Measurements of the Vapor Pressures of Difluoromethane, 1-Chloro-1,2,2,2-tetrafluoroethane, and Pentafluoroethane

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We present new measurements of the vapor pressures of difluoromethane (R32) from 235 to 265 K, of 1-chloro-1,2,2,2-tetrafluoroethane (R124) from 220 to 286 K, and of pentafluoroethane (R125) from 218 to 286 K. Measurements were made in two ebulliometers, one of glass and one of metal. Overall, pressures ranged from 13 to about 950 kPa. We also present vapor pressures of R125, calculated via thermodynamic relationships, for temperatures down to 170 K (2.3 kPa). We study the azeotropic mixture of R125 with chloropentafluoroethane (R115), and we correct our data for a small R115 impurity.

Introduction

Information about vapor pressures is important initially to qualify potential candidates as working fluids in refrigeration machinery. Later, it is also very useful in calculating the thermodynamic properties necessary for the design of that machinery. These are subjects of much current interest due to the imminent ban on the familiar chlorofluorocarbons (CFC's) and the eventual phase-out of the hydrochlorofluorocarbons (HCFC's) as working fluids.

One major current problem involves finding replacements for chlorodifluoromethane (R22) and its azeotropic mixture with chloropentafluoroethane (R115), known as R502. It is generally conceded that their replacements will be binary or ternary mixtures. Also, it is expected that difluoromethane (R32) and pentafluoroethane (R125) will be components in some of these mixtures. In addition, 1-chloro-1,2,2,2-tetrafluoroethane (R124) may find some use as an interim replacement for dichlorotetrafluoroethane (R114) in specialized applications and as a component in ternary mixtures.

Very few measurements for the vapor pressures of these substances have been published in the literature. Wilson et al. (*1*) presented a vapor pressure curve for R125 but not the experimental data upon which it was based. McLinden (*2*) published a curve for R125 derived from the unpublished data of Shankland et al. (*3*). Recently, Baroncini et al. (*4*) reported new vapor pressures for R125 in our temperature range. For R124 vapor pressures have been given by Kubota et al. (*5*) and by Shankland et al. (*6*). For R32 there are the data of Malbrunot et al. (*7*) and our own recent low-pressure data, Weber and Goodwin (*8*). Overall, however, more data are needed for all three fluids in order to characterize the thermodynamic properties sufficiently well for refrigeration cycle calculations.

We present here new vapor pressure data for these three fluids which were measured in two ebulliometers, one of glass and the other of metal. In general, pressures ranged from 13 to about 950 kPa, and temperatures ranged from 218 up to 286 K. For R125 we also give vapor pressures at lower temperatures, down to the triple point, which were calculated by utilizing recently available thermodynamic measurements (*9*, *10*). We will give a brief description of the apparatus, experimental results, and comparisons with the available data. Because R125 forms an azeotrope with chloropentafluoroethane (R115), we were unable to obtain a highly purified sample. We therefore present a brief study of some of the properties of this binary system in order to adjust our data to compensate for the R115 impurity.

Experimental Section

The data were measured in two comparative ebulliometers. The first was made of glass and was of the Ambrose type (*11*). It has been described several times previously (*12*, *13*); thus, we give only the briefest description here. Two boilers, one containing a refrigerant and the other containing a reference fluid, were connected through a manifold. Heat was supplied to each boiler with a capsule-type heater. Water was used as the reference fluid, and we made use of the new equation for the vapor pressure of water on the ITS-90 scale (*14*) from the International Association for the Properties of Water and Steam (IAPWS) to determine the system pressure. Two long-stem platinum resistance thermometers were used to measure the temperature of the condensing vapor in each boiler with an uncertainty of 2-3 mK. The resistance of the thermometers was measured with a digital multimeter which had been calibrated with a standard resistor. All temperatures are reported on the ITS-90 temperature scale. Helium gas from a low-pressure supply filled the connecting manifold and a 15-L ballast tank used to establish and stabilize the system pressure. A total reflux condenser at the top of each boiler prevented the samples from escaping into the manifold and also provided a well-defined interface between the samples and the helium gas. In contrast with mechanical differential pressure indicators, these fluid interfaces were completely free of problems due to mechanical friction and hysteresis. Two liquid-nitrogen-filled cold traps in the manifold ensured that the two samples were isolated from each other. The reference boiler was surrounded by thermal insulation, and the refrigerant boiler was placed inside a thermostated shield, which was maintained 15 K colder than the boiler. Because of the glass constriction, this system was restricted to pressures less than about 260 kPa.

The other ebulliometer was of all-metal construction, and it was designed to be used at pressures to 3000 kPa or more. A more complete description will be given in a future publication. The overall system had the same general design as the glass apparatus above. The same thermometers and multimeter were used. The boilers were of a different design, however. They utilized a vapor-lift pump which directed a stream of saturated liquid and vapor onto

Table 1. Vapor Pressure of R124 Measured with the Glass Ebulliometer and Deviation Calculated from Eq 1

T/K	P/kPa	$100(P - P_{\mathrm{calc}})/P$	T/K	P/kPa	$100(P - P_{\mathrm{calc}})/P$
221.807	13.6130	−0.050	222.957	14.6194	0.016
223.005	14.6632	0.019	224.068	15.6360	−0.010
225.082	16.6252	0.031	226.970	18.5812	−0.007
228.600	20.4246	−0.009	230.427	22.6687	0.005
231.986	24.7343	−0.005	233.583	27.0102	0.002
235.514	29.9858	0.010	237.217	32.8220	0.001
239.027	36.0745	0.011	241.623	41.1778	−0.011
243.488	45.2053	0.013	243.917	46.1688	0.004
245.626	50.1826	−0.013	247.696	55.4309	0.007
249.587	60.5849	−0.004	251.187	65.2398	−0.008
252.720	69.9698	−0.003	254.415	75.5090	0.000
255.598	79.5825	0.007	257.300	85.7325	0.002
258.405	89.9264	0.004	260.959	100.2270	0.000
263.330	110.5960	−0.010	265.432	120.4910	−0.003
267.404	130.3800	−0.004	269.214	140.0030	−0.003
271.077	150.4810	−0.004	272.883	161.1860	−0.015
274.270	169.8530	−0.006	275.821	179.9460	0.019
279.074	202.4830	−0.020	280.273	211.3800	−0.003
282.200	226.2590	0.003	283.872	239.7530	−0.014
286.098	258.8290	0.027			

Table 2. Vapor Pressure of R32 Measured in the Metal Ebulliometer

T/K	P/kPa	T/K	P/kPa
235.8680	200.3467	249.2701	349.5576
236.3426	204.5835	252.7461	399.6118
236.4328	205.4252	255.9006	449.6160
236.8755	209.4426	258.7972	499.6660
237.0920	211.4449	261.4802	549.7127
237.3066	213.4556	261.4939	550.9834
237.9465	219.4503	263.9999	600.1012
240.9516	249.4776	266.3463	650.1570
245.3832	299.5250		

the thermometer well. Thus, they measured boiling temperatures rather than the condensing temperatures measured by the glass apparatus. Both boilers were surrounded by thermostated shields. In place of the ballast tank, we used a commercial pressure controller which maintained the pressure constant to within ±0.02 kPa at the steady state. The temperature in each boiler was measured to within ±5 mK. Because of the high pressures and temperatures involved, water was not used as the reference fluid. Instead, we used 1,1-dichloro-2,2,2-trifluoroethane (R123) since its vapor pressure had been accurately determined (*15*).

Since the primary measurements in this work are the temperatures of the two boilers, the quality of the results is determined by the accuracy with which we can make these relative temperature measurements and also by the uncertainty in the vapor pressures of the reference fluids. In the glass apparatus the uncertainty in the relative temperature measurements is 2−3 mK, while in the metal apparatus it is approximately 5 mK. The uncertainty in the vapor pressure of water is taken to be 0.01%, and for the R123 reference fluid the uncertainty is about 0.03%.

The samples of R124 and R32 were extremely pure as received from the suppliers, who stated that their mole fractions were 0.999 85 and 0.9999, respectively. The sample of R125 contained 0.26 mol % R115 which could not be removed by distillation. We made no attempt to purify it.

Results

R124. These results were all measured with the glass apparatus. Temperatures ranged from 221.8 to 286 K, and pressures varied from 13.6 to 258.8 kPa. A total of 41 data were measured. The results are given in Table 1. An Antoine equation,

$$\ln(P/\mathrm{kPa}) = A + B/(T/\mathrm{K} + C) \tag{1}$$

was fit to the data, and the parameters were found to be $A = 13.9390$, $B = -2073.45$, and $C = -38.7613$. The resulting fit is shown in Figure 1. The standard relative deviation was 0.014% in P, and only two data deviated by more than 0.03%; one of these was at the lowest temperature (221.8 K, 0.05%). This temperature is about the lowest at which this apparatus should be used; this limit was imposed primarily by the temperature to which we could cool the thermostated shield around the boiler.

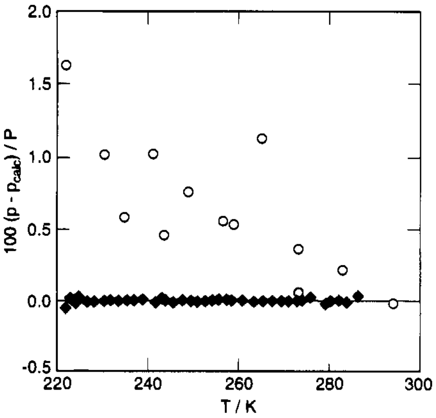

Figure 1. Deviations of the vapor pressure data for R124 from eq 1: (◆) this work, (○) Shankland et al. (*6*).

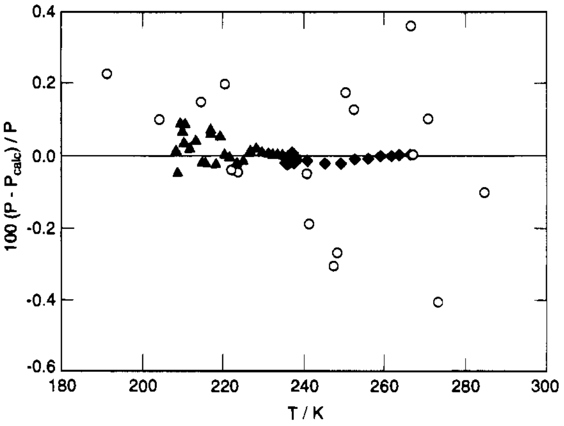

Figure 2. Deviations of the vapor pressure data for R32 from eq 2: (▲) glass ebulliometer (*8*), (◆) this work, (○) Malbrunot et al. (*7*).

R32. The results reported here were measured with the metal ebulliometer. Temperatures ranged from 236 to 266 K, and pressures varied from 200 to 650 kPa. These data bridge the gap between our previously published results from the glass apparatus (*8*), 208−237 K, and static measurements made in our Burnett apparatus (*16*) from 268 to 348 K. The values are given in Table 2. We have represented the present results and the previously published results from (*8*) with a Wagner-type equation,

$$\ln(P/P_c) = T_c/T(a_1\tau + a_1\tau^{1.5} + a_3\tau^{2.5} + a_4\tau^5) \tag{2}$$

with $\tau = 1 - T/T_c$, $a_1 = -7.461\,49$, $a_2 = 1.736\,87$, $a_3 = -1.986\,56$, $a_4 = -2.617\,09$, $P_c = 5793.1$ kPa, and $T_c = 351.36$ K. The deviations are shown in Figure 2. The present data have a very small bias, $\langle 100\delta P/P\rangle = -0.011$, relative to our other data, and they have a precision of 0.006% in P. Since each of the sets of data was measured

Table 3. Vapor Pressure of R125 Measured in the Glass Ebulliometer and Corrected for R115 Impurity

T/K	P/kPa	T/K	P/kPa
218.792	73.7126	219.205	75.3215
219.474	76.3836	220.007	78.5190
220.203	79.3263	220.410	80.1824
220.582	80.9071	220.944	82.4188
221.213	83.5673	221.603	85.2409
222.284	88.2381	222.882	90.9315
222.763	90.3914	223.369	93.1839
224.079	96.5393	225.009	101.0860
226.286	107.5412	226.466	108.4831
226.395	108.1079	227.078	111.7351
227.518	114.1230	228.513	119.6434
229.292	124.1224	230.091	128.8654
231.855	139.8029	234.007	154.1359
233.844	153.0023	235.051	161.4451
236.660	173.3154	238.277	185.8698
237.790	182.0049	239.307	194.2497
240.815	207.0155	242.545	222.4285
244.329	239.2461	245.192	247.7090
245.674	252.5508	246.063	256.5198
246.600	262.0412		

Table 4. Vapor Pressure of R125 Measured in the Metal Ebulliometer and Corrected for R115 Impurity

T/K	P/kPa	T/K	P/kPa
240.020	200.1229	240.022	200.1850
241.183	210.2029	241.188	210.2158
242.305	220.2253	242.306	220.2320
243.388	230.2275	244.430	240.2426
244.430	240.2426	245.419	249.9650
245.419	249.9790	245.444	250.2599
245.474	250.5039	245.476	250.5483
246.399	259.9810	246.412	260.0570
247.360	270.0630	247.381	270.2980
247.381	270.2980	248.288	280.0820
249.190	290.0960	249.206	290.2942
249.222	290.4186	250.068	300.1200
250.957	310.4795	254.153	350.1990
254.182	350.4960	257.816	400.2377
257.839	400.5773	261.144	450.2082
261.171	450.6728	261.800	460.5563
264.205	500.1448	264.228	500.6106
264.254	500.9383	265.126	515.9830
266.796	545.8929	266.523	540.9030
267.063	550.7690	267.065	550.9314
267.071	550.9314	267.072	550.4842
267.088	551.0399	268.420	575.6622
269.712	600.8394	269.722	600.7377
269.738	601.2446	269.739	601.2157
270.987	625.8146	272.203	650.9417
272.204	650.8647	272.205	650.9263
272.206	650.9724	272.211	650.9259
273.402	675.7031	274.555	701.0124
274.561	700.9632	276.783	750.9170
276.787	751.0706	278.905	801.1823
280.924	850.9024	282.858	901.2540
284.711	951.1840	284.712	951.5946
284.714	951.5741		

in a different apparatus with a different technique, this excellent level of agreement gives confidence in the accuracy of our results.

R125. We report here three sets of data for R125 vapor pressures. The first set was measured in the glass apparatus. A total of 39 data were measured, and they ranged in temperature and pressure from 218 to 246 K and from 74 to 262 kPa, respectively. The second set consisted of 65 data measured in the metal apparatus. Temperatures ranged from 240 to 285 K and pressures from 200 to 951 kPa. Results from the first and second sets are displayed in Tables 3 and 4, respectively. Both sets have been adjusted for the small R115 impurity mentioned above, using a technique given in the next section. These two sets have internal precisions of 0.010% and 0.021%, respectively. Because of the high volatility of R125 and the low-temperature limitation of the apparatus, we were unable to make measurements at pressures lower than 74 kPa. Therefore, for the third set of data we have taken advantage of the availability of new thermodynamic results for R125, namely, ideal-gas heat capacities, $C_p°$, derived from speed-of-sound measurements of Gillis (*9*) and saturated liquid heat capacities, C_σ, from Magee (*10*). These data, together with estimates for the virial coefficients of R125 (*17*) and a value for the vapor pressure at a reference temperature, allow the calculation of vapor pressures at lower temperatures from standard thermodynamic relationships and from the condition of equality of the Gibbs free energy across the two-phase boundary. The reference temperature was taken to be 220 K, and the corresponding pressure from the smoothed experimental values was 78.505 kPa. Table 5 gives the calculated results at 5 K intervals for temperatures down to 170 K, which is near the triple point. Densities of the saturated vapor, which are byproducts of this calculation, are also given. These three sets were represented with the *ad hoc* equation

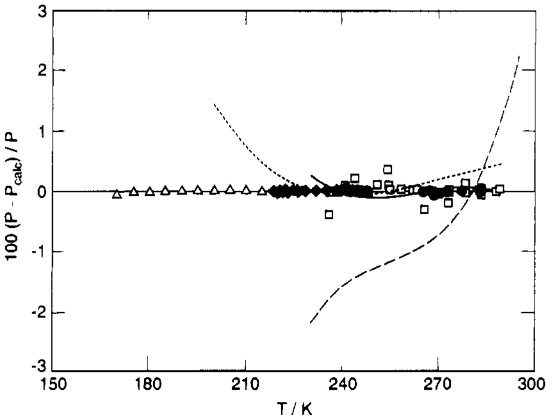

Figure 3. Deviations of the vapor pressure data for R125 from eq 3: (◆) this work, glass ebulliometer, (●) this work, metal ebulliometer (only half of data shown for clarity), (△) this work, calculated values, (□) Baroncini et al. (*4*), (—) curve from ref 4, (- - -) curve of Wilson et al. (1), (— — —) curve of McLinden (*2*).

Table 5. Vapor Pressures and Vapor Densities of R125 Calculated from Thermodynamic Data

T/K	P/kPa	$10^3\rho_v$/(mol·dm^{-3})	T/K	P/kPa	$10^3\rho_v$/(mol·dm^{-3})
170	2.303	1.634	200	24.636	15.057
175	3.660	2.525	205	33.762	20.214
180	5.640	3.790	210	45.450	26.693
185	8.453	5.537	215	60.187	34.719
190	12.350	7.895	220	78.505	44.543
195	17.629	11.012			

$$\ln(P/\text{kPa}) = A + T_c/T(a_1\tau + a_2\tau^4 + a_3\tau^9) \qquad (3)$$

with $\tau = (1 - T/T_c)$. The parameters were found to be $a_1 = -6.8413$, $a_2 = -4.2181$, $a_3 = -0.7594$, $A = 8.1727$, and $T_c = 339.30$. This equation is valid in the temperature range 170–290 K. The resulting fit to the data is shown in Figure 3. All of the measured data are fit extremely well. The calculated data are represented within their estimated uncertainty, $|\delta P/\text{Pa}| = 10^{-4}(P/\text{Pa}) + 3$. The standard deviation is 0.016% in P.

Impurity Adjustments

The R124 and R32 samples were very pure, but the R125 sample contained a 0.26 mol % impurity which was identified by the supplier as R115. This binary system forms a positive azeotrope (*18*), and for this reason it is

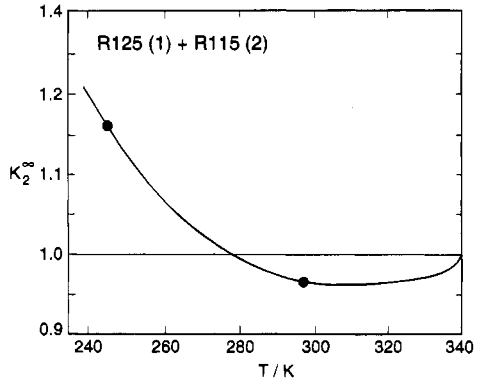

Figure 4. Infinite dilution partition coefficient of R115 dissolved in R125.

very difficult to separate the components. We therefore made a limited study of the phase equilibrium properties of this system in order to determine appropriate adjustments for the vapor pressure results.

The metal ebulliometer was designed for this type of study. Both boilers were loaded with samples of the impure R125, and the helium pressure was set at 250 kPa. The heaters were turned on, and the system reached a steady-state condition at a temperature of about 245 K. After a careful determination of the temperature of each boiler, a small amount of pure R115 was injected into one boiler. The amount of R115 used raised the total impurity level from 0.26 to 0.75 mol %, as measured with a gas chromatograph. When the steady state had been reached again, the temperature change in this boiler was measured and the quantity $(dT/dx_2)_p$ was determined to be -3.67 K, where x_2 is the mole fraction of the R115 solute. Then, assuming that the vapor phase is an ideal mixture of ideal gases, the partition coefficient may be estimated from

$$K_2^\infty \cong 1 - (\mathrm{d}\ln P_1^\sigma/\mathrm{d}T)(\mathrm{d}T/\mathrm{d}x_2)_p \qquad (4)$$

where $K_2^\infty = y_2/x_2$ is the ratio of the mol fractions of R115 in the vapor and liquid phases at infinite dilution, and P_1^σ is the vapor pressure of pure R125. In eq 4 several fugacity coefficients have been set to unity, but the result should be sufficiently accurate for our purposes. The partition coefficient was found to be 1.16 at 245 K. The fact that this value is greater than unity is indicative of a positive azeotrope since R115 is less volatile than R125.

Since K_2^∞ is a function of temperature, it was necessary to make more than one determination of its value. A second measurement was made at ambient temperature by using a gas chromatograph to analyze the composition of the two phases in the sample supply cylinder, which was assumed to be in thermodynamic equilibrium. Measurement of the peak area ratios, solute/solvent, in each phase eliminated the need to determine response factors. A value of $K_2^\infty = 0.956 \pm 0.024$ was found at 297 K.

The results are shown in Figure 4. These two values plus a knowledge of the general behavior of $K(T)$ are sufficient to generate the curve shown in the figure. It can be seen that K remains close to unity, and for this reason separation by distillation would be difficult. The behavior may be approximated by the function

$$K_2^\infty = \exp[a(T_c - T)^{1/3}/T] + b(T_c - T)^{3.2} \qquad (5)$$

with $a = -4.38$ and $b = 1.15 \times 10^{-7}$, and $T_c = 339.3$ K. In eq 5 the first term is taken from the model of Japas and Levelt-Sengers (*19*), and the second is empirical. This curve, which is shown in the figure, should be useable in the range 220 K to T_c. The point of incipient azeotropy is seen to be about 280 K.

Knowledge of K_2^∞ as a function of temperature made it possible to correct the measured vapor pressure data for the impurity. The ebulliometers measure temperature at constant pressure, and therefore it is appropriate to make adjustments of the form δT $(=T_{\mathrm{adj}} - T_{\mathrm{exp}})$ rather than δP. Since the glass apparatus measured condensing temperature and the metal apparatus measured boiling temperature, their corrections were different. For the metal ebulliometer,

$$\delta T = (K_2^\infty - 1)P_1^\sigma x_2/(\mathrm{d}P_1^\sigma/\mathrm{d}T) \qquad (6)$$

where P_1^σ is the experimental vapor pressure. The adjustment was zero near 280 K, and it increased to 0.010 K at 245 K, corresponding to a pressure adjustment of $100\delta P/P = -0.042$. For the glass ebulliometer,

$$\delta T = (K_2^\infty)^{-1}(K_2^\infty - 1)P_1^\sigma x_2/(\mathrm{d}P_1^\sigma/\mathrm{d}T) \qquad (7)$$

At 245 K $\delta T = 0.009$ K and at 220 K $\delta T = 0.015 \pm .002$ K, corresponding to a pressure adjustment of $100\delta P/P = -0.07$. The adjustments given by eqs 6 and 7 have been applied to derive the values in Tables 4 and 3, respectively.

Comparisons

We have compared our results with the rather sparse results available in the literature. For R124 those from Kubota et al. (*5*) only overlap our results at the highest temperatures, 278–286 K, where their pressures are higher than those calculated with eq 1 by 1–7%. The Shankland et al. (*6*) results, seen in Figure 1, agree somewhat better; deviations vary from 0 at 286 K to +1.5% at 218 K. In the case of R32, Figure 2 shows that the results of Malbrunot et al. (*7*) agree with our results within the precision of their measurements, approximately ±0.4%, and show no apparent bias. Agreement with our earlier low-pressure results (*8*) is excellent in the region of overlap, ±0.01% at 237 K. The results for R125 are shown in Figure 3. The curve given by Wilson et al. (*1*) exhibits systematic deviations but remains within the bounds ±0.4% from 218 to 286 K. At lower temperatures, however, their curve is systematically higher than our calculated values in Table 5, by more than 2% at 190 K. The curve of McLinden, which was based on older data (*3*), shows large systematic deviations from our curve, reaching −2% at 228 K. The recent results of Baroncini et al. (*4*) agree with our results quite well. Deviations of most of their values are within ±0.1%, which is within their standard deviation, and there is no apparent bias or systematic deviation in the range 235–286 K. They reported that their sample contained 0.19 mol % R115 and 0.01 mole %N_2. Adjustment for these impurities would improve the agreement somewhat. Comparison of K_2^∞ in Figure 4 with phase equilibrium calculations using a Peng–Robinson (P–R) equation of state indicated that the P–R equation reproduced values of $K_2^\infty(T)$ quite well with a binary interaction parameter, $k_{12} = 0.046$, for the system R125/R115.

Acknowledgment

We are indebted to J. Magee and to K. Gillis for allowing us to make use of their data prior to publication.

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Received for review February 24, 1994. Accepted May 9, 1994.[⊗] Apparatus development for this work was supported in part by the Division of Engineering and Geosciences, Office of Basic Energy Sciences, U.S. Department of Energy. The measurements for this work were supported in part by U.S. Department of Energy Grant Number DE-FG02-91CE23810: Materials Compatibility and Lubricants Research (MCLR) on CFC-Refrigerant Substitutes, and in-kind contributions from the air-conditioning and refrigeration industry. The samples were supplied by E. I. du Pont de Nemours and Co.

[⊗] Abstract published in *Advance ACS Abstracts,* August 1, 1994.