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Isochoric *PVTx* Measurements for the Carbon Dioxide + 1,1,1,2-Tetrafluoroethane Binary System

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ABSTRACT: *PVTx* measurements for carbon dioxide (CO_2, R744) + 1,1,1,2-tetrafluoroethane (CF_3CH_2F, R134a) for both two-phase and superheated vapor regions are presented. The measurements were taken with a constant volume apparatus at temperatures ranging from (233 to 373) K and pressures from (111 to 2915) kPa along 11 isochores. The data obtained in the two-phase region were used to derive vapor−liquid equilibrium (VLE) parameters using a flash method with the Carnahan−Starling−De Santis equation of state (CSD EOS). The results were compared with data existing in the literature. The results from the superheated region were compared with those predicted from the CSD EOS adopting parameters derived from the two-phase region data. A complete set of data was also compared with the REFPROP 9.0 prediction.

INTRODUCTION

Because of its triple-point temperature of 216.58 K, carbon dioxide is no longer a feasible solution for vapor compression cycles intended for use at lower temperatures. An obvious solution to overcome this drawback could be a blend containing carbon dioxide. For this reason, recently, our attention turned to systems composed of hydrofluorocarbons (HFCs) in mixtures with CO_2.

In previous studies, the P, V, T, and x properties of the CO_2 + R41, CO_2 + R116, CO_2 + R125, CO_2 + R32, CO_2 + R23, and CO_2 + R152a systems were measured by an isochoric method,[1−3] the Burnett method,[4−8] and a solid−liquid equilibrium apparatus.[9,10] In this paper, the *PVTx* properties of the CO_2 + R134a binary system are studied with a constant volume apparatus.

Vapor−liquid equilibrium (VLE) data of this binary system were already measured by different laboratories.[11−13] The measurements performed by Silva-Oliver and Galicia-Luna[11] covered a temperature range from (329 to 354) K, while the measurements performed by Duran-Valencia et al.[12] covered a lower temperature range from (252.95 to 292.95) K. More recently, Lim et al.[13] measured VLE data from (323 to 342) K. To our knowledge no experimental results have been published in the open literature on the superheated vapor region of this specific binary system. Isochoric measurements were consequently taken, covering both the two phase and the superheated vapor region, with temperatures spanning from (233 to 373) K and pressures spanning from (111 to 2915) kPa. VLE parameters were derived from data in the two-phase region, applying the Carnahan−Starling−De Santis equation of state (CSD EOS).[14] Data obtained from the superheated region were also compared with the predictions obtained with the CSD EOS.

Table 1. Measurements at Bulk Compositions z_1 and Masses m_1 and m_2 for the CO_2 (1) + R134a (2) System over the Temperature Range ΔT and Pressure Range ΔP and Binary Interaction Parameters K_{12}

series	z_1	ΔT/K	ΔP/kPa	n/mol	m_1/g	m_2/g	K_{12}
1	0.146	243−363	153−961	0.0953	0.610	8.312	−0.034
2	0.163	233−343	111−766	0.0800	0.573	6.840	−0.012
3	0.268	243−373	228−1160	0.1113	1.314	8.312	−0.026
4	0.373	233−343	214−952	0.0994	1.631	6.357	−0.020
5	0.456	243−373	365−1380	0.1322	2.656	7.335	−0.020
6	0.466	248−343	379−1051	0.1093	2.241	5.960	−0.020
7	0.646	243−373	591−1875	0.1796	5.108	6.482	−0.015
8	0.729	238−343	629−1773	0.1877	6.025	5.181	−0.017
9	0.747	243−373	687−1763	0.1674	5.502	4.326	−0.018
10	0.807	233−343	709−2504	0.2719	9.659	5.347	−0.012
11	0.899	233−343	846−2915	0.3190	12.615	3.304	−0.009
						avg	−0.018

EXPERIMENTAL SECTION

Chemicals. Carbon dioxide was supplied by Sol SpA. R134a was donated by Ausimont Spa (Milan, Italy). Their purity was checked by gas chromatography, using a thermal conductivity detector, and found to be 99.99 % and 99.98 %, respectively, basing all estimations on an area response.

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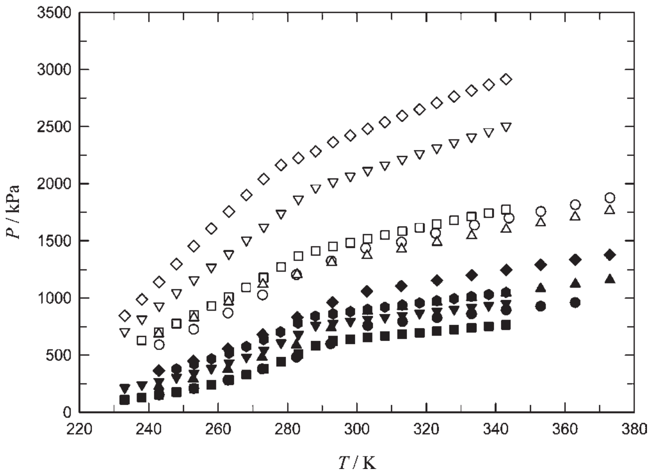

Figure 1. Experimental $P-T$ data: ●, $z_1 = 0.146$; ■, $z_1 = 0.163$; ▲, $z_1 = 0.268$; ▼, $z_1 = 0.373$; ♦, $z_1 = 0.456$; ●, $z_1 = 0.466$; ○, $z_1 = 0.646$; □, $z_1 = 0.729$; △, $z_1 = 0.747$; ▽, $z_1 = 0.807$; ◊, $z_1 = 0.899$.

Apparatus. The setup has already been described elsewhere,[15] so it is only briefly outlined here. It is a classical constant volume setup, with a stainless steel sphere of about 253 cm^3 of volume.

The main changes made to the original apparatus[15,16] concerned the twin thermostatic baths filled with different silicone oils (Baysilone M10 and Baysilone M100, Bayer). After charging with the sample mixture, the setup could be operated over two temperature ranges, approximately from (210 to 290) K and from (290 to 360) K, depending on which bath was used.

The thermostatic baths were easy to move thanks to the new system configuration. The spherical cells and pressure transducer were immersed in one of the two thermostatic baths. An auxiliary thermostat was used to reach below-ambient temperatures. The cell volume was estimated (as explained elsewhere[15]) to be (273.5 ± 0.3) cm^3 at room temperature.

The pressure and temperature data acquisition systems were identical to those of the previous apparatus.[15,16] A PID device was used to control the temperature, which was measured using a calibrated resistance thermometer; the total uncertainty of the temperature measurements was 0.02 K. The uncertainty in the pressure measurements stems from the uncertainty of the transducer and null indicator system and the pressure gauges. The uncertainty of the digital pressure indicator (Ruska, model 7000) is 0.003 % of its full scale (6000 kPa). Temperature fluctuations due to bath instability can also affect the total uncertainty in the pressure measurement, which was nonetheless found to be less than 1 kPa.

Experimental Procedure. Mixtures were prepared using the gravimetric method. First of all, the pure samples were charged in different bottles, degassed to remove noncondensable gases and air, and then weighed with an analytical balance (uncertainty of 0.3 mg). After evacuating the cell, the bottles were then emptied into the cell immersed in the bath. The mixtures were charged in the vapor phase. The bottles were weighed again, and the mass of the charge was calculated from the difference between the two masses. The dispersion of the mass inside the duct was estimated to be between (0.01 and 0.06) g, depending on the charging

Table 2. Experimental Molar Volumes V as a Function of Pressure P and Temperature T at Overall Composition z_1 in the Two-Phase Region for the CO_2 (1) + R134a (2) System

T	P	V	T	P	V
K	kPa	$dm^3 \cdot mol^{-1}$	K	kPa	$dm^3 \cdot mol^{-1}$
	$z_1 = 0.146$			$z_1 = 0.646$	
243.02	153.1	2.864	242.99	591.2	1.520
252.95	208.9	2.865	252.93	726.6	1.521
262.80	281.5	2.866	262.85	869.5	1.522
272.69	379.2	2.867	272.81	1028.1	1.522
282.54	482.4	2.869	282.59	1204.9	1.523
	$z_1 = 0.163$			$z_1 = 0.729$	
233.03	110.9	3.408	237.95	629.3	1.454
238.01	129.9	3.409	242.92	701.1	1.455
242.96	151.8	3.410	247.95	776.9	1.455
247.96	177.5	3.411	253.04	852.9	1.455
252.98	207.6	3.411	258.09	932.2	1.456
258.02	242.5	3.412	263.07	1010.5	1.456
263.11	283.0	3.413	268.08	1093.5	1.456
268.13	329.1	3.414	273.09	1180.0	1.457
273.14	382.1	3.414	278.12	1272.0	1.457
278.12	441.6	3.415	283.11	1370.1	1.457
283.10	509.3	3.416	237.95	629.3	1.454
288.09	585.1	3.417		$z_1 = 0.747$	
	$z_1 = 0.268$		242.89	687.1	1.631
242.88	228.1	2.452	253.03	828.5	1.631
252.80	291.7	2.454	263.00	968.4	1.632
262.78	376.0	2.455	272.88	1121.1	1.633
272.67	481.6	2.456		$z_1 = 0.807$	
282.52	589.0	2.457	233.06	708.7	1.004
	$z_1 = 0.373$		238.02	817.7	1.004
233.08	214.4	2.746	243.00	931.8	1.004
238.03	241.7	2.747	248.00	1046.2	1.004
242.97	271.2	2.747	253.01	1159.8	1.005
247.98	304.8	2.748	258.04	1272.0	1.005
253.00	342.7	2.749	263.08	1387.9	1.005
258.02	384.6	2.749	268.13	1503.8	1.005
263.09	432.0	2.750	273.13	1620.3	1.006
268.13	484.7	2.750	278.13	1740.3	1.006
273.13	543.2	2.751	283.12	1864.1	1.006
278.13	609.0	2.752		$z_1 = 0.899$	
283.12	682.6	2.752	233.09	846.2	0.855
	$z_1 = 0.456$		238.02	988.7	0.856
242.93	365.3	2.065	242.97	1140.0	0.856
252.88	450.4	2.065	247.99	1297.6	0.856
262.88	556.2	2.066	253.00	1454.5	0.856
272.84	679.7	2.067	258.02	1607.1	0.856
282.84	831.6	2.068	263.08	1756.4	0.857
	$z_1 = 0.466$		268.12	1900.2	0.857
247.97	379.1	2.498	273.13	2039.9	0.857
253.06	422.6	2.498			
258.09	468.1	2.499			
263.12	518.6	2.499			
268.13	575.2	2.500			

Table 2. Continued

T	P	V	T	P	V
K	kPa	$dm^3 \cdot mol^{-1}$	K	kPa	$dm^3 \cdot mol^{-1}$
273.10	636.6	2.500			
277.97	704.1	2.501			
283.03	781.2	2.502			

temperature, pressure, and molar mass of the fluid, and finally subtracted from the total mass of the sample. The uncertainty in mixture preparation was estimated to be constantly lower than 0.001 in mole fraction.

After reaching the experimental temperature, the mixing pump was activated for about 15 min and, next, the mixture was allowed to stabilize for about 20 min before data recording. After having charged each mixture composition, the temperature was increased step by step.

RESULTS AND DISCUSSION

The temperature and pressure ranges are shown in Table 1, along with the mixture's composition and the number of moles charged. The $P-T$ isochoric sequence is also shown in Figure 1.

Based on the analysis of the slope of each T,P sequence, the experimental points were each attributed either to the superheated or two-phase region. Table 2 shows the experimental data within the VLE boundary, while Table 3 contains the $PVTx$ data.

VLE Derivation. The method used to derive VLE data from the isochoric measurements using the CSD EOS was described elsewhere.[17] The method involves deriving the VLE parameters for each data point in the two-phase region using the "flash method" with the CSD EOS. For this method to be applied to isochoric data, we also need the volumetric properties of both phases, which were calculated from the CSD EOS. T, z_i, and n (number of moles charged) were kept constant during the correlation with the isochoric cell volume from the gravimetric calibration. The objective function:

$$Q = \sum_i \left(\frac{dP}{P}\right)^2 \tag{1}$$

was minimized tuning the K_{12} value. The correlation gives also the parameters of VLE (pressure and composition of both phases) which were considered, obviously, as dependent variables.

Figure 2 shows the scatter diagram of the relative pressure deviations which are almost temperature-independent. The pressure deviations were found to be within ± 1 % for all series, except for a few points at lower temperatures measured for the two series with the higher number of moles (series 10 and 11). The same trend of results was obtained comparing the experimental data together with the REFPROP 9.0[18] prediction, as shown in Figure 3. It is worth noting that the REFPROP model is very accurately predicting the present binary system behavior, considering also that the mixing parameters were estimated. The obtained binary interaction parameters are reported in Table 1. The average of the value is $K_{12} = -0.018$, with a statistical uncertainty of ± 0.005.

To compare our data with the literature, we performed data reduction through minimization of the objective function

Table 3. Experimental Molar Volumes V as a Function of Pressure P and Temperature T at Composition z_1 in the Superheated Vapor Region for the CO_2 (1) + R134a (2) System

T	P	V	T	P	V
K	kPa	$dm^3 \cdot mol^{-1}$	K	kPa	$dm^3 \cdot mol^{-1}$
	$z_1 = 0.146$			$z_1 = 0.646$	
292.44[a]	599.9[a]	2.870[a]	292.60[a]	1325.4[a]	1.524[a]
303.11	758.8	2.871	302.51	1435.8	1.524
313.12	794.0	2.872	312.82	1487.6	1.525
323.08	828.4	2.874	322.64	1565.2	1.526
333.05	862.1	2.875	333.86	1636.1	1.526
343.01	895.4	2.876	343.82	1698.1	1.527
352.96	928.3	2.878	353.00	1754.7	1.528
362.91	960.8	2.879	362.96	1815.6	1.528
	$z_1 = 0.163$		372.90	1875.8	1.529
293.10	625.9	3.417		$z_1 = 0.729$	
298.10	640.6	3.418	288.10	1413.3	1.458
303.08	655.1	3.419	293.11	1449.4	1.458
308.08	669.6	3.420	298.10	1483.2	1.458
313.05	683.6	3.420	303.09	1516.5	1.459
318.03	697.6	3.421	308.07	1549.7	1.459
323.03	711.6	3.422	313.06	1582.6	1.459
328.01	725.5	3.423	318.04	1615.0	1.460
333.01	739.3	3.423	323.03	1647.4	1.460
337.98	752.9	3.424	328.01	1679.3	1.460
342.97	766.4	3.425	332.99	1711.2	1.461
	$z_1 = 0.268$		337.99	1742.8	1.461
292.60[a]	739.2[a]	2.458[a]	342.96	1774.0	1.461
303.11	885.3	2.459		$z_1 = 0.747$	
313.11	926.8	2.460	282.73[a]	1207.0[a]	1.634[a]
323.09	966.9	2.461	292.74	1311.5	1.634
333.05	1006.4	2.462	302.93	1371.2	1.635
343.01	1045.1	2.463	312.88	1428.5	1.636
352.98	1084.4	2.464	323.09	1486.7	1.636
362.94	1122.6	2.465	333.06	1542.9	1.637
372.88	1160.1	2.466	343.02	1598.5	1.638
	$z_1 = 0.373$		352.98	1653.8	1.639
288.11	761.3	2.753	362.90	1708.3	1.639
293.11	779.0	2.753	372.88	1763.1	1.640
298.10	797.1	2.754		$z_1 = 0.807$	
303.09	814.9	2.755	288.11	1963.7	1.006
308.07	832.4	2.755	293.11	2015.0	1.006
313.06	849.9	2.756	298.09	2065.5	1.007
318.04	867.5	2.756	303.08	2115.7	1.007
323.07	884.8	2.757	308.06	2165.2	1.007
328.02	901.6	2.758	313.05	2214.6	1.007
333.00	918.5	2.758	318.04	2263.7	1.007
337.99	935.3	2.759	323.02	2312.3	1.008
342.97	951.9	2.759	328.01	2360.6	1.008
	$z_1 = 0.456$		333.00	2408.7	1.008
292.94[a]	963.9[a]	2.069[a]	337.98	2456.3	1.008
302.89	1059.8	2.070	342.97	2504.0	1.009
312.77	1106.8	2.071		$z_1 = 0.899$	
323.09	1155.0	2.072	278.12	2162.7	0.857

Table 3. Continued

T	P	V	T	P	V
K	kPa	$dm^3 \cdot mol^{-1}$	K	kPa	$dm^3 \cdot mol^{-1}$
333.06	1201.0	2.073	283.11	2223.8	0.857
343.02	1246.4	2.074	288.09	2283.3	0.858
352.96	1291.3	2.075	293.11	2362.8	0.858
362.92	1335.8	2.075	298.10	2421.7	0.858
372.86	1379.8	2.076	303.08	2480.1	0.858
	$z_1 = 0.466$		308.06	2537.9	0.858
288.02	842.2	2.502	313.04	2593.4	0.858
293.11	862.1	2.503	318.04	2650.2	0.859
298.10	881.7	2.503	323.00	2706.8	0.859
303.08	901.0	2.504	328.01	2762.2	0.859
308.08	920.1	2.504	333.00	2814.5	0.859
313.07	939.1	2.505	338.00	2866.6	0.859
318.04	958.0	2.505	342.98	2914.8	0.860
323.02	976.7	2.506			
328.01	995.3	2.507			
333.00	1013.8	2.507			
337.98	1032.0	2.508			
342.98	1050.8	2.508			

[a] Not considered in the regression.

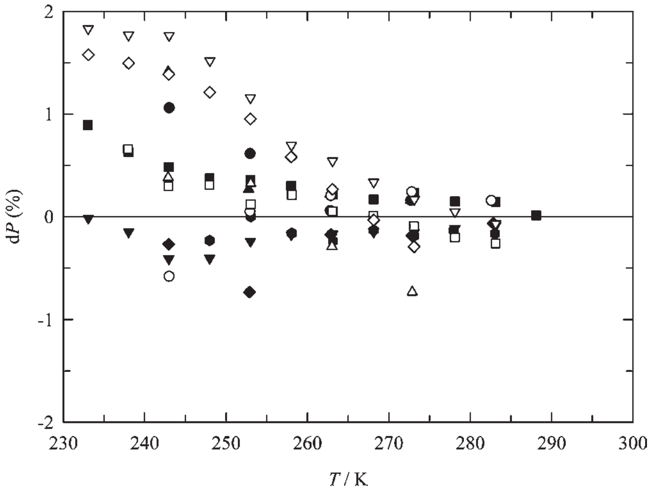

Figure 2. Pressure deviations between experimental values and those calculated with the CSD EOS[14] for the CO_2 (1) + R134a (2) system in the two-phase region. Symbols are denoted as in Figure 1.

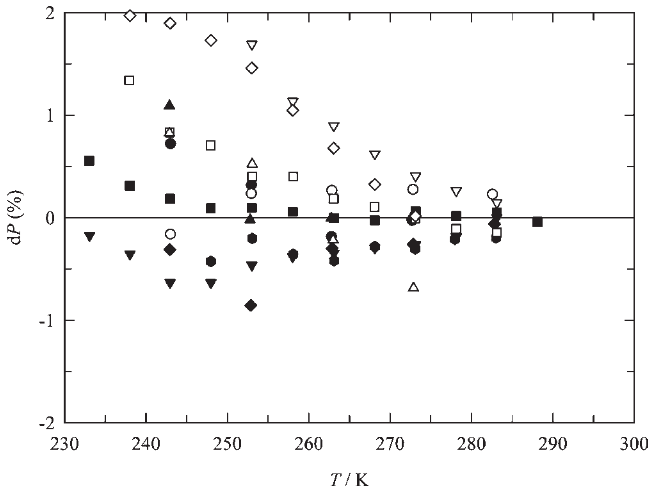

Figure 3. Pressure deviations between experimental values and those calculated with the REFPROP 9.0[18] for the CO_2 (1) + R134a (2) system in the two-phase region. Symbols are denoted as in Figure 1.

Table 4. Summary of Deviation in Pressure for the Literature Sources Obtained with an Average Value of $K_{12} = -0.011$

deviation	present work	ref 11	ref 12	ref 13
AD (dP %)	0.55	0.30	−1.64	0.30
AAD (dP %)	0.70	1.97	1.59	3.12

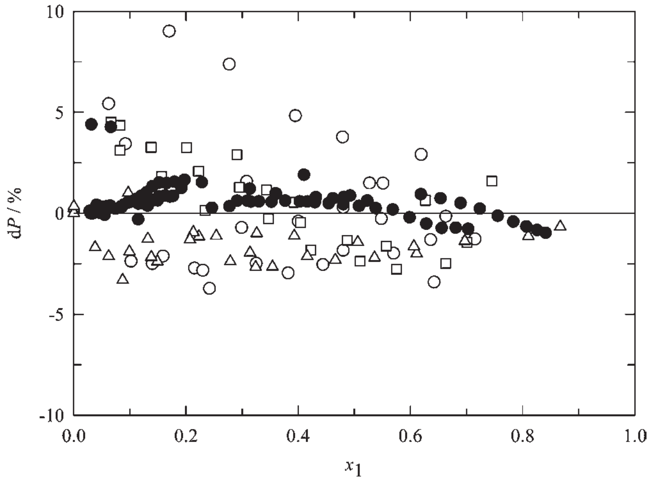

Figure 4. Pressure deviations versus bubble-point composition x_1 between experimental values and those calculated with the CSD EoS[14] with $K_{12} = -0.011$ for the CO_2 (1) + R134a (2) system in the two-phase region: ●, present work; □, Silva-Oliver and Galicia-Luna;[11] △, Duran-Valencia et al.;[12] ○, Lim et al.[13]

including all literature data and combining them as one set with our isochoric data in the two-phase region and assuming at first that K_{12} was independent of temperature. In this way an average value of $K_{12} = -0.011$ was found, and results are reported in Table 4. Results are also shown in Figures 4 and 5, where deviations are reported for the different sources versus bubble-point composition, x_1 and temperature, respectively. From the analysis of results, in our opinion the assumed model, even with only one adjusted temperature-independent parameter, is able to represent all data considered close to their real experimental accuracy, as observed deviations are randomly distributed, both plotted versus bubble-point composition, x_1, and temperature. In addition, from Figure 4 it is also possible to see that deviations for data from Silva-Oliver and Galicia-Luna[11] are slightly s-shaped but generally within 2 to 3 %, while measurements performed by Duran-Valencia et al.[12] showed a generally negative deviation. Data measured by Lim et al.[13] showed a positive deviation, increasing with the bubble point of the first component composition. In addition,

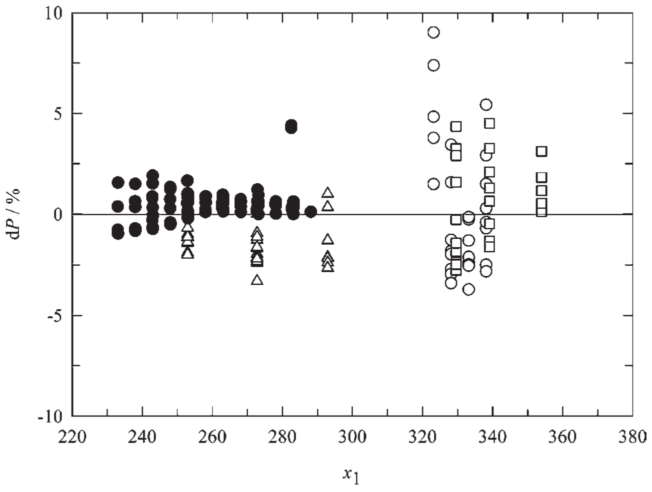

Figure 5. Pressure deviations versus temperature between experimental values and those calculated with the CSD EoS[14] with $K_{12} = -0.011$ for the CO_2 (1) + R134a (2) system in the two-phase region. Symbols are denoted as in Figure 4.

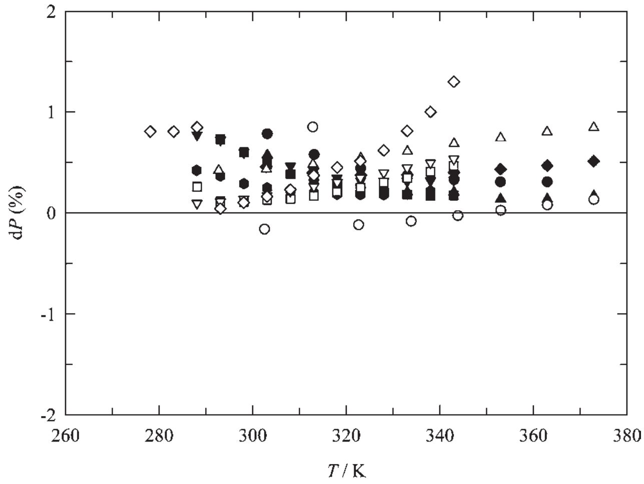

Figure 6. Pressure deviations between experimental values and those calculated with the CSD EOS[14] for the CO_2 (1) + R134a (2) system in the superheated vapor region. Symbols are denoted as in Figure 1.

the Lim et al.[13] at 343.15 K data showed unexpected phase behavior (for some, $x_1 > y_1$) and was not considered in the final data reduction.

PVTx. Since there are no published data on the superheated vapor region for the binary systems considered, pressures at the superheated region were also compared with the CSD EOS[14] and REFPROP 9.0[18] predictions. In this case, the averaged value of $K_{12} = -0.018$ obtained with the flash method in the two-phase region data was used for the CSD EOS[14] calculations. Not considering the points in the proximity of the change of phase that showed much higher deviations (these points were denoted in Table 3 with an asterisk), an AAD (dP%) = 0.37 % was obtained, while better results were achieved comparing the experimental findings with the REFPROP 9.0[18] prediction, obtaining an AAD (dP%) = 0.13 %. Results are summarized in Figures 6 and 7.

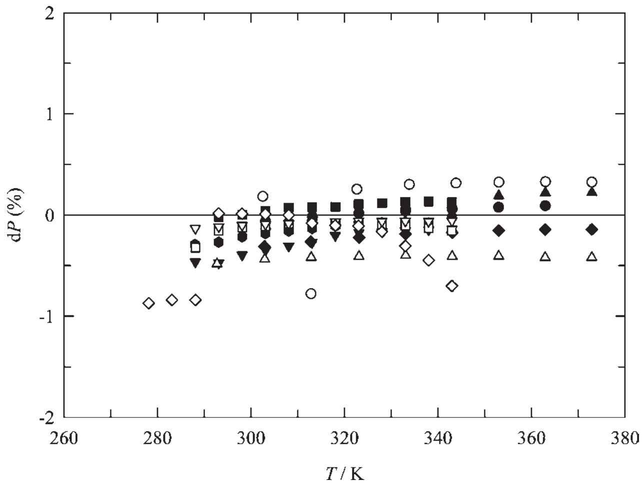

Figure 7. Pressure deviations between experimental values and those calculated with the REFPROP 9.0[18] for the CO_2 (1) + R134a (2) system in the superheated vapor region. Symbols are denoted as in Figure 1.

CONCLUSIONS

An isochoric apparatus has been used to obtain *PVTx* measurements on CO_2 + R134a. The binary interaction parameters were derived from experimental data in the two-phase region, applying the flash method and the Carnahan−Starling−De Santis equation of state. Existing literature data were compared with the two-phase measurements. The *PVTx* data were compared by the CSD EOS and by REFPROP 9.0 prediction.

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