

Volumetric Properties of Carbon Dioxide + Sulfur Hexafluoride at High Pressures

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Volumetric properties of binary mixtures of carbon dioxide and sulfur hexafluoride have been determined using a specially designed high-pressure variable-volume view cell. The data are reported for mixtures containing 0, 9, 20.2, 31, 35.6, 50, and 100% by mass sulfur hexafluoride over a temperature range from 296 to 430 K at pressures up to 70 MPa. At these conditions, depending upon the mixture composition, densities are observed to vary in the range from 0.7 to 1.6 g/cm³.

Introduction

Theoretical and experimental investigation of fluid mixtures at high pressures has been a focus of intense research over the past several years. The documentation of the PVT behavior of fluid mixtures and the effect of temperature and pressure on their densities at high pressures are particularly important for processes at near critical and supercritical conditions (Kiran and Levelt Sengers, 1994). This is particularly because many properties of the supercritical fluids are correlated and scale best with density. From a practical perspective, binary fluid mixtures are of interest to many processes since introduction of the second component to the primary process or processing fluid may permit regulation or improvement of selectivity, solubility, reactivity, recovery, and recycle.

The present paper is concerned with the volumetric properties of sulfur hexafluoride and its binary mixtures with carbon dioxide. Sulfur hexafluoride (SF₆) has been of interest in chromatography (Kiran, 1983; Hellgeth *et al.*, 1988), the electrical power industry (Cole and de Reuck, 1990) and polymer separations (Super *et al.*, 1993). Even though there are extensive data for the density of carbon dioxide (Vargaftik, 1975; Angus *et al.*, 1976; Ely, 1986; Duschek *et al.*, 1990; Kiran *et al.*, 1995), data for SF₆ are more limited (DeZwaan and Jonas, 1975; Cole and de Reuck, 1990; Abbaci and Sengers, 1990; Gilgen *et al.*, 1992; Blanke *et al.*, 1993). As for their binary mixtures, data are scarce and are limited to a few compositions (in the range from 49 to 95% SF₆ by mass) at relatively low temperatures (in the range 294–338 K) and at pressures below 40 MPa (Super *et al.*, 1993). In the present study, data for these mixtures are reported over the full composition range covering a temperature range from 297 to 430 K and pressures up to 70 MPa. This is a part of an ongoing program at the University of Maine on polymer processing with binary fluid mixtures at high pressures (Kiran, 1994; Xiong and Kiran, 1994; Kiran and Gokmenoglu, 1994, 1995). We have already reported the volumetric properties of carbon dioxide + pentane (Kiran *et al.*, 1995) and will be reporting data for other mixtures such as carbon dioxide + toluene (Pöhler and Kiran, 1995) and carbon dioxide + acetone in future papers.

Experimental Section

a. Experimental Apparatus. The experimental system details have been described in our previous publica-

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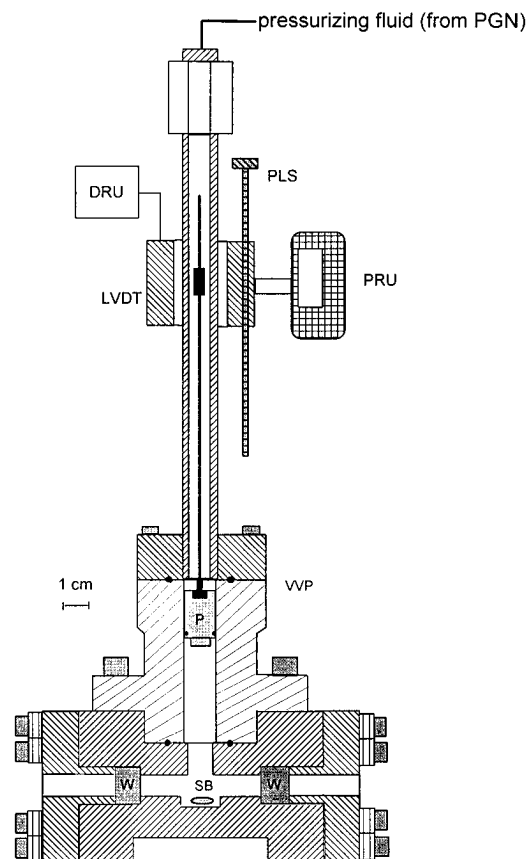


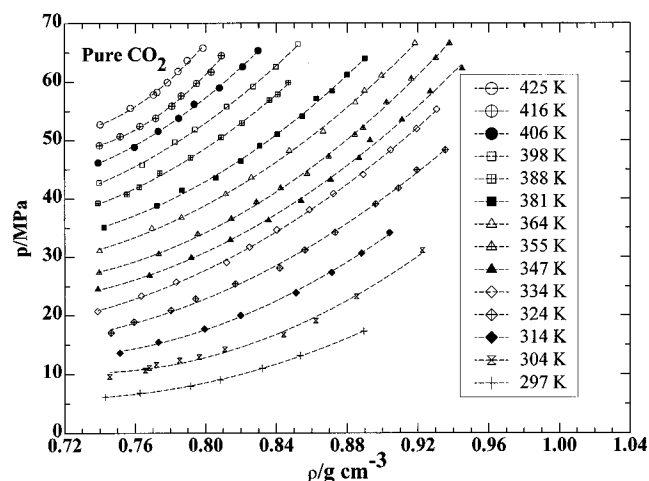
Figure 1. Variable-volume view cell. W = sapphire window, SB = stirring bar, P = piston, DRU = digital read-out unit, LVDT = linear variable differential transformer, PLS = position locator screw, PRU = position read-out unit, PGN = pressure generator, and VVP = variable-volume part.

tions (Kiran *et al.*, 1995). It consists of a high-pressure variable-volume view cell, a pressure generator (PGN), a solvent delivery manifold, and the associated controls and display modules. The system is operable at pressures up to 70 MPa and temperatures up to 473 K.

The variable-volume view cell (Figure 1) is equipped with two sapphire windows for either visual observations or optical detection. The cell content is mixed with a magnetic stirrer and a Teflon-coated stirring bar (SB) inside the cell cavity. The cell pressure and the temperature are monitored with a flush-mount pressure/temperature transducer with an accuracy of ± 0.03 MPa and ± 0.5 K, respectively. The transducer is in direct contact with the fluid inside the cell. The cell is heated with four cartridge heaters

Table 1. Density Data for Pure CO₂

ρ /(g/cm ³)	p /MPa	ρ /(g/cm ³)	p /MPa	ρ /(g/cm ³)	p /MPa	ρ /(g/cm ³)	p /MPa	ρ /(g/cm ³)	p /MPa
<i>T</i> = 297 K		<i>T</i> = 304 K		<i>T</i> = 314 K		<i>T</i> = 324 K		<i>T</i> = 334 K	
0.7431	6.11	0.7455	9.59	0.7512	13.62	0.7462	17.04	0.7386	20.72
0.7625	6.80	0.7656	10.66	0.7731	15.47	0.7591	18.89	0.7633	23.33
0.7914	7.95	0.7678	11.11	0.7993	17.68	0.7803	20.87	0.7832	25.70
0.8081	9.02	0.7718	11.59	0.8196	19.97	0.7942	22.82	0.8117	29.04
0.8320	10.97	0.7851	12.31	0.8510	23.81	0.8167	25.36	0.8248	31.64
0.8534	13.12	0.7962	12.89	0.8711	27.31	0.8418	28.07	0.8401	34.61
0.8892	17.27	0.8107	14.21	0.8880	30.62	0.8560	31.19	0.8586	38.06
		0.8440	16.70	0.9037	34.12	0.8731	34.18	0.8721	40.81
		0.8622	19.08			0.8958	39.08	0.8889	44.11
		0.8852	23.19			0.9088	41.76	0.9043	48.28
		0.9223	31.11			0.9192	44.87	0.9190	51.93
						0.9353	48.34	0.9303	55.26
<i>T</i> = 347 K		<i>T</i> = 355 K		<i>T</i> = 364 K		<i>T</i> = 381 K		<i>T</i> = 388 K	
0.7388	24.48	0.7395	27.34	0.7398	31.06	0.7423	35.08	0.7388	39.25
0.7679	26.83	0.7732	30.52	0.7693	34.91	0.7722	38.80	0.7553	40.74
0.7918	29.89	0.7954	33.91	0.7862	36.74	0.7866	41.42	0.7622	41.97
0.8139	32.89	0.8144	36.53	0.8113	40.76	0.8056	43.53	0.7738	44.37
0.8354	36.25	0.8285	39.40	0.8257	43.61	0.8198	46.43	0.7914	47.00
0.8539	39.59	0.8423	41.79	0.8471	48.19	0.8304	49.04	0.8087	50.51
0.8706	43.22	0.8573	44.21	0.8664	51.47	0.8404	50.99	0.8204	52.93
0.8866	46.94	0.8695	47.21	0.8844	56.44	0.8545	54.10	0.8359	56.84
0.8927	49.97	0.8842	50.94	0.8897	58.49	0.8624	57.11	0.8406	57.82
0.9108	53.50	0.8889	52.10	0.8992	61.03	0.8713	58.42	0.8467	59.85
0.9268	58.29	0.9021	56.44	0.9182	66.59	0.8798	61.21		
0.9448	62.21	0.9160	60.51			0.8899	63.98		
		0.9299	64.02						
		0.9377	66.57						
<i>T</i> = 398 K		<i>T</i> = 406 K		<i>T</i> = 416 K		<i>T</i> = 425 K			
0.7395	42.76	0.7389	46.18	0.7396	49.12	0.7400	52.74		
0.7638	45.78	0.7593	48.82	0.7513	50.68	0.7572	55.51		
0.7825	49.69	0.7729	51.56	0.7625	52.41	0.7702	57.79		
0.7938	51.85	0.7846	53.78	0.7715	53.81	0.7722	58.22		
0.8118	55.75	0.7934	56.19	0.7808	55.89	0.7782	59.89		
0.8267	59.17	0.8076	58.95	0.7860	57.61	0.7842	61.82		
0.8396	62.55	0.8208	62.58	0.7948	59.74	0.7895	63.64		
0.8522	66.42	0.8295	65.34	0.8029	61.68	0.7984	65.77		
				0.8088	64.47				

**Figure 2.** Variation of the density of carbon dioxide with pressure at selected temperatures.

heaters inserted symmetrically into the cell body, and the temperature is controlled with an Omega controller which uses an RTD probe.

At a given temperature, the cell volume (and hence the pressure in the cell) can be changed by moving a piston (P) inside the variable-volume part (VVP) of the cell. The position of the piston is detected by a linear variable differential transformer (LVDT) and a digital read-out unit (DRU) which responds to the location of a ferromagnetic slug connected to the piston with a rigid stainless-steel rod. When the internal volume of the cell is at its maximum (i.e., piston is all the way up), the LVDT coil is moved using the position locator screw (PLS) until the digital readout unit indicates zero which corresponds to the ferromagnetic slug being at the exact center of the LVDT coil. At this point, the reading of the position readout unit (PRU) is set to zero. When the piston moves downward to a new

position (i.e., smaller cell volume), the LVDT coil is moved (with PLS) to locate its new position which is again identified, with the signal from the DRU becoming zero. The distance traveled from the initial zero reference point is directly read from the PRU with an accuracy of ± 0.013 mm.

b. Operational Procedure. Density determinations are based on the measurement of the cell volume at any given temperature and pressure. The fluid to be tested is charged to the cell at room temperature from a fluid transfer vessel which has been previously filled with the fluid (about 50–100 g) of interest. The amount charged during pumping is monitored from the decrease in the weight of the transfer vessel. The weight is recorded with a Mettler balance (model 6100) with an accuracy of ± 0.01 g.

In loading the cell with two fluids, SF₆ which has a lower vapor pressure than CO₂ is charged first. After charging the cell with SF₆, the cell is removed and weighed on a Sartorius balance (16 kg capacity with 0.1 g accuracy) for further verification of the exact amount loaded. The cell is then connected to the system. A new transfer vessel filled with carbon dioxide is connected to the solvent charge line. The line is purged with carbon dioxide before the valve to the cell is opened and carbon dioxide is pumped into the cell. The amount charged is again monitored from the weight change of the transfer vessel. The cell, now loaded with both fluids, is once again removed and weighed for accurate determination of the exact amounts loaded. Typically, about 18 g of fluid mixture is loaded into the cell.

After loading, the cell content is stirred and heated to the desired temperature. While the piston is at the top of the variable-volume cell (which is assured by not applying pressure on the pressure generation line behind the piston), the zero reference point of the position readout unit (PRU),

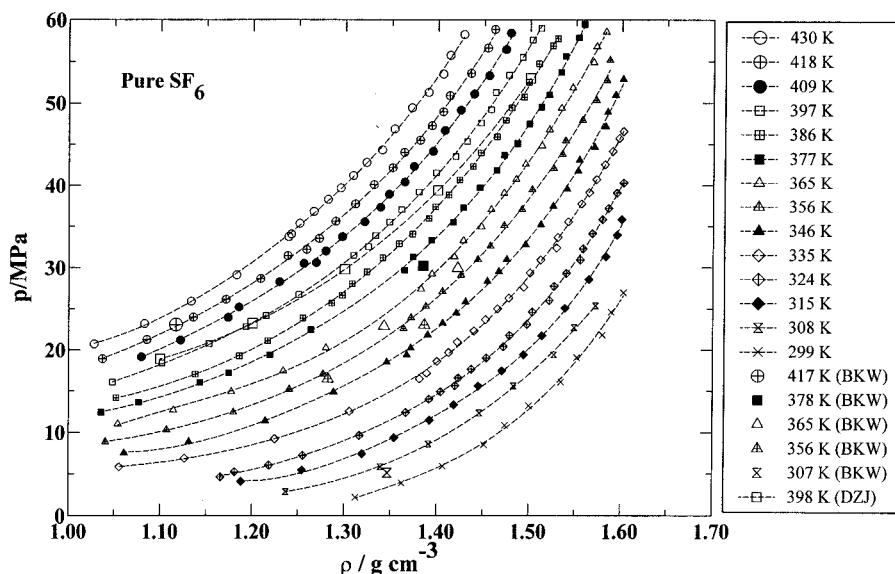


Figure 3. Variation of the density of sulfur hexafluoride with pressure at selected temperatures. BKW = data from Blanke *et al.* (1993), and DZJ = data from DeZwinn and Jonas (1975).

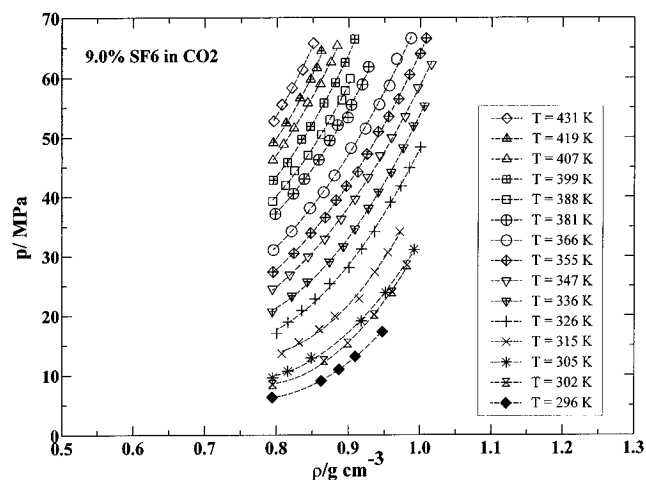


Figure 4. Variation of the density of the carbon dioxide + sulfur hexafluoride mixture (9.0% by mass SF₆) with pressure at selected temperatures.

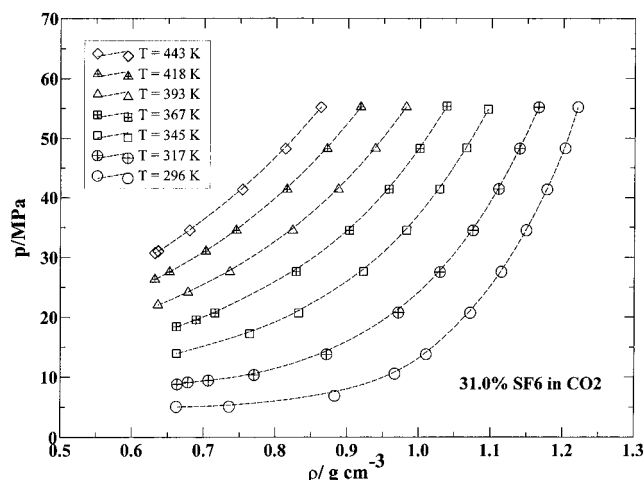


Figure 6. Variation of the density of the carbon dioxide + sulfur hexafluoride mixture (31.0% by mass SF₆) with pressure at selected temperatures.

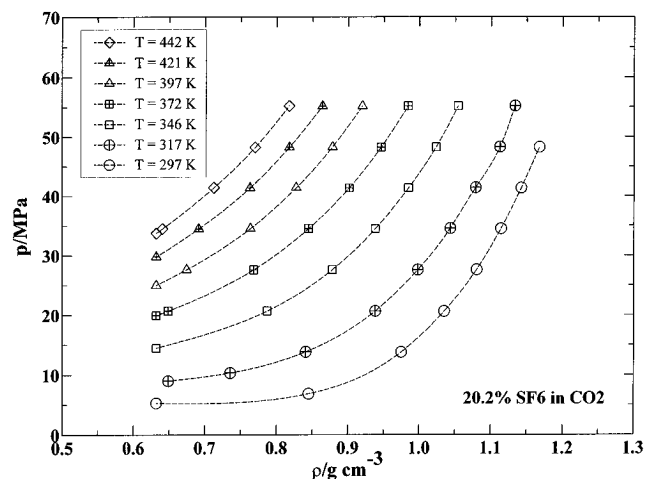


Figure 5. Variation of the density of the carbon dioxide + sulfur hexafluoride mixture (20.2% by mass SF₆) with pressure at selected temperatures.

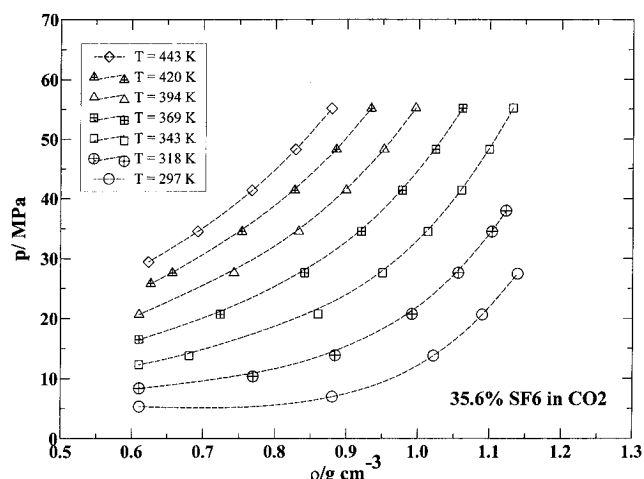


Figure 7. Variation of the density of the carbon dioxide + sulfur hexafluoride mixture (35.6% by mass SF₆) with pressure at selected temperatures.

corresponding to the maximum internal volume of the cell (which is $22.43 \pm 0.05 \text{ cm}^3$), is established. After thermal equilibration is achieved, the piston is moved by applying pressure from the pressure generator to bring the system to a new pressure. [Here, *n*-pentane is used as the pressurizing fluid behind the piston.] The new position of

the piston, and thus the internal volume of the cell corresponding to the prevailing pressure, is determined by moving the LVDT with the aid of the position locator screw. All accessible pressures up to 70 MPa at a given temperature are mapped in a similar fashion. From the total amount of the fluid that has been charged, and the

Table 2. Density Data for Pure Sulfur Hexafluoride

ρ /(g/cm ³)	p /MPa	ρ /(g/cm ³)	p /MPa	ρ /(g/cm ³)	p /MPa	ρ /(g/cm ³)	p /MPa	ρ /(g/cm ³)	p /MPa
$T = 299$ K		$T = 308$ K		$T = 315$ K		$T = 324$ K			
1.3127	2.18	1.2370	2.84	1.1882	4.08	1.1655	4.62	1.4784	21.78
1.3622	3.90	1.3392	5.86	1.2545	5.45	1.1812	5.23	1.4973	23.13
1.4063	5.93	1.3909	8.63	1.3194	7.44	1.2185	6.06	1.5033	24.60
1.4513	8.54	1.4460	12.34	1.3540	9.38	1.2554	7.23	1.5218	26.02
1.4738	10.87	1.4828	15.63	1.3920	11.46	1.3165	9.63	1.5269	27.72
1.4998	13.22	1.5269	19.45	1.4185	13.34	1.3665	12.40	1.5402	29.31
1.5347	16.12	1.5489	22.72	1.4448	15.61	1.3916	14.02	1.5553	30.94
1.5521	19.09	1.5721	25.37	1.4705	17.48	1.4039	14.93	1.5583	32.31
1.5798	21.82			1.4938	19.44	1.4196	15.64	1.5685	34.12
1.5892	24.59			1.5138	21.73	1.4232	16.58	1.5792	35.83
1.6025	26.95			1.5389	25.06	1.4370	17.68	1.5859	37.21
				1.5647	28.58	1.4534	19.04	1.5949	39.08
				1.5831	31.35	1.4719	20.42	1.6022	40.34
				1.5954	33.95				
				1.6002	35.85				
$T = 335$ K		$T = 346$ K		$T = 356$ K		$T = 365$ K		$T = 377$ K	
1.0554	5.86	1.0608	7.53	1.0404	8.877	1.0539	11.03	1.0357	12.41
1.1268	6.90	1.1314	8.90	1.1072	10.32	1.1144	12.69	1.0767	13.62
1.2247	9.25	1.2144	11.38	1.1796	12.42	1.1773	14.91	1.1430	16.02
1.3057	12.53	1.2883	14.88	1.2404	15.21	1.2336	17.44	1.1742	17.23
1.3812	16.49	1.3458	18.52	1.2769	17.08	1.2799	20.25	1.2191	19.38
1.3888	17.17	1.3672	19.43	1.3636	22.58	1.3824	27.40	1.2636	22.43
1.4000	18.62	1.3715	20.25	1.3717	23.91	1.3942	29.25	1.3643	29.68
1.4125	19.70	1.3899	21.83	1.3877	25.28	1.4183	31.31	1.3736	31.32
1.4218	20.98	1.4059	23.25	1.4057	27.10	1.4279	33.29	1.3933	33.31
1.4400	22.31	1.4207	24.49	1.4256	29.11	1.4474	34.97	1.4169	35.51
1.4525	23.69	1.4306	25.85	1.4439	30.98	1.4581	37.07	1.4277	37.29
1.4654	25.02	1.4541	28.36	1.4572	32.63	1.4719	39.07	1.4455	39.76
1.4777	26.32	1.4666	29.55	1.4721	35.13	1.4847	40.79	1.4640	41.88
1.4936	27.64	1.4750	31.00	1.4913	37.10	1.4950	42.63	1.4717	43.74
1.5005	29.38	1.4953	32.80	1.5023	39.51	1.5128	44.86	1.4859	45.13
1.5121	30.95	1.5121	35.23	1.5256	42.13	1.5210	46.80	1.4990	47.51
1.5292	32.41	1.5254	37.51	1.5352	43.85	1.5339	49.41	1.5123	49.53
1.5308	33.67	1.5404	39.60	1.5386	45.44	1.5463	51.89	1.5207	51.02
1.5420	35.57	1.5524	41.80	1.5569	47.95	1.5688	54.89	1.5336	53.71
1.5564	37.78	1.5540	43.14	1.5718	50.34	1.5721	56.80	1.5383	55.64
1.5647	39.14	1.5702	44.69	1.5834	52.72	1.5823	58.57	1.5526	57.91
1.5724	40.74	1.5820	47.17	1.5865	55.18			1.5588	59.46
1.5817	42.56	1.5842	48.91						
1.5901	44.19	1.5929	50.98						
1.5971	45.74	1.6011	52.89						
1.6017	46.56								
$T = 386$ K		$T = 397$ K		$T = 409$ K		$T = 418$ K		$T = 430$ K	
1.0521	14.17	1.0480	16.06	1.0794	19.16	1.0368	18.93	1.0279	20.72
1.1381	17.04	1.1022	18.40	1.1220	21.21	1.0853	21.25	1.0827	23.18
1.1861	19.29	1.1530	20.76	1.1735	23.93	1.1357	23.95	1.1334	25.91
1.2168	21.11	1.1932	22.79	1.1853	25.20	1.1712	26.14	1.1826	29.11
1.2547	23.85	1.2148	24.12	1.2292	28.27	1.2087	28.66	1.2389	33.76
1.2860	25.66	1.2502	26.70	1.2556	30.50	1.2382	31.44	1.2417	34.06
1.2972	26.63	1.3093	31.46	1.2690	30.61	1.2585	32.19	1.2509	35.36
1.3077	28.02	1.3254	32.54	1.2792	32.02	1.2720	33.53	1.2661	36.82
1.3239	29.48	1.3328	33.86	1.2970	33.74	1.2896	35.63	1.2814	38.29
1.3410	31.19	1.3480	35.51	1.3210	35.56	1.3108	37.76	1.2950	39.70
1.3583	32.89	1.3608	37.04	1.3380	37.33	1.3309	40.12	1.3083	41.22
1.3728	34.07	1.3795	39.18	1.3474	38.95	1.3510	42.19	1.3229	42.82
1.3905	35.97	1.3978	41.55	1.3641	40.44	1.3632	44.04	1.3396	44.35
1.3974	37.40	1.4189	43.57	1.3738	42.35	1.3805	45.51	1.3528	46.85
1.4118	38.85	1.4315	45.35	1.3944	44.17	1.3931	47.26	1.3715	49.42
1.4234	40.70	1.4464	47.58	1.4072	46.70	1.4052	48.94	1.3895	51.29
1.4379	42.35	1.4572	49.21	1.4243	49.14	1.4125	50.89	1.4052	53.50
1.4464	44.00	1.4624	51.30	1.4388	51.12	1.4354	53.59	1.4129	55.75
1.4640	45.93	1.4767	53.37	1.4551	53.33	1.4536	56.64	1.4277	58.26
1.4733	47.92	1.4913	55.48	1.4733	56.46	1.4612	58.89		
1.4796	49.44	1.5023	57.58	1.4784	58.43				
1.4931	50.74	1.5116	59.02						
1.4980	52.53								
1.5093	54.72								
1.5243	56.90								
1.5297	57.75								

information on volume at different T and P , densities of the mixture are calculated. After the mapping of volumes is completed at a given temperature, the cell is heated to a new temperature and the procedure is repeated. When tests are completed, the pressure in the pressure generation line is released, and the fluid in the cell is discharged after cooling.

Using this system, volume changes are determined with an accuracy of ± 0.0025 cm³. From comparisons with density data for carbon dioxide and pentane, density measurements have been on the average shown to be accurate to $\pm 1.2\%$ (Kiran *et al.*, 1995).

c. Materials. Sulfur hexafluoride was instrument grade (Scott Specialty Gases) with a purity of 99.99%. Carbon dioxide was bone-dry grade with an eductor tube (Airco) with a purity of 99.8%. They were used without further purification.

Results and Discussions

a. Densities of the Pure Fluids. The results of density measurements for pure carbon dioxide and sulfur hexafluoride are shown in Figures 2 and 3. Numerical data are given in Tables 1 and 2. For carbon dioxide, data have been collected up to 65 MPa in the temperature range from

Table 7. Density Data for Sulfur Hexafluoride + Carbon Dioxide [50.0% by Mass SF₆]

ρ /(g/cm ³)	p /MPa	ρ /(g/cm ³)	p /MPa	ρ /(g/cm ³)	p /MPa	ρ /(g/cm ³)	p /MPa	ρ /(g/cm ³)	p /MPa
$T = 296$ K		$T = 323$ K		$T = 348$ K		$T = 398$ K		$T = 423$ K	
0.8133	6.08	0.8022	11.39	0.7922	17.18	0.7781	27.88	0.7721	33.10
1.0112	9.02	0.9512	15.98	0.8611	19.76	0.8292	31.15	0.8053	35.60
1.0781	12.49	1.0120	19.14	0.9263	23.19	0.8732	34.52	0.8411	38.68
1.1224	15.82	1.0610	22.83	0.9822	26.95	0.9171	38.36	0.8772	42.12
1.1463	18.02	1.0842	25.04	1.0271	30.70	0.9542	42.11	0.9123	45.77
		1.1033	26.95	1.0632	34.47	0.9883	46.06	0.9454	49.73
		1.1234	29.10	1.0962	38.51	1.0171	49.75	0.9752	53.68
		1.1471	32.04	1.1231	42.31	1.0441	53.56	1.0021	57.40
						1.0692	57.39	1.0260	61.19
						1.0912	60.93	1.0473	64.64
						1.1100	64.65		

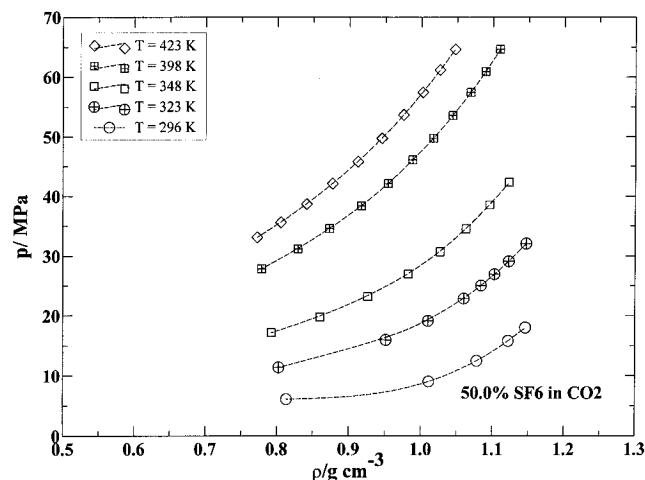
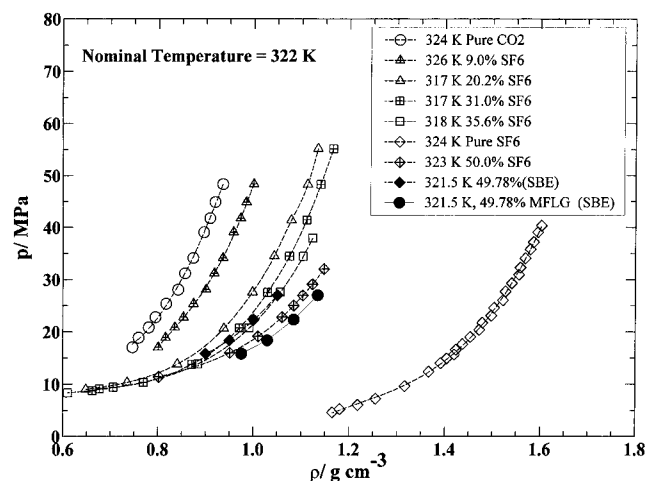
**Figure 8.** Variation of the density of the carbon dioxide + hexafluoride mixture (50.0% by mass SF₆) with pressure at selected temperatures.**Figure 9.** Variation of the density of the carbon dioxide + hexafluoride mixture with composition and pressure at a fixed temperature. SBE = data from Super *et al.* (1993).

Figure 9 shows a comparative plot of pressure versus density for different mixtures at comparable temperatures. The densities increase with increasing SF₆ content of the mixture. Since at the conditions investigated, SF₆ densities are higher than the highest density for carbon dioxide, the mixtures do not show the density crossovers that were encountered in the carbon dioxide + pentane system (Kiran *et al.*, 1995).

Density information on SF₆ + CO₂ mixtures have been reported at low temperatures (in the range from 295 to 336 K) for selected compositions (23 mol % or 49.78% by mass and higher SF₆ contents) (Super *et al.*, 1993). The measurements were conducted by a flotation technique in which the density of the mixture is assigned by determining the flotation point of small metal balls of known density.

The same authors have also reported predictions by the mean-field lattice-gas theory (MFLG). The present data cover mixtures with 50% or lower SF₆ content. Their experimental data for 49.78% SF₆ and their prediction for the same mixture by mean-field lattice gas theory are also included in Figure 9. The data differ by about 6% from the present measurements. Their MFLG predictions compare better with the present data. Even though not shown in the figure, comparisons of the density data for this mixture at 295 K display similar differences.

Conclusions

Densities of carbon dioxide + sulfur hexafluoride mixtures over the entire composition range have been determined at temperatures in the range from 296 to 430 K and at pressures up to 70 MPa. It is shown that, at these conditions, depending upon the mixture composition, densities change from 0.7 to 1.6 g/cm³. The density increases with increasing sulfur hexafluoride content of the mixture.

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Received for review October 18, 1995. Accepted December 22, 1995.® This research has in part been supported by the National Science Foundation (Grant CTS-P310232).

JE950260+

® Abstract published in *Advance ACS Abstracts*, February 15, 1996.