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Dew and Bubble Point Measurements for Carbon Dioxide–Propane Mixtures

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Dew and bubble point measurements were made for binary mixtures of carbon dioxide and propane over a temperature range of 210.9–349.8 K with carbon dioxide mole fractions ranging from 0.076 to 0.442 and for pressures up to 5.86 MPa. Dew point measurements were also made for a ternary mixture of carbon dioxide, propane, and methane having a carbon dioxide mole fraction of 0.048 and a propane mole fraction of 0.229.

Introduction

Design of low-temperature processes for removal of carbon dioxide from natural gas or refinery gas requires the capability to make accurate predictions of vapor–liquid equilibria properties of carbon dioxide–hydrocarbon mixtures. Systematically developed phase equilibria data for binary mixtures of carbon dioxide and hydrocarbons can be used as a base for development of accurate correlations for the prediction of phase equilibria properties of multicomponent mixtures. In this study, dew and bubble points were measured for five binary mixtures of carbon dioxide and propane and dew points were measured for a ternary mixture of carbon dioxide, propane, and methane.

Previous investigators (*1–7*) have studied the carbon dioxide–propane system and the results of these studies provided the basis for identification of regions where additional data are needed. Vapor–liquid equilibria data are not available over the complete concentration range below 244 K.

Experimental Section

Experimental details which have been presented elsewhere (*8*) are summarized below.

Equipment. Figure 1 shows a schematic diagram of the apparatus. All valves, piping, and fittings were constructed from 316 stainless steel and the equilibrium cells were constructed from heavy-duty Pyrex glass.

Figure 2 shows dew point and bubble point cells that were constructed from heavy-duty Pyrex glass with an outer diameter of 19 mm, and inner diameter of 11 mm, and an approximate volume of 29 cm^3. Each of the cells contained a 400 series stainless steel ball which was used to mix the contents of the cell by means of a U-shaped magnet which is raised and lowered manually.

The equilibrium cell was connected to the steel tubing of the system through a metal-to-glass connector. The stainless steel connector was designed to have a small volume and to avoid leaks which might develop because of the different coefficients of thermal expansion of glass and metal. A Teflon O ring which was the only contact point between the metal and glass served as a reliable seal and an excellent vibration absorber.

Methods for obtaining a low temperature, controlling it at a constant value, and measuring it accurately have been reported before (*9*). The equilibrium cell was placed in a 0.009 46-m^3 Dewar flask filled with either commerical isopentane for low-temperature measurements (122–288 K) or ethylene glycol for measurements at higher temperatures (277–355 K).

Table I. Dew Points for Carbon Dioxide-Propane Mixture with y_{CO_2} = 0.076

press., MPa	temp, K	press., MPa	temp, K
0.059	270.4	0.820	291.5
0.076	225.4	0.965	297.0
0.096	232.0	1.192	306.0
0.131	238.6	1.379	310.9
0.145	242.9	1.578	316.8
0.196	247.2	1.781	322.0
0.245	254.4	2.037	327.8
0.289	263.7	2.292	333.7
0.283	263.7	2.581	339.8
0.414	271.7	2.578	339.8
0.414	271.7	2.892	346.4
0.520	277.6	2.892	346.5
0.520	277.5	3.243	349.8
0.820	291.6		

Table II. Bubble Points for Carbon Dioxide-Propane Mixture with x_{CO_2} = 0.076

press., MPa	temp, K	press., MPa	temp, K
0.289	213.2	1.217	283.2
0.303	217.8	1.327	287.9
0.307	217.8	1.327	287.5
0.345	224.8	1.609	298.1
0.407	230.3	1.606	298.1
0.455	235.9	1.761	303.2
0.448	235.9	1.761	303.7
0.524	241.3	2.037	310.9
0.589	248.7	2.037	310.9
0.689	254.7	2.440	322.1
0.758	260.9	2.440	322.0
0.882	266.4	2.847	329.6
1.006	271.2	3.216	338.7
1.061	277.6	3.574	345.2
1.227	283.2		

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 equipment.

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.

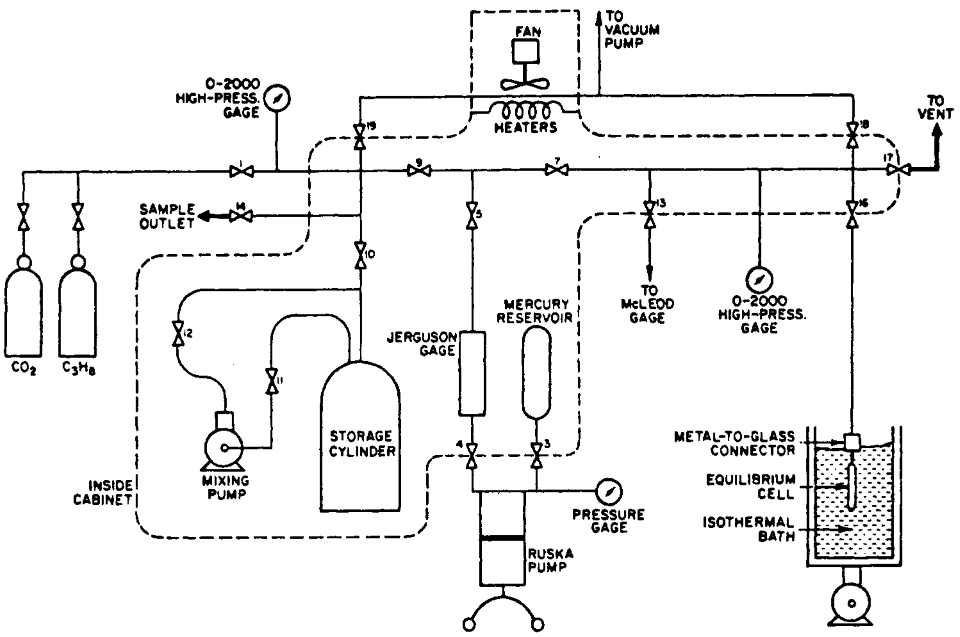
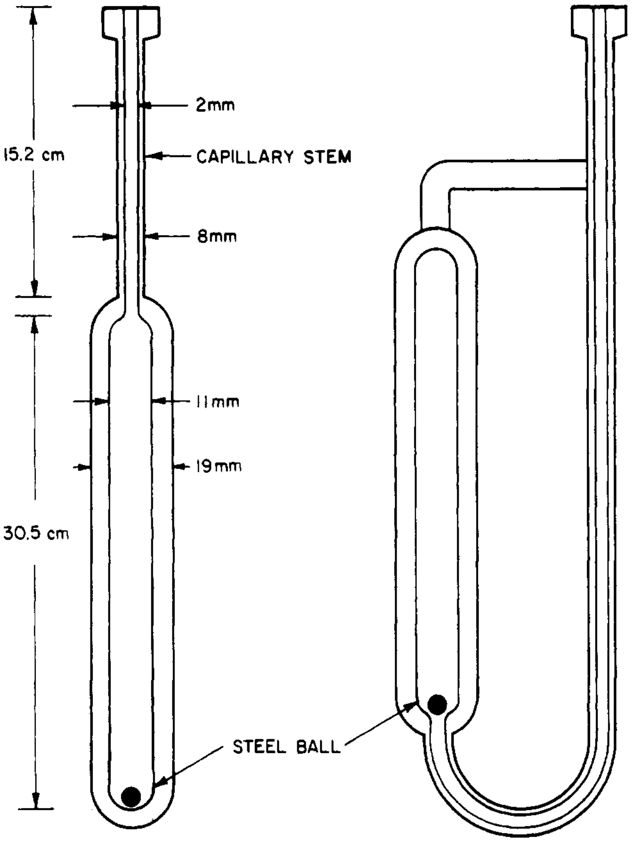


Figure 1. Schematic diagram of apparatus.



a. DEW POINT CELL b. BUBBLE POINT CELL

Figure 2. Dew and bubble point cells.

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.

The mixtures used in this study had, at certain pressures, dew point temperatures higher than room temperature. Thus, all tubing, the storage cylinder, the mixing pump, and the Jerguson gauge were installed in a cabinet which could be heated to 350 K in order to prevent condensation in the lines and to maintain the uniformity of the composition of the gas charged into the equilibrium cell. The cabinet could be heated by 12 electric resistance heaters to obtain the desired temperature.

The storage cylinder which was a high-pressure seamless steel gas cylinder with a volume of approximately 0.040 m^3 was equipped with a sample mixing manifold. The manifold consisted of a piping arrangement to provide inlet 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 pressure in the storage cylinder and in the Jerguson gauge and the pressure in the equilibrium cell were measured with two high-precision 0–13.79-MPa pressure gauges. The reported accuracy of the measurements was 0.1% of the full-scale reading.

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 4 (Figure 1) was closed and valve 3 was opened to bring the pressure of the mercury pump to atmospheric pressure.

Procedure. Mixture components were added to the storage cylinder in approximately the desired proportion and in an amount sufficient to make dew and bubble point measurements over the required range of temperatures and pressures. Once the components were charged into the storage cylinder, the gas mixture was circulated through the mixing pump for about 10 days. During the mixing period samples were taken periodically to be analyzed by gas chromatography until no significant difference in successive sample compositions was observed.

Before each run, the entire system except for the storage cylinder was leak tested. Leak testing was followed by complete evacuation of the part of the system which was under pressure. System evacuation was obtained by using the vacuum pump with valves 5, 7, 9, 13, 16, 18, and 19 open and all

Table III. Dew Points for Carbon Dioxide-Propane Mixture with $y_{CO_2} = 0.181$

press., MPa	temp, K	press., MPa	temp, K
0.062	216.3	0.986	294.3
0.072	219.5	1.117	299.1
0.086	222.4	1.117	298.7
0.107	227.6	1.131	299.8
0.127	233.2	1.241	303.2
0.169	238.7	1.227	303.2
0.210	244.3	1.406	307.2
0.262	249.8	1.565	311.1
0.317	255.4	1.565	311.8
0.386	260.9	1.668	313.9
0.469	266.5	1.902	318.4
0.558	272.0	1.902	317.9
0.658	277.6	2.047	322.4
0.800	286.2	2.454	330.4
0.800	285.7	2.964	338.7
0.841	288.7	3.371	344.3
0.937	291.5	3.843	349.8
0.934	291.5		

Table IV. Bubble Points for Carbon Dioxide-Propane Mixture with $x_{CO_2} = 0.181$

press., MPa	temp, K	press., MPa	temp, K
0.303	210.9	1.606	277.6
0.362	216.5	1.606	277.9
0.427	222.0	1.792	283.2
0.496	227.6	1.792	283.2
0.572	233.2	1.792	283.4
0.662	238.7	1.978	288.7
0.658	238.7	2.199	294.3
0.758	244.3	2.209	294.3
0.755	244.3	2.413	299.8
0.855	249.8	2.413	299.8
0.855	249.6	2.633	305.4
0.986	255.4	2.874	310.9
0.986	255.4	2.879	311.5
1.110	260.9	3.136	316.5
1.110	261.0	3.419	322.0
1.248	266.5	3.701	327.6
1.482	272.0	3.984	333.2
1.475	272.0	4.308	338.7
1.478	272.0	4.608	344.3

other valves closed. (See Figure 1.) Once the complete evacuation was achieved, valves 18 and 19 were closed. Then a small amount of the mixture was introduced into the system and evacuated again. This procedure was repeated following any change of equilibrium cell.

Depending on the sensitivity of the pressure-temperature curve to either pressure or temperature, either of two modes of operation were employed to obtain measurements. At low temperatures, the curve is sensitive to pressure; as a result the constant-temperature operation leads to more accurate measurements. At high temperatures, the curve is sensitive to temperature, which suggests the use of the constant-pressure method.

The visual observation techniques employed in this work for the determination of dew and bubble points have been successfully employed by a previous investigator (*9*).

In the constant-temperature operation for the determination of the dew point, the temperature of the cell was maintained at a preselected value. A previous investigator (*8*) recommended a 5-min period to allow the temperature of the contents of the equilibrium cell to reach the temperature of the bath fluid. This criterion was used in this work whenever the temperature of the cabinet was less than 311 K. When the temperature of the cabinet was higher, a 7-min period was used to allow the thermal equilibrium between the cell contents and the bath fluid.

Once thermal equilibrium was reached, the pressure was increased by means of the Jerguson gauge until a thin liquid ring was seen around the steel ball when it was in contact with the neck of the equilibrium cell. During the process, the contents of the cell were continuously agitated by raising and lowering the U-magnet and, hence, moving the steel ball up and down.

Once the liquid ring was observed, the pressure in the equilibrium cell was decreased slightly and the mixture agitated. This procedure was repeated until the thin ring disappeared. The pressure was then increased until the ring was observed again. At this point the temperature and pressure were recorded as the dew point parameters of the mixture. Increasing the pressure beyond this point resulted in the formation of droplets in the cell. Usually the liquid ring would appear and disappear within a pressure change of 0.007 MPa.

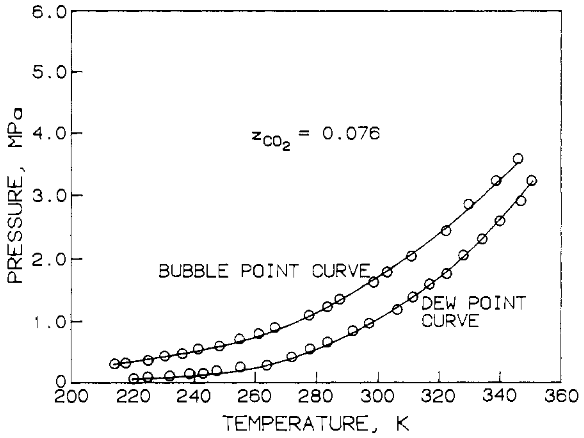


Figure 3. Dew and bubble points for carbon dioxide-propane mixture; $z_{CO_2} = 0.076$.

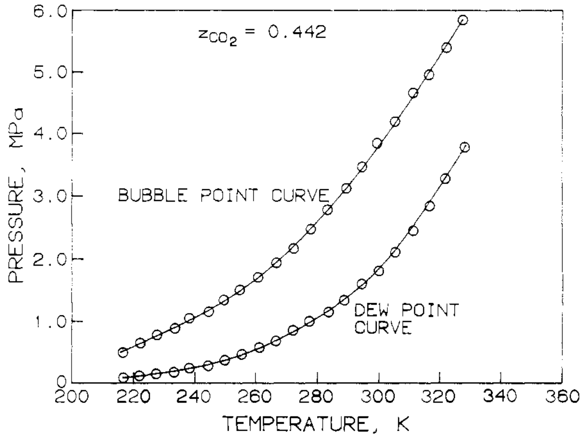


Figure 4. Dew and bubble points for carbon dioxide-propane mixture; $z_{CO_2} = 0.442$.

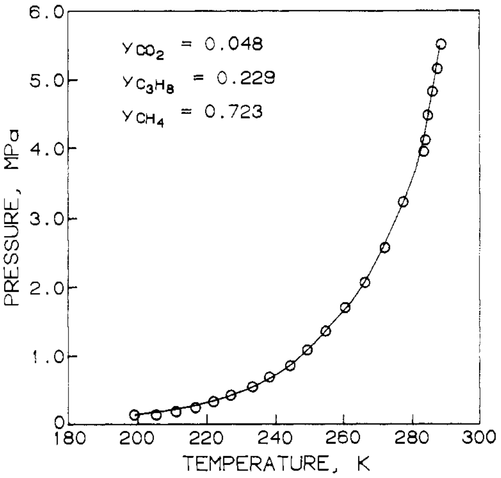


Figure 5. Dew points for carbon dioxide-propane-methane mixture.

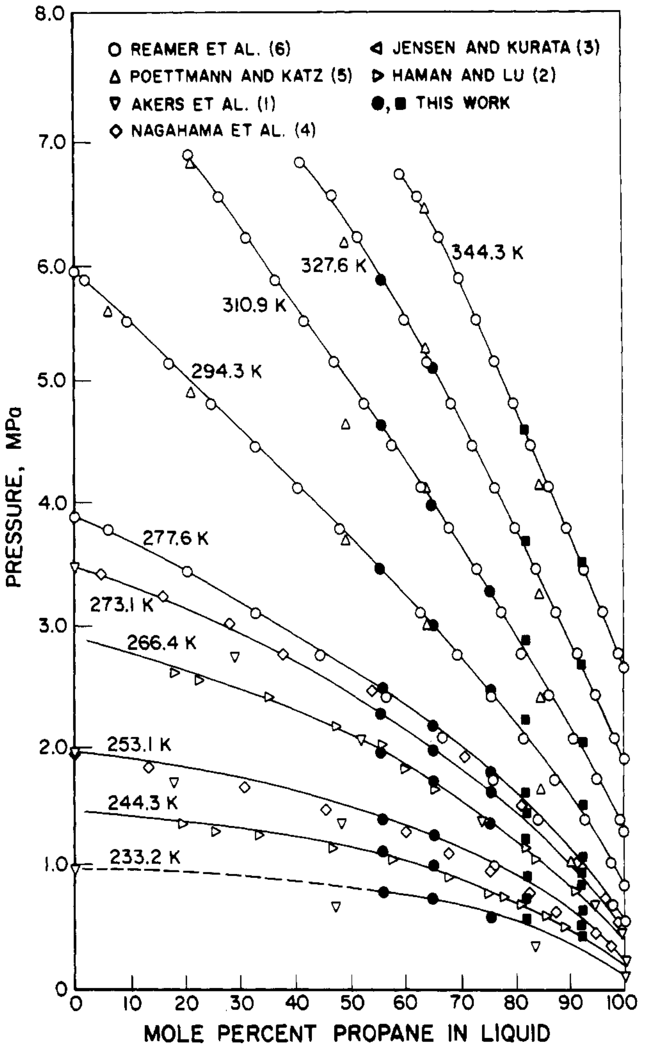


Figure 6. Comparison of bubble point data for carbon dioxide–propane system.

In constant-pressure operation, the pressure of the cell was maintained at a preselected value. Once the desired pressure was reached, the temperature was decreased until a liquid ring appeared. During the process, the contents of the cell were thoroughly agitated. When the ring appeared, the temperature was increased until the liquid ring disappeared. Then the temperature was decreased again until the formation of a thin ring was observed at the contact line of the glass neck and the steel ball. These values of pressure and temperature were recorded as the dew point of the mixture. Usually the ring would appear and disappear within a temperature change of ±0.05 K.

In the constant-temperature operation for the determination of the bubble point, the temperature of the cell was maintained at a preselected value. The pressure of the cell was increased while the mixture was agitated. More gas was transferred to the equilibrium cell and the pressure increased. This process was repeated until a very large bubble of vapor was seen at the top of the cell. After this a small amount of gas mixture was introduced into the equilibrium cell and the contents agitated again until a new equilibrium state was reached with a smaller bubble at the top of the cell.

During the above period the contents of the cell were agitated continuously. The transfer of gas to the cell was continued until the size of the vapor bubble was reduced and finally disappeared. Once the bubble disappeared, the pressure in the equilibrium cell was decreased slightly and the mixture agitated. This procedure was repeated until the bubble appeared again. The pressure was then increased again until the bubble disappeared one more time. The pressure and the temperature were recorded at this point and were considered to be the bubble point of the mixture. Typically, bubble point pressures were reproducible to ±0.007 MPa.

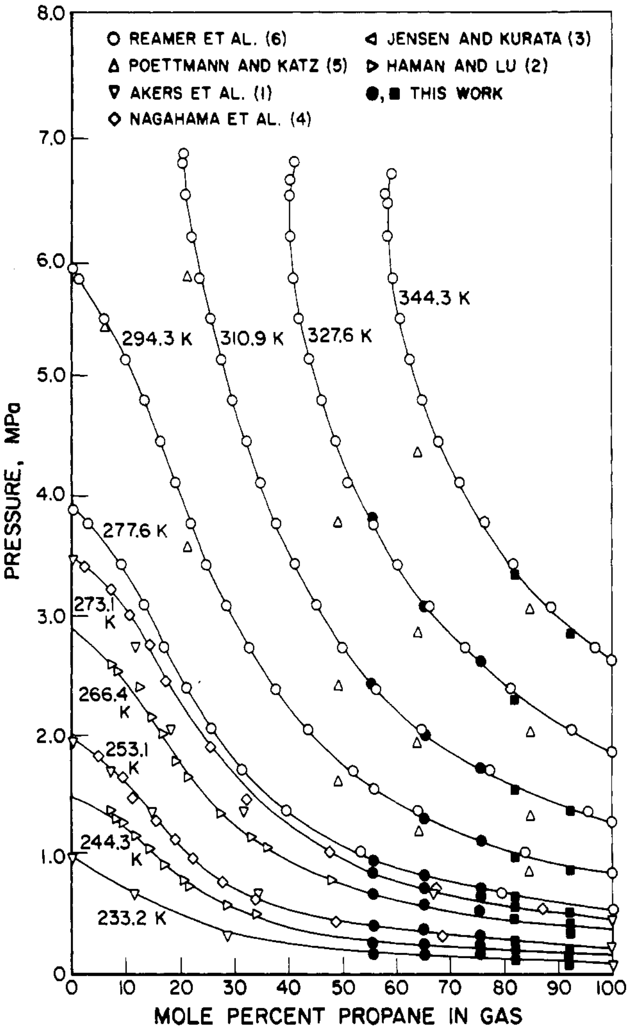


Figure 7. Comparison of dew point data for carbon dioxide–propane system.

Table V. Dew Points for Carbon Dioxide–Propane Mixture with $y_{CO_2} = 0.244$

press., MPa	temp, K	press., MPa	temp, K
0.083	216.5	0.724	272.0
0.090	216.5	0.717	277.6
0.103	222.0	0.827	277.6
0.117	222.0	0.837	283.2
0.124	227.6	0.979	288.7
0.152	227.6	0.979	288.7
0.165	233.2	1.117	294.3
0.179	233.2	1.296	299.8
0.193	238.7	1.554	305.4
0.207	238.7	1.744	310.9
0.255	244.3	2.006	316.5
0.310	249.8	2.309	322.0
0.379	255.4	2.619	327.6
0.455	260.9	2.650	327.6
0.538	266.5	2.950	333.2
0.631	266.5	2.981	333.2

In constant-pressure operation, the cell was cooled to bring the mixture into the two-phase region while the pressure ws held constant by introducing more gas to the cell. As cooling continued, the amount of liquid formed in the cell increased until a very small bubble of gas was observed at the top of the cell. The contents of the cell were agitated continuously until the

Table VI. Bubble Points for Carbon Dioxide–Propane Mixture with $x_{CO_2} = 0.244$

press., MPa	temp, K	press., MPa	temp, K
0.334	216.5	1.778	277.6
0.424	222.0	1.792	277.6
0.482	227.6	1.964	283.2
0.579	233.2	1.968	283.2
0.675	238.7	2.192	288.7
0.786	244.3	2.192	288.7
0.917	249.8	2.457	294.3
1.058	255.4	2.695	299.8
1.199	260.9	2.985	305.4
1.379	266.5	3.267	310.9
1.385	266.5	3.543	316.5
1.578	272.0	3.846	322.0

Table VII. Dew Points for Carbon Dioxide–Propane Mixture with $y_{CO_2} = 0.349$

press., MPa	temp, K	press., MPa	temp, K
0.076	216.5	0.965	283.2
0.103	222.0	1.130	288.7
0.131	227.6	1.323	294.3
0.172	233.2	1.530	299.8
0.214	238.7	1.744	305.4
0.269	244.3	2.020	310.9
0.331	299.8	2.333	316.5
0.403	255.4	2.702	322.0
0.496	260.9	2.709	322.0
0.592	266.5	3.102	327.6
0.710	272.0	3.102	327.6
0.689	272.0	3.557	333.2
0.827	277.6	3.571	333.2

Table VIII. Bubble Points for Carbon Dioxide–Propane Mixture with $x_{CO_2} = 0.349$

press., MPa	temp, K	press., MPa	temp, K
0.448	216.5	2.164	277.6
0.538	222.0	2.416	283.2
0.648	227.6	2.702	288.7
0.751	233.2	2.992	294.3
0.882	238.7	3.316	299.8
1.020	244.3	3.650	305.4
1.179	249.8	3.991	310.9
1.344	255.4	4.360	316.5
1.534	260.9	4.370	316.5
1.720	266.5	4.722	322.0
1.937	272.0	4.715	322.0
1.930	272.0	5.121	327.6
2.171	277.6		

Table IX. Dew Points for Carbon Dioxide–Propane Mixture with $y_{CO_2} = 0.442$

press., MPa	temp, K	press., MPa	temp, K
0.076	216.5	0.979	277.6
0.096	222.0	1.137	283.2
0.131	227.6	1.344	288.7
0.176	233.2	1.568	294.3
0.221	238.7	1.827	299.8
0.290	244.3	2.109	305.4
0.358	249.8	2.447	310.9
0.455	255.4	2.826	316.5
0.558	260.9	2.826	316.5
0.676	266.5	3.288	322.0
0.817	272.0	3.281	322.0
0.831	272.0	3.812	327.6
0.965	277.6		

Table X. Bubble Points for Carbon Dioxide–Propane Mixture with $x_{CO_2} = 0.442$

press., MPa	temp, K	press., MPa	temp, K
0.472	216.5	2.220	272.0
0.572	222.0	2.492	277.6
0.572	222.0	2.488	277.6
0.686	227.6	2.785	283.2
0.813	233.2	3.123	288.7
0.958	238.7	3.460	294.3
0.962	238.7	3.846	299.8
1.124	244.3	4.198	305.4
1.303	249.8	4.653	310.9
1.496	255.4	4.978	316.5
1.496	255.4	4.980	316.5
1.713	260.9	5.409	322.0
1.951	266.5	5.411	322.0
2.217	272.0	5.859	327.6

Table XI. Dew Points for a Carbon Dioxide–Propane–Methane Mixture with $y_{CO_2} = 0.048$ and $y_{C_3H_8} = 0.229$

press., MPa	temp, K	press., MPa	temp, K
0.103	199.8	2.089	266.5
0.141	205.4	2.592	272.0
0.138	205.4	2.585	272.0
0.193	210.9	3.233	277.6
0.248	216.5	3.219	277.6
0.324	222.0	3.977	283.2
0.424	227.6	4.136	283.4
0.544	233.2	4.480	284.9
0.696	238.7	4.825	286.2
0.869	244.3	5.170	287.5
1.082	249.8	5.514	289.1
1.358	255.4	5.514	289.0
1.703	260.9		

bubble disappeared. When this occurred, the temperature was increased until the bubble reappeared. Then the temperature was decreased again until the bubble disappeared once more. At the given pressure, the temperature at which the bubble disappeared was identified as the bubble point temperature.

In the experiments in which solid formation was observed, the pressure was kept constant at a preselected value and the temperature was lowered until tiny solid crystals were observed. The pressure and temperature at this point were recorded. The temperature was then increased and the temperature at which the solids disappeared was also recorded.

Chemicals Used. High-quality, research-grade gases were used for the preparation of the mixtures and as references for the gas chromatograph. All gases were purchased from the Matheson Co. and the reported purities of the carbon dioxide, propane, and methane were respectively 99.995%, 99.99%, and 99.99%.

Mixture Analysis. Gas chromatography was used for the determination of mixture compositions. The chromatography unit consisted of an Aerograph A90-P3 gas chromatograph with a thermal conductivity detector. For binary mixtures, the column was made from 0.635-cm-diameter stainless steel tubing of 0.33-m length filled with 80–100 mesh Porapack-Q, which is sensitive to air, methane, propane, and carbon dioxide. The column used for the ternary mixture was constructed from 0.317-cm-diameter stainless steel tubing of 0.30-m length filled with 100–120 mesh Porapack-T, which is also sensitive to air, methane, propane, and carbon dioxide but allows a better separation between the methane and carbon dioxide peaks. The retention time and peak area were recorded by a Hewlett-Packard Series 3370 integrator. A Sargent SR recorder connected to the integrator was used for visual observation of peak shape, separation, and base line. The carrier gas was a high purity grade helium with a reported purity of at least 99.995%. The estimate of the standard deviation of the mole fraction measurement was 0.001.

Results and Discussion

The experimentally determined dew and bubble points for the carbon dioxide–propane mixtures are presented in Tables I–X and are shown graphically for two of the mixtures in Figures 3 and 4. Dew points for the ternary mixture of carbon dioxide, propane, and methane are presented in Table XI and shown graphically in Figure 5. 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.

Pressure–composition diagrams were constructed for 10 isotherms by using the data from the literature and the dew and bubble point data obtained in this work. In order to incorporate the data of this work, the pressure values were taken from the intersection of the given constant-temperature lines with the dew and bubble point curves for each mixture. Figure 6 presents *P–x* curves for several temperatures and Figure 7 presents *P–y* curves for several temperatures. In general, good agreement was obtained between the data presented in this investigation and the data of the other investigators.

Solid formation experiments were conducted over the pressure range 0.124–0.165 MPa. The experiments were conducted for each of the binary mixtures investigated in this study. The temperature of a given mixture was lowered at constant pressure until solid crystals appeared. The mixtures were then heated at constant pressure until the solids disappeared. In general the appearance temperature was several degrees lower than the disappearance temperature. The temperatures at which solid appeared range from 183.1 to 198.3 K and the temperatures at which solid disappeared varied from 194.3 to 201.4 K. Solid formation temperatures increased as the carbon dioxide concentration increased.

Registry No. Propane, 74-98-6; methane, 74-82-8; carbon dioxide, 124-38-9.

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Solubility of Carbon Dioxide in Aqueous Diethanolamine Solutions at Elevated Temperatures and Pressures

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The solubility of carbon dioxide in 10, 20, and 30 wt % aqueous diethanolamine (DEA) solutions has been determined for elevated temperatures (100–205 °C) and CO_2 partial pressures (70–4000 kPa). The results were acquired by a simple experimental procedure and agree, on the average, within 10% of literature values reported for more moderate temperatures and pressures.

Introduction

Aqueous diethanolamine (DEA) solutions are used extensively for the removal of H_2S and CO_2 from raw natural gas and other light hydrocarbon mixtures. Knowledge of the equilibrium solubility of these acid gases in DEA solution is essential for the proper design and operation of absorption and desorption equipment. Furthermore, many gas treating plants experience problems with DEA degradation, i.e., the irreversible reaction of DEA with CO_2. To understand degradation, which is particularly serious at elevated temperatures and pressures, it is also necessary to have a good knowledge of CO_2 solubility in DEA solutions. The solubility data presented in this paper were acquired for use in a recent study on DEA degradation (*1*).

Data on CO_2 solubility in aqueous DEA solutions have been reported by several investigators, but no results have been published for elevated temperatures and pressures. Table I summarizes the ranges of experimental variables investigated by previous researchers. The present study provides CO_2 solubility data under the following conditions: DEA concentration 10–30 wt %; Temperature, 100–205 °C; CO_2 partial pressure, 70–4000 kPa.

Table I. Summary of Experimental Conditions Used by Previous Researchers

researcher	DEA concn, wt %	temp, °C	CO_2 partial press., kPa
Bottoms (*3*)	50	25–55	1.33–101.33
Mason and Dodge (*4*)	5–80	0–75	1.33–101.33
Reed and Wood (*5*)	25	100–140	241.3–1571.3
Dow (*6*)	5–40	20–100	1.33–400
Murzin and Leites (*7*)	5–80	20–90	0.0133–93.33
Lee et al. (*8*)	5–50	25–120	0.689–5707
Lawson and Garst (*9*)	25	38–121	2.0–4266

Apparatus

Aqueous DEA solutions were prepared by mixing distilled water and commercial-grade DEA which had a purity in excess of 98%. The carbon dioxide had a minimum purity of 99.5%.

The equipment used in this work consisted essentially of a 600-mL stirred, stainless steel autoclave (supplied by the Parr Instrument Co., Moline, IL, Model 4560; see Figure 1). The autoclave, which was equipped with an electric heating mantle, could be operated at temperatures ranging from ambient to 400 °C. The temperature was controlled to within ±0.5 °C by an automatic controller whose output was displayed on a digital

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