

# Partitioning of Phenylalkanols between Micelles and Water Studied by Limiting Interdiffusion Coefficients in Water and Tetradecyltrimethylammonium Bromide Solutions<sup>†</sup>

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The Taylor dispersion method was used to measure limiting interdiffusion coefficients for benzyl alcohol, 2-phenylethanol, 3-phenylpropan-1-ol, 4-phenylbutan-1-ol, and 5-phenylpentan-1-ol in water and tetradecyltrimethylammonium bromide micellar solutions at 298.2 K. These data were used to obtain the degree of binding of the phenylalkanols to the micelles. Partition coefficients of the phenylalkanols between the micellar pseudophase and the bulk aqueous phase and values of the standard Gibbs energy for transfer from the bulk aqueous phase to the micellar pseudophase were calculated. The results are compared with literature values for the sodium dodecyl sulfate and hexadecyltrimethylammonium micellar systems.

## Introduction

Although diffusion coefficients of alkanols in water have been measured by many workers,<sup>1–10</sup> the absence of data for alkanols having phenyl group is quite surprising. Because of their UV absorption, phenylalkanols could be used as probes to investigate solute–solvent interactions even at dilute concentrations. One of the purposes of the present study is to provide diffusion coefficients for phenylalkanols in water. The second purpose is to obtain partition coefficients of phenylalkanols between micellar and bulk aqueous phases. Partition of alkanols between micelles and water has been of interest for a long time.<sup>11–31</sup> To obtain micelle–water partition coefficients, a number of methods have been used, such as solute vapor pressure,<sup>12</sup> depression of the critical micelle concentration (CMC),<sup>13–18</sup> differential UV absorption,<sup>19</sup> NMR self-diffusion,<sup>20–22</sup> NMR paramagnetic relaxation,<sup>23,24</sup> calorimetry,<sup>25,26</sup> potentiometry,<sup>27</sup> micellar liquid chromatography,<sup>28,29</sup> and fluorescence<sup>30</sup> methods. Although the partition of benzyl alcohol has been studied for aqueous solutions of sodium dodecyl sulfate (SDS),<sup>15,16,19,21,23,26</sup> dodecyltrimethylammonium bromide (DTAB),<sup>15,16,22,23</sup> and hexadecyltrimethylammonium bromide (CTAB),<sup>18</sup> the data for higher homologues are quite limited.<sup>18,19</sup>

Stilbs<sup>20–22</sup> obtained the degree of binding,  $p$ , of a number of solutes to SDS and DTAB micelles in D<sub>2</sub>O by measuring self-diffusion coefficients of the solutes in the micellar solutions. This is a neat technique because the value of  $p$  can be obtained at any concentration of the solute or micelle by a simple calculation using self-diffusion coefficients. It is particularly powerful in determining the  $p$  value for a solute at some high concentration, which may be necessary in many practical applications. In this method, however, systems having a very low solute concentration cannot easily be studied, and the method is not quite suited to obtain thermodynamic partition coefficients, especially when the activity coefficient of the solute varies with the concentration in the micellar pseudophase. Using

the Taylor dispersion method, we can determine limiting interdiffusion coefficients, where the solute concentration is quite low in comparison with the surfactant concentration in the micellar state. In this study, we measured limiting interdiffusion coefficients for five phenylalkan-1-ols in water and aqueous tetradecyltrimethylammonium bromide (TTAB) solutions. From these data, we obtained values of the degree of binding of the phenylalkanols to the TTAB micelles. Furthermore, we obtained partition coefficients of phenylalkanols between the micellar pseudophase and the bulk aqueous phase because they can be compared with literature values obtained even at different concentrations of the solute and the surfactant.

## Experimental Section

Phenylalkanols of the highest purity available were obtained from Wako Pure Chemical Industries (Wako), Alfa Aesar, or Tokyo Chemical Industry (TCI) and used as received. The chemicals and their purities as determined by the manufacturers using capillary gas chromatography (cGC) or GC were benzyl alcohol (Wako, 100.0 %, cGC), 2-phenylethanol (Wako, 99.9 %, cGC), 3-phenylpropan-1-ol (Wako, 98.8 %, cGC), 4-phenylbutan-1-ol (Alfa Aesar, 99.3 %, GC), and 5-phenylpentan-1-ol (TCI, 99.1 %, cGC). TTAB (TCI, 98.7 %) did not show any minimum near the CMC in the surface tension–concentration curve obtained using a du Nouy tensiometer and was used as received. Pyrene (Wako, > 98 %) was recrystallized using petroleum ether. Water was distilled twice after deionization.

Diffusion coefficients were measured using the Taylor dispersion method.<sup>32–35</sup> In this method, a small amount of a solution is injected into a solvent flowing through a capillary tube under laminar flow conditions. The solute is dispersed along the tube as a result of diffusion and convection. When the following condition is satisfied,

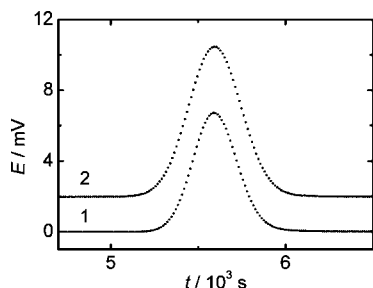
$$6.9 \ll \frac{Ua}{D} \ll \frac{4L}{a} \quad (1)$$

where  $U$  is the average flow speed of the carrier solution,  $a$  is the radius of the capillary,  $D$  is the (binary) diffusion coefficient, and  $L$  is the distance in which the greater part of the change in concentration takes place, the concentration of the solute,  $c$ , at

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**Figure 1.** Recorder output voltage as a function of time after the sample injection: 1, Benzyl alcohol in water; 2, benzyl alcohol in 0.04 mol·dm<sup>-3</sup> TTAB solution.

a sufficiently long time,  $t$ , after the solute injection can be expressed as a function of the position as<sup>32,33</sup>

$$c = \frac{M}{\pi a^2 (4\pi kt)^{1/2}} \exp\left(-\frac{x_1^2}{4kt}\right) \quad (2)$$

in which  $M$  is the number of moles of the solute injected,  $x_1 = x - Ut$ , where  $x$  is the distance from the injector, and  $k$  is given by

$$k = \frac{a^2 U^2}{48D} \quad (3)$$

Experimentally, it is more convenient to detect the concentration at a fixed position. The concentration at the detector, which is placed at  $l = Ut_R$ , where  $t_R$  is the residence time of the solute in the tube, is expressed as a function of time as

$$c(t) = \frac{2M}{\pi a^3 U} \left(\frac{3D}{\pi t}\right)^{1/2} \exp\left[-\frac{12D(t - t_R)^2}{a^2 t}\right] \quad (4)$$

Although the above result is for a straight tube, conditions to justify the use of eq 4 for a coiled tube have been fully described.<sup>34</sup> An HPLC pump (Hitachi L-7110) was used to pass either water or 0.04 mol·dm<sup>-3</sup> TTAB solution through a Teflon capillary tube (0.5 mm i.d., 20 m in length) made into a coil 0.35 m in diameter. With an HPLC injector (Rheodyne-7125), 20 mm<sup>3</sup> of an aqueous solution containing either a phenylalkanol (ca. 0.001 mol·dm<sup>-3</sup>) or 0.04 mol·dm<sup>-3</sup> TTAB + phenylalkanol (ca. 0.001 mol·dm<sup>-3</sup>) was injected into the carrier stream. At the end of the stream, the phenylalkanol concentration was detected using a UV-vis detector (Hitachi L-7420) at a wavelength between (256 and 261) nm. The detector signal was transferred to a computer via a Shimadzu CBM-10A communications bus module. Other experimental details have been described elsewhere.<sup>36-38</sup> The detector signal (output voltage) was fitted to the expression<sup>10,39</sup>

$$v(t) = A_0 + A_1 t + A_2 t^{-1/2} \exp\left[-A_3 \frac{(t - A_4)^2}{t}\right] \quad (5)$$

to obtain values for  $A_0$  through  $A_4$  using data analysis software (Microcal, Origin). Diffusion coefficients were calculated from the relation  $D = A_3 a^2 / 12$ .

## Results and Discussion

Figure 1 shows dispersion peaks for benzyl alcohol (1) in water and (2) in 40 mM TTAB solution. The recorder signal was obtained every 6 s, but the data are plotted every 12 s for clarity. Sample solutions were injected  $N$  times ( $5 \leq N \leq 10$ ) for each solute (phenylalkanol). Each dispersion peak was fitted to eq 5, and the  $D$  value was obtained from the  $A_3$  value. The

**Table 1.** Limiting Interdiffusion Coefficients of Phenylalkanols ( $D$ ) in Water and 0.04 mol·dm<sup>-3</sup> TTAB Solutions at 298.2 K and Degrees of Binding of the Solute to the Micelles ( $p$ )

solute	$10^9 \cdot D / \text{m}^2 \cdot \text{s}^{-1}$		
	in water	in TTAB	$p$
benzyl alcohol	0.855 ± 0.017	0.584 ± 0.010	0.347 ± 0.019
2-phenylethanol	0.781 ± 0.014	0.454 ± 0.007	0.463 ± 0.015
3-phenylpropan-1-ol	0.722 ± 0.019	0.262 ± 0.002	0.711 ± 0.009
4-phenylbutan-1-ol	0.664 ± 0.018	0.173 ± 0.001	0.833 ± 0.006
5-phenylpentan-1-ol	0.532 ± 0.009	0.105 ± 0.001	0.933 ± 0.003
pyrene		0.0746 ± 0.0007	1 <sup>a</sup>

<sup>a</sup> This value is assumed.

average  $D$  value for the  $N$  determinations for each solvent is listed in Table 1 together with its standard deviation; the uncertainty in the value of  $A_3$  in eq 5 for each peak was smaller than the uncertainty associated with the  $D$  value shown in Table 1. Solutions injected contained  $1 \cdot 10^{-3}$  mol·dm<sup>-3</sup> phenylalkanol. As they flowed through the capillary tube, the solutes were further diluted by a factor of 10 to 50 at the end of the capillary. Therefore, the results obtained can be taken as limiting interdiffusion coefficients. Our value of  $D$  for benzyl alcohol in water ( $0.830 \cdot 10^{-9}$  m<sup>2</sup>·s<sup>-1</sup>) may be compared to the value ( $0.803 \cdot 10^{-9}$  m<sup>2</sup>·s<sup>-1</sup>) obtained by Stilbs in D<sub>2</sub>O.<sup>21</sup> It is surprising that no other aqueous solution data are available, since there are many data for alkanols without a phenyl group.<sup>1-10</sup>

From the diffusion coefficients of phenylalkanol in water,  $D_w$ , and in micellar solution,  $D$ , we can obtain the degree of binding,  $p$ , of the solute to the micelle using the equation<sup>20-22,40</sup>

$$D = (1 - p)D_w + pD_m \quad (6)$$

where  $D_m$  is the diffusion coefficient of the micelle. The diffusion coefficient of pyrene in the TTAB solution was taken as the  $D_m$  value because essentially all of the pyrene molecules are solubilized in the micelles.<sup>37,40</sup> The resulting  $p$  values are also listed in Table 1, and they are seen to increase with increasing alkyl chain length, as expected. From the  $p$  values, we can obtain the partition coefficient of the solute, which in terms of mole fractions is defined as

$$K_x = \frac{x_m}{x_w} \quad (7)$$

where  $x_m$  is the mole fraction of the solute in the micellar pseudophase and  $x_w$  is the mole fraction of the solute in the bulk aqueous phase. In terms of molarity, the partition coefficient is defined as

$$K_c = \frac{c_m}{c_w} \quad (8)$$

where  $c_m$  and  $c_w$  are the molarities of the solute in the micellar pseudophase and bulk aqueous phase, respectively. The  $K_x$  values can be calculated from the  $p$  values using

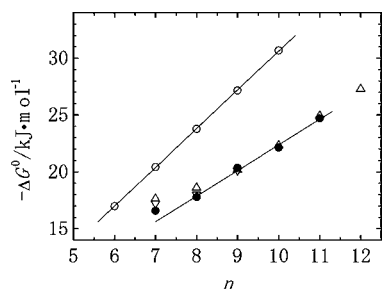
$$K_x = \frac{p}{1 - p} \cdot \frac{n_w}{n_m} \quad (9)$$

where  $n_w$  is the total number of moles (water + monomeric surfactant + solute) in the bulk aqueous phase and  $n_m$  is the total number of moles (surfactant + solute) in the micellar pseudophase. It should be noted that in the present study, the solute concentration (less than  $1 \cdot 10^{-3}$  mol·dm<sup>-3</sup>) was much lower than the micellar surfactant concentration, [TTAB] - CMC, where [TTAB] was 0.0400 mol·dm<sup>-3</sup> and the CMC was 0.0035 mol·dm<sup>-3</sup>. Therefore,  $n_m$  can be approximated as the

**Table 2. Partition Coefficients  $K_x$  and  $K_c$  of Phenylalkanols between Micelles and Water**

solute	system <sup>a</sup>	surfactant composition	$K_x$	$K_c$	source
benzyl alcohol	TTAB + H <sub>2</sub> O	0.04 mol·dm <sup>-3</sup>	799 ± 67	44 ± 4	this work
benzyl alcohol	DTAB + H <sub>2</sub> O		470		refs 15 and 16
benzyl alcohol	DTAB + D <sub>2</sub> O (308 K)	0.16 mol·dm <sup>-3</sup>	790 <sup>b</sup>	45 <sup>b</sup>	ref 22
benzyl alcohol	DTAB + D <sub>2</sub> O (308 K)	0.16 mol·kg <sup>-1</sup>	605 <sup>c</sup>		ref 23
benzyl alcohol	CTAB + H <sub>2</sub> O		1041 ± 186		ref 18
benzyl alcohol	SDS + H <sub>2</sub> O		440		refs 15 and 16
benzyl alcohol	SDS + H <sub>2</sub> O		730 ± 30 <sup>d</sup>		ref 26
benzyl alcohol	SDS + H <sub>2</sub> O		1220		ref 19
benzyl alcohol	SDS + D <sub>2</sub> O	0.24 mol·kg <sup>-1</sup>	432 <sup>e</sup>		ref 23
benzyl alcohol	SDS + D <sub>2</sub> O	0.24 mol·dm <sup>-3</sup>	450 <sup>f</sup>	33 ± 5	ref 21
2-phenylethanol	TTAB + H <sub>2</sub> O	0.04 mol·dm <sup>-3</sup>	1295 ± 76	71 ± 4	this work
2-phenylethanol	CTAB + H <sub>2</sub> O		1420 ± 254		ref 18
2-phenylethanol	SDS + H <sub>2</sub> O		1810		ref 19
3-phenylpropan-1-ol	TTAB + H <sub>2</sub> O	0.04 mol·dm <sup>-3</sup>	3690 ± 160	201 ± 9	this work
3-phenylpropan-1-ol	CTAB + H <sub>2</sub> O		3374 ± 603		ref 18
3-phenylpropan-1-ol	SDS + H <sub>2</sub> O	0.012–0.070 mol·dm <sup>-3</sup>	3390		ref 19
4-phenylbutan-1-ol	TTAB + H <sub>2</sub> O	0.04 mol·dm <sup>-3</sup>	7495 ± 290	410 ± 16	this work
4-phenylbutan-1-ol	SDS + H <sub>2</sub> O		8100		ref 19
5-phenylpentan-1-ol	TTAB + H <sub>2</sub> O	0.04 mol·dm <sup>-3</sup>	21100 ± 990	1150 ± 54	this work
5-phenylpentan-1-ol	SDS + H <sub>2</sub> O		23200		ref 19

<sup>a</sup>  $T = 298.2$  K unless otherwise noted. <sup>b</sup> Calculated by us from the tabulated  $p$  value of 0.69 using eqs 9 and 11, assuming that the partial molar volume of DTAB in the micellar state in D<sub>2</sub>O is 290 cm<sup>3</sup>·mol<sup>-1</sup> and the CMC value of DTAB in D<sub>2</sub>O is 0.015 mol·dm<sup>-3</sup>. <sup>c</sup> Calculated by us from the tabulated  $p$  value of 0.64 assuming that the CMC value of DTAB in D<sub>2</sub>O is 0.015 mol·kg<sup>-1</sup>. <sup>d</sup> Converted by us by multiplying by 55.5 the original values of  $K_{x,m} = x^{m^m}/m^m$ , where  $x^m$  is the mole fraction of the solute and  $m^m$  is the molality of the solute in the bulk aqueous phase. <sup>e</sup> Calculated by us from the tabulated  $p$  value of 0.67 assuming that the CMC value of SDS in D<sub>2</sub>O is 0.008 mol·kg<sup>-1</sup>. <sup>f</sup> Calculated by us from the tabulated  $p$  value of 0.67 and the partial molar volume of 250 cm<sup>3</sup>·mol<sup>-1</sup> for SDS in the micellar state.



**Figure 2.** Standard Gibbs energies for transfer of phenylalkanols from the bulk aqueous phase to the micellar pseudophases and of 1-alkanols from the bulk aqueous phase to the respective bulk 1-alkanol phases as functions of the solute carbon number: ●, phenylalkanols + TTAB + water (this study); △, phenylalkanols + SDS + water (ref 19); ▽, phenylalkanols + CTAB + water (ref 18); ○, 1-alkanols + 1-alkanols + water (ref 43).

number of moles of the surfactant in the micellar phase (i.e., 0.0365 mol per 1 dm<sup>3</sup> of solution). The  $n_w$  value can be approximated as the number of moles of water molecules per 1 dm<sup>3</sup> of solution and was calculated using

$$n_w = \frac{1000 - V_s([\text{TTAB}] - \text{CMC})}{18.02} \quad (10)$$

where  $V_s$  is the partial molal volume of TTAB in the micellar state (i.e., 330 cm<sup>3</sup>·mol<sup>-1</sup>).<sup>41,42</sup> The  $K_c$  value can be calculated using

$$K_c = \frac{p}{1-p} \cdot \frac{V_w}{V_m} \quad (11)$$

where  $V_w$  is the volume of the bulk aqueous phase and  $V_m$  is the volume of the micellar pseudophase. The  $V_w$  value was computed as 1000 -  $V_m$ , where the  $V_m$  value was estimated using

$$V_m = V_s([\text{TTAB}] - \text{CMC}) \quad (12)$$

Values of both  $K_x$  and  $K_c$  are listed in Table 2 together with literature values.

As far as we searched, there are no data available for the TTAB micellar system. For benzyl alcohol, our value of  $K_x$  is larger than that obtained for the DTAB + H<sub>2</sub>O system at 298.2 K by Treiner<sup>15,16</sup> using a CMC depression method, close to that obtained for the DTAB

+ D<sub>2</sub>O system at 308 K by Stilbs<sup>22</sup> using NMR self-diffusion measurements, and smaller than that obtained for the CTAB + H<sub>2</sub>O system at 298.2 K by Abu-Hamdiyyah<sup>18</sup> using the CMC depression method. Literature values are also listed for the SDS + H<sub>2</sub>O and SDS + D<sub>2</sub>O systems, but the values vary by as much as a factor of 3. For 2-phenylethanol, our value of  $K_x$  is close to that obtained by Abu-Hamdiyyah<sup>18</sup> for the CTAB + H<sub>2</sub>O system and smaller than that obtained by Kawamura et al.<sup>19</sup> for the SDS + H<sub>2</sub>O system. For 3-phenylpropan-1-ol, our value is fairly close to those obtained by Abu-Hamdiyyah<sup>18</sup> for CTAB + H<sub>2</sub>O and by Kawamura et al.<sup>19</sup> for SDS + H<sub>2</sub>O. For 4-phenylbutan-1-ol and 5-phenylpentan-1-ol, our values are quite close to those obtained by Kawamura et al.<sup>19</sup>

From the  $K_x$  values, we calculated values of the standard Gibbs energy for transfer of the solutes from the bulk aqueous phase to the micellar pseudophase using the relation

$$\Delta G^\circ = -RT \ln K_x \quad (13)$$

where  $R$  is the gas constant and  $T$  is the absolute temperature. The results are plotted in Figure 2 as a function of  $n$ , the number of carbons in the solute (closed circles). From 2-phenylethanol ( $n = 8$ ) to 5-phenylpentan-1-ol ( $n = 11$ ), the plot gives a fairly good linear relationship, which is expressed by

$$-\Delta G^\circ/\text{kJ} \cdot \text{mol}^{-1} = (-0.16 \pm 1.11) + (2.25 \pm 0.12)n \quad (14)$$

Also plotted in Figure 2 for comparison are  $\Delta G^\circ$  values obtained from the solubility of 1-alkanols in water,<sup>43,44</sup> for which the linear fit is expressed by

$$-\Delta G^\circ/\text{kJ} \cdot \text{mol}^{-1} = (-3.52 \pm 0.14) + (3.41 \pm 0.02)n \quad (15)$$

The dependence on the number of carbons for the transfer of phenylalkanols to the micellar pseudophases (2.25 kJ mol<sup>-1</sup>) is 66% of that for the transfer of the 1-alkanols to the respective bulk 1-alkanol phases (3.41 kJ mol<sup>-1</sup>).

In Figure 2, data for the transfer of phenylalkanols to the SDS<sup>19</sup> and CTAB<sup>18</sup> micellar phases are also plotted. In the cases of 3-phenylpropan-1-ol, 4-phenylbutan-1-ol, and 5-phenylpentan-1-ol ( $n = 9$  to 11), our results for the TTAB system are surprisingly

close to those for the SDS system,<sup>19</sup> though the methods used were quite different. In the cases of benzyl alcohol and 2-phenylethanol ( $n = 7$  and 8), the  $\Delta G^\circ$  values ( $K_x$  values) obtained by the three groups differ from each other. It may be expected that the partition coefficient for a given nonpolar solute should increase with increasing surfactant chain length, as is the case for nonpolar gases in micelles of sodium alkyl sulfates.<sup>45</sup> In the case of a nonpolar coordination compound having an aromatic nature, tris(acetylacetonato)cobalt(III), the solubility increases greatly with increasing chain length in anionic sodium alkyl sulfate micelles, but the chain-length dependence is very weak in cationic alkyltrimethylammonium bromide micelles.<sup>46</sup> We are currently studying the effects of alkyl chain length and the nature of polar head groups on the partition coefficients of the phenylalkanol.

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