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Density, Viscosity, and Vapor Pressure for Binary Mixtures of Tricyclo [5.2.1.$0^{2.6}$] Decane and Diethyl Carbonate

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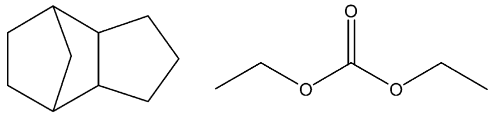
The density, viscosity, and vapor pressure for binary mixtures of tricyclo [5.2.1.$0^{2.6}$] decane (JP-10), a high-energy density hydrocarbon fuel, with diethyl carbonate are presented. Excess volume, V_m^E, and viscosity deviation, $\Delta\eta$, for the binary mixtures have been calculated. Positive values of V_m^E, negative values of $\Delta\eta$, and positive deviations from Raoult's law for the vapor pressures are observed. The deviation results are discussed in terms of the changes of molecular interactions in the mixtures, and they are fitted with the Redlich−Kister equation. The viscosities have also been correlated with several semiempirical equations. These physical property data and calculations provide valuable information on the development and application of new high-energy density hydrocarbon fuels.

Introduction

Modern aviation fuels must meet several critical requirements, such as large volumetric energy content or heat value, high storage stability, low viscosity, toxicity, and freeze point, and acceptable flash point.[1] A pure polycyclic hydrocarbon, tricyclo [5.2.1.$0^{2.6}$] decane, or *exo*-tetrahydrodicyclopentadiene, shown in Figure 1a, is a high-energy density hydrocarbon fuel, which is usually named JP-10. JP-10 takes advantage of the increased energy storage available through strained cyclic geometry and presently serves as the missile fuel in the U.S.[2] Because appropriate properties are necessary for flow and ignition of the hydrocarbon fuels under operating conditions, there is increasing interest in adding oxygenated compounds such as ethers, alcohols, and esters to different hydrocarbon fuels to adjust their physical properties. Dialkyl carbonates are sometimes used as fuel additives because of the high oxygen contents and the octane-enhancing and pollution-reducing capabilities.[3,4] The hydrocarbon fuels with alkyl carbonates as additives would have better injection delivery and spray, higher oxygen contents, and excellent octane values.[5]

In this work, detailed measurements are conducted over the whole composition range on density and viscosity at 298.15 K and vapor pressure at various temperatures for the binary mixtures of tricyclo [5.2.1.$0^{2.6}$] decane (JP-10) and diethyl carbonate (DEC) (Figure 1b). The data could be used in the development of models for mixtures of hydrocarbons with oxygenates. The results could provide reference information on the additive choice and the property optimization of high-energy density hydrocarbon fuels.



1a 1b

Figure 1. Molecular structures of (a) tricyclo [5.2.1.$0^{2.6}$] decane (JP-10) and (b) diethyl carbonate (DEC).

Table 1. Components of JP-10 Sample

component	wt %
tricyclo [5.2.1.$0^{2.6}$] decane	97.08
bicyclo [4.4.0] decane	1.72
tricyclo [3.3.1.$1^{3,7}$] decane	1.19

Table 2. Physical Property of Investigated Samples

	T_b/K		ρ (298.15 K)/g·cm^{-3}		η (298.15 K)/mPa·s	
compound	exptl	lit.	exptl	lit.	exptl	lit.
JP-10	458.09	457.76[a]	0.9314	0.9318[b]	2.739	2.767[b]
DEC	398.51	399.32[c]	0.9690	0.9690[d,e]	0.764	0.750[g]
				0.9692[f]		0.749[h]
				0.96921[g]		0.756[i]
				0.9691[h]		

[a] Ref 8. [b] Ref 9. [c] Ref 10. [d] Ref 11. [e] Ref 12. [f] Ref 13. [g] Ref 14. [h] Ref 15. [i] Ref 16.

Experimental Section

Materials. The sample of JP-10 was obtained from Liming Research Institute of Chemical Industry, China. The predominant compositions of the sample were analyzed by a Hewlett-Packard 6890/5973 GC/MS, which was equipped with a DB-17 column and programmed from (333 to 453) K at a rate of 10 K·min^{-1} with an initial isothermal period of 5 min. On the basis of MS information, the structure of each compound corresponding to the GC peak was determined according to the confidence and similarity from the NIST98.L database. The relative contents of the components were obtained by the peak area normalization method. The results are listed in Table 1. DEC with more than 98.5 wt % purity, purchased from Sinopharm Chemical Reagent Company, was dried over 0.4 nm molecular sieves and degassed by ultrasound before use. We prepared binary mixtures of JP-10 with DEC by directly weighing the constituent components with a precision of 0.0001 g using a Mettler Toledo AL204 balance. The uncertainty of mole fraction was estimated to be ± 1·10^{-4}.

Density and Viscosity Measurement. Densities, ρ, of pure components and their binary mixtures were measured by a vibrating-tube digital densimeter (DMA55 Anton Paar) ther-

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Table 3. Density, ρ, Excess Volume, V_m^E, Viscosity, η, and Viscosity Deviation, $\Delta\eta$, for JP-10 (1) + DEC (2) Binary Mixtures at 298.15 K

x_2	ρ $\mathrm{g \cdot cm^{-3}}$	V_m^E $\mathrm{cm^3 \cdot mol^{-1}}$	η mPa·s	$\Delta\eta$ mPa·s
0.0000	0.9314	0.0000	2.739	−0.000
0.1141	0.9331	0.2989	2.140	−0.374
0.2235	0.9353	0.5080	1.757	−0.541
0.3303	0.9381	0.6263	1.496	−0.591
0.4355	0.9413	0.6939	1.292	−0.587
0.5360	0.9448	0.7112	1.149	−0.531
0.6336	0.9488	0.6617	1.026	−0.462
0.7288	0.9534	0.5384	0.938	−0.362
0.8207	0.9582	0.3942	0.861	−0.257
0.9109	0.9635	0.1996	0.814	−0.126
1.0000	0.9690	0.0000	0.764	0.000

mostatted by a circulating-water bath with a precision of 0.01 K. The densimeter was calibrated with dry air and freshly twice-distilled water. The overall uncertainty of the density dependent on the temperature control of the water bath, the concentration, and the vibrating-tube digital densimeter was estimated to be $\pm 1 \cdot 10^{-4}\ \mathrm{g \cdot cm^{-3}}$.

The dynamic viscosity, η, is calculated from the kinematic viscosity, ν, and the density, ρ

$$\nu = \eta/\rho \tag{1}$$

The kinematic viscosities were determined using a Ubbelohde viscometer. The viscometer was filled with 15 cm^3 solution and was submerged in a thermostatic bath with a resolution of 0.01 K. The flow time was measured with a stopwatch to an accuracy of 0.01 s. The viscometer was calibrated with twice-distilled water. We reported each viscosity value of the fluid by averaging over three consecutive runs. The flow time was reproducible to be ± 0.2 s, and the uncertainty of viscosity was within ± 0.002 mPa·s.

Vapor Pressure Measurement. Inclined ebulliometers with pumplike stirrers, shown in Figure 2, were used to measure the vapor pressure. The composition analysis for the ebulliometer is expressed as the following equation[6]

$$\frac{x_0 - x}{x} = \frac{(R + \alpha)(K - 1)}{1 + R} \tag{2}$$

where x_0 is the feed composition, x is the liquid-phase mole fraction, R is the reflux ratio under steady state, α is the liquid holdup factor, $\alpha = (N_g - RN_1)/N$, N_g and N_1 are the amounts of substance retained in the vapor and liquid phases, respectively, N is the total amount of substance of the system, K is the phase equilibrium constant ($K = y/x$), and y is the vapor-phase mole fraction. When R approaches zero, $\alpha = N_g/N$, and eq 2 is simplified to the form of the quasi-static method

$$\frac{x_0 - x}{x} = (K - 1)\alpha \tag{3}$$

The inclined structure of the ebulliometer can reduce the effect of the height of liquid, and the stirring function is useful for circulation of the solution. As a result, the reflux ratio, R, and liquid holdup, α, can be reduced. The value of α for the ebulliometer system is controlled to about 10^{-4}, and K is usually less than 10, so x and x_0 have very similar values, and the liquid

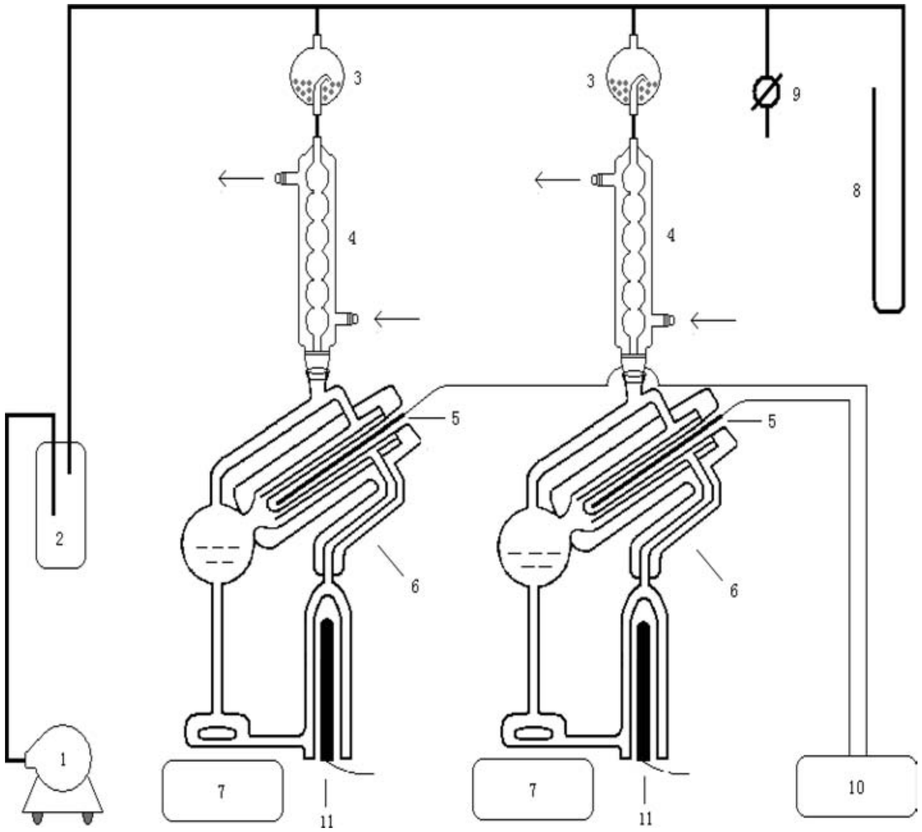

Figure 2. System for the determination of vapor pressure: 1, vacuum pump; 2, trap; 3, drying vessels; 4, condensators; 5, platinum resistance thermometers; 6, inclined ebulliometers; 7, magnetic stirrers; 8, mercury manometer; 9, pressure control valve; 10, digital multimeter; 11, heaters.

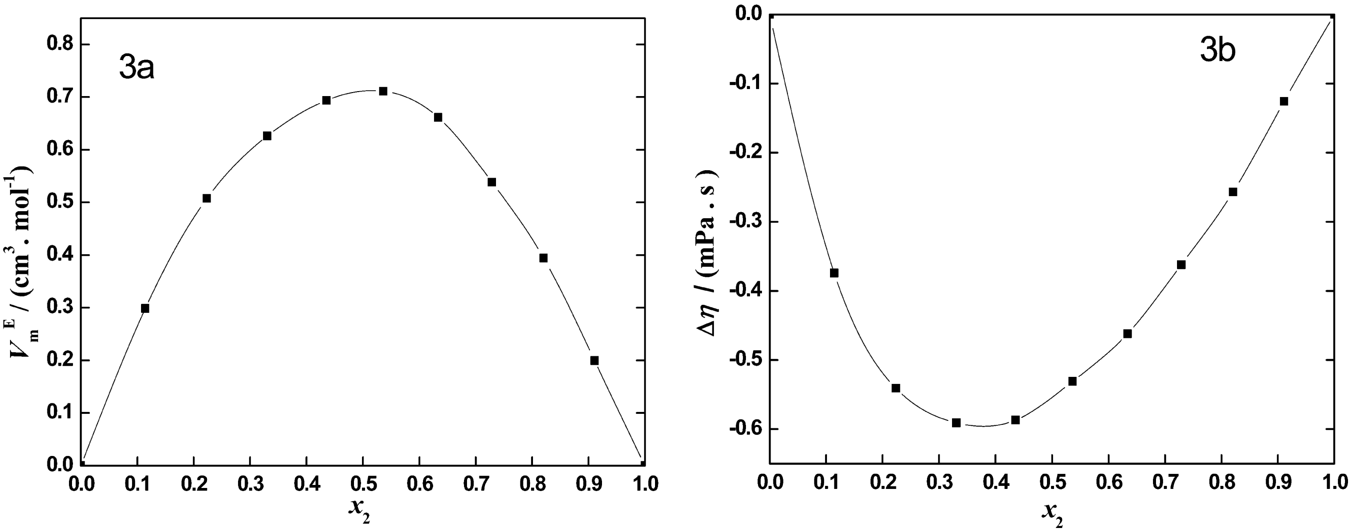

Figure 3. Plots of (a) excess molar volume and (b) viscosity deviation versus mole fraction for JP-10 (1) + DEC (2) binary mixtures at 298.15 K.

Table 4. Correlation Parameters and Standard Deviation, σ, for V_m^E and Δη by the Redlich−Kister Equation

property	A_1	A_2	A_3	A_4	A_5	σ
V_m^E/cm^3·mol^{-1}	2.83912	−0.02012	−0.05115	−0.45384	−0.15632	0.007
Δη/mPa·s	−2.2163	1.0435	−0.8174	0.5207	0.1370	0.004

composition is supposed to be a constant. The ebulliometer is used for the measurements on the bubble-point vapor pressure of mixtures.

The vapor−liquid equilibrium (VLE) temperatures of a sample and a reference material (ethanol) in two separate ebulliometers were measured under the same pressure. The pressure could be calculated from the boiling temperature of ethanol and its well-known pressure−temperature behavior,[7] and it avoided the necessity of measuring the equilibrium pressure directly with a mercury manometer. The temperatures with uncertainty of less than 0.01 K were recorded with two standard platinum resistance thermometers (Yunnan Instrument Factory, China) connected to Keithley 195A digital multimeters. The vapor pressures at various temperatures for each sample were determined over the equilibrium pressure range from about (8 to 102) kPa. The uncertainty of the vapor pressure was ± 0.1 kPa.

Results and Discussion

Density and Viscosity Data. The physical properties of the samples of JP-10 and DEC are compared with reference data[8−16] in Table 2, where T_b is the normal boiling point, ρ is the density, and η is the viscosity.

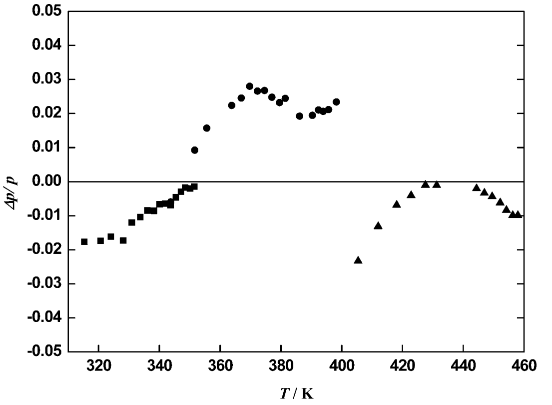

Figure 4. Comparison of measured vapor pressures with literature values ($\Delta p = p - p_{\text{calcd}}$): ■, ethanol (ref 7); ●, DEC (ref 18); ▲, JP-10 (ref 19).

The data of density and viscosity along with the calculated values of excess volume, V_m^E, and viscosity deviation, Δη, for the binary mixtures at 298.15 K are given in Table 3. Excess volumes and viscosity deviations are obtained from eqs 4 and 5

$$V_m^E = \frac{M_1x_1 + M_2x_2}{\rho} - \left(\frac{M_1x_1}{\rho_1} + \frac{M_2x_2}{\rho_2}\right) \tag{4}$$

$$\Delta\eta = \eta - (x_1\eta_1 + x_2\eta_2) \tag{5}$$

where M_i, x_i, ρ_i, and η_i are the molecular weight, the mole fraction, the density, and the viscosity of the pure component, i, respectively. ρ and η are the density and the viscosity of the binary mixture, respectively.

The excess volume or viscosity deviation as a function of composition was fitted to the Redlich−Kister type equation[17]

$$Y = x_1x_2\sum_{i=1}^{k} A_i(x_1 - x_2)^{i-1} \tag{6}$$

where Y implies V_m^E or Δη. A_i is the polynomial coefficient. The correlated results are given in Table 4, in which the tabulated standard deviation, σ, is defined as

$$\sigma = [\sum (Y - Y_{\text{calcd}})^2/(n - k)]^{1/2} \tag{7}$$

where n is the number of datum points, k is the number of estimated parameters, Y is the experimental value, and Y_{calcd} is the calculated value from eq 6.

The excess volume, V_m^E, and the viscosity deviation, Δη, against the mole fraction for the investigated binary mixtures at 298.15 K are shown in Figure 3. It is observed that the excess

Table 5. Bubble-Point Vapor Pressure Data for JP-10 (1) + DEC (2) Binary System

$x_2 = 0.0000$		$x_2 = 0.0457$		$x_2 = 0.0689$		$x_2 = 0.0909$		$x_2 = 0.1141$	
T/K	p/kPa	T/K	p/kPa	T/K	p/kPa	T/K	p/kPa	T/K	p/kPa
393.98	15.0	378.76	14.4	371.40	12.8	365.94	9.9	358.34	8.4
396.02	16.6	384.65	20.9	378.90	19.5	373.86	17.8	368.84	16.2
401.42	20.6	387.71	24.9	384.34	25.2	383.76	29.8	376.94	24.2
413.88	30.3	391.46	29.6	390.61	32.6	390.73	39.5	382.16	30.3
418.34	34.8	399.61	41.1	396.90	41.0	393.96	44.1	386.59	36.1
426.86	44.9	400.38	42.4	400.47	46.1	394.52	44.9	389.40	40.0
431.22	50.7	404.13	47.8	405.34	53.5	399.29	51.8	392.75	44.8
434.56	55.8	409.71	56.4	408.76	58.9	402.95	57.1	395.17	48.6
436.68	59.2	411.23	58.6	412.74	65.3	409.47	67.3	401.89	59.5
439.31	63.2	415.41	65.2	415.92	70.8	410.78	69.0	405.64	65.8
441.13	66.6	420.84	73.5	420.01	77.6	415.30	75.8	407.19	68.8
442.7	69.5	423.96	78.3	423.57	84.1	418.93	81.4	410.99	75.4
442.81	69.8	427.54	83.8	432.47	100.5	423.77	88.5	413.96	81.0
444.37	72.9	430.18	87.9			428.27	95.0	414.64	82.4
446.13	75.9	438.75	100.6			431.99	100.3	423.25	99.2
445.73	75.1							423.62	100.0
450.08	83.9								
457.66	101.1								

$x_2 = 0.3328$		$x_2 = 0.5348$		$x_2 = 0.7291$		$x_2 = 0.9564$		$x_2 = 1.0000$	
T/K	p/kPa	T/K	p/kPa	T/K	p/kPa	T/K	p/kPa	T/K	p/kPa
350.65	10.7	348.88	12.6	345.94	12.8	340.58	11.4	343.77	13.8
360.88	18.6	356.93	17.9	355.51	19.3	349.18	16.8	351.67	19.3
368.81	26.7	365.68	25.7	364.08	27.0	349.36	17.0	355.63	22.7
374.42	33.3	372.71	33.6	369.84	33.5	357.63	24.0	358.29	26.0
377.91	37.9	377.66	40.1	374.00	38.9	364.56	31.5	363.84	31.3
384.37	46.9	382.91	48.0	378.62	45.7	369.56	37.9	366.98	35.2
390.08	55.4	387.33	55.3	382.10	51.4	374.22	44.7	369.75	39.1
397.38	66.9	390.58	61.3	385.33	57.1	378.43	51.7	372.28	42.8
401.12	73.4	394.19	68.4	388.67	63.6	380.84	56.3	374.61	46.5
402.31	75.3	396.90	74.0	391.94	70.5	383.20	60.9	377.05	50.5
406.92	83.6	396.87	74.8	394.78	77.0	385.70	66.0	379.59	55.0
409.39	88.0	402.35	86.4	398.14	85.3	391.24	78.7	381.49	58.8
411.79	92.2	405.65	94.5	401.01	92.7	393.15	83.6	383.37	63.1
416.32	100.8	407.86	100.3	403.98	101.0	395.91	90.9	383.57	63.5
						398.30	98.0	386.18	68.5
						399.63	101.0	387.61	72.8
								390.35	78.5
								392.39	84.0
								393.93	88.2
								395.72	93.4
								398.28	101.5

Table 6. Correlation Results of Vapor Pressure by the Antoine Equation for JP-10 (1) + DEC (2) Binary Mixtures[a]

		Antoine equation coefficients				
x_2	n	A	B	C	AAD/kPa	ARD/%
0.0000	18	11.7067	2158.84	153.53	0.21	0.57
0.0457	15	6.2226	176.60	329.10	0.05	0.15
0.0689	13	7.1481	346.45	296.02	0.03	0.05
0.0909	15	6.2026	177.79	320.41	0.06	0.11
0.1141	16	7.4370	396.28	283.68	0.05	0.09
0.3328	14	7.2947	384.78	272.82	0.08	0.26
0.5348	14	10.9335	1508.40	169.39	0.11	0.24
0.7291	14	13.1847	2564.12	104.76	0.02	0.03
0.9564	16	12.9115	2349.02	116.36	0.09	0.16
1.0000	21	13.7415	2780.96	93.69	0.26	0.51

[a] AAD = $(1/n)\sum_{i=1}^{n} |p - p_{\text{calcd}}|_i$; ARD = $(1/n)\sum_{i=1}^{n} |((p - p_{\text{calcd}})/p)\cdot 100\ \%|_i$.

molar volumes are positive and the viscosity deviations are negative over the entire composition range. The DEC molecule with high oxygen content contains strong polar groups of C=O and C−O, and there exist strong dipole−dipole interactions among the DEC molecules. When the rigid and nonpolar JP-10 molecules are mixed with the DEC molecules, the electrostatic interactions are weakened, and the distance between two polar molecules in the binary mixtures becomes larger. The free volume increases, and it leads to the positive values of V_m^E and negative values of $\Delta\eta$.

Table 7. Parameters in the Wilson Model for Vapor Pressure Correlation of JP-10 (1) + DEC (2) Binary System

T/K	Λ_{12}	Λ_{21}
378.15	0.477	0.439
383.15	0.346	0.543
388.15	0.263	0.645
393.15	0.211	0.735
398.15	0.171	0.833

Vapor Pressure Data and Phase Diagrams. The deviations of vapor pressures from the literature data of ethanol,[7] DEC,[18] and JP-10[19] are shown in Figure 4. Vapor pressures at various temperatures for ten binary mixtures of JP-10 and DEC are listed in Table 5.

A nonlinear regression method is used to fit the vapor pressure data to the Antoine equation

$$\ln p/\text{kPa} = A - \frac{B}{T/\text{K} - C} \tag{8}$$

where p is the vapor pressure, T is the equilibrium temperature, and A, B, and C are constants. Table 6 gives the Antoine constants, together with the average absolute deviation (AAD) and average relative deviation (ARD).[20] The fractional deviations of the vapor pressures are shown in Figure 5.

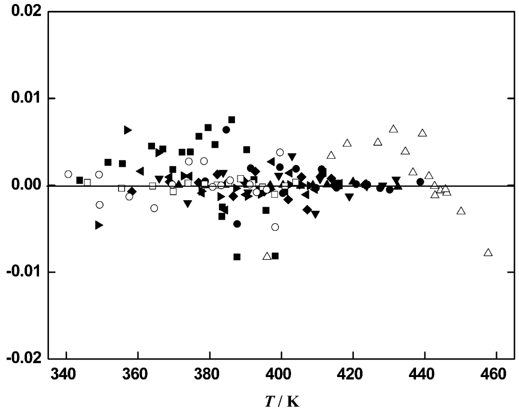

Figure 5. Fractional deviations of vapor pressure correlation with the Antoine equation for JP-10 (1) + DEC (2) binary mixtures ($\Delta p = p - p_{\text{calcd}}$): ■, $x_2 = 0.0000$; ●, $x_2 = 0.0457$; ▲, $x_2 = 0.0689$; ▼, $x_2 = 0.0909$; ◆, $x_2 = 0.1141$; left solid triangle, $x_2 = 0.3328$; right solid triangle, $x_2 = 0.5348$; □, $x_2 = 0.7291$; ○, $x_2 = 0.9564$; △, $x_2 = 1.0000$.

From the correlation results, the $p-x$ phase diagrams and pressure deviations from the linear addition values (Raoult's law) for the binary system are shown in Figure 6. It is indicated that the mixtures show positive deviations from Raoult's law. The maximum pressure departure from the linear addition value over the experimental range is about 26 kPa at 378.15 K.

The VLE data at constant temperature are correlated using the Wilson model[21] of the liquid-phase activity coefficients, γ_i

$$\ln \gamma_i = -\ln(x_i - \Lambda_{ij}) + x_j[\Lambda_{ij}/(x_i + \Lambda_{ij}x_j) - \Lambda_{ji}/(x_j + \Lambda_{ji}x_i)] \quad (i \neq j) \quad (9)$$

where Λ_{ij} and Λ_{ji} are adjustable parameters.

At vapor−liquid equilibrium,

$$y_i p = x_i \gamma_i p_i^* \quad (10)$$

where x_i and y_i are the liquid and vapor phase mole fractions, respectively, p is the total pressure, and p_i^* is the vapor pressure of pure component, i. The objective function (OF)[22] used is

$$\text{OF} = \sum \left(1 - \frac{p_{\text{calcd}}}{p}\right)^2 \quad (11)$$

$$I = \int_0^1 [\ln(\gamma_1/\gamma_2)]\, dx_1 \quad (12)$$

We obtained the optimum binary parameters by minimizing the OF, and the thermodynamic consistency of the VLE data has been checked by means of the area test using eq 12 with $|I| \leq 0.01$. Correlated parameters of the Wilson model are listed in Table 7. Representations of the correlated VLE data are shown in Figure 6 with the solid lines, which are in good agreement with the experimental values.

Viscosity Data Correlation. Knowledge of the viscosity for pure liquids and mixtures and application of the viscosity models are useful for practical and theoretical purposes. Numerous equations for viscosity correlation have been introduced.[23−26] In this work, four typical semiempirical equations are employed to correlate the experimental viscosity data of the binary system. The Kendall−Monroe equation,[23] which has no adjustable parameter, is expressed as

$$\eta = (x_1\eta_1^{1/3} + x_2\eta_2^{1/3})^3 \quad (13)$$

The single-parameter correlations of Hind, Grunberg−Nissan, and Frenkel are, respectively[26]

$$\eta = x_1^2\eta_1 + x_2^2\eta_2 + 2x_1x_2A_{12} \quad (14)$$

$$\ln \eta = x_1 \ln \eta_1 + x_2 \ln \eta_2 + x_1x_2A_{12} \quad (15)$$

$$\ln \eta = x_1^2 \ln \eta_1 + x_2^2 \ln \eta_2 + 2x_1x_2 \ln A_{12} \quad (16)$$

where A_{12} is an interaction parameter. The fractional deviations, correlation parameters, and the standard deviation are shown in Figure 7. The best one of the four correlation methods for the binary mixtures is the Frenkel equation.

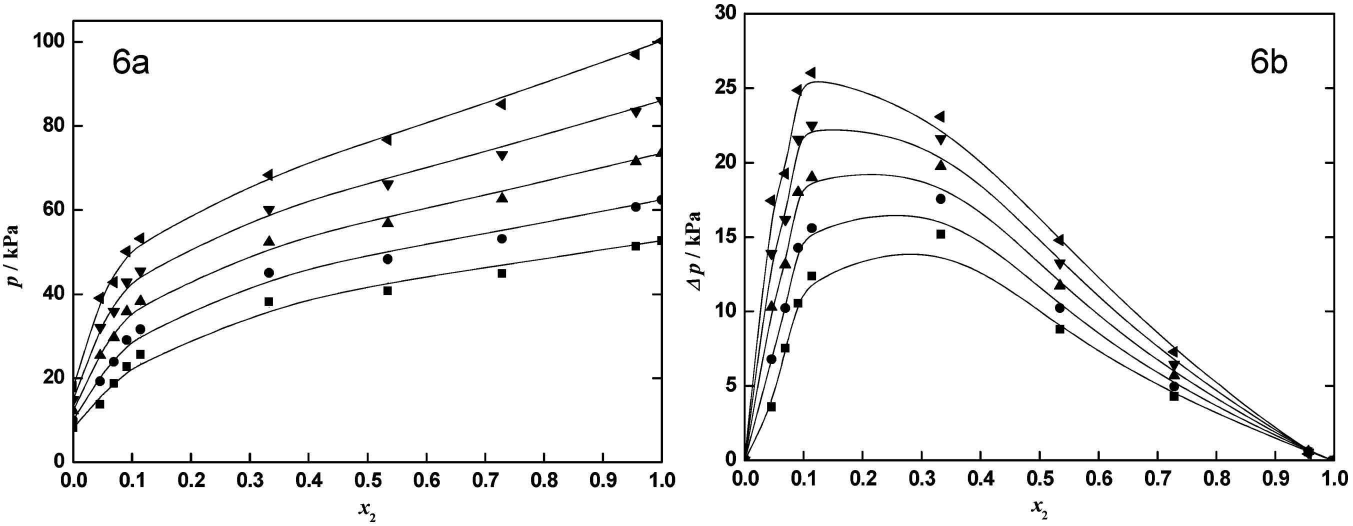

Figure 6. (a) Pressure−composition ($p-x$) diagrams and (b) pressure departure from Raoult's law for JP-10 (1) + DEC (2) binary mixtures ($\Delta p = p - p_{\text{calcd}}$). ■, 378.15 K; ●, 383.15 K; ▲, 388.15 K; ▼, 393.15 K; left solid triangle, 398.15 K; −, correlated results with Wilson model.

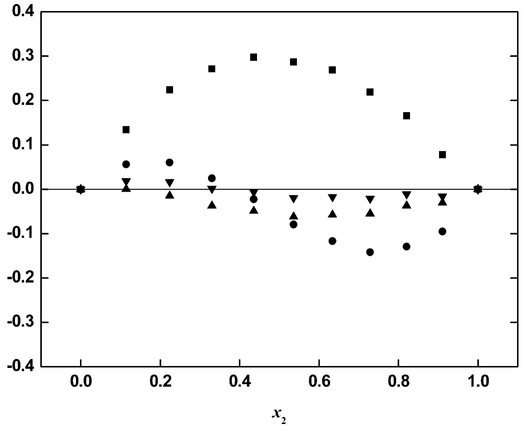

Figure 7. Fractional deviations of viscosity correlation with several empirical equations for JP-10 (1) + DEC (2) binary mixtures at 298.15 K ($\Delta\eta = \eta - \eta_{\text{calcd}}$): ■, Kendall−Monroe ($\sigma = 0.27$); ●, Hind ($A_{12} = 0.5$; $\sigma = 0.09$); ▲, Grunberg−Nissan ($A_{12} = -1.0$; $\sigma = 0.05$); ▼, Frenkel equation ($A_{12} = 0.96$; $\sigma = 0.02$).

Conclusions

Densities, viscosities, and vapor pressures at different temperatures for binary mixtures of tricyclo [5.2.1.0$^{2.6}$] decane (JP-10) with diethyl carbonate (DEC) have been measured. Excess volume, V_m^E, and viscosity deviation, $\Delta\eta$, for the binary mixtures have been calculated. Positive values of V_m^E, negative values of $\Delta\eta$, and positive deviations from Raoult's law of the vapor pressures are observed, and these deviation results can be explained in terms of the changes of molecular interactions. Several calculated deviation functions fitted with the Redlich−Kister equation and the viscosities correlated with semiempirical equations are performed with valuable information. The thermodynamic data and calculations are helpful for adjusting or controlling the volatility, fluidity, and combustibility of high-energy density hydrocarbon fuels.

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