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Binary Mutual Diffusion Coefficients of Aqueous Solutions of β-Cyclodextrin at Temperatures from 298.15 to 312.15 K

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Binary mutual diffusion coefficients (interdiffusion coeffcients) have been measured for aqueous solutions of β-cyclodextrin (β-CD) at concentrations from (0.002 to 0.008) mol·dm^{-3}. The effect of temperature, from (298.15 to 312.15) K, was also analyzed. The concentration dependence of D is discussed on the basis of Hartley's equation. The activation energy for the diffusion process, 18719 J·mol^{-1}, was calculated from data of D at different temperatures. This activation energy is in good agreement with that estimated using Stokes−Einstein equation (18644 J·mol^{-1}). Activity coefficients for aqueous β-CD solutions were also estimated from Hartley equation and experimental D.

Introduction

Aqueous solutions of cyclodextrins (CDs) have become important systems in fundamental and applied research due to their wide range of applications related to chemical synthesis and catalysis, analytical chemistry, and pharmaceutical chemistry.[1−3] CDs are cyclic oligosaccharides[4,5] having a truncated cone structure with a hydrophilic external surface and a relatively nonpolar cavity. CDs form inclusion complexes with a wide variety of molecules and ions.[6−10] With inorganic salts, both the anions and cations are involved in these complexes.[9,10] Thus, a significant property of CDs is their ability to include other organic or inorganic compounds into their cavities,in both the solid state and solution, to form inclusion complexes. These binding interaction results have been used in areas as diverse as enzymatic synthesis,[11] drug delivery,[12] catalysis,[13] and energy transfer studies.[14] The theoretical basis of such interactions has been analyzed[15] to understand how CDs may affect the structures of compounds and their solutions.[16]

The most common pharmaceutical application of cyclodextrins is to increase the solubility of poorly soluble drugs and to engineer slow-release delivery systems for drug molecules. Consequently, the diffusion properties of aqueous CD solutions are of considerable interest. Diffusion coefficients for aqueous CD solutions at 298.15 K have been reported,[17,18] but relevant data for the same concentrations at other temperatures—especially at 310.15 K for in vivo pharmaceutical applications—are scarce in the literature.[17] In the present study, mutual diffusion (interdiffusion) coefficients D measured by the Taylor dispersion method are reported for aqueous solutions of β-CD at concentrations from (0.002 to 0.008) mol·dm^{-3} and at temperatures from (298.15 to 312.15) K. Diffusion measurements at higher concentrations were not made because of the low solubility of β-CD (0.016 mol·dm^{-3} at 298.15 K). The concentration dependence of D is compared with data obtained by optical interferometry[18] at 298.15 K. In addition, the accuracy of the Taylor diffusion measurements is assessed by measuring binary mutual diffusion coefficients for aqueous solutions of potassium chloride and sucrose at 298.15 K for comparison with previously reported D values measured by accurate optical interferometric and conductometric techniques.[19,20]

Experimental Section

Materials. β-CD supplied by Sigma had a water mass fraction of 13.1 % (determined by drying to constant mass at 420 K in a nitrogen atmosphere). The solutions for the diffusion measurements were prepared by weighing an appropriate amount of β-CD and dissolved in bi-distilled water using calibrated volumetric flasks. The β-CD concentration, in molarity, was calculated taking into account the percentage of water mentioned above. The solutions were freshly prepared and deaerated for about 30 min before each set of runs.

Procedure. Dispersion methods for diffusion measurements are based on the work carried out by Taylor in 1953 and 1954[21−23] on the dispersion of small amounts of solution injected into laminar carrier streams of solvent or solution of different composition, flowing through a long capillary tube.[24−27] The length of the Teflon dispersion tube used in the present study was measured directly by stretching the tube in a large hall and using two high-quality theodolytes and appropriate mirrors to accurately focus on the tube ends. This technique gave a tube length of 3.2799 (± 0.0001) × 10^3 cm, in agreement with less-precise check measurements using a good-quality measuring tape. The radius of the tube, 0.05570 (± 0.00003) cm, was calculated from the tube volume obtained by accurately weighing (resolution 0.1 mg) the tube when empty and when filled with distilled water of known density.

At the start of each run, a six-port Teflon injection valve (Rheodyne, model 5020) was used to introduce 0.063 cm^3 of solution into the laminar carrier stream of slightly different composition. A flow rate of 0.17 cm^3·min^{-1} was maintained

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Table 1. Mean Diffusion Coefficients (D) at 298.15 K Measured by Taylor Dispersion for 0.100 mol·dm^{-3} Test Solutions and Previously Reported Literature values (D_{Lit})[a]

	Δc	D	S_{D}[b]	D_{Lit}	$D - D_{\mathrm{Lit}}$
solute	mol·dm^{-3}	10^{-9} m^2·s^{-1}	10^{-9} m^2·s^{-1}	10^{-9} m^2·s^{-1}	D_{Lit} %
sucrose	0.05	0.526	0.001	0.522	0.8
KCl	0.05	1.809	0.001	1.838	−1.6

[a] D_{Lit} represents the literature diffusion coefficients values.[28−32] [b] S_{D} is the standard deviation of that mean.

by a metering pump (Gilson model Minipuls 3) to give retention times of about 8 × 10^3 s. The dispersion tube and the injection valve were kept at 298.15 K and 303.15 K (± 0.01 K) in an air thermostat.

Dispersion of the injected samples was monitored using a differential refractometer (Waters model 2410) at the outlet of the dispersion tube. Detector voltages, $V(t)$, were measured at accurately timed 5 s intervals with a digital voltmeter (Agilent 34401 A) with an IEEE interface. Binary diffusion coefficients were evaluated by fitting the dispersion equation

$$V(t) = V_0 + V_1 t + V_{\max} (t_{\mathrm{R}}/t)^{1/2} \exp[-12D(t - t_{\mathrm{R}})^2/r^2 t] \quad (1)$$

to the detector voltages. The additional fitting parameters were the mean sample retention time t_{R}, peak height $V_{\max}$, baseline voltage V_0, and baseline slope V_1.

The concentrations of the injected solutions ($\bar{c} + \Delta c$) and the carrier solutions ($\bar{c}$) differed by 0.004 mol·dm^{-3} or less. Solutions of different composition were injected into each carrier solution to confirm that the measured diffusion coefficients were independent of the initial concentration difference and therefore represented the differential value of D at the carrier-stream composition.

Tests of the Dispersion Equipment. Binary aqueous sucrose and KCl solutions were used to test the operation of the dispersion equipment. These systems were chosen because their diffusion coefficients are accurately known from conductometric and optical interferometric measurements (uncertainties < 0.5 %)[28−32] and span a useful range of D values from about 0.3 × 10^{-9} m^2·s^{-1} to 1.8 × 10^{-9} m^2·s^{-1}. Table 1 gives the concentrations of the test solutions, the mean D values at 298.15 K determined from 4 to 6 replicate dispersion profiles, and their relative deviations from the literature D values. The reproducibility of these results was usually within ± 1 %. Comparison of the results suggests an uncertainty of 1 to 2 % for the D values reported here, which is typical for Taylor dispersion measurements.

Results and Discussion

The Taylor dispersion equipment was used to measure diffusion coefficients for β-CD in aqueous solutions at 298.15 K, 303.15 K, 308.15 K, and 312.15 K and concentrations from (0.002 to 0.008) mol·dm^{-3}. Table 2 gives the average D value for each carrier solution determined from four profiles generated by injecting samples that were more or less concentrated than the carrier solution. Good reproducibility was obtained, within ± 1 %. In Figure 1, the average D values at each composition and temperature are plotted against the β-CD concentration. Comparison of our results at 298.15 K with the interferometric D values for aqueous β-CD reported by Paduano et al.[18] (Table 2) suggests an acceptable uncertainty of (1 to 2) % for the Taylor D values.

Table 2. Mutual Diffusion Coefficients of Aqueous β-Cyclodextrin Solutions (D)[a] **Measured by the Taylor Technique at Different Temperatures (T) and Concentrations, $\bar{c}$**

T = 298.15 K				
$\bar{c}$	Δc	D	S_{D}[b]	100 ($D - D_{\mathrm{Lit}}$)
mol·dm^{-3}	mol·dm^{-3}	10^{-9} m^2·s^{-1}	10^{-9} m^2·s^{-1}	D_{Lit}[c]
0.002	0.002	0.324	0.003	0.9
0.004	0.004	0.323	0.001	1.2
0.006	0.004	0.321	0.003	1.3
0.008	0.004	0.318	0.002	0.0

T = 303.15 K		T = 308.15 K		T = 312.15 K	
D	S_{D}[b]	D	S_{D}[b]	D	S_{D}[b]
10^{-9} m^2·s^{-1}	10^{-9} m^2·s^{-1}	10^{-9} m^2·s^{-1}	10^{-9} m^2·s^{-1}	10^{-9} m^2·s^{-1}	10^{-9} m^2·s^{-1}
0.389	0.005	0.430	0.006	0.456	0.005
0.388	0.004	0.428	0.005	0.454	0.006
0.381	0.004	0.427	0.006	0.454	0.002
0.378	0.004	0.423	0.006	0.453	0.002

[a] Each tabulated D value is the average of four replicate measurements. [b] S_{D} is the standard deviation of that mean. [c] 100($D - D_{\mathrm{Lit}}$)/D_{Lit} is the relative difference in percent between the Taylor D values and the D_{Lit} values reported by Paduano et al.[18]

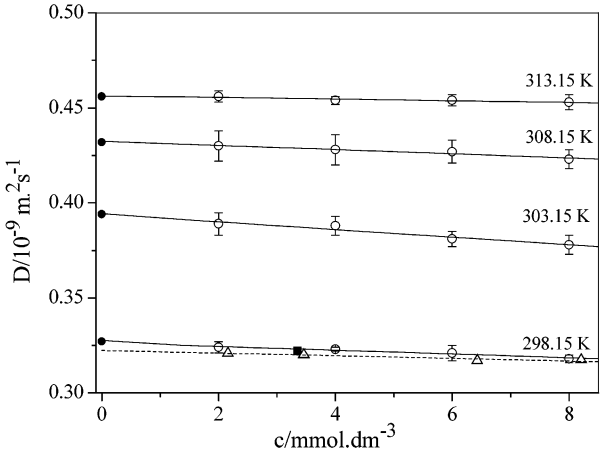

Figure 1. Mutual diffusion coefficients of aqueous β-cyclodextrin solutions: ○, this work; △, Paduano et al.;[18] ■, Longsworth;[17] and ● D^0 (see Table 3). The solid and dashed lines are fitted eq 2.

Table 3. Least-Squares Values of Parameters D^0 and A for the Concentration Dependence of D (eq 2)

T/K	D^0	A	σ[a]
298.15	0.326	−1.00	7.07×10^{-4}
298.15	0.322[b]	−0.69[b]	6.90×10^{-4}[b]
303.15	0.394	−2.00	1.73×10^{-3}
308.15	0.432	−1.10	9.49×10^{-4}
312.15	0.456	−0.45	5.92×10^{-4}

[a] Standard deviations. [b] From the D_{Lit} values reported by Paduano et al.[18]

The concentration dependence of the measured diffusion coefficients is accurately represented (standard deviation < 1 %) by the linear equation:

$$D/10^{-9}\ \mathrm{m^2{\cdot}s^{-1}} = D^0[1 + A(c/\mathrm{mol{\cdot}dm^{-3}})] \quad (2)$$

D^0 is the diffusion coefficient at infinite dilution. The least-squares values of D^0 and parameter A are listed in Table 3.

Concentration Dependence of D. The concentration dependence of the mutual diffusion coefficient for dilute solutions of nonionic, nonassociating solutes is given by Hartley's equation:[32]

$$D/10^{-9}\ \mathrm{m^2{\cdot}s^{-1}} = D^0\left(1 + \frac{\mathrm{d}\ln\gamma}{\mathrm{d}\ln c}\right)_{T,P} \quad (3)$$

Table 4. Comparison of the Thermodynamic *B* Coefficients Evaluated from the Taylor and Gouy Diffusion Data and from the Osmotic Data Reported by Miyajima

T/K	B	T/K	B
298.15	-1.00^a	303.15	-2.00^a
298.15	-0.69^b	308.15	-1.10^a
298.15	-1.17^c	312.15	-0.45^a

[a] From our D values (Table 2) and eq 5 (i.e., $A = B$). [b] From the D_{Lit} values reported by Paduano et al.[18] (Table 2) and eq 5 (i.e., $A = B$). [c] From the osmotic data values reported by Miyajima et al.[33]

Table 5. Activity Coefficients (γ) Evaluated from Equation 4 Using the Thermodynamic *B* Coefficients Indicated in Table 4

	T = 298.15 K			T = 303.15 K	T = 308.15 K	T = 312.15 K
c/mol·dm^{-3}	γ	γ^a	γ^b	γ	γ	γ
0.002	0.9980	0.9986	0.9977	0.9960	0.9978	0.9991
0.004	0.9960	0.9972	0.9953	0.9920	0.9956	0.9982
0.006	0.9940	0.9959	0.9930	0.9881	0.9934	0.9973
0.008	0.9920	0.9945	0.9907	0.9841	0.9912	0.9964

[a] Values evaluated from the Gouy diffusion data.[18] [b] Values evaluated from the osmotic data reported by Miyajima et al.[33]

where γ is the thermodynamic activity coefficient of the solute. The Hartley equation, though very useful, is limited to the analysis of diffusion in dilute solutions because variations in the viscosity with concentration and the counterflow of solvent relative to the solute are neglected.

For dilute aqueous solutions of α-cyclodextrin and γ-cyclodextrin, the molarity c and molality m composition scales are nearly identical numerically. The activity coefficients data reported by Miyajima et al.[33] are accurately represented by the equation

$$\ln \gamma = B(c/\text{mol·dm}^{-3}) \tag{4}$$

with $B = -1.17$ at 298.15 K. We make the reasonable assumption that eq 4 can also be used to represent the activity coefficients of dilute aqueous solutions of β-CD, as suggested by Paduano et al.[18] Combining Hartley's equation and eq 4 gives

$$D/10^{-9}\ \text{m}^2\text{·s}^{-1} = D^0(1 + B(c/\text{mol·dm}^{-3})) \tag{5}$$

for the predicted concentration dependence of the mutual diffusion coefficient of dilute aqueous solutions of β-CD.

If activity data are available for the evaluation of $\text{d}\ln \gamma/\text{d}c = B$, then eq 4 can be used to calculate the concentration dependence of D. By inverting this procedure, activity coefficients can be estimated from the concentration dependence of D using $\text{d}(D/D^0)/\text{d}c = B$ and eq 4 (Tables 4 and 5). To check the reliability of this procedure, which rests on several assumptions, we estimated B values from our diffusion coefficients and those obtained by Paduano et al.[18] and from the activity data reported by Miyajima et al.[33] As shown in Table 4, the different sets of B values are in acceptable agreement. The decrease in the magnitude of B with increasing temperature may reflect decreasing solute−solute interactions. As shown in Table 5, the activity coefficients calculated from our diffusion coefficients, from the Gouy diffusion data, and from the osmotic data are in good agreement (± 0.1 %).

Temperature Dependence of D. To see if the changes in D with temperature for aqueous β-CD follow the well-known Stokes−Einstein equation:[32]

$$D^0 = k_{\text{B}}T/6\pi\eta^0 a \tag{6}$$

Table 6. Hydrodynamic Radius *a* of *β*-cyclodextrin (from eq 6) at Temperatures from (298.15 to 312.15) K

T K	$10^{16}\,D^0\eta^0/T$ m·s^{-1}·kg·K^{-1}	a nm
274.15	9.49^a	0.772
286.15	9.65^a	0.759
298.15	9.74	0.752
303.15	10.3	0.706
308.15	10.1	0.726
310.15	9.80^a	0.747
312.15	9.72	0.753

[a] Evaluated using Longsworth's D values and η values at 0.00335 mol·dm^{-1}.[17]

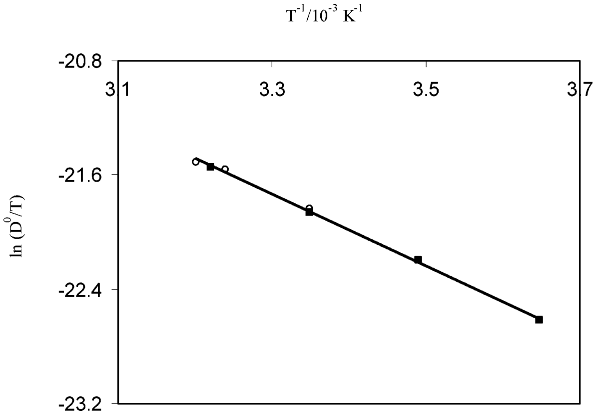

Figure 2. Variation of the logarithm of the limiting diffusion coefficients with the reciprocal temperature: ○, limiting diffusion coefficients (this work); ■, D values at 0.00335 mol·dm^{-3}.[17]

Table 6 gives the values of $D^0\eta^0/T$ and the effective hydrodynamic radius a for infinitely dilute β-CD solutions. k_{B} and η^0 are Boltzmann's constant and the viscosity of pure water at temperature T.[34] The variations in $D^0\eta^0/T$ and a with temperature are, in general, relatively small, < 3 %, which is within the precision of the diffusion measurements. The Stokes−Einstein equation therefore gives a reliable account of the variation in D^0 over the temperature range used in the present study. Moreover, our a values are very similar to those reported by Longsworth[17] (Table 6). In fact, Evans et al.[35] have obtained similar results with other systems.

D values measured at different temperatures can be used to evaluate activation energies E_D for diffusion by using the Eyring relation:

$$E_{\text{D}} = -R\ \text{d}\ln(D^0/T)/\text{d}(1/T) \tag{7}$$

where R is the gas constant. Figure 2 shows that logarithm of the limiting D^0 values plotted against $1/T$ is linear (correlation coefficient = 0.9954). For Figure 2, we used our data and those obtained by Longsworth.[17] The activation energy calculated from the slope of the limiting is 18719 J·mol^{-1}, which is similar in magnitude to values obtained for other aqueous nonelectrolytes of similar size and structure.[17] Moreover, the calculated activation energy is only slightly different from the value $-R[\text{d}\ln(T/\eta^0)/\text{d}(1/T)] = 18644$ J.mol^{-1} suggested by the Stokes−Einstein relation.

Conclusions

Diffusion coefficients measured for aqueous solutions of β-CD provide transport data necessary to model the diffusion in pharmaceutical applications. From the diffusion coefficients, activity coefficients and activation energies have been evaluated that provide a better understanding of the thermodynamic and transport properties of this important carbohydrate in aqueous solutions.

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