

MEASUREMENT AND ESTIMATION OF VAPOR PRESSURES OF DIMETHYL, DIETHYL, DIISOPROPYL AND DIBUTYL SUCCINATES AT REDUCED PRESSURE

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Key Words: Vapor Pressure, Dimethyl Succinate, Diethyl Succinate, Diisopropyl Succinate, Dibutyl Succinate

Vapor pressures of dimethyl, diethyl, diisopropyl and dibutyl succinates were measured in the ranges of 0.6–100, 0.6–100, 0.4–100 and 0.5–15 kPa respectively. The percent root mean square deviations of pressure of the four succinates by fitting the Cragoe equation were 0.36, 0.34, 0.28 and 0.94 respectively.

When, in addition to the information for the molecular structure of a substance of interest the value of the normal boiling point, T_b , was known, the Lee-Kesler and Gomez-Thodos equations with values of $\theta (= T_b/T_c)$ and P_c obtained from the Joback method were separately applied to the estimation of vapor pressures for the succinates. The equations mentioned above were also extended successfully to the estimation of vapor pressures where only the value of any one datum point at the reduced pressure, in addition to knowledge of the molecular structure, was known.

Introduction

Few experimental vapor pressure data for dialkyl succinates have been published, and their accuracy is sometimes not enough to be useful in chemical practice.

The vapor pressures of dimethyl, diethyl, diisopropyl and dibutyl succinates were measured in the respective pressure ranges of 0.6–100, 0.6–100, 0.4–100 and 0.4–15 kPa. The results were analyzed by

fitting the Chebyshev polynomial and the Cragoe, Frost-Kalkwarf and Antoine equations. It would be convenient in the design and operation of various kinds of chemical plants to be able to determine the vapor pressures of all the substances of interest from molecular structures alone, but no method for doing so has yet been developed.

Knowing only one point of vapor pressure for each of these succinates, we tried to estimate the pressures by using the Lee-Kesler and Gomez-Thodos equations combined with $\theta (= T_b/T_c)$ and P_c from the Joback method.

* Received December 25, 1991. Correspondence concerning this article should be addressed to H. Katayama.

1. Experimental

Special-grade materials from Tokyo Kasei Co. Ltd. were used without further purification. The purity of each substance was determined by gas chromatographic analyses with a 4 mm-I.D. and 2 m-long column of 5% silicone SE-52 on celite. In all cases it exceeded 99%.

Table 1 shows the densities and refractive indexes compared with published values.^{3,5,13,16,19,20}

The experimental apparatus and procedure have been described elsewhere.^{11,12} The accuracy of the pressure gauge used was ± 2 Pa for the 10–34 kPa range, and ± 1 Pa for the 0.4–10 kPa range. The accuracy of atmospheric pressure measurement, by a Fortin mercury barometer, was ± 0.01 kPa. For dimethyl and diethyl succinates the temperatures were measured on a mercury thermometer set, described previously, with an accuracy of ± 0.04 K. The temperatures of diisopropyl and dibutyl succinates were measured using a platinum resistance thermometer F25 from Chino Co., Ltd., Tokyo with an accuracy of ± 0.05 K and a resolution of 0.0001 K.

Dibutyl succinate decomposed during heating under atmospheric pressure, and the condensate of the succinate changed to a substance of sherbet consistency in the circulation reservoir. Some portion of the succinate was thought to change into succinic acid in the solid state. The vapor pressure was measured at 15–0.4 kPa to avoid decomposition during the experiment.

2. Results and Discussion

The experimental results are presented in **Table 2**, and are plotted in **Fig. 1** along with the values taken from the literature.^{3,5,9,14,20}

Table 1. Densities ρ and refractive indexes n_D of four succinates at 293.15 K

	ρ [kg/m ³]		n_D [—]	
	Exptl	Lit.	Exptl ^{*3)}	Lit.
Dimethyl succinate	1119.87 ^{*1)}	1119.7 ¹⁶⁾ 1119.8 ²⁰⁾	1.42035	1.4195 ¹⁹⁾ 1.4196 ⁵⁾
Diethyl succinate	1040.44 ^{*1)}	1040.6 ³⁾ 1040.4 ¹³⁾ 1040.2 ¹⁹⁾	1.42076	1.4198 ¹²⁰⁾ 1.4201 ³⁾
Diisopropyl succinate	984.7 ^{*2)}	984.7 ²⁰⁾	1.41807	1.4177 ¹²⁰⁾
Dibutyl succinate	976.8 ^{*2)}	976.7 ¹⁶⁾ 976.0 ³⁾	1.42963	1.4298 ¹⁶⁾ 1.4298 ³⁾

*1) Pycnometers (50 ml) newly made from reference to Schroeder *et al.*¹⁸⁾ were used with error of ± 0.02 [kg/m³].

*2) Commercial Ostwald pycnometers (10 ml) were used with error of ± 0.2 [kg/m³].

*3) A dipping refractometer from Carl Zeiss Jena Co. was used with error of ± 0.00002 unit.

2.1. Correlation of the results

To examine the reliability of the experimental results, they were fitted to the Chebyshev polynomial¹¹⁾ and to the Cragoe equation,⁴⁾ as shown in Appendix 1, and to the Frost-Kalkwarf⁸⁾ and Antoine equations. The latter equations were omitted because of their lesser importance in the discussion below.

Here the least-square fit of $t \ln(P)$ vs. t was selected as the function to be minimized, because the fit of $T \ln(P)$ vs. T was found to give a far inferior

Table 2. Vapor pressures of four succinates

Dimethyl succinate					
P [kPa]	T [K]	P [kPa]	T [K]	P [kPa]	T [K]
101.42	468.73	6.000	385.31	1.301	354.68
32.000	429.48	5.000	381.28	1.201	353.25
30.000	427.60	4.000	376.46	1.101	351.78
25.000	422.18	2.000	362.46	1.001	350.15
15.000	407.99	1.901	361.58	0.901	348.51
10.000	397.60	1.801	360.58	0.801	346.52
9.001	394.88	1.701	359.67	0.701	344.61
8.001	392.02	1.601	358.49	0.601	342.18
7.001	389.01				
Diethyl succinate					
P [kPa]	T [K]	P [kPa]	T [K]	P [kPa]	T [K]
99.84	488.03	6.002	402.59	1.501	372.95
32.000	448.23	5.002	398.37	1.401	371.49
30.002	446.19	4.000	393.42	1.301	370.14
25.000	440.59	3.001	387.02	1.201	368.78
20.000	434.01	2.001	378.47	1.101	366.97
15.000	425.91	1.901	377.44	1.001	365.32
10.000	415.17	1.801	376.53	0.901	363.53
9.000	412.58	1.701	375.40	0.701	359.12
8.002	409.71	1.601	374.23	0.601	356.56
7.000	406.22				
Diisopropyl succinate					
P [kPa]	T [K]	P [kPa]	T [K]	P [kPa]	T [K]
99.62	496.83	3.000	394.28	1.200	375.70
32.000	456.62	2.000	385.73	1.100	374.10
25.000	449.16	1.900	384.67	1.000	372.35
20.000	442.30	1.800	383.60	0.900	370.43
10.000	423.15	1.700	382.44	0.800	368.40
9.000	420.33	1.600	381.21	0.700	366.09
8.000	417.21	1.500	379.96	0.600	363.53
7.000	413.82	1.400	378.66	0.500	360.67
5.000	405.62	1.300	377.22	0.400	357.22
Dibutyl succinate					
P [kPa]	T [K]	P [kPa]	T [K]	P [kPa]	T [K]
15.000	475.72	2.000	426.35	0.900	409.66
10.000	464.30	1.700	422.75	0.800	407.36
5.000	447.67	1.500	420.04	0.700	404.46
3.500	438.82	1.300	417.00	0.600	401.98
3.000	435.72	1.101	413.63	0.500	397.82
2.500	431.45	1.001	411.79		

correlation.

Table 3 shows the percent root mean square deviations of pressure (prms) using the Chebyshev polynomial with 3–10 parameters, and the Frost-Kalkwarf and Antoine equations. The polynomial

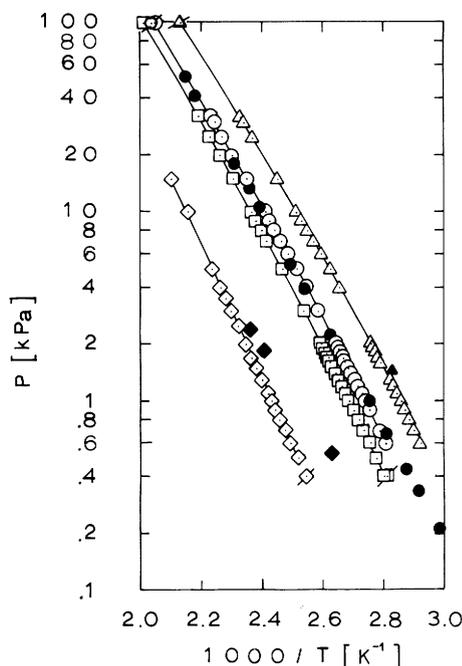


Fig. 1. Vapor pressures of four succinates vs. $1/T$. \triangle , \circ , \square and \diamond : dimethyl, diethyl, diisopropyl and dibutyl succinates respectively, in this work. Dimethyl succinate (\blacktriangle : Kharasch *et al.* (1945), \triangle : Vogel (1948)). Diethyl succinate (\bullet : Hieber and Reindl (1940), \odot : French (1947)). Diisopropyl succinate (\square : Vogel (1948)). Dibutyl succinate (\blacklozenge : Contzen-Crowet (1926), \diamond : Vogel (1948)).

All the solid lines were drawn on the basis of the Cragoe equation with the constants of Table 4.

with four parameters, i.e. the Cragoe equation, gives a good correlation. The constants of the Cragoe equation are listed in **Table 4**.

The larger deviation of dibutyl succinate may be due to the unstable and fluctuating circulation flow of vapor condensate during the experiment, induced by the high viscosity of the condensate.

2.2 Estimation of vapor pressure from normal boiling point alone

The vapor pressure of substances having high boiling points are not often known over a wide range of pressure, and sometimes only a few datum points are available. It is convenient that we can estimate the values of vapor pressure over a wide range from few data. Using only the values of the normal boiling points for the succinates, estimations of the boiling points at lower vapor pressures have been made by using the Lee-Kesler^{15,17)} and Gomez-Thodos^{7,8,17)} equations.

The Lee-Kesler equation is expressed as follows for nonpolar hydrocarbons.

$$\ln(P_r) = f^{(0)}(T_r) + \omega f^{(1)}(T_r) \quad (1)$$

$$f^{(0)}(T_r) = 5.92714 - 6.09648/T_r - 1.28862 \ln(T_r) + 0.169347 T_r^6 \quad (2)$$

$$f^{(1)}(T_r) = 15.2518 - 15.6875/T_r - 13.4721 \ln(T_r) + 0.43577 T_r^6 \quad (3)$$

where T_r ($=T/T_c$) is a reduced temperature, P_r ($=P/P_c$) a reduced pressure. T_c critical temperature K, P_c critical pressure kPa, and ω an acentric factor.

Substituting $T_r = \theta$ and $P_r = 101.325/P_c$ in Eq. (1), Lee and Kesler obtained the value of ω as follows:

Table 3. The percent root mean square deviations of pressure (prms)^{*1)} using the Chebyshev polynomials, and the Frost-Kalkwarf and Antoine equations

	Chebyshev polynomials								Frost-Kalkwarf Eq.	Antoine Eq.
	number of parameters									
	3	4 ^{*2)}	5	6	7	8	9	10		
Dimethyl succinate	2.42	0.36	0.34	0.34	0.31	0.31	0.31	0.30	0.43	0.79
Diethyl succinate	2.44	0.34	0.32	0.31	0.31	0.31	0.31	0.31	0.41	0.49
Diisopropyl succinate	2.13	0.28	0.28	0.27	0.21	0.21	0.19	0.18	0.40	0.98
Dibutyl succinate	1.04	0.94	0.94	0.72	0.71	0.66	0.64	0.47	0.94	1.31

*1) prms was defined as $100[\sum_i ((P_{\text{exptl}} - P_{\text{calcd}})/P_{\text{exptl}})^2 / n]^{1/2}$. n : number of data.

*2) These values are identical with those of the Cragoe equation.

Table 4. Cragoe constants of four succinates

	A_c	B_c	C_c	D_c
Dimethyl succinate	-1.49762	-135.8949	0.471814×10^{-1}	-0.631502×10^{-4}
Diethyl succinate	-2.52986	-145.3541	0.507060×10^{-1}	-0.668404×10^{-4}
Diisopropyl succinate	-1.57359	-218.8291	0.429489×10^{-1}	-0.490376×10^{-4}
Dibutyl succinate	-7.29122	-74.4031	0.679076×10^{-1}	-0.824560×10^{-4}

Table 5. Critical properties of four succinates calculated by the Ambrose and Joback methods

	θ		Ref.	P_c [kPa]		$\omega^{*1)}$
	Ambrose	Joback		Ambrose	Joback	
Dimethyl succinate	0.7105	0.7142		3070	3140	0.6040
Diethyl succinate	0.7319	0.7370	0.7404* ²⁾	2510	2560	0.7043
Diisopropyl succinate	0.7449	0.7496		2140	2210	0.7448
Dibutyl succinate	0.7665	0.7773		1840	1796	0.9262

*¹⁾ The values were obtained from Eq. (4) with the values of θ and P_c of the Joback.

*²⁾ The value was obtained from the values of T_b and T_c compiled by Reid *et al.*¹⁷⁾

$$\omega = [\ln(101.325/P_c) - f^{(0)}(\theta)] / f^{(1)}(\theta) \quad (4)$$

where $\theta (= T_b/T_c)$ is a reduced temperature of the normal boiling point T_b .

The other equation used is of Gomez and Thodos, which is shown in Appendix 2.

If we know the value of θ in addition to the values of T_c and P_c , we can calculate any point of vapor pressure from Eqs. (1)–(4), and from Eqs. (A-5)–(A-8) separately.

To obtain the values of θ and P_c for the succinates, the Ambrose²⁾ and Joback¹⁰⁾ methods, which are based on group contribution techniques, were used. According to the Joback method,

$$\theta = 0.584 + 0.965 \sum \Delta_\theta - (\sum \Delta_\theta)^2 \quad (5)$$

$$P_c = 100.0 (0.113 + 0.0032 n_A - \sum \Delta_p)^{-2} \quad (6)$$

where n_A is the number of atoms in the molecule. The dimensionless quantities Δ_θ and Δ_p , which are shown in a table, represent the increments of the group contributions for various atoms or groups of atoms.

The values of θ and P_c for the succinates are shown in **Table 5**. The θ value of the Joback is larger than that of the Ambrose by 0.5–1.4%, and the P_c value of the Joback is larger than that of the Ambrose by 50–70 kPa except for dibutyl succinate. We have a comparable value of θ of diethyl succinate only with that cited by Reid *et al.*¹⁷⁾ The θ value of the Joback resembles that of Reid *et al.*, but that of the Ambrose is smaller than the latter. For all the succinates, the values of θ and P_c estimated from the Joback method were therefore used in the discussion below.

The values of ω , which are also listed in **Table 5**, were calculated from Eq. (4) by using the values of θ and P_c of the Joback.

Substituting $P = 101.325$ kPa in the Cragoe equation with the constants of **Table 4** yields the values of T_b for dimethyl, diethyl and diisopropyl succinates as 468.68, 488.63 and 497.50 K respectively. Then the values of T_b , and of θ from the Joback method, give the values of $T_c (= T_b/\theta)$ for the succinates as 656.2, 663.0 and 663.7 K respectively.

The results of calculations using the Lee-Kesler and Gomez-Thodos equations are plotted in **Fig. 2** and

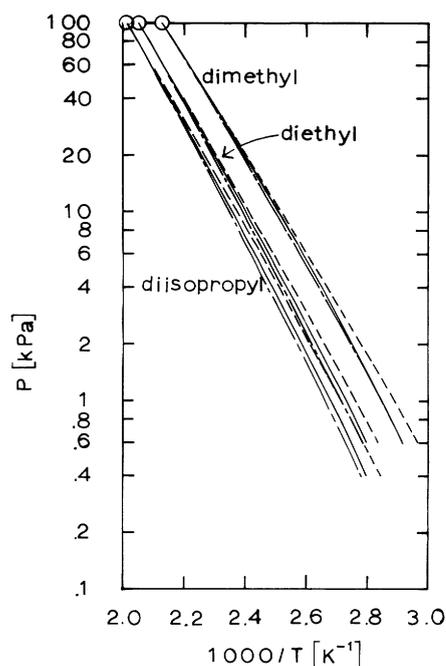


Fig. 2. Estimation of vapor pressures for three succinates under the assumption that for each compound the normal boiling point was known beforehand. \circ : normal boiling point. Solid lines: drawn from the experimental values. Broken lines: obtained from the Lee-Kesler equation. Dotted lines: obtained from the Gomez-Thodos equation

the deviations are summarized in **Table 6**. The temperatures calculated from the Lee-Kesler equation are larger than the experimental values over the entire pressure range; the temperature differences in the pressure range of 30–0.6 kPa are 1–3.1 K. The temperatures calculated from the Gomez-Thodos equation are smaller than the experimental ones; the lower pressures give in the larger temperature differences, and the differences in 30–0.6 kPa are 0.01–5.3 K.

2.3 Estimation of vapor pressure from any one vapor pressure-temperature point

In the section above, the estimation of vapor pressure was carried out from the normal boiling point. However, the normal points for some substances are not known, or cannot be frequently measured because of decomposition during heating,

Table 6. Differences between calculated temperatures, from the Lee-Kesler and Gomez-Thodos equations, and experimental ones. Where normal boiling point is known beforehand.

	Pressure [kPa]											
	0.600		1.000		2.000		6.000		10.000		30.000	
	LK* ¹⁾	GT* ²⁾										
	ΔT [K]		ΔT [K]		ΔK [K]		ΔT [K]		ΔT [K]		ΔT [K]	
Dimethyl succinate	-0.27	5.28	-1.11	4.04	-1.68	2.86	-1.90	1.51	-1.65	1.13	-1.08	0.39
Diethyl succinate	-2.80	3.68	-3.07	2.90	-3.14	2.06	-2.67	1.16	-2.43	0.72	-1.59	0.01
Diisopropyl succinate	-2.33	4.47	-2.68	3.56	-2.73	2.69	-2.45	1.45	-1.88	1.34	-1.10	0.42

$\Delta T = T_{\text{expt}} - T_{\text{calcd}}$, T_{expt} : experimental or smoothed values.

*¹⁾ comparison with values calculated from the Lee-Kesler equation.

*²⁾ comparison with values calculated from the Gomez-Thodos equation.

as in the case of dibutyl succinate. When we know only one vapor pressure-temperature datum point of a reduced pressure for the substance of interest, it would be more convenient if we could estimate vapor pressures from that point and a knowledge of the molecular structure.

This was attempted by use of Eqs. (1) and (A-5). The values of θ , P_c and ω for the succinates are already available from Table 5. We emphasize again that all the values were obtained from a knowledge of the molecular structures alone.

Since the value of T_b is unknown, and the critical temperature T_c is not obtainable directly from $T_c = T_b/\theta$, the value of T_c is separately obtained through iterated calculations of Eqs. (1)–(3) and of Eqs. (A-5)–(A-8) at one datum point given. As an example, for dimethyl, diethyl and diisopropyl succinates the point of 7.000 kPa was taken as the point known beforehand and the point of 3.000 kPa for dibutyl succinate. The values of T_c obtained from the Lee-Kesler equation are 653.30, 658.89, 658.24 and 695.38 K for dimethyl, diethyl, diisopropyl and dibutyl succinates respectively. Those from the Gomez-Thodos equation are 658.71, 664.76, 665.90 and 705.18 K respectively. The values of T_c by these two equations are generally different from each other.

The results of estimation are shown in Fig. 3 and Table 7. For dimethyl, diethyl and diisopropyl succinates in the range of the pressure lower than 7.000 kPa, the results of the Lee-Kesler equation are superior to those of the Gomez-Thodos equation, but at higher pressures the former is inferior to the latter. For dibutyl succinate the opposite case is true.

Conclusion

The vapor pressures of four succinates were measured at reduced pressures, and the results were correlated by the Chebyshev polynomial and by the Cragoe, Frost-Kalkwarf and Antoine equations to examine the reliability of the data.

When the normal boiling point is already known

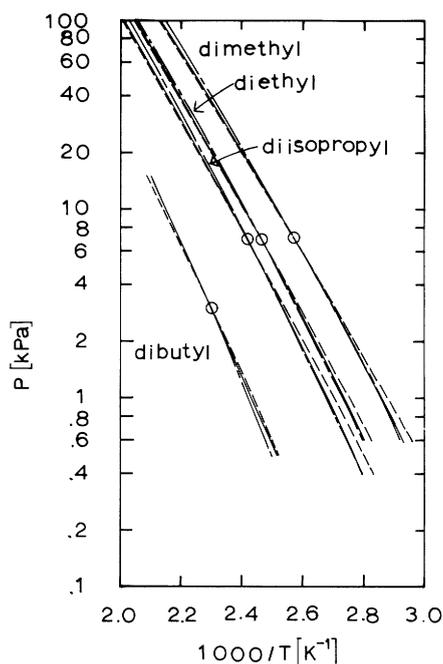


Fig. 3. Estimation of vapor pressures for four succinates under the assumption that for each compound one vapor pressure-temperature point was known beforehand.

For dimethyl, diethyl and diisopropyl succinates the points known beforehand are those of 7.000 kPa, and for dibutyl succinate the point is that of 3.000 kPa. They are represented by the mark \circ in the figure. All the lines (solid, broken and dotted) have the same meanings as in Fig. 2.

values, the Lee-Kesler and Gomez-Thodos equations combined with the values of θ and P_c obtained by the Joback method were applied to the estimation of vapor pressures of the succinates.

Next, this estimation procedure was extended to the case of any single boiling point at which the vapor pressure is known. The values of the vapor pressure estimated showed good agreement with the experimental values in a wide range of lower or higher boiling points than the datum boiling point for which the vapor pressure was known beforehand.

When we need the vapor pressure for the substance

Table 7. Differences between calculated temperatures, from the Lee-Kesler and Gomez-Thodos equations, and experimental ones. Where value at one datum point is known beforehand*¹⁾

	Pressure [kPa]											
	0.600		1.000		3.000		10.000		30.000		Atmospheric pressure	
	LK* ²⁾	GT* ³⁾	LK* ²⁾	GT* ³⁾								
	ΔT [K]	ΔT [K]										
Dimethyl succinate	1.25	3.98	0.46	2.71	-0.11	1.03	0.13	-0.34	0.83	-1.24	2.10	-1.77
Diethyl succinate	-0.46	2.84	-0.67	2.04	-0.37	0.91	0.29	-0.26	1.32	-1.04	3.47	-1.32
Diisopropyl succinate	-0.26	3.27	-0.56	2.33	-0.38	0.97	0.52	-0.06	1.48	-1.00	2.78	-1.66
Dibutyl succinate	-1.60	1.20	-0.93	1.10	0.00	0.00	-0.50	-3.00				

$\Delta T = T_{\text{exptl}} - T_{\text{calcd}}$, T_{exptl} : experimental or smoothed values.

*¹⁾ The points given are the point of 7.000 kPa for dimethyl, diethyl and diisopropyl succinates, and that of 3.000 kPa for dibutyl succinate.

*²⁾ comparison with values calculated from the Lee-Kesler equation.

*³⁾ comparison with values calculated from the Gomez-Thodos equation.

of interest at a reduced pressure, if we know the value of any one point of the pressure as well as the molecular structure of the substance, the estimation procedure proposed here is applicable to such substance as this.

Appendix 1

The Chebyshev polynomial is:

$$t \ln(P) = a_0/2 + \sum a_i E_i(x) \quad (\text{A-1})$$

where P is in kPa, t ($= T - 273.15$) in K, T in K, $E_1(x) = x$, $E_2(x) = 2x^2 - 1$, $E_i(x) = 2xE_{i-1}(x) - E_{i-2}(x)$, and x is a function of temperature defined as

$$x = \frac{2T - (T_{\text{max}} + T_{\text{min}})}{T_{\text{max}} + T_{\text{min}}} \quad (\text{A-2})$$

where T_{max} and T_{min} are respectively the maximum and minimum temperatures of the substance. The polynomial with four parameters is equal to the Cragoe equation, which is

$$\ln(P) = A_c + B_c/t + C_c t + D_c t^2 \quad (\text{A-3})$$

or

$$t \ln(P) = A_c t + B_c + C_c t^2 + D_c t^3 \quad (\text{A-4})$$

Appendix 2

The Gomez-Thodos equation is as follows:

$$\ln(P_r) = \beta \left(\frac{1}{T_r^m} - 1 \right) + \gamma (T_r^7 - 1) \quad (\text{A-5})$$

They derived the equation of β from substitution of $T_r = \theta$ and $P_r = 101.325/P_c$ in Eq. (A-5).

$$\beta = \frac{\gamma(\theta^7 - 1)}{1 - \theta^{-m}} - \frac{\ln(101.325/P_c)}{1 - \theta^{-m}} \quad (\text{A-6})$$

For a polar substance that does not hydrogen-bond, m and γ have been expressed as:

$$m = 0.466 T_c^{0.166} \quad (\text{A-7})$$

$$\gamma = 0.08594 \exp(7.462 \times 10^{-4} T_c) \quad (\text{A-8})$$

Nomenclature

A_c, B_c, C_c, D_c	Cragoe constants defined by Eq. (A-3)	
a_0, a_i	= constants of the Chebyshev polynomial defined by Eq. (A-1)	
$E_1(x)$	= $-x$	[—]
$E_2(x)$	= $-2x^2 - 1$	[—]
$E_i(x)$	= $2xE_{i-1}(x) - E_{i-2}(x)$	[—]
m	= value defined as Eq. (A-7)	[—]
P	= pressure	[kPa]
P_c	= critical pressure	[kPa]
P_r	= reduced pressure ($= P/P_c$)	[—]
T	= temperature	[K]
T_b	= temperature at the normal boiling point	[K]
T_c	= critical temperature	[K]
T_r	= reduced pressure ($= T/T_c$)	[—]
t	= $T - 273.15$	[K]
x	= $(2T - (T_{\text{max}} + T_{\text{min}}))/(T_{\text{max}} + T_{\text{min}})$	[—]
β	= value defined as Eq. (A-6)	[—]
γ	= value defined as Eq. (A-8)	[—]
ω	= acentric factor obtained from Eq. (4)	[—]
θ	= reduced temperature ($= T_b/T_c$)	[—]

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