higher cyclohexane concentrations.

Glossary

h^E	excess enthalpy
g^E	excess Gibbs free energy
R	gas constant
T	temperature
x_i	mole fraction of component i
P	pressure
a_{ij}	adjustable parameter for i-j interactions
h_{ij}	adjustable parameter for i-j interactions
G_{ij}	nonrandomness weighting factor defined by eq 5
b_{ij}	adjustable parameter for i-j interactions
T_{ref}	reference temperature for EBLCM model
z	coordination number

Greek Letters

α	defined in eq 6
β	defined in eq 7
γ_i	activity coefficient of component i
δ	defined in eq 8
λ	defined in eq 9

Registry No. Cyclohexane, 110-82-7; 2-propanol, 67-63-0.

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Vapor-Liquid Equilibria in Mixtures Containing Carbon Dioxide, Toluene, and 1-Methylnaphthalene

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Vapor-liquid equilibrium compositions were determined at pressures ranging from 259 to 14 450 kPa for three systems: the binaries carbon dioxide-toluene and carbon dioxide-1-methylnaphthalene; and the ternary carbon dioxide-toluene-1-methylnaphthalene. Binary equilibrium compositions are reported at 353.15, 383.15, and 413.15 K for the carbon dioxide-toluene system, and 353.15 and 413.15 K for the carbon dioxide-1-methylnaphthalene system. The liquid-phase solubility of carbon dioxide is found to be greater in toluene than in 1-methylnaphthalene. The ternary system was investigated at 353.15 and 413.15 K and with two initial liquid molar ratios of 1-methylnaphthalene to toluene: 9 to 1, and 4 to 1. The presence of toluene in the ternary system is found to increase the liquid-phase solubility of carbon dioxide. The vapor-phase solubility of 1-methylnaphthalene at 353.15 K is determined to be greater because of the presence of toluene, while the solubility at 413.15 K is found to be inhibited by the presence of toluene.

Introduction

Carbon dioxide is found in abundance in the natural gas and petroleum industries. Heavy aromatic compounds are also frequently encountered in these industries. The efficient design of separation and other processing equipment often requires accurate high-pressure vapor-liquid equilibrium data. High-pressure binary vapor-liquid equilibrium data on carbon dioxide-aromatic systems are scarce, and ternary equilibrium data on carbon dioxide-aromatic-aromatic systems are virtually nonexistent.

In this paper, we report vapor-liquid equilibrium data on three systems: the binary carbon dioxide-toluene; the binary carbon dioxide-1-methylnaphthalene; and the ternary carbon dioxide-toluene-1-methylnaphthalene. Equilibrium data are reported at temperatures of 353.15 (80.0 °C), 383.15 (110.0 °C), and 413.15 (140.0 °C) K, and pressures ranging from about 207 (30 psia) to 14 480 (2100 psia) kPa. The ternary system is investigated to determine how small amounts of toluene, when added to the carbon dioxide-1-methylnaphthalene system, affect the equilibrium compositions.

Ng and Robinson (*1*) report similar data on the carbon dioxide-toluene system at temperatures of 311.26 (38.11 °C), 352.56 (79.44 °C), 393.71 (120.56 °C), and 477.04 (203.89 °C) K. Tremper and Prausnitz (*2*) report the solubility of carbon dioxide in 1-methylnaphthalene at low pressures and temperatures up to 473.15 K (200 °C). Sebastian and co-workers (*3*) report the vapor-liquid equilibrium of the carbon dioxide-1-methylnaphthalene system at temperatures of 463.05 (189.9 °C), 543.45 (270.3 °C), 623.55 (350.4 °C), and 703.55 (430.4 °C) K, but pressures ranging from 2070 (300 psia) to only 5170 (750 psia) kPa. The only data found in the literature for a

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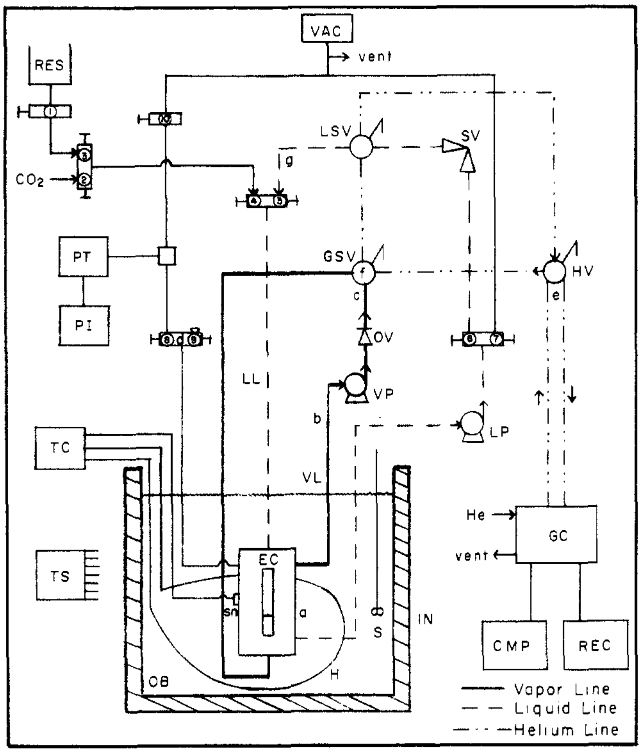

Figure 1. Schematic diagram of experimental apparatus: (CMP) computer; (EC) equilibrium cell; (GC) gas chromatograph; (GSV) gas sampling valve; (H) heater; (He) helium; (HV) helium switch valve; (IN) insulation; (LL) liquid line; (LP) liquid pump; (LSV) liquid sampling valve; (OB) oil bath; (OV) one-way valve; (PI) pressure indicator; (PT) pressure transducer; (REC) chart recorder; (RES) liquid reservoir; (S) stirrer; (sn) temperature sensor; (SV) safety valve; (TC) temperature controller; (TS) thermocouple switch; (VAC) vacuum pump; (VL) vapor line; (VP) vapor pump; (1–10) valve numbers; and (a–g) thermocouples.

ternary system similar to carbon dioxide–toluene–1-methylnaphthalene are those reported by Yang and co-workers (*4*). They studied the ternary system of carbon dioxide–butylbenzene–2-methylnaphthalene and report the liquid-phase solubility of carbon dioxide at temperatures of 308.15 (35 °C) and 323.15 (50 °C) K and pressures upto 6080 kPa (880 psia).

Experimental Section

The experimental apparatus used in this work is shown in Figure 1. The equilibrium cell has an internal volume of about 100 cm^3 and is equipped with two transparent glass windows. The pressure limits for the cell are 10 860 kPa (1575 psia) at 588.15 K (315 °C), and 14 690 kPa (2130 psia) at 311.15 K (38 °C). Equilibrium is attained rapidly (10–15 min) because the vapor and liquid phases are both circulated. The vapor is circulated at a flow rate of about 60 cm^3/min using a electromagnet-driven piston-type pump. The vapor circulation pump design is similar to that described by Street and Erickson (*5*). The liquid is circulated by using a high-pressure pump at a flow rate of about 6 cm^3/min. The equilibrium cell is contained in a constant-temperature oil bath. The oil bath is surrounded by about 2 in. of Fiberglas insulation; 100- and 200-W immersible electric heaters are placed in the oil bath. The bath temperature is controlled to within ±0.1 °C with a temperature controller. All piping lines are 316 stainless steel tubing with an outer diameter of $^1/_8$ in. and a wall thickness of 0.035 in. All lines outside the oil bath are heated with heavy-duty heating tape. Temperatures throughout the system are determined relative to a water–ice mixture with copper–constantan thermocouples. The pressure of the system is measured with a strain gage pressure transducer and a digital indicator. The maximum error is ±10 kPa.

Vapor and liquid samples are injected directly into the gas chromatograph using sampling valves placed in both the vapor and liquid lines. External sampling loops of 20 and 250 μL are used in the vapor sampling valve. The larger loop is used when the vapor composition of the heavy component is very small. The liquid sampling valve has an external loop of 0.5 μL. Helium carrier gas transports the vapor and liquid samples directly to a gas chromatograph.

The gas chromatograph, connected to a chart recorder and a mircoprocessor, uses a thermal conductivity detector. The microprocessor integrates peak area using a 12-bit analog-to-digital converter. The thermal conductivity detector was calibrated for each pure component by injecting known amounts and determining peak areas. Curves showing moles vs. peak area were then constructed and fitted to third-order polynomials in peak area. Compositions are determined by using these equations to calculate the moles of each compound present in the sample.

Injections from each phase were usually repeated at least twice for each equilibrium point. The average of the individual compositions is reported as the equilibrium composition. The repeatability of the analysis for the carbon dioxide–toluene system was generally within a mole fraction of 0.002. The liquid-phase compositions for the carbon dioxide–1-methylnaphthalene system were reproducible to within a mole fraction of 0.001. The vapor-phase compositions for this system are reproducible to within a mole fraction of 0.000 02 at 353.15 K (80.0 °C), and 0.0001 at 413.15 K (140.0 °C). The liquid-phase compositions for the ternary system are reproducible to within a mole fraction of 0.002. The vapor-phase compositions for the ternary system are reproducible to within a mole fraction of 0.000 04 at 353.15 K (80.0 °C), and 0.0002 at 413.15 K (140.0 °C). The accuracy of the equilibrium compositions is believed to be within 1.5 times the reproducibility.

Experiments were performed on the ternary system by charging the cell with a toluene–1-methylnaphthalene mixture of known mole fraction. The cell was then pressurized with carbon dioxide. Data were taken with initial 1-methylnaphthalene to toluene mole ratios of 9 to 1 and 4 to 1.

The carbon dioxide used in this study is supplied by Matheson (Coleman Instrument Grade), with a minimum purity of 99.99%. The toluene used is Baker Analyzed, with a purity of 99.9%. The 1-methylnaphthalene, supplied by Aldrich Chemical Co., has a purity of 99%. These were used without further purification.

A more detailed description of the experimental apparatus and procedures is given by Morris (*6*).

Experimental Results and Discussion

The experimental vapor–liquid equilibrium compositions and *K* values for the binary system carbon dioxide–toluene are reported in Table I. These data are given for temperatures of 353.15 (80.0 °C), 383.15 (110.0 °C), and 413.15 (140.0 °C) K. The pressures range from 259 (37.5 psia) to 13 170 (1910 psia) kPa. The data for these three isotherms are shown graphically in Figure 2 as a P–x–y diagram. Each of the isotherms is extrapolated to the critical point. The isotherms are also extrapolated to a toluene mole fraction of 1 by using the vapor pressure data reported by Vargaftik (*7*). In Figure 3, the equilibrium data are plotted as a *K* factor vs. pressure diagram.

The isothermal liquid compositions shown in Figure 2 are linear at conditions remote from the mixture critical point. This indicates that the carbon dioxide–toluene system nearly follows Henry's law. Figure 2 shows that the solubility of carbon dioxide in toluene decreases with an increase in temperature. The critical pressure of the mixture increases with temperature over the range of temperatures investigated. The compositions of the critical points in the temperature range of 353.15–413.15 K (80.0–140.0 °C) are between a carbon dioxide mole fraction

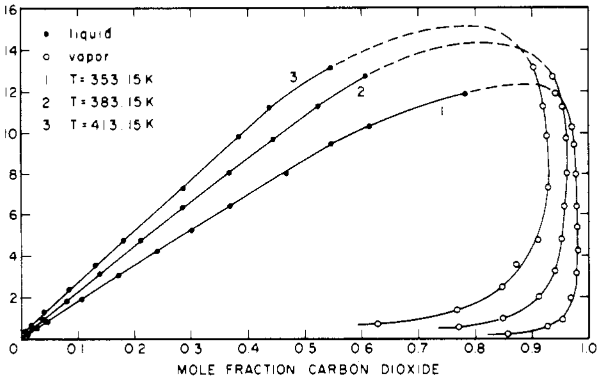

Figure 2. Pressure-composition diagram for carbon dioxide-toluene system.

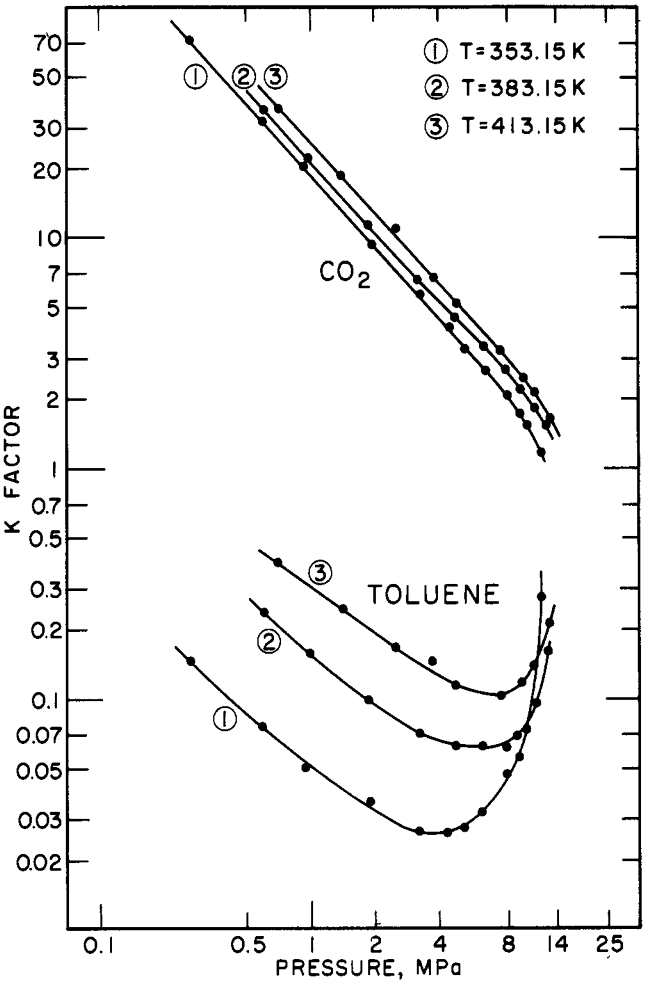

Figure 3. *K* factor-pressure diagram for carbon dioxide-toluene system.

of 0.7 and 0.9. The carbon dioxide composition at the critical point decreases with an increase in temperature.

Ng and Robinson (*1*) report high-pressure vapor-liquid equilibrium data for the carbon dioxide-toluene system at a temperature of 352.6 K (79.44 °C). Their data for this isotherm agree well with the data reported here for 353.15 K (80.0 °C). A comparison of the two is shown in Figure 4. The data are generally in agreement to within a mole fraction of 0.02.

The experimental vapor-liquid equilibrium data for the binary system carbon dioxide-1-methylnaphthalene are presented in Table II. Data are reported at isotherms of 353.15 (80.0 °C) and 413.15 (140.0 °C) K with pressures ranging from 1220 (177 psia) to 14450 (2096 psia) kPa. Figure 5 shows the solubility of carbon dioxide in 1-methylnaphthalene in the form

Table I. Vapor-Liquid Equilibrium Data for Carbon Dioxide-Toluene System

P, kPa	$x(CO_2)$	$y(CO_2)$	$K(CO_2)$	K(Tol)
T = 353.15 K				
259	0.0120	0.856	71.4	0.146
579	0.0287	0.926	32.3	0.0758
905	0.0463	0.952	20.5	0.0505
1950	0.105	0.968	9.18	0.0361
3125	0.170	0.978	5.76	0.0262
4290	0.239	0.980	4.11	0.0264
5350	0.300	0.981	3.27	0.0276
6475	0.366	0.980	2.68	0.0314
8040	0.468	0.975	2.08	0.0467
9495	0.546	0.974	1.78	0.0573
10335	0.613	0.971	1.58	0.0754
11930	0.783	0.939	1.20	0.279
T = 383.15 K				
593	0.0217	0.771	35.6	0.234
972	0.0385	0.847	22.0	0.156
1905	0.0807	0.908	11.3	0.100
3180	0.140	0.938	6.70	0.0722
4815	0.210	0.951	4.53	0.0622
6405	0.284	0.955	3.37	0.0624
8120	0.366	0.961	2.63	0.0617
9735	0.443	0.961	2.17	0.0698
11300	0.522	0.953	1.83	0.0973
12800	0.607	0.936	1.54	0.163
T = 413.15 K				
710	0.0176	0.623	35.4	0.384
1340	0.0409	0.766	18.7	0.244
2465	0.0835	0.847	10.1	0.167
3590	0.130	0.872	6.70	0.147
4790	0.177	0.908	5.12	0.112
7315	0.286	0.928	3.18	0.102
9865	0.383	0.926	2.42	0.119
11315	0.437	0.921	2.11	0.140
13170	0.546	0.904	1.66	0.212

Table II. Vapor-Liquid Equilibrium Data for Carbon Dioxide-1-Methylnaphthalene System

P, kPa	$x(CO_2)$	$y(CO_2)$	$K(CO_2)$	K(1-Mn)
T = 353.15 K				
1815	0.0825	0.999 663	12.1	0.000 367
2930	0.132	0.999 718	7.59	0.000 325
4980	0.205	0.999 730	4.89	0.000 339
8280	0.323	0.999 431	3.10	0.000 840
11265	0.414	0.998 34	2.41	0.002 83
14425	0.488	0.996 00	2.04	0.007 81
T = 413.15 K				
1220	0.0459	0.994 22	21.7	0.006 06
2250	0.0772	0.996 15	12.9	0.004 17
4220	0.132	0.997 37	7.58	0.003 03
6000	0.183	0.997 55	5.44	0.003 00
8410	0.244	0.997 20	4.09	0.003 70
11935	0.330	0.996 05	3.02	0.005 89
14450	0.381	0.994 55	2.61	0.008 77

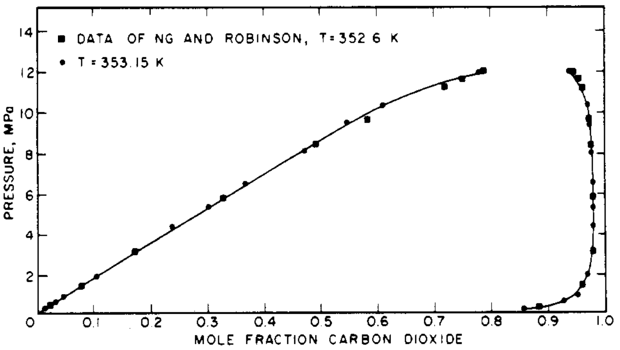

Figure 4. Comparison of measured experimental data of carbon dioxide-toluene system with that of Ng and Robinson (*1*).

Table III. Vapor–Liquid Equilibrium Data for Carbon Dioxide–Toluene-1-Methylnaphthalene ($Z_{1\text{-MN}}/Z_{\text{Tol}}$ = 9) System

P, kPa	$x(CO_2)$	$y(CO_2)$	x(Tol)	y(Tol)	x(1-Mn)	y(1-Mn)	$K(CO_2)$	K(Tol)	K(1-MN)
T = 353.15 K									
2165	0.1024	0.997 589	0.0583	0.001 536	0.8393	0.000 875	9.74	0.0263	0.001 14
4235	0.1969	0.998 223	0.0526	0.000 994	0.7505	0.000 783	5.07	0.0189	0.001 04
6530	0.2857	0.998 249	0.0471	0.000 978	0.6672	0.000 773	3.49	0.0208	0.001 16
9865	0.4154	0.997 17	0.0363	0.001 45	0.5483	0.001 38	2.40	0.0400	0.002 51
13080	0.4999	0.994 40	0.0298	0.002 78	0.4703	0.002 82	1.99	0.0931	0.006 01
T = 413.15 K									
2605	0.0902	0.991 46	0.0603	0.005 87	0.8495	0.002 67	11.00	0.0973	0.003 15
5880	0.1986	0.994 11	0.0493	0.003 69	0.7521	0.002 20	5.00	0.0749	0.002 93
9300	0.2919	0.993 92	0.0471	0.003 52	0.6610	0.002 56	3.41	0.0746	0.003 87
12790	0.3812	0.991 69	0.0364	0.004 22	0.5824	0.004 09	2.60	0.116	0.007 02

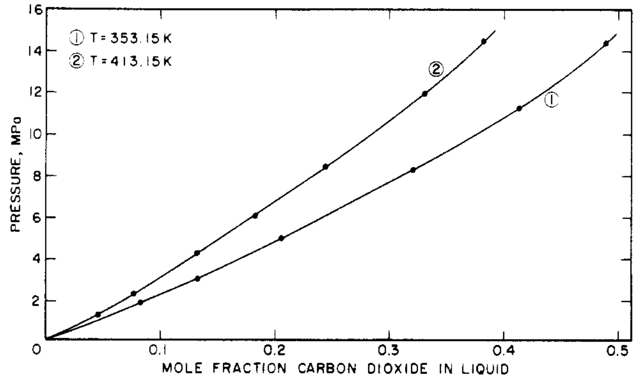

Figure 5. Pressure–liquid composition diagram for carbon dioxide–1-methylnaphthalene system.

of a P–x diagram, and Figure 6 shows the equilibrium vapor compositions of carbon dioxide in the form of a P–y diagram. A K factor vs. pressure plot for these data is shown in Figure 7.

The solubility of carbon dioxide in 1-methylnaphthalene is found to decrease with an increase in temperature. The critical pressures of the two isotherms appear to be high enough so that an extrapolation to these points cannot be made. The curvature in Figure 5 suggests that the carbon dioxide–1-methylnaphthalene system exhibits negative deviations from Raoult's law. The data reported by Sebastian and co-workers (*3*), at higher temperatures and pressures up to 5170 kPa (750 psia), show similar behavior.

Negative deviations from Raoult's law occur when the liquid-phase activity coefficient of the light component is less than 1. This normally occurs when the energy of attraction between molecules in the mixture are greater than the sum of the attractive energies between the pure components, or when the size difference between the molecules in the mixture is large. The entropic (size) effect is believed to be the cause of the negative deviations in the carbon dioxide–1-methylnapthalene system. For the carbon dioxide–toluene system, the enthalpic and entropic terms nearly cancel, resulting in the straight-line behavior shown in Figure 2. Calculations using regular solution theory with Flory–Huggins entropy of mixing roughly predict these types of behavior.

A comparison of Figures 2 and 5 indicates that carbon dioxide is more soluble in toluene than in 1-methylnaphthalene. Also, the critical pressures of the carbon dioxide–1-methylnaphthalene system are much higher than the carbon dioxide–toluene system.

The ternary system was investigated to determine how small amounts of toluene added to the carbon dioxide–1-methylnaphthalene system affect the equilibrium compositions. The primary interest was to determine the solubility enhancement of 1-methylnaphthalene in the vapor phase, compared to the binary carbon dioxide–1-methylnaphthalene system.

The ternary equilibrium data with an initial 1-methylnaphthalene to toluene mole ratio of 9 to 1 is reported in Table

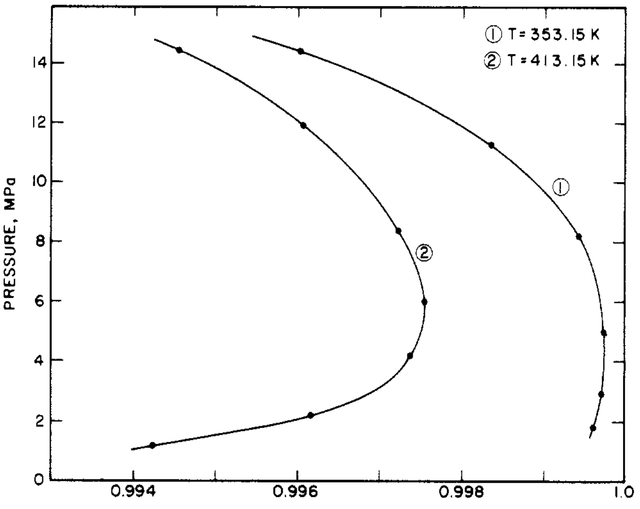

Figure 6. Pressure–vapor composition diagram for carbon dioxide–1-methylnaphthalene system.

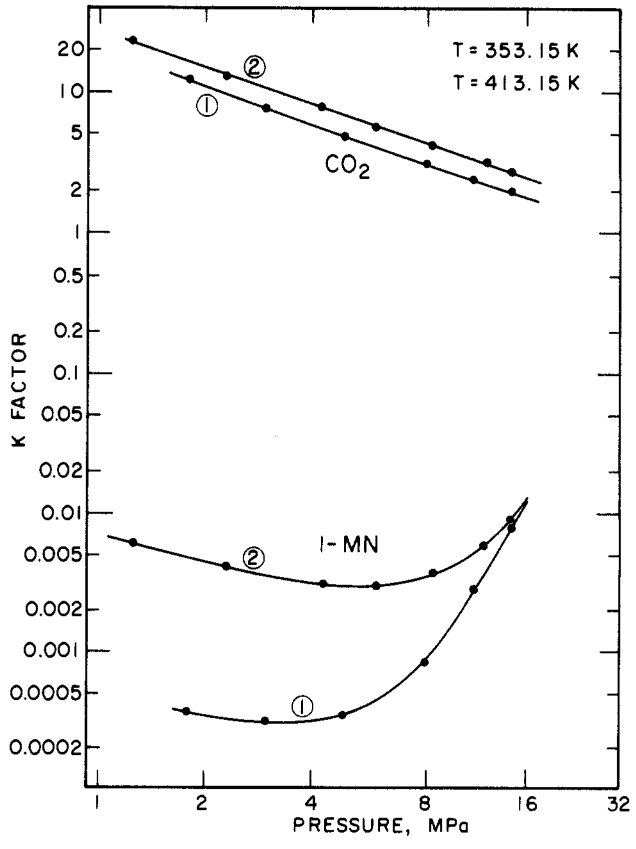

Figure 7. K factor–pressure diagram for carbon dioxide–1-methylnaphthalene system.

Table IV. Vapor-Liquid Equilibrium Data for Carbon Dioxide-Toluene-1-Methylnaphthalene ($Z_{1\text{-MN}}/Z_{\text{Tol}} = 4$) System

P, kPa	$x(CO_2)$	$y(CO_2)$	x(Tol)	y(Tol)	x(1-MN)	y(1-Mn)	$K(CO_2)$	K(Tol)	K(1-MN)
				T = 353.15 K					
3060	0.1518	0.997 367	0.0976	0.001 983	0.7506	0.000 65	6.57	0.0203	0.000 865
5330	0.2431	0.997 760	0.0864	0.001 720	0.6705	0.000 52	4.10	0.0199	0.000 776
10220	0.4567	0.995 25	0.0665	0.002 89	0.4768	0.001 86	2.18	0.0435	0.003 90
13940	0.5442	0.990 06	0.0550	0.006 44	0.4008	0.003 50	1.82	0.117	0.008 73
				T = 413.15 K					
2670	0.0968	0.986 69	0.1010	0.010 68	0.8022	0.002 63	10.20	0.106	0.003 27
6275	0.2200	0.991 66	0.0878	0.006 21	0.6922	0.002 13	4.51	0.0708	0.003 07
9715	0.3249	0.991 50	0.0756	0.006 07	0.5995	0.002 43	3.05	0.0802	0.004 06
13295	0.4106	0.988 25	0.0620	0.007 30	0.5274	0.004 45	2.41	0.118	0.008 44

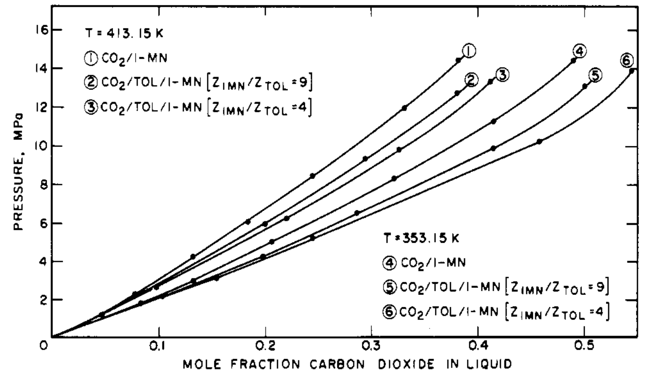

Figure 8. Solubility of carbon dioxide in various liquid mixtures of toluene and 1-methylnaphthalene.

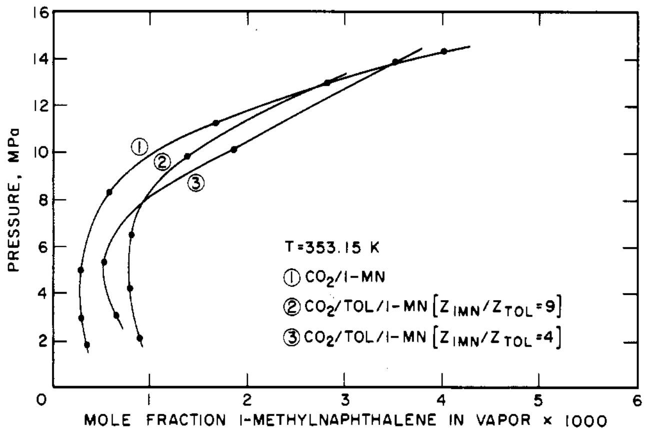

Figure 9. Vapor-phase solubility of 1-methylnaphthalene in the ternary system at 353.15 K.

III. Table IV reports the ternary equilibrium compositions with an initial 1-methylnaphthalene to toluene mole ratio of 4 to 1. These data are reported at temperatures of 353.15 (80.0 °C) and 413.15 (140.0 °C) K with pressures ranging from 2165 (314 psia) to 13 940 (2022 psia) kPa.

Figure 8 shows a comparison between the liquid-phase solubility of carbon dioxide in the ternary system and the binary system carbon dioxide–1-methylnaphthalene. As expected, the presence of toluene increases the solubility of carbon dioxide in the liquid phase. Figure 9 shows a comparison between the vapor-phase solubility of 1-methylnaphthalene in the ternary system and the binary system carbon dioxide–1-methylnaphthalene at 353.15 K (80.0 °C). At this temperature and for pressures less than 13 790 kPa (2000 psia), the presence of toluene increases the vapor-phase solubility of 1-methylnaphthalene. At pressures less than 6895 kPa (1000 psia) the initial liquid mole ratio of 9 to 1 produces greater vapor-phase solubilities of 1-methylnaphthalene than the 4 to 1 initial molar ratio. At higher pressures this relation is reversed.

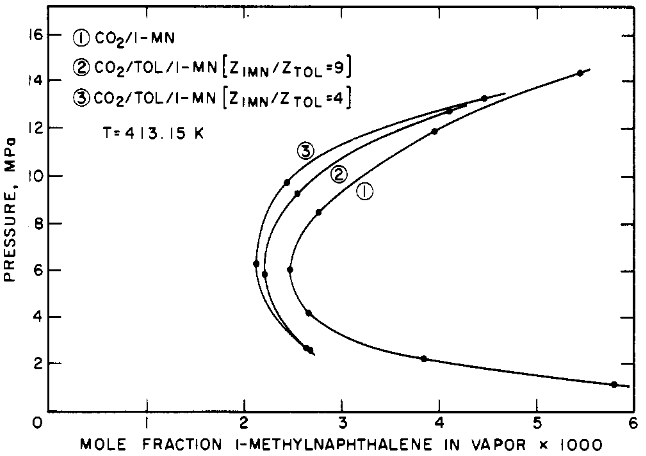

Figure 10. Vapor-phase solubility of 1-methylnaphthalene in the ternary system at 413.15 K.

Figure 10 shows a comparison between the vapor-phase solubility of 1-methylnaphthalene in the ternary system and in the binary system carbon dioxide-1-methylnaphthalene at 413.15 K (140.0 °C). The presence of toluene at this temperature causes a decrease in the vapor-phase solubility of 1-methylnaphthalene over the full range of pressures tested. The initial molar ratio of 9 to 1 has greater solubilities than the initial molar ratio of 4 to 1.

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Registry No. CO_2, 124-38-9; TOL, 108-88-3; 1-MN, 90-12-0.

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