59	0.050
233	0.127	0.957$^+$	1.3461	1.0071	2.30	26.5	7.54	0.049
455	0.240	0.966	1.3359	1.0135	2.12	13.5	4.03	0.048
484	0.256	0.968$^+$	1.3344	1.0144	2.10	12.6	3.78	0.043
498	0.263	0.968$^+$	1.3337	1.0148	2.09	12.3	3.68	0.044
727	0.379	0.974	1.3023	1.0227	2.01	7.82	2.57	0.042
734	0.381	0.972	1.3200	1.0230	1.91	7.78	2.55	0.045
956	0.490	0.972	1.3040	1.0322	1.75	5.57	1.98	0.055
960	0.498	0.974	1.3037	1.0325	1.73	5.47	1.96	0.052
1239	0.624	0.972	1.2760	1.0438	1.57	4.10	1.56	0.075
1244	0.625	0.971	1.2756	1.0490	1.57	3.68	1.55	0.077
1426	0.715	0.963	1.2505	1.0631	1.47	2.95	1.35	0.130
1428	0.719	0.963	1.2500	1.0633	1.46	2.94	1.34	0.132
1537	0.769	0.954	1.2296	1.0781	1.43	2.47	1.24	0.199
1545	0.775	0.952	1.2276	1.0799	1.42	2.43	1.23	0.213
1684	0.847	0.905	1.1753	1.1290	1.54	1.77	1.07	0.621
250.0 °F								
164	0.073$^+$	0.819	1.3266	1.0066	2.55	42.4	11.2	0.195
164	0.074	0.819$^+$	1.3263	1.0066	2.55	42.4	11.1	0.196
450	0.193$^+$	0.921	1.3159	1.0141	2.36	15.1	4.77	0.098
454	0.195	0.915$^+$	1.3157	1.0142	2.36	15.3	4.69	0.106
701	0.296$^+$	0.936	1.3063	1.0216	2.19	9.40	3.16	0.091
705	0.296	0.936$^+$	1.3050	1.0217	2.20	9.36	3.16	0.091
837	0.345	0.939[c]					2.72	0.093
906	0.376	0.942	1.2950	1.0289	2.08	6.90	2.51	0.093
917	0.378$^+$	0.942	1.2945	1.0291	2.08	6.85	2.49	0.093
935	0.384	0.941$^+$	1.2936	1.0299	2.07	6.69	2.45	0.096
1250	0.500$^+$	0.939	1.2739	1.0434	1.91	4.65	1.88	0.122
1261	0.503	0.939$^+$	1.2731	1.0440	1.91	4.59	1.87	0.123
1505	0.589$^+$	0.934	1.2532	1.0589	1.81	3.50	1.59	0.161
1514	0.594	0.934$^+$	1.2523	1.0595	1.80	3.46	1.57	0.163
1746	0.686	0.914	1.2274	1.0798	1.70	2.76	1.33	0.274
1920	0.761$^+$	0.878	1.1917	1.1135	1.73	2.17	1.15	0.511
1931	0.768	0.882$^+$	1.1881	1.1170	1.74	2.08	1.15	0.509
399.3 °F								
254	0.042	0.344					8.23	0.684
440	0.113	0.566					5.03	0.489
720	0.219	0.681					3.11	0.408
940	0.303	0.113					2.35	0.412
1228	0.416	0.768					1.70	0.501
1408	0.512	0.662					1.29	0.693
1439	0.555	0.638					1.15	0.813

[a] Relative to vacuum at 6328 Å. [b] Calculated from refractive index and composition data cm^3/(g-mol). [c] Check point obtained on constant volume cell. [d] (+) Interpolated isobarically from the other equilibrium phase.

Table II. Critical Locus Data for the Carbon Dioxide-n-Heptane Binary System

temp, °F	pressure, psia
99.5	1130
175.0	1710
250.0	1988
399.3	1456

Table III. Refractive Index Data for n-Heptane Liquid Relative to Vacuum at 6328 Å

100 °F		175 °F		250 °F	
pressure, psia	refractive index	pressure, psia	refractive index	pressure, psia	refractive index
246	1.3788	222	1.3567	220	1.3327
423	1.3796	410	1.3578	426	1.3345
624	1.3804	622	1.3591	622	1.3361
816	1.3812	826	1.3602	824	1.3378
1034	1.3822	1022	1.3613	1032	1.3394
1516	1.3841	1532	1.3640	1547	1.3430
2008	1.3860	2030	1.3665	2018	1.3461
2496	1.3878	2510	1.3688	2504	1.3491

Table IV. Molar Refractivity Values at Zero Pressure for Carbon Dioxide and n-Heptane

	$R_{LL}{}^0$, cm³/(g-mol)	
temp, °F	CO_2	nC_7
100	6.65	33.60
175	6.65	34.01
250	6.65	34.20

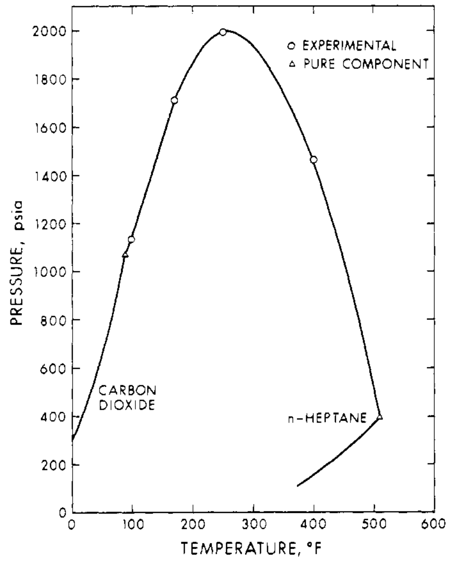

Figure 3. Critical locus for the carbon dioxide-n-heptane binary system.

and n-heptane using the Benedict-Webb-Rubin (*1*) equation of state were used to calculate the molar refractivity at each of the three temperatures according to the Lorentz-Lorenz relationship:

$$R_{LL} = \frac{n^2 - 1}{n^2 + 2} \frac{M}{\rho}$$

The values of $R_{LL}{}^0$, the refractivity at zero pressure, were obtained by fitting the values of R_{LL} with pressure at each temperature. The resulting values are reported in Table IV.

The molal volume of each equilibrium phase was calculated using the measured experimental phase refractive indices together with the molal average refractivity based on the measured phase compositions and molal refractivities of the pure materials at the equilibrium pressure and temperature. The results of these calculations are reported in Table I.

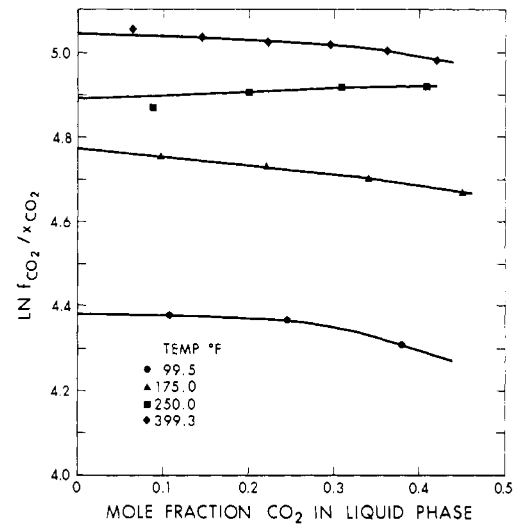

Figure 4. Graphical determination of Henry's constants for carbon dioxide in the carbon dioxide-n-heptane binary system.

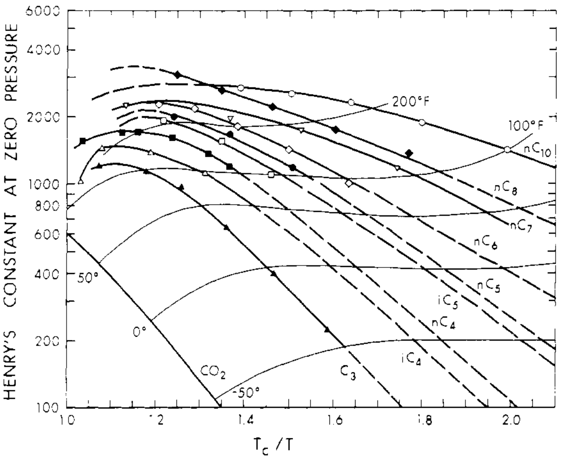

Figure 5. Henry's constants for carbon dioxide in paraffin hydrocarbons.

Table V. Henry's Constants at Zero Pressure for Carbon Dioxide in n-Heptane

temp, °F	Henry's constant, psia
99.5	1167
175.0	1719
250.0	1944
399.3	2279

Discussion

It is frequently of interest to know the Henry constant for the supercritical components when dealing with vapor-liquid equilibrium calculations for multicomponent mixtures. Furthermore, the Henry constants evaluated from experimental data are often a good indicator of the reliability of the data. In the system under investigation in this work, carbon dioxide is supercirtical at all temperatures studied.

Henry's constants for carbon dioxide in n-heptane were evaluated from the experimental data using the method and program described by Christiansen and Fredenslund (*7*). A plot of the carbon dioxide vapor-phase fugacity divided by the liquid-phase concentration at the collocation points as a function of liquid-phase concentration is shown in Figure 4. A Poynting correction was applied to the values at zero concentration to obtain the Henry constants at zero pressure for each temperature. These are presented in Table V and are illustrated in the composite graph given in Figure 5 which includes Henry's constants for carbon dioxide in other n-paraffin hydrocarbons.

These values were all calculated using similar methods. The sources of information for the other carbon dioxide–paraffin systems were as follows: carbon dioxide–propane, Poettman and Katz (*12*); carbon dioxide–isobutane, Besserer and Robinson (*4*); carbon dioxide–*n*-butane Olds et al. (*11*); carbon dioxide–isopentane, Besserer and Robinson (*6*); carbon dioxide–*n*-pentane, Besserer and Robinson (*5*); carbon dioxide–*n*-hexane, Gupta and Friesen (*9*); carbon dioxide–*n*-octane, Schneider et al. (*14*); carbon dioxide–*n*-decane, Reamer and Sage (*13*).

It will be noted from this figure that the data on the Henry constants for carbon dioxide in most of the paraffin hydrocarbons follows a consistent pattern. A possible exception is in the case of carbon dioxide in *n*-octane where the low-temperature data lie outside the curve for *n*-decane. The value obtained in this work for carbon dioxide in *n*-heptane at 250 °F appears a bit low.

The experimental data were subjected to a consistency test using the method of Christiansen and Fredenslund (*7*). Of the 63 points used in the test, 53 met the criterion that $|Y_{\text{calcd}} - Y_{\text{exptl}}| \leq 0.010$. At 399.3 °F, only two of the seven data points met the criterion, probably because of the inability of the equation of state to properly describe the fugacity of both phases so near the critical region.

Glossary

K_{CO_2}	equilibrium ratio for carbon dioxide
K_{nC_7}	equilibrium ratio for normal heptane
M	molecular weight
n	refractive index
n_L	refractive index of liquid
n_V	refractive index of vapor
P	pressure
R_{LL}	molal refractivity as defined by the Lorentz–Lorenz, relationship
$R_{LL}{}^0$	molal refractivity at zero pressure
V_L	liquid molal volume
V_V	vapor molal volume
x	mole fraction of component in the liquid phase
y	mole fraction of component in the vapor phase
ρ	density

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Equilibrium Phase Properties of the Nitrogen–Isobutane System

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The compositions of the equilibrium vapor and liquid phases have been experimentally determined for the nitrogen–isobutane binary system at temperatures of 0, 50.1, 99.9, 150.0, 199.7, and 250.0 °F and at pressures up to the critical point for the system or to about 3000 psia. Refractive index measurements for each of the equilibrium phases were also made at temperatures of 0, 50.1, and 99.9 °F. The experimental data were used to calculate the equilibrium phase molar volumes at these conditions and to calculate the equilibrium ratio for nitrogen and isobutane in the binary system. The Henry constant for nitrogen in isobutane was calculated at each temperature.

Introduction

Nitrogen is commonly found in natural gases and other reservoir fluids and consequently its presence must be considered whenever the thermodynamic properties or phase behaviors of these fluids are being evaluated. Most modern equations of state or generalized correlation procedures make use of binary interaction parameters between unlike molecules such as nitrogen and paraffin hydrocarbons. The phase behavior of many of the nitrogen–hydrocarbon binaries has been determined and consequently the necessary binary interaction parameters and Henry's constants are known. However, no data have been found in the literature on the behavior of the nitrogen–isobutane binary system. For this reason it was considered worthwhile to study the equilibrium phase properties of this system at temperatures in the 0–250 °F range at pressures up to about 3000 psia.

Experimental Method

The experimental methods used in this work were similar to those used for the studies on nitrogen in normal pentane and nitrogen in isopentane recently reported by Kalra et al. (*8*) and Krishnan et al. (*10*). The work at 50.1 and 99.9 °F was carried out in a double piston-type variable volume cell which permitted the removal of both equilibrium liquid and vapor samples and also the direct measurement of the refractive index of each phase. The working pressure of the cell was 3000 psia and the working volume ranged from about 10 to 175 mL. This equipment and the method of operation have been described in detail by Besserer and Robinson (*2*, *3*).

The work at 0 °F was carried out in a double-windowed constant-volume cell which also permitted the removal of equilibrium vapor and liquid samples and the measurement of the refractive index of each phase. Equilibrium was attained by agitating with an externally coupled Teflon-coated magnet. The working pressure of this cell was about 4000 psia and the

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