Critical Point and Vapor Pressure Measurements for Nine Compounds by a Low Residence Time Flow Method

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Critical point measurements consisting of critical temperatures and critical pressures have been performed on nine compounds by a flow method with ultralow residence times. These compounds along with their Chemical Abstract Service Registry Numbers (provided by the author) are bis(2-aminoethyl)amine (111-40-0), 2-(2-aminoethylamino)ethanol (111-41-1), 1,4-butanediol (110-63-4), 2-(2-butoxyethoxy)ethyl acetate (124-17-4), 2-(2-ethoxyethoxy)ethyl acetate (112-15-2), 2-methyl-1,3-propanediol (2163-42-0), phenyl acetate (122-79-2), 1,3-propanediol (504-63-2), and propylene carbonate (108-32-7). Vapor pressure measurements are also included for these nine compounds.

Introduction

This work is part of an ongoing investigation of the critical properties for compounds of industrial interest selected in 1997 by sponsors of Project 851 of the Design Institute for Physical Property Data (DIPPR) of the American Institute of Chemical Engineers. This paper reports experimental measurements of the critical properties for nine compounds studied in a flow apparatus with ultralow residence times. Vapor pressure measurements on these nine compounds are also reported.

Experimental Section

The flow apparatus and procedures used for these measurements have been described previously.[1] Table 1 reports measured purities and water contents for the compounds studied in this work. The purities were measured at Wiltec by means of gas chromatography. The water analyses were performed by Karl Fischer titration. Care was taken to purchase the compounds at high purity. Water and dissolved gases were removed by distilling off a small amount of material under vacuum, but no further attempts were made to purify the chemicals. The supplier and Chemical Abstract Service Registry Numbers (CASRN) of each chemical are also listed.

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Temperatures were measured on the ITS-90. Temperature and pressure measurement devices were calibrated to within ±0.1 K and ±0.7 kPa, respectively. The reported critical temperatures and pressures are estimated to have uncertainties of ±2 K and ±2%, respectively. The vapor pressure measurements are estimated to be uncertain to within ±2%.

Results and Discussion

Table 2 presents the measured critical point properties for the nine compounds included in this study. When available, values are compared with values from the literature. Table 2 also lists the estimated critical properties for these compounds. These estimates were made using Joback's method[2] and the boiling points measured as part of this project. Due to the lower residence times possible with this method, these newly measured values are believed to be more reliable than the previous values. Gas chromatographic analyses performed on many of the compounds after the measurements were performed showed no signs of significant decomposition.

Tables 3−11 present vapor pressure data on the nine compounds included in this study. These tables list the measured temperature and the measured and correlated pressures. The reported correlations of the measured data demonstrate the internal consistency of the measured data and show, when applicable, that these data agree with data previously measured on these compounds. The Riedel

Table 1. Source and Purity of Materials

compound	CASRN[b]	analyzed purity/mass %[a] Wiltec	analyzed purity/mass %[a] supplier	water/mass %	supplier
bis(2-aminoethyl)amine	111-40-0	99.7	99.8	0.13	Aldrich
2-(2-aminoethylamino)ethanol	111-41-1	98.8	99.9	0.07	Aldrich
1,4-butanediol	110-63-4	99.4	99[c]	0.04	Aldrich
2-(2-butoxyethoxy)ethyl acetate	124-17-4	99.6	99[c]	0.05	Aldrich
2-(2-ethoxyethoxy)ethyl acetate	112-15-2	98.8	98[c]	0.07	Aldrich
2-methyl-1,3-propanediol	2163-42-0	99.0	98.5	0.01	Aldrich
phenyl acetate	122-79-2	99.9	99.3	0.01	Aldrich
1,3-propanediol	504-63-2	99.6	98.8	0.13	Aldrich
propylene carbonate	108-32-7	99.8	99.99	0.00	Aldrich

[a] On a water-free basis. [b] Provided by the author. [c] These are product specifications instead of actual analyses.

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Table 2. Results of Critical Point Measurements

compound	T_c/K meas	T_c/K previous	T_c/K est[a]	P_c/MPa meas	P_c/MPa previous	P_c/MPa est[a]
bis(2-aminoethyl)amine	709.8	709[b]	676.1	4.38	4.3[b]	4.60
2-(2-aminoethylamino)ethanol	739.2		694.3	4.65		4.82
1,4-butanediol	723.8	727[c]	667.7	5.52	6.22[c]	4.97
2-(2-butoxyethoxy)ethyl acetate	693.9	681[c]	683.0	2.15	3.15[c]	2.05
2-(2-ethoxyethoxy)ethyl acetate	673.5	663[c]	658.5	2.59	2.73[c]	2.46
2-methyl-1,3-propanediol	708.0		651.1	5.35		5.03
phenyl acetate	685.7		684.6	3.59		3.67
1,3-propanediol	718.2	722[b]	657.5	6.55	6.3[b]	5.72
propylene carbonate	762.7		771.1	4.14		4.59

[a] Estimated according to the method of Joback.[2] [b] VonNiederhausern et al.[3] [c] Wilson et al.[4]

Table 3. Vapor Pressure of Bis(2-aminoethyl)Amine

T/K	P/kPa meas	P/kPa corr[a]	% dev	T/K	P/kPa meas	P/kPa corr[a]	% dev
370.50	2.000[b]	2.025	−1.27	615.0	1379	1363	1.13
385.13	3.995[b]	4.014	−0.48	615.2	1380	1369	0.77
391.62	5.331[b]	5.331	0.00	668.8	2757	2751	0.23
401.30	7.990[b]	7.975	0.20	668.8	2758	2754	0.14
408.56	10.67[b]	10.62	0.40	687.6	3448	3431	0.50
414.36	13.31[b]	13.24	0.48	688.0	3448	3451	−0.08
420.46	16.65[b]	16.57	0.51	696.0	3791	3775	0.43
425.52	19.94[b]	19.83	0.55	696.2	3793	3788	0.12
432.15	25.03[b]	24.90	0.55	704.6	4138	4157	−0.46
432.16	25.03[b]	24.90	0.52	704.6	4138	4157	−0.46
432.18	25.04[b]	24.92	0.49	704.8	4137	4166	−0.70
438.82	31.17[b]	31.04	0.44	704.8	4137	4166	−0.70
445.53	38.57[b]	38.43	0.36	705.0	4240	4180	1.43
452.26	47.36[b]	47.24	0.26	705.6	4240	4203	0.88
459.04	57.80[b]	57.72	0.14	707.0	4240	4269	−0.68
465.86	70.10[b]	70.09	0.01	707.0	4240	4273	−0.80
472.72	84.51[b]	84.62	−0.12	707.0	4310	4273	0.85
479.62	101.3[b]	101.6	−0.27	707.4	4309	4273	0.85
486.55	120.8[b]	121.3	−0.40	708.2	4309	4287	0.50
493.52	143.2[b]	144.0	−0.55	708.4	4309	4330	−0.50
500.54	169.0[b]	170.2	−0.69	709.0	4377	4335	−0.62
507.61	198.5[b]	200.2	−0.84	709.2	4378	4364	0.31
514.70	232.0[b]	234.2	−0.98	709.8	4377	4373	0.11
521.83	270.0[b]	272.9	−1.10				

[a] Riedel parameters: $a = 77.31858$, $b = -9473.84$, $c = -7.46276$, $d = 2.500214 \times 10^{-18}$. [b] Steele et al.[6] (used in correlation).

Table 4. Vapor Pressure of 2-(2-Aminoethylamino)ethanol

T/K	P/kPa meas	P/kPa corr[a]	% dev	T/K	P/kPa meas	P/kPa corr[a]	% dev
447.45	12.37[b]	12.50	−1.04	727.2	3793	3815	−0.59
453.65	15.82[b]	15.82	0.04	728.0	3793	3865	−1.92
460.85	20.94[b]	20.56	1.84	729.4	3962	3939	0.57
468.65	26.61[b]	26.94	−1.24	729.8	3962	3962	−0.01
475.55	34.34[b]	33.84	1.45	730.2	3962	3985	−0.58
483.55	42.04[b]	43.28	−2.95	730.2	3962	3991	−0.73
488.25	50.30[b]	50.20	0.19	730.4	3964	4003	−0.97
491.35	57.65[b]	55.02	4.57	732.0	4135	4098	0.92
498.85	66.47[b]	68.18	−2.57	734.2	4151	4226	−1.81
504.05	78.19[b]	78.66	−0.60	735.6	4309	4320	−0.27
719.2	3447	3400	1.35	735.8	4308	4333	−0.58
719.4	3446	3415	0.91	736.2	4305	4352	−1.09
725.8	3792	3744	1.27	737.6	4482	4450	0.71
726.8	3791	3793	−0.07	737.8	4481	4463	0.40
726.8	3791	3799	−0.21	738.8	4655	4530	2.68

[a] Riedel parameters: $a = 170.2815$, $b = -16653.1$, $c = -20.2672$, $d = 8.917558 \times 10^{-18}$. [b] Taken from Daubert[7] (used in correlation).

equation[5] was used to correlate the data in this study and is

$$\ln(P/\text{Pa}) = a + \frac{b}{T/\text{K}} + c\,\ln(T/\text{K}) + d(T/\text{K})^6 \qquad (1)$$

Table 5. Vapor Pressure of 1,4-Butanediol

T/K	P/kPa meas	P/kPa corr[a]	% dev	T/K	P/kPa meas	P/kPa corr[a]	% dev
416.30	3.952[b]	3.914	0.96	561.8	437.1	441.4	−0.99
421.03	4.961[b]	4.930	0.62	562.2	437.8	445.1	−1.67
428.02	6.863[b]	6.844	0.29	584.6	692.9	689.1	0.56
437.15	10.24[b]	10.27	−0.29	585.0	692.9	695.5	−0.37
445.54	14.54[b]	14.62	−0.51	603.2	969.4	961.3	0.83
447.23	15.53[b]	15.65	−0.81	602.8	965.3	953.2	1.25
448.76	16.54[b]	16.65	−0.63	602.4	966.0	946.6	2.00
450.22	17.49[b]	17.65	−0.88	624.4	1371	1358	0.96
452.35	19.04[b]	19.19	−0.79	625.2	1379	1375	0.26
454.19	20.46[b]	20.62	−0.77	626.0	1379	1393	−1.00
456.27	22.15[b]	22.33	−0.83	624.6	1378	1363	1.14
462.38	27.79[b]	28.07	−1.02	624.8	1378	1367	0.78
466.65	32.45[b]	32.77	−0.99	689.8	3446	3461	−0.45
475.96	48.84[b]	45.28	7.29	691.0	3445	3517	−2.08
479.95	51.23[b]	51.72	−0.95	697.2	3792	3817	−0.65
501.43	100.5[b]	100.4	0.13	698.4	3791	3872	−2.13
501.49	100.9[b]	100.6	0.33	703.4	4136	4134	0.04
522.0	176.5	175.7	0.45	704.4	4138	4194	−1.35
525.4	193.7	191.6	1.10	714.0	4826	4753	1.53
546.4	311.0	316.6	−1.82	715.2	4827	4821	0.13
546.8	311.0	318.7	−2.51	723.8	5516	5390	2.30

[a] Riedel parameters: $a = 154.7833$, $b = -15978.3$, $c = -17.9329$, $d = 6.068987 \times 10^{-18}$. [b] Palczewska-Tulinska et al.[8] (used in correlation).

Table 6. Vapor Pressure of 2-(2-Butoxyethoxy)ethyl Acetate

T/K	P/kPa meas	P/kPa corr[a]	% dev	T/K	P/kPa meas	P/kPa corr[a]	% dev
513.0	84.81	81.87	−3.46	609.4	593.0	597.0	−0.68
515.0	85.49	85.91	−0.49	632.0	855.0	861.1	−0.71
540.0	157.2	156.0	0.78	654.2	1179	1203	−2.07
540.8	157.2	158.4	−0.78	672.8	1572	1572	−0.03
570.0	287.5	291.7	−1.44	673.0	1571	1577	−0.36
591.0	424.7	433.5	−2.06	691.0	2068	2024	2.14
589.4	423.3	420.3	0.71	691.8	2068	2046	1.06
590.8	423.3	431.1	−1.84	693.8	2150	2105	2.11

[a] Riedel parameters: $a = 100.0441$, $b = -11866.6$, $c = -10.5249$, $d = 4.255466 \times 10^{-18}$.

where a, b, c, and d are parameters. The values obtained for these parameters by a least-squares regression are given as a footnote with each table. In the case of 1,3-propanediol, some of the data reported by Stein[10] were not used to obtain the correlation listed in Table 10. The deviations for the higher temperature data of Stein suggest that decomposition may have been occurring.

Conclusion

Reliable critical properties have been determined for nine compounds of industrial significance. These data are also

Table 7. Vapor Pressure of 2-(2-Ethoxyethoxy)ethyl Acetate

	P/kPa				P/kPa		
T/K	meas	corr[a]	% dev	T/K	meas	corr[a]	% dev
293.15	0.013[b]	0.013	1.71	597.8	852.2	846.3	0.69
484.6	94.46	95.74	−1.35	598.6	852.2	855.3	−0.37
488.4	100.7	104.8	−4.14	618.2	1162	1142	1.75
512.0	177.2	180.4	−1.80	619.4	1162	1161	0.06
531.6	267.5	270.3	−1.04	639.8	1550	1537	0.81
553.4	406.8	406.6	0.06	661.8	2108	2052	2.63
554.0	407.5	411.7	−1.04	663.0	2105	2084	0.99
573.6	570.9	575.9	−0.88	672.6	2417	2355	2.59
573.6	571.6	576.8	−0.92				

[a] Riedel parameters: $a = 81.60059$, $b = -9600.36$, $c = -8.14715$, $d = 4.242500 \times 10^{-18}$. [b] Wikoff et al.[9] (used in correlation).

Table 8. Vapor Pressure of 2-Methyl-1,3-propanediol

	P/kPa				P/kPa		
T/K	meas	corr[a]	% dev	T/K	meas	corr[a]	% dev
489.2	114.4	114.9	−0.36	613.6	1513	1517	−0.31
487.8	114.4	110.5	3.49	613.0	1510	1503	0.44
489.6	113.8	116.1	−2.09	612.6	1509	1494	0.97
487.8	111.0	110.1	0.78	634.0	2068	2060	0.38
521.6	262.0	262.3	−0.12	634.2	2068	2066	0.13
521.8	263.4	263.5	−0.06	654.4	2760	2738	0.78
536.6	359.2	368.4	−2.55	654.8	2760	2753	0.25
537.2	363.4	373.2	−2.72	678.6	3798	3759	1.00
553.2	518.5	522.7	−0.82	679.6	3797	3802	−0.14
553.4	521.2	523.8	−0.49	698.8	4835	4822	0.26
571.8	755.0	748.5	0.86	699.4	4837	4851	−0.28
572.2	757.0	752.6	0.58	705.2	5182	5206	−0.46
592.8	1091	1085	0.54	705.2	5180	5206	−0.50
593.6	1092	1100	−0.69	707.8	5353	5363	−0.19
612.8	1509	1501	0.50	708.0	5354	5376	−0.41

[a] Riedel parameters: $a = 99.44587$, $b = -11595.9$, $c = -10.3564$, $d = 3.127100 \times 10^{-18}$.

Table 9. Vapor Pressure of Phenyl Acetate

	P/kPa				P/kPa		
T/K	meas	corr[a]	% dev	T/K	meas	corr[a]	% dev
465.8	93.08	94.04	−1.04	606.0	1303	1314	−0.82
490.2	171.7	169.1	1.51	629.8	1816	1811	0.27
490.6	171.7	171.0	0.39	630.2	1816	1823	−0.38
513.6	281.3	279.9	0.52	629.8	1817	1811	0.34
514.0	281.3	281.6	−0.09	630.6	1817	1833	−0.83
539.4	457.1	457.4	−0.07	652.8	2430	2422	0.33
539.6	457.1	459.1	−0.43	653.4	2430	2443	−0.53
561.4	670.9	669.5	0.21	674.8	3174	3155	0.59
562.0	670.2	675.0	−0.72	675.8	3174	3192	−0.59
585.2	972.2	971.8	0.04	681.6	3442	3416	0.75
585.4	972.2	976.2	−0.41	682.4	3442	3452	−0.29
605.2	1304	1299	0.35	685.2	3593	3562	0.87

[a] Riedel parameters: $a = 71.29997$, $b = -8616.02$, $c = -6.73640$, $d = 3.357878 \times 10^{-18}$.

useful in the evaluation of the applicability of current predictive techniques as well as in developing better correlations for estimating critical temperatures and pressures. Accurate vapor pressures are also included for these nine compounds.

Literature Cited

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Table 10. Vapor Pressure of 1,3-Propanediol

	P/kPa				P/kPa		
T/K	meas	corr[a]	% dev	T/K	meas	corr[a]	% dev
480.65	86.18[b]	85.82	0.43	611.45	1665[c]	1515	9.04
489.65	113.1[b]	111.3	1.56	621.15	1976[c]	1770	10.41
500.85	145.5[b]	151.3	−4.02	624.95	2073[c]	1879	9.36
511.85	201.3[b]	201.3	0.03	653.25	3160[c]	2866	9.31
522.65	260.6[b]	262.4	−0.69	656.65	3536[c]	3008	14.95
532.15	326.1[b]	327.7	−0.49	700.8	5515	5433	1.49
542.65	420.6[b]	414.2	1.51	701.8	5516	5503	0.24
551.65	482.6[b]	501.9	−4.00	704.0	5688	5658	0.52
560.35	621.2[b]	600.0	3.42	705.4	5688	5752	−1.13
562.55	622.6[b]	627.0	−0.70	705.4	5860	5760	1.71
571.85	810.1[b]	751.8	7.20	706.8	5860	5862	−0.04
571.95	764.6[b]	753.3	1.49	709.4	6032	6050	−0.29
574.45	817.0[b]	790.0	3.31	715.2	6205	6513	−4.96
578.45	854.3[b]	851.6	0.31	715.4	6378	6521	−2.25
582.45	990.0[c]	916.9	7.40	716.6	6378	6619	−3.77
591.65	1195[c]	1082	9.47	716.0	6549	6578	−0.44
601.45	1366[c]	1282	6.19				

[a] Riedel parameters: $a = 91.33574$, $b = -11145.1$, $c = -9.20254$, $d = 3.140965 \times 10^{-18}$. [b] Stein[10] (used in correlation). [c] Stein[10] (not used in correlation).

Table 11. Vapor Pressure of Propylene Carbonate

	P/kPa				P/kPa		
T/K	meas	corr[a]	% dev	T/K	meas	corr[a]	% dev
368.57	0.669[b]	0.661	1.23	432.07	9.879[b]	9.845	0.35
370.89	0.732[b]	0.744	−1.61	435.69	11.18[b]	11.17	0.07
371.67	0.753[b]	0.774	−2.70	437.71	11.98[b]	11.97	0.06
373.14	0.835[b]	0.833	0.24	441.97	13.80[b]	13.82	−0.13
375.80	0.953[b]	0.949	0.40	444.49	14.99[b]	15.02	−0.23
378.47	1.066[b]	1.081	−1.40	449.01	17.33[b]	17.40	−0.40
381.52	1.253[b]	1.250	0.26	455.60	21.28[b]	21.41	−0.63
384.37	1.433[b]	1.429	0.28	459.10	23.64[b]	23.84	−0.86
388.53	1.736[b]	1.729	0.38	462.07	25.84[b]	26.08	−0.92
391.57	1.990[b]	1.982	0.42	668.6	1380	1407	−1.98
395.66	2.389[b]	2.372	0.74	668.6	1380	1409	−2.11
398.77	2.739[b]	2.711	1.02	725.6	2758	2741	0.62
402.50	3.186[b]	3.172	0.43	762.0	3964	4020	−1.40
406.57	3.775[b]	3.751	0.66	760.4	3966	3959	0.18
410.69	4.462[b]	4.426	0.81	762.2	3961	4032	−1.80
414.61	5.201[b]	5.163	0.74	763.6	4135	4091	1.07
419.07	6.167[b]	6.125	0.67	762.2	4138	4028	2.64
426.83	8.202[b]	8.165	0.46	762.6	4137	4045	2.22

[a] Riedel parameters: $a = 69.98825$, $b = -9331.73$, $c = -6.46065$, $d = 1.726568 \times 10^{-18}$. [b] Hong et al.[11] (used in correlation).

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