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Vapor Pressures of 1-Methyl-2-pyrrolidone, 1-Methyl-azepan-2-one, and 1,2-Epoxy-3-chloropropane

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The saturated vapor pressures were measured for samples, of mole fraction purity of 99.98 %, of 1-methyl-2-pyrrolidone (NMP), 1-methyl-azepan-2-one (*N*-methylcaprolactam), and 1,2-epoxy-3-chloropropane (epichlorohydrin) over wide temperature ranges by comparative ebulliometry. Measurements were made over the temperature range of (380.73 to 475.72) K for 1-methyl-2-pyrrolidone, (402.95 to 510.60) K for *N*-methylcaprolactam, and (315.67 to 388.29) K for epichlorohydrin, respectively. The experimental data were correlated, and overall measures of data reproducibility are given. The results are compared with literature data.

Introduction

Three oxygen compounds (1-methyl-2-pyrrolidone (NMP), 1-methyl-azepan-2-one (*N*-methylcaprolactam), and 1,2-epoxy-3-chloropropane (epichlorohydrin)) being of industrial interest were considered. These compounds, for example, are involved in the caprolactam production process. The reliable simulation of the entire unit, among other data, requires reliable saturated vapor pressures of all compounds in a relatively wide temperature range.

In this work, the saturated vapor pressures of NMP over the temperature range from (380.73 to 475.72) K, of 1-methyl-azepan-2-one over the temperature range from (402.95 to 510.60) K, and of 1,2-epoxy-3-chloropropane over the temperature range from (315.67 to 388.29) K were measured and correlated with the Antoine equation.

Experimental Section

Chemicals. Samples (of 1-methyl-2-pyrrolidone, 1-methyl-azepan-2-one, and 1,2-epoxy-3-chloropropane) of certified mole fraction purity of 99.98 % were used as supplied by CHEMIPAN R & D Laboratories (Warsaw, Poland).

Apparatus and Procedure. Saturated vapor pressure was measured over a temperature range of (380.73 to 475.72) K for NMP, (402.95 to 510.60) K for 1-methyl-azepan-2-one, and (315.67 to 388.29) K for 1,2-epoxy-3-chloropropane, respectively. The comparative ebulliometric technique comprising a dynamic twin-ebulliometer assembly was used as described elsewhere.[1] Temperature was measured on ITS-90 with a platinum resistance thermometer (Leeds & Northrup, model 8163-C) operated in conjunction with a Mueller bridge (Leeds & Northrup, type G-2) and an electronic null detector (Leeds & Northrup, model 9834). To provide replicate data, each equilibrium point was measured six times. The maximum uncertainty in the temperature measurement and the associated pressure inconstancy were estimated at ± 5 mK and ± 6.7 Pa, respectively. To obtain the fits to the Antoine equation

for 1-methyl-2-pyrrolidone

$$\ln(P/\mathrm{kPa}) = 14.65738 - \frac{4112.28}{T/\mathrm{K} - 66.866} \quad (1)$$

for 1-methyl-azepan-2-one

$$\ln(P/\mathrm{kPa}) = 13.80793 - \frac{3800.85}{T/\mathrm{K} - 96.957} \quad (2)$$

for 1,2-epoxy-3-chloropropane

$$\ln(P/\mathrm{kPa}) = 14.49820 - \frac{3249.98}{T/\mathrm{K} - 60.169} \quad (3)$$

the maximum likelihood method was used as described in more detail elsewhere.[1,2] Tables 1 (for 1-methyl-2-pyrrolidone), 2 (for 1-methyl-azepan-2-one), and 3 (for 1,2-epoxy-3-chloropropane) list the observed (T_i, P_i) data pairs, their estimated precision measures (σ_{T_i}, σ_{P_i}), and the deviations (ΔT_i, ΔP_i) between the observed and calculated variables. The ΔT_i and ΔP_i values allow us to check whether the values obtained for error variances are appropriate by assessing whether the variations in these fall properly within their (computed) confidence intervals. Because neither ΔT_i nor ΔP_i alone is adequately representative as an overall measure of reproduction for an individual data point, we suggested[1]

$$\kappa_i^s = \mathrm{sgn}(\Delta T_i)\cdot\kappa_i/\hat{\sigma} \quad (4)$$

where, for the present case

$$\kappa_i = [(\Delta P_i/\sigma_{P_i})^2 + (\Delta T_i/\sigma_{T_i})^2]^{1/2} \quad (5)$$

as an overall measure of data point reproduction, where κ_i is the distance between the *i*th observed and estimated data points in the (P, T) space and σ_T and σ_P are adopted as length units. The κ_i^s values are seen (eq 4) to be scaled with respect to $\hat{\sigma}$ (i.e., to the standard deviation of κ_i given by)

$$\hat{\sigma} = \left[\sum_{i=1}^{n} \kappa_i^2/(n-3)\right]^{1/2} \quad (6)$$

where n is the number of experimental points. The sign of ΔT_i has been attributed to κ_i to have the experimental point located

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Table 1. Experimental Temperatures, T, Orthobaric Pressures, P, Precision Measures, σ_T and σ_P, Calculated Residuals, ΔT ($=T - T_{calcd}$) and ΔP ($=P - P_{calcd}$), and Overall Measures of Data Point Reproduction, κ^s, of 1-Methyl-2-pyrrolidone

T/K	σ_T/K	ΔT/K	P/kPa	σ_P/kPa	ΔP/kPa	κ^s
380.73	0.009	0.00	4.742	0.005	0.005	−0.60
383.12	0.010	−0.01	5.240	0.005	0.009	−1.10
386.38	0.007	−0.01	5.980	0.003	0.007	−1.43
387.92	0.012	−0.01	6.361	0.007	0.008	−0.70
389.88	0.009	0.01	6.862	0.002	−0.001	0.52
393.04	0.009	−0.01	7.775	0.003	0.005	−1.26
400.46	0.013	−0.02	10.287	0.002	0.001	−0.76
403.85	0.007	0.01	11.625	0.003	−0.006	1.62
406.68	0.005	0.01	12.871	0.003	−0.007	1.64
408.96	0.004	0.00	13.962	0.001	0.000	0.50
411.42	0.005	0.00	15.218	0.006	0.002	−0.23
413.77	0.011	0.00	16.500	0.003	0.001	−0.28
416.81	0.004	0.00	18.277	0.005	−0.007	0.91
420.94	0.003	0.00	20.973	0.002	0.000	−0.13
424.65	0.004	0.01	23.650	0.002	−0.002	1.01
427.56	0.005	−0.01	25.964	0.003	0.003	−1.00
432.29	0.004	0.01	30.076	0.001	0.000	0.94
435.85	0.005	−0.01	33.540	0.003	0.002	−0.73
439.12	0.004	0.00	36.990	0.008	0.007	−0.54
441.74	0.003	−0.01	39.963	0.004	0.008	−1.42
444.04	0.006	−0.01	42.732	0.005	0.006	−1.17
446.64	0.005	0.00	46.025	0.004	0.000	−0.09
451.08	0.007	−0.01	52.176	0.007	0.006	−0.80
457.46	0.003	0.00	62.110	0.006	−0.008	0.93
475.72	0.006	0.01	99.402	0.004	−0.001	0.56

Table 2. Experimental Temperatures, T, Orthobaric Pressures, P, Precision Measures, σ_T and σ_P, Calculated Residuals, ΔT ($=T - T_{calcd}$) and ΔP ($=P - P_{calcd}$), and Overall Measures of Data Point Reproduction, κ^s, of 1-Methyl-azepan-2-one

T/K	σ_T/K	ΔT/K	P/kPa	σ_P/kPa	ΔP/kPa	κ^s
402.95	0.014	−0.06	4.019	0.002	0.008	−2.31
405.13	0.033	−0.05	4.390	0.007	0.013	−0.92
411.63	0.029	−0.06	5.651	0.002	0.001	−0.88
415.53	0.010	0.01	6.531	0.003	−0.002	0.35
420.70	0.007	0.00	7.906	0.003	0.000	−0.00
424.29	0.005	0.01	8.986	0.001	−0.002	1.34
428.38	0.009	0.00	10.376	0.002	−0.001	0.19
430.35	0.016	−0.03	11.119	0.003	0.003	−0.78
434.66	0.008	0.01	12.838	0.001	−0.001	0.75
436.35	0.012	−0.02	13.596	0.001	0.000	−0.77
443.09	0.008	0.02	16.887	0.001	0.000	0.77
450.68	0.011	0.00	21.388	0.002	0.000	−0.17
458.30	0.013	0.00	26.820	0.003	0.000	0.13
462.79	0.015	0.02	30.499	0.004	−0.002	0.60
468.95	0.005	−0.02	36.269	0.002	0.003	−1.45
479.21	0.013	0.00	47.694	0.007	0.001	−0.15
510.60	0.004	0.00	101.397	0.012	−0.012	0.49

"below" or "above" the response curve. When systematical errors are absent, the sign of κ_i^s should be randomly distributed, and absolute values should be about unity; large κ_i values ($\gg$1) may indicate outliers. An easy measure of the randomness of κ_i^s is the number of sign changes test. If two neighboring κ_i^s values have opposite signs, then one speaks of a sign change. The total number of sign changes should be roughly equal to $n/2 \pm (n/2)^{1/2}$ (limits at 68 % probability level). The corresponding values are 11 (9 to 15) for NMP, 11 (6 to 10) for 1-methyl-azepan-2-one, and 10 (9 to 15) for 1,2-epoxy-3-chloropropane, where values in parentheses are limits calculated at the 68 % probability level.

The calculated deviations (Tables 1 to 3) in the observed temperature (ΔT) and pressure (ΔP) show a consistently statistical pattern, and especially with regard to temperature, they do not rise in the vicinity of the normal boiling point. This shows that the substance is thermally stable and shows no signs of decomposition as the temperature is increased.

Table 3. Experimental Temperatures, T, Orthobaric Pressures, P, Precision Measures, σ_T and σ_P, Calculated Residuals, ΔT ($=T - T_{calcd}$) and ΔP ($=P - P_{calcd}$), and Overall Measures of Data Point Reproduction, κ^s, of 1,2-Epoxy-3-chloropropane

T/K	σ_T/K	ΔT/K	P/kPa	σ_P/kPa	ΔP/kPa	κ^s
315.67	0.005	0.00	5.916	0.002	−0.002	0.49
316.53	0.010	−0.01	6.183	0.004	0.004	−0.47
318.81	0.010	−0.02	6.915	0.002	0.002	−0.78
321.90	0.010	0.00	8.015	0.029	0.003	−0.04
324.03	0.004	0.00	8.859	0.002	0.001	−0.34
327.03	0.003	0.00	10.175	0.001	0.001	−0.67
330.21	0.004	−0.01	11.745	0.001	0.001	−0.73
334.22	0.003	0.00	14.006	0.002	0.002	−0.66
334.77	0.005	0.02	14.324	0.001	−0.001	1.75
337.79	0.003	0.00	16.305	0.004	−0.003	0.29
339.71	0.002	0.01	17.663	0.002	−0.007	1.72
343.98	0.005	0.00	21.048	0.006	−0.003	0.22
345.99	0.002	0.00	22.818	0.003	0.001	−0.07
347.15	0.005	−0.01	23.904	0.004	0.006	−0.88
350.87	0.004	0.00	27.615	0.003	0.000	0.09
352.30	0.001	0.00	29.158	0.003	−0.010	1.46
354.46	0.002	−0.01	31.661	0.001	0.001	−1.50
357.56	0.001	0.00	35.494	0.008	−0.019	0.94
359.92	0.002	0.00	38.699	0.001	−0.001	0.65
363.28	0.003	−0.01	43.680	0.005	0.018	−1.97
367.42	0.003	0.00	50.425	0.002	−0.001	0.40
371.36	0.009	0.01	57.634	0.007	−0.003	0.51
375.06	0.001	0.00	65.197	0.004	0.009	−1.02
388.29	0.002	0.00	98.816	0.004	−0.004	0.68

Results and Discussion

Saturated vapor pressures for NMP, 1-methyl-azepan-2-one, and 1,2-epoxy-3-chloropropane can be compared with corresponding literature data. Deviations between the experimental data, both the newly measured and that taken from the literature, and the values calculated with relevant equations with parameters fitted to the new data versus temperature are presented in Figures 1 to 3.

For 1-methyl-2-pyrrolidone and temperatures higher than 360 K, Pavlov et al.[3] reported values of the boiling temperatures at (26.66 and 101.32) kPa; Kalinichenko and Yarym-Agaev[4] reported values of the boiling point at (385.0, 395.7, 403.3, and 408.2) K; Gierycz et al.[5] reported values of the boiling point in the range (333.24 to 393.55) K; Zudkevitch et al.[6] reported values of the boiling point at (393.15 and 443.15) K; Gupta and Rawat[7] reported the value of the boiling point at 433.15 K; Giles et al.[8] reported values of the boiling point at (373.15 and 423.15) K; Linek et al.[9] reported saturated vapor pressures in the range (330.00 to 373.17) K; Fischer and Gmehling[10] reported values of boiling points at (six points in the vicinity, ± 0.05, of) (363.56 and 413.37) K; Noll et al.[11] reported values of the boiling point at (351.01 and 380.24) K; Blanco et al.[12] reported saturated vapor pressures in the range (414.45 to 475.10) K; and Domańska and Łachwa[13] reported the value of the boiling point at 373.15 K. Antoine parameters are reported by Riddick et al.,[14] by Dykyj et al.,[15] and in the Floppy Book.[16] The saturated vapor pressure equation is also reported in the ChemCAD Data Base.[17] There exists agreement, within experimental error, of our data with those of Gierycz et al., Giles et al., Linek et al., and Noll et al. Other data, except for those of Blanco et al., can be divided into two groups. The first group consists of the data of Kalinichenko and Yarym-Agaev and Zudkevitch et al. These data exhibit systematic negative deviations from our values. The second group consists of the data of Pavlov et al., Gupta and Rawat, and Fischer and Gmehling with systematic positive deviations from our values. Data in these two groups have deviations calculated using our Antoine parameters as a reference in opposite directions when compared to each other. Similar disagreement is observed (up

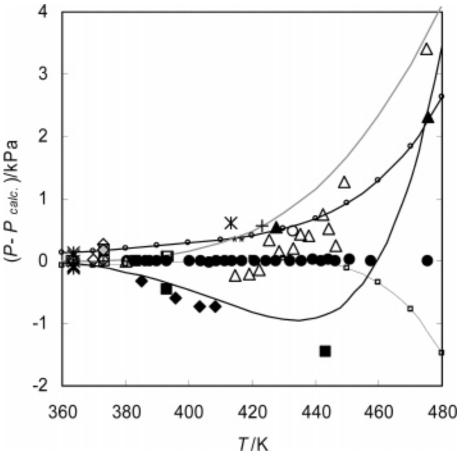

Figure 1. 1-Methyl-2-pyrrolidone deviations between the experimental saturated vapor pressures and the values calculated with eq 1 vs temperature: ●, this work; ▲, Pavlov et al.;[3] ◆, Kalinichenko and Yarym-Agaev;[4] □, Gierycz et al.;[5] ■, Zudkevitch et al.;[6] ○, Gupta and Rawat;[7] +, Giles et al.;[8] ◇, Linek et al.;[9] *, Fischer and Gmehling;[10,] gray triangle, Noll et al.;[11] △, Blanco et al.;[12] gray diamond, Domańska and Łachwa.[13] Solid black line calculated from the Antoine parameters reported by Riddick et al.;[14] line with small squares calculated from the Antoine parameters reported by Dykyj et al.;[15] line with small circles calculated from the Antoine parameters taken from the Floppy Book;[16] gray line calculated from the saturated vapor pressure equation taken from the ChemCAD Data Base.[17]

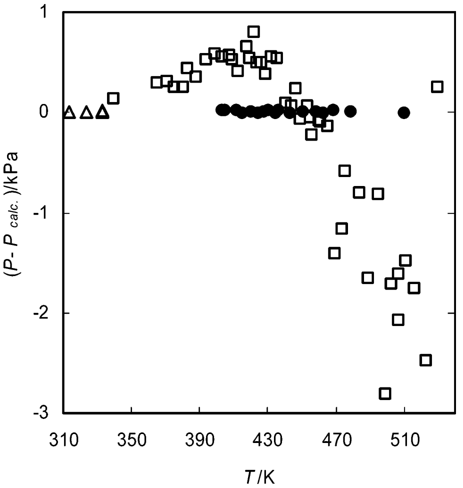

Figure 2. 1-Methyl-azepan-2-one deviations between the experimental saturated vapor pressures and the values calculated with eq 2 vs temperature: ●, this work; □, Shcherbina et al.[18]

to 435 K) for data calculated using equations with literature parameters except for values calculated using the parameters of Svoboda (reported by Dykyj et al.)[15] which agreed excellently up to 440 K, and then systematic negative deviations are observed.

For 1-methyl-azepan-2-one, Shcherbina et al.[18] reported saturated vapor pressures at (339.95 to 529.45) K. These data exhibit systematic deviations from our data. In the temperature range up to 420 K, our data are lower than those of Shcherbina et al. and increase with temperature. These deviations are of 0.799 kPa (8.8 %) at $T = 421.95$ K. In the temperature range (420 to 520) K, deviations decrease to -2.815 kPa (3.8 %) at a temperature of 498.95 K. The point, at 529.45 K, has again positive deviation of 0.255 kPa (0.17 %). It should be noted that the data of Shcherbina et al. exhibit considerable scatter (cf. Figure 2). Fitting with the Antoine equation results in $\sigma(P/\text{kPa}) = 0.376$ and $100 \cdot \sigma(\delta P/P) = 3.91$. Other literature data at temperatures lower than 339 K have been ignored when preparing the residual graph given in Figure 2.

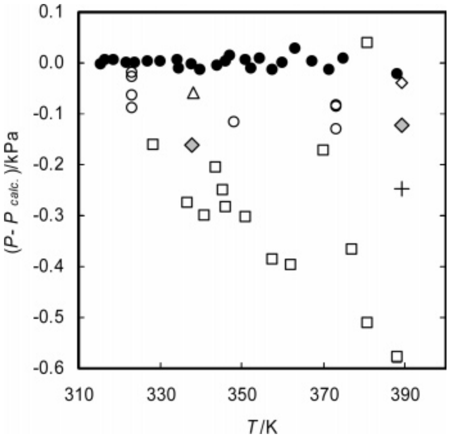

Figure 3. 1,2-Epoxy-3-chloropropane deviations between the experimental saturated vapor pressures and the values calculated with eq 3 vs temperature: ●, this work; □, Urbancova;[19] ○, Giles et al.;[8] ◇, Klon-Palczewska et al.;[20] gray diamond, Denisova et al.;[21] Δ, Jonasson et al.;[22] +, Weast and Grasselli.[24]

For 1,2-epoxy-3-chloropropane, Urbancova[19] reported saturated vapor pressures in the range (328.35 to 388.15) K; Klon-Palczewska et al.[20] reported a value of the normal boiling point; Denisova et al.[21] reported values of the boiling point at (337.65 and 389.17) K; Jonasson et al.[22] reported the value of the boiling point at 338.15 K; and Giles et al.[8] reported values of the boiling point at (323.15, 348.15, and 373.15) K. Data of Urbancova exhibit systematic deviations from our data; it should be noted that as absolute deviations increased the corresponding relative deviations decreased. Data of Giles et al. agreed with our data within two times the error in pressure reported by the authors. Also, the remaining data, being systematically below our data, agreed reasonably well. In refs 22 and 23, values of the normal boiling point are reported. The first value, 288.35 K, reported after a Union Carbide Corporation product bulletin (1968), significantly deviates from our and other estimates for the normal boiling point; therefore, this point has been ignored when preparing the residual graph given in Figure 3. Antoine parameters reported by Riddick et al.[14] and Dykyj et al.[25] were fitted to data of Urbancova and therefore are ignored in the figure.

Registry Numbers Supplied by the Authors. 1,2-Epoxy-3-chloropropane, 106-89-8; 1-methyl-azepan-2-one, 2556-73-2; 1-methyl-2-pyrrolidone, 872-50-4.

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