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The aggregartion and catalytic activity of amphiphilic calix[4]resorcinolarenes and phenols in hydrolysis of phosphonous esters in water—dimethylformamide media

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The amphiphilic calix[4]resorcinolarenes, aminomethylated calix[4]resorcinolarenes, *o*-aminomethylphenols, and their quaternary derivatives in water—dimethylformamide media (10—75 vol.% DMF) form aggregates, which catalyze the hydrolysis of phosphonous esters. The ability to self-association and the catalytic activity of the aggregates depend on the hydrophobicity of the amphiphilic compound, the pH of solution, and the content of DMF.

Key words: micelle formation, kinetics, hydrolysis, aggregates, critical micellization concentrations, amphiphilic compounds, aminomethylated calix[4]resorcinolarenes, phenols, phosphonic esters.

Earlier[1—3] we have found that the catalytic activity of highly organized water—organic solvent systems based on calix[4]resorcinolarenes and their derivatives in reactions with esters of phosphorus acids (EPA) depends mainly on the kind and properties of the aggregates formed. Recently, an interest to functionalized micelles sharply increased.[4,5] The self-assembling and properties of the aggregates of calix[4]resorcinolarenes are due[1,2] to amphiphilicity, *i.e.*, the presence of both a hydrophobic hydrocarbon radical on the lower rim of the cavity of calix[4]resorcinolarenes and a charge on the upper rim, as well as to a solvent nature. Water-soluble calix[4]arenes (n = 4—8) containing the sulfonic and ammonium head groups are known to form aggregates similarly to common ionic surfactants.[6—8] The critical aggregation concentrations depend on the length of the hydrocarbon radical, the number of aromatic rings (n), and the conformation of calixarenes.

The aim of this work is a study of the aggregation of *o*-aminomethylphenols (AMP, **1—8**) and their quaternary derivatives (QAMP, **9**, **10**), calix[4]resorcinolarene (**11**), and aminomethylated calix[4]resorcinolarenes (AMC, **12—14**) with different hydrophobicities in water—dimethylformamide media (10—75 vol. % DMF), and the effect of the solvent composition and the pH of solutions on their micelle-forming ability and catalytic activity in reactions with ethyl-*p*-nitrophenyl chloromethyl phosphonite (**15**).

Experimental

Compounds **1—8**,[9] **9** and **10**,[10] **11**,[11], **12—14**,[12] and substrate **15** [13] were synthesized according to earlier reported procedures. The parameters of recrystallized triphenylphosphate (**16**) and *p*-nitrophenol (**17**) corresponded to the literature values.[14] The surface tension (σ) of solutions was measured on a VT torsion balance by the ring detachment method, and electroconductivity of solutions (χ) was estimated on a CDM-2d conductometer at 25 °C. Temperature was maintained with a U-1 thermostat with an accuracy of ±0.1 °C. To prepare ionized species, the micelle formation for compounds **1—14** was studied in the 4—8- or 1—2-fold excess of NaOH or HCl for AMC or AMP and QAMP, respectively. The reaction kinetics for these compounds with substrate **15** was studied by spectophotometry and ^{31}P NMR spectroscopy under more than 10-fold excess of a nucleophile (AMP, QAMP, and AMC) with respect to substrate **15**. The reactions were monitored spectrophotometrically on a Specord UV—Vis spectrophotometer by the variation of the absorption band of the *p*-nitrophenolate formed (λ = 400 nm) at 25 °C and the concentration of compound **15** of $5 \cdot 10^{-5}$ mol L^{-1}. The ^{31}P NMR spectra were recorded on a Bruker MSL 400 instrument (161.97 MHz, 85% H_3PO_4 as the reference).

The apparent rate constants for hydrolysis (k_{app}) were determined from the first order equation. The constants of binding of substrates by the aggregates formed with a catalyst (K_b), the critical aggregation concentrations (critical micellization concentration) (CMC), and the reaction rate constants for aggregates (k_m) were calculated from the plots of k_{app} vs. the nucleophile concentration. For this purpose, the known equation[15] was used:

$$k_{app} = (k_{H_2O} + k_m K_b C_{surf})/(1 + K_b C_{surf}), \qquad (1)$$

where k_{H_2O} (s^{-1}) is the rate constant for the reaction in the water—DMF system, C_{surf} (mol L^{-1}) is the concentration of AMC, AMP, or QAMP after subtraction of the CMC for these compounds.

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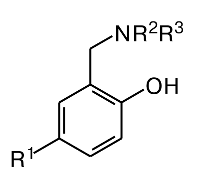
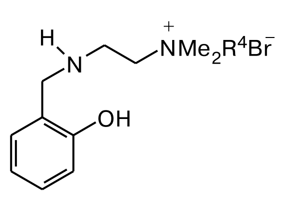
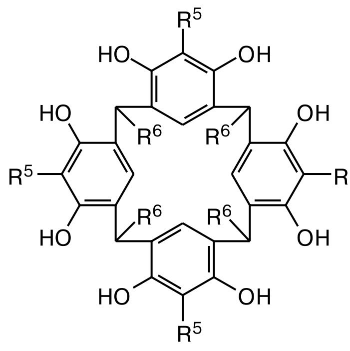
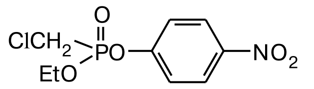

AMP **1—8**


QAMP **9**, **10**


11, AMC **12—14**


15

	R^1	R^2	R^3		R^4	R^5	R^6
1	H	Me	Me	**9**	$C_{14}H_{29}$	—	—
2	C_9H_{19}	Me	Me	**10**	$C_{16}H_{33}$	—	—
3	H	H	Bu	**11**	—	H	C_9H_{19}
4	H	H	C_8H_{17}	**12**	—	CH_2NMe_2	C_9H_{19}
5	H	H	$C_{11}H_{23}$	**13**	—	CH_2NEt_2	$C_{11}H_{23}$
6	H	Bu	Bu	**14**	—	CH_2NEt_2	C_9H_{19}
7	H	C_6H_{13}	C_6H_{13}				
8	H	C_9H_{19}	C_9H_{19}				

Results and Discussion

It is known that AMP[16] and AMC[2] incorporate strong intramolecular hydrogen bonds (IHB) OH...N that are responsible for the formation of zwitterionic species. These compounds are characterized by a complicated system of the acid-base equilibria. As a consequence, the AMP can occur in the form protonated at the N atom (pH 3—5), a neutral or zwitterionic species (pH 6—9), and a phenolate species (pH $\geq$ 9), depending on the pH of a medium. The zwitterionic species of AMC[2] occurs in a wide pH range of 5—10. Hence, the hydrophobized AMP and AMC are the amphiphilic compounds capable of aggregate formation.

We studied by tensiometry the self-assembling of AMC, AMP, and QAMP as a function of their structure and pH in the water—dimethylformamide solution (30 vol.% DMF).[5—8] Table 1 presents the CMC values of the compounds under study at the pH values providing the maximal accumulation of the protonated, zwitterionic, and phenolate species. Compounds **1**, **3**, and **6** containing the Me and Bu radicals do not form aggregates in the range of concentrations $\leq$0.1 mol L^{-1}. Note that compounds **7** and **8** containing two alkyl substituents at the N atom form aggregates only in an acidic medium (CMC = $7 \cdot 10^{-3}$ and $2 \cdot 10^{-3}$ mol L^{-1}, respectively). The CMC values for AMPs **4** and **5** containing one long alkyl chain at the N atom are comparable to the CMC value for

Table 1. Values of CMC for AMP **2—5**, **7**, and **8**, QAMP **9** and **10**, calix[4]resorcinolarene **11** and AMC **12—14** at various pHs (DMF (30 vol.%)—H_2O)

Compound	CMC/mol L^{-1}		
	pH 3—4	pH 7—8	pH 9—11 [a]
2	$4.5 \cdot 10^{-3}$	$5.4 \cdot 10^{-4}$	$4.5 \cdot 10^{-4}$ ($2 \cdot 10^{-4}$)
4	$3.0 \cdot 10^{-3}$	$1.2 \cdot 10^{-3}$	$1.1 \cdot 10^{-3}$ ($8 \cdot 10^{-4}$)
5	$1 \cdot 10^{-3}$	$4.5 \cdot 10^{-4}$	$3.2 \cdot 10^{-4}$
7	$7 \cdot 10^{-3}$	—	—
8	$2 \cdot 10^{-3}$	—	—
9	—	$8 \cdot 10^{-3}$	$6.0 \cdot 10^{-4}$ ($2.8 \cdot 10^{-4}$)
10	—	$1.2 \cdot 10^{-3}$	$3.5 \cdot 10^{-4}$ ($3.5 \cdot 10^{-4}$)
11	—	—	$1.0 \cdot 10^{-4}$ ($6 \cdot 10^{-5}$)
12	$6 \cdot 10^{-5}$	$5 \cdot 10^{-5}$	$8 \cdot 10^{-5}$ ($2 \cdot 10^{-5}$)
12[b]	$1 \cdot 10^{-4}$	—	$1 \cdot 10^{-4}$
12[c]	$8 \cdot 10^{-5}$	—	$8 \cdot 10^{-5}$
12[d]	$1 \cdot 10^{-4}$	—	$1 \cdot 10^{-4}$
13	$2 \cdot 10^{-4}$	$3 \cdot 10^{-4}$	$9 \cdot 10^{-4}$
14	—	$5 \cdot 10^{-5}$	—

[a] The CMC values calculated from the kinetic data presented in Fig. 1 (see Table 2) are shown in parentheses.

[b—d] The CMC values are obtained conductometrically at the DMF concentrations of 10,[b] 50,[c] and 75 vol.%.[d]

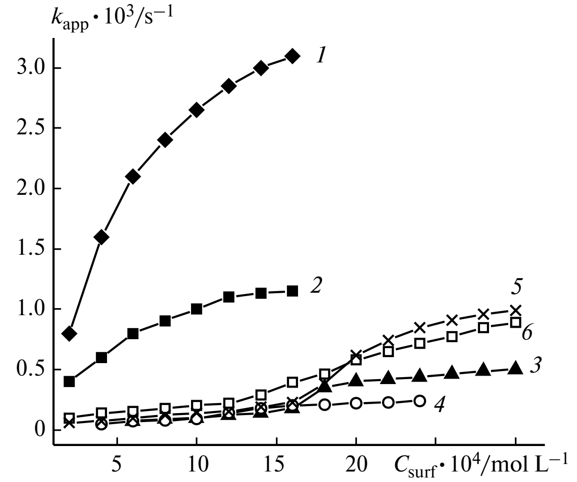


Fig. 1. Apparent rate constants (k_{app}) for the reaction of substrate **15** with compounds **12** (*1*), **11** (*2*), **4** (*3*), **2** (*4*), **9** (*5*), and **10** (*6*) *vs.* their concentrations (C_{surf}); DMF (30 vol.%)—H_2O, pH 10, 298 K.

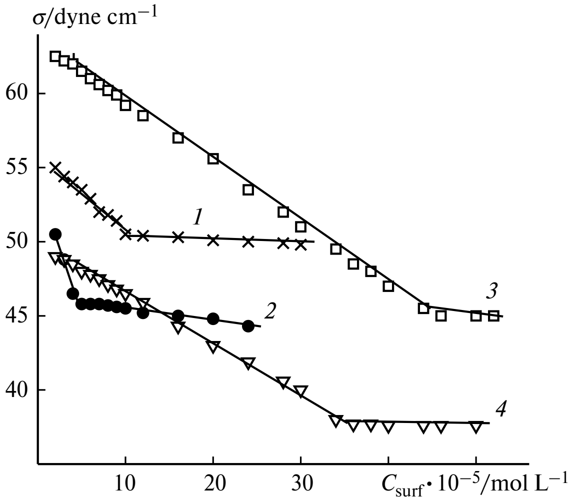


Fig. 2. Variation of the surface tension of the systems as a function of the concentrations of calix[4]resorcinolarene **11** (*1*), AMC **12** (*2*), AMP **4** (*3*), and QAMP **10** (*4*); DMF (30 vol.%)—H_2O.

AMP **2** with a similar radical in the benzene ring. Thus, the number of the alkyl substituents affects substantially the micelle formation of the amphiphilic AMP. The CMC values for QAMPs **9** and **10**, containing a fragment of cationic surfactants, and those for the hydrophobized AMPs **2**, **4**, and **5** slightly differ from each other (see Table 1). However, QAMPs decrease the surface tension (σ) of solutions more sharply than AMPs (Fig. 2). Note that the anion and zwitterionic species of AMP form aggregates easier than the cation species (see Table 1).

The CMC values found for AMCs **12** and **14** (R^6 = C_9H_{19}) are nearly one order in magnitude lower than those for AMP **2** (R^1 = C_9H_{19}) at close pH values. The CMC values for AMC **13** and AMP **5** ($R^1 = R^6 = C_{11}H_{23}$) are of the same order. The higher CMC values for AMC **13** are likely due to less favorable hydrophilic-lypophilic balance compared to AMCs **12** and **14** similarly to the situation observed for gemini surfactants.[17] Contrary to this, AMP and QAMP behave similarly to common surfactants, *i.e.*, the CMC values decrease[5,15,18] with an increase in the hydrocarbon radical length. Hence, the study of the surface tension of solutions showed that the amphiphilic AMP, QAMP, and AMC form aggregates in aqueous DMF (30 vol.%). However, the AMCs whose structures combine the features of both gemini surfactants[17] and amphiphilic macrocycles[5] exhibit specific behavior that is typical of both kinds of surfactants, *i.e.*, the low CMC at comparatively small radical length as typical of macrocycles and an increase in the CMC values with the radical length as is observed for the gemini surfactants.

Table 2. Parameters of the reaction of substrate **15** with AMPs **2** and **4**, QAMPs **9** and **10**, calix[4]resorcinolarene **11**, and AMC **12**

Compound	CMC • 10^4 /mol L^{-1}	k_m • 10^3 /s^{-1}	K_b /L mol^{-1}
2	2	0.52	210
4	8	0.78	180
9	2.8	2.6	100
10	2.7	0.95	170
11	0.6	1.7	2120
12	0.2	4.4	1570

Note. The reaction conditions: pH 10, 25 °C, DMF (30 vol.%)—H_2O.

The study of the electroconductivity of solutions (χ) of AMP **4** and AMC **12** as a function of their concentration by a known procedure[5—8] at various DMF concentrations in aqueous solutions (Fig. 3) showed that AMP **4** forms aggregates only at the DMF concentration of 10 and 30 vol.% and AMC **12** does it at the concentrations of 10—75 vol.%. These compounds are not dissolved in water and do not form micelles in DMF. The CMC values for AMP **4** (pH 10) in binary media increase with an increase in the DMF concentration reaching 6 • 10^{-4} and 1.2 • 10^{-3} mol L^{-1}, respectively. The CMC values for AMC **12** (pH 11) somewhat decrease (to ~50 vol.% DMF) and slightly depend on the pH of solutions (see Table 1, Figs. 3 and 4). In all cases, this compound likely exists in one ionized form, namely, as a zwitterion, as has been shown in the study reported earlier.[2]

To compare the micelle-forming ability of both common surfactants and amphiphilic AMC and AMP, we measured σ and χ of solutions of the cationic surfactant, cetyltrimethylammonium bromide (CTAB), anionic surfactant sodium dodecylsulfate (SDS), and non-ionic surfactant Triton-X-100 as functions of their concentrations in the binary solvent water—DMF in the range of 10—75 vol.%. Figure 4 presents the CMC values for CTAB, SDS, Triton-X-100, and AMC **12** vs. the DMF concentration. These dependences for ionic surfactants, CTAB and SDS, are extremal similarly to a variation[19] in

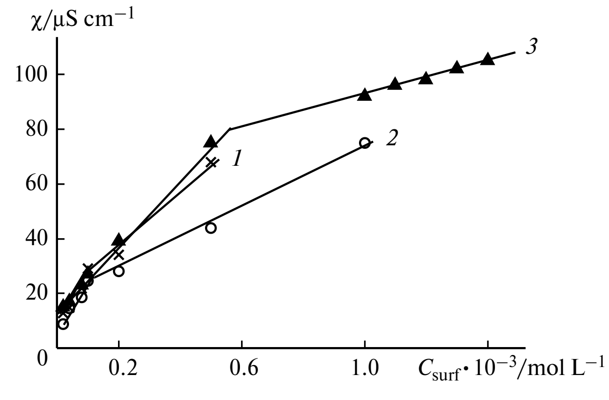


Fig. 3. Electroconductivity (χ) of the systems as a function of the concentrations of AMC **12** (*1*, *2*) and AMP **4** (*3*) at the DMF concentrations of 10 (*1*, *3*) and 75 vol.% (*2*).

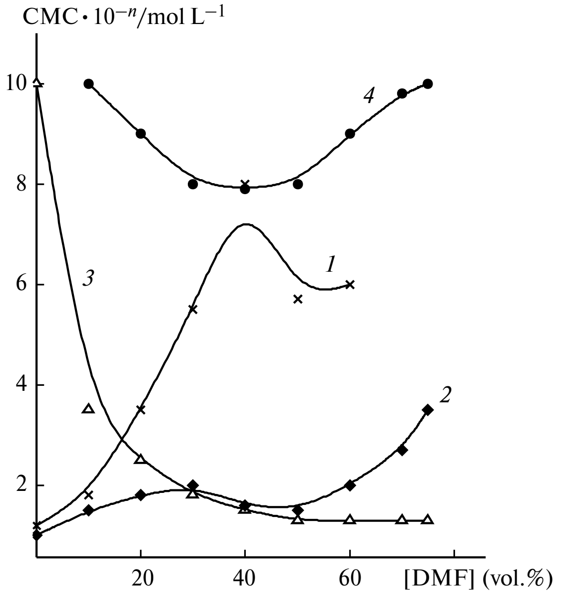


Fig. 4. Variation in CMC of CTAB (*1*, n = 3), SDS (*2*, n = 2), Triton-X-100 (*3*, n = 4), and AMC **12** (*4*, n = 5) with the DMF concentration.

viscosity in the water—DMF system. Like urea, DMF is an organic additive that increases the CMC of aqueous solutions of surfactants due to changes in the hydrogen-bonded structure of water.[18,20] At the same time, as can be seen in Fig. 4, the CMC of the non-ionic surfactant Triton-X-100 decreases by almost one order of magnitude with an increase in the DMF concentration. The stabilization of the micellar state in this case can be explained by inclusion of the DMF molecules, similarly to urea,[18] into the micelle of Triton-X-100. Comparison of the CMC for both common surfactants and AMC (see Fig. 4) gives evidence of the high ability of the latters to self-assembling: the CMC for AMC **12** ($5 \cdot 10^{-5}$—$1 \cdot 10^{-4}$ mol L^{-1}) are considerably lower than those for CTAB and SDS, which alter with the DMF concentration in the regions of $(2—8) \cdot 10^{-3}$ and $(1—3) \cdot 10^{-2}$ mol L^{-1}, respectively. As was mentioned above, low CMC values ($1 \cdot 10^{-4}$—$5 \cdot 10^{-6}$ mol L^{-1}) are typical of amphiphilic macrocyclic compounds.[5] In addition, contrary to common surfactants, the CMC for AMC are independent in fact of the viscosity of the binary mixture water—DMF. This can indicate the higher viscosity of the AMC aggregates compared to micelles of common surfactants and agrees with the data obtained[7] in the study of the micelle formation in water-soluble calix[6]arenes, as well as the different structure of the aggregates. It has been shown for the AMC aggregates,[7] that the lyotropic liquid-crystal structures are formed from the bilayers as precursors in the CMC region, whereas common surfactants form spherical micelles in the CMC region.[21] A trend to the CMC lowering for AMC **12** with an increase in the DMF concentration up to ~50 vol.% can indicate the inclusion of the solvent molecules in the aggregate structure.

Difference in the behavior of the AMC aggregates was also found in the study of the effect of the additives of triphenylphosphate (**16**) and *p*-nitrophenol (**17**) ($C_{16,17} = 1 \cdot 10^{-4}$ mol L^{-1}), which simulate the interaction of the EPA with micelles in the course of chemical reaction, on the self-assembling of AMC **14**, AMP **4**, and Triton-X-100 in the 30% aqueous DMF. Additional inflection points appear on the plots of the surface tension vs. the concentrations of AMP or Triton-X-100 (CMC-2), which point to micellar transitions, *i.e.*, the formation of different kinds of micelles;[22] CMC-1 decreases in these systems by 3—4 times and become equal to $4 \cdot 10^{-4}$ and $4 \cdot 10^{-5}$ mol L^{-1}, respectively (Fig. 5). The effect of compounds **16** and **17** on aggregation in these systems is the same and does not influence the AMC aggregation in fact (see Fig. 5); CMC in the systems remains unchanged.

Earlier we have shown by the ^{31}P NMR that AMP and AMC in the water-alcohol and micellar solutions of surfactants catalyze the hydrolysis of EPAs. In particular, the interaction of AMP[23] and AMC[2,3] with *p*-nitrophenyl-diphenyl phosphate occurs in two stages: in the first stage, phosphorylated AMP or AMC are formed, which are further hydrolyzed to the corresponding acids in the slower stage. A drop in the intensity of the substrate signal in the ^{31}P NMR spectra is observed. The drop is accompanied by the appearance of the signal of phosphorylated intermediate, whose intensity initially increases and then decreases. Simultaneously, the signal of the acid arises and its intensity increases in time.

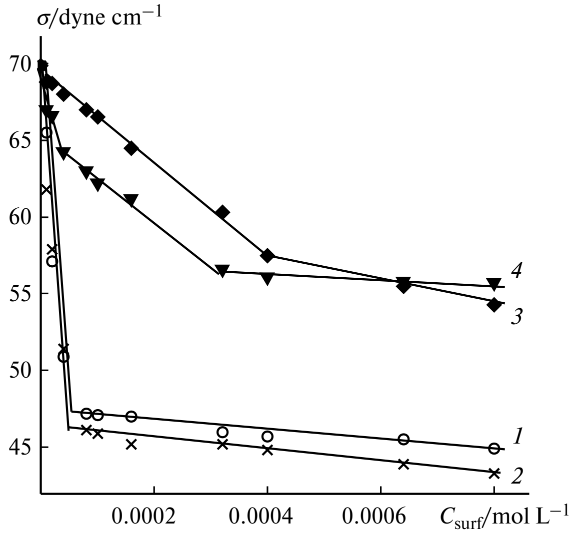


Fig. 5. Variation of the surface tension (σ) of the systems as a function of the concentrations of AMC **14** (*1*, *2*), AMP **4** (*3*), and Triton-X-100 (*4*) in the absence (*1*) and in the presence (*2—4*) of compound **16**; $C_{16} = 1 \cdot 10^{-4}$ mol L^{-1}, DMF (30 vol.%)—H_2O.

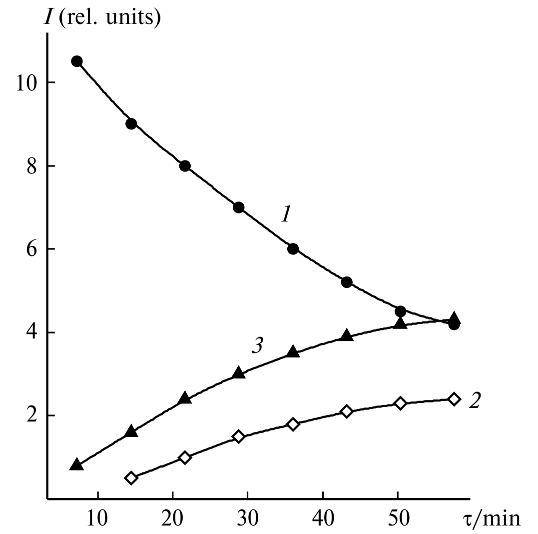


Fig. 6. Variation of the intensities of the ^{31}P NMR spectra at δ 17.6 (*1*), 16.6 (*2*), and 13.6 (*3*) in the course of the reaction of QAMP **9** ($4 \cdot 10^{-2}$ mol L^{-1}) with substrate **15** ($4 \cdot 10^{-3}$ mol L^{-1}) at pH 8 and temperature 311 K; DMF (30 vol.%)—H_2O.

The ^{31}P NMR study of the reaction of **15** with non-micelle-forming AMP **6** and micelle-forming AMP **4**, QAMP **9**, and AMC **13** in the 30% aqueous DMF showed that the EPA hydrolysis in the micellar systems based on AMP, QAMP, and AMC occurs likely through different mechanisms. Irrespectively of the aggregation state, AMPs interact with substrate **15** (δ 17.3 ppm) *via* the above mechanism to form phosphorylated AMP (δ 16.6), followed by its hydrolysis to the acid (δ 13.3). The examination of the ^{31}P NMR spectra of the reaction mixtures of **15** with QAMP (Fig. 6) recorded at different reaction times showed that the interaction occurs simultaneously through two pathways, *i.e.*, the transesterification and hydrolysis of the substrate occur in parallel. The hydrolysis of compound **15** in the presence of AMC **12** is not accompanied by the formation of an intermediate. The difference in the reaction mechanisms of compound **15** with AMP and AMC is likely due to the fact that the phosphorylation of the macrocyclic phenols AMCs proceeds more slowly than that of simple phenols.[24] The substrate bound by the AMC aggregate is hydrolyzed faster than it undergoes transesterification.

The spectrophotometric study of the reaction kinetics for substrate **15** with calix[4]resorcinolarene **11**, AMC **12**, AMPs **2** and **4**, and QAMPs **9** and **10** confirmed the formation of aggregates of these compounds, namely, the kinetic curves have a "saturation" shape that is typical of micelle-catalyzed reactions[15] (see Fig. 1). A similar plot of k_{app} against the concentration of the non-micelle-forming AMP **3** is linear, and the bimolecular rate constant for this reaction is 0.2 L mol^{-1} s^{-1}. Table 2 presents the parameters of the micelle-catalyzed reaction of substrate **15** with the compounds under study, namely, CMC, K_b, and k_m. As follows from the comparison of the CMC values obtained by tensiometry, conductometry, and the kinetic method (see Tables 1 and 2), they are lower in the latter case. This is likely due to the effect of the substrate additives on the micellar system. The reactivities of functional aggregates formed by calix[4]resorcinolarenes, AMP, and QAMP, which hydrolize **15** *via* different mechanisms, differ dramatically (see Fig. 1 and Table 2). The CMC of the aggregates of calix[4]resorcinolarenes is one order of magnitude lower and K_b is one order of magnitude higher than those for AMP.

Hence, the aggregates of macrocyclic amphiphiles, calix[4]resorcinolarenes containing hydroxy and amino groups, are formed much more easily and bind substrates more strongly than the aggregates of *o*-aminomethylphenols; they exhibit much higher hydrolytic activity at low concentrations.

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