

Reactivity of Inorganic α -Nucleophiles in Acyl Group Transfer Processes in Water and Surfactant Micelles: I. Systems Based on Organic Complexes of Tribromide Anion

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Abstract—Systems based on organic complexes of tribromide anion generate upon dissolution in water nucleophile–oxidant couple HOBr/BrO[−] and accelerate hydrolysis of ethyl 4-nitrophenyl ethylphosphonate, diethyl 4-nitrophenyl phosphate, and 4-nitrophenyl *p*-toluenesulfonate by a factor of 15–90 in the presence of cationic surfactant micelles. As in water, hypobromite ion in surfactant micelles acts as α -nucleophile, and the magnitude of the α -effect almost does not change in going from water to micelles. Micellar effects of surfactants are determined by the nucleophilicity of hypobromite ion in surfactant micelles and by solubilization of the substrate and BrO[−], which largely depend on the counterion concentration in the micelle surface layer. The main factor responsible for the observed acceleration is increased reactant concentration in the micellar pseudophase.

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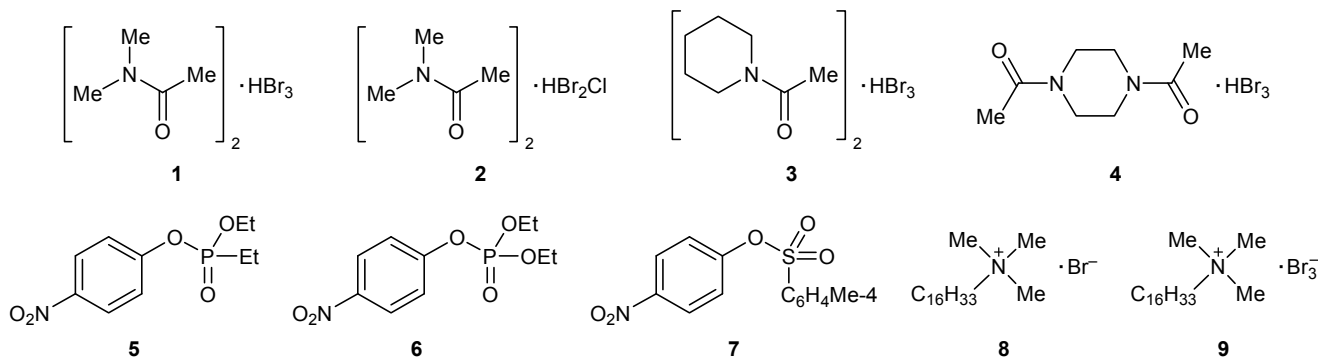
Inorganic α -nucleophiles, i.e., molecular entities possessing a lone electron pair in the α -position with respect to the nucleophilic center, show anomalously high reactivity in acyl group transfer processes, which exceeds 10^2 – 10^5 times the reactivity of “normal” nucleophiles with a comparable basicity (so-called α -effect) [1–3]. Although this effect has been studied over more than 50 years, no common opinion on its nature has been formulated so far [1–10]. Presumably, the possibility of manifestation of the α -effect is determined by a number of factors, including stabilization of the transition state due to intramolecular general acid–base catalysis [4, 5], enhanced thermodynamic stability of the products [5, 6], solvation [1], nucleophile basicity [2, 3, 7], hybridization of the electrophilic center [1, 8], and others [1, 3, 9, 10]. The contribution of each factor may change depending on the reaction series and conditions. Growing popularity of using organized solutions (surfactant micelles [3, 11–15], microemulsions [16], ionic liquids [17, 18], etc. [3, 19]) as reaction medium reasonably raises the problem of manifestation of the α -effect therein.

From the practical viewpoint, studies in this line are important since α -nucleophiles are highly efficient

reagents for decomposition of toxic phosphorus acid esters [3, 19]. Taking into account that decontamination solutions often contain surfactants [3, 11–15, 19], data on variation of the α -effect in these media would be useful for the prediction of their efficiency and optimization of their composition and properties.

A typical inorganic α -nucleophile is hypobromite anion [1–3, 20], and the conjugate hypobromous acid (HOBr) is a strong oxidant [3, 20–22]; therefore, the system HOBr/BrO[−] can be regarded as a nucleophile–oxidant couple ensuring not only nucleophilic cleavage of acyl-containing substrates but also further decomposition of the products. A required ratio of the nucleophilic cleavage and oxidation channels can readily be achieved by variation of the concentrations of HBrO and BrO[−], which depend on pH. Despite favorable combination of properties, the use of systems based on the HOBr/BrO[−] nucleophile–oxidant couple is limited due to instability and corrosiveness of the initial reactants [20].

Development of new convenient sources, bis(di-alkylamide) hydrogen tribromides **1–4**, and studies of the nucleophilic reactivity of the HOBr/BrO[−] system made it possible to estimate advantages and prospects



of using organic complexes. Bis(dialkylamide) hydrogen tribromides are characterized by high active bromine content and are stable, readily soluble in water, and relatively safe in handling. They dissociate in water with formation of tribromide ion whose hydrolysis generates HOBr, BrO^- , or HOBr/ BrO^- , depending on pH [20–23].

We previously showed that bis(dialkylamide) hydrogen trihalides 1–4 are efficient reagents for decomposition of 4-nitrophenyl esters 5–7 derived from phosphonic, phosphoric, and *p*-toluenesulfonic acids in water [20].

In this work we studied the reaction kinetics and compared nucleophilic reactivities of hypobromite ion generated from different precursors (complexes 1, 3, and 9) in the presence of cationic surfactant micelles in order to elucidate how the composition of organic complexes affects the nucleophilicity of BrO^- anion, estimate the efficiency of substrate and reagent binding to the micellar pseudophase, reveal specificity of the α -effect of hypobromite ion in micellar systems, and assess the relation between the substrate structure (compounds 5–7) and the observed micellar effects.

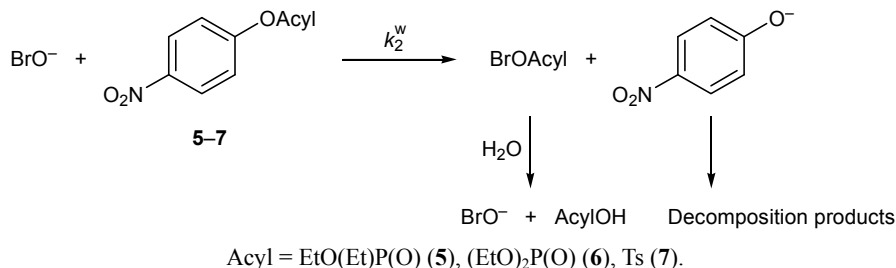
As cationic surfactant we used a classical representative of this group of compounds, cetyl(trimethyl)ammonium bromide (8), as well as cetyl(trimethyl)ammonium tribromide (9). On the one hand, surfactant 9 in aqueous medium gives rise to micelles analogous to those formed by 8; on the other hand, it acts as

a source of BrO^- ions due to the presence of reactive tribromide counterion [20–23].

Kinetics of decomposition of esters 5–7 with organic complexes 1, 3, and 9 in the presence of cationic surfactant micelles. Attack of hypobromite ion on the electron-deficient center of substrates 5–7 leads to the formation of 4-nitrophenoxide ion which undergoes further decomposition by the action of HOBr (Scheme 1) [20–23]. Therefore, while studying the nucleophilicity of BrO^- ion, the acidity of the medium was maintained at such a level that the pseudofirst-order rate constant did not change over at least 10 reaction half-times. Specifically, the reaction with ester 5 was carried out at $\text{pH} \geq 11.0$, and with esters 6 and 7, at $\text{pH} \geq 11.5$.

The dependences of the apparent second-order rate constants $k_{2, \text{app}} = k_{\text{obs}}/[\text{BrO}^-]$, $\text{L mol}^{-1} \text{s}^{-1}$, versus concentration of 8 and 9 (at a constant pH value) for the reactions of 5–7 with hypobromite ions generated from complexes 1, 3, and 9 were typical of nucleophilic substitution reactions occurring in two phases, micellar (m) and aqueous (w) (Figs. 1–4). Decomposition of substrate S in the presence of surfactant micelles is a fairly complicated process including alkaline hydrolysis and reaction of esters with hypobromite ion in both phases. This process can be represented by Scheme 2, where $k_{2, \text{BrO}^-}^{\text{m}}$ and $k_{2, \text{BrO}^-}^{\text{w}}$ ($\text{L mol}^{-1} \text{s}^{-1}$) characterize nucleophilic reactivity of BrO^- ; $k_{2, \text{OH}^-}^{\text{m}}$ and $k_{2, \text{OH}^-}^{\text{w}}$ ($\text{L mol}^{-1} \text{s}^{-1}$) characterize nucleophilicity of OH^- ion [under the given conditions, the concentration of

Scheme 1.



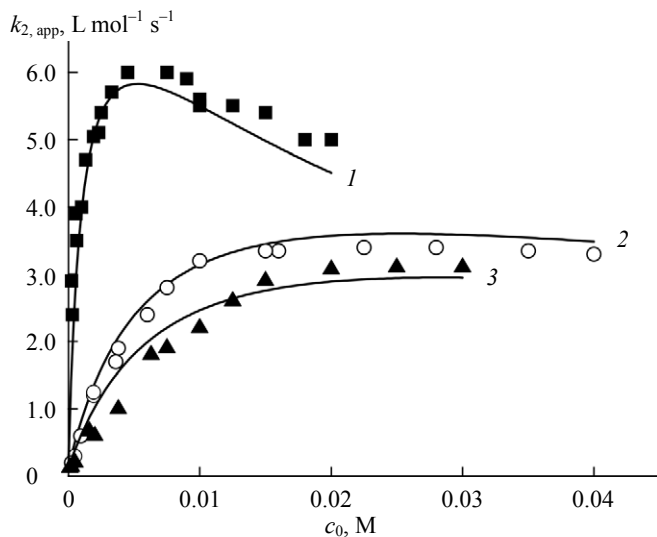


Fig. 1. Plots of the rate constants for the reaction of ethyl 4-nitrophenyl ethylphosphonate (**5**) with hypobromite ion versus surfactant concentration; water, pH 11.20, 25°C. Source of active bromine: (1) cetyl(trimethyl)ammonium tribromide (**9**), (2) bis(*N,N*-dimethylacetamide) hydrogen tribromide (**1**), and (3) bis(*N*-acetylpiperidine) hydrogen tribromide (**3**).

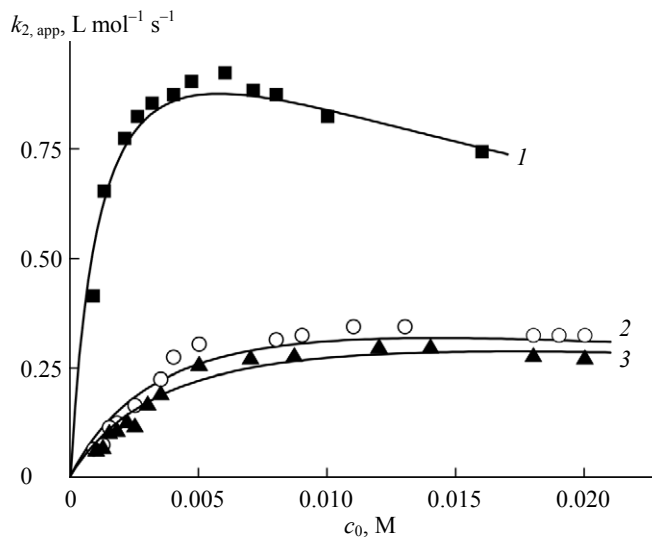


Fig. 2. Plots of the rate constants for the reaction of diethyl 4-nitrophenyl phosphate (**6**) with hypobromite ion versus surfactant concentration; water, pH 11.70, 25°C. Source of active bromine: (1) cetyl(trimethyl)ammonium tribromide (**9**), (2) bis(*N,N*-dimethylacetamide) hydrogen tribromide (**1**), and (3) bis(*N*-acetylpiperidine) hydrogen tribromide (**3**).

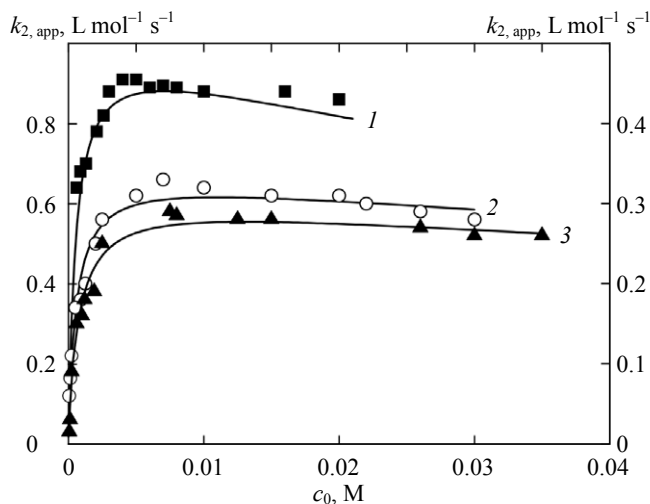


Fig. 3. Plots of the rate constants for the reaction of 4-nitrophenyl 4-toluenesulfonate (**7**) with hypobromite ion versus surfactant concentration; water, pH 11.50, 25°C. Source of active bromine: (1) cetyl(trimethyl)ammonium tribromide (**9**), (2) bis(*N,N*-dimethylacetamide) hydrogen tribromide (**1**), and (3) bis(*N*-acetylpiperidine) hydrogen tribromide (**3**).

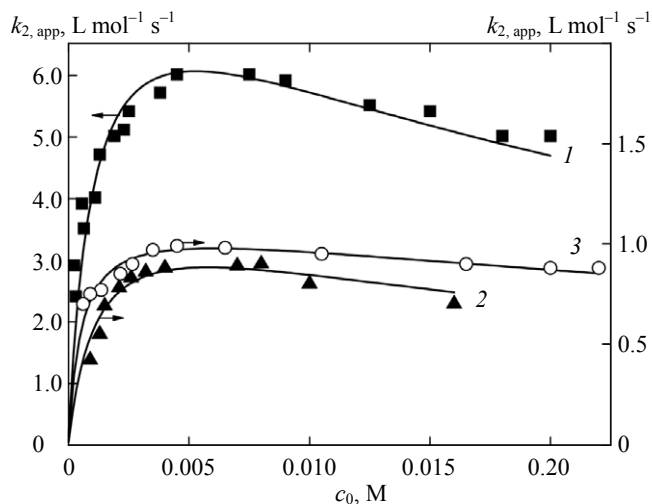


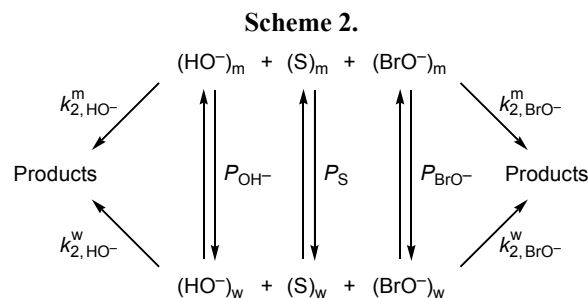
Fig. 4. Plots of the rate constants for the reactions of esters (1) **5** (pH 11.20), (2) **6** (pH 11.70), and (3) **7** (pH 11.50) with hypobromite ion versus concentration of cetyl(trimethyl)ammonium tribromide (**9**); water, 25°C.

hydroxide ions remained constant (pH = const)]; and P_S , P_{OH^-} , and P_{BrO^-} are the micelle–water partition coefficients of the substrate and hydroxide and hypobromite ions, respectively [20].

In terms of a simple pseudophase model [15, 20, 24] with account taken of distribution of the substrate and hypobromite and hydroxide ions, the kinetics of

the decomposition of esters **5–7** in the presence of surfactants **8** and **9** can be described by Eq. (1):

$$k_{obs} = \frac{k_{2,OH^-}^m (1/V_m) K_{OH^-} K_S c + k_{2,OH^-}^w [HO^-]_0}{(1 + K_{OH^-} c)(1 + K_S c)} + \frac{k_{2,BrO^-}^m (1/V_m) K_{BrO^-} K_S c + k_{2,BrO^-}^w [BrO^-]_0}{1 + K_{BrO^-} c)(1 + K_S c)} \quad (1)$$



Here, c (M) is the concentration of micellized surfactant [$c = c_0 - \text{CMC}$, where CMC (M) is the critical micelle concentration]; cV_m and $(1 - cV_m)$ are the volume fractions of the micelle and aqueous phases; $K_S = (P_S - 1)V_m$, $K_{\text{BrO}^-} = (P_{\text{BrO}^-} - 1)V_m$, and $K_{\text{OH}^-} = (P_{\text{OH}^-} - 1)V_m$ (L/mol) are equilibrium constants for binding of the substrate and hypobromite and hydroxide ions, respectively; and V_m (L/mol) is the partial volume of surfactant which was assumed to be equal to 0.37 L/mol [11, 13, 15, 20].

Analysis of the kinetic data showed that the contribution of alkaline hydrolysis of substrates to the observed rate constants is insignificant; therefore, in most cases the results were processed without considering alkaline hydrolysis.

Table 1 contains the parameters characterizing the reactions of hypobromite ion with substrates 5–7 in the presence of surfactants 8 and 9.

Micellar effects of surfactants in the reactions of esters 5–7 with hypobromite ion. Cationic surfactants like 8 and 9 usually give rise to so-called micellar catalysis of nucleophilic substitution (Table 2, $\Delta_{\text{exp}} = k_{\text{obs}}^m/k_{\text{obs}}^w$) [3, 11, 15]. Different factors may be responsible for increase of the observed rate constants [11].

In our kinetic experiments, the pH value was maintained at >11.0 ; therefore, there are no reasons to believe that the observed effects are partially related to shift of the acid ionization constant of hypobromous acid $\{pK_a^{\text{HOBr}}(\text{H}_2\text{O}) = 8.7$ [25]}.

The nucleophilicity of BrO^- in the micellar pseudo-phase change insignificantly: both increase and reduction of the reactivity are observed (Table 1, k_{2,BrO^-}^m), and the k_{2,BrO^-}^m and k_{2,BrO^-}^w values differ by no more than twofold. This character of variation of the nucleophilicity of BrO^- indicates that there is no radical change of the properties of the medium in going from the aqueous phase to micellar pseudophase. On the whole, $k_{2,\text{BrO}^-}^m \geq k_{2,\text{BrO}^-}^w$ for esters 5 and 6, and $k_{2,\text{BrO}^-}^m \leq k_{2,\text{BrO}^-}^w$ for substrate 7. In the latter case, reduction of the nucleophilicity of BrO^- may be favored by different efficiencies of substrate and nucleophile binding to surfactant micelles, which leads to separation of the active sites of the substrate and nucleophile (Table 1; cf. K_S and K_{BrO^-} for the reaction with ester 7). In the micellar pseudophase formed by surfactant 9, k_{2,BrO^-}^m

Table 1. Kinetic and thermodynamic parameters for the reactions of esters 5–7 with hypobromite ions in the presence of surfactants 8 and 9 (water, 25°C)

Source of active bromine	Surfactant	k_{2,BrO^-}^w , L mol ⁻¹ s ⁻¹ [20]	k_{2,BrO^-}^m , L mol ⁻¹ s ⁻¹	K_S , L/mol	K_{BrO^-} , L/mol
Ethyl 4-nitrophenyl ethylphosphonate (5)					
1 ^a	8	0.13	0.17±0.04	150	12±3
3 ^b	8	0.13	0.24	150	10
9	9	0.13	0.08±0.01	900	42±8
Diethyl 4-nitrophenyl phosphate (6)					
1 ^c	8	0.01	0.008±0.002	200	26±7
3 ^b	8	0.01	0.011	200	15
9	9	0.01	0.012±0.001	800	43±8
4-Nitrophenyl 4-toluenesulfonate (7)					
1 ^c	8	0.017	0.008±0.002	1600	13±3
3 ^b	8	0.017	0.007	1200	17
9	9	0.017	0.016±0.001	1800	25±3

^a $[\text{BrO}^-]_0 = \text{const} = 0.01$ M.

^b The kinetic and thermodynamic parameters were taken from [21, 22].

^c $[\text{BrO}^-]_0 = \text{const} = 0.019$ M.

Table 2. Micellar effects of surfactants **8** and **9** in the reactions of esters **5–7** with hypobromite ion (water, 25°C)

Source of active bromine	Surfactant	Δ_1^a	Δ_2^a	Δ_{theor}	Δ_{exp}^b
Ethyl 4-nitrophenyl ethylphosphonate (5)					
1	8	1.31	20	26	26
3^c	8	1.85	17	32	24
9	9	0.62	78	47	46
Diethyl 4-nitrophenyl phosphate (6)					
1	8	0.84	37	31	35
3^c	8	1.10	25	28	29
9	9	1.15	80	88	91
4-Nitrophenyl 4-toluenesulfonate (7)					
1	8	0.47	30	14	13
3^c	8	0.41	37	15	15
9	9	0.94	54	51	53

^a The parameter Δ_1 corresponds to the first multiplier in Eq (2), and Δ_2 , to the second multiplier.

^b $\Delta_{\text{exp}} = k_{\text{obs, BrO}^-}^m / k_{\text{obs, BrO}^-}^w$ at c_{opt} , pH = const.

^c Data of [21, 22].

increases for esters **5** and **6**, whereas the k_{2, BrO^-}^m and k_{2, BrO^-}^w values for **7** are approximately equal.

The most important factor determining the magnitude of micellar effects of surfactants is concentrating of substrate and nucleophile in micelles. Just the concentrating effects in most cases are responsible for increase of the observed rate constants. In fact, the degree of acceleration is described by Eq. (2) in terms of the pseudophase distribution model [13, 20, 24]:

$$\Delta = \frac{k_{2, \text{app}}^m}{k_{2, \text{BrO}^-}^w} = \frac{k_{2, \text{BrO}^-}^m}{k_{2, \text{BrO}^-}^w} \frac{K_S K_{\text{BrO}^-}}{V_m (\sqrt{K_S} + \sqrt{K_{\text{BrO}^-}})^2} = \Delta_1 \Delta_2 \quad (2)$$

Here, $k_{2, \text{app}}^m = k_{\text{obs, BrO}^-}^m / [\text{BrO}^-]$, Δ_1 , and Δ_2 characterize the contributions of the nucleophilicity and reactant concentrating to the increase of the reaction rate in going from water to the micellar pseudophase (Δ).

Comparison of the nucleophilicity of BrO^- toward substrates **5–7** in the aqueous and micellar phases (Tables 1 and 2) suggests that the observed acceleration effects cannot be rationalized solely by variation of the reactivity of the nucleophile (Table 2; $\Delta_{\text{exp}} = k_{\text{obs, BrO}^-}^m / k_{\text{obs, BrO}^-}^w$; $\Delta_{\text{theor}} = \Delta_1 \Delta_2$). As noted above, both increase and reduction of the nucleophilicity of hypobromite ion are observed (Δ_1 , Table 2). On the other hand, the concentrating effect [the second multiplier Δ_2

in Eq. (2)] ensures ~20–80-fold increase of the reaction rate. Micellar effects (Δ_{theor} , Table 2) increase ~15–90 times and reach the maximum value when micelle-forming surfactant **9** is used as a source of BrO^- ions. The Δ_{theor} and Δ_{exp} values under the optimal conditions agree well with each other provided that the concentrations of hypobromite ion in the aqueous and micellar phases and acidities of the latter are equal (Table 2). Thus, the main factor responsible for acceleration of the reactions in the presence of surfactants **8** and **9** is reactant concentrating effect in the micellar pseudophase.

Effects of the substrate nature and source of BrO^- on the micellar effects of surfactants. Depending on the BrO^- precursor, the magnitude of micellar effects changes in the series **9** > **3** \approx **1** for all substrates **5–7**, and the differences range from 1.5 to 3 times (Table 2; Δ_{theor} and Δ_{exp}). This result seems somewhat surprising since the micellar pseudophase of surfactants **8** and **9** is formed by cetyl(trimethyl)ammonium, i.e., their micelles should have similar structures. Less significant acceleration of the reaction in systems based on surfactant **8** and organic tribromide complexes **1** and **3**, as compared to **9**, is most likely to be related to increased concentration of counterions (Br^-), which leads to partial neutralization of the surface

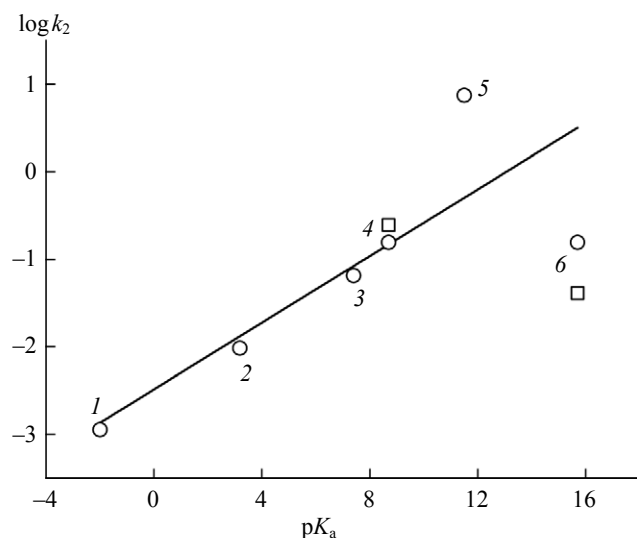


Fig. 5. Brønsted plots for the reactions of inorganic nucleophiles (1) NH₂OH, (2) F⁻, (3) ClO⁻, (4) BrO⁻, (5) HOO⁻, and (6) HO⁻ with ethyl 4-nitrophenyl ethylphosphonate (5) in water (light circles) and in the presence of cetyl(trimethyl)ammonium bromide (8) (light squares); 25°C.

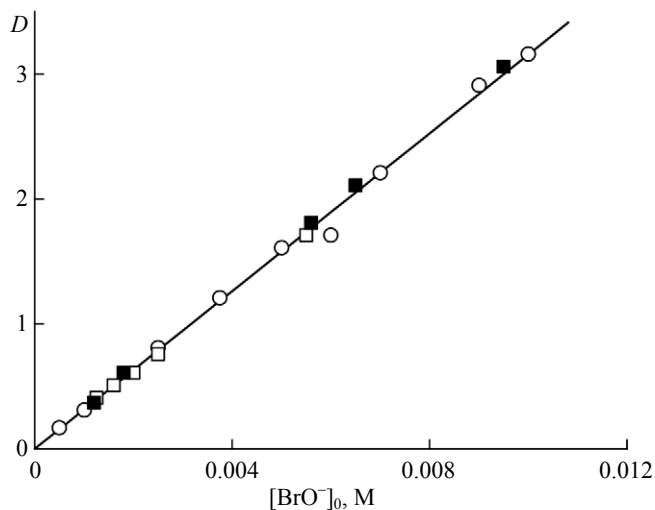


Fig. 6. Plot of the optical density at λ 330 nm versus concentration of hypobromite ion ($[\text{BrO}^-]_0$, M) generated from bis(*N,N*-dimethylacetamide) hydrogen tribromide (1) [the concentration of BrO⁻ was determined by iodometric titration at pH 11.15 (light circles) and pH 11.70 (light squares)] and cetyl(trimethyl)ammonium tribromide (9) [the concentration of BrO⁻ was calculated from the analytical surfactant concentration; pH 11.50 (dark squares)]; water, 25°C.

charge of micelles. Therefore, destabilizing effect of the repulsion between similarly charged head groups decreases, which is accompanied by reduction of the surface potential in absolute value and variation of the effect of the micellar pseudophase on the physico-chemical properties and reactivity of the solubilized

reactants. Decrease of the observed rate of the reaction of BrO⁻ with substrates 5–7 in systems 1/8 and 3/8 compared to 9 is accompanied by reduction of the ability of the system to concentrate nucleophile species in the micelle surface layer, as follows from the variation of K_{BrO^-} [Table 1; K_{BrO^-} (9) > K_{BrO^-} (1/8) \approx K_{BrO^-} (3/8)]. As a result, the catalytic effect weakens. Analogous character of the counterion effect was observed previously for other nucleophilic reactions, in particular for alkaline hydrolysis of esters [10, 11].

The second factor that may be responsible for micellar effects of 8 is the nature of the organic component of complexes 1 and 3. Concurrent binding of the substrate and organic component of the complex by surfactant micelles cannot be excluded. Nevertheless, the contribution of that factor to micellar effects is likely to be insignificant as compared to the effect of counterion concentration. This follows from the similarity of substrate binding constants K_S for systems 1/8 and 3/8 and surfactant 9 (Table 1).

Finally, particular attention should be given to the relation between the substrate structure and micellar effects of surfactants. The highest nucleophilic reactivity of BrO⁻ in water was observed in the reaction with ester 5 and was comparable with those for substrates 6 and 7: $k_{2, \text{BrO}^-}^{\text{w}}(\text{5}) \gg k_{2, \text{BrO}^-}^{\text{w}}(\text{7}) \geq k_{2, \text{BrO}^-}^{\text{w}}(\text{6})$. A similar nucleophilicity series was observed for the micellar pseudophase: $k_{2, \text{BrO}^-}^{\text{m}}(\text{5}) \gg k_{2, \text{BrO}^-}^{\text{m}}(\text{6}) \approx k_{2, \text{BrO}^-}^{\text{m}}(\text{7})$. On the other hand, the magnitude of micellar effects also depends on the substrate hydrophobicity. For example, almost twofold reduction of the nucleophilicity of BrO⁻ toward ester 7 was found for systems 1/8 and 3/8, but stronger substrate concentrating (K_S , Table 1) makes micellar effects comparable with those observed in the decomposition of esters 5 and 6 (Table 2).

As we already noted, hypobromite ion is a typical α -nucleophile. It reacts with esters 5–7 in water at rates comparable to the rate of alkaline hydrolysis, though its basicity is lower by 8 orders of magnitude than the basicity of hydroxide ion (Fig. 5). Hypobromite ion retains its anomalously high reactivity in the micellar pseudophase formed by surfactants 8 and 9, while the magnitude of the α -effect changes insignificantly (Fig. 5). Taking into account that the nucleophilicity of hypobromite ion in cationic surfactant micelles conforms to the Brønsted relationship for inorganic α -nucleophiles in water, there are reasons to assert that the α -effect of BrO⁻ ion in water and surfactant micelles is determined by similar factors.

In conclusion, it should be emphasized that, among various classes of surfactants, those possessing a reactive ion, as well as functionalized surfactants [12–15], attract particular interest since they make it possible to achieve considerable micellar effects without introduction of additional reagents. An undoubted advantage of systems based on organic complexes with tribromide ion is that they are convenient in handling and versatile reagents capable of acting as nucleophile, oxidant, or brominating agent.

EXPERIMENTAL

Bis(*N,N*-dimethylacetamide) hydrogen tribromide (**1**), bis(*N*-acetylpiperidine) hydrogen tribromide (**3**), and cetyl(trimethyl)ammonium tribromide (**9**) were synthesized according to the procedure described in [20]. The active bromine content of complexes **1** and **3** (96–98%) was determined by iodometric titration. The molar absorption coefficient of hypobromite ion was determined from the concentration dependence of the optical density of a series of solutions of tribromide **1** (Fig. 6). The concentration of hypobromite ion in aqueous solutions of complexes **3** and **9** was determined by spectrophotometry at λ 330 nm ($\varepsilon = 300 \pm 20 \text{ L mol}^{-1} \text{ cm}^{-1}$). The difference between the theoretical and experimentally found concentrations of hypobromite ions in solutions of tribromides **3** and **9** did not exceed 5%.

Esters **5** and **7** were synthesized and purified as described in [20]. Commercial diethyl 4-nitrophenyl phosphate (**6**) ($\geq 90\%$, Aldrich), cetyl(trimethyl)ammonium bromide (**8**) ($\geq 99.0\%$, Aldrich), and inorganic reagents of analytical and ultrapure grades were used without additional purification. All solutions were prepared using doubly distilled water just before kinetic measurements and were adjusted to required pH values by adding a small amount of concentrated aqueous potassium hydroxide or HCl. The acidity of the medium was measured before and after each kinetic run with a Metrohm 744 pH meter. The kinetics of nucleophilic cleavage of esters **5–7** was monitored by accumulation of 4-nitrophenoxide ion (water, 25°C, λ 400–420 nm; Thermo Electron Genesys 10S UV spectrophotometer). The initial substrate concentration ($< 5 \times 10^{-5} \text{ M}$) was always much lower than the initial nucleophile concentration. The substrate (as a concentrated solution in methanol) was added to a spectrophotometric cell through a capillary. The observed pseudofirst-order rate constants k_{obs} (s^{-1}) were deter-

mined from the change of the optical density with time using Eq. (3) [13, 20]:

$$\ln(D_{\infty} - D_{\tau}) = \ln(D_{\infty} - D_0) - k_{\text{obs}}\tau, \quad (3)$$

where D_0 , D_{τ} , and D_{∞} are the optical densities at the initial moment, time τ , and by the end of the process, respectively. The linear dependences were processed by the least-squares method, and the obtained values were characterized by mean-square deviations.

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