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Henry's Law Constants and Infinite Dilution Activity Coefficients of Propane, Propene, Butane, 2-Methylpropane, 1-Butene, 2-Methylpropene, *trans*-2-Butene, *cis*-2-Butene, 1,3-Butadiene, Dimethylether, Chloroethane, 1,1-Difluoroethane, and Hexane in Tetrahydropyran

Yoshimori Miyano,*,† Sigemichi Uno,‡ Katsumi Tochigi,‡ Satoru Kato,§ and Hiroshi Yasuda||

Department of Chemistry and Bioscience, Kurashiki University of Science and the Arts, 2640 Nishinoura, Tsurajimacho, Kurashiki 712-8505, Japan, Department of Materials and Applied Chemistry, Nihon University, 1-8-14, Kandasurugadai, Chiyodaku, Tokyo, 101-8308, Japan, Department of Applied Chemistry, Graduate School of Engineering, Tokyo Metropolitan University, 1-1, Minamiohsawa, Hachiohji 192-0397, Japan, Corporate R&D Center, Showa Denko K.K., 5-1, Ogimachi, Kawasaki-ku, Kawasaki, Kanagawa, 210-0867, Japan

Henry's law constants and infinite dilution activity coefficients of propane, propene, butane, 2-methylpropane, 1-butene, 2-methylpropene, *trans*-2-butene, *cis*-2-butene, 1,3-butadiene, dimethylether, chloroethane, 1,1-difluoroethane, and hexane in tetrahydropyran in the temperature range of (250 to 330) K were measured by a gas stripping method, and partial molar excess enthalpies and entropies were evaluated from the activity coefficients. A rigorous formula for evaluating the Henry's law constants from the gas stripping measurements was used for these highly volatile mixtures. The estimated uncertainties are about 2 % for the Henry's law constants and 3 % for the infinite dilution activity coefficients. The Henry's law constants followed the order of the increasing Henry's law constant with decreases in the normal boiling point temperature of the solutes except for polar solutes. The partial molar excess entropies and enthalpies of the solutes at infinite dilution in tetrahydropyran are smaller than those in benzene. These excess properties of unsaturated hydrocarbons in tetrahydropyran are smaller than those of saturated hydrocarbons.

Introduction

A systematic study of gas solubilities including the Henry's law constant is useful in providing data required for the design of absorption processes as well as in developing solution thermodynamics. Tetrahydropyran (THP) is a six-membered ring ether. Although similar cyclic ethers such as tetrahydrofuran and 1,4-dioxane have been widely used as industrial materials, THP has not yet been used. The reason might be that it was difficult to produce a sufficient amount of pure THP for industrial use. However, Showa Denko K.K. has recently established a new THP production process[1] starting with hydrogen, acetaldehyde, and acrolein, satisfying 99.9 % purity. The physicochemical properties of THP are as follows:

(1) THP separates from the water, and the mutual solubilities are 2.5 wt % in water and 8.1 wt % in THP at 298 K. On the other hand, tetrahydrofuran and 1,4-dioxane dissolve in the water completely.

(2) THP is low toxic material.

(3) THP was stable under strong acidic conditions such as 1 wt % H_2SO_4 under reflux conditions for 7 h, and its peroxides hardly were formed at room temperature for 30 days.[2]

Though THP has many advantages described above, the thermodynamic data for THP are very scarce in the literature. Especially, the solubilities of gases in THP are not available in the literature. The purpose of this work is to measure Henry's law constants of the low molecular weight hydrocarbons and their isomers in THP and to identify their thermodynamic characteristics.

In this work, the Henry's law constants in THP for 13 solutes (propane, propene, butane, 2-methylpropane, 1-butene, 2-methylpropene, *trans*-2-butene, *cis*-2-butene, 1,3-butadiene, dimethylether, chloroethane, 1,1-difluoroethane, and hexane) are measured with the gas stripping method originally proposed by Leroi et al.,[3] and the infinite dilution activity coefficients of the solutes and the partial molar excess enthalpies and entropies are evaluated. The vapor pressures of THP are also measured.

Theory

The gas stripping method, originally proposed by Leroi et al.,[3] is based on the variation in the vapor-phase composition when the highly diluted solute in a liquid mixture in an equilibrium cell is stripped from the solution by the flow of inert gas (helium). The composition of the gas leaving the cell is periodically sampled and analyzed by gas chromatography. The peak area (S) of the solute decreases exponentially with the volume of inert gas flowing out of the cell according to the following rigorous relation.[4]

$$\ln\frac{S}{S_0} = \left(\frac{(H_g/\varphi_g^V)}{(1+\alpha)P_s^{sat} - \alpha(H_g/\varphi_g^V)} - 1\right)\ln\left(1 - \frac{(1+\alpha)P_s^{sat} - \alpha(H_g/\varphi_g^V)}{ZRTn_{s,0}^L + V_{GP,0}(H_g/\varphi_g^V)}V\right) \quad (1)$$

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* Corresponding author. E-mail: miyano@chem.kusa.ac.jp. Fax: +81-86-440-1062.

† Kurashiki University of Science and the Arts.

‡ Nihon University.

§ Tokyo Metropolitan University.

|| Corporate R&D Center, Showa Denko K.K..

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where S_0 is the peak area of the solute at time $t = 0$; P_s^{sat} is the saturated vapor pressure of the pure solvent (s); H_g is the Henry's law constant of the solute (g); Z is the compressibility factor of the saturated vapor in the cell; and φ_g^V represents the fugacity coefficient of the solute in the vapor phase. Superscripts V and L denote vapor and liquid phases, respectively. The $n_{s,0}^L$ is the number of moles of solvent in the liquid phase in the cell at $t = 0$. V is the volume of the saturated gas flowing out of the cell, and $V_{GP,0}$ is the initial volume of the vapor phase in the cell. The volume (V_{GP}) of the vapor phase in the cell at time t slightly depends on the gas volume withdrawn from the cell, and it is expressed as

$$V_{GP} = V_{GP,0} + \alpha V \qquad \alpha = \frac{P_s^{sat}\upsilon_s^{L,sat}}{ZRT} \tag{2}$$

where $\upsilon_s^{L,sat}$ is the liquid molar volume of the solvent at saturation; R is the gas constant; and T is the absolute temperature.

From eq 1, the Henry's law constant of the solute can be obtained from the slope of the line in a log−log plot as follows. The initial value of the Henry's law constant was set equal to zero, and the logarithm on the right side in eq 1 was calculated. By a process of successive iterations, the value of the Henry's law constant was determined.

The infinite dilution activity coefficient of the solute (γ^∞) can be obtained from the following relationships

$$H_g \equiv \lim_{x_g \to 0} \frac{f_g^V}{x_g} = f_g^{L,0}\gamma^\infty = f_g^{sat}\lambda\gamma^\infty = \varphi_g^{sat}P_g^{sat}\lambda\gamma^\infty \tag{3}$$

$$\gamma^\infty = \frac{H_g}{\varphi_g^{sat}P_g^{sat}\lambda} \tag{4}$$

where x_g is the mole fraction of the solute in the liquid phase; f_g^V is the fugacity of the solute in the vapor phase; $f_g^{L,0}$ is the fugacity of the pure solute in the reference state (pure liquid at system temperature and pressure); f_g^{sat} and φ_g^{sat} are, respectively, the fugacity and the fugacity coefficient of the solute at saturation; and P_g^{sat} is the saturated vapor pressure of the pure solute. λ is the Poynting correction factor and is well approximated as

$$\lambda \approx \exp\left(\frac{(P - P_g^{sat})\upsilon_g^{L,sat}}{RT}\right) \tag{5}$$

where P is the system pressure. The vapor pressures and the saturated liquid densities for pure solutes were taken from a reference.[5] The densities of THP were estimated from experimental data[6] and the modified Rackette technique.[7] The thermodynamic properties appearing in the above equations (φ_g^V, φ_g^{sat}, Z, etc.) were calculated from the virial equation of state truncated after the second virial coefficient. For propane and propene, however, the virial equation of state truncated after the third virial coefficient was used for this purpose because of its high volatility. The details of the calculation methods were described in the previous papers.[4,8]

Experimental Section

Details of the experimental apparatus were presented in our earlier papers.[4,9] About 36 cm^3 of THP was introduced into the equilibrium cell, having a volume of about 40 cm^3, and the quantity was determined by mass with an accuracy of 1 mg (0.003 %). The equilibrium cell was then immersed in a constant-temperature bath filled with ethylene glycol + water and connected to a supply of helium. The temperature was controlled to within ± 0.02 K and measured with a quartz thermometer (Hewlett-Packard Co., model 2804A) with an uncertainty of 0.01 K (0.01 %). About 2 cm^3 of the solute gas was introduced into the equilibrium cell by a syringe. Helium then began to flow into the equilibrium cell at a flow rate of about 2 cm^3·min^{-1}, which was measured with a mass flow meter (Kofloc Co., model 3300) with an uncertainty of 1 %. The pressure in the cell was approximately equal to atmospheric pressure measured by a pressure transducer (Paroscientific, Inc., Digiquartz pressure transducer model 215A, and Tsukasa Sokken Co. Ltd., Digiquartz Pressure Computer model 600S) to an uncertainty of 10 Pa (0.01 %).

Table 1. Vapor Pressures of Tetrahydropyran

T/K	P^s/kPa
257.96	0.98
260.07	1.13
270.28	2.16
279.82	3.70
289.94	6.28
290.00	6.30
300.01	10.23
332.98	40.00
341.09	53.33
347.72	66.66
353.38	79.99
358.37	93.33
360.22	98.66

The uncertainty of this measurement for the Henry's law constants may be considered to be within 2 %; it mostly depends on the uncertainty of the mass flow meter (1 %). The total uncertainty of the other measured variables such as the temperature, the pressure, and the mass of the solvent was estimated to be better than 1 %.

Materials. THP was supplied by Showa Denko K.K. with a specified minimum purity by GC of 99.7+ %. Butane, 2-methylpropane, 1-butene, and 2-methylpropene were supplied by Takachiho Kagaku Kogyo with specified minimum mass fraction purities of 0.998, 0.99, 0.99, and 0.99, respectively. Propane and propene were supplied by Japan Fine Products with specified minimum mass fraction purities of 0.995 and 0.995, respectively. *cis*-2-Butene, *trans*-2-butene, chloroethane, 1,1-difluoroethane, and 1,3-butadiene were supplied by Aldrich Chemicals with specified minimum purities by GC of 99+ %, 99+ %, 99.7+ %, 98+ %, and 99+ %, respectively. Dimethyl ether and hexane were supplied by Tokyo Kasei Kogyo Co. with a specified minimum purity of 99+ % and 96+ % by GC. All materials were used without further purification.

Results and Discussion

To evaluate the Henry's law constants from the gas stripping experiment, the vapor pressures of THP are necessary. The vapor pressures of THP were measured by Giner et al.[10] at the temperature range of (298 to 328) K and by Cabani et al.[11] at 298 K. The vapor pressures of THP at the temperature range of (258 to 360) K were measured in this work by a static method[12] at lower temperatures than 300 K and by a hold-up compensable ebulliometer method[13] at higher temperatures than 330 K and indicated numerically in Table 1. The results were correlated by the Antoine equation as follows

$$\ln P^s = 14.20363 - 3031.415/(T - 44.751) \tag{6}$$

Table 2. Henry's Law Constants and Infinite Dilution Activity Coefficients of Solutes in Tetrahydropyran[a]

T/K	H_g/kPa	γ^∞	P_g^{sat}/kPa	φ_g^{sat}	T/K	H_g/kPa	γ^∞	P_g^{sat}/kPa	φ_g^{sat}	$\Delta H^{E,\infty}/RT_{300}$	$\Delta S^{E,\infty}/R$
Propane										0.52	−0.11
250.06	428	2.08	219.0	0.94	309.98	1920	1.84	1274.1	0.85		
270.12	780	1.98	433.2	0.92	329.97	2710	1.79	1984.3	0.82	dev. = 0.20 %	
289.97	1270	1.90	770.3	0.89							
Propene										−0.06	−0.42
250.06	362	1.42	273.5	0.94	309.98	1760	1.44	1530.6	0.84		
270.12	680	1.42	532.3	0.91	329.97	2520	1.44	2365.7	0.80	dev. = 0.21 %	
289.97	1140	1.43	934.6	0.87							
Butane										0.59	−0.01
250.06	79	2.04	39.4	0.98	309.98	560	1.78	347.0	0.92		
270.12	173	1.94	92.2	0.96	329.97	890	1.72	591.7	0.89	dev. = 0.09 %	
289.97	326	1.85	187.7	0.94							
2-Methylpropane										0.56	−0.09
250.06	131	2.14	63.2	0.97	309.98	810	1.88	485.4	0.90		
270.12	272	2.04	140.0	0.95	329.97	1230	1.82	802.0	0.87	dev. = 0.27 %	
289.97	486	1.94	272.6	0.93							
1-Butene										0.03	−0.29
250.06	68	1.39	49.9	0.98	309.98	520	1.38	419.6	0.91		
270.12	153	1.39	114.8	0.96	329.97	840	1.38	706.8	0.88	dev. = 0.17 %	
289.97	296	1.38	230.1	0.94							
2-Methylpropene										0.05	−0.27
250.06	69	1.39	50.8	0.98	309.98	530	1.38	426.2	0.91		
270.12	155	1.39	116.5	0.96	329.97	850	1.37	718.6	0.88	dev. = 0.20 %	
289.97	300	1.38	233.6	0.94							
trans-2-Butene										0.07	−0.27
250.06	52	1.43	36.8	0.98	309.98	427	1.40	332.4	0.92		
270.12	119	1.41	87.2	0.97	329.97	700	1.40	568.0	0.90	dev. = 0.38 %	
289.97	236	1.40	178.9	0.95							
cis-2-Butene										0.12	−0.16
250.00	42.8	1.36	31.9	0.98	309.98	372	1.31	307.3	0.93		
270.07	100	1.33	77.5	0.97	330.00	630	1.32	533.5	0.90	dev. = 0.54 %	
289.98	204	1.32	162.6	0.95							
1,3-Butadiene										−0.48	−0.52
250.06	43.1	0.95	46.2	0.98	309.98	382	1.05	399.6	0.92		
270.12	101	0.97	107.5	0.96	329.97	650	1.09	677.7	0.89	dev. = 0.86 %	
289.97	207	1.01	217.4	0.94							
Dimethylether										0.07	−0.16
250.00	135	1.29	108.9	0.97	309.98	890	1.26	811.8	0.89		
270.07	284	1.26	238.7	0.95	330.00	1390	1.26	1330.7	0.86	dev. = 0.62 %	
289.98	530	1.26	460.6	0.92							
Chloroethane										−0.28	−0.21
250.00	19.1	0.90	21.4	0.99	309.98	209	0.94	235.3	0.95		
270.07	47.7	0.89	54.7	0.98	330.00	375	0.98	420.7	0.92	dev. = 2.18 %	
289.98	103	0.89	120.0	0.97							
1,1-Difluoroethane										−0.21	−0.71
250.06	166	1.57	110.1	0.96	309.98	1230	1.69	845.8	0.88		
270.12	376	1.64	244.3	0.94	329.97	1880	1.66	1397.3	0.85	dev. = 1.40 %	
289.97	720	1.67	475.4	0.91							
Hexane										0.68	0.14
290.00	24.5	1.76	14.0	0.99	319.99	78	1.65	48.2	0.97		
299.89	36.7	1.71	21.7	0.98	330.02	107	1.61	68.7	0.97	dev. = 0.33 %	
309.98	54	1.67	32.9	0.98							

[a] dev.: average deviation defined by eq 8.

where P^s/kPa is the vapor pressure of THP and T is the absolute temperature. The uncertainty of the measured vapor pressures may be considered to be within 0.3 %. The vapor pressures measured by Giner et al. and Cabani et al. are about 1.7 % smaller than those calculated from eq 6. This discrepancy may be caused by the difference of the purities of THP (they used THP with specified minimum purity of 99 %).

The Henry's law constants and the infinite dilution activity coefficients measured in this work are numerically indicated in Table 2. All experiments were conducted under atmospheric pressure, and the estimated fugacity coefficients of the solute in the vapor phase and the compressibility factors of the vapor were around unity ($\varphi_g^V = 1.00$, $Z = 1.00$) for all systems. However, for the evaluation of the infinite dilution activity coefficients, the nonideality of gases at saturation is not negligible. For highly volatile solutes such as propene, the vapor pressure at $T = 330$ K is about $P = 2400$ kPa, and the nonideality (φ_g^{sat}) should be evaluated. The estimated uncertainty for the vapor-phase fugacity coefficient at saturation was about 1 % as discussed in a previous paper[4] (the difference between the calculated values from the virial equation and the Soave equation of state[14] was less than 1 %). Therefore, the uncertainty of the obtained infinite dilution activity coefficients may be considered to be no greater than 3 %.

The Henry's law constants of nonpolar gases in liquids generally follow the order of increasing Henry's law constant with decreases in the normal boiling point temperature of the solute. As shown in Figure 1, the order of the Henry's law constants of the 13 solutes in THP at $T = 290$ K generally follows the above relationship except for some polar solutes (propene ($\mu = 0.4$ D), 2-methylpropene ($\mu = 0.5$ D), 1-butene ($\mu = 0.3$ D), *cis*-2-butene ($\mu = 0.3$ D), dimethylether ($\mu = 1.3$ D), 1,1-difluoroethane ($\mu = 2.3$ D), and chloroethane ($\mu = 2.0$ D)) where μ is the dipole moment ($1\ \text{D} = 3.162 \cdot 10^{-25}\ \text{J}^{1/2} \cdot \text{m}^{3/2}$).

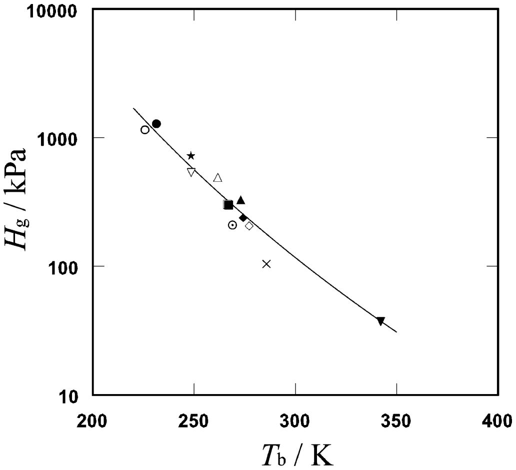

Figure 1. Relationship between the Henry's law constant of a solute in tetrahydropyran at 290 K and the boiling point temperature of the solute: ●, propane; ○, propene; ▲, butane; △, 2-methylpropane; ■, 1-butene; □, 2-methylpropene; ◆, *trans*-2-butene; ◇, *cis*-2-butene; ⊙, 1,3-butadiene; ▽, dimethylether; x, chloroethane; ★, 1,1-difluoroethane; ▼, hexane.

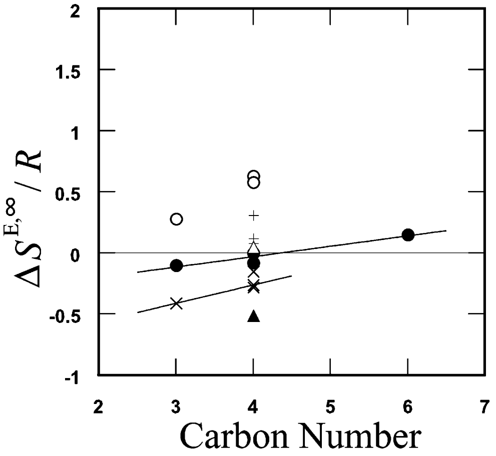

Figure 2. Partial molar excess entropies of solutes in tetrahydropyran: ●, alkanes; x, alkenes; ▲, alkadienes; ○, alkanes in benzene; +, alkenes in benzene; △, alkadienes in benzene.

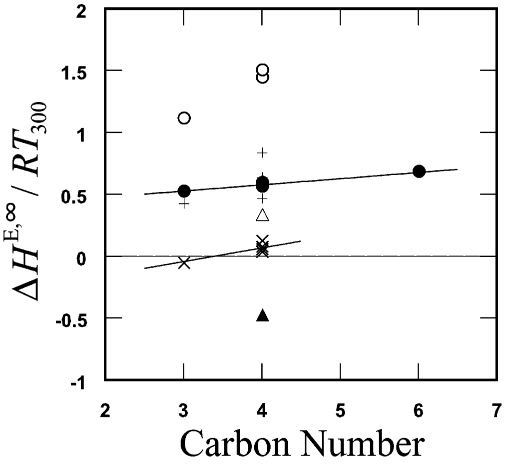

Figure 3. Partial molar excess enthalpies of solutes in tetrahydropyran: ●, alkanes; x, alkenes; ▲, alkadienes; ○, alkanes in benzene; +, alkenes in benzene; △, alkadienes in benzene.

The Henry's law constants of polar solutes in nonpolar or polar solvents generally become smaller than the expected values based on the simple relationship, because of the dipole-induced−dipole or dipole−dipole interactions.

The activity coefficient relates to the partial molar excess properties as follows

$$\ln \gamma^{\infty} = \frac{\Delta H^{E,\infty}}{RT} - \frac{\Delta S^{E,\infty}}{R} \tag{7}$$

where $\Delta H^{E,\infty}$ and $\Delta S^{E,\infty}$ are the partial molar excess enthalpy and the partial molar excess entropy of the solute at infinite dilution, respectively. In the temperature range of (250 to 330) K, the partial molar excess properties were assumed to be constants, and the infinite dilution activity coefficients were correlated by eq 7. From the correlations, the partial molar excess properties can be obtained as the slopes and the intercepts of the linear relations. The reduced partial molar excess properties ($\Delta H^{E,\infty}/RT_{300}$ and $\Delta S^{E,\infty}/R$) are also indicated in Table 2, where $T_{300} = 300$ K. The average deviation of the infinite dilution activity coefficients in these correlations is defined as follows

$$\sigma = \sqrt{\frac{1}{N}\sum_{j=1}^{N}\left[\frac{\gamma^{\infty}_{j,\mathrm{calcd}} - \gamma^{\infty}_{j}}{\gamma^{\infty}_{j}}\right]^{2}} \tag{8}$$

where N is the number of data points; γ^{∞}_{j} is the experimentally obtained infinite dilution activity coefficient; and $\gamma^{\infty}_{j,\mathrm{calcd}}$ is the infinite dilution activity coefficient calculated from eq 7. The average deviations for all systems are within the uncertainty of the experimental value (3 %), and the maximum deviation is 2.2 % for the chloroethane + THP system.

In Figures 2 and 3, the $\Delta S^{E,\infty}/R$ and $\Delta H^{E,\infty}/RT_{300}$ values of the hydrocarbons in THP are, respectively, plotted vs the carbon number of the solutes. These figures also include the excess properties of the same solutes in benzene.[15] Figure 2 shows that the $\Delta S^{E,\infty}/R$ values of alkanes in benzene are positive and larger than those in THP, implying that a solute molecule introduced into benzene destroys the parallel planar structures formed in benzene. Meanwhile, the $\Delta S^{E,\infty}/R$ values of the alkanes in THP are close to zero, implying that the degree of the order formed around the alkane molecule in THP is almost the same as that in the pure liquid. Therefore, the degree of the parallel planar structures formed in THP is lower than those in benzene. Butane and 2-methylpropane have almost the same molecular surface areas; therefore, these two solutes may destroy the parallel structures formed in the solvent to the same degree. However, as shown in Table 2, the $\Delta S^{E,\infty}/R$ value of 2-methylpropane in THP is negative and smaller than that of butane, indicating that the order of the parallel framework formed in pure liquid 2-methylpropane, i.e., the standard state, is lower than that in butane. Similarly, the $\Delta S^{E,\infty}/R$ values of C_4 hydrocarbons decrease in the order of butane > 1-butene > butadiene, implying that the larger the number of carbon−carbon double bonds in the hydrocarbon molecules, the lower the degree of the molecular order in their pure liquid states.

Figure 3 shows that the $\Delta H^{E,\infty}/RT_{300}$ values of the alkanes in benzene are positive and larger than those in THP, demonstrating that the alkane molecule surrounded by the distorted planar structures in benzene undergoes weaker attractive molecular interactions than in THP. The $\Delta H^{E,\infty}/RT_{300}$ values of 1,3-butadiene in THP are negative, implying that attractive molecular interactions at infinite dilution of 1,3-butadiene in THP are stronger than in its pure liquid state.

In Table 3, the $\Delta S^{E,\infty}/R$ and $\Delta H^{E,\infty}/RT_{300}$ values of butane are listed for four different solvents. Table 3 shows that the $\Delta S^{E,\infty}/R$ value in THP is close to zero; therefore, the molecular order formed in THP is much weaker than the parallel

Table 3. $\Delta S^{E,\infty}/R$ and $\Delta H^{E,\infty}/RT_{300}$ Values for Butane

solvent	$\Delta S^{E,\infty}/R$	$\Delta H^{E,\infty}/RT_{300}$
THP	−0.01	0.59
hexadecane[16]	0.28	0.15
benzene[15]	0.6	1.5
water[16]	−11.5	−1.6

framework formed in hexadecane. The notably negative $\Delta S^{E,\infty}/R$ value in water, −11.5, reflects the strong hydrophobic effects formed around butane molecules. Table 3 demonstrates that the excess free energy of butane at infinite dilution in THP is dominated by the enthalpy term, which is much different from the entropy control in alkane/water systems and short carbon-chain alkane/long carbon-chain alkane systems.

Conclusions

Henry's law constants, the infinite dilution activity coefficients, and the partial molar excess properties of the 13 solutes in THP at $T = (250 \text{ to } 330)$ K have been obtained from gas stripping measurements.

The Henry's law constant could be directly obtained from this experimental method. The Henry's law constants of the nonpolar solutes in THP have followed the order of the increasing Henry's law constant with decreases in the normal boiling point temperature of the solute.

The partial molar excess entropies and the enthalpies of hydrocarbons at infinite dilution in THP are smaller than those in benzene. The $\Delta H^{E,\infty}/RT_{300}$ and the $\Delta S^{E,\infty}/R$ values decrease in the order of alkanes > alkenes > alkadienes.

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