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Infinite-Dilution Partition and Activity Coefficients for Alkanes and Bromo- and Chloroalkanes in Squalene-Dinonyl Phthalate Mixtures at 30 °C

Ghassan A. Oweimreen,* Yahya T. Al-Janabi, and Foad Y. Al-Hawaj

Department of Chemistry, King Fahd University of Petroleum and Minerals, Dhahran 31261, Saudi Arabia

Using gas-liquid chromatography, we obtained infinite-dilution partition and activity coefficients at 30.0 °C for *n*-hexane, *n*-heptane, 3-methylpentane, 2,4-dimethylpentane, and bromo- and chloroalkanes in pure squalene, in pure dinonyl phthalate, and in three mixtures thereof. Where appropriate, the results are contrasted with those from previous studies. The values of the partition coefficients were tested for their agreement with the Purnell-Andrade equation and with an equation derived by Laub, Martire, and Purnell on the basis of conventional models of solution. From fits of the data to the latter equation, solute-solvent and solvent-solvent interaction parameters were calculated.

Introduction

For a variety of gas-liquid chromatographic (GLC) systems in which a volatile solute component (A) is distributed between a binary nonvolatile solvent mixture (X + Y) and the gas phase, Purnell and de Andrade (1) find that the linear relation

$$K_{R(M)}^0 = \varphi_X K_{R(X)}^0 + \varphi_Y K_{R(Y)}^0 \quad (1)$$

is obeyed within experimental error. $K_{R(X)}^0$ and $K_{R(Y)}^0$ are the infinite-dilution solute partition coefficients in pure solvents X and Y, respectively. $K_{R(M)}^0$ is the infinite-dilution solute partition coefficient in a mixture where the volume fractions of X and Y are φ_X and φ_Y , respectively. On the other hand, Laub, Martire, and Purnell (2) derive the expression

$$\ln K_{R(M)}^0 = \varphi_X \ln K_{R(X)}^0 + \varphi_Y \ln K_{R(Y)}^0 + (V_X/V_Y)\chi_{Y(X)}\varphi_X\varphi_Y \quad (2)$$

where V represents molar volumes and $\chi_{Y(X)}$ is the Flory-type interaction parameter characterizing X-Y interactions. Throughout this paper, the letters B, C, and D will refer to the solvents squalene (SQA), dinonyl phthalate (DNP), and squalene (SQA), respectively. The letter A will refer to the solute.

Although the model (2) on which eq 2 is based requires $\chi_{Y(X)}$ to be independent of the nature of the solute, experimental evidence (2, 10, 11) shows that $\chi_{Y(X)}$ depends on solute-solvent interaction. For a given $K_{R(X)}^0/K_{R(Y)}^0$ ratio, eq 2 predicts curvature in $K_{R(M)}^0$ versus φ_X plots that can deviate negatively (for $\chi_{Y(X)} \leq 0$) or positively (for $\chi_{Y(X)} > 0$) from linearity, with near linearity possible if the value of $\chi_{X(Y)}$ (whether positive or neg-

Table I. Properties of the Pure Components at 30.00 °C

	$V/$ ($\text{cm}^3 \text{mol}^{-1}$)	$P_A^0/$ mmHg	$-B_{AA}/$ ($\text{cm}^3 \text{mol}^{-1}$)
solutes			
hexane	132.53	187.10	1808
heptane	148.39	58.38	2717
3-methylpentane	131.52	232.91	1768
2,4-dimethylpentane	150.92	122.75	2390
tetrachloromethane	97.70	143.00	1451
trichloromethane	80.86	238.77	1142
dichloromethane	64.99	529.97	803
1,2-dichloropropane	98.88	63.24	2544
1,2-dichloroethane	79.91	99.81	1650
1,2-dibromoethane	87.05	15.26	
1-bromobutane	109.02	52.71	
solvents			
squalene (D)	484.37		
dinonyl phthalate (C)	434.82		

ative) is close to zero (2). While eq 1 has been used in the window diagram method (3-5) for optimizing chromatographic separations, there is no doubt now (2, 6-11) that, in general, it is an approximate description of the true physical situation.

In our previous study (11), we obtained infinite-dilution partition and activity coefficients at 30.0 °C from GLC for *n*-pentane and six haloalkane solutes in squalene (SQA)-dinonyl phthalate (DNP) solvent mixtures. In this paper, we report values of infinite-dilution partition and activity coefficients at 30.0 °C from GLC for *n*-hexane, *n*-heptane, 3-methylpentane, 2,4-dimethylpentane, and chloro- and bromoalkanes in squalene (SQA)-dinonyl phthalate (DNP) solvent mixtures. The results are tested for their agreement with eqs 1 and 2 and are analyzed in the light of the treatment given in ref 2 to obtain solute-solvent and solvent-solvent interaction parameters.

Experimental Section

Squalene (SQA) and di-*n*-nonyl phthalate (DNP) were used as received from Phase Separations Ltd. (U.K.). High-purity solutes were also used as received from standard suppliers. The stationary liquids were deposited on Chromosorb W (60/80 mesh, acid-washed and silanized with dimethylchlorosilane) solid support from solutions in methylene chloride. The methylene chloride was removed by rotary evaporation. Traces of methylene chloride were removed from the coated supports by placing them in a vacuum dessicator for several days. The free-flowing powders were then packed in copper columns of 1-m length and 0.25-in. o.d. The weight percent of the liquid phase employed was in the neighborhood of 10% and was

* Author to whom correspondence should be addressed.

Table II. Partition Coefficients (K_R^0) as Functions of Volume Fractions (φ_{DNP}) at 30.00 °C

solute	K_R^0 at various φ_{DNP}				
	(pure SQE) 0.000	0.250	0.500	0.750	(pure DNP) 1.000
<i>n</i> -hexane	282.7	269.9	244.0	224.2	199.6
<i>n</i> -heptane	856.7	812.2	728.2	660.7	588.7
3-methylpentane	232.2	221.9	220.1	184.5	166.1
2,4-dimethylpentane	386.8	366.8	330.0	298.8	268.2
tetrachloromethane	679.6	655.7	609.2	579.8	533.8
trichloromethane	415.6	502.1	566.3	645.9	710.9
dichloromethane	134.4	169.0	196.7	231.3	257.7
1,2-dichloropropane	858.3	1016	1115	1242	1378
1,2-dichloroethane	499.8	628.1	726.4	844.1	961.7
1,2-dibromoethane	3156	3558	3827	4113	4354
1-bromobutane	1088	1196	1250	1320	1355

Table III. Solute Partition Coefficients with Pure Squalene, Pure Squalane, and Pure Dinonyl Phthalate Solvents at 30.00 °C

solute	$K_{R(B)}^0$ earlier studies	$K_{R(D)}^0$ this study	$K_{R(C)}^0$	
			earlier studies	this study
<i>n</i> -hexane	304.4 ^a	282.7	197.1 ^a	199.6
<i>n</i> -heptane	926.7 ^a	856.7	578.9 ^a	588.7
tetrachloromethane	466.2 ^b	679.6	527.8 ^b	533.8
trichloromethane	225.8 ^c	415.6	690.3 ^c	710.9
dichloromethane	77.08 ^c	134.4	244.8 ^c	257.7
1,2-dichloroethane	285.3 ^c	499.8	909.3 ^c	961.7

^aReference 2. ^bReference 8. ^cReference 11.

determined from the weight loss of three separately ashed samples (ca. 1 g each) of coated support (12). Packings with volume fractions of DNP (φ_c) equal to 0.25, 0.50, and 0.75 were also prepared. The solutes studied are listed in Table I.

Table I gives the molar volumes of the pure liquid components and the saturation vapor pressure and virial coefficients of the solutes at 30.0 °C. The solute molar volumes were obtained from Dreisbach's (13) compilation, while second virial coefficients were calculated from known critical constants (13), and a refined corresponding states relation (14, 15). The molar volumes of SQE and DNP were determined from density measurements with an Anton Paar digital precision system (16). Mixtures of SQE and DNP were found to have an effectively zero volume of mixing.

The gas chromatographic system used and the experimental conditions have already been described elsewhere (11). Also described elsewhere (12, 17) are details of the procedure followed to obtain partition coefficients. Sample size variation established the attainment of the infinite-dilution condition (17, 18). Varying solvent/support ratio indicated that interfacial effects were absent (17, 19). The percent standard deviation in $K_{R(S)}^0$, calculated from the standard relation

$$K_{R(S)}^0 = jF_c t'_R / V_s \quad (3)$$

was found (12) to be 1.2%. V_s is the volume of the stationary liquid in the column, j is the compressibility factor, F_c is the corrected flow rate, and t'_R is the retention time adjusted for column dead space ($t'_R = t_R - t_A$). Use of helium as carrier gas at low inlet pressures made virial corrections trivial, and the measured partition coefficients $K_{R(S)}$ correspond to those at zero column pressure drop, $K_{R(S)}^0$. In Table II, K_R^0 values as functions of the volume fraction of DNP are given.

Results and Discussion

The partition coefficients for a number of solutes in pure SQA ($K_{R(B)}^0$), in pure SQE ($K_{R(D)}^0$), and pure DNP ($K_{R(C)}^0$) are given in Table III. For trichloromethane, dichloromethane, and 1,2-

Table IV. Infinite-Dilution Solute Activity Coefficients with Pure Squalene and with Pure Dinonyl Phthalate at 30.00 °C

solute	$\gamma_{A(D)}$	$\gamma_{A(C)}$	
		this study	earlier studies
<i>n</i> -hexane	0.749	1.188	1.203; ^a 1.201 ^b
<i>n</i> -heptane	0.783	1.279	1.300; ^a 1.313 ^b
3-methylpentane	0.735	1.153	
2,4-dimethylpentane	0.831	1.347	
tetrachloromethane	0.405	0.576	0.584 ^c
trichloromethane	0.397	0.261	0.267; ^d 0.257 ^c
dichloromethane	0.559	0.326	0.334; ^d 0.329 ^c
1,2-dichloropropane	0.719	0.506	
1,2-dichloroethane	0.783	0.459	0.482; ^d 0.450 ^c
1,2-dibromoethane	0.807	0.656	
1-bromobutane	0.678	0.609	

^aReference 2. ^bReference 21. ^cReference 8. ^dReference 11.

dichloroethane, the $K_{R(C)}^0$ values from this study are higher, on average, than those from our earlier study (11) by 4.6%. An estimated 1.2% error in each of these studies along with differences between the two DNP samples, obtained from different manufacturers, account for this difference. That the DNP sample used in this study is different from the previously used sample (11) is evidenced by its lower (by 0.26%) molar volume. On the other hand, our $K_{R(C)}^0$ values for *n*-hexane, *n*-heptane, and tetrachloromethane are in very good agreement (about 1.4% on average) with those obtained for earlier studies (2, 8). Confidence in the results of this study ($K_{R(D)}^0$ and $K_{R(C)}^0$ values) arises from the very good agreement of the $K_{R(C)}^0$ values with earlier studies (2, 8, 11). This allows us to compare $K_{R(D)}^0$ and $K_{R(B)}^0$ values. In addition, the differences between $K_{R(D)}^0$ and $K_{R(B)}^0$ are quite large and can only be due to the differences in structure between the solvents SQE and SQA, respectively. As Table III shows, the polar chloroalkanes are retained longer in SQE (which contains double bonds) than in the saturated SQA. For the polar chloroalkanes, $K_{R(C)}^0 > K_{R(D)}^0 > K_{R(B)}^0$. The opposite is true for the nonpolar solutes *n*-hexane and *n*-heptane where $K_{R(C)}^0 < K_{R(D)}^0 < K_{R(B)}^0$. Tetrachloromethane, which is nonpolar like *n*-alkanes (thus, $K_{R(D)}^0 > K_{R(C)}^0$) yet contains polar C-Cl bonds like the polar chloromethanes (thus $K_{R(D)}^0 > K_{R(B)}^0$), exhibits the overall trend $K_{R(D)}^0 > K_{R(C)}^0 > K_{R(B)}^0$. Having $K_{R(D)}^0 > K_{R(B)}^0$ for the nonpolar tetrachloromethane molecule is indicative of interactions between the chlorine atoms and squalene. To our knowledge, partition coefficients for the solutes used in this study with SQE as solvent are not available in the literature. Such data are needed if SQE, a solvent intermediate in polarity relative to DNP and SQA, is to be considered as a component of stationary-phase mixtures for optimizing separations by GLC.

The solute (A) partition coefficient with pure DNP (C) and pure SQE (D) were converted to corrected activity coefficients $\gamma_{A(C)}$ (or $\gamma_{A(D)}$) via the relation

$$\gamma_{A(C)} = \frac{RT}{V_C p_A^0 K_{R(C)}^0} \exp \left\{ \frac{-(B_{AA} - V_A) p_A^0}{RT} \right\} \quad (4)$$

p_A^0 and B_{AA} are the bulk solute pressure and virial coefficient, T is the column temperature, and V_C (or V_D) is the molar volume of the stationary phase. Where B_{AA} values are not known (see Table I), the exponential correction term in eq 4, which is generally very close to unity, was disregarded. The results, given in Table IV, show the very good agreement of the $\gamma_{A(C)}$ values from this study with earlier careful measurements (2, 8, 11, 21).

Plots of $K_{R(M)}^0$ versus φ_C are presented for *n*-heptane (Figure 1a) and for representative haloalkane solutes (Figure 1b-d). The curvature and asymmetry of the plots are similar to those previously encountered (2, 10, 11). They show that the infinite-dilution partition coefficients for all the solutes examined in

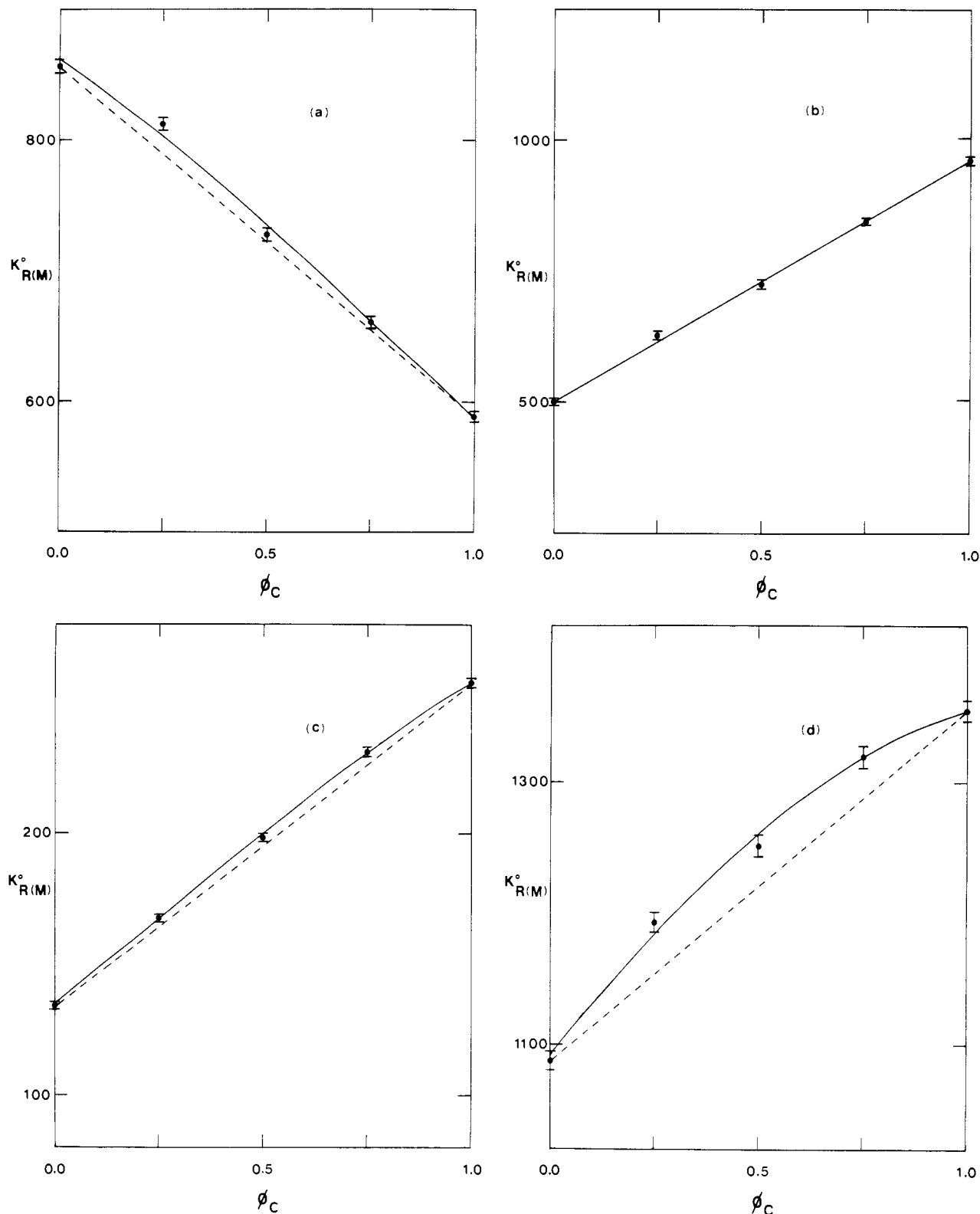


Figure 1. Partition coefficients as a function of volume fraction of dinonyl phthalate in squalene at 30.00 °C for (a) *n*-heptane, (b) 1,2-dichloroethane, (c) dichloromethane, and (d) 1-bromobutane. Points with 1.2% error bars are experimental points; solid curves are obtained with eq 5; dashed lines join the partition coefficients for pure squalene and pure dinonyl phthalate. The solid straight line in b is a linear fit of the experimental data for 1,2-dichloroethane.

this study agree, within experimental error, with values calculated from eq 2, which allows for interaction between the two solvents. In addition, the results for the 1,2-dichloroethane dichloromethane, trichloromethane, and 1,2-dichloropropane also gave nearly linear plots of $K_{R(M)}^{\circ}$ versus ϕ_C , and a linear least-squares analysis of their results gave correlation coeffi-

icients of 0.9994, 0.9990, 0.9988, and 0.9981, respectively. In our earlier study (11), these solutes have been shown to be associated with DNP and their association constants K_{AC} were evaluated.

Presented in Table V are $\chi_{A(D)}$ and $\chi_{A(C)}$, the solute interaction parameters in pure SQE and in pure DNP, respectively, and

Table V. Interaction Parameters at 30.00 °C

solute	$\chi_{A(D)}$	$\chi_{A(C)}$	$\chi_{C(D)}$	av % diff ^a
<i>n</i> -hexane	0.281	0.665	0.463	0.84
ref 2		0.663		
ref 6		0.674		
ref 21		0.645		
<i>n</i> -heptane	0.245	0.663	0.376	0.62
ref 2		0.674		
3-methylpentane	0.267	0.640	0.373	0.67
2,4-dimethylpentane	0.293	0.703	0.341	0.70
dichloromethane	0.561	-0.070	1.780	0.84
ref 11		0.058		
trichloromethane	0.343	-0.477	1.076	0.94
ref 11		-0.408		
tetrachloromethane	-0.100	0.165	0.354	0.51
1,2-dichloroethane	0.723	0.100	1.189	0.92
ref 11		0.187		
1,2-dibromoethane	0.682	0.387	0.721	0.50
1,2-dichloropropane	0.464	0.028	0.582	1.14
1-bromobutane	0.328	0.139	0.575	0.49

^a Average percent difference between $K_{R(M)}^0$ calculated from eq 5 with the above parameters and observed $K_{R(M)}^0$.

$\chi_{C(D)}$, the interaction parameter characterizing DNP–SQE interactions. They were obtained by analyzing the data for the solutes studied in accordance with eq 2 written in the form (2)

$$\ln K_{R(M)}^0 = \ln K_{R(D)}^0 + \left\{ \left(\frac{V_A}{V_C} - \frac{V_A}{V_D} \right) + \left(\chi_{A(D)} - \chi_{A(C)} + \frac{V_A}{V_C} \chi_{C(D)} \right) \right\} \varphi_C - \frac{V_A}{V_C} \chi_{C(D)} \varphi_C^2 \quad (5)$$

Only the $\chi_{A(C)}$ values may be compared with earlier studies since no previous measurements were made with SQE as solvent. Table V contrasts the $\chi_{A(C)}$ values obtained in this study using SQE–DNP mixtures with the $\chi_{A(C)}$ values of previous studies (2, 6, 11, 21) using SQA–DNP mixtures. We note that the agreement of the $\chi_{A(C)}$ values with previous studies is better for *n*-hexane and *n*-heptane than for the chloroalkanes. This is probably because chloroalkanes interact somewhat more strongly with SQE than with SQA while the interactions of *n*-alkanes with SQE and SQA are nearly equal. Table V also shows that the $\chi_{C(D)}$ values (which according to the model in ref 2 should be independent of the nature of the solute) are appreciably larger for the polar haloalkane solutes than for alkanes. Similar results have been previously encountered (2, 10, 11), and a least-squares-fit of them according to the relation (2)

$$\ln K_{R(M)}^0 = \ln K_{R(D)}^0 + \left\{ \left(\frac{V_A}{V_C} - \frac{V_A}{V_D} \right) + (\chi_{A(D)} - \chi'_{A(C)}) \right\} \varphi_C + \frac{V_A}{V_C} \chi_{C(D)} \varphi_D \varphi_C + \ln \left(1 + \frac{K_{AC}}{V_C} \varphi_C \right) \quad (6)$$

gave K_{AC} , the association constant of a weak 1:1 A–C complex, and $\chi'_{A(C)}$, the parameter characterizing solute–solvent interaction in the absence of complex formation. The $\chi'_{A(C)}$ values obtained for the solutes in these systems closely approached the $\chi_{A(C)}$ values for the noncomplexing alkane solutes, which indicated that a 1:1 complex forms between each of the solutes and the polar solvent.

From a least-squares fit of our results for the haloalkane solutes according to eq 6, values of K_{AC} and $\chi'_{A(C)}$ were obtained (see Table VI). Table VI shows that $\chi'_{A(C)}$ values for haloalkanes do not approach the $\chi_{A(C)}$ values for alkanes (see Table V). In addition, K_{AC} values from this study (using SQE–DNP mixtures) differ, for the same solutes, from K_{AC} values obtained from an earlier study (11) (using SQA–DNP mixtures). This and the absence of a clear trend in the K_{AC} values from

Table VI. Best-Fit Interaction Parameters and Association Constants for Haloalkane Solutes at 30.00 °C

solute	$\chi_{A(D)}$	$\chi'_{A(C)}$	K_{AC}	av % diff ^a
trichloromethane	0.036	0.044	0.30	0.59
dichloromethane	0.563	0.582	0.40	0.65
1,2-dichloropropane	0.465	0.358	0.17	0.51
1,2-dichloroethane	0.726	0.672	0.34	0.64
1,2-dibromoethane	0.683	0.767	0.20	0.46
1-bromobutane	0.329	0.456	0.16	0.65

^a Average percent difference between $K_{R(M)}^0$ calculated from eq 6 with the above parameters and observed $K_{R(M)}^0$.

this study indicate the existence of interactions between the haloalkane solutes and SQE. Unfortunately, the effect of such interactions cannot be examined in the presence of the more polar DNP solvent. Interactions between SQE and the haloalkane solutes will be examined in a future study involving SQE–SQA mixtures. Nevertheless, the fact that the haloalkane–SQE interactions are much weaker than the haloalkane–DNP interactions is obvious from the nearness of the $\chi_{A(D)}$ and $\chi'_{A(C)}$ values (see Table VI).

Conclusions

Partition and activity coefficients for alkanes and chloro- and bromoalkanes were obtained at infinite dilution in SQE–DNP mixtures. The results for all the solutes considered in this study agree, within experimental error, with eq 2. In addition, 1,2-dichloroethane, dichloromethane, and trichloromethane gave virtually linear $K_{R(M)}^0$ vs φ_C plots.

Trends in the partition coefficients of the solutes in SQE, SQA, and DNP were identified and rationalized. The trend for the polar chloroalkanes was $K_{R(C)}^0 > K_{R(D)}^0 > K_{R(B)}^0$. This trend is reversed for the nonpolar alkanes. Tetrachloromethane exhibited the trend $K_{R(D)}^0 > K_{R(C)}^0 > K_{R(B)}^0$. That $K_{R(D)}^0$ and $K_{R(C)}^0$ are greater than $K_{R(B)}^0$ is attributed to the polar character of the C–Cl bonds. That $K_{R(D)}^0$ is greater than $K_{R(C)}^0$ is attributed to the overall nonpolar character of the tetrachloromethane molecule.

The various solute–solvent and solvent–solvent interaction parameters were calculated on the basis of the treatment given in ref 2. While the results indicate that weak 1:1 complexes form between the polar haloalkane solutes and DNP, no clear trend was observed among their association constants, K_{AC} . This is attributed to simultaneous interactions of different strengths, between the haloalkane solutes and SQE. It is hoped that an assessment of these haloalkane–SQE interactions may be made from a future study involving SQE–SQA solvent mixtures.

Registry No. SQE, 111-02-4; DNP, 84-76-4; hexane, 110-54-3; heptane, 142-82-5; 3-methylpentane, 96-14-0; 2,4-dimethylpentane, 108-08-7; tetrachloromethane, 56-23-5; trichloromethane, 67-66-3; dichloromethane, 75-09-2; 1,2-dichloropropane, 78-87-5; 1,2-dichloroethane, 107-06-2; 1,2-dibromoethane, 106-93-4; 1-bromobutane, 109-65-9.

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Correction

Simple Apparatus for Vapor-Liquid Equilibrium Measurements with Data for the Binary Systems of Carbon Dioxide with *n*-Butane and Isobutane

Lloyd A. Weber (*J. Chem. Eng. Data* **1989**, *34*, 171-175).

Data from Table III were inadvertently omitted from the published article. The additional data are as follows:

Table III. Experimental Results for the CO₂ + *n*-C₄H₁₀ System (Component 1, CO₂)

<i>P</i> /bar	<i>x</i> ₁ /%	<i>y</i> ₁ /%
<i>T</i> = 344.26 K		
76.98	63.2	78.4
79.44	66.4	77.4
80.46	68.1	75.8
81.1	69.4	74.6