Effect of structural preorganization on the reactivity of carbazoylmethyl derivatives of pyrogallol and calix[4]pyrogallol

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The aggregation behavior of carbazoylmethyl derivatives of pyrogallol and calix[4]pyrogallol was studied by conductometry and dynamic light scattering in water—DMF media. The formation of mixed nanoaggregates in the presence of cationic surfactants was revealed. The effect of structural preorganization of these compounds on their reactivity in the acylation by 4-nitrophenylacetate and the catalytic activity in the hydrolysis of phosphorus acid esters was studied.

Key words: micelles, critical micelle concentration, kinetics, hydrolysis, acylation, hydrazides, cationic surfactants, calix[4]pyrogallol, organophosphorus compounds.

Calixarenes are typical objects of supramolecular chemistry capable of noncovalent binding of organic compounds.[1—5] The application of calixarenes is known[6—9] in analytical chemistry, ecology, lithography, in the creation of film materials, catalysts, *etc*. We have previously obtained[10—13] data on the self-organization of amphiphilic calixarenes and showed[9,14—16] a substantial effect of aggregation on their catalytic activity in the reactions with phosphorus and carboxylic acid esters.

We have recently[17—19] reported the synthesis and characterization of new calix[4]arenes functionalized by the carbazoylmethyl groups. Acyl hydrazides find use as drugs, complexones, and photo- and thermochromic materials.[20—22] At the same time, they are key reactants in the syntheses of many organic compounds.[23—25] The synthesized hydrazide derivatives of calix[4]resorcinol and calix[4]pyrogallol exist in the boat conformation. For all calix[4]arenes under study, including their model compounds,[17,19] the *trans*-structure of the amide moiety of the hydrazide group was found.

Solubility of calix[4]phenols with acetylhydrazide groups in organic solvents, in particular, in chloroform, made it possible to study their extraction properties toward a series of transition metal ions.[18] This study showed that the high extraction ability of these compounds compared to that of their structural unit, namely, 4-*tert*-butylphenoxyacetylhydrazide, is caused by the preorganization of the hydrazide groups on the calixarene platform resulting in the positive cooperative effect in binding of metal ions. An important influence of the cooperative

effect of the functional groups on the catalytic properties was observed[26] for the supramolecular models of enzymes.

In the present work, we studied the acylation of the NH_2 groups of the hydrazine fragments of calix[4]pyrogallol **1** and its structural analog **2** with 4-nitrophenylacetate (*p*-nitrophenylacetate, PNPA) as the acyl group donor. This was done in order to reveal the
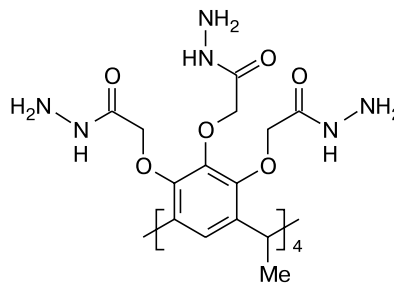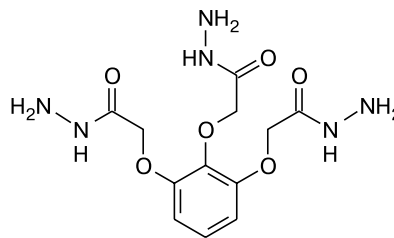
role of structural preorganization of the hydrazide groups and the effect of aggregation on the reactivity and catalytic activity. The hydrolysis of *P,P*-bis(chloromethyl)-*O*-(4-nitrophenyl)phosphinate (BCNPP) catalyzed by compound **1** was also studied in aqueous DMF (30 vol.%) in a wide concentration range.

Calix[4]pyrogallol **1** containing the hydrophilic acetylhydrazide groups and hydrophobic aryl fragments is insoluble in lowly polar solvents but is soluble in water.[17] Its structural analog **2** possess similar properties. Therefore, these compounds are of interest in studying the influence of aggregation and preorganization of the functional groups on their reactivity in aqueous and aqueous-organic media.

Experimental

Commercial PNPA (Fluka) was used; BCNPP was synthesized according to a known procedure.[27] The reactions of ethyl bromoacetate with pyrogallol and calix[4]pyrogallol afforded esters of the corresponding arene-1,2,3-triyl-tris(hydroxyacetic acids), which were transformed by the subsequent treatment with excess hydrazine hydrate into hydrazides **1** and **2** according to a procedure described by us earlier.[17]

All measurements were carried out in a water—DMF (30 vol.%) medium at 30 °C. Electrical conductivity was measured on a CDM-2d conductometer (Denmark). The electrical conductivity of bidistilled water used for the preparation of solutions of the reactants was taken as the zero value. The effective radii of the aggregates were determined on a Photocor Complex dynamic light scattering instrument and calculated from the diffusion coefficients by the Stokes—Einstein equation for spherical particles of the same size with allowance for the viscosity of the solutions under study. For each system the hydrodynamic radius was determined as the arithmetical average of the results of ten measurements.

The kinetics of the acylation and hydrolysis processes was studied by spectrophotometry on a Specord UV—Vis instrument at the initial substrate concentrations $5 \cdot 10^{-5}—1 \cdot 10^{-4}$ mol L^{-1} and different pH values at 30 °C in a water—DMF (30 vol.%) medium. The reaction course was monitored by the change in the absorbance of solutions at a wavelength of 400 nm (formation of the 4-nitrophenolate anion). The conversion was higher than 90%. The apparent rate constants of the first-order reaction (k_{app}, s^{-1}) were calculated by the regression method using the equation

$$\ln(D_\infty - D_t) = -0.434 k_{app} t + \text{const}, \tag{1}$$

where D_∞ and D_t are the absorbances upon the cessation of the reaction and at the time t.

The kinetic dependences were analyzed by the equation of the pseudo-phase model of micellar catalysis[28a]

$$k_{app} = [k_w + k_m K_s (C_{Surf} - CMC)]/[1 +$$
$$+ K_s (C_{Surf} - CMC)], \tag{2}$$

where k_{app} is the apparent rate constant of the pseudo-first order reaction, s^{-1}; k_w and k_m are the rate constants of the pseudo-first

order reaction in the solvent bulk and in the micellar phase, s^{-1}; K_s is the binding constant of the substrate with the micelle, L mol^{-1}; C_{Surf} is the surfactant concentration, mol L^{-1}; CMC is the critical micelle concentration, mol L^{-1}.

Results and Discussion

The aggregation of the derivatives of calix[4]pyrogallol **1** and pyrogallol **2** was studied by conductometry and dynamic light scattering. The curve of the dependence of the electrical conductivity on the concentration of compound **1** (Fig. 1) contains two inflections corresponding to the onset of aggregation (CMC$_1$ = $1.2 \cdot 10^{-5}$ mol L^{-1}) and structural rearrangement in the system (CMC$_2$ = $1.5 \cdot 10^{-3}$ mol L^{-1}). According to the light scattering data, the radius of the aggregates of calix[4]pyrogallol **1** at a concentration of $2.5 \cdot 10^{-3}$ mol L^{-1} is 137 nm, whereas the radius is 141 nm at a concentration of $1 \cdot 10^{-3}$ mol L^{-1}, *i.e.*, in the region from CMC$_1$ to CMC$_2$. Evidently, the structure of the aggregates is formed due to intermolecular hydrogen bonding (IMHB) of the —C=O...H—N— and —N—H...N— types, which were observed in calix[4]arene hydrazides and their monomeric analogs in the solid state and in solutions according to X-ray diffraction and IR and NMR spectroscopic data.[17,19,29,30] Struc-
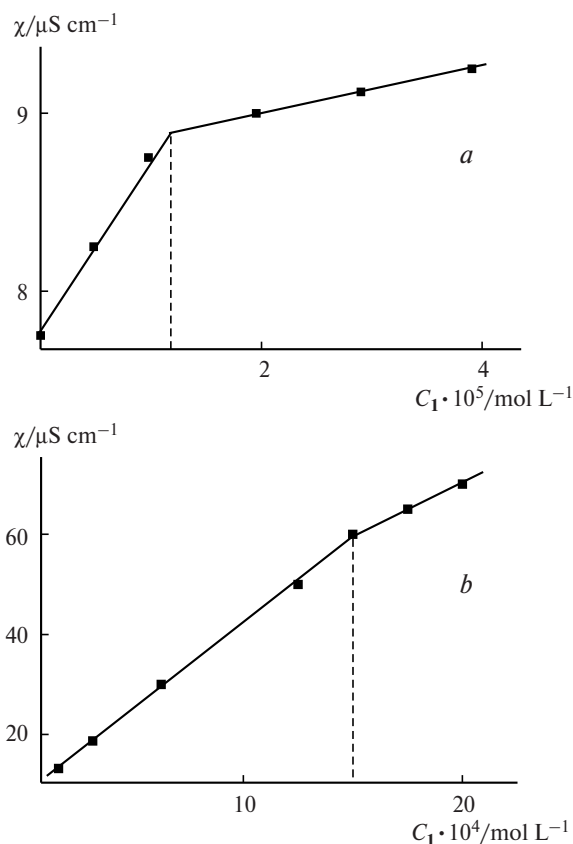

Fig. 1. Plots of the electrical conductivity *vs* concentration of compound **1** in the region of CMC$_1$ (*a*) and CMC$_2$ (*b*).

tural arrangements in the system can be caused by a change in the shape or sizes of the associates. The carbazoylmethyl derivative of pyrogallol **2** is also aggregated in a water—DMF medium (CMC = $3 \cdot 10^{-3}$ mol L^{-1}).

It is known[31—33] that unsubstituted calix[4]pyrogallols in both the solid state and solutions form stable hexameric aggregates. This process can additionally involve water molecules or metal cations. The nanospheroidal structures obtained from these compounds are stable even in polar water-containing media.[31] However, the size of these aggregates (diameter less than ~19 nm)[32,33] is substantially smaller than the size of the structures in our case. The substitution of the hydrogen atoms of the hydroxy groups for the carbazoylmethyl moieties favors, most likely, the formation of a more complex and saturated network of hydrogen bonds. This can be a main reason for the enhanced stability of the aggregates in our case and for their considerable sizes.

The kinetics of the acylation of derivatives **1** and **2** by PNPA in a water—DMF medium was studied (Scheme 1).

Scheme 1
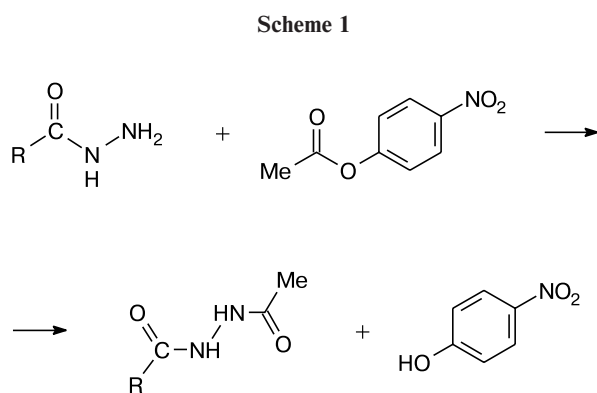
The dependence of the apparent rate constant (k_{app}, s^{-1}) of the acylation of compounds **1** and **2** on the pH value is presented in Fig. 2. These data show that the reactivity of the macrocycle is much higher than that of its monomeric analog. The plot of the apparent rate constant *vs* concentration of **1** is nonlinear and reaches a plateau (Fig. 3). This confirms the formation of associates of compound **1** capable of pre-reaction binding of the
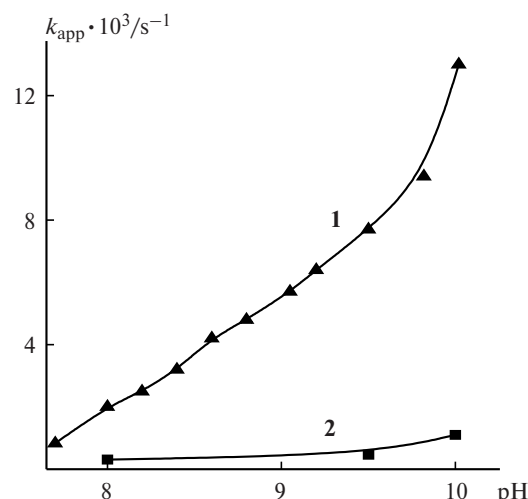

Fig. 2. Dependence of k_{app} of the acylation of compounds **1** and **2** on pH ($C_1 = 1.5 \cdot 10^{-3}$ mol L^{-1}, $C_2 = 6 \cdot 10^{-3}$ mol L^{-1}).
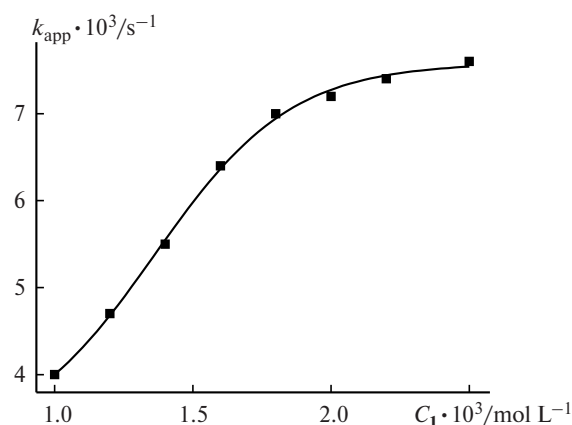

Fig. 3. Dependence of k_{app} of the acylation on the concentration of calix[4]pyrogallol **1** (pH = 9.5).

substrate (aggregate—substrate complex) due to noncovalent interactions similarly to the formation of the enzyme—substrate and micelle—substrate complexes.[28b,34] The data shown in Fig. 3 were processed using Eq. (2) of the pseudo-phase model of micellar catalysis. The parameters obtained are given in Table 1. Rather high binding constant of PNPA ($K_s = 1300$ L mol^{-1}) with the forming aggregates of calix[4]pyrogallol **1** suggests that the factor

Table 1. Parameters of the acylation of calix[4]pyrogallol **1** by PNPA in the absence and in the presence of CSurf (pH = 9.5)

System	CMC/mol L^{-1}	$k_m \cdot 10^2$/s^{-1}	K_s/L mol^{-1}	k_m/k_0
1—H$_2$O—DMF	$5.8 \cdot 10^{-4}$	1.1	$1.3 \cdot 10^3$	22[a]
CSurf$_1$—**1**[b]—H$_2$O—DMF	$3.6 \cdot 10^{-5}$	3.1	$9.5 \cdot 10^2$	4[c]
CSurf$_2$—**1**[b]—H$_2$O—DMF	$1.2 \cdot 10^{-4}$	4.4	$1.3 \cdot 10^3$	6[c]

[a] Acceleration of the rate constant of the acylation of compound **2** ($6 \cdot 10^{-3}$ mol L^{-1}) at pH = 9.5 ($k_0 = 5 \cdot 10^{-4}$ s^{-1}).
[b] $C_1 = 1.5 \cdot 10^{-3}$ mol L^{-1}.
[c] Acceleration over the rate constant of the acylation of compound **2** ($6 \cdot 10^{-3}$ mol L^{-1}) in the absence of CSurf at pH = 9.5 ($k_0 = 7.5 \cdot 10^{-3}$ s^{-1}).

Table 2. The CMC values (mol L^{-1}) for CSurf in the absence (I) and presence (II) of calix[4]pyrogallol **1** ($C_1 = 1.5 \cdot 10^{-3}$ mol L^{-1})

Parameter	CSurf$_1$		CSurf$_2$		CSurf$_3$	
	I	II	I	II	I	II
CMC$_1$	$5 \cdot 10^{-4}$	$5 \cdot 10^{-5}$	$6 \cdot 10^{-4}$	$3 \cdot 10^{-5}$	$2 \cdot 10^{-5}$	$1 \cdot 10^{-5}$
CMC$_2$	$6.5 \cdot 10^{-3}$	$2 \cdot 10^{-4}$	$7 \cdot 10^{-3}$	$1.5 \cdot 10^{-4}$	$1.2 \cdot 10^{-4}$	—

of reactant concentrating makes the main contribution to the process under study.

The reactivity of the monomeric structural analog **2** in the acylation at the concentration exceeding by 4 times the concentration of macrocycle **1** (see Table 1) is by ~20 times lower. This is distinctly seen from the data in Fig. 2 exhibiting the plots of k_{app} *vs* pH for the reactions of compounds **1** and **2** with PNPA.

At present, binary mixtures of amphiphilic compounds forming combined nanoaggregates with specific, including catalytic, properties are of great interest for controlling the rates of chemical processes.[35-38] Calix[4]pyrogallol **1** was acylated in micellar solutions of cationic surfactants (CSurf) containing the hydroxyalkyl groups: cetyldimethyl(2-hydroxypropyl)ammonium (CSurf$_1$) and cetylmethyldi(2-hydroxyethyl)ammonium (CSurf$_2$) bromides. In this case, the systems based on calix[4]pyrogallol **1** and CSurf consist of the components capable of self-associating. In addition, compound **1** contains the carbazoylmethyl groups that can be involved in chemical processes.

Calix[4]pyrogallol **1** was found to affect the CMC of the detergents decreasing them. The CMC values of the cationic surfactants in the absence and presence of **1** are presented in Table 2. Figure 4 shows as an example the dependence of the electrical conductivity on the CSurf$_1$ concentration in the presence of compound **1**. The decrease in the CMC upon the addition of macrocycle **1** to the system, according to published data,[39] favors the formation of combined aggregates due to hydrogen bonds of the hydrazide fragments with the OH groups of the molecules or micelles of CSurf. It can be assumed that these bonds are stronger than the IMHB between the hydrazide groups of the molecules in compound **1**. This can decompose an aggregate of calix[4]pyrogallol, after which its molecules interact with the CSurf micelles to form combined structures.

The size of the aggregates in a calix[4]pyrogallol **1** ($1 \cdot 10^{-3}$ mol L^{-1})—CSurf$_2$ ($4 \cdot 10^{-4}$ mol L^{-1})—water—DMF system was determined by dynamic light scattering and showed a decrease in the radius of the aggregates of calix[4]arene **1** in the presence of CSurf$_2$ (from 141 to 125 nm). It is known[9,40] that for the cationic surfactants the radius of the micelles in water and in a water—DMF (30 vol.%) medium is ~2.5—3 nm.

The plots of k_{app} of the acylation of **1** with PNPA *vs* CSurf concentration are presented in Fig. 5. It is seen that the rate of the process in the micellar systems increases by 4—6 times. The results of quantitative processing of the nonlinear dependences $K_{app} = f(C_{CSurf})$ by Eq. (2) of the pseudo-phase model are given in Table 1. The binding constants of the substrate (PNPA) in CSurf$_2$ solutions
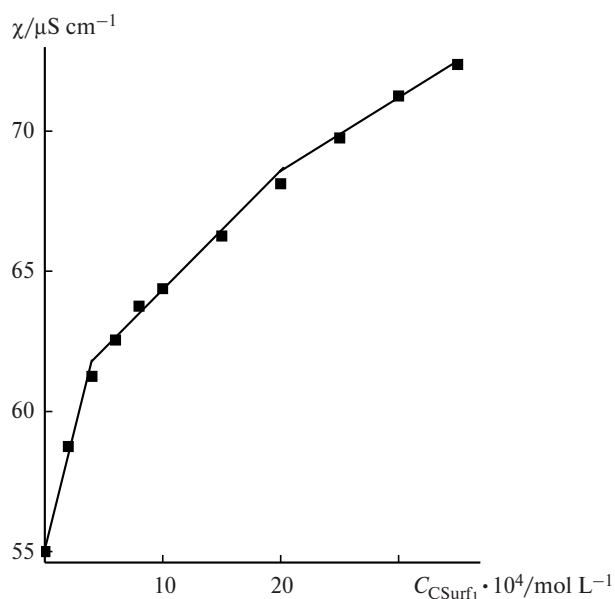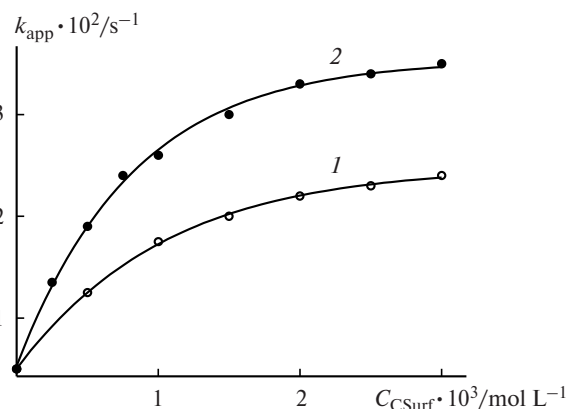

Fig. 4. Dependence of the electrical conductivity on the CSurf$_1$ concentration in the presence of calix[4]pyrogallol **1** ($C_1 = 1.5 \cdot 10^{-3}$ mol L^{-1}).

Fig. 5. Plots of k_{app} of the acylation of calix[4]pyrogallol **1** *vs* concentration of CSurf$_1$ (*1*) and CSurf$_2$ (*2*); water—DMF (30 vol.%), pH = 9.5.

Table 3. Parameters of the hydrolysis of BCNPP

System	pH	$k_0{}^a$/s^{-1}	$k_m \cdot 10^{-2}$ /s^{-1}	K_s /L mol^{-1}	CMC /L mol^{-1}	k_m/k_0
1—H$_2$O—DMF	9.5	$1.58 \cdot 10^{-2}$	9.6	1260	$2.6 \cdot 10^{-3}$	6
CSurf$_3$—**1**^{b}—H$_2$O—DMF	9.0	$5 \cdot 10^{-3}$	4.1	2400	$1.6 \cdot 10^{-5}$	8

^{a} Rate constant of the hydrolysis of BCNPP in the absence of compound **1** and surfactant.
^{b} $C_1 = 3.5 \cdot 10^{-3}$ mol L^{-1}.

(1300 L mol^{-1}) are higher than K_s in the system based on CSurf$_1$ (950 L mol^{-1}), suggesting the direct participation of the hydroxyalkyl groups of CSurf in the binding.

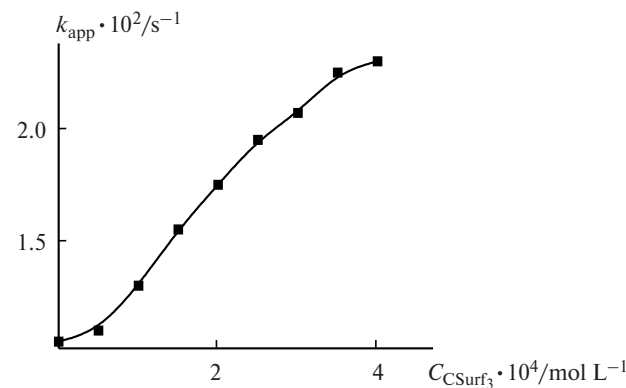The catalytic activity of calix[4]pyrogallol **1** was studied using the hydrolysis of BCNPP as an example (Scheme 2).

Scheme 2

(ClCH$_2$)$_2$P(O)OC$_6$H$_4$NO$_2$-4 + H$_2$O →

→ (ClCH$_2$)$_2$P(O)OH + HOC$_6$H$_4$NO$_2$-4
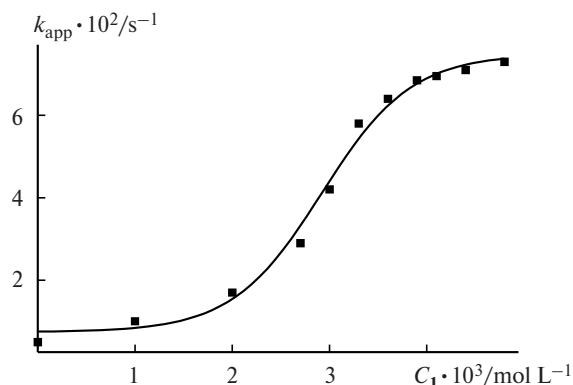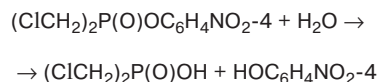
Fig. 6. Dependence of k_{app} of the hydrolysis of BCNPP on the concentration of calix[4]pyrogallol **1** (pH = 9.0).
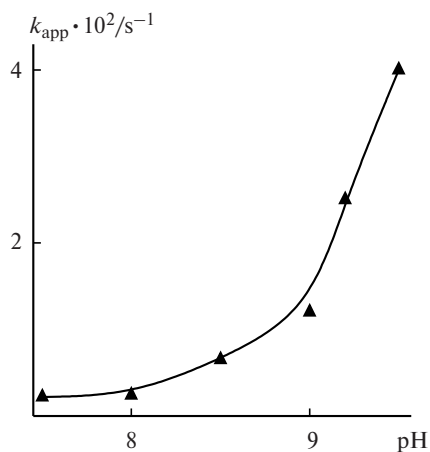
Fig. 7. Dependence of k_{app} of the hydrolysis of BCNPP on pH in the presence of calix[4]pyrogallol **1** ($C_1 = 3.0 \cdot 10^{-3}$ mol L^{-1}).

The formation of bis(chloromethyl)phosphinic acid ($\delta = 27.6$) in this reaction was confirmed by the ^{31}P NMR method. The chemical shift of BCNPP in a water—DMF medium is 32.2 ppm.

The plots of the apparent rate constant of the hydrolysis of BCNPP *vs* concentration of calix[4]pyrogallol **1** and *vs* pH are shown in Figs 6 and 7, respectively. The results of quantitative processing of the data in Fig. 6 using Eq. (2) are given in Table 3. The hydrolysis in the presence of compound **1** is accelerated by 6 times compared to the alkaline hydrolysis of BCNPP under the same conditions. The acceleration of the hydrolysis in the presence of structural analog **2** in a concentration of $1.6 \cdot 10^{-2}$ mol L^{-1} (pH = 9.5) is by ~2 times lower ($k_{app} = 1.8 \cdot 10^{-2}$ s^{-1}) than that in the presence of calix[4]pyrogallol **1** ($k_{app} = 4 \cdot 10^{-2}$ s^{-1}) in the concentration ~5-fold lower ($3 \cdot 10^{-3}$ mol L^{-1}).

The effect of a geminal surfactant, *viz.*, hexylidene-1,6-bis(dimethylcetylammonium) dibromide (CSurf$_3$), on the hydrolysis of BCNPP catalyzed by compound **1** (Fig. 8) and the influence of the latter on the CMC$_1$ of this surfactant (see Table 2) were revealed. In a CSurf—macrocycle **1** mixed system an increase in the binding constant of the substrate compared to the system in the absence of a detergent is observed. However, the combined aggregates exert a catalytic effect that differs insignificantly from the catalytic activity of the aggregates of calix[4]pyrogallol **1** itself (see Table 3).

Thus, aggregate formation due to intermolecular hydrogen bonds between the carbazoyl groups of calix[4]-

Fig. 8. Dependence of k_{app} of the hydrolysis of BCNPP on the concentration of CSurf$_3$ in the presence of calix[4]pyrogallol **1** ($C_1 = 3.5 \cdot 10^{-3}$ mol L^{-1}, pH = 9.0).

pyrogallol **1** molecules increases its reactivity in the acylation and the catalytic activity in the hydrolysis of phosphorus acid esters compared to the reactivity of its structural unit **2**. The formation of the combined aggregates of calix[4]pyrogallol **1** with the cationic surfactant enhances its ability to acylation and catalytic effect.

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