Densities, Viscosities, Refractive Indices, and Surface Tensions for 12 Flavor Esters from $T = 288.15$ K to $T = 358.15$ K

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Densities, viscosities, refractive indices, and surface tensions for 12 flavor esters were measured from 288.15 K to 358.15 K at atmospheric pressure. The esters studied were ethyl acetoacetate, isoamyl acetate, ethyl isovalerate, methyl benzoate, ethyl caproate, ethyl benzoate, benzyl acetate, isoamyl butyrate, ethyl salicylate, benzyl propionate, ethyl phenylacetate, and ethyl caprylate. Densities were determined using a vibrating-tube density meter, and viscosities were measured with an automatic Ubbelohde capillary viscometer. Refractive indices were measured using a digital Abbe-type refractometer, and surface tensions were measured using the Wilhelmy-plate method. The experimental data were correlated by temperature-dependent equations.

Introduction

The thermophysical study of esters is of increasing interest due to their wide usage in flavoring, perfumery, artificial essences, and cosmetics. Esters are also important solvents in pharmaceutical, paint, and plastic industries. Several studies for binary mixtures of the thermophysical properties of ester compounds have been conducted in the recent years.[1−6] However, detailed investigation of the properties such as density, viscosity, refractive index, and surface tension over a wide range of temperature for pure flavor esters are still scare in the literature.

Therefore, in the present paper we undertake to obtain reliable density, viscosity, refractive index, and surface tension data for 12 important flavor esters in the temperature range of 288.15 K to 358.15 K at atmospheric pressure. The flavor esters chosen in this study were ethyl acetoacetate, isoamyl acetate, ethyl isovalerate, methyl benzoate, ethyl caproate, ethyl benzoate, benzyl acetate, isoamyl butyrate, ethyl salicylate, benzyl propionate, ethyl phenylacetate, and ethyl caprylate. Among these substances, the density and viscosity data have been reported previously for isoamyl acetate, ethyl isovalerate, and isoamyl butyrate at temperatures from (293.15 to 343.15) K.[7] To our knowledge, no other data on these properties over a wide range of temperature are available in the open literature.

Experimental Section

Materials. The chemicals used were of analytical grade and were used without further purification. The purity of these chemicals was analyzed by gas chromatography (Perkin-Elmer Autosystem) using a flame ionization detector with a 60 m × 0.53 mm capillary column packed by Stabilwax. High-purity helium was used as the carrier gas. The mass percent purities as determined by the major peak areas on gas chromatography together with the sources and CAS Registry Nos. (CASRN) of chemicals are given in Table 1.

Apparatus and Procedure. Samples were prepared by mass in a 50 cm^3 Erlenmeyer flask provided with a ground glass joint stopper, using a Precisa 262SMA balance with an uncertainty of $\pm 3 \times 10^{-5}$ g. Densities were measured with an Anton Paar DMA-5000 vibrating-tube density meter (Anton-Paar, Graz, Austria) with an accuracy of 5×10^{-6} g·cm^{-3} in the range (0 to 3) g·cm^{-3}, which was thermostatically controlled to within ± 0.01 K in the range (273.15 to 363.15) K. Calibration was performed periodically under atmospheric pressure, in accordance with specifications, using double-distilled water and dry air. The uncertainty of the density measurements was estimated to be less than $\pm 3 \times 10^{-5}$ g·cm^{-3}.

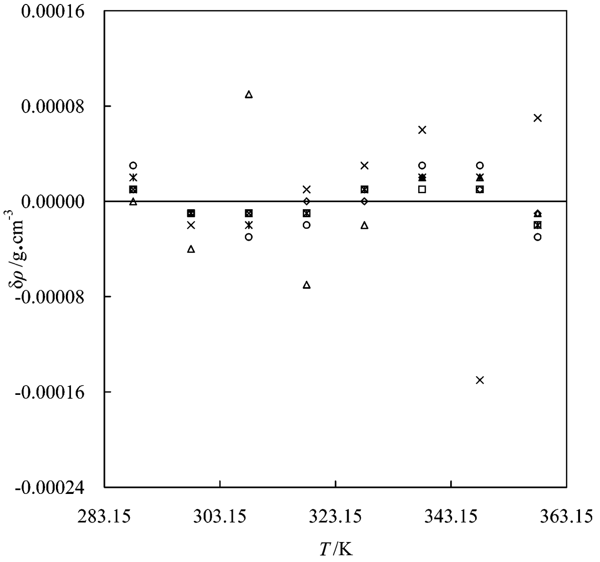

Figure 1. Variation of densities with temperature for six flavor esters: ◇, ethyl phenylacetate; □, benzyl acetate; △, methyl benzoate; ×, ethyl caproate; ∗, ethyl caprylate; ○, ethyl isovalerate. Solid curves were calculated from eq 2.

The kinematic viscosities were determined with the commercial Ubbelohde capillary viscometers (Cannon Instrument Co., State College, PA) of (0.36, 0.47, 0.53, and 0.83) mm in diameter. The viscometer was kept in a Lauda D20 KP thermostat controlled to ± 0.01 K with a proportional-integral-differential regulator. A computer-controlled measuring system (Lauda, Lauda-Königshofen, Germany)

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Table 1. Sources and Mass Fraction (*w*) Purities of the Esters Used in This Study

compounds	molecular formula	sources	CASRN	100*w*
ethyl acetoacetate	$C_6H_{10}O_3$	Acros (U.S.A.)	141-97-9	99.2
isoamyl acetate	$C_7H_{14}O_2$	Tedia (U.S.A.)	123-92-2	99.3
ethyl isovalerate	$C_7H_{14}O_2$	Acros (U.S.A.)	108-64-5	99.0
methyl benzoate	$C_8H_8O_2$	Lancaster (England)	93-58-3	99.2
ethyl caproate	$C_8H_{16}O_2$	Acros (U.S.A.)	123-66-0	99.7
ethyl benzoate	$C_9H_{10}O_2$	Acros (U.S.A.)	93-89-0	99.8
benzyl acetate	$C_9H_{10}O_2$	Acros (U.S.A.)	140-11-4	99.6
isoamyl butyrate	$C_9H_{18}O_2$	Acros (U.S.A.)	106-27-4	99.4
ethyl salicylate	$C_9H_{10}O_3$	Acros (U.S.A.)	118-61-6	99.6
benzyl propionate	$C_{10}H_{12}O_2$	TCI (Japan)	122-63-4	99.4
ethyl phenylacetate	$C_{10}H_{12}O_2$	Acros (U.S.A.)	101-97-3	99.4
ethyl caprylate	$C_{10}H_{20}O_2$	Acros (U.S.A.)	106-32-1	99.5

with an uncertainty of ± 0.01 s was used for flow time measurement. The range of the flow time for the liquids investigated varied from 200 s to 880 s. The kinematic viscosities (ν) were determined according to

$$\nu = k(t - \theta) \tag{1}$$

where k is the viscometer constant, t is the flow time, and θ is the Hagenbach correction. The absolute viscosity (η) was then calculated from the density by the relation $\eta = \nu\rho$. The values of k, which were determined by calibrating with pure water at working temperatures, are 0.000906 ± 0.000001, 0.002913 ± 0.000002, 0.004655 ± 0.000002, and 0.009070 ± 0.000003 for the capillary viscometers with 0.36 mm, 0.47 mm, 0.53 mm, and 0.83 mm diameter, respectively. The value θ, which is dependent on the flow time and the size of capillary, was taken from the tables supplied by the manufacturer. Triplicate measurements of flow times were reproducible within ± 0.01 %. The uncertainty of the viscosity measurement was estimated to be less than ± 0.6 %.

Refractive indices (n_D) were measured with a digital Abbe refractometer RX-5000 (Atago, Tokyo, Japan), which works at the wavelength (589 nm) corresponding to the D-line of sodium. The temperature was controlled to ± 0.05 K with circulating thermostat water to a jacketed sample vessel. Calibration was performed periodically under atmospheric pressure using double-distilled water. The uncertainty of the refractive index measurement was estimated to be less than ± 0.00002 units.

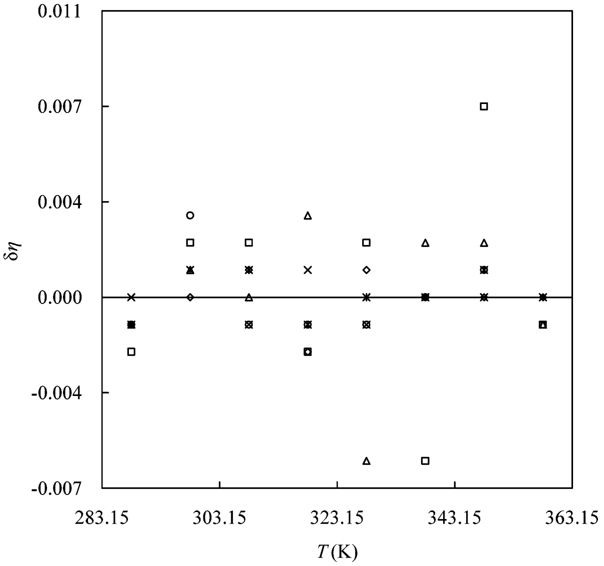

Figure 2. Variation of viscosities with temperature for six flavor esters: ◇, ethyl phenylacetate; □, benzyl acetate; △, methyl benzoate; ×, ethyl caproate; $*$, ethyl caprylate; ○, ethyl isovalerate. Solid curves were calculated from eq 3.

Surface tensions (σ) were measured with an automatic surface tension meter model CBVP-A3 (Kyowa, Japan), which works with the Wilhelmy-plate method. The platinum plate was thoroughly cleaned and flame-dried before each measurement. Calibration was performed periodically under atmospheric pressure, in accordance with specifications, using two 200 mg calibration masses. All liquids were thermostatically controlled to within ± 0.05 K with a circulating thermostat water to a jacketed sample vessel. The uncertainty of surface tension measurement was estimated to be ± 0.2 mN·m^{-1}.

All measurements described above were performed at least three times under atmospheric pressure (100.8 ± 0.2) kPa, and an average of at least three measurements was calculated for each temperature. The uncertainty in the liquid composition was estimated to be ± 1 × 10^{-4}.

Results and Discussion

The experimental densities, viscosities, refractive indices, and surface tensions of 12 flavor esters from T = (288.15 to 358.15) K together with the literature values are given in Table 2. From this table, it can be seen that the experimental values are generally in agreement with those from the literature. The deviations between our experimental data and the literature values may have resulted from the differences in the experimental apparatus, procedure, and purities of compounds used.

The experimental densities, refractive indices, and surface tensions of pure esters were correlated using a

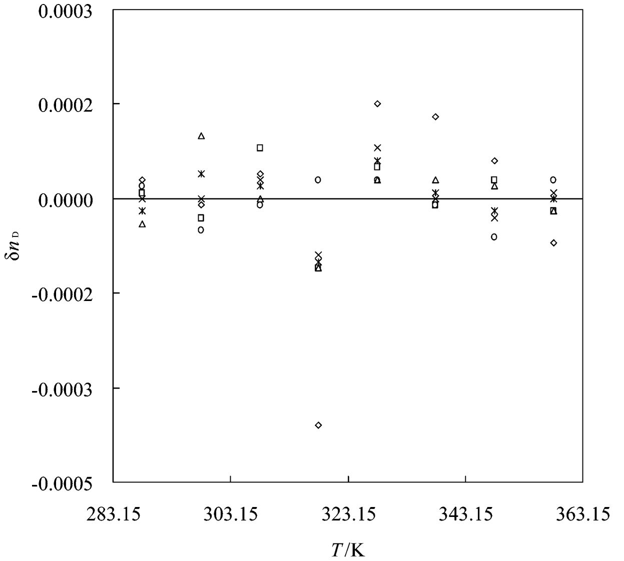

Figure 3. Variation of refractive indices with temperature for six flavor esters: ◇, ethyl phenylacetate; □, benzyl acetate; △, methyl benzoate; ×, ethyl caproate; $*$, ethyl caprylate; ○, ethyl isovalerate. Solid curves were calculated from eq 2.

Table 2. Experimental Densities (ρ), Viscosities (η), Refractive Indices (n_D), and Surface Tensions (σ) for 12 Flavor Esters at Temperatures from (288.15 to 358.15) K

	ρ		η				σ			ρ		η				σ	
	$g \cdot cm^{-3}$		$mPa \cdot s$		n_D		$mN \cdot m^{-1}$			$g \cdot cm^{-3}$		$mPa \cdot s$		n_D		$mN \cdot m^{-1}$	
T/K	exptl	lit	exptl	lit	exptl	lit	exptl	lit	T/K	exptl	lit	exptl	lit	exptl	lit	exptl	lit
Ethyl Acetoacetate																	
288.15	1.03392		1.902		1.42091		33.3		328.15	0.99226		0.981		1.40386		29.1	
298.15	1.02345	1.0208[2]	1.581	1.5081[9]	1.41658	1.4189[2]	32.3	31.3[8]	338.15	0.98158		0.847		1.39949		28.2	
		1.02126[9]							348.15	0.97105		0.736		1.39510		27.0	
308.15	1.01300	1.0102[2]	1.344	1.239[2]	1.41246	1.4120[2]	31.1		358.15	0.96046		0.644		1.39061		26.1	
318.15	1.00254		1.144		1.40803		30.0										
Isoamyl Acetate																	
288.15	0.87772		0.940		1.40288		25.4		318.15	0.84817	0.86732[7]	0.627	0.638[7]	1.38895		22.4	
298.15	0.86791	0.86621[3]	0.804	0.781[3]	1.39836		24.3		328.15	0.83822	0.86014[7]	0.559	0.567[7]	1.38447		21.6	
		0.87601[7]		0.827[7]					338.15	0.82819	0.85405[7]	0.484	0.507[7]	1.37971		20.6	
		0.8664[9]		0.7895[9]					348.15	0.81807		0.434		1.37487		19.6	
308.15	0.85807	0.85585[3]	0.704	0.747[3]	1.39365		23.4		358.15	0.80785		0.390		1.36995		18.7	
		0.87310[7]		0.724[7]													
Ethyl Isovalerate																	
288.15	0.86966		0.853		1.39880		24.4		328.15	0.82971	0.84235[7]	0.504	0.516[7]	1.37978		20.7	
298.15	0.85978	0.86401[7]	0.739	0.752[7]	1.39401		23.5		338.15	0.81952	0.83631[7]	0.452	0.457[7]	1.37492		19.6	
308.15	0.84983	0.85598[7]	0.642	0.659[7]	1.38931		22.5		348.15	0.80920		0.408		1.37002		18.7	
318.15	0.83981	0.84739[7]	0.566	0.581[7]	1.38458		21.6		358.15	0.79873		0.367		1.36524		17.6	
Methyl Benzoate																	
288.15	1.09323	1.09262[4]	2.297	2.304[4]	1.51915		38.6				1.0739[10]		1.491[11]				
		1.09334[8]		2.298[8]							1.0739[11]						
298.15	1.08392	1.0850[1]	1.851	1.918[1]	1.51467	1.51466[4]	37.2		318.15	1.06462	1.06428[4]	1.297	1.270[4]	1.50514	1.5048[11]	34.8	
		1.0836[4]		1.823[4]		1.51457[9]					1.0641[11]		1.253[11]				
		1.083[5]		1.8578[8]		1.5152[11]			328.15	1.05482		1.108		1.50058		33.7	
		1.0839[10]		1.810[11]					338.15	1.04499		0.970		1.49584		32.5	
		1.0837[11]							348.15	1.03523		0.852		1.49107		31.3	
308.15	1.07434	1.0756[1]	1.531	1.663[1]	1.50993	1.5101[11]	35.8		358.15	1.02560		0.750		1.48624		30.4	
		1.07399[4]		1.504[4]													
Ethyl Caproate																	
288.15	0.87564		1.102		1.40950		26.4		328.15	0.83805		0.630		1.39164		22.7	
298.15	0.86629		0.940		1.40504		25.4		338.15	0.82855		0.562		1.38705		21.8	
308.15	0.85691		0.814		1.40055		24.5		348.15	0.81899		0.504		1.38239		21.0	
318.15	0.84750		0.711		1.39597		23.6		358.15	0.80934		0.452		1.37768		20.0	
Ethyl Benzoate																	
288.15	1.05078		2.444		1.50760		35.7		328.15	1.01340		1.169		1.48946		31.5	
298.15	1.04142	1.041[5]	1.971	1.945[8]	1.50328	1.5046[11]	34.6	34.8[8]	338.15	1.00406		1.016		1.48470		30.2	
		1.0421[11]		1.936[11]					348.15	0.99470		0.890		1.48004		29.3	
308.15	1.03207	1.0325[11]	1.623	1.591[11]	1.49863	1.5001[11]	33.3		358.15	0.98531		0.784		1.47526		28.4	
318.15	1.02273	1.0229[11]	1.368	1.332[11]	1.49391	1.4955[11]	32.5										
Benzyl Acetate																	
288.15	1.06011		2.530		1.50439		37.7		328.15	1.02267		1.229		1.48623		33.2	
298.15	1.05075	1.0515[9]	2.056		1.49982		36.4		338.15	1.01329		1.059		1.48157		32.2	
		1.050[12]							348.15	1.00389		0.941		1.47701		31.1	
308.15	1.04139		1.703		1.49539		35.3		358.15	0.99445		0.826		1.47233		30.1	
318.15	1.03203		1.432		1.49064		34.1										
Isoamyl Butyrate																	
288.15	0.86772		1.317		1.41259		25.7		328.15	0.83147	0.84231[7]	0.724	0.646[7]	1.39506		22.4	
298.15	0.85869	0.86204[7]	1.111	0.967[7]	1.40815		24.7		338.15	0.82235	0.83545[7]	0.641	0.575[7]	1.39055		21.6	
308.15	0.84962	0.85708[7]	0.952	0.836[7]	1.40374		24.0		348.15	0.81319		0.573		1.38600		20.5	
318.15	0.84055	0.85011[7]	0.827	0.731[7]	1.39927		23.1		358.15	0.80396		0.511		1.38139		19.7	
Ethyl Salicylate																	
288.15	1.13472		3.694		1.52481		37.5		328.15	1.09594		1.530		1.50646		33.4	
298.15	1.12500		2.831		1.52022		36.3		338.15	1.08624		1.305		1.50191		32.0	
308.15	1.11529		2.253		1.51573		35.2		348.15	1.07660		1.126		1.49744		31.0	
318.15	1.10560		1.842		1.51116		34.4		358.15	1.06683		0.984		1.49296		30.2	
Benzyl Propionate																	
288.15	1.03686		2.550		1.49950		36.0		328.15	0.99993		1.294		1.48171		31.6	
298.15	1.02760		2.123		1.49498		34.8		338.15	0.99072		1.114		1.47711		30.5	
308.15	1.01838		1.787		1.49074		33.6		348.15	0.98149		0.964		1.47252		29.5	
318.15	1.00915		1.515		1.48611		32.4		358.15	0.97225		0.840		1.46768		28.5	
Ethyl Phenylacetate																	
288.15	1.03635		3.002		1.49971		35.9		328.15	0.99893		1.383		1.48149		31.6	
298.15	1.02696		2.384		1.49513		34.6		338.15	0.98961		1.191		1.47688		30.6	
308.15	1.01760		1.948		1.49063		33.5		348.15	0.98028		1.037		1.47224		29.4	
318.15	1.00826		1.624		1.48583		32.8		358.15	0.97095		0.908		1.46747		28.6	
Ethyl Caprylate																	
288.15	0.87079		1.697		1.41991		27.2		328.15	0.83621		0.892		1.40294		23.8	
298.15	0.86215		1.411		1.41560		26.4		338.15	0.82753		0.784		1.39857		22.8	
308.15	0.85352		1.193		1.41137		25.5		348.15	0.81858		0.694		1.39418		22.0	
318.15	0.84487		1.027		1.40702		24.7		358.15	0.81005		0.617		1.38974		21.4	

Table 3. Correlation Results from Eq 2 for Density Data

compounds	a	$b \times 10^4$	$c \times 10^9$	$\alpha \times 10^5$ / $\mathrm{g{\cdot}cm^{-3}}$
ethyl acetoacetate	1.3202	−9.4927	−153.57	7.9
isoamyl acetate	1.1297	−7.7589	−342.86	2.4
ethyl isovalerate	1.1126	−7.0697	−472.62	3.2
methyl benzoate	1.3469	8.0716	−251.79	13
ethyl caproate	1.1239	−7.9309	−237.50	2.0
ethyl benzoate	1.3184	−9.2367	−17.262	1.8
benzyl acetate	1.3242	−8.9989	−58.333	1.5
isoamyl butyrate	1.1155	−8.1919	−141.07	2.1
ethyl salicylate	1.4149	−9.7525	9.5237	3.1
benzyl propionate	1.3037	−9.2880	9.5238	1.3
ethyl phenylacetate	1.3101	−9.6317	45.238	1.3
ethyl caprylate	1.1092	−7.9401	−115.48	8.3

Table 4. Correlation Results from Eq 3 for Viscosity Data

compounds	A	$B \times 10^{-3}$	$C \times 10^2$	D	$\alpha \times 10^4$ / mPa·s
ethyl acetoacetate	−77.33	3.285	−2.393	12.97	41
isoamyl acetate	−302.2	9.150	−8.579	52.11	66
ethyl isovalerate	−221.6	7.330	−5.769	37.55	18
methyl benzoate	−563.3	17.90	−14.01	95.77	34
ethyl caproate	−322.6	10.28	−8.378	54.95	11
ethyl benzoate	−412.7	13.71	−10.00	69.71	21
benzyl acetate	−151.3	6.368	−3.047	24.53	50
isoamyl butyrate	−257.8	8.633	−6.530	43.60	11
ethyl salicylate	−431.9	15.11	−9.660	72.15	41
benzyl propionate	−69.92	3.125	−2.198	11.71	18
ethyl phenylacetate	−543.8	17.51	−13.42	92.31	13
ethyl caprylate	−309.6	10.28	−7.767	52.41	8.2

Table 5. Correlation Results from Eq 2 for Refractive Indices

compounds	a	$b \times 10^4$	$c \times 10^8$	$\alpha \times 10^5$
ethyl acetoacetate	1.5269	−3.1700	−17.738	7.1
isoamyl acetate	1.5127	−3.1009	−24.643	8.9
ethyl isovalerate	1.5236	−3.9571	−12.976	4.4
methyl benzoate	1.6398	−3.7661	−14.524	7.2
ethyl caproate	1.5207	−3.3179	−18.809	7.9
ethyl benzoate	1.6195	−3.2824	−20.833	8.7
benzyl acetate	1.6279	−4.0523	−8.0952	7.0
isoamyl butyrate	1.5250	−3.4740	−14.940	9.3
ethyl salicylate	1.6626	−4.9559	6.1310	6.7
benzyl propionate	1.5999	−2.6465	−29.107	8.9
ethyl phenylacetate	1.6228	−4.0149	−8.9286	10
ethyl caprylate	1.5295	−3.4139	−13.631	8.3

Table 6. Correlation Results from Eq 2 for Surface Tension Data

compounds	a	$b \times 10^2$	$c \times 10^5$	$\alpha \times 10^2$ / $\mathrm{mN{\cdot}m^{-1}}$
ethyl acetoacetate	73.4	−16.8	10.1	9.4
isoamyl acetate	56.3	−11.8	3.57	6.7
ethyl isovalerate	42.4	−3.51	−9.52	5.1
methyl benzoate	97.3	−27.4	24.4	11
ethyl caproate	57.3	−12.1	4.76	4.7
ethyl benzoate	78.7	−18.5	12.5	13
benzyl acetate	87.5	−22.6	18.4	8.3
isoamyl butyrate	41.9	−3.44	−7.74	11
ethyl salicylate	71.4	−12.8	3.57	16
benzyl propionate	88.0	−24.1	20.8	9.1
ethyl phenylacetate	73.0	−15.0	7.14	15
ethyl caprylate	59.2	−13.1	7.14	10

temperature-dependent equation with the following form:

$$Y = a + b(T/\mathrm{K}) + c(T/\mathrm{K})^2 \tag{2}$$

where Y refers to $\rho/\mathrm{g{\cdot}cm^{-3}}$, n_D, or $\sigma/\mathrm{mN{\cdot}m^{-1}}$; a, b, and c are fitted parameters. The viscosity data of pure esters were regressed using

$$\ln(\eta/\mathrm{mPa{\cdot}s}) = A + \frac{B}{(T/\mathrm{K})} + C(T/\mathrm{K}) + D\ln(T/\mathrm{K}) \tag{3}$$

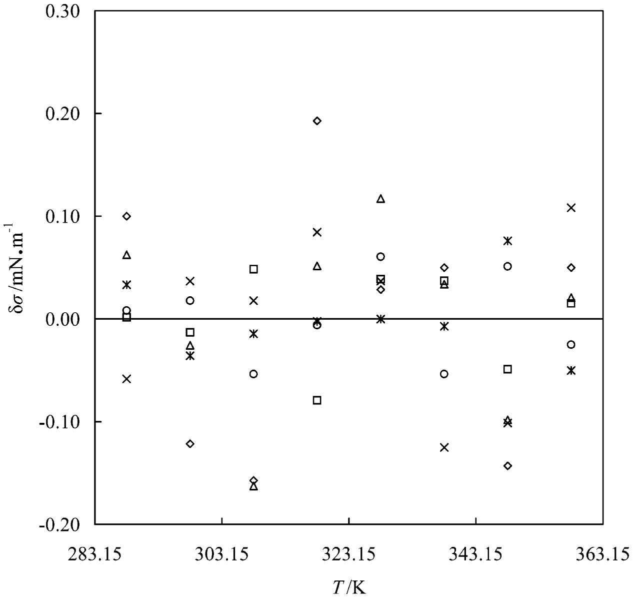

Figure 4. Variation of surface tensions with temperature for six flavor esters: ◇, ethyl phenylacetate; □, benzyl acetate; △, methyl benzoate; ×, ethyl caproate; ∗, ethyl caprylate; ○, ethyl isovalerate. Solid curves were calculated from eq 2.

where A, B, C, and D are fitted parameters. The values of fitted parameters were determined by a nonlinear regression analysis based on the least-squares method and are summarized along with the standard deviations (α) between the experimental and fitted values of the respective functions in Tables 3 to 6. The standard deviation is defined by

$$\alpha = \left[\sum_{i=1}^{m} \frac{(Y_i^{\mathrm{exptl}} - Y_i^{\mathrm{calcd}})^2}{m - p}\right]^{1/2} \tag{4}$$

where m is the number of experimental points and p is the number of adjustable parameters. The α values lie between 1.3×10^{-5} g·cm^{-3} and 1.3×10^{-4} g·cm^{-3}, between 8.2×10^{-4} mPa·s and 6.6×10^{-3} mPa·s, between 4.4×10^{-5} and 1.0×10^{-4}, and between 0.047 mN·m^{-1} and 0.16 mN·m^{-1} for ρ, η, n_D, and σ, respectively. The largest α values are corresponding to methyl benzoate, isoamyl acetate, ethyl phenylacetate, and ethyl salicylate for ρ, η, n_D, and σ, respectively. Figures 1 to 4 show the deviations between the experimental data and the calculated values versus temperature for ρ, η, n_D, and σ of six flavor esters. It can be seen that eqs 2 and 3 can be used to represent the experimental data very well.

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