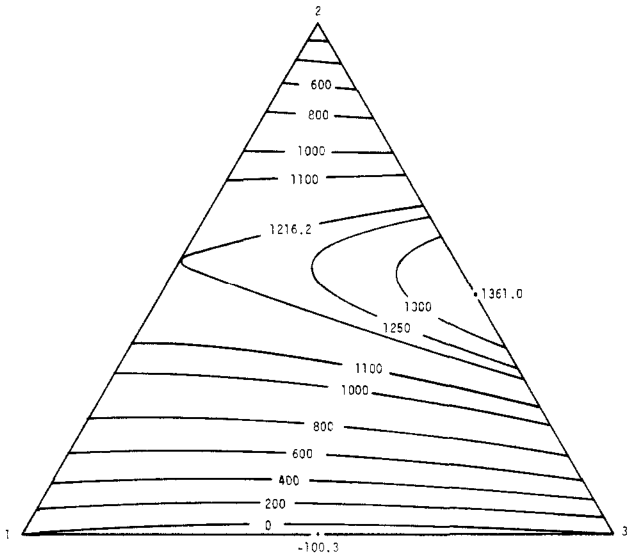

Figure 4. Lines of constant G^E (J/mol) for the ethylene glycol (1)/acetonitrile (2)/water (3) system at 50 °C.

Glossary

A_{ij}, A_{ji}	parameters in eq 1
B_{ij}	second virial coefficient
C_0, C_1, C_2	parameters in eq 2
G^E	excess Gibbs function, liquid phase
g	G^E/RT
P	total pressure
P_i^{sat}	vapor pressure of pure i
R	universal gas constant
T	absolute temperature
V_i^L	molar volume of pure liquid i
x	mole fraction, liquid phase

Greek Letters

λ_{ij}, λ_{ji}	parameters in eq 1
η_{ij}, η_{ji}	parameters in eq 1

Registry No. Ethylene glycol, 107-21-1; acetone, 67-64-1; acetonitrile, 75-05-8.

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Dew Point Measurements for Nitrogen–Propane and Nitrogen–Butane Mixtures

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Dew point measurements were made for binary mixtures containing nitrogen and propane, *n*-butane, or isobutane. The temperature range was 188.2–350.4 K and the pressure range was 0.083–6.897 MPa. Nitrogen mole fractions ranged from 0.090 to 0.910.

Introduction

Accurate phase equilibria data for mixtures of nitrogen and hydrocarbons are needed to improve predictive procedures for estimation of properties of mixtures in natural gas processes. As part of an ongoing effort, dew point data were determined for binary mixtures of nitrogen with propane, *n*-butane, and isobutane. Measurements were made in ranges where data are not currently available.

Previous investigators have presented vapor–liquid equilibria data for the nitrogen–propane system (*1–4*), the nitrogen–*n*-butane system (*5–8*), and the nitrogen–isobutane system (*9*). These studies provided a data base to identify regions where additional data are necessary.

Experimental Section

Equipment. A schematic diagram for the dew point apparatus is shown in Figure 1. All of the piping, valves, and fittings are constructed from 316 stainless steel, and each piece was washed with acetone and flushed with nitrogen before assembly.

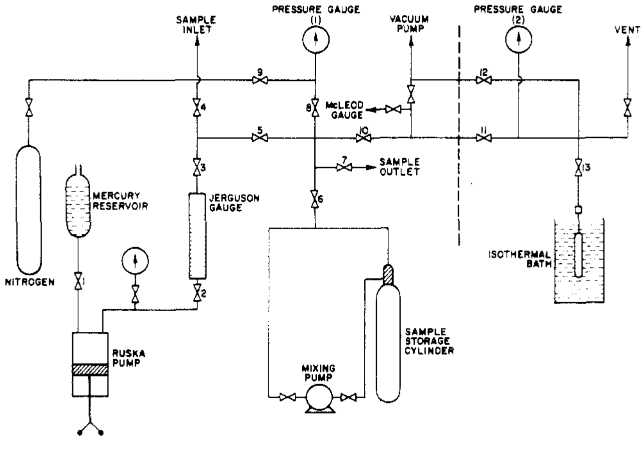

Figure 1. Schematic diagram of dew point equipment.

To preserve the uniformity of the gas composition charged into the cell, the system, with the exception of the cell, its

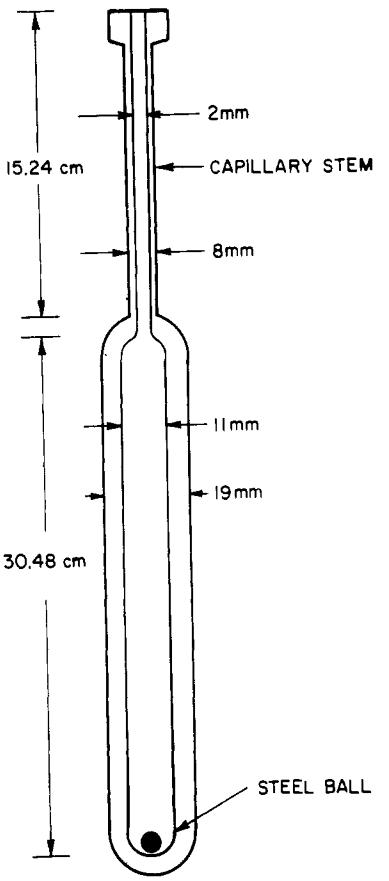

Figure 2. Dew point cell.

constant-temperature bath, and mercury pump, was enclosed in the constant-temperature cabinet, where a relatively high temperature well above the highest dew point of the mixture was maintained.

The dew point cells were constructed from heavy duty Pyrex glass with an outer diameter of 19 mm and an inner diameter of 11 mm. The volume of the cells was approximately 29 cm^3. Each cell contained a 400 series stainless steel ball which provided agitation of the contents of the cell by manual movement of a U-shaped magnet. The manufacturer's recommended maximum working pressure was 13.79 MPa. All the cells that were used in the experiments were pressure tested under a pressure of 8.37 MPa for a period of 24 h, and no failure was observed. The cell was connected to steel tubing of the system through a capillary glass (2-mm bore, 8-mm outer diameter, 15.24-cm length) tube to a glass-to-metal connector. A schematic diagram of a dew point cell is shown in Figure 2.

The glass-to-metal connector, which was fabricated from stainless steel, was designed to have a minimum dead volume and be capable of absorbing the vibrations generated by the moving parts of the system. A rubber O ring was provided to prevent the steel ring from having direct contact with the glass flange, and acted as an effective seal at high pressures and temperatures. The small volume between the liquid level in the cell and the valve reduced the diffusion in the line.

Methods for obtaining a low temperature, controlling it at a constant value, and measuring it accurately have been reported before (*10*). The equilibrium cell was placed in a 0.009 46-m^3 Dewar flask filled with either commercial isopentane for low-temperature measurements (122–288 K) or ethylene glycol for measurements at higher temperatures (277–355 K).

The Dewar flask was provided with three windows which permitted visual observation of the glass cell and its contents. A gasket, constructed of several rubber rings, at the top of the Dewar acted as a seal and prevented direct contact of the bath fluid with the surface of the higher temperature equipment cabinet which contained the storage cylinder, the mixing pump, the Jerguson gauge, and system tubing.

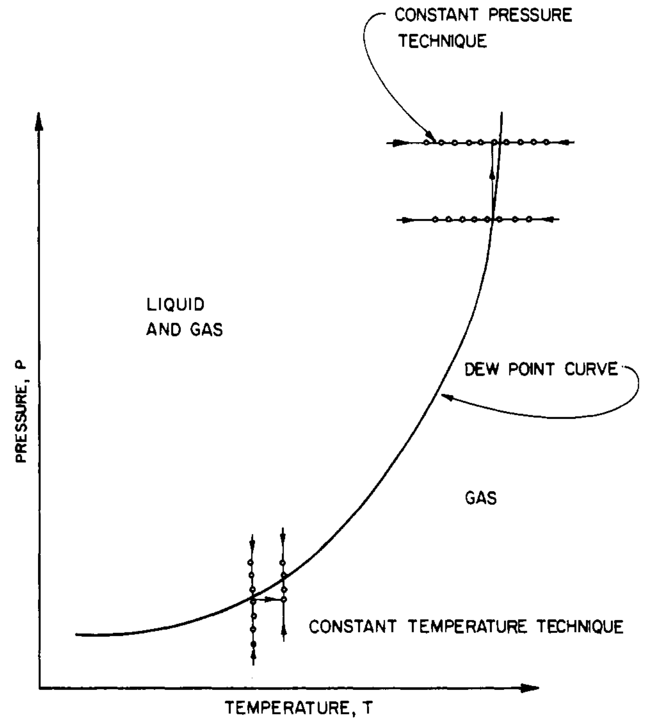

Figure 3. Constant-pressure technique and constant-temperature technique.

The temperature of the bath was controlled by the combined action of a cooling coil and electric resistance heater. The refrigerant for the cooling coil was liquid nitrogen. The flow of liquid nitrogen, and therefore the rate of cooling, was controlled by the two valves placed at the exit of the tank. One of these valves was for rapid cooling and the other one for maintaining small flow rates as required for constant-temperature operation.

The copper tube heater which is the source of heat was regulated by a sensor inserted inside the Dewar flask. The sensor was connected to a Bayley temperature controller Model 114, which is capable of controlling the temperature in the range 73.1–373.1 K to ±0.01 K. A powerstat accompanied this unit.

The bath fluid was stirred continuously during a run to ensure a uniform temperature throughout the fluid. It was assumed that the temperature of the cell contents was the same as that of the bath fluid.

The temperature of the bath was measured with a Leeds and Northup Model 8167-25-B platinum resistance thermometer. The thermometer was calibrated against a National Bureau of Standards reference. The resistance of the thermometer was measured by a Leeds and Northup Model 8054 resistance thermometer bridge. The accuracy of the bridge is 0.005 Ω or ±0.05% of the reading, whichever is larger. This corresponds to a temperature accuracy of ±0.05 K.

Two high-precision gauges were employed to measure pressure. One gauge (PG1) measured the pressure of the Jerguson gauge and tubing network in the sample section. The other gauge (PG2) measured the cell pressure.

These gauges were provided by Heise Bourdon Tube Co., Inc., and were capable of measuring the pressure to 13.79 MPa. The reported accuracy of measurements is 0.1% of the full-scale reading.

The mixture was stored in a high-pressure steel storage cylinder with a volume of approximately 0.040 m^3. The cylinder was seamless and equipped with a sample mixing manifold. The manifold consisted of a piping arrangement to provide inlet

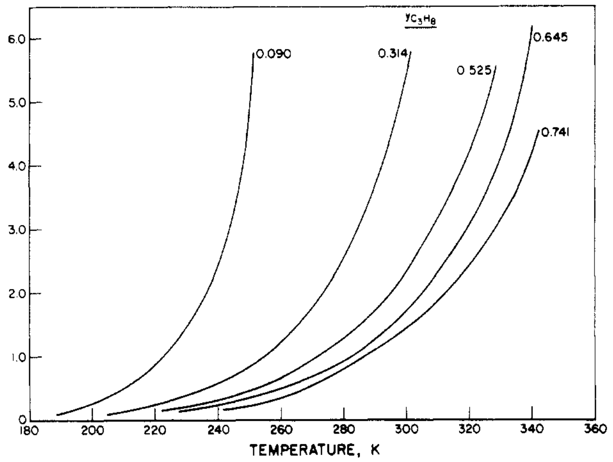

Figure 4. Dew points for nitrogen–propane mixtures.

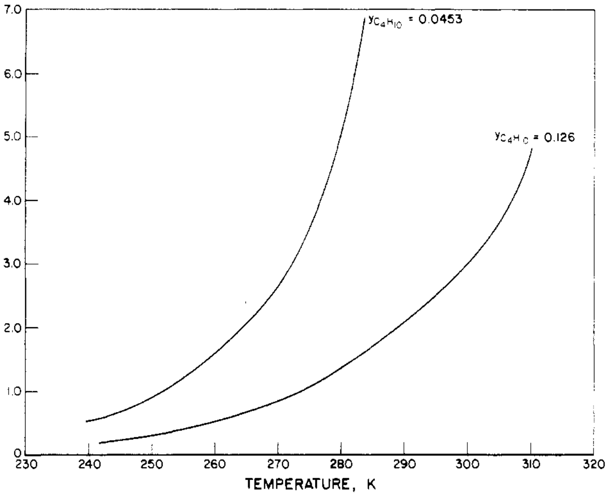

Figure 5. Dew points for nitrogen–*n*-butane mixtures.

and outlet facilities for a recirculating mixing pump, and a port for a thermocouple assembly. The pump is an oscillating, free piston magnetically driven pump.

The system pressure can be increased in two ways. The pressure in the phase cell can be increased by transferring more material from the storage cabinet to the cell, provided the pressure in the storage cylinder is higher than that in the system. The pressure of the system can also be increased by displacing mercury, by means of the Ruska pump, into the Jerguson gauge. The valve at the bottom of the Jerguson gauge was opened for that purpose. A mercury reservoir was provided to develop large changes in pressure with the mercury pump. To transfer mercury from the mercury reservoir which is at atmospheric pressure, valve 2 (Figure 1) was closed and valve 1 was opened to bring the pressure of the mercury pump to atmospheric pressure. The pressure of the mercury pump was measured by a 20.68-MPa bourdon tube gauge pressure.

***Procedure*.** For preparation of a mixture with the desired composition, the storage cylinder and other parts were washed with acetone, reconnected, leak tested, and held under vacuum for about 24 h. The hydrocarbon was introduced first into the storage cylinder and then a sufficient amount of nitrogen was added until the desired composition was reached. The basis for preparing a mixture with a particular composition was partial pressure which served as a relatively accurate procedure. With this procedure we could make mixtures with a maximum deviation of ±1% from the specified composition.

The components were mixed by an electric mixing pump, usually requiring about 1 week of continuous mixing. During the mixing, the composition was measured by both gas chromatography and mass spectroscopy analysis. Usually the difference in compositions for the first mixture measured on the 9th and 11th days was less than 0.2%, which indicated complete mixing. Subsequent mixtures which were prepared by adding nitrogen required only 4–6 days of mixing time.

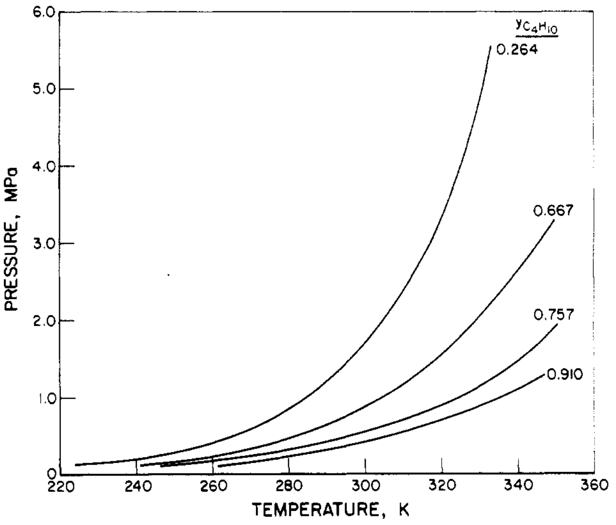

Figure 6. Dew points for nitrogen–isobutane mixtures.

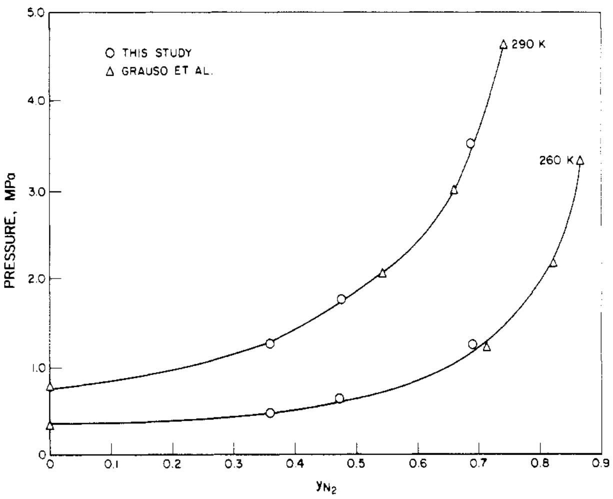

Figure 7. Comparison of nitrogen–propane system with other data.

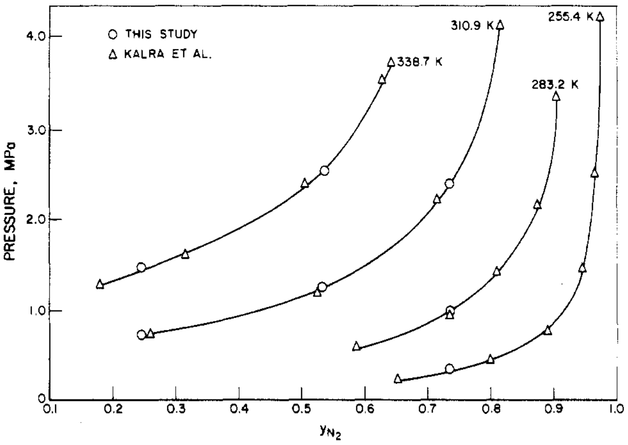

Figure 8. Comparison of nitrogen–isobutane system with other data.

Prior to beginning a run for each mixture, the entire tubing network was flushed with nitrogen to remove all traces of contaminants and leak tested. Prior to a run the entire system, except the storage cylinder, was evacuated. After charging a small amount of gas mixture, we evacuated the system again.

Table I. Dew Points for Propane–Nitrogen Mixture with $y_{C_3H_8} = 0.090$

press., MPa	temp, K	press., MPa	temp, K
0.083	188.2	1.944	235.9
0.158	192.1	2.344	239.3
0.200	197.1	2.750	242.1
0.283	203.2	3.267	244.3
0.393	208.2	3.474	245.9
0.558	214.0	3.998	248.2
0.800	220.4	4.742	249.8
1.082	225.4	5.363	251.5
1.282	228.7	5.769	251.8
1.592	232.6		

Table II. Dew Points for Propane–Nitrogen Mixture with $y_{C_3H_8} = 0.314$

press., MPa	temp, K	press., MPa	temp, K
0.096	204.8	2.178	276.5
0.138	210.9	2.371	278.7
0.200	216.5	2.571	280.9
0.255	222.1	2.792	283.2
0.331	227.6	3.019	285.4
0.400	233.2	3.274	287.6
0.503	238.7	3.488	289.3
0.634	244.3	4.136	293.7
0.793	249.8	4.536	295.9
0.993	255.4	4.853	297.6
1.220	260.9	5.204	299.3
1.475	265.9	5.625	300.9
1.709	269.5	5.783	301.5
1.937	273.2		

Table III. Dew Points for Propane–Nitrogen Mixture with $y_{C_3H_8} = 0.525$

press., MPa	temp, K	press., MPa	temp, K
0.124	222.1	1.592	288.2
0.158	226.5	1.737	290.9
0.186	230.4	1.889	293.7
0.214	233.7	2.061	296.5
0.255	237.1	2.164	298.2
0.303	240.9	2.330	300.4
0.345	244.3	2.481	302.6
0.386	247.6	2.702	305.4
0.434	250.4	2.888	307.6
0.496	253.7	3.088	309.8
0.551	256.5	3.302	312.1
0.634	260.9	3.536	314.3
0.744	264.8	3.757	316.2
0.841	268.7	4.370	322.1
0.972	272.6	4.618	323.7
1.096	275.9	4.839	325.1
1.241	280.4	5.328	327.6
1.420	284.3	5.652	328.7

Table IV. Dew Points for Propane–Nitrogen Mixture with $y_{C_3H_8} = 0.645$

press., MPa	temp, *K*	press., MPa	temp, K
0.103	227.6	1.578	296.8
0.138	232.1	1.861	302.6
0.165	235.4	2.213	308.7
0.200	238.7	2.502	313.2
0.234	242.6	2.840	317.6
0.296	247.6	3.157	321.5
0.372	253.2	3.474	324.8
0.462	259.2	3.839	328.2
0.586	265.4	4.294	332.1
0.731	272.1	4.749	335.4
0.903	278.7	5.501	339.0
1.034	283.7	5.549	339.3
1.241	290.4	5.687	340.0

Table V. Dew Points for Propane–Nitrogen Mixture with $y_{C_3H_8} = 0.741$

press., MPa	temp, K	press., MPa	temp, K
0.131	241.5	1.647	305.9
0.200	247.6	2.061	314.8
0.303	255.7	2.302	319.8
0.441	264.3	2.799	326.5
0.544	270.4	3.102	330.4
0.662	275.9	3.453	334.3
0.793	280.9	3.819	337.6
0.965	286.5	4.301	341.5
1.061	291.5	4.508	342.7
1.358	298.7		

Table VI. Dew Points for *n*-Butane–Nitrogen with $y_{C_4H_{10}} = 0.0453$

press., MPa	temp, K	press., MPa	temp, K
0.526	239.5	3.451	273.9
0.692	245.3	3.802	276.4
1.036	253.2	4.082	276.6
1.383	257.9	4.123	277.0
1.425	257.9	4.140	276.6
2.044	265.7	4.828	278.8
2.076	265.7	4.829	278.8
2.554	269.7	5.177	280.8
2.692	269.9	5.515	280.7
2.761	269.9	5.518	280.6
2.761	270.1	6.222	282.6
3.382	273.9	6.621	283.2
3.423	274.1	6.897	283.5

Table VII. Dew Points for *n*-Butane–Nitrogen with $y_{C_4H_{10}} = 0.126$

press., MPa	temp, K	press., MPa	temp, K
0.158	241.5	2.033	290.0
0.262	246.5	2.350	293.3
0.345	252.6	2.564	296.2
0.482	258.7	2.757	297.6
0.689	265.4	2.888	299.3
0.827	269.3	3.129	301.5
0.875	270.4	3.398	303.7
1.020	273.7	3.867	306.5
1.151	276.8	4.205	307.9
1.296	279.3	4.405	308.7
1.461	281.9	4.618	309.6
1.737	286.1	4.784	310.4

Table VIII. Dew Points for Isobutane–Nitrogen Mixture with $y_{C_4H_{10}} = 0.264$

press., MPa	temp, K	press., MPa	temp, K
0.101	224.1	1.463	295.8
0.108	225.8	1.652	298.9
0.136	232.8	1.787	302.2
0.167	236.1	1.959	304.8
0.184	239.8	2.073	306.7
0.218	243.8	2.328	310.0
0.287	249.5	2.600	313.8
0.343	256.1	2.852	316.7
0.401	259.8	3.086	318.7
0.460	262.9	3.334	320.3
0.580	269.0	3.727	322.9
0.698	274.4	3.944	324.3
0.835	279.5	4.065	324.9
0.942	283.2	4.447	327.2
1.022	285.3	4.820	329.6
1.221	290.3	5.150	331.9
1.335	293.2	5.512	333.3

Table IX. Dew Points for Isobutane–Nitrogen Mixture with $y_{C_4H_{10}} = 0.467$

press., MPa	temp, K	press., MPa	temp, K
0.101	240.8	0.922	302.3
0.115	243.8	1.025	305.9
0.126	245.4	1.115	309.0
0.153	249.8	1.228	311.8
0.170	252.9	1.380	315.9
0.187	254.8	1.532	319.6
0.225	259.8	1.669	322.3
0.274	264.8	1.794	324.8
0.298	267.9	1.925	328.2
0.336	270.9	2.097	331.8
0.380	275.4	2.235	334.0
0.384	275.4	2.400	337.1
0.425	278.2	2.407	337.1
0.470	280.9	2.455	337.9
0.539	284.8	2.586	340.1
0.618	288.7	2.769	342.9
0.687	292.1	3.007	345.9
0.746	295.4	3.276	349.9
0.828	298.7		

Table X. Dew Points for Isobutane–Nitrogen Mixture with $y_{C_4H_{10}} = 0.757$

press., MPa	temp, K	press., MPa	temp, K
0.101	246.7	0.715	310.9
0.115	251.9	0.742	312.6
0.122	254.3	0.825	316.5
0.132	256.6	0.887	319.3
0.153	259.8	0.935	321.5
0.163	262.2	1.035	325.4
0.177	265.4	1.094	327.6
0.208	269.8	1.156	329.8
0.229	273.2	1.290	334.3
0.253	276.5	1.366	336.5
0.301	281.5	1.373	336.5
0.356	286.5	1.439	338.7
0.411	292.1	1.549	341.5
0.480	297.6	1.628	343.2
0.560	302.6	1.680	344.8
0.563	302.6	1.790	347.6
0.649	307.6	1.921	350.4

Table XI. Dew Points for Isobutane–Nitrogen Mixture with $y_{C_4H_{10}} = 0.910$

press., MPa	temp, K	press., MPa	temp, K
0.108	261.2	0.477	305.4
0.125	264.3	0.536	309.8
0.132	266.5	0.539	309.8
0.139	267.7	0.605	314.3
0.160	271.5	0.639	316.5
0.163	271.5	0.687	319.2
0.177	275.4	0.736	322.1
0.194	277.6	0.784	324.3
0.215	280.4	0.822	326.5
0.243	283.7	0.863	329.0
0.260	285.9	0.911	330.9
0.274	287.6	0.977	333.7
0.305	291.5	1.011	335.4
0.308	291.5	1.080	338.2
0.356	295.4	1.153	341.5
0.377	297.6	1.149	341.5
0.408	299.8	1.211	344.3
0.439	302.6	1.290	347.6

Prior to charging, valves 4, 7, 10, and 9 were closed and valves 3, 5, 8, and 6 were opened to allow the flow of gas to the Jerguson gauge (see Figure 1). The Jerguson gauge acted as a storage for a mixture as well as a pressure booster.

Depending on the sensitivity of the pressure–temperature curve to either pressure or temperature, two modes of operation were carried out to obtain measurements. At low temperatures, the curve is sensitive to pressure, which leads to constant-temperature operation. At high temperatures, the curve is sensitive to temperature, which leads to constant-pressure operation as shown in Figure 3.

At low temperature, before charging the gas into the phase cell, the temperature of the bath fluid was brought to the desired temperature. At high temperature, the pressure of the gas was usually boosted to approximately the desired pressure before it was charged to the equilibrium cell.

The visual observation technique to determine dew points has been successfully employed by previous investigators (*10*, *11*).

In constant-temperature operation, the temperature of the cell was kept constant at a preselected value and the pressure was changed until droplets of liquid formed on the stainless steel ball. When the steel ball made contact with the glass neck, the liquid wetting the glass surface was visible. The pressure was then lowered until the liquid disappeared. Thereafter, the pressure in the cell was increased by 0.014-MPa increments until a liquid ring was observed. The pressure was decreased slightly and the contents were agitated. If the liquid ring did not disappear, the pressure was decreased further until no liquid ring could be observed. at this point, pressure was increased by 0.0034-MPa increments until a very thin liquid ring was observed. Further increase in pressure resulted in the formation of droplets on the wall of the cell. The appearance of the thin ring at the intersection of the glass neck and the steel ball was identified as the dew point.

In the constant-pressure operation, the pressure of the cell was maintained at a desired value by transferring more gas to the cell while it was rapidly cooled, until liquid droplets were observed. Then the temperature of the cell was increased by 1 K increments. When the temperature seemed to approach the dew point temperature, the rate of heating was decreased and the contents of the cell were thoroughly agitated. When the temperature of the cell was very close to the dew point temperature, the temperature was lowered in 0.05 K increments, until the dew point was reached. Again, the formation of the thin ring at the intersection of the glass neck and the steel ball was chosen as the criterion for visual determination of the dew point.

Chemicals Used. High-quality, research-grade gases were used for the standard mixtures and samples. High-purity nitrogen furnished by Union Carbide was reported to contain 99.995% nitrogen. Research-grade propane, *n*-butane, and isobutane were purchased from Matheson Co, and were reported to have purities of 99.99%, 99.97%, and 99.996%, respectively.

Mixture Analysis. Gas chromatography was used for the determination of mixture compositions. The chromatograph unit consisted of a Barber Colman gas chromatograph series 5000 with a thermal conductivity detector. The column was made from 0.635-cm-diameter stainless steel tubing of 0.182-m length filled with 80–100 mesh Porapack-Q. A Hewlett-Packard series 3370 integrator recorded retention time and peak area. Visual observation of the peak shape, separation, and base line was made by a Unicorder, Model 20, connected to the integrator. The estimate of the standard deviation of the mole fraction measurement was in all cases within ±0.001.

Mixture compositions were determined by comparing gas chromatograph output areas with those of a standard mixture. The standard mixture was prepared by first charging the lower molecular weight component to a stainless steel bomb and determining the mass of the component added with a Seko mass balance with an accuracy of ±0.001 g. Then the higher molecular weight component was added to the bomb and its mass was determined with the Seko balance. For each mixture composition determination, chromatographs were determined for the sample mixture and the standard mixture and the sample composition was calculated from the equation

$$y_i^{\text{sample}} = (A_i^{\text{sample}}/A_i^{\text{standard}})y_i^{\text{standard}} / \{(A_i^{\text{sample}}/A_i^{\text{standard}})y_i^{\text{standard}} + (A_j^{\text{sample}}/A_j^{\text{standard}})y_j^{\text{standard}}\}$$

Results and Discussion

The experimentally determined dew points for the nitrogen–propane mixtures, the nitrogen–*n*-butane mixtures, and the nitrogen–isobutane mixtures are presented in Tables I–XI. The results are presented graphically in Figures 4–6. The maximum uncertainty in pressure measurements was estimated to be ±0.015 MPa and the maximum uncertainty in temperature measurements was estimated to be ±0.05 K.

Figure 7 shows a comparison between the isothermal pressure–composition measurements by Grauso et al. (*4*) for the nitrogen–propane system and the measurements made in this study. Figure 8 shows a similar comparison between the nitrogen–isobutane data of Kaira et al. (*9*) and the data obtained in this study. Although the agreement is good, the range of overlapping data is small and the dew point temperatures from this work used in the comparison do not correspond exactly to the temperatures of the other studies.

Registry No. Nitrogen, 7727-37-9; propane, 74-98-6; butane, 106-97-8; isobutane, 75-28-5.

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Dew and Bubble Point Measurements for a Methane–Ethane–Propane Mixture

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Dew and bubble point measurements were made for a ternary mixture of methane, ethane, and propane (z_{CH_4} = 0.8511; $z_{C_2H_6}$ = 0.1007). The temperature range of measurements was 144–245 K and the pressure range was 0.689–7.235 MPa. A complete phase boundary curve was developed. The mixture critical point was determined to be 224.21 K and 6.850 MPa.

Introduction

The purpose of this paper is to present experimentally determined dew and bubble points for a mixture of methane, ethane, and propane which establishes a complete phase boundary for the mixture. These data can be used in the development and testing of correlations for prediction of properties under conditions of vapor–liquid equilibrium.

Some vapor–liquid equilibrium data for the methane–ethane–propane system are presented by Price and Kobayashi (*1*). They employed a vapor circulation method and determined vapor- and liquid-phase compositions by infrared spectroscopy and gas–liquid partition chromatography. Vairogs et al. (*2*) presented the results of a phase equilibria study for a multicomponent mixture containing methane, ethane, and propane.

Dew and bubble point measurements for simulated natural gas mixtures were presented by Gonzalez and Lee (*3*). Techniques for the determination of dew points were presented by Van Poolen (*4*) and Bloomer et al. (*5*).

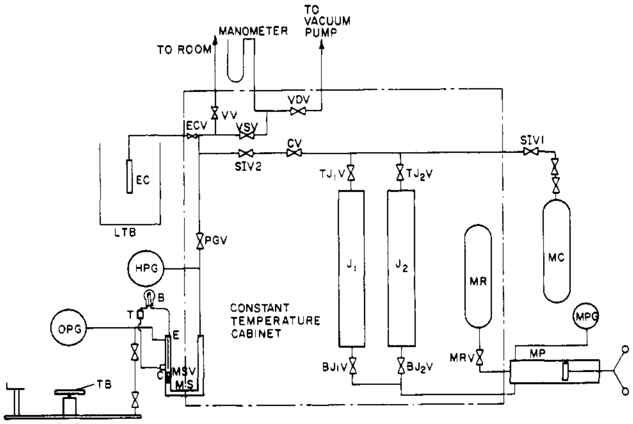

Figure 1. Schematic diagram of apparatus.

Experimental Section

Equipment. A schematic diagram of the dew and bubble equipment is presented in Figure 1. The equipment is a modified version of the phase equilibrium apparatus employed by Bloomer and Parent (*6*).

Dew and bubble points were determined with visual aid in Pyrex glass equilibrium cells (Figure 1, EC). The glass equilibrium cell used to determine the dew points is similar in design to that used by Van Poolen (*4*). The approximate volume of