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Henry's Constants and Infinite Dilution Activity Coefficients of Propane, Propene, Butane, Isobutane, 1-Butene, Isobutene, *trans*-2-Butene, and 1,3-Butadiene in 1-Butanol and 2-Butanol from (250 to 330) K

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Henry's constants (or Henry's law constants) and the infinite dilution activity coefficients of propane, propene, butane, isobutane, 1-butene, isobutene, *trans*-2-butene, and 1,3-butadiene in 1-butanol and 2-butanol in the temperature range of (250 to 330) K were measured by a gas stripping method. A rigorous formula for evaluating the Henry's constants from the gas stripping measurements was used for the data reduction of these highly volatile mixtures. The accuracy is about 2% for the Henry's constants and 3% for the estimated infinite dilution activity coefficients. In the evaluation of the infinite dilution activity coefficients, the nonideality of the solute cannot be neglected, especially at higher temperatures. The estimated uncertainty of the infinite dilution activity coefficients includes 1% for nonideality.

Introduction

A systematic study of gas solubilities including the Henry's constant is useful in providing design data for absorption processes as well as indirectly aiding the analysis of molecular interactions in solutions.

Although a large number of alkane, alkene + alcohol solubility data have been published, few data are available for C_4 gases such as butane, 1-butene, and their isomers + alcohol mixtures. The solubility data will be useful in developing prediction methods. Especially for group contribution methods, it may be necessary to take into account the differences between isomers. To estimate the gas solubilities from a molecular theory or molecular simulation, however, an accurate intermolecular potential is necessary. The Henry's constant is directly related to the residual chemical potential of the solute at infinite dilution, which is evaluated from the intermolecular potential between a solute molecule and a solvent molecule. Therefore, the Henry's constant is a suitable macroscopic property for testing the intermolecular potential between different kinds of molecules.

Oxygenates have been used widely as fuel additives to increase the octane number. For these oxygenate processes, solubility data for hydrocarbons + alcohols mixtures are needed both in the design of the production facilities and also in the determination of the properties of final products.

The gas stripping method originally proposed by Leroi et al.[1] has been used to measure the activity coefficients at infinite dilution of solutes in nonvolatile solvents when the vapor pressures of solutes are negligibly small.[2,3] In previous work,[4−6] Henry's constants for propane, propene, butane, isobutane, 1-butene, isobutene, *trans*-2-butene, and 1,3-butadiene in methanol, 1-propanol, and 2-propanol were measured with this method. For these highly volatile solutes and solvents, a rigorous expression was derived for data reduction.

In this work, Henry's constants for propane, propene, butane, isobutane, 1-butene, isobutene, *trans*-2-butene, and 1,3-butadiene in 1-butanol and 2-butanol in the temperature range of (250 to 330) K are measured by the gas stripping method using the rigorous expression.

Theory

The gas stripping method originally proposed by Leroi et al.[1] is based on the variation of the vapor-phase composition when the highly dilute 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 by the following rigorous relation:[4,5]

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

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), and H_g is the Henry's constant of the solute, defined as follows:

$$H_g = \lim_{x_g \to 0} \frac{f_g^V}{x_g} \quad (2)$$

where f_g^V is the fugacity of the solute in the vapor phase at the system temperature and pressure, x_g is the mole fraction of solute in the liquid phase. Z is the compressibility factor of the saturated vapor in the cell, and φ_g^V represents the fugacity coefficients of the solute (g) in the vapor phase. Superscripts V and L denote vapor and liquid phases, respectively. $n_{s,0}^L$ is the number of moles of solvent

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in the liquid phase in the cell at $t = 0$. $V_{GP,0}$ is the initial volume of the vapor phase in the cell, and V the volume of the saturated gas flowing out of the cell. The volume of the vapor phase in the cell at time t, V_{GP}, slightly depends on the gas volume withdrawn from the cell and is given by

$$V_{GP} = V_{GP,0} + \alpha V \qquad \alpha = \frac{P_s^{sat} v_s^{L,sat}}{ZRT} \tag{3}$$

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

From eq 1, the Henry's constants of solutes can be obtained as the slope of the line in a log−log plot as follows: The initial value of the Henry's constant was set equal to zero, and the logarithm on the right-hand side in eq 1 was calculated. Then the new value of the Henry's constant was obtained. These calculations were iterated until a fixed value of the Henry's constant was obtained.

The infinite dilution activity coefficient of the solute, γ^∞, can be obtained from the following relationship:

$$H_g = f_g^{L,o}\gamma^\infty = f_g^{sat}\lambda\gamma^\infty = \varphi_g^{sat} P_g^{sat}\lambda\gamma^\infty \tag{4}$$

$$\gamma^\infty = \frac{H_g}{\varphi_g^{sat} P_g^{sat}\lambda} \tag{5}$$

where P is the Poynting correction factor and is well approximated as

$$\lambda \approx \exp\left(\frac{(P - P_g^{sat}) v_g^{L,sat}}{RT}\right) \tag{6}$$

where P is the system pressure. The vapor pressures and saturated liquid densities for pure substances were evaluated from the data book.[7] The thermodynamic properties appearing in the above equations ($\varphi_g{}^V$, φ_g^{sat}, Z, etc.) were calculated by 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 paper.[4]

The volume of the saturated gas flowing out of the equilibrium cell, V, can be evaluated from

$$(P - P_s^{sat})V + \frac{S_0}{KC_2(C_1 + 1)}[(1 - C_2V)^{C_1+1} - 1] = ZRTn_{He} \tag{7}$$

$$C_1 = \frac{(H_g/\sigma_g^V)}{(1 + \alpha)P_s^{sat} - \alpha(H_g/\varphi_g^V)} - 1 \qquad C_2 = \frac{(1 + \alpha)P_s^{sat} - \alpha(H_g/\varphi_g^V)}{ZRTn_{s,0}^L - V_{GP,0}(H_g/\varphi_g^V)} \tag{8}$$

where n_{HE} is the number of moles of helium flowing out of the cell, which was measured by a mass flow meter. K is the proportionality constant between the peak area of the solute detected by gas chromatography and its partial pressure. The values of K for all solutes were determined as follows: The pressure of a solute gas in the sampler for gas chromatography, which was placed in a constant-temperature air bath, was measured by a pressure transducer (Paroscientific, Inc., Digiquartz Pressure Transducer, model 215A and Tsukasa Sokken Co. Ltd., Digiquartz Pressure Computer, model 600S) to an accuracy of 10 Pa. Then the solute gas in the sampler was analyzed by gas chromatograpy, and the peak area for the solute gas was obtained. The relation between the pressures of the solute gas and the peak area was obtained.

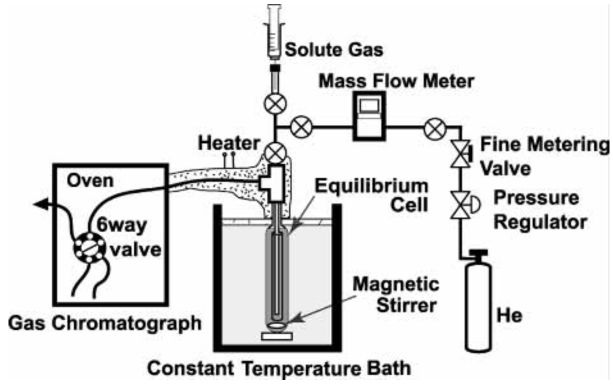

Figure 1. Schematic diagram of the experimental apparatus.

Experimental Section

Apparatus and Procedure. A similar experimental apparatus to that described previously[4] was used to measure the Henry's constants and is shown in Figure 1. About 36 cm^3 of the solvent (1-butanol or 2-butanol) was introduced into the equilibrium cell (volume ~40 cm^3), and the accurate quantity was determined by mass. Then the equilibrium cell was 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 by a quartz thermometer (Hewlett-Packard Co., model 2804A) with an accuracy of 0.01 K. About 1 cm^3 of the solute gas was introduced into the equilibrium cell by a syringe. The amount of solute gas introduced into the cell was adjusted to keep the mole fraction lower than 10^{-4} in solution. Then, helium began to flow into the equilibrium cell at a flow rate of about 2 cm^3·min^{-1}, which was measured by a mass flow meter (Koflok Co., model 3300) with a maximum flow rate of 2 cm^3·min^{-1} and an accuracy of 1%. The flow rate was controlled by a fine metering valve (double needles, Swagelok Co., SS2-D). The gas flowing out of the equilibrium cell was kept at a higher temperature than that of the bath to avoid any condensation and was introduced into a gas chromatograph (Hitachi Ltd., model G-3000, with double FID detectors) to measure the solute peak area, S. Sampling for gas chromatography was performed every 60 min and continued for about 20 h. The pressure in the cell, the temperature of the bath, and the total number of moles of helium, n_{HE}, which could be obtained from the integration of the flow rate, were also measured every 60 min. The pressure in the cell was approximately equal to atmospheric pressure, and atmospheric pressure was measured by a pressure transducer (Digiquartz Pressure Transducer, model 215A) to an accuracy of 10 Pa.

Figure 2 shows the equilibrium cell that contains a magnetic stirrer and an inner glass tube; both provide good mixing of the liquid. At low temperatures, however, it becomes difficult to mix the liquid because of the increased viscosity. For highly viscous fluids, the liquid above the inner glass tube cannot be stirred well. To get reproducible experimental results for these viscous fluids, it was important to reduce the liquid dead volume above the inner glass tube.

The accuracy of this measurement of the Henry's constants may be considered to be within 2%; it depends mostly on the accuracy of the mass flow meter.

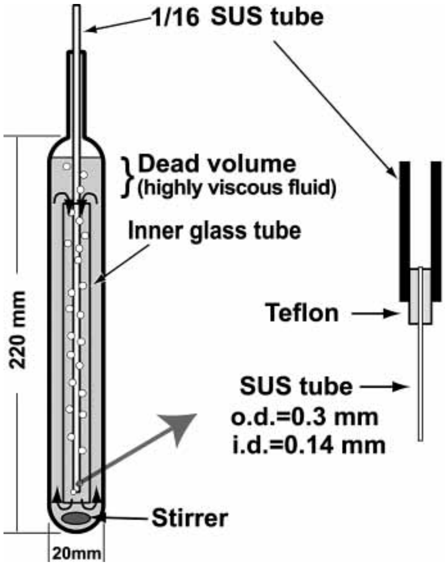

Figure 2. Equilibrium cell and dead volume in the liquid phase.

Table 1. Henry's Constants and Infinite Dilution Activity Coefficients of Solutes in 1-Butanol

T/K	H_g/kPa	γ^∞	T/K	H_g/kPa	γ^∞
	propane			1-butene	
250.01	746	3.6	250.01	168	3.5
270.00	1420	3.6	270.00	372	3.4
290.01	2380	3.6	290.01	731	3.4
310.00	3770	3.6	310.00	1260	3.3
330.00	5400	3.6	330.00	1970	3.2
	propene			isobutene	
249.99	770	3.0	250.01	168	3.4
270.00	1441	3.0	270.04	377	3.4
290.01	2420	3.0	290.01	733	3.4
310.00	3710	3.0	310.01	1260	3.3
330.00	5250	3.0	330.00	1980	3.2
	butane			*trans*-2-butene	
250.01	159	4.1	250.01	123	3.4
270.04	363	4.1	270.00	284	3.4
290.01	716	4.1	290.01	571	3.4
310.01	1260	4.0	310.00	1020	3.3
330.00	2010	3.9	330.00	1630	3.3
	isobutane			1,3-butadiene	
250.01	269	4.4	250.01	134	3.0
270.04	583	4.4	270.04	302	2.9
290.01	1080	4.3	290.01	598	2.9
310.01	1830	4.3	310.01	1050	2.9
330.00	2780	4.1	330.00	1660	2.8

Table 2. Henry's Constants and Infinite Dilution Activity Coefficients of Solutes in 2-Butanol

T/K	H_g/kPa	γ^∞	T/K	H_g/kPa	γ^∞
	propane			1-butene	
249.97	730	3.6	249.97	180	3.7
269.99	1380	3.5	269.99	390	3.6
290.00	2360	3.5	290.00	743	3.5
310.00	3630	3.5	310.00	1230	3.3
330.00	5040	3.3	330.00	1870	3.1
	propene			isobutene	
249.97	793	3.1	249.99	181	3.7
269.99	1470	3.1	270.00	394	3.5
290.00	2430	3.1	290.00	747	3.4
310.00	3670	3.0	310.00	1240	3.2
330.00	4910	2.8	330.00	1860	3.0
	butane			*trans*-2-butene	
249.96	162	4.2	249.97	133	3.7
270.00	363	4.1	269.99	301	3.6
290.00	708	4.0	290.00	584	3.5
310.00	1220	3.9	310.00	1000	3.3
330.00	1860	3.6	330.00	1550	3.1
	isobutane			1,3-butadiene	
249.97	269	4.4	249.99	158	3.5
270.00	572	4.3	270.00	349	3.4
290.00	1060	4.2	290.00	656	3.2
310.00	1740	4.1	310.00	1100	3.0
330.00	2550	3.8	330.00	1670	2.8

Materials. Butane, isobutane, 1-butene, and isobutene 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 at specified minimum mass fraction purities of 0.995 and 0.995, respectively. *trans*-2-Butene and 1,3-butadiene were supplied by Aldrich Chemicals at specified minimum mass fraction purities of 0.99 and 0.99, respectively. 1-Butanol and 2-butanol were supplied by Kishida Chemical Co. with specified minimum mass fraction purities of 0.995 and 0.995, respectively.

Results and Discussion

The Henry's constants and infinite dilution activity coefficients measured in this work are numerically indicated in Tables 1 and 2 for the 1-butanol and 2-butanol systems, respectively. Because all experiments were conducted under atmospheric pressure, the estimated fugacity coefficients of the solute in the vapor phase and the compressibility factors of the vapor were around unity ($\varphi_g^V = 1$, $Z = 1$) 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 should be evaluated. As described in the previous section, the nonideality of the solute was calculated by the virial equation of state. To examine the viability of the virial equation of state at these high pressures, the Soave equation of state[8] was used for comparative purposes to calculate the fugacity coefficients of the solute at saturation. The calculated results from the virial equation of state truncated after the third virial coefficients agree with that calculated from the Soave equation of state to within 1%. This means that the evaluated activity coefficients slightly depend on the estimation method for the nonideality, and it is difficult to judge which estimation method is better. Therefore, the accuracies of the obtained infinite dilution activity coefficients in Table 1 may be considered to be no greater than 3%.

Figures 3 and 4 show the temperature dependence of the Henry's constants of the eight gases in 1-butanol and 2-butanol, respectively, in the temperature range of (250 to 330) K. Hayduk et al.[9] measured the gas solubilities of propene, butane, isobutane, and isobutene in 1-butanol at 298.15−343.15 K and 101 kPa. The gas solubilities in 1-butanol at 101 kPa were also measured for propane and butane by Hayduk and Castaneda,[10] for butane and isobutane by Blais and Hayduk,[11] and for butane by Miyano and Hayduk.[12] In general, it is difficult to estimate the Henry's constants from gas solubility data at atmospheric pressure. The gas solubility data are usually measured at intermediate concentration, and the smoothed data are obtained from the correlation with an activity coefficient equation. Then the Henry's constants can be obtained from the extrapolation of the equation to infinite dilution. The Henry's constants so obtained will depend strongly on the equation. For comparative purposes, however, these Figures contain the extrapolated values, which were estimated with the Margules equation.[13]

Laakkonen et al.[14] measured the vapor−liquid equilibria (VLE) for 1-butene + 2-butanol at 326.18 K and 60−220 kPa. Gros et al.[15] measured the high-pressure VLE for propane + 2-butanol and propene + 2-butanol at 330−368

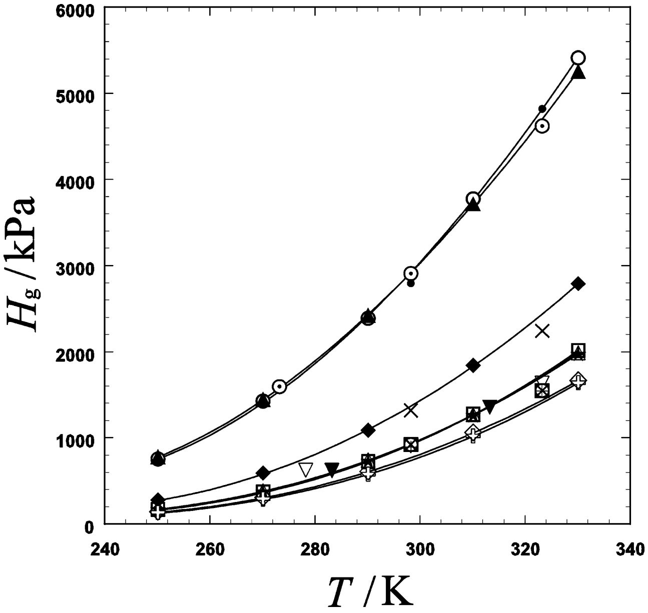

Figure 3. Henry's constants of eight gases in 1-butanol: ○, propane; ▲, propene; □, butane; ◆, isobutane; ★, 1-butene; △, isobutene; open plus symbols, *trans*-2-butene; ◇, 1,3-butadiene; ⊙, propane;[10] ●, propene;[9] ▽, butane;[10] ▼, butane;[12] squares with x's, butane;[11] ×, isobutane;[11] +, isobutene[9].

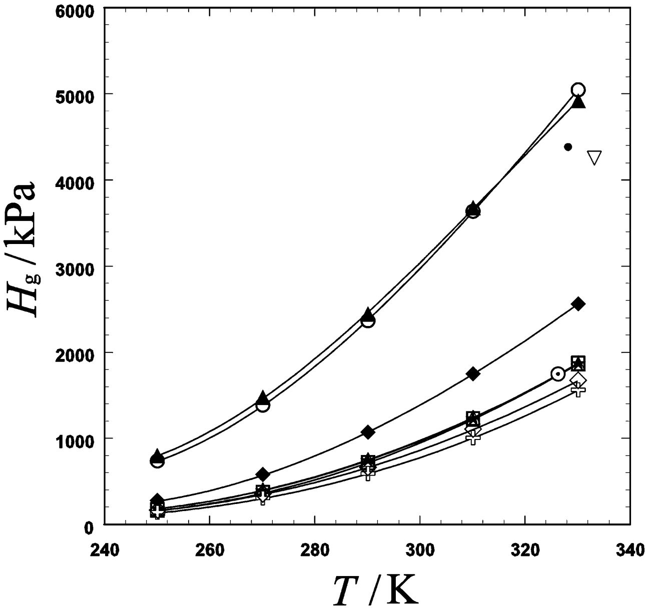

Figure 4. Henry's constants of eight gases in 2-butanol: ○, propane; ▲, propene; □, butane; ◆, isobutane; ★, 1-butene; △, isobutene; open plus symbols, *trans*-2-butene; ◇, 1,3-butadiene; ⊙, 1-butene;[14] ●, propane;[15] ▽, propene[15].

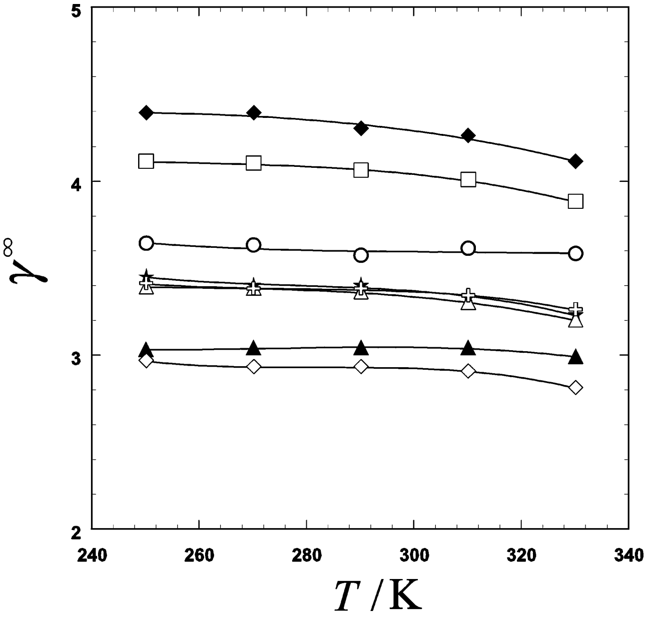

Figure 5. Infinite dilution activity coefficients of eight gases in 1-butanol: ○, propane; ▲, propene; □, butane; ◆, isobutane; ★, 1-butene; △, isobutene; open plus symbols, *trans*-2-butene; ◇, 1,3-butadiene.

K and 1000−4000 kPa. Using these VLE data, the Henry's constants were evaluated according to the definition of the Henry's constant:

$$f_g^{V} = H_g x_g \text{ at infinite dilution} \qquad x_g \to 0 \tag{9}$$

When we use the fugacity coefficient, we can get

$$P y_g = p_g = (H_g/\varphi_g^{V})\, x_g \tag{10}$$

where y_g is the mole fraction of the solute in the vapor phase, p_g is the partial pressure of the solute in the vapor phase, and x_g is the mole fraction of the solute in the liquid phase. From eq 10, the value of H_g/φ_g^{V} can be obtained as the slope of the plot of the solute partial pressure versus the solute mole fraction in the liquid phase at infinite dilution. As described previously, the fugacity coefficients of the solute at infinite dilution can be taken to be unity, and the Henry's constant can be obtained as the slope.

The Henry's constants measured in this work agree well with some of the estimated values from the gas solubilitiy data and also agree well with the estimated values from the VLE data by Laakkonen et al.,[14] which contain the data at lower pressures than atmospheric. However, the estimated Henry's constants from the high-pressure VLE data measured by Gros et al.[15] deviate about 20% from those in this work. This may depend on the lack of the VLE data at low concentration; the minimum mole fraction of the solutes in their data was 0.29.

The order of the Henry's constants at 290 K in 1-butanol and 2-butanol is propene (T_b =225.5 K) > propane (231.1 K) > isobutane (261.4 K) > isobutene (266.2 K) >1-butene (266.9 K) > butane (272.7 K) > 1,3-butadiene (268.7K) > *trans*-2-butene (274.0 K). The gas solubilities for simple systems generally follow the simple relationship that the Henry's constants of nonpolar gases in nonpolar solvents generally follow the order of increasing Henry's constant with decreases in the normal boiling point temperature (T_b) of liquefied gas. In a strongly polar solvent, however, the Henry's constants of polar gases such as propene, 1-butene, and isobutene decrease in general because of the dipole-pair intermolecular interaction. The Henry's constants of the eight gases follow a simple relationship in 1-butanol and 2-butanol, except for 1,3-butadiene. This means that 1-butanol and 2-butanol, both polar solvents, can be approximately treated as nonpolar solvents.

1,3-Butadiene, which is nonpolar, is smaller than others because it has two double bonds, which will make it easier to insert the molecule into a liquid. This may be the reason that 1,3-butadiene does not follow the relationship.

Figures 5 and 6 shows the temperature dependence of the infinite dilution activity coefficients for the same systems. From these Figures, it seems that the infinite dilution activity coefficients for C_4 gases can be classified into three groups. The first group is alkane, butane, and

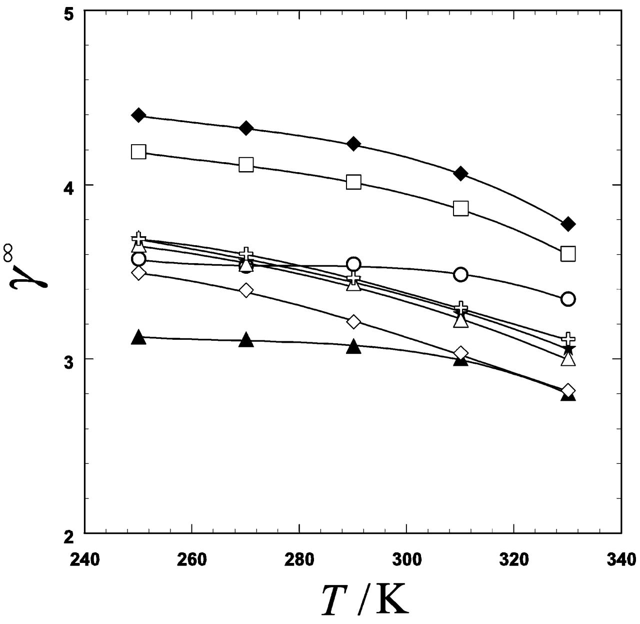

Figure 6. Infinite dilution activity coefficients of eight gases in 2-butanol: ○, propane; ▲, propene; □, butane; ◆, isobutane; ★, 1-butene; △, isobutene; open plus symbols, *trans*-2-butene; ◇, 1,3-butadiene.

its isomers. The second is alkene, 1-butene, and its isomers, and the last group is dialkene and 1,3-butadiene. Each group shows very similar values of the infinite dilution activity coefficients. The order of the infinite dilution activity coefficients is alkane > alkene > dialkene for C_4 gases. Also, we observe that the temperature dependencies of the activity coefficients for C_4 gases in 2-butanol are greater than those in 1-butanol, the activity coefficients of 1,3-butadiene in 2-butanol are more similar to those of the alkene group than that in 1-butanol, and finally the smoothed lines for C_4 and C_3 gases are mostly parallel.

Conclusions

Henry's constants and the infinite dilution activity coefficients of the eight gases in 1-butanol and 2-butanol have been obtained from gas stripping measurements at temperatures from (250 to 330) K. The Henry's constant did not depend on the nonideality of the solute and solvent for the systems studied in this work. The activity coefficients, however, strongly depended on the nonideality of the solute in the reference state.

The accuracy of Henry's constants measured in this work is within 2% over the entire temperature range. The accuracy of the infinite dilution activity coefficients, however, is within 3%, which is worse than that for Henry's constants because of the inaccuracy of the estimated nonideality of gases, especially for highly volatile gases such as propane and propene.

List of Symbols

f	fugacity
H	Henry's constant
K	proportionality constant for gas chromatography
n	number of moles
P	pressure
R	gas constant
S	peak area detected by GC
T	absolute temperature
t	time
V	volume
V_{GP}	volume of vapor phase in the cell
v	molar volume
x	mole fraction in the liquid phase
y	mole fraction in the vapor phase
Z	compressibility factor

Greek Letters

α	volume factor defined by eq 2
γ	activity coefficient
λ	Poynting correction factor
φ	fugacity coefficient

Subscripts

0	initial value at time = 0
g	solute (gas component)
s	solvent

Superscripts

°	property at a reference state
∞	property at infinite dilution
L	property in the liquid phase
V	property in the vapor phase
sat	property at saturation

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