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Phase Equilibria in Hydrocarbon Systems.

Volumetric and Phase Behavior of the *n*-Decane-CO₂ System

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Experimental measurements of the volumetric and phase behavior of this binary system are reported at pressures up to 10,000 pounds per square inch in the temperature interval between 40° and 460° F.

THE CONTINUED INTEREST of the production division of the petroleum industry in the use of CO₂ as at least one component of miscible displacement of hydrocarbon fluids within underground reservoirs makes a specific understanding of the volumetric and phase behavior of binary systems involving CO₂ and aliphatic hydrocarbons of importance. The volumetric behavior of the methane-CO₂ system has been studied at pressures up to 10,000 p.s.i.a. in the temperature interval between 100° and 460° F. (16). The partial volumetric behavior of the components of this system has also been established (12). Likewise the volumetric behavior of the ethane-CO₂ system has been investigated at pressures up to 10,000 p.s.i.a. at temperatures between 100° and 460° F. (17). The two foregoing studies did not involve any phase behavior investigations. More intensive studies of the propane-CO₂ (20) and *n*-butane-CO₂ (11) systems have been made. These investigations were carried out in both the homogeneous and heterogeneous regions at pressures up to 10,000 p.s.i.a. in the temperature interval between 40° and 460° F. in the case of the propane-CO₂ system and from 100° to 460° F. for the *n*-butane-CO₂ system. Throughout the above ranges of pressure and temperature only two phases have made their appearance. The volumetric behavior and the vapor pressure of CO₂ have been established by Michels and Michels (10) and Meyer and Van Dusen (8), respectively. Likewise, the volumetric behavior of CO₂ near the critical point has been studied in some detail by Michels, Blaisse, and Michels (9). The volumetric behavior at the higher pressures and temperatures has also been investigated (4). Earlier studies of binary systems

of paraffin hydrocarbons and CO₂ were carried out by Kuenen (5, 6, 7), while the work of Poettman and Katz (13) confirmed the general behavior found by Kuenen. Poettmann and Katz (13) also reviewed to some extent the literature relating to binary mixtures of CO₂ and hydrocarbons.

There has been only limited work on systems involving CO₂ and pure hydrocarbons of higher molecular weight. For this reason, a study has been completed upon the volumetric and phase behavior of the *n*-decane-CO₂ system at temperatures from 40° to 460° F. and at pressures up to 10,000 p.s.i.a. throughout the entire range of composition.

APPARATUS AND PROCEDURE

The apparatus and techniques employed in this investigation are similar to those used in an earlier study (21). Procedure consisted of introducing known weights of *n*-decane and CO₂ into a stainless steel container, the effective volume of which was varied by the introduction or withdrawal of mercury. The temperature of the stainless steel container was controlled by immersion in an agitated oil bath.

The temperature of measurement was related to the international platinum scale by use of a strain-free, platinum resistance thermometer. The latter instrument has been compared with the indications of a similar device calibrated by the National Bureau of Standards. Appropriate corrections for the influence of changes in room temperature upon the resistance of the coils of the Mueller bridge used in these measurements were made. Experience

over a period of several decades indicates that the temperature of the stainless steel container was known within 0.1° F. relative to the international platinum scale throughout the temperature interval between 40° and 460° F. Throughout the greater part of this interval it is believed that the temperature of measurement was related to the international temperature scale within 0.05° F. Pressure was determined by means of a balance whose construction has been described in some detail (21). This instrument was calibrated at least annually against the vapor pressure of samples of CO₂ at the ice point. The variation in calibration of this balance over a 20-year period has been less than 0.05% (19). From this experience it appears that the pressures were established within 0.01% or 0.2 p.s.i., throughout the entire range of this investigation. The quantity of mercury introduced or withdrawn from the stainless steel vessel was determined from the changes in elevation of an air-mercury interface in a connected auxiliary stainless steel vessel which was maintained at a constant temperature near that of the room. Experience indicates that the volume occupied by the mixture under investigation was established with an uncertainty of not more than 0.15% except for a few states in the liquid phase. In the latter instances the uncertainty may have been as large as 0.3%. The weight of CO₂ introduced was established by gravimetric methods. The quantity of *n*-decane was determined from volumetric measurements of the quantity introduced into the above-described stainless steel vessel before the CO₂ was added. It is believed that the weight of the *n*-decane was established within 0.1% while the weight of CO₂ introduced was determined within 0.02%.

The pressures corresponding to bubble point for each of the experimentally studied mixtures were determined from discontinuities in the isothermal, volume-pressure relationships which were associated with bubble point. To establish the composition of the dew-point gas, samples of this coexisting phase were withdrawn under nearly isothermal, isobaric conditions. Near the critical states samples of the liquid phase were also withdrawn. Relative quantities of *n*-decane and CO₂ in the samples were determined by a partial condensation procedure (14) carried out under such conditions that the relative volatility of the *n*-decane and the CO₂ differed by more than a factor of 100. It is believed that the composition of the mixtures employed

was established within 0.0005 mole fraction CO₂ at each of the states investigated. The withdrawal of the samples in the heterogeneous region was accomplished with less than 0.5 p.s.i. change in pressure and 0.02° F. change in temperature. It should be recognized that this withdrawal is carried out without agitation and it is not believed that such small variations in pressure reflected a change in the composition from that established at the initial equilibrium.

MATERIALS

The CO₂ utilized in this study was prepared by the thermal decomposition of purified sodium bicarbonate. The gas evolved was dried by passage over calcium chloride and anhydrous calcium sulfate at a pressure of 1 atmosphere and then sublimed at liquid nitrogen temperature. The final product was collected in a steel container such as has been described (21). The vapor pressure agreed with that established by Bridgeman (2) within 0.03%. On the basis of the method of preparation and the measured value of the vapor pressure it is believed that the CO₂ employed contained less than 0.0005 mole fraction of impurity.

The *n*-decane was obtained as research grade from the Phillips Petroleum Co. with a reported purity of 0.9934 mole fraction *n*-decane. The hydrocarbon was dried over metallic sodium and evacuated to a pressure of 0.0001 inch of mercury at liquid nitrogen temperature. The specific weight of substantially air-free material was 45.3394 pounds per cubic foot at 77° F. as compared to a value of 45.337 reported (1) for an air-saturated sample at the same temperature. The index of refraction relative to the D-lines of sodium at 77° F. was 1.4097 as compared to a value of 1.40967 reported (1) for an air-saturated sample. Experience with *n*-decane in other investigations indicates that the sample employed for this study probably did not contain more than 0.0066 mole fraction of materials other than *n*-decane and that these impurities were other compounds containing ten carbon atoms per molecule.

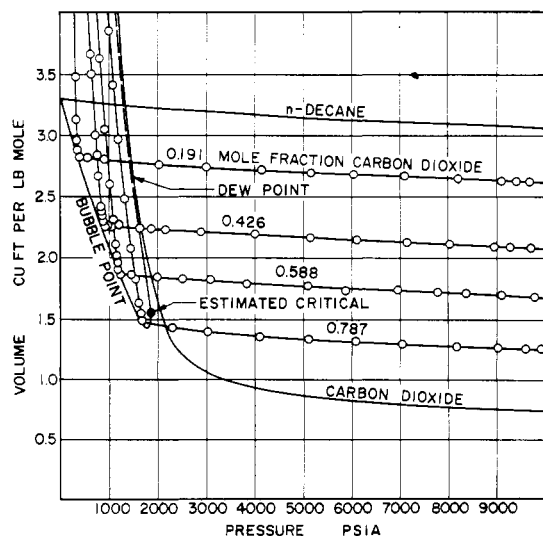


Figure 1. Volumetric measurements at 160° F.

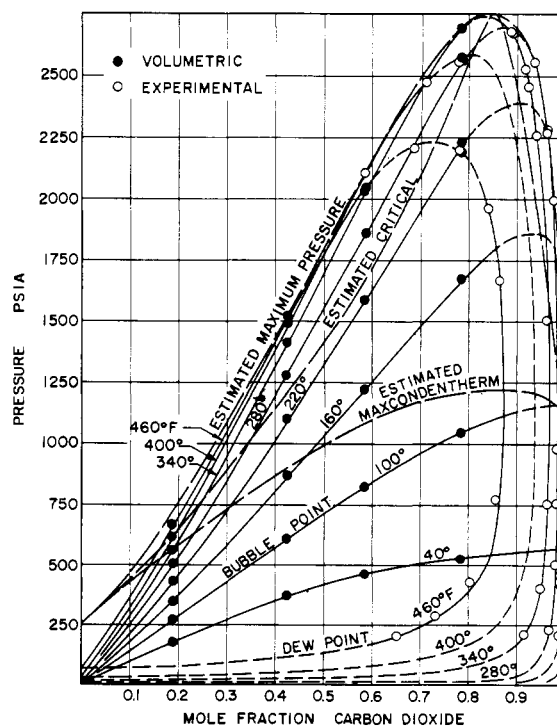


Figure 2. Pressure-composition diagram

Table I. Properties of Coexisting Gas and Liquid Phases

Pressure P.S.I.A.	Dew Point		Bubble Point		Equilibrium Ratio	
	Mole Fraction, CO ₂	Volume cu. ft./lb. Mole	Mole Fraction, CO ₂	Volume cu. ft./lb. Mole	CO ₂	n-Decane ^a
			40° F.			
0.004 ^b	0.0000	...	0.0000	3.055	...	1.000
50	0.9998	...	0.0545	2.934	18.34	0.000150
100	0.9999	...	0.1086	2.811	9.210	0.000112
150	0.9999	...	0.1627	2.688	6.146	0.000119
200	0.9999	24 ^c	0.2178	2.561	4.590	0.000128
250	0.9999	18.6	0.2747	2.430	3.640	0.000138
300	0.9999	15.04	0.3355	2.289	2.980	0.000150
350	0.9999	12.46	0.4046	2.130	2.471	0.000168
400	0.9999	10.52	0.4765	1.962	2.098	0.000191
450	0.9999	8.97	0.5685	1.749	1.758	0.000232
500	0.9999	7.71	0.7117	1.421	1.404	0.000347
550	0.9999	6.61	0.9135	0.970	1.095	0.00116
566.5 ^d	1.0000	0.781	1.0000	0.781	1.000	1.00000
			100° F.			
0.073 ^b	0.0000	°	0.0000	3.166	...	1.00000
100	0.9986	...	0.0730	2.994	13.68	0.00151
200	0.9990	...	0.1437	2.825	6.950	0.00117
300	0.9991	...	0.2129	2.662	4.693	0.00114
400	0.9992	13.°	0.2816	2.499	3.548	0.00111
500	0.9993	9.48	0.3513	2.335	2.845	0.00111
600	0.9993	7.37	0.4220	2.165	2.368	0.00121
700	0.9993	5.82	0.4950	1.991	2.019	0.00139
800	0.9993	4.60	0.5712	1.811	1.750	0.00163
900	0.9993	3.58	0.6510	1.633	1.534	0.00201
1000	0.9993	2.748	0.7430	1.446	1.345	0.00272
1100	0.9992	2.004	0.8641	1.253	1.156	0.00589
1160 ^f	0.9946	1.506	0.9946	1.506	1.000	1.00000
1082 ^g	0.9993
			160° F.			
0.40 ^b	0.0000	°	0.0000	3.302	...	1.0000
200	0.9961	...	0.1118	3.017	8.910	0.00439
400	0.9972	...	0.2127	2.763	4.688	0.00356
600	0.9975	9.5 ^c	0.3077	2.534	3.242	0.00361
800	0.9976	6.65	0.3988	2.316	2.501	0.00399
1000	0.9976	4.91	0.4876	2.101	2.046	0.00468
1250	0.9972	3.50	0.6000	1.834	1.662	0.00700
1500	0.9942	2.532	0.7132	1.599	1.394	0.0202
1750	0.9822	1.834	0.8295	1.448	1.184	0.104
1860 ^f	0.9477	1.537	0.9477	1.537	1.000	1.000
1098 ^g	0.9976
			220° F.			
1.59 ^b	0.0000	°	0.0000	3.443	...	1.0000
200	0.9892	...	0.0931	3.187	10.62	0.0119
400	0.9926	...	0.1774	2.972	5.595	0.00900
600	0.9933	11 ^c	0.2549	2.781	3.897	0.00899
800	0.9938	7.94	0.3273	2.605	3.036	0.00922
1000	0.9938	6.08	0.3964	2.437	2.507	0.0103
1250	0.9931	4.59	0.4798	2.234	2.070	0.0133
1500	0.9912	3.60	0.5602	2.045	1.769	0.0200
1750	0.9877	2.886	0.6394	1.864	1.545	0.0341
2000	0.9789	2.354	0.7173	1.712	1.365	0.0746
2250	0.9640	1.937	0.7968	1.605	1.210	0.177
2392 ^f	0.9046	1.685	0.9046	1.685	1.000	1.000
1120 ^g	0.9938
			280° F.			
5.08 ^b	0.0000	°	0.0000	3.585	...	1.0000
200	0.9677	...	0.0796	3.373	12.16	0.0351
400	0.9797	...	0.1548	3.174	6.330	0.0240
600	0.9830	...	0.2240	2.996	4.388	0.0219
800	0.9848	9.0 ^c	0.2879	2.836	3.421	0.0214
1000	0.9850	6.98	0.3476	2.689	2.834	0.0230
1250	0.9849	5.37	0.4183	2.514	2.354	0.0260
1500	0.9831	4.30	0.4878	2.347	2.015	0.0330
1750	0.9804	3.53	0.5576	2.184	1.758	0.0443
2000	0.9748	2.959	0.6277	2.029	1.553	0.0677
2250	0.9633	2.511	0.6954	1.891	1.385	0.120
2500	0.9436	2.152	0.7635	1.803	1.236	0.238
2692 ^f	0.8705	1.865	0.8705	1.865	1.000	1.000
1145 ^g	0.9850

(Continued on page 511)

Table I. Properties of Coexisting Gas and Liquid Phases

Pressure P.S.I.A.	Dew Point		Bubble Point		Equilibrium Ratio	
	Mole Fraction, CO ₂	Volume cu. ft./lb. Mole	Mole Fraction, CO ₂	Volume cu. ft./lb. Mole	CO ₂	<i>n</i> -Decane ^a
			340° F.			
13.49 ^b	0.0000	...	0.0000	3.742	...	1.0000
200	0.9182	...	0.0683	3.569	13.44	0.0878
400	0.9517	...	0.1372	3.391	6.938	0.0560
600	0.9617	...	0.2018	3.223	4.765	0.0480
800	0.9657	9.8 ^c	0.2623	3.065	3.681	0.0465
1000	0.9672	7.66	0.3195	2.919	3.027	0.0482
1250	0.9674	5.96	0.3865	2.753	2.503	0.0531
1500	0.9652	4.82	0.4510	2.596	2.140	0.0634
1750	0.9614	4.01	0.5156	2.445	1.865	0.0797
2000	0.9548	3.39	0.5795	2.306	1.648	0.107
2250	0.9427	2.910	0.6461	2.181	1.459	0.162
2500	0.9220	2.520	0.7170	2.092	1.286	0.276
2732 ^d	0.8430	2.139	0.8430	2.139	1.000	1.000
1174 ^e	0.9675
			400° F.			
31.19 ^b	0.0000	...	0.0000	3.962	...	1.000
200	0.8231	...	0.0578	3.804	14.24	0.188
400	0.9017	...	0.1238	3.631	7.282	0.112
600	0.9255	...	0.1861	3.466	4.972	0.0916
800	0.9346	...	0.2445	3.319	3.822	0.0866
1000	0.9377	8.2 ^c	0.2993	3.183	3.133	0.0889
1250	0.9379	6.46	0.3650	3.022	2.570	0.0978
1500	0.9353	5.26	0.4304	2.867	2.173	0.114
1750	0.9292	4.38	0.4952	2.720	1.877	0.140
2000	0.9177	3.72	0.5605	2.586	1.638	0.187
2250	0.8978	3.22	0.6287	2.477	1.428	0.275
2500	0.8545	2.764	0.7178	2.415	1.190	0.516
2587 ^d	0.8050	2.544	0.8050	2.544	1.000	1.000
1203 ^e	0.9380
			460° F.			
64.72 ^b	0.0000	...	0.0000	4.229	...	1.000
200	0.6548	...	0.0450	4.103	14.55	0.361
400	0.8116	...	0.1104	3.922	7.350	0.212
600	0.8578	...	0.1730	3.755	4.958	0.172
800	0.8684	...	0.2308	3.609	3.762	0.171
1000	0.8724	8.7 ^c	0.2868	3.475	3.042	0.179
1250	0.8739	6.85	0.3550	3.322	2.462	0.196
1500	0.8706	5.58	0.4206	3.178	2.070	0.223
1750	0.8608	4.66	0.4880	3.033	1.764	0.272
2000	0.8347	3.93	0.5717	2.939	1.460	0.386
2224 ^d	0.7280	3.17	0.7280	3.17	1.000	1.000
1220 ^e	0.8740

^a Due to number of significant figures reported for dew-point gas composition, some discrepancies in values of the equilibrium ratio may exist at this temperature. ^b Vapor pressure of *n*-decane. ^c Dew-point volumes involve a somewhat larger uncertainty since they were established from volumetric measurements in the two-phase region. ^d Vapor pressure of CO₂. ^e Dew-point volumes of

n-decane expressed in cubic feet per pound-mole: at 100° F. = 82,200; at 160° F. = 16,660; at 220° F. = 4339; at 280° F. = 1447; at 340° F. = 591. Values based upon calorimetric vaporization measurements of Couch *et al.* (3). ^f Estimated critical state. ^g Estimated maxcondentherm.

Table II. Properties at the Unique States in the *n*-Decane-CO₂ System

Mole Fraction CO ₂	Pressure P.S.I.A.	Temperature ° F.	Pressure P.S.I.A.	Temperature ° F.	Pressure P.S.I.A.	Temperature ° F.
	Critical		Maxcondentherm		Maximum Pressure	
0.0	304 ^a	655 ^a				
0.1	(448) ^b	(641.5) ^b	(429) ^b	(642.0) ^b	(469) ^b	(632.25) ^b
0.2	(647)	(623.5)	(573)	(625.5)	(754)	(593.25)
0.3	(890)	(601.5)	(725)	(606.3)	(1094)	(547.5)
0.4	(1140)	(579.0)	(864)	(588.25)	1430	502.5
0.5	(1410)	(553.75)	(990)	(568.5)	1794	455.0
0.6	(1720)	(520.75)	(1098)	(545.75)	2151	407.5
0.7	2101	475.0	(1170)	(520.25)	2474	367.0
0.8	2564	405.75	1209	490.75	2712	336.0
0.9	2442	227.0	1219	442.0	2696	309.0
1.0	1069.87 ^c	87.80 ^c				

^a Critical state of *n*-decane (1). ^b Values in parentheses involve larger uncertainties than the other entries in the table. ^c Critical state of CO₂ (4).

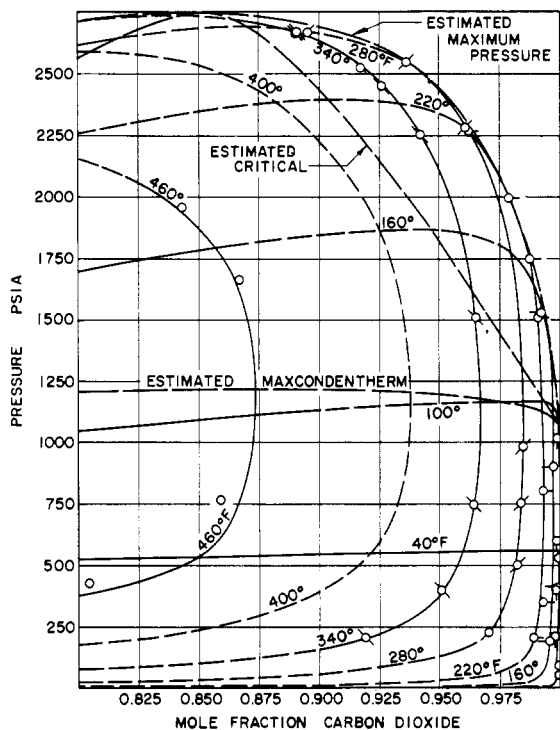


Figure 3. Experimental composition of dew-point gas

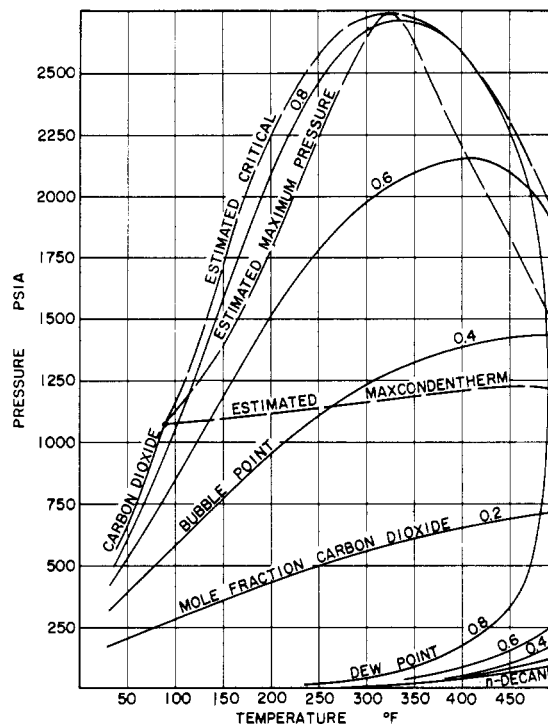


Figure 4. Pressure-temperature diagram

EXPERIMENTAL RESULTS

Four mixtures of CO_2 and n -decane were investigated. The data obtained, coupled with the available information upon the volumetric behavior of n -decane (15) and of CO_2 (4) are sufficient to establish the volumetric and phase behavior of this system. The molal volumes of CO_2 , n -decane, and the four mixtures of CO_2 and n -decane are presented in Figure 1 as a function of pressure at 160°F . The experimental points for each of the four mixtures have been included as an illustration of the density of the experimental data. The standard error of estimate of the experimental data shown on this figure was 0.006 cubic foot per pound-mole, assuming all of the uncertainty lay in the determination of the molal volume rather than in the measurement of the pressure, temperature, or composition of the mixture.

Experimental information similar to that presented in Figure 1 was obtained for temperatures between 40° and 460°F . The detailed experimental data for each temperature of measurement are available (18). The smoothed data for even values of composition, pressure, and temperature are also available (18).

Figure 2 presents all the experimental measurements employed to establish the phase behavior of the n -decane- CO_2 system. The volumetric measurements were used primarily to establish the behavior at bubble point, while the directly determined compositions were employed to determine the behavior of the dew-point gas. In order to illustrate the detail of these measurements there is shown in Figure 3 the behavior of the dew-point gas on a much enlarged composition scale, except for three measurements at 460°F . A detailed record of the experimentally determined compositions is available (18). The data are within the experimental uncertainty of measurement.

Table I records the properties of the coexisting phases taken from the smooth curves of Figures 2 and 3 for the several temperatures investigated. Included also are the

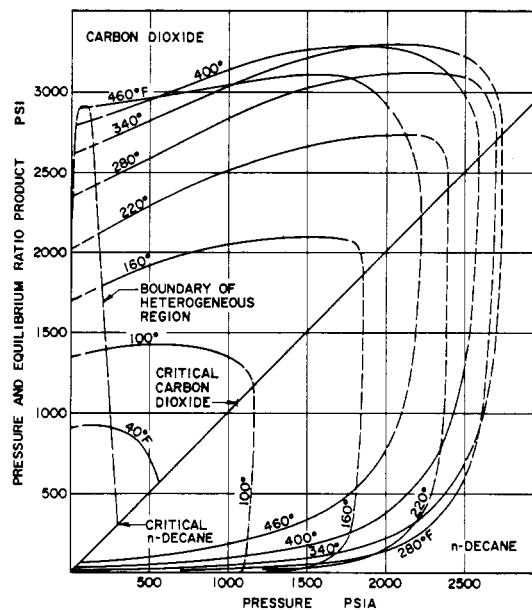


Figure 5. Equilibrium ratios for n -decane and CO_2

equilibrium ratios. The standard error of estimate of the experimentally determined composition data presented in Table I was 0.0039 mole fraction. It should be recognized that the standard error of estimate assumes that all of the uncertainty is in the experimental determination of the composition and not in the values of the prevailing pressure and temperature. There apparently is no other investigation of the phase behavior of the n -decane- CO_2 system upon which the comparison can be based.

The influence of pressure and temperature upon the boundaries of the heterogeneous region for the four experimentally investigated mixtures of n -decane and CO_2 is

shown in Figure 4. No experimental points have been included since the curves represent interpolated values obtained from Figures 2 and 3. The behavior is similar to that encountered in other binary systems containing CO₂ (11, 16, 17, 20). Some of the properties at the unique states of the *n*-decane-CO₂ system are presented as a function of composition in Table II.

In order to illustrate still further the nature of the phase behavior of this system, there is shown in Figure 5 the product of the pressure and equilibrium ratio for both *n*-decane and CO₂ as a function of pressure for eight temperatures between 40° and 460° F.

A large number of other diagrams, particularly those pertaining to the volumetric behavior of this system, could be presented. However, the behavior is similar to that encountered in other binary systems with CO₂ and a hydrocarbon as components.

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Vapor Liquid Equilibrium in the Methane-*n*-Decane-Nitrogen System

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Experimental data on the compositions of the conjugate phases are presented. These data are presented in the form of isotherms at 100, 160, 220, 280° F. At each of these isotherms equilibrium data were obtained at 1000, 2000, 3000, 4000, and 5000 p.s.i.a., with the nitrogen concentration varying from 0% to 100% in the vapor phase.

SINCE NITROGEN occurs extensively in petroleum fluids, a knowledge of its equilibrium data is of importance in the petroleum industry. *K* values for nitrogen are reported in the N.G.A.A. data book (3) up to a convergence pressure of 5000 p.s.i.a. Jacoby (5) in his review of the *K* values of an earlier edition of this book, with reference

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to the nonhydrocarbon *K* value, emphasizes the need for more experimental data on the effect of nitrogen on the hydrocarbon *K* values.

In this work the methane-*n*-decane-nitrogen system was investigated at temperatures of 100, 160, 220, and 280° F. At each of the isotherms, equilibrium data were obtained at 1000, 2000, 3000, 4000 and 5000 p.s.i.a. with the nitrogen concentration varying from 0% to 100% in the vapor phase.