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The Vapor Pressure of 1, 1-Dichloro-2, 2, 2-Trifluoroethane (R123)

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The vapor pressure of 1, 1-dichloro-2, 2, 2-trifluoroethane (R123) has been measured at temperatures between 256.4 and 453.8 K by ebulliometric and static techniques. These results have been combined to obtain a correlation for the vapor pressure from 256.4 K to the critical temperature.

KEY WORDS: 1, 1-dichloro-2, 2, 2-trifluoroethane; ebulliometry; R123; refrigerant; vapor pressure.

1. INTRODUCTION

Recently, we published our ebulliometric measurements of the vapor pressure of 1, 1, 1, 2-tetrafluoroethane (R134a) [1], chlorodifluoromethane (R22) [1], and 1, 1-dichloro-1-fluoroethane (R141b) [2, 3]. In this paper we report vapor-pressure measurements, made both by direct and comparative ebulliometry and by a static method, for 1, 1-dichloro-2, 2, 2-trifluoroethane (R123) at pressures in the range 14.31 kPa to 3.49 MPa, corresponding to temperatures from 256.4 to 453.8 K. Our group has published three papers concerning the vapor pressure of R123 previously [3–5]. The present work covers a wider temperature range and shows that the previous static measurements [4], which had been used in all our previous correlations, were affected by the presence of a volatile impurity. We consider the measurements reported here to be definitive, because all the results obtained with two different samples and two different techniques are in concordance. Throughout the remainder of the text, for the sake of brevity, these compounds are referred to with the numbering scheme used by the refrigeration industry and shown in parentheses above [6].

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2. EXPERIMENTAL

2.1. Ebulliometer

Two ebulliometric techniques have been used for the measurements reported here. For pressures in the range 14 to 202 kPa we have used a comparative ebulliometer, with which the condensing temperature of the substance under study and that of a reference material, water, are measured when the two liquids are boiling at the same pressure. A second ebulliometer was used for condensation temperatures between 300 and 374 K; in this apparatus the pressure was measured directly. Both apparatuses and procedures have been described in detail elsewhere [1–3]; only the important features are discussed here.

The comparative apparatus, which was similar to that described by Ambrose *et al.* [7, 8], has been discussed previously [1, 3] and was used without modification. Condensation temperatures in the comparative ebulliometers were measured on the International Temperature Scale of 1990 (ITS-90) [9] with two long-stem platinum resistance thermometers. The stabilities of the thermometers were checked in a triple point of water cell after the measurements were completed.

The second ebulliometer was constructed from sapphire and stainless steel and sealed with viton o-rings. In the remainder of this text this apparatus is referred to as the sapphire boiler. Two sets of results were obtained with the same boiler but different gauges to measure and different arrangements to control the pressure. In the first set of measurements the pressure of the buffer gas was stabilized by a 10-dm^3 ballast volume. The pressures were measured by a quartz (Paroscientific Model 42KT)[2] pressure transducer with a imprecision of 0.01 kPa and an inaccuracy of 0.3 kPa. When we compared this device to an argon-lubricated piston gauge, we found differences of less than $0.0004p$ at pressures between 100 and 750 kPa. For the second set of measurements the pressure was controlled automatically (D&H Model PC1, operated in its control mode) and measured using another quartz transducer (D&H Model RPM1), with a precision and accuracy similar to those of the transducer described above. At each state point the temperature and pressure were measured 30 times with the data acquisition system and the mean as (ln p) was taken before changing the pressure. After completing both sets of vapor pressure

[2] In order to describe materials and experimental procedures adequately, it is occasionally necessary to identify commercial products by manufacturers' name or label. In no instance does such identification imply endorsement by the National Institute of Standards and Technology, nor does it imply that that particular product or equipment is necessarily the best available the best available for the purpose.

measurements, the two quartz transducers were compared with each other and, with the manufacturer's calibration, found to differ by a constant offset of 1.1 kPa. By comparing these devices to a calibrated barometer, the discrepancies were reduced to less than 0.01 kPa, a level more than adequate for our purpose and well within the manufacturer's quoted accuracy. In the sapphire boiler helium was used in the first set of measurements and argon in the second, as the buffer gas. Based on the observations of Ambrose *et al.* [7, 8], using two different buffer gases for ebulliometry should not give rise to any significant systematic error. The condensation temperatures were measured with a 100-Ω platinum resistance thermometer, which was calibrated (on ITS-90) against the standard long-stem thermometers discussed above. It was found to have an inaccuracy of less than 0.01 K. Before commencing measurements the ebulliometer with the sample ampoule, containing dried and degassed material (see Section 2.2 below), attached to a side arm was evacuated, at a temperature of 300 K, using a rotary vacuum pump until the pressure was below 1 Pa.

2.2. Burnett Measurements

Static vapor-pressure measurements were performed with an automated Burnett apparatus developed at the National Institute of Standards and Technology (NIST). This apparatus has been described in detail elsewhere [10–13]. Temperatures were measured on ITS-90 with an inaccuracy of 1 mK by a platinum resistance thermometer. Pressures were measured with a fused quartz Bourdon gauge, separated from the sample by a precise differential capacitance manometer [10] which was operated as a null instument. When the Bourdon gauge was calibrated against an argon-lubricated balance, the transducer was found to have an inaccuracy and instability of 0.1 kPa.

In our earlier static work with R134a [1] and R123 [4], the sample was degassed in an ampoule by a repeated freezing, pumping, and thawing process until the pressure was invariant. At that time, for R123, Weber [4] indicated, based solely on the measured pressure, a mole fraction of impurity of air of 1×10^{-5}. However, it has been suggested that this technique is not adequate and that vacuum distillation or sublimation is always more effective at removing air [7]. The methods of vacuum sublimation have been discussed in the literature [7, 14]. To eliminate water and air from our sample, we adopted the following procedure. First, the gas-phase sample was dried by passing it slowly over a grade 0.4–nm molecular sieve, previously baked at 500 K for 48 h. The sample was collected downstream of the drier, where it was degassed by vacuum sublimation with a liquid

Table I. Vapor Pressures p and Deviations $\Delta p_1 = p - p(\text{calc.})$ from Eq. (1) and $\Delta p_2 = p - p(\text{calc.})$ from Eq. (2) at Temperatures T for 123

T (K)	p (kPa)	Δp_1 (Pa)	Δp_2 (Pa)	T (K)	p (kPa)	Δp_1 (Pa)	Δp_2 (Pa)	T (K)	p (kPa)	Δp_1 (Pa)	Δp_2 (Pa)
					Comparative ebulliometry						
256.399	14.310	3	1	276.434	37.852	1	4	291.801	71.733^a	1	5
259.769	17.062	−4	−5	277.573	39.804^a	2	5	295.528	82.763^a	−15	−12
259.923	17.202^a	0	0	279.277	42.871	−1	2	298.771	93.456^a	9	12
262.445	19.556	1	2	281.661	47.486	2	5	301.481	103.170^a	3	3
264.828	22.015^a	2	3	284.383	53.225	−2	1	304.700	115.732^a	11	12
266.320	23.671	−6	−5	286.338	57.691	7	10	308.552	132.238^a	−44	−46
269.660	27.774	1	2	287.631	60.791^a	1	3	313.430	155.835^a	−2	11
271.536	30.317^a	1	3	288.492	62.940	8	10	318.126	181.464^a	0	−21
273.098	32.572	−2	1	290.640	68.538	0	1	321.587	202.360^a	23	−18
					Sapphire ebulliometer						
300.616	100.07^b		92	331.690	274.20		143	353.349	491.57^b		−13
301.784	104.27		−27	333.382	287.83^b		87	353.935	499.19		326
306.706	124.22		90	333.625	289.76^b		10	357.804	549.21		220
312.121	149.27		44	334.738	299.20		118	361.401	599.21		316
314.314	160.57^b		127	340.298	349.19		20	361.648	602.73^b		289
316.854	174.27		30	345.266	399.21		126	364.790	649.20		253

Sapphire boiler								
321.063	199.26	159	345.233	399.19	454	367.931	699.03	962
321.071	199.24	89	345.604	402.81[b]	144	369.477	724.18	940
323.689	215.89[b]	−52	349.771	449.19	346	370.975	749.18	910
324.895	224.18	147	349.767	449.21	413	372.425	774.14	1032
324.912	224.24	92	352.534	481.87[b]	281	373.864	799.19	830
328.456	249.33	44						
Static								
333.142	285.599	−171	373.118	785.011	−183	413.123	1756.560	273
338.101	328.571[a]	−101	373.127	785.200[a]	−151	418.090	1920.056	−619
338.157	328.992	−191	378.130	876.721	−117	418.127	1922.265	324
343.114	376.751	−91	383.113	975.358	−259	423.047	2096.589	450
348.077	429.523[a]	−91	388.133	1083.091	−180	423.133	2098.484	−803
348.126	430.038	−124	393.121	1198.673	−80	428.119	2288.711	566
353.126	488.679[a]	−154	393.122	1198.940	163	428.193	2291.213	170
353.150	489.029	−100	398.108	1323.079	−59	433.116	2490.682	284
358.116	553.047	−144	398.122	1322.424	−1076	433.176	2493.146	237
358.134	553.318[a]	−116	403.115	1456.870	−593	437.933	2698.539	75
363.105	623.531[a]	−154	403.121	1457.629	−1	443.171	2940.590	199
363.157	624.258	−196	408.113	1601.212	−256	448.067	3182.075	−500
368.107	700.663	−238	408.134	1602.147	53	452.618	3423.406	239
368.112	700.825[a]	−156	413.062	1754.131	−206	453.791	3487.698	−174

[a] Values obtained with a separate aliquot of batch 6A9101.

[b] Results obtained with sample batch 1988 and the Paroscientific transducer.

nitrogen-cooled finger. This process was repeated three times. A positive displacement pump was then filled with the liquid and a 30-cm^3 aliquot distilled into the apparatus, which had been cooled to 273 K.

2.3. Sample Purity

Distilled water from the laboratory supply was used as the reference material in the comparative ebulliometer. R123 was obtained from Halocarbon Laboratories in two 3.6×10^{-3}-m^3 screw-top containers (from batch numbers 1988 and 6A9101), each with a stated lowest mole fraction purity of 0.9995. A gas chromatographic mass spectrum of the crude sample from batch 6A9101 was performed with a 60-m capillary column containing a nonpolar stationary phase, polymethylsiloxane, for the separation [15]. No chlorofluorocarbon impurities, including 1, 2-dichloro-1, 2, 2-trifluoroethane (R123a), the principal impurity in R123 as manufactured and used, were detected [15]. Karl Fisher coulombic titration of the stock material from batch 6A9101 for water returned a mole fraction of 8.3×10^{-5} [15]. Gas chromatographic analysis (with a thermal conductivity detector and a 3-m column packed with Carbopack and 5% Fluorcol stationary phase operating at 383 K) of batch 1988 indicated the presence in the crude sample of three impurities: water with a mole fraction of $(23 \pm 2) \times 10^{-6}$, R123a, and other unidentified hydrocarbons, both with mole fractions of less than 1×10^{-6} [16]. The sample was dried and degassed by the methods described in Section 2.2. A single sample was recovered from the Burnett cell after the measurements. No air was detected in it by gas chromatographic techniques. Samples from batch 6A9101 were used for the comparative ebulliometry, the second set of sapphire boiler results, and the static measurements reported here. An aliquot of batch 1988 was used for the first set of sapphire boiler measurements (10 values identified by *b* in Table I). This batch was also used for many other measurements including earlier static vapor pressure measurements [4], refractive index [17], surface tension [18], critical properties [19], our earlier ebulliometry [3], and finally, gaseous speed of sound measurements [20].

3. RESULTS

The comparative ebulliometric measurements covered the range 14.3 to 202 kPa, which corresponds to boiling temperatures of 256.4 to 321.6 K. The results were obtained with two separate aliquots of batch 6A9101; the runs are identified in Table I. The vapor pressures of water, the reference fluid, were calculated from the boiling temperatures with the Wagner [21–24] equation given by Goodwin et al. [1].

The values of the vapor pressure obtained from both ebulliometric and the static experiments are listed in Table I, together with deviations from the Antoine, Eq. (1), and Wagner, Eq. (2), smoothing equations, determined at each of the temperatures. In Table I we also identify, with a superscript *b*, those values determined with sample batch 1988 and the Paroscientific quartz pressure transducer. Small corrections have been applied to account for the fluid head in each ebulliometer [5]; for our comparative ebulliometry we calculated a static head correction factor of 1.000174.

4. ANALYSIS AND DISCUSSION

Our vapor pressures determined by the comparative ebulliometric technique and listed in Table I may be represented by the Antoine equation

$$\ln p = 13.91135 - 2381.21/(T - 44.747), \tag{1}$$

where p is in kPa and T is in K. The standard deviation of the fit was 11 Pa, or 3.2 mK in temperature. The normal boiling temperature, at a pressure of 101.325 kPa, calculated from Eq. (1) is (300.984 ± 0.005) *K* and agrees exactly with the value reported by Weber [3], while that of Kubota *et al.* [25] lies 0.284 K below our result; all values were compared on ITS-90 [9, 26] with the conversion formula from Ref. 27.

To provide a Wagner [21–24] equation for the fluid range from 256 K to the critical temperature, we combined our ebulliometric and static measurements listed in Table I with the earlier comparative ebulliometric results of Weber [3]. In the regression analysis, each experimental observation was weighted: the comparative results by $1.4 \times 10^{-3}\, dp/dT$, combined in quadrature with the uncertainty in the vapor pressure of water, while the direct ebulliometric measurements were weighted by $0.17\ \text{kPa}/p$ and the static measurements by $0.15\ \text{kPa}/p$, where 0.17 and 0.15 kPa are estimates of the uncertainty in the pressure measurements respectively. Using the adaptive analysis described previously [1] to select powers of τ, where $\tau = (1 - T/T_c)$, from a bank of terms, the best representation was found to be

$$\ln(p) = 8.205701 + (-7.399609\tau + 1.69998\tau^{1.5} - 2.38621\tau^{2.5} - 3.4009\tau^{5.0})\, T_c/T \tag{2}$$

with the critical temperature T_c of 456.831 K [19] and where p is in kPa. The weighted standard deviation of the fit was 341 Pa, or 3.1×10^{-4} in $\ln p$; the results are shown as deviations from Eq. (2) in Fig. 1. At

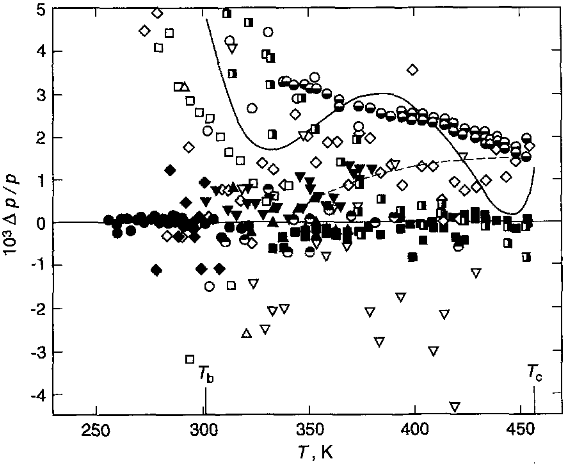

Fig. 1. Fractional deviations $\Delta p/p = \{p - p(\text{calc.})\}/p$ of the experimental vapor pressures from Eq. (2) for R123. T_b is the boiling temperature at a pressure of 101.325 kPa. (●) This work, comparative ebulliometry; (▲) this work, sapphire boiler, sample batch 1988; (▼) this work, sapphire boiler, sample batch 6A9101; (■) static measurements, sample batch 6A9101; (◆) comparative ebulliometry, Ref. 3; (◒) Ref. 4; (▽) Ref. 25; (◓) Ref. 28; (◇) Ref. 29; (◨) Ref. 30; (○) Ref. 5; (△) Ref. 31; (□) Ref. 32; (——) Ref. 34; (----) Ref. 4, corrected for a 7×10^{-5} mole fraction of air.

temperatures between 256.4 and 321.6 K the fractional difference between the vapor pressure calculated from Eq. (1) and that calculated from Eq. (2) is always less than 2×10^{-4}. The two sets of comparative ebulliometry reported here and identified in Table I are indistinguishable for all practical purposes. The precise ebulliometric measurements of Weber [3] are also shown in Fig. 1. They are clearly in excellent agreement with our measurements, although they show more scatter. The lowest datum from Ref. 3 at 271.55 K is in error by more than $0.012p$, and this discrepant result was omitted from the regression analysis. The pressures measured in the sapphire boiler with two samples differ fractionally by less than 2×10^{-4} and are well within the uncertainty assigned to each quartz transducer. Because the results obtained from our comparative ebulliometry and those from the sapphire boiler are in good agreement, we do not attribute a significant part of the scatter in the measurements to the presence of different impurities in the batches of R123.

There are several other determinations of the vapor pressure of R123 in the literature, and as Fig. 1 shows, most that are available at temperatures above 350 K are in good agreement with Eq. (2); all values were compared on ITS-90 [27]. For example, at temperatures between 310 and 420 K, the measurements of Qian *et al.* [28], using a sample with a mole fraction purity of 0.9993, are in good agreement with Eq. (2). Their 12 results with this sample show deviations from Eq. (2) that span from -1.1 kPa at 420 K (about $0.00056p$) to 210 Pa at 330 K (about $0.0008p$). All of these differences are well within the estimated accuracy of their pressure measurements. The static measurements of Yamashita *et al.* [29] at temperatures between 273 and 453 K deviate from Eq. (2) by 204 Pa at 278.15 K (about $0.005p$) and -106 Pa (about $-0.0005p$) at 323.14 K, well within their estimated uncertainty. The values reported by Kubota *et al.* [25] above 308.14 K lie within their quoted $0.01p$ accuracy but, at lower temperatures, diverge in a systematic manner consistent with the presence of a small quantity of volatile impurity to be 1.75 kPa (about $0.05p$) high at 273 K. The more recent measurements by Oguchi *et al.* [30], at temperatures in the range 243.15 to 453.12 K, are in excellent agreement with Eq. (2) at temperatures above 373 K, with differences of less than $\pm 0.001p$, but at lower temperatures they diverge, to lie $0.069p$ high at 253.16 K, in a manner that may be consistent with the presence of a volatile impurity. The earlier measurements of Morrison and Ward [5], which were not included in our analysis, consistently show deviations between $0.0022p$ and $0.0054p$. The measurements of Maezawa *et al.* [31] lie between $0.04p$ above, at 280 K, and $0.026p$ below, at 350 K, Eq. (2); only two of the eight pressures reported in Ref. 31 are small enough to be shown on the scale of Fig. 1. The static measurements due to Baroncini *et al.* [32] deviate from Eq. (2) by $0.0008p$ at 338.09 K and diverge systematically to be 0.01_2p above Eq. (2) at 260.05 K; the differences are as expected for a sample containing a volatile impurity. The precise static measurements of Weber [4], shown in Fig. 1, differ systematically at all temperatures. At 338.126 K (the lowest temperature) his result differs by $0.0034p$ from Eq. (2) and by $0.002p$ at 453.097 K (the highest temperature in Ref. 4). At 373.127 K Weber's result is 2.3 kPa above that given in Table I. Such errors in static vapor pressure measurements could arise from the presence of a volatile impurity [7] or errors in the pressure measurement. The uncertainty in the pressure measurements contributes fractionally only about 1.5×10^{-4} to the error in the vapor pressure but the effects of impurities are more difficult to assess. In Ref. 4 no chemical analysis was performed on a sample recovered from the Burnett experiment; thus we were unable to identify a volatile impurity that might have given rise to the divergence at lower pressures. We suspect that it might have been air. The effect of residual air on the vapor pressure of 2, 2, 2-trifluoroethanol has been discussed quantitatively by Kabata *et al.* [33]. Their measurements, which were performed with once-, twice, and thrice-degassed samples, clearly show the effects of air impurities on the vapor pressure; in Ref. 33 the sample was degassed by repeated freezing pumping and thawing. These results support our hypothesis that the positive divergence from Eq. (2), which increases with decreasing temperature, that we observed for the vapor pressure reported in Refs. 4, 25, and 32 may be attributed to the presence of a volatile impurity such as air. Based solely on the differences discussed above, we applied a simple correction for an air impurity with a mole fraction of about 7×10^{-5} to the values reported by Weber [4]. This correction was calculated assuming that all the air behaved as a perfect gas, and has a negligible solubility in the liquid phase, and that the vapor occupied two-thirds of the cell volume. The corrected results are shown in Fig. 1 and, at the lowest temperatures, are in concordance with the present work. The correlation given by the Japanese Association of Refrigeration (JAR) [34] was derived from information independent of ours [29, 30, 35–37]. It is 709 Pa (about $0.02_2 p$) high at 273 K and 793 Pa (about $0.00024p$) high at 450 K compared with Eq. (2). The earlier correlation of McLinden *et al.* [38] agrees with Eq. (2) within their estimated uncertainty of $0.01p$.

Assuming that the critical temperature of 456.831 K (on ITS-90) recommended by Weber and Levelt Sengers [19] is correct, we calculate from Eq. (2) a critical pressure of (3661.8 ± 0.3) kPa. This value is 12.2 kPa below the value given in Ref. 19 from extrapolation of the vapor pressure [19] of an impure sample. The critical pressure reported in Ref. 19 was printed in error and the correct value should have been 3668 kPa, which is 6.2 kPa above our result. The critical pressure of Yamashita *et al.* [29] lies 13.2 kPa above the present value, and it is within 2.6 times their uncertainty, while the critical pressure recommended by JAR [34] is 4.0 kPa above the critical pressure calculated from Eq. (2).

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