

repulsions between the charged carboxylic groups and results in the formation of mixed microdomains.<sup>38b</sup> Effect III should bring about a decrease of  $I_1/I_3$  but would occur only in PSX solutions with  $X \geq 4$ .

Obviously, for PS1 and PS2 the changes of  $I_1/I_3$  are essentially due to effect I. The decreases of  $I_1/I_3$  in the presence of PS1 or PS2 are only slightly less steep than in pure water. A partial recoiling of the copolymer (effect III) would have no impact on the change of  $I_1/I_3$ , as pyrene is not bound by PS1 or PS2 in the whole  $\alpha$  range (see Figures 7 and 9 at very low  $C_S$ ).

On the contrary, PS16 appears to correspond to a situation where partition is unimportant. Indeed the value of  $I_1/I_3$  in the absence of DTAB in Figure 18, at  $C_{PS} = 2 \times 10^{-4}$  M, is close to that found at  $C_{PS} = 10^{-2}$  M.<sup>29</sup> This indicates that pyrene is nearly completely solubilized in the microdomains and that effect I is negligible. The increase of  $I_1/I_3$  with  $C_S$  reflects the progressive enrichment of the microdomains in DTAB (effect II). Recall that in PS16 most repeat units appear to be forming microdomains.<sup>30</sup> Thus effect III discussed above is negligible. Note that the  $I_1/I_3$  vs  $C_S$  plot for PS16 extends to a  $C_S$  value slightly below that where a DTAB-PS16 complex precipitates out.

PS4, PS6, and PS10 correspond to intermediate situations. A minimum is expected to result from the superimposition of effects I and II in the variation of  $I_1/I_3$  with  $C_S$  at high  $C_S$  where effect II becomes predominant. This minimum is observed for the three copolymers. Evidence for the contribution of effect III can be found in the results for PS10. Microdomains are known to exist in PS10 solutions, but at  $\alpha = 1.00$  microdomain formation involves only 15% repeat units.<sup>30</sup> Nevertheless as for PS16 the  $I_1/I_3$  value at  $C_S = 0$  indicates that pyrene is nearly completely solubilized within these domains. Thus effect I contributes only little to the change of  $I_1/I_3$  at low  $C_S$ . This is borne out by the increase of  $I_1/I_3$  with  $C_S$  at very low  $C_S$ . One would therefore expect a monotonous increase of  $I_1/I_3$  from the value of 1.10 in the absence of DTAB to 1.45 at very high  $C_S$ . The leveling off of  $I_1/I_3$  and its subsequent decrease in Figure 18 reveal that a new effect sets in where additional repeat units become involved in microdomains, together with the added DTAB. These additional repeat units can only be those not involved in microdomains at low  $C_S$ . Their

involvement corresponds to a recoiling of PS10 (effect III). When most of the free repeat units have been used up by this process,  $I_1/I_3$  is expected to increase again with  $C_S$ , as observed.

A similar process is likely to occur in PS4 and PS6 solutions. In the latter,  $I_1/I_3$  slowly decreases up to  $C_S = 3 \times 10^{-5}$  M then decreases faster up to  $2 \times 10^{-4}$  M DTAB. These two concentrations probably determine the range where PS6 recoils.

## Conclusions

The above study of the binding of DTAB to a series of homologous but increasingly hydrophobic copolymers has clearly shown that the cooperativity in the binding is modulated by the hydrophobicity of the copolymer. As one goes from hydrophilic to hydrophobic copolymers, the binding constant increases (binding takes place at lower surfactant concentrations) and the binding goes from cooperative to anticooperative. This behavior has been explained in terms of the difference in free energies for the binding of an oncoming surfactant to a copolymer site where it interacts with another bound surfactant and for the binding to a site where it interacts with the copolymer alkyl side chains self-assembled in microdomains. This difference decreases and changes sign as the length of the side chain increases. For the copolymers used, the range of fraction of occupied sites where the binding is cooperative is restricted to about 0.4. In systems where the binding is cooperative, the comparison of the changes of  $I_1/I_3$  with  $C_S$  to the binding isotherms reveals that  $I_1/I_3$  begins to decrease at the concentration  $C^*_S$  where binding starts and that  $I_1/I_3$  becomes nearly constant at  $C^{**}_S$  where binding is no longer cooperative. The concentration  $C^{**}_S$  may be considered as the cmc of DTAB in the presence of PSX.

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## Mutual Diffusion Coefficients in the Water-Rich Region of Water/Phenol Mixtures and Their Relation to Aggregate Formation

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Mutual diffusion coefficients in the one-phase water-rich region of the phase diagram of the phenol/water system (0–10 wt % phenol) were measured using the Taylor dispersion technique, at several temperatures and mole fractions. The values range from 0.71 to  $1.88 \times 10^{-9}$  m<sup>2</sup>/s. In order to obtain evidence about the formation of aggregates of pseudomicelles in this system, as it is invoked in the interpretation of bulk and surface properties, correlation lengths of the concentration fluctuations have been calculated at 328 K using the diffusion data and measured viscosities. They agree with the assumption of aggregate formation in the bulk of the solution at a phenol weight fraction about 7–8 wt %.

### Introduction

Systematic studies of aqueous solutions of amphiphilic molecules with a small aliphatic tail, hence with no clearly defined amphiphathy to be considered as conventional surfactants, are quite interesting. A remarkable example is the 2-butoxyethanol (2BE) + water (W) mixture.<sup>1-5</sup> Here, the concentration dependence

of its surface and bulk properties is very similar to those associated with micellization of normal surfactants. The analogous behavior of 2BE + W with well-established micellar systems has suggested<sup>1-5</sup> that there is some form of organization or aggregation in the bulk of the solution. Hence, the 2BE can be classified as a borderline surfactant.

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**TABLE I: Mutual Diffusion Coefficients ( $D_{12} \times 10^9$ , m<sup>2</sup>/s) for the Phenol + Water Mixtures at Different Temperatures ( $T$ ) and Mole Fractions ( $X_{ph} \times 10^{-3}$ )**

$T$ , K	infinite dilution		2 wt % <sup>a</sup>		4 wt % <sup>a</sup>		6 wt % <sup>a</sup>		8 wt % <sup>a</sup>	
	$X_{ph}$	$D_{12}$	$X_{ph}$	$D_{12}$	$X_{ph}$	$D_{12}$	$X_{ph}$	$D_{12}$	$X_{ph}$	$D_{12}$
296.5	1.5733	0.94	3.9137	0.77	7.9325	0.74				
303.0	1.7587	1.12	3.9095	0.92	7.9339	0.88	12.079	0.71		
308.0	1.6435	1.22	3.9091	1.04	7.9328	0.99				
313.0	1.6977	1.33	3.9104	1.24	7.9327	1.07	12.085	0.85		
318.0	1.7209	1.49	3.9095	1.34	7.9374	1.19				
323.0	1.1858	1.62	3.9117	1.45	7.9389	1.30				
328.0	1.6943	1.75	3.9101	1.65	7.9305	1.50	12.081	1.21	16.394	0.85
343.0	1.4364	1.88	3.9113	1.75						

<sup>a</sup> Approximate concentration in phenol weight fraction.

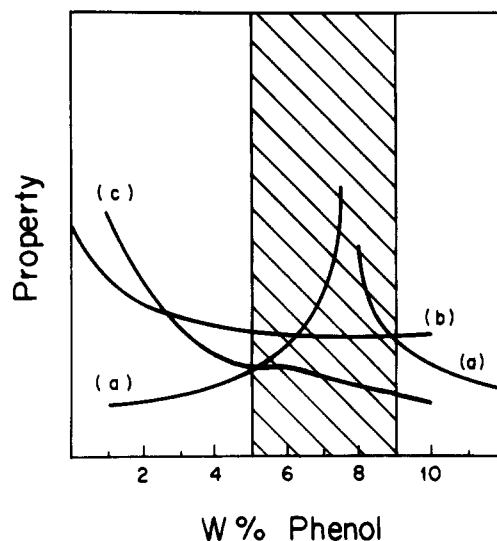
Another system that bears a strong resemblance to the system just mentioned above is phenol + W, although the concentration dependence of its properties is not so enhanced, so this system can be classified even below the surfactant borderline typified by the 2BE + W system. J. Gracia et al.<sup>6</sup> undertook the study of the phenol + W mixture as an example of a mixture that combines aggregate formation at low concentrations followed by an immiscibility region at higher phenol concentrations. These workers studied the interrelations between bulk and interfacial phase behavior in this system, and they found two different regimes in the one-phase water-rich region of the phase diagram located below the critical consolute point. The first regime begins with pure water, and as the phenol concentration is increased, a monolayer of phenol at the water/air interface is formed. In the second regime, they assumed that some form of phenol aggregates occur at a concentration around 7–8 wt % phenol. They based their conclusions on the behavior of surface tension, conductivity, and gradual abatement of foam stability in the mixture, as the phenol concentration is increased. Also, they examined the temperature dependence of the angle of contact in the two-liquid region, and all the results were consistent with the idea of aggregate formation.

In Figure 1, the narrow area where the aggregate formation is supposed to take place is presented, with the concentration dependence of the properties used in ref 2 to support the aggregate formation assumption.

The purpose of this paper is to present mutual diffusion coefficient (MDC) data measured at several temperatures (296.5–333 K) and mole fractions (ca. infinite dilution to 16.3937  $\times 10^{-3}$ ), in the water-rich mixtures of phenol + W, using the Taylor dispersion technique. This data can give more information that supports the assumption about the formation of aggregates or pseudomicelles in solution, improving our understanding in this system.

### Instrument Design and Operation

The Taylor dispersion technique<sup>11</sup> is based on the dispersion of an injected binary-mixture pulse in a laminar flowing stream of the same mixture at slightly different composition, by the joint action of convection and molecular diffusion. Under proper conditions, the pulse concentration profile will eventually become normal, and the center of gravity of the profile will move with the mean velocity of the laminar flow. The theory for the development of ideal equipment to measure MDC's using this method is reviewed by Alizadeh et al.<sup>12</sup> In addition to this, they presented a detailed criteria for the design of a practical apparatus for measuring the MDC. In the present paper, we followed that work in order to design a measuring instrument, and the details of our instrument were presented in ref 1.



**Figure 1.** Concentration dependence (schematic) at 333 K for the phenol + water system: foam column height (a), surface tension (b), and molal conductivity (c). All from ref 6.

Alizadeh et al.<sup>12</sup> found expressions for the first ( $\bar{i}$ ) and second ( $\sigma^2$ ) temporal moments of the distribution of the dispersed pulse for an ideal instrument and a set of corrections for this ideal instrument in order to include practical limitations. They found that the ideal moments have to be corrected according to

$$\bar{i} = \bar{i}_{\text{exp}} + \sum \delta \bar{i}_i \quad \text{and} \quad \sigma^2 = \sigma_{\text{exp}}^2 + \sum \delta \sigma_i^2$$

where  $\bar{i}_{\text{exp}}$  and  $\sigma_{\text{exp}}^2$  denote the experimentally determined moments and  $\delta \bar{i}_i$  and  $\delta \sigma_i^2$  are the corrections applied to them. For details see refs 12 and 1.

The values of  $\bar{i}_{\text{exp}}$  and  $\sigma_{\text{exp}}^2$  were determined with a nonlinear fitting program of the digitized numbers coming from the differential refractometer, in the region where it has a linear response to the concentration difference between the cells. This task was done with a data acquisition board (Lab Master DMA, Scientific Solutions, Inc.) and a Printaform PC. The use of an interface and nonlinear fitting was unavoidable, since as one approaches the coexistence line the Gaussian curve coming from the refractometer is not very sharp, due to fluctuations in the mixture.

### Experimental Section

Phenol of 99% purity supplied by the J. T. Baker Co. was further purified by sublimation. Analysis by gas chromatography gave a value of 99.998% purity. The other fluid, water, was doubly distilled water. The binary mixtures were prepared with an estimated error in the quoted mole fractions to be less than  $1 \times 10^{-4}$ . Special care was taken in order to degas the binary mixtures in an ultrasonic cleaner without inducing concentration changes.

Measurements of the MDC's were carried out in mixtures of phenol and water at different temperatures in the one-phase water-rich region. The results are shown in Table I. It is difficult to estimate the precision involved in the determination of MDC's by the technique described above. Hence, we followed the common practice of employing the reproducibility of the results of a series

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of experiments, under nominally identical experimental conditions, as a measure of the precision of the determinations. We determined the values of  $\bar{t}_{\text{exp}}$  and  $\sigma_{\text{exp}}^2$  as mentioned above, and after doing the corrections discussed in refs 12 and 1, we obtained a precision of 2.5%.

The ranges in concentration and temperature where MDC's were measured were limited by experimental drawbacks related to the features of the phenol + W system. One problem was the bubble formation upon sample injection within the six-port Valco high-pressure liquid chromatograph injection valve at temperatures above 328 K and for concentrations above 3 wt % phenol. This is probably due to instantaneous increments of pressure in the sample pulse during the process of charging and injecting, then promoting the  $L_1$ -V phase transition. The limit for concentrations above 8 wt % phenol for measurements at 328 K is imposed by the coexistence line  $L_1$ - $L_2$ -V, ca. 11-12 wt % phenol, as given in ref 10. However, in our unsuccessful attempt at 10 wt %, it was impossible to prevent the  $L_1$ - $L_2$ -V transition. Given the vertical form of the coexistence curve at those concentrations, small changes in temperature promote the phase transition, hence our failure may be due to thermal instabilities in the connections from the diffusion tube to the detector. Another possibility may be that the reported phase diagram<sup>10</sup> is not precise enough there. It is likely that both phenomena contributed to the onset of phase separation.

For a series of phenol + W mixtures, kinematic viscosities were measured at 328 K with a Canon-Fenske viscosimeter. These kinematic viscosities were converted into dynamic viscosities through the measurement of densities with a precision pycnometer, at the concentrations where MDC's were measured.

### Results and Discussion

Table I reports the MDC's for the phenol + W mixtures at several temperatures and phenol mole fractions ( $X_{\text{ph}}$ ). The present work as far as we know gives the first picture of the MDC's behavior in the one-phase water-rich region of this system. There is only a reported value at infinite dilution (298 K) given by Sharma and Kalia<sup>13</sup> using an estimative technique. Our results are higher than this value.

In the concentration interval displayed in Table I at 328 K, the MDC behavior of phenol + water mixtures is not clearly characterized by the presence of two distinctly different regimes, due to aggregate formation, as can be observed for the case of a borderline surfactant such as 2BE.<sup>1</sup> If there is a passage from one regime to the other, then it seems to occur smoothly over the phenol concentration region where bulk and surface properties display an important change (dashed area of Figure 1). This may be consistent with the fact that aggregate formation is not so important here as in the 2BE case. The enhancement of other properties is also diminished with respect to the 2BE case; hence diffusion could not be an exception. Another point is that the onset of phase separation does not permit study of a sufficiently wide concentration range to determine whether the MDC's eventually become constant as in the 2BE case.

The decrease of the MDC's is not by itself strong evidence for an increasing number of phenol-water interactions in solution and therefore for some kind of organization present in the solution like aggregates or pseudomicelles. The experimental information<sup>6,7</sup> about this system can be summarized as follows: There is a first regime that corresponds to the build up of a monolayer of phenol at the water/air interface. The development of the dilute adsorbed layer progressively decreases the surface tension and increases the foam stability. The molar conductivity of the bulk solution also decreases. The second regime is characterized by the constancy of the surface tension, bulk conductivity, and a reversal in the behavior of foam stability, i.e. foaminess decreasing with increasing phenol concentration. In order to explain the origin of this behavior, two distinct processes corresponding to different, although related, physical situations must be considered. In the first one, an event occurring mainly at the water-rich/air interface gives

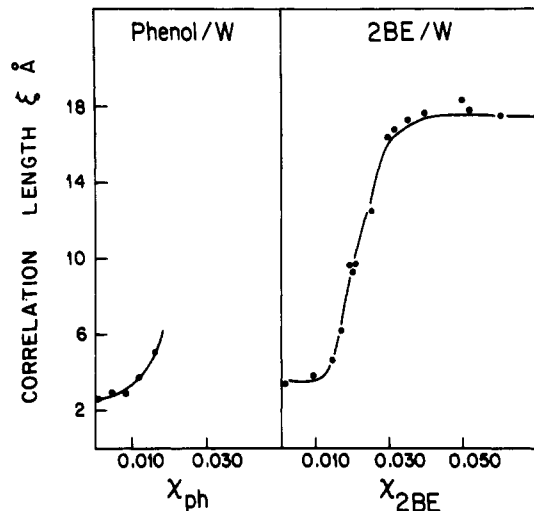


Figure 2. Correlation length of concentration fluctuations vs the phenol mole fraction at 328 K and vs the 2BE mole fraction at 298 K (from ref 1). The vertical line is to aid visualization only.

rise to the described behavior. Here, a possibility is prewetting.<sup>8</sup> In the second one, a process occurs mainly at the bulk of the solution that modifies the behavior of the water-rich/air interface, through a competition between adsorption and segregation from the interface. Such a process could be aggregation. The first situation can be excluded since no partial-to-perfect wetting transition takes place;<sup>6-8</sup> hence no prewetting is expected. On the other hand, at the coexistence line, the temperature dependence of the contact angle between the two liquids and their own vapor decreases from values around  $43^\circ$  when  $T = 293$  K to zero at  $T_c$ , with a clear transition around 311 K.<sup>6</sup> The composition attained in the water-rich liquid at this transition temperature gives again 7-8 wt %.<sup>6</sup> This can be attributed to a competing process in the bulk of the solution that does not permit it to reach the perfect wetting. The assumption that some form of phenol aggregation occurs could explain not only the temperature dependence of the angle of contact but also the behavior of surface tension and of conductivity and the gradual abatement of foam stability. In the same way, as organization in the mixture increases, the mobility of phenol is reduced. This could explain the decrease of the MDC. As far as we know, there are no measurements of apparent molar heat capacities or partial molar enthalpies on this system, except at infinite dilution,<sup>9</sup> to give stronger support to the aggregate formation assumption.

The MDC's given in Table I allow the calculation of the correlation length<sup>1,5,14</sup> ( $\xi$ ) of the concentration fluctuations in these mixtures. This quantity gives us information about the processes occurring in the bulk of the solution as the phenol concentration is increased. Assuming that velocity fluctuations are statistically independent of concentration fluctuations,<sup>14</sup>  $\xi$  is related to the MDC through<sup>15</sup>

$$\xi = kT/6\pi\eta D_{12} \quad (1)$$

In Figure 2, calculated values of  $\xi$  through the use of eq 1, the MDC's, and the shear viscosity data are displayed as a function of the concentration, at 328 K. As a point of comparison, the correlation length of the 2BE + W system in the equivalent region of aggregate formation is presented too. At the very low phenol concentrations, the value of the correlation length (ca. 2.71 Å)

(14) The  $\xi$  values calculated from eq 1 may be different from the correlation length usually defined as

$$\xi = \frac{1}{6} \int r^2 \phi(r) d^3r / \int \phi(r) d^3r, \quad \phi = \langle \Delta x(o) \Delta x(r) \rangle$$

for solutions far from the critical point. But, the concentration dependence of this "apparent" correlation length has been useful in several studies of aggregation. See: Euliss, G. W.; Sorensen, C. M. *J. Chem. Phys.* **1984**, *80*, 4767. See also refs 5 and 1.

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is practically the same as the radius  $r$  for the phenol molecule in solution (2.77 Å). This value can be obtained using the virial coefficient data given by Matteoll and Leporl<sup>16</sup> for the virial expansion of the osmotic pressure in this system. When the mole fraction of phenol is ca 0.008,  $\xi$  starts to increase rapidly, until it has been doubled at a phenol concentration of ca. 0.016. The magnitude of this correlation length when compared to the molecular radius indicates a slight association between phenol and water molecules. This agrees with the aggregate formation assumption at those concentrations.

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In conclusion, the whole picture of the aggregate formation as it is invoked in the interpretation of bulk and surface properties in phenol + W agrees with the information extracted from the MDC's presented in this paper. In addition, it is important to note that MDC measurements are sensible enough to give part of the picture of the organization in the bulk of the solution in this kind of borderline systems.

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## Extension of Kohler-Strnad Viscosity Model for Ionic Rod-Shaped Micelles of Low Axial Ratio. Application to Sodium Deoxycholate Micelles

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An extension of Kohler-Strnad model (*J. Phys. Chem.* 1990, 94, 7628) for the evaluation of viscosity measurements of ionic surfactants forming rod-shaped micelles of low axial ratio is proposed. The method is applied to sodium deoxycholate micelles in aqueous solution of sodium chloride, chlorate, and salicylate at different electrolyte and surfactant concentrations. It has been observed that the difference between the fractional counterion association at the two terminal hemispherical caps and at the cylindrical part of the rod micelles, and the standard work for the reversible transfer of a surfactant ion between both parts, depend on the anion of the electrolyte used.

### Introduction

Viscosity measurements of bile salts solutions have been published in a number of unrelated papers by several authors.<sup>1-11</sup> In general, all authors have found that viscosity depends on pH, electrolyte concentration (normally NaCl), temperature, buffer used to keep the pH constant, and bile salt concentration. At pH values close to neutrality several authors have found that the viscosity depends on the elapsed time from the preparation of solution,<sup>1,2,6</sup> but it becomes constant after a time (typically 1 hour). This behavior was attributed to a thixotropic effect (due to the mechanical influence of the viscometer)<sup>1</sup> and was interpreted in terms of a polymer-like aggregation and degradation of the aggregates as a consequence of the shearing stress.<sup>6</sup>

Also common is the observation of different regions for the dependence of viscosity with bile salt concentration. For NaDC, NaC and NaDHC (see Glossary for abbreviations) at the lowest bile salt concentrations, Fontell<sup>3</sup> observes a behavior of the viscosity typical for electrolyte solutions; since at intermediate concen-

trations the viscosity obeys Vand's<sup>12</sup> equation, he accepts that the micelles are spherical in shape and highly hydrated. Finally, this author deduces that micelles are anisometric at the highest concentrations of bile salts. Similarly, Vochten and Joos<sup>2</sup> observed the existence of a second cmc (which is dependent on NaCl concentration) and Sesta et al.<sup>8</sup> observed a break point in the expected straight line according to Vand's<sup>12</sup> equation which was interpreted in terms of a primary superaggregation. Güveli<sup>9</sup> has evaluated the viscosity for NaDC from the apparent specific volume suggesting the existence of secondary aggregates. A similar conclusion was obtained by the same author<sup>10</sup> for NaTDC from his evaluation of Schurtenberger et al.<sup>13</sup> results of light scattering. He also concluded that the secondary micelles (formed at [NaTDC] > 40 g/dm<sup>3</sup> and [NaCl] = 0.8 M) are rodlike in shape and flexible.

Other viscosity measurements imply the study of mixed micelles of bile salts and tetralkylammonium bromides carried out by Barry and Gray,<sup>5</sup> who found a minimum in the viscosity for an equimolecular mixture of both surfactants, or the studies by Sesta et al.<sup>8</sup> in water solutions of urea.

It is clear from this short review on viscosity for bile salts solutions that although important conclusions have been obtained, most of the information is essentially qualitative or semiquantitative in nature. This in fact reflects the state of art in the theoretical background necessary for interpretation of viscosity data. Recently, Kohler and Strnad<sup>14</sup> have published a new evaluation of viscosity measurements of dilute solutions of ionic surfactants (for references of previous models see the literature

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