The influence of hydrophobic amines on hydrolysis of bis(*p*-nitrophenyl) methylphosphonate in micellar solutions of cetylpyridinium bromide

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The kinetics of hydrolysis of bis(*p*-nitrophenyl) methylphosphonate in the presence of primary aliphatic amines in aqueous micellar solutions of cetylpyridinium bromide was studied. The reaction proceeds *via* two routes, alkaline hydrolysis and amine-catalyzed hydrolysis according to the general basic catalysis mechanism. The contributions of these routes and the catalytic effect of micelles depend on the hydrophobicity of the amines. The formation of different types of micelles was found, and their characteristic parameters were determined by tensiometry and high-resolution ^{1}H NMR spectroscopy with a magnetic field pulse gradient.

Key words: kinetics, hydrolysis, bis(*p*-nitrophenyl) methylphosphonate, amines, micelles, cetylpyridinium bromide, ^{1}H NMR spectroscopy, diffusion.

Micellar catalysis is a nontraditional method of affecting the reactivity of organic compounds.[1,2] Elucidation of the factors that determine the efficiency of micellar catalysis is a topical task. The line of investigation of micelle formation and micellar catalysis is tightly connected with the problems of enzymology, because most processes involving enzymes occur in highly organized systems, namely, cell membranes and subcellular species. Micellar solutions can serve as convenient models for the investigation of *in vitro* processes occurring in a living cell.[3]

Functionalization of micelles of cationic surfactants by incorporation of long-chain amines (*i.e.*, the formation of mixed aggregates bearing a fixed active site responding to the variation of the medium pH and the component ratio) can result in the design of biomimetic systems. These systems exhibit catalytic activity in nucleophilic cleavage processes, in particular, in the transfer of acyl and phosphoryl groups.

This work is a continuation of our study dealing with the catalytic properties of micellar systems consisting of a surfactant and a primary aliphatic amine.[4,5] It is aimed at elucidating the relationship between the structures of microaggregates and their influence on the rate and mechanism of cleavage of ester bonds. For this purpose, using bis(*p*-nitrophenyl) methylphosphonate (**1**) as the substrate, we studied the kinetics of hydrolysis of esters of tetracoordinated phosphorus in the presence of primary amines with nonbranched chains in aqueous micellar solutions of cetylpyridinium bromide (CPB). A number of characteristic parameters of the system confirming the formation of micellar aggregates of various types were determined by independent physical methods (tensiometry and high-resolution ^{1}H NMR spectroscopy with a magnetic field pulse gradient).

Experimental

The solvents and amines were purified by standard procedures. CPB samples were twice reprecipitated with ether from ethanol. Phosphonate **1** was synthesized and purified by a procedure reported previously.[6]

The formation of the *O*-(*p*-nitrophenyl) methylphosphonic acid monoanion upon hydrolysis of compound **1** in solutions of CPB in the presence of decylamine is proved by the fact that its chemical shift in the ^{31}P NMR spectra (22.3 ppm) coincides with the chemical shift of the product of alkaline hydrolysis of this substrate (Bruker MSL operating at 161.97 MHz). The formation of this monoanion was also detected by potentiometric titration of the reaction mixture in experiments with equimolar amounts of the amine and the substrate using the material balance equations.

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The reaction kinetics was studied by spectrophotometry on a Specord UV-VIS instrument at 25 °C based on the variation of the optical density of solutions at 400 nm (the formation of the *p*-nitrophenoxide anion). The initial substrate concentration was $5 \cdot 10^{-5}$ mol L^{-1} and the degree of conversion was >90%. The required pH values in the systems were attained by adding hydrochloric acid and determined using a pH-340 instrument.

The observed pseudo-first-order rate constants (k_{obs}) were determined from the plot $\log(A_\infty - A_\tau) = -0.434\, k_{obs} + \text{const}$, where A_τ and A_∞ are the optical densities of the solutions at time τ and after completion of the reaction, respectively. The k_{obs} values were calculated by the least-squares methods. The second-order rate constants (k_2) for *n*-octylamine in the absence of CPB were calculated from the linear section of the plot for k_{obs} *vs* concentration of the amine (C_{am}) using the equation $k_2 = (k_{obs} - k_0)/C_{am} \cdot \alpha$, where k_0 is the rate constant for alkaline hydrolysis of the substrate, found as the intercept on the ordinate axis in the plot of this dependence at a given pH, and α is the fraction of the neutral (reactive) form of the amine under the conditions of the kinetic experiment. The α value was found by potentiometric titration.

The binding constants of the substrate (K_{bond}), the critical micelle concentrations (CMC), and the rate constants for the micellar phase (k_m) were calculated from the equation for the pseudophase model of micellar catalysis[2]

$$k_{obs} = \frac{k_m K_{bond} C_{det} + k_0}{1 + K_{bond} C_{det}}, \qquad (1)$$

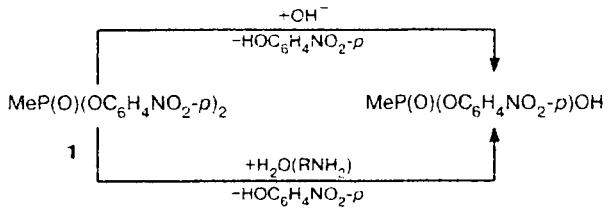
where C_{det} is the surfactant concentration corrected for the CMC and k_0 is the reaction rate constant in the absence of a surfactant.

The surface tension of solutions (σ) was determined by the ring detachment method using a du Noüy tensiometer at 20 °C.

The ^{1}H NMR spectra were recorded on a Tesla BS 576A high-resolution spectrometer at a proton resonance frequency of 100 MHz. The spectrometer was equipped with a homemade magnetic field pulse gradient unit, which enabled creation of a field gradient of up to 50 G cm^{-1}. Specific features of using this equipment and some technical approaches to the study of microemulsions were described previously.[7,8] The proton spectra were recorded and the diffusion coefficients were measured at 30 °C. Water consisting of 95% (v/v) D_2O and 5% (v/v) doubly distilled H_2O was used as the bulk phase of the micellar solutions.

Results and Discussion

The major processes involved in the cleavage of esters of phosphorus-containing acids in amine-containing aqueous solutions are alkaline hydrolysis and hydrolysis catalyzed by amines according to a general basic catalysis mechanism.[9,10]



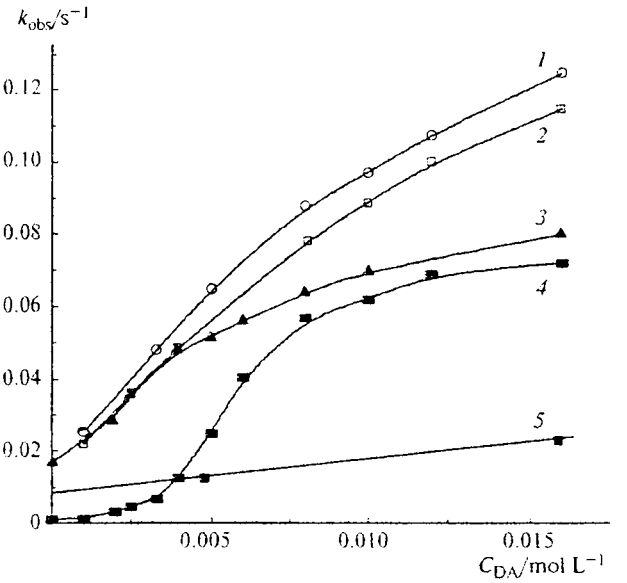


Fig. 1. Observed rate constant for the hydrolysis (k_{obs}) of ester **1** *vs* concentration of *n*-decylamine (25 °C): C_{CPB} = 0.05 mol L^{-1} at variable pH (*1*); 0.02 mol L^{-1} at variable pH (*2*); 0.015 mol L^{-1} at pH 9.4 (*3*); 0 at pH 9.4 (*4*); *n*-octylamine: 0 at pH 10.4 (*5*).

The contribution of aminolysis in the case of primary amines is negligibly small.[10] The efficiency of general basic catalysis in aqueous solutions, according to the Brønsted equation, is proportional to the nucleophile basicity. The observed rate constant (k_{obs}) obeys a linear dependence on the concentrations of hydrophilic and short-chain amines over a broad range of amine concentrations.[10]

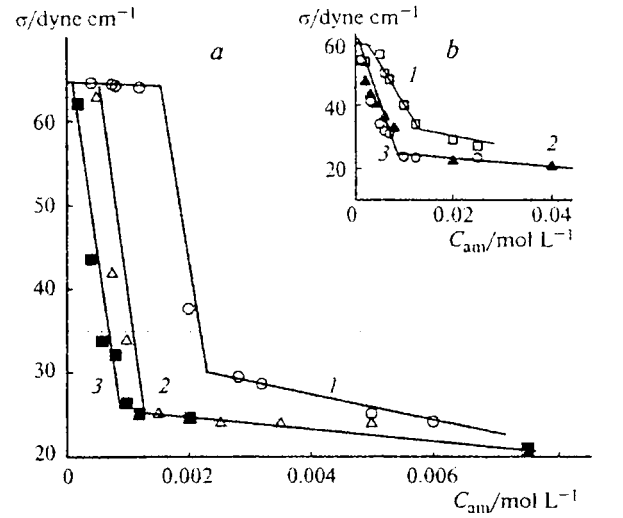


Fig. 2. Surface tension of solutions (σ) of amines *vs* concentration (C_{am}) for various degrees of protonation (α) (20 °C). *a*, *n*-Decylamine at α (%) = 25 (*1*), 50 (*2*), 100 (*3*); *b*, *n*-octylamine at α (%) = 25 (*1*), 50 (*2*), 100 (*3*).

Table 1. CMC values for primary aliphatic amines in aqueous solutions at different degrees of protonation (20 °C)

Amine	Degree of protonation (%)	CMC/mol L^{-1}
n-Decylamine	0	0.00095
	25	0.0010
	50	0.0013
	75	0.0027
n-Octylamine	0	0.008
	50	0.010
	75	0.0135

In the case of hydrophobic amines disposed to aggregation (for example, *n*-decylamine), the process can be complicated due to the formation of micelles, which influence the rate of hydrolysis. The pattern of variation of the observed rate constant for hydrolysis of ester **1** as a function of *n*-decylamine concentration (C_{DA}) reflects the changes in the system under study (Fig. 1). The initial linear section corresponds to the premicellar region. The change in the curvature of the $k_{obs} = f(C_{DA})$ plot is due to the micelle formation process; flattening-out of the curve suggests that the micelles become saturated with the substrate molecules. It should be noted that *n*-octylamine is not prone to association in the same concentration range; the $k_{obs} = f(C_{am})$ plot is linear up to 0.015 mol L^{-1} at pH 9.4—10.4 (see, *e.g.*, Fig. 1, curve *5*) and the bimolecular rate constant (k_2) is 0.7 L mol^{-1} s^{-1}.

The fact of micelle formation by hydrophobic amines was confirmed by measurement of the surface tension in their aqueous solutions (Fig. 2). The CMC values for *n*-decylamine are approximately an order of magnitude lower than those for *n*-octylamine. In addition, they depend substantially on the degree of neutralization of the amine (Table 1): protonated amines are least prone to form micellar aggregates, while nonprotonated amines exhibit the highest tendency for aggregation. Apparently, the micelle formation is hampered by electrostatic repulsion between the positively charged and slightly shielded ammonium head groups. The CMC values obtained confirm that the conditions of kinetic experiments in the systems with *n*-octylamine correspond to the premicelle region, while in the case of *n*-decylamine, they are also extended to the region of micelle formation, which influences the pattern of variation of k_{obs} as a function of the amine concentration (see Fig. 1).

In the cleavage of phosphonate **1** in aqueous solutions containing CPB and an amine, acceleration of both alkaline hydrolysis and the process catalyzed by the general basic mechanism should be expected. This is, first of all, due to the fact that the substrate and an amine are solubilized by micelles due to hydrophobic interactions. In addition, OH^- ions are accumulated on the positively charged micellar surface due to electrostatic attraction. The influence of CPB on the rate of hydrolysis of **1** following an increase in the *n*-decylamine concentration is due to both a change in the pH of the solution (from ~9 to 11) and the increase in the concentration of the nucleophile, which activates the water molecules participating in the cleavage of ester bonds (see Fig. 1, curves *1* and *2*). In order to evaluate the influence of the amine concentration on the rate of the process, the kinetic experiments were carried out at a constant pH value (see Fig. 1, curve *3*). In this case, the acidic properties of compounds are enhanced in cationic micelles, due to their selective solubilizing capacity with respect to acidic or basic species. The apparent change in the pK can contribute significantly to the micellar kinetic effect by changing the concentration of the reactive form of the reactant. CPB micelles increase the proportions of neutral amines,[4,5] which results in higher activities of nucleophiles in the reactions under study.

Table 2. Micellar parameters of hydrolysis of **1** in the CPB medium in the presence of primary *n*-alkylamines (25 °C)

Nucleophile	C_{am}/mol L^{-1}	pH	K_{bond} /L mol^{-1}	CMC[a] /mol L^{-1}	k_m/s^{-1}	k_m/k_0[b]
n-Butylamine	0.005	10.4	330	0.001	0.185	30
	0.01	10.4	300	0.0011	0.197	32
n-Octylamine	0.0025	9.4	195	0.0001	0.0306	77
	0.005	9.4	267	0.0002	0.0329	82
	0.01	9.4	364	0.0001	0.0385	96
	0.02	9.4	350	0.0001	0.0407	98
	0.005	10.4	310	0.0003	0.20	35
n-Decylamine[c]	0.0010	9.4	80	0.0001	0.060	150
	0.0018	9.4	92	0.0001	0.0826	206
	0.0025	9.4	87	0.00007	0.121	300
NaOH		10.4	330	0.0006	0.20	36

[a] For CPB, CMC = 0.0006 mol L^{-1}.[11] [b] k_0 is the rate constant for alkaline hydrolysis at a given pH without a surfactant. [c] In the absence of CPB at pH 9.4 for *n*-decylamine, K_{bond} = 46 mol L^{-1}, CMC = 0.001 mol L^{-1}, k_m = 0.21 s^{-1}.

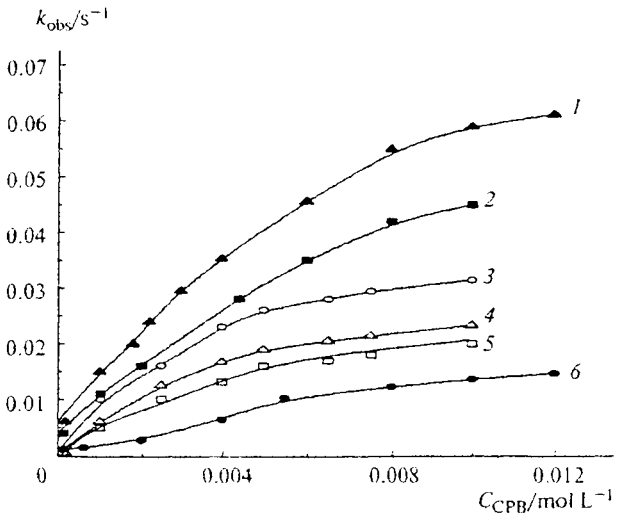


Fig. 3. Observed rate constant for the hydrolysis (k_{obs}) of ester **1** in the presence of *n*-alkylamines *vs* CPB concentration (pH 9.4, 25 °C): decylamine, C_{DA}/mol L^{-1} = 0.0025 (*1*), 0.0018 (*2*), 0.001 (*6*); octylamine, C_{OA}/mol L^{-1} = 0.020 (*3*), 0.005 (*4*), 0.0025 (*5*).

To elucidate the influence of amines on the hydrolytic cleavage of **1** in aqueous micellar solutions of CPB, the kinetics of this process was studied in the presence of amines of different hydrophobicities. The dependences of k_{obs} on the surfactant concentration for hydrolysis of **1** in the presence of *n*-butylamine and *n*-octylamine at pH 10.4 or in dilute solutions of NaOH are virtually identical and barely depend on the amine concentration, as shown by quantitative processing of the $k_{obs} = f(C_{CPB})$ plots in terms of Eq. (1) (Table 2). This implies apparently that micelle-catalyzed alkaline hydrolysis predominates under these conditions and amine acts only as a buffering agent, which ensures sufficiently high pH values.

The role of alkaline hydrolysis can be diminished and the contribution of the amine-catalyzed reaction following a general basic catalysis pattern can be elucidated if the concentration of the OH^- ions decreases. However, at lower pH, the content of neutral *n*-butylamine sharply decreases (at pH 9.4 and C_{CPB} = 0.01 mol L^{-1}, α = 0.04), which precludes investigation of this reaction under the given conditions.

In the case of more hydrophobic *n*-octylamine, pK_a decreases[5] in micellar solutions of CPB and the fraction of the neutral amine proves to be rather high (at pH 9.4 and C_{CPB} = 0.01 mol L^{-1}, $\alpha \approx 0.15$ to 0.2). The $k_{obs} = f(C_{CPB})$ plots for the hydrolysis of **1** in the presence of *n*-octylamine at pH 9.4 change slightly upon variation of the content of amine in the reaction medium (Fig. 3). This suggests that in this case, too, alkaline hydrolysis is the predominant route. Meanwhile, the rate of cleavage of **1** in micellar solutions of CPB in the presence of *n*-decylamine depends appreciably on the amine concentration (see Fig. 3). Thus, *n*-decylamine seems to act as a basic catalyst.

The results of processing of the kinetic data (see Fig. 3) in terms of Eq. (1) show that the addition of hydrophobic amines facilitates the micelle formation of CPB and thus decreases the CMC (see Table 2), which is a typical indication of the formation of mixed micelles.[1] *n*-Butylamine, which is not solubilized by CPB micelles, somewhat increases the CMC by exerting a slight negative influence on micelle formation.

As the length of the alkyl chain in the amine increases, binding of ester **1** by CPB micelles somewhat diminishes due to the change in the properties of the mixed micellar aggregates. Despite the low K_{bond} values for the substrate in the presence of *n*-decylamine, the efficiency of micellar catalysis (k_m/k_0) is higher (~4 times) than in the case of *n*-octylamine (see Table 2). In micellar solutions of CPB, alkaline hydrolysis can occur in parallel with the amine-catalyzed process corresponding to general basic catalysis; therefore, the k_m values are effective constants. The close k_m values found for *n*-butyl- and *n*-octylamine at pH 10.4 indicate that the processes follow virtually the same pathway, *i.e.*, micellar-catalyzed alkaline hydrolysis of **1**.

The specific kinetic behavior of *n*-decylamine in the presence of CPB can be explained by assuming that the properties of aggregates change upon the formation of mixed micelles. To confirm the formation of these species by an independent method, high-resolution ^1H NMR spectroscopy with a magnetic field pulse gradient was used; this allows one not only to record the NMR spectra of the chemical components of multicomponent systems such as microemulsions but also to study the diffusion decay of individual lines and to determine the diffusion coefficients of the components of the mixture.[7,13]

The NMR spectrum of an aqueous solution of CPB (C_{CPB} = 0.05 mol L^{-1}) both in the absence and in the presence of *n*-decylamine up to a concentration of 0.032 mol L^{-1} contains separate lines due to protons of the Me (δ 0.78) and $(-CH_2-)_n$ groups (δ 1.2), signals for aromatic protons (δ 8.24, 8.73, and 9.19), and a signal due to water protons (δ 4.78). The spectrum also exhibits weak lines due to methylene protons in the α-position relative to the Me group (δ 2.08) and methylene protons at the N atom (δ 4.73).

The diffusion coefficients of the individual components of the system (D_i) were determined using the signals of the methylene protons and the water protons. These signals are sufficiently intense and well resolved, unlike the relatively weak signals of the methyl and aromatic protons or the methylene protons of the CH_2—N group, which overlap with the water proton signal.

Although the signals overlap somewhat, measurement of the diffusion coefficient of water (D_w) presents no difficulty because the signal for the water protons is much more intense than that of the CH_2—N group of CPB; in addition, D_w is two orders of magnitude greater than the diffusion coefficient of CPB, which is found from the line with δ 1.2. This line contains contribu-

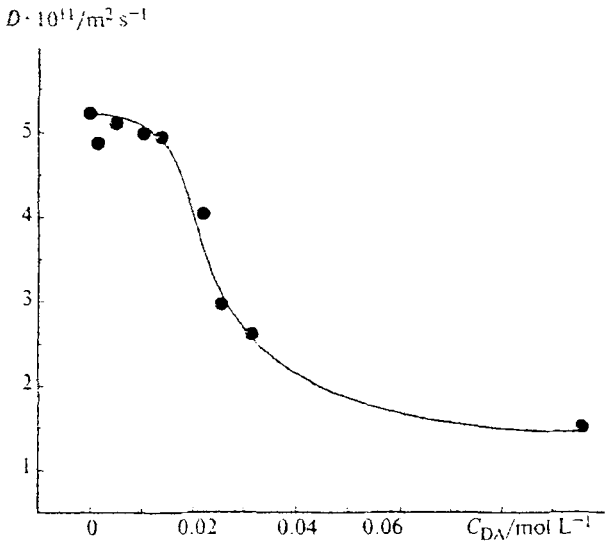


Fig. 4. Observed self-diffusion coefficient (D) in mixed CPB—*n*-decylamine micelles *vs* concentration of the amine (C_{CPB} = 0.05 mol L^{-1}, 30 °C).

tions of the methylene protons of CPB and *n*-decylamine, which cannot be separated because the line shows one-exponential diffusion decay. The fact that this decay cannot be resolved into two exponents indicates that the diffusion coefficients of CPB and *n*-decylamine are close or identical. This implies diffusion of both components within a single structural aggregate, which may be due to incorporation of *n*-decylamine molecules into CPB micelles.

The diffusion coefficient of water depends slightly on the content of *n*-decylamine in the system. In the absence of the amine, $D_w = 2.1 \cdot 10^{-9}$ $m^2 s^{-1}$, while at the maximum content of the amine it decreases to $1.9 \cdot 10^{-9}$ $m^2 s^{-1}$.

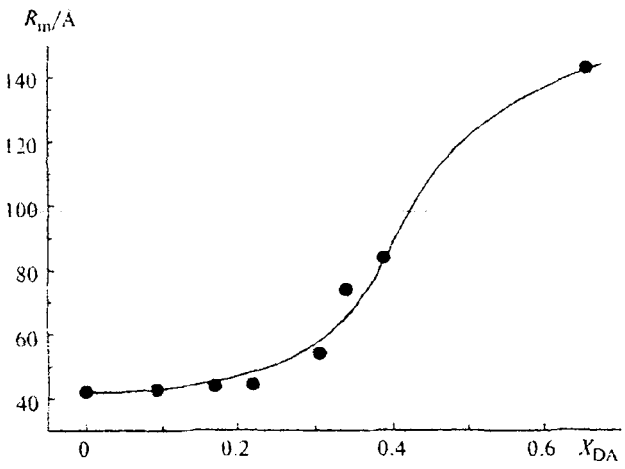


Fig. 5. Effective radius (R) of mixed micelles *vs* molar fraction (X) of the amine (X_{DA}+ X_{CPB}= 1, 30 °C).

The diffusion coefficient of CPB is $5.23 \cdot 10^{-11}$ $m^2 s^{-1}$. Since we are dealing with a dilute system, the Stokes—Einstein equation can be used to determine the effective radius (R) of the kinetic species in which CPB diffuses. With allowance for direct collisions of species with one another, we obtain the following equation[14]

$$R = (1 - 2\phi)kT/6\pi\eta D, \qquad (2)$$

where k is the Boltzmann constant, T is the absolute temperature, η is the solvent viscosity, ϕ is the volume fraction of the solute, and D is the diffusion coefficient. The viscosity of D_2O at 30 °C, equal to 1.033 cP, was used in the calculations. Calculation using Eq. (2) showed that in the absence of *n*-decylamine, CPB diffuses within aggregates with an effective hydrodynamic radius of 42 Å.

When *n*-decylamine is introduced in a micellar solution of CPB, the diffusion coefficient of micelles decreases. Figure 4 shows the variation of the micelle diffusion coefficient as a function of the concentration of *n*-decylamine (the variation of the catalytic properties of this system is shown in Fig. 1, curve *1*). It can be seen that, as the content of *n*-decylamine increases, D decreases first smoothly and then sharply. Figure 5 presents the dependence of the micelle radius on the CPB : *n*-decylamine ratio at a constant CPB concentration, equal to 0.05 mol L^{-1}. The micelle radius sharply increases when the system contains one *n*-decylamine molecule per three or fewer CPB molecules. In a micellar solution of CPB saturated with *n*-decylamine, in which we were able to carry out diffusion measurements, the micelle radius is twice as large as that in the initial system.

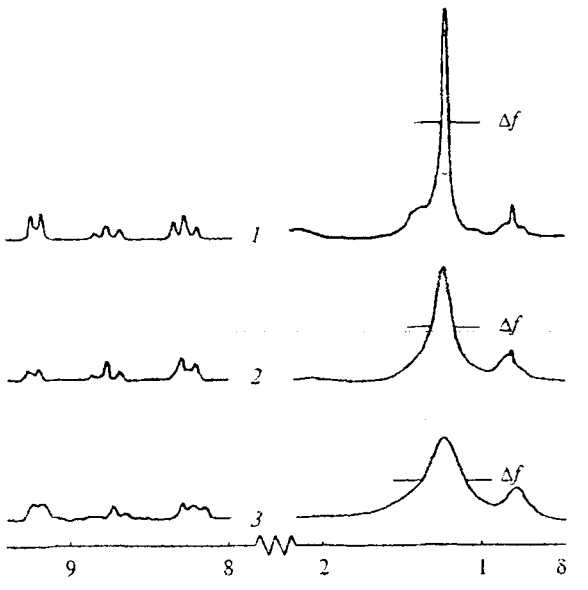


Fig. 6. Width of the $(-CH_2-)_n$ signal (δ 1.2) *vs* composition of mixed micelles (C_{CPB} = 0.05 mol L^{-1}): in the absence of *n*-decylamine (*1*) and with C_{DA}/mol L^{-1} = 0.025 (*2*) and 0.05 (*3*).

Yet another evidence for the formation of mixed micelles from CPB and *n*-decylamine is provided by NMR spectra. As the content of *n*-decylamine increases, all signals except for the signal of water are broadened (Fig. 6). The increase in the line width, *i.e.*, a decrease in the spin—spin relaxation time, points to a limitation of the mobility of molecule fragments of both components of the system, which is due to incorporation of *n*-decylamine molecules between the CPB molecules in the micelles.

An absolutely different behavior is observed for *n*-butylamine, added to a solution of CPB (0.05 mol L^{-1}) in concentrations of up to 0.27 mol L^{-1}. The NMR data indicate that in this case, amine is not incorporated in the CPB micelles. A well-resolved line due to the methylene protons at the N atom in *n*-butylamine (δ 2.64) appears in the NMR spectrum; the diffusion decay can be conveniently monitored on this line. No line broadening, typical of the systems with *n*-decylamine, is observed in this case.

The radius calculated from Eq. (2) using the diffusion coefficient of *n*-butylamine in this system ($7.39 \cdot 10^{-10}$ $m^2 s^{-1}$) is equal to ~3 Å. This value is quite suitable for characterizing individual molecules of butylamine. The diffusion coefficient of CPB, determined, as in the case with *n*-decylamine, using the signal due to the CH_2 group protons (δ 1.2), is $5.59 \cdot 10^{-11}$ $m^2 s^{-1}$. Calculations from Eq. (2) show that CPB moves within a particle with a radius of 38 Å, which is very close to the radius of a CPB micelle without amines added.

Thus, the fact of formation of mixed micellar aggregates containing *n*-decylamine and CPB was confirmed by the kinetic method and by ^1H NMR spectroscopy. Some characteristic parameters were determined, namely, the critical micelle concentration, the diffusion coefficient, and the effective hydrodynamic radius. The catalytic properties of primary amine—CPB systems with various component ratios and their influence on the mechanism of hydrolytic cleavage of phosphorus-containing esters are evaluated: in the presence of short-chain amines, alkaline hydrolysis of **1** is the predominant reaction pathway, whereas in the case of *n*-decylamine, the contribution of hydrolysis catalyzed by the general basic mechanism increases due to the formation of mixed micelles with CPB.

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