Part I

PHASE EQUILIBRIA
MOLECULAR TRANSPORT
THERMODYNAMICS

Vapor-Liquid Equilibria of the Helium-Nitrogen System

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This report presents more extensive experimental data on the helium-nitrogen system, compares the results with those reported by other investigators, and presents this information in useful form for engineering calculations. A windowed, phase equilibrium apparatus was employed to obtain phase equilibria data at 96 experimental conditions for temperatures from 76.5° to 120° K. and pressures from 200 to 2000 p.s.i.a. A compilation of experimental vapor-liquid values and equilibrium constants, *K*, for helium and nitrogen are included. Average deviation of experimental composition values from smoothed values is 0.01 and 0.38 mole % for liquid and vapor phases, respectively. The data obtained in this work agree only qualitatively with the data of Gonikberg and Fastowsky (±6%); however, there is unusually good agreement with those of Kharakhorin (±1.5%). Buzyna's data were not sufficiently detailed for satisfactory comparisons.

PHASE DATA on the helium-nitrogen system at low temperatures and high pressures are limited. The earliest investigation of this system was reported by Fedoritenko and Ruhemann (*5*, *10*). Experimental phase data were obtained at temperatures of 64°, 78°, 90°, and 108° K. and pressures from 75 to 2200 p.s.i.a. by employing both static and flow methods. Tabular data were not presented.

Gonikberg and Fastowsky (*7*) experimentally studied the solubility of helium in nitrogen by employing the circulation method at pressures up to 4300 p.s.i.a. and at temperatures of 78.0°, 90.1°, and 109° K. The results reported by these investigators are questionable because of the means employed in obtaining vapor phase data.

More extensive results have been published by Kharakhorin (*8*) at temperatures from 68° to 11.5° K. and pressures up to 3300 p.s.i.a. This author presented a detailed discussion of experimental techniques employed and accuracy of the experimental data obtained. Also, comparisons were made with previous studies of helium binary systems.

Buzyna (*3*) recently studied the behavior of the helium-nitrogen system by employing the dew point-bubble point and static methods. Vapor-liquid composition values were presented for pressures of 170, 330, 500, 800, 1000 p.s.i.a. and temperatures from 77.3° to 122.8° K. Reverse-order solubility phenomena were observed in the liquid phases in various temperature ranges and at all pressures investigated.

Comparisons of the phase equilibria data obtained by the Helium Research Center with those of previous investigators are made for the common isotherm, 90° K. Accurate comparisons of the results obtained in this study with those of Fedoritenko and Ruhemann (*5*) were difficult because they did not present numerical data. The vapor-liquid data obtained in the present study deviate from those of Kharakhorin by an average of 1.5% and from those of Gonikberg and Fastowsky by an average of 6.0%.

EXPERIMENTAL

The helium used in this study had a minimum purity of at least 99.995%, as evidenced from analysis of concentrated impurity samples (*9*). The average impurities were 9.8 p.p.m. Ne, 9.9 p.p.m. N_2, 0.2 p.p.m. O_2, 0.2 p.p.m.

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H_2, and 3.7 p.p.m. H_2O. The nitrogen had a minimum purity of at least 99.9% as shown by mass spectrometer analyses. Both gases were used without further purification.

Gas cylinders were evacuated and purged with nitrogen a minimum of three times. Mixtures were then prepared with compositions of 11.5, 25.1, 37.8, 46.6, and 75.5% helium.

The essential features of the apparatus employed in this study have been described in detail by Brandt and Stroud (*2*). Basically, the apparatus consists of a windowed, phase equilibrium cell, which can be maintained at any desired pressure up to 4000 p.s.i. and at any temperature between the ambient temperature and 76.5° K.

Temperatures within the cell were controlled with a proportional temperature-controller–recorder and measured with a precision potentiometer in junction with a copper-constantan thermocouple. The thermocouple was calibrated by taking e.m.f. readings at various pressures from the normal boiling point to the critical temperature of pure nitrogen. These vapor pressure data were compared with published values (*1, 4, 11*) and are accurate within 0.2° K. Pressures were regulated by a reducing regulator and measured with a rotating deadweight gage. The temperature control was within ±0.5° K., and the pressure was within 1 p.s.i. of the reported value.

When the cell had cooled to the desired temperature, the binary mixture was set at the desired pressure and admitted to the cell. Stirring the cell contents facilitated the establishment of equilibrium conditions.

Samples of each phase were analyzed with a gas chromatograph. Prepared mixtures of helium and nitrogen covering the range of 0 to 100% helium were used to calibrate the chromatograph and were standardized by two methods. Mixtures having helium concentrations from 1 to 100% were analyzed by means of a mass spectrometer, with an accuracy of ± 0.2 mole %, and mixtures containing less than 1% helium were analyzed by a charcoal absorption method (*6*), with an accuracy of ± 0.03 mole %.

Samples were taken directly into the chromatograph with capillary tubing. An average of three analyses were made of each phase at the desired equilibrium conditions. The equilibrium chamber was emptied after each determination. Subsequent equilibrium conditions were established at the same pressure by further cooling of the cell until one isobar had been completed over the entire temperature range of this study.

Ninety-six equilibrium conditions were established and more than 500 samples analyzed. Analyses obviously in error were rejected, and the determinations were repeated.

RESULTS

Vapor-liquid equilibrium behavior of the helium-nitrogen system was investigated for temperatures from 76.5° to 120° K. in increments of five degrees and at pressures of approximately 200, 400, 600, 800, 1000, 1200, 1400, 1600, 1800, and 2000 p.s.i.a. The results of the experimental vapor-liquid observations are presented in Table I.

Isothermal and isobaric diagrams are shown in Figures 1 and 2, respectively. The liquid phase of Figure 2 is presented in Figure 3 on an expanded scale. Large-scale plots of our original data were used in cross-checking the results and for selecting the smoothed values presented in the several charts and in Table II.

Smoothed equilibrium constants, $K = y/x$, for helium and nitrogen are given in Table III. Values in the table were derived for each isotherm by computing X, the average value for the liquid, and Y, the average value for the vapor, from data presented in Table I, and plotting these values *vs.* pressure. Then values were determined at even isobars 200, 400, . . . by reading directly from the plots, and these values were used to obtain the K data given in Table III. To illustrate the effects of pressure on the equilibrium constants, experimental values of K for helium are presented graphically.

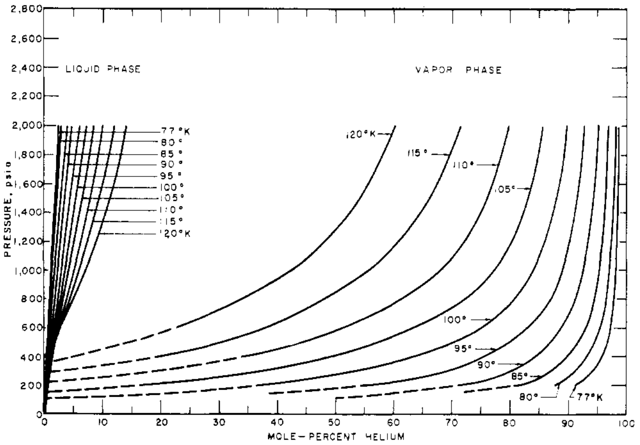

Figure 1. Pressure-composition diagram

DISCUSSION

Figure 1 is a pressure-composition diagram of both vapor and liquid phases. For the 77° and 80° K. isotherms, pressure has an almost neglible effect on composition above 600 p.s.i.a. Although the lower pressure ranges were not investigated, the isotherms in this region were closed by extrapolation of the dew point curves. The zero-helium intercepts of the dew point curves correspond to the vapor pressure of nitrogen. In the liquid phase, each lower temperature isotherm crosses all higher temperature isotherms at some pressure less than 625 p.s.i.a.

Isobars are shown in Figure 2 as functions of temperature *vs.* composition. For pressures less than the critical pressure of nitrogen (493 p.s.i.a.), the curves close at a temperature corresponding to the vapor pressure of nitrogen at the zero-helium intercept. The 200 and 400 p.s.i.a. isobars show zero intercepts at 109° and 121.4° K. respectively. For pressures between 500 and 2000 p.s.i.a., the curves wil close at temperatures less than the critical temperature of nitrogen (126.1° K.), unless retrograde condensation occurs.

A temperature-composition diagram of the liquid phase is presented in Figure 3 on an expanded scale. The 800

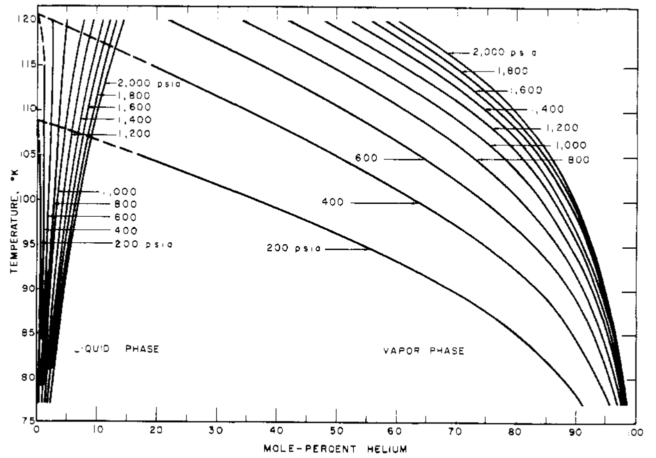

Figure 2. Temperature-composition diagram

Table I. Experimental Vapor-Liquid Equilibrium Values

Pressure P.S.I.A.	Helium, Mole %	
	Liquid	Vapor
	76.5° K.	
202	0.27	90.98
	0.26	91.13
	0.27	91.24
	0.26	91.13
	0.27	91.07
	0.26	
402	0.36	95.87
	0.32	95.58
	0.33	95.90
		95.58
601	0.60	96.97
	0.65	97.06
	0.60	97.02
808	0.93	97.59
	0.89	97.59
	0.92	97.59
1002	1.07	97.96
	1.07	97.96
	1.07	97.96
1206	1.30	98.28
	1.32	98.37
	1.31	98.32
1395	1.54	98.26
	1.52	98.35
	1.49	98.18
1603	1.72	98.42
	1.72	98.30
	1.73	
1800	1.92	98.44
	1.94	98.44
	1.92	
	1.91	
2006	2.13	98.58
	2.15	98.62
	2.12	98.58
	85° K.	
202	0.41	81.03
	0.37	80.95
	0.39	80.67
	0.39	
404	. . .	89.88
	. . .	89.69
	. . .	90.14
604	1.10	92.82
	1.02	92.81
	1.01	92.84
	0.96	
807	1.51	94.73
	1.47	94.29
	1.46	94.41
		94.43
1002	1.77	95.13
	1.71	95.00
	1.78	94.97
	1.72	
1202	2.20	95.80
	2.19	95.74
	2.30	95.80
1396	2.47	96.40
	2.50	96.32
	2.48	96.07
1610	2.90	96.24
	2.90	96.27
	80° K.	
202	0.32	88.06
	0.31	87.76
	0.31	87.91
	0.31	87.99
399	0.53	93.41
	0.46	93.41
	0.45	93.41
599	0.76	95.76
	0.75	95.85
	0.74	95.67
807	1.13	96.45
	1.14	96.55
	1.14	96.57
1001	1.36	96.92
	1.37	97.07
	1.33	97.12
	1.38	96.97
1206	1.62	97.22
	1.65	97.19
	1.63	97.24
	1.62	
1395	1.88	97.76
	1.89	97.67
	1.89	97.64
	1.89	
1603	2.16	97.72
	2.13	97.80
	2.15	97.62
1801	2.50	97.62
	2.38	97.86
2002	2.73	98.20
	2.62	98.24
	2.63	98.15
	2.64	
	90° K.	
201	0.41	69.92
	0.40	69.61
	0.42	69.34
402	0.97	84.06
	0.96	83.61
		84.17
604	1.33	87.68
	1.34	88.18
	1.32	88.11
803	1.88	91.27
	1.80	91.43
	1.80	
1000	2.26	92.32
	2.21	92.52
	2.26	92.13
1201	2.88	93.38
	2.79	93.35
	2.80	93.35
	2.74	
1400	3.14	94.05
	3.12	93.98
	3.19	
1607	3.80	94.52
	3.80	94.49
	3.81	
	3.82	
	85° K. (*Cont.*)	
1800	3.16	96.48
	3.05	96.57
	3.46	96.54
	3.18	96.59
	3.46	96.49
1999	3.54	96.89
	3.59	96.80
	3.65	96.79
	95° K.	
201	0.51	55.12
	0.48	54.61
	0.48	55.12
	0.45	
403	1.15	76.59
	1.03	77.42
		77.08
605	1.73	83.36
	1.67	83.42
	1.55	
803	2.21	86.82
	2.18	86.63
	2.20	86.88
996	2.76	89.13
	2.71	89.22
	2.70	89.29
1204	3.67	90.05
	3.36	90.15
	3.41	90.03
	3.19	
1398	4.09	90.80
	4.12	91.25
	4.04	91.49
	4.07	
1603	4.75	91.99
	4.63	92.16
	4.53	92.13
		92.28
1799	5.28	92.35
	5.02	92.35
	5.15	92.30
	5.06	
	5.23	
1996	5.63	92.99
	5.59	92.94
	5.61	93.08
	105° K.	
204	0.29	16.60
	0.25	16.80
	0.27	16.77
	0.27	16.68
400	1.05	49.68
	1.05	49.56
	1.03	49.48
602	2.11	63.15
	2.16	63.37
	2.13	63.40
805	3.12	71.77
	3.19	70.81
	3.15	70.94
		71.66
1000	4.29	75.94
	4.00	76.02
	3.87	75.94
	4.30	
	90° K. (*Cont.*)	
1797	4.15	94.64
	4.26	94.62
	4.24	94.54
	3.98	94.89
	4.00	
	3.95	
	4.31	
2000	4.61	95.11
	4.56	95.11
	4.58	
	100° K.	
204	0.43	35.26
	0.47	36.45
	0.45	36.38
	0.44	35.35
400	. . .	62.58
	. . .	63.31
	. . .	62.93
602	1.97	75.49
	1.98	75.97
	1.96	75.75
		75.81
802	2.78	81.01
	2.71	81.07
		81.11
992	4.13	83.64
	3.16	83.76
	3.35	
	3.31	
	3.30	
1200	4.38	85.92
	4.38	85.89
1396	5.08	86.82
	5.06	86.72
	5.07	
1606	5.87	88.35
	5.85	88.26
		88.28
1797	6.31	88.55
	6.24	88.34
	6.25	88.32
2007	6.96	89.44
	6.72	89.22
	110° K.	
400	1.07	34.09
	1.03	33.83
	1.00	34.09
	1.02	
603	2.36	50.63
	2.39	50.73
	2.34	50.78
	2.52	
803	3.78	59.57
	3.75	61.68
	3.72	60.14
		60.68
		62.12
1001	5.01	66.63
	4.79	66.93
	5.01	66.60
	5.05	

(Continued on page 476)

Table I. Experimental Vapor-Liquid Equilibrium Values

Pressure P.S.I.A.	Helium, Mole % Liquid	Helium, Mole % Vapor	Pressure P.S.I.A.	Helium, Mole % Liquid	Helium, Mole % Vapor
	105° K. (*Cont.*)			110° K. (*Cont.*)	
			1201	6.17	69.62
				6.06	69.70
1206	5.12	80.94		6.33	69.60
	5.38	80.14			
	5.16	81.32	1403	7.31	73.86
	5.10	81.02		7.32	73.96
				7.30	72.98
1406	6.17	82.50			73.18
	6.08	82.53			
			1606	8.18	77.49
1603	7.36	83.15		8.24	76.98
	7.28	83.03		8.17	77.15
	7.29				77.53
	7.10				
			1806	9.40	78.86
1799	7.90	84.02		8.59	78.76
	6.88	83.57		8.95	78.86
	7.60	84.16		9.08	
	8.00	83.40		9.19	
2001	8.17	84.84	2003	10.13	79.25
	8.49	84.84		9.50	79.50
	8.32	84.65		9.50	79.44
	8.14			9.84	
				10.18	
	115° K.			120° K.	
400	1.02	18.64	602	2.54	22.10
	0.99	18.56		2.52	22.02
	1.03	18.64		2.53	22.20
	1.03	18.54			
			806	4.93	35.39
602	2.56	37.24		4.76	35.45
	2.47	36.79		4.90	34.28
	2.49	36.96		5.00	33.40
	2.50	36.75		5.14	34.73
	2.59				
			1008	7.70	41.89
801	4.37	47.34		7.24	42.28
	4.40	46.25		7.39	41.75
	4.33	46.50		7.63	
	4.37	48.38			
			1199	9.13	48.42
1009	6.41	55.62		9.09	48.90
	5.70	55.13		9.22	48.90
	6.22	54.93			
	5.50	55.27	1400	10.26	51.78
	6.03			10.23	52.41
				10.28	52.41
1200	7.29	61.09			51.78
	7.16	60.79			
		61.39	1604	11.88	56.17
				11.96	56.36
1402	8.85	64.98		11.91	56.32
	9.09	64.58		11.83	56.14
	7.70	65.10			56.16
	8.75				
			1798	12.61	58.03
1605	9.64	67.83		13.11	58.24
	9.49	67.60		13.49	58.13
	9.74	67.69		12.77	
1803	10.95	70.08	2003	14.36	60.62
	10.17	69.73		13.59	60.36
	11.40	69.98		14.16	60.57
2005	11.56	71.97			
	12.07	72.64			
	11.73	71.50			
		71.92			

p.s.i.a. and higher isobars exhibit reverse-order solubility behavior, showing increasing solubility with temperature for any given pressure. Reverse-order solubility is also shown by the 200, 400, and 600 p.s.i.a. isobars until a temperature of maximum solubility is reached at 95°, 100.6°, and 116.5° K., respectively. After these maxima are reached, regular solubility behavior is observed with increasing temperature (decreasing solubility with increasing temperature).

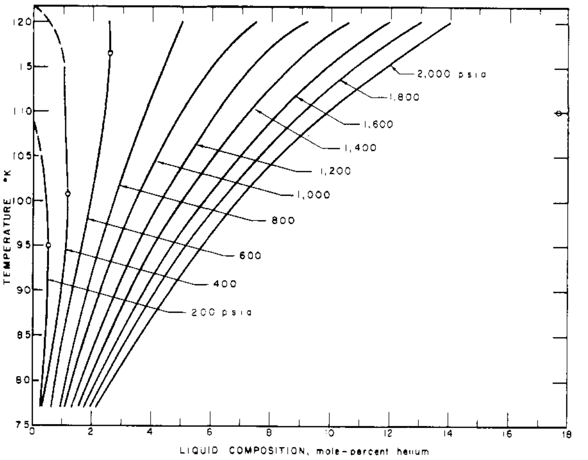

Figure 3. Temperature-composition diagram, liquid phase

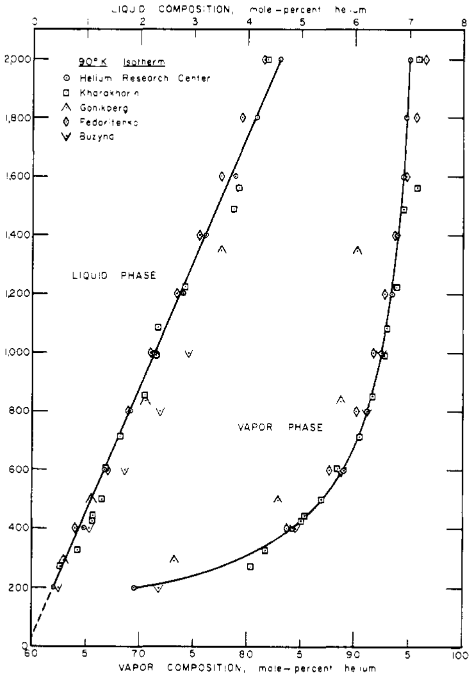

Figure 4. Comparison of Buzyna, Fedoritenko, Gonikberg, Kharakhorin, and Helium Research Center's data at 90° K.

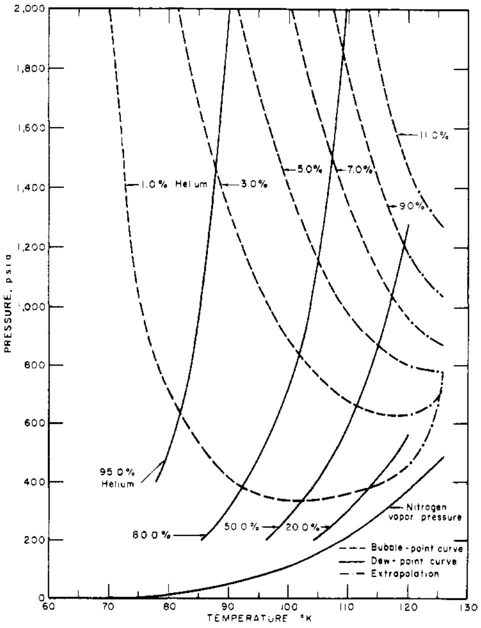

Figure 5. Pressure-temperature diagram

Table II. Smoothed Composition Values, Helium, Mole %

Temp., °K.	Pressure (P.S.I.A.) 200	400	600	800	1000	1200	1400	1600	1800	2000
					Liquid Phase					
77	0.26	0.33	0.62	0.91	1.08	1.31	1.52	1.72	1.92	2.14
80	0.31	0.49	0.74	1.12	1.36	1.64	1.89	2.14	2.44	2.66
85	0.39	0.76	1.04	1.44	1.78	2.22	2.51	2.90	3.25	3.61
90	0.40	0.96	1.34	1.83	2.26	2.80	3.21	3.76	4.15	4.60
95	0.48	1.09	1.64	2.23	2.76	3.48	4.08	4.66	5.15	5.65
100	0.42	1.16	1.92	2.70	3.40	4.32	5.04	5.80	6.30	6.89
105	0.27	1.04	2.15	3.15	4.14	5.18	6.12	6.95	7.59	8.27
110	...	1.03	2.40	3.74	4.98	6.19	7.26	8.24	9.08	9.86
115	...	1.02	2.53	4.34	5.98	7.26	8.63	9.84	10.9	11.8
120	...	...	2.53	4.98	7.46	9.14	10.6	11.9	13.0	14.0
					Vapor Phase					
77	91.1	95.7	97.0	97.6	98.0	98.3	98.3	98.4	98.4	98.6
80	87.6	93.3	95.6	96.5	97.0	97.2	97.7	97.7	97.8	98.2
85	80.6	89.8	92.9	94.4	95.1	95.7	96.3	96.3	96.5	96.8
90	69.6	84.2	89.0	91.2	92.4	93.4	93.9	94.4	94.7	95.0
95	54.3	75.4	83.1	86.8	88.9	90.3	91.1	91.7	92.3	92.7
100	36.3	63.4	75.0	80.7	84.1	86.2	87.5	88.4	89.1	89.7
105	16.6	49.1	63.8	72.2	77.3	80.3	82.3	83.4	84.8	85.5
110	...	33.7	51.6	61.2	67.9	72.2	75.0	76.8	78.4	79.7
115	...	18.7	37.2	48.2	55.6	60.8	64.8	67.4	69.6	71.4
120	...	...	22.2	34.0	42.2	48.1	52.5	55.8	58.2	60.2

Table III. Smoothed Equilibrium Constants, *K*, for Helium and Nitrogen

Temp., °K.	K_{He} 200	400	600	800	1000	1200	1400	1600	1800	2000
76.5	341	287	156	107	91.55	75.05	64.64	57.19	51.27	46.29
80	283	195	128	84.67	71.34	59.64	51.69	45.45	40.07	37.34
85	208	...	89.25	63.82	54.61	42.95	38.81	33.19	29.61	26.97
90	170	86.7	66.16	49.91	41.21	33.34	29.85	24.80	22.91	20.77
95	114	70.67	50.54	39.45	32.80	26.11	22.35	19.86	17.93	16.57
100	79.9	...	38.45	29.58	24.26	19.61	17.11	15.07	14.10	13.06
105	62.8	47.66	29.72	22.64	18.44	15.58	13.48	11.44	11.03	10.24
110	...	33.01	21.13	16.22	13.45	11.25	10.05	9.426	8.701	8.077
115	...	18.24	14.66	10.84	9.253	8.461	7.545	7.046	6.433	6.108
120	...	...	8.739	7.000	5.603	5.327	4.892	4.725	4.472	4.318
					$K_{N_2} \times 10^2$					
76.5	8.91	4.28	3.00	2.43	2.062	1.702	1.767	1.669	1.591	1.441
80	12.1	6.62	4.27	3.520	3.021	2.826	2.355	2.340	2.276	1.849
85	19.2	...	7.255	5.623	5.058	4.316	3.835	3.852	3.587	3.288
90	30.5	16.2	12.17	8.821	7.856	6.831	6.174	5.718	5.612	5.125
95	45.3	23.22	16.89	13.52	11.09	10.27	9.195	8.242	8.086	7.480
100	64.4	...	24.75	19.47	16.88	14.75	13.94	12.43	12.38	11.45
105	83.5	50.96	37.49	29.62	25.06	20.19	18.62	18.23	17.54	16.59
110	...	66.69	50.50	40.69	35.02	32.36	28.59	24.74	23.28	22.85
115	...	82.44	64.69	55.05	47.60	41.94	38.41	35.72	33.74	31.73
120	...	...	79.91	68.75	62.73	56.42	53.61	49.68	48.13	45.81

Gonikberg and Fastowsky (*7*). There is good agreement between the data of this study and that of Kharakhorin (*8*). The agreement with Buzyna's (*3*) data is satisfactory for the vapor phase, but poor for the liquid phase.

A comparative picture of the data obtained in this study with those of previous investigations is given in Figure 4 for the 90° K. isotherm. This figure shows only qualitative agreement between the data. Buzyna's data for helium in the liquid phase show the greatest deviation (23.5%); in the vapor phase, Gonikberg's data deviate by 3.9%. Although only one isotherm is presented, it is representative of the disagreement that exists among the data of previous investigations.

The work reported here is in satisfactory agreement with that of Fedoritenko and Ruhemann (*5*) and that of

A pressure-temperature diagram for the coexisting phases is presented in Figure 5. Minima are observed in the bubble point curves for mixtures containing three mole % helium or less. These minima are observed for pressures less than 625 p.s.i.a. The dew point curves have positive slopes and asympotically approach the critical temperature of nitrogen. Phase separation was not observed in this study at temperatures greater than 124° K.

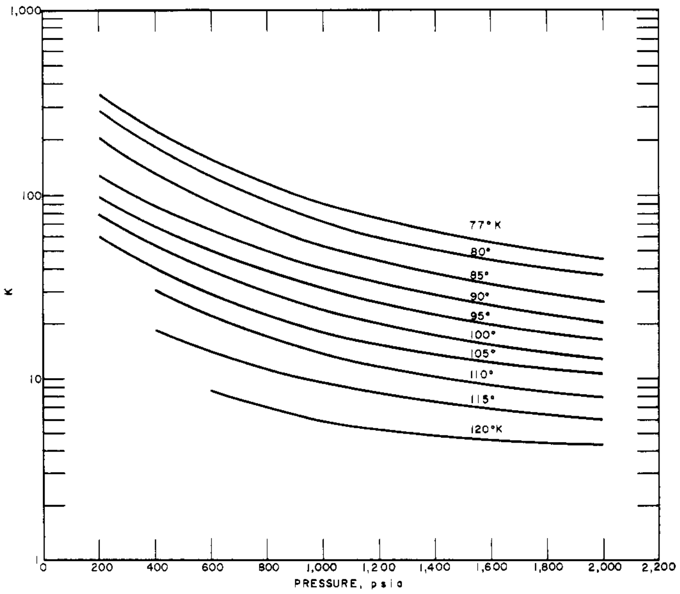

Figure 6. Equilibrium constant-pressure diagram for helium

Isothermal equilibrium constants for helium are shown as functions of pressure in Figure 6. For a given pressure, the K value for helium is greatest at the lowest temperature. For any given isotherm, the K values increase with decreasing pressure.

This study represents a more thorough investigation than previously reported (*3, 5, 7, 8, 10*) on the helium-nitrogen system at temperatures from 76.5° to 120° K. and pressures up to 2000 p.s.i.a. The average reproducibility of all vapor phase data was within ±0.13 mole %. The corresponding value for the liquid phase was within ±0.07 mole %.

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Vapor-Liquid Equilibria in a Natural Gas-Condensate-Nitrogen System

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This paper describes an experimental investigation on the effect of nitrogen on the vapor-liquid equilibrium ratios of hydrocarbons in the natural gas-condensate system. Experimental work was carried out under constant temperature conditions at 100° F. and 220° F. At each temperature, experiments were conducted at pressures of 500, 1000, 1500, 2000, and 3000 p.s.i.a. Nitrogen concentration in the vapor phase was varied from 10% to 50% at each percentage. The equilibrium constants for hydrocarbons and for nitrogen are shown.

THIS PAPER describes an experimental investigation on the effect of nitrogen on the vapor-liquid equilibrium ratios of the hydrocarbons in a natural gas-condensate system. Nitrogen is often found in appreciable concentrations in petroleum fluids, either as a natural occurrence or as a result of the application of secondary recovery techniques.

K-values for complex hydrocarbon systems have been reported in the literature (*7, 16*). Poettman (*11*), and Poettman and Katz (*12*) have reported K-values for CO_2 in similar hydrocarbon systems. Jacoby and Rzasa (*6*) list K-values for nitrogen, methane, CO_2, ethane and hydrogen sulfide in mixtures of all these components in heavier hydrocarbons. Eilerts and coworkers (*3*) have published a report on the effect of nitrogen and other gases and light hydrocarbons on the phase-boundary pressures of complex systems. In addition several binary systems of various hydrocarbons with nitrogen have been investigated (*1, 2, 14*) as well as the ternary system n-butane-methane-nitrogen (*15*).

EXPERIMENTAL

The experimental work was carried out under constant temperature conditions at 100° F. and at 220° F. At each temperature, experiments were conducted at pressures of 500, 1000, 1500, 2000 and 3000 p.s.i.a. The nitrogen concentration in the vapor phase was varied from 10% to 50% at each pressure.

The fluids were brought to equilibrium in a 35-ml. windowed stainless steel cell. In order to attain equilibrium the vapor was circulated from the top of the cell through a magnetic-piston pump and into the bottom of the cell so that it bubbled up through the liquid layer.

The equilibrium phases at each different set of conditions of temperature, pressure and composition were sampled and analyzed in a Beckman Thermotrac temperature programmer. The samples were withdrawn from the equilibrium cell into stainless steel high pressure tubing which was connected to the cell. Pressure in the equilibrium cell was maintained constant by injecting mercury into the bottom of the cell during the sampling procedure.

Finally each sample was allowed to expand into a glass flask. The size of each sample was regulated so that the pressure in the flask was 10 to 20 cm. of Hg. In addition

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