

Synergistic Effect in the Catalysis of Arylsulfonylation of Phenols and Arenecarboxylic Acids by the System Pyridine *N*-Oxide–Triethylamine in Dioxane

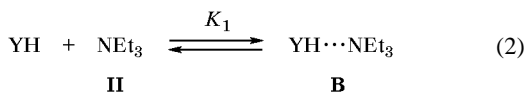
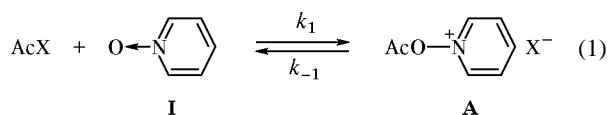
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Received April 26, 2000

Abstract—The kinetics of arylsulfonylation of substituted nitrophenols and 2-(4-dimethylaminophenylazo)-benzoic acid with 4-toluenesulfonyl chloride in dioxane in the presence of pyridine *N*-oxide–triethylamine were studied. The observed synergistic effect was explained by interaction of two intermediates, *N*-tosyloxy-pyridinium chloride (nucleophilic catalysis) and phenol(or acid)–triethylamine complex.

In the previous studies [1–5] we found that the catalytic system pyridine *N*-oxide (**I**)–triethylamine (**II**) exerts a powerful synergistic effect in acyl transfer reactions. By kinetic study of the phosphorylation of substituted phenols [$\text{AcX} = (\text{EtO})_2\text{POCl}$]; $\text{YH} = \text{ArOH}$] in dioxane [5] we obtained direct proofs that the observed synergistic effect is related to the high rate of the reaction between intermediates **A** and **B** which are responsible for nucleophilic and general base catalysis, respectively [reactions (1)–(3)].



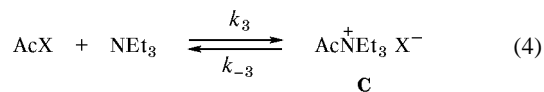
AcX is an acylating agent, and YH is a nucleophilic reagent.

It should be emphasized that, depending on the acidity and concentration of substituted phenol, the rate-determining stage changes from reaction (3) for less acidic phenols to (1) for more acidic phenols. Strong acceleration effects produced by mixtures of catalysts attract interest from the practical viewpoint, e.g., in the development of chemical systems for decomposition of ecotoxicants [6]. Therefore, studies

of synergistic effect in reactions of other substrates as potential models of ecotoxicants seem to be important.

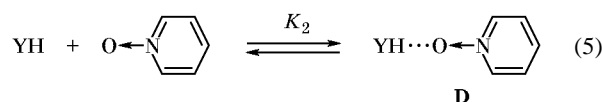
In the present work we examined the kinetics of sulfonylation of 4-nitrophenol (**IIIa**), 2,5-dinitrophenol (**IIIb**), 2,4-dinitrophenol (**IIIc**), and 2-(4-dimethylaminophenylazo)benzoic acid (**IV**) with 4-toluenesulfonyl chloride in the presence of pyridine *N*-oxide–triethylamine in dioxane at 25°C. As previously [5], we follow the concept of synergistic effect given by Eqs. (1)–(3).

The sulfonylation of phenols and carboxylic acid **IV** with 4-toluenesulfonyl chloride in the presence of triethylamine alone was studied previously [5]. It was found that the reaction follows a general base catalysis mechanism with participation of complex **B** and substrate AcX, regardless of the acidity of the YH reagent. Nucleophilic activation of the substrate according to Eq. (4) does not contribute much to the overall rate of the process (cf. [7]).



Therefore, when the sulfonylation is carried out in the presence of a mixture of catalysts **I** and **II**, any pathway involving intermediate **C** can be neglected. The rate of sulfonylation of phenols and carboxylic acids with 4-toluenesulfonyl chloride in the presence of pyridine *N*-oxide (**I**) as a single catalyst in dioxane is so low that it cannot be measured (provided that

the concentration of **I** is comparable with the concentration of catalyst **II**). This is not surprising, for pyridine *N*-oxide (**I**) showed a weak catalytic effect as compared to triethylamine (**II** in the benzoylation [2, 3] and phosphorylation [5] of weak nucleophiles such as phenols and carboxylic acids. However, in the acylation of stronger bases (such as aromatic amines) the catalytic activity of pyridine *N*-oxide exceeds that of triethylamine by more than an order of magnitude [8]. The reason is that in reactions with weak nucleophiles substrate activation according to Eq. (1) with formation of intermediate **A** is insufficient to reduce the energy barrier to a considerable extent. Here, simultaneous activation of both the substrate and the nucleophile YH is necessary. However, H-complex **D** formed by Eq. (5) from *N*-oxide **I** and reagent YH [9] does not ensure effective proton transfer in the reactions $\text{AcX} + \mathbf{D}$ or $\mathbf{A} + \mathbf{D}$ because of the low basicity of catalyst **I** [10].



Therefore, we can exclude pathways involving formation of complex **D** when considering catalytic process in the presence of bases **I** and **II**. It seems reasonable to prefer pathway (3) which involves the reaction between complexes **A** and **B**. In order to calculate the equilibrium concentrations of these complexes, the equilibrium constants $K_{\text{eq}} = k_1/k_{-1}$, K_1 , and K_2 must be known. The constant K_{eq} cannot be measured; according to [11–13], it can be estimated at about <0.1 l/mol. Then, the equilibrium concentration of intermediate **A** is given by Eq. (6):

$$[\mathbf{A}] = K_{\text{eq}} [\text{AcX}]_0 [\mathbf{I}]_0, \quad (6)$$

where the subscript “0” refers to the initial concentration. In the general case, the equilibrium concentrations of complexes formed by triethylamine (**II**) with phenols **III** and acid **IV** should be calculated by Eq. (7), taking into account equilibria (2) and (5):

$$[\mathbf{B}] = \frac{K_1 [\mathbf{II}]_0 [\text{YH}]_0}{1 + K_1 [\mathbf{II}]_0 + K_2 [\mathbf{I}]_0} = \alpha_{\mathbf{II}} [\text{YH}]_0. \quad (7)$$

Here, $\alpha_{\mathbf{II}}$ is the fraction of YH bound into complex **B** [Eq. (8)]:

$$\alpha_{\mathbf{II}} = \frac{K_1 [\mathbf{II}]_0}{1 + K_1 [\mathbf{II}]_0 + K_2 [\mathbf{I}]_0}. \quad (8)$$

The formation constants of the H-complexes of **II** with phenols **IIIa–IIIc** and acid **IV** (K_1) were determined by us previously [3, 5] using UV spectroscopy, and the corresponding K_2 values for the complexes with pyridine *N*-oxide (**I**), using IR spectroscopy (see Experimental). The results are given in Table 1 together with the $\text{p}K_a(\text{H}_2\text{O})$ values of proton donors.

Except for the complex with 4-nitrophenol (**IIIa**), K_1 values are considerably greater than K_2 (cf. K_1/K_2 ratios in Table 1). This may be due to predominant formation of ionic complexes $\text{Et}_3\text{NH}^+ \cdots \text{Y}^-$ by proton donors YH with triethylamine (**II**) and of molecular complexes $\text{C}_5\text{H}_5\text{NO} \cdots \text{HY}$ with pyridine *N*-oxide (**I**). The complexes of 4-nitrophenol (**IIIa**) with both amine **II** and *N*-oxide **I** have a molecular structure, and the K_1 and K_2 values are comparable.

It was surprising that K_2 values decrease as the acidity of proton donor (phenols **IIIa–IIIc**) increases. The complex formation constants K_1 for amine **II** change in the normal way: they increase as the acidity of nucleophile rises. This difference, as well as considerably lower values of K_2 relative to K_1 for phenols **IIIb** and **IIIc**, may be attributed to competing formation of intra- and intermolecular hydrogen bonds. When a molecular complex is formed, the negative contribution of intramolecular hydrogen bond in the initial state of proton donor is greater than in the formation of ionic complex (with amine **II**).

The values of K_1 and K_2 given in Table 1 were used to calculate the concentration of complex **B** and fraction of the nucleophile bound thereto by Eqs. (7) and (8). Except for the reaction with 4-nitrophenol (**IIIa**), the term $K_2[\mathbf{I}]_0$ in the denominator is almost insignificant (taking into account the initial concentrations $[\mathbf{I}]_0$ and $[\mathbf{II}]_0$).

The rate of reactions with 4-nitrophenol **IIIa** and acid **IV** in the presence of catalytic system **I–II** was monitored following the disappearance of the free phenol (or acid). The initial concentrations met the condition $[\text{AcX}]_0 \gg [\text{YH}]_0 \ll ([\mathbf{I}]_0 + [\mathbf{II}]_0)$. The apparent pseudofirst-order rate constants k_{ap} (s^{-1}) did not change during the process.

In keeping with the mechanism of synergistic effect, involving reaction (3) as the rate-determining stage, the gain Δk_{ap} (s^{-1}) in the reaction rate is given by Eq. (9) [taking into account Eqs. (6)–(8)] [2, 3, 5]:

$$\begin{aligned} \Delta k_{\text{ap}} &= K_{\text{eq}} k_2 \alpha_{\mathbf{II}} [\text{AcX}]_0 [\mathbf{I}]_0; \\ \Delta k_{\text{ap}} &= k_{\text{ap}} - k_{\text{ap}}^{\mathbf{II}}. \end{aligned} \quad (9)$$

Here, $k_{\text{ap}}^{\mathbf{II}}$ is the apparent pseudofirst-order rate constant in the presence of amine **II** alone. It was

Table 1. Formation constants of H-complexes of phenols **IIIa–IIIc** and acid **IV** with triethylamine (**II**) (K_1) and pyridine *N*-oxide (**I**) (K_2) (dioxane, 25°C)

Proton donor	p <i>K</i> _a (H ₂ O)	<i>K</i> ₁ , l/mol [3, 5]	<i>K</i> ₂ , l/mol	<i>K</i> ₁ / <i>K</i> ₂	$\nu(\text{OH})$, cm ⁻¹	ϵ , l mol ⁻¹ cm ⁻¹
IIIa	7.15	3.17±0.09	6.08±0.83	0.52	3226	272±4
IIIb	5.22	132±19	3.08±0.08	43	3262	110±5
IIIc	4.11	1095±55	1.80±0.16	608	3235	109±2
IV	5.22	6.9±1.5	0.90±0.05 [3]	8.8		

Table 2. Kinetic parameters of the reactions of 4-toluenesulfonyl chloride and diethyl chlorophosphate with nitrophenols **IIIa–IIIc** and acid **IV** in the presence of catalyst mixture **I/II** (dioxane, 25°C)

Comp. no.	<i>K</i> ₁ , l/mol [5]	(EtO) ₂ POCl [5]			4-MeC ₆ H ₄ SO ₂ Cl		
		$k_4 = k_2 K_{\text{eq}}$, l ² mol ⁻² s ⁻¹	$k_1 \times 10^2$, l mol ⁻¹ s ⁻¹	$(k_2/k_{-1}) \times 10^{-4}$, l/mol	$k_4 = k_2 K_{\text{eq}}$, l ² mol ⁻² s ⁻¹	$k_1 \times 10^2$, l mol ⁻¹ s ⁻¹ ^a	$(k_2/k_{-1}) \times 10^{-4}$, l/mol
IIIc	1095±55	–	2.03±0.28	–	370±140	3.7±1.6	10.1±1.2
IIIb	132±19	>103	2.38±0.08	>10	310±120	7.9±3.0	3.9±0.9
IIIa	3.17±0.09	224±17	–	0.903	40.2±5.7	–	0.69 ^b
IV	6.9±1.5 [3]	290±12	–	1.05	42.6±6.8	–	0.74 ^b

^a The average k_1 value for the two series is $(5.8 \pm 2.3) \times 10^{-3}$ l mol⁻¹ s⁻¹.

^b Calculated using the average k_1 value.

calculated by the formula $k_{\text{ap}}^{\text{II}} = k_2^0 \alpha_{\text{II}} [\text{AcX}]_0$, where $k_2^0 = 0.0723 \pm 0.0068$ and 0.0767 ± 0.0086 l mol⁻¹ s⁻¹ for phenol **IIIa** and acid **IV**, respectively [7].

Figure 1 illustrates the synergistic effect in the catalytic system pyridine *N*-oxide–triethylamine. It is seen that k_{ap} values can be regarded as a quantitative measure of the synergistic effect since they differ only slightly from Δk_{ap} .

Equation (9) was checked for validity in two series of experiments. The first series was carried out at a constant substrate concentration ($[\text{AcX}]_0 = \text{const}$) and variable overall concentration of the catalysts, and in the second series the overall concentration of the catalysts was maintained constant ($[\text{I}]_0 + [\text{II}]_0 = \text{const}$), while the substrate concentration was varied. A satisfactory agreement was observed between the values of $k_4 = K_{\text{eq}} k_2$ calculated by Eq. (9) for phenol **IIIa** and acid **IV** on variation of $[\text{AcX}]_0$ from 15.3×10^{-2} to 1.53×10^{-2} M and of $([\text{I}]_0 + [\text{II}]_0)$ from 2.3×10^{-2} to 9.3×10^{-2} M (total of 30 experiments with acid **IV** and 14 experiments with phenol **IIIa** were performed). The average values of k_4 are given in Table 2.

The rate of sulfonylation of dinitrophenols **IIIb** and **IIIc** was monitored following the disappearance of complex **B** at the same reactant concentration ratio

as above: $[\text{AcX}]_0 \gg [\text{YH}]_0 \ll ([\text{I}]_0 + [\text{II}]_0)$. Unlike the preceding system where the concentration of complex **B** changed exponentially with time; an analogous pattern for dinitrophenols was observed only at a definite part of the kinetic curve (Fig. 2). In the reactions with phenols **IIIb** and **IIIc**, a clearly exponential dependence starts, respectively, when ~80 or ~30% conversion of the complex is attained (Fig. 2,

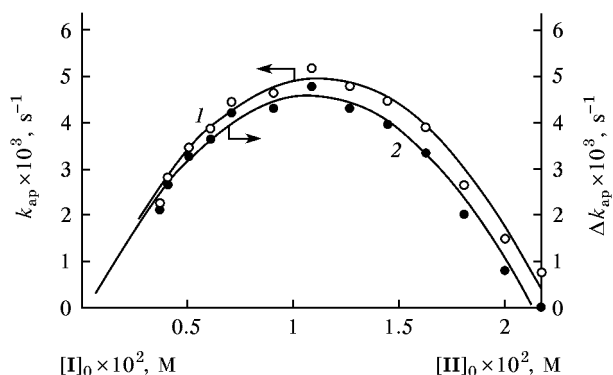


Fig. 1. Plots of (1) k_{ap} and (2) Δk_{ap} versus composition of the catalytic system *N*-oxide **I**–amine **II** ($[\text{I}]_0 + [\text{II}]_0 = \text{const}$) in the reaction of 4-toluenesulfonyl chloride (**AcX**) with acid **IV** (dioxane, 25°C, $[\text{I}]_0 + [\text{II}]_0 = 0.0217$, $[\text{AcX}]_0 = 0.0693$ M).

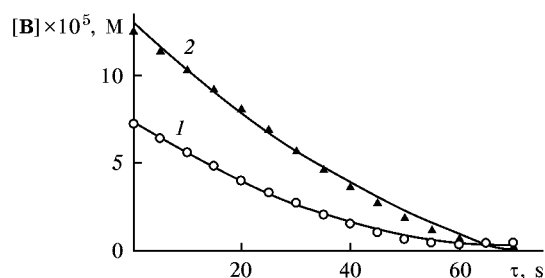


Fig. 2. Kinetic curves for the disappearance of complex **B** in the reaction of 4-toluenesulfonyl chloride (AcX) with dinitrophenols **IIIc** and **IIIb** in the presence of a mixture of catalysts **I** and **II** (dioxane, 25°C): (1) 2,4-dinitrophenol (**IIIc**), $[\text{AcX}]_0 = 0.0348$, $[\text{I}]_0 = 0.0248$, $[\text{II}]_0 = 0.0328$ M; (2) 2,5-dinitrophenol (**IIIb**), $[\text{AcX}]_0 = 0.0177$, $[\text{I}]_0 = 0.0583$, $[\text{II}]_0 = 0.0302$ M.

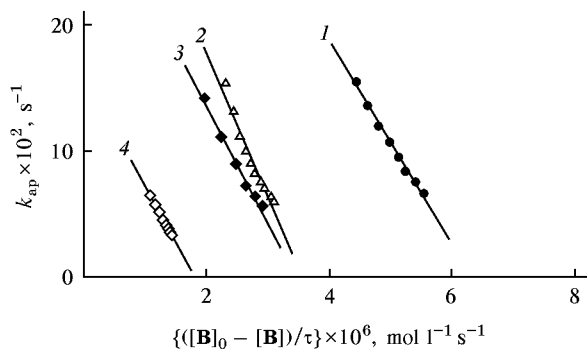


Fig. 3. Plots of k_{ap} versus $([\text{B}]_0 - [\text{B}])/\tau$ for the reaction of 4-toluenesulfonyl chloride (AcX) with 2,4-dinitrophenol (**IIIc**) in the presence of catalytic system **I–II** (dioxane, 25°C); $[\text{AcX}]_0 = 0.0216$, $[\text{II}]_0 = 0.0328$ M; $[\text{I}]_0$: (1) 0.0745, (2) 0.0647, (3) 0.0497, (4) 0.0199 M.

curves 1 and 2). The initial parts of the kinetic curves (up to ~80 and ~30% conversion, respectively) are described by pseudofirst-order equation (10) [5]:

$$[\text{B}] = [\text{B}]_0 - \alpha_{\text{II}} k^0 \tau = \alpha_{\text{II}} [\text{YH}]_0 - k \tau. \quad (10)$$

Here, $[\text{B}]_0$ is the initial concentration of complex **B** ($\tau = 0$), k_0 ($\text{mol l}^{-1} \text{s}^{-1}$) is the pseudozero-order rate constant in YH, and $k = k_0 \alpha_{\text{II}}$. Analogous kinetic curves were observed previously in the phosphorylation of the same phenols with diethyl chlorophosphate in dioxane in the presence of the same catalytic system [5]. The observed pattern was proved to result from change of the rate-determining stage, depending on the acidity of phenol and its concentration. At the initial linear part of the kinetic curve (Fig. 2) the rate-determining stage is the reaction of AcX with *N*-oxide **I** to give intermediate **A** (k_1), and the exponential part

corresponds to the rate-determining reaction between complexes **A** and **B** (k_2). Therefore, the kinetic curves were treated as described in [5]. The reaction rate at the initial linear part is given by Eq. (11):

$$k = k_1 [\text{AcX}]_0 [\text{I}]_0 \alpha_{\text{II}}. \quad (11)$$

Equation (11) was used to analyze the kinetic data obtained for dinitrophenol **IIIb** for which the kinetic plot in the coordinates of Eq. (10) was linear up to ~80% conversion of the substrate. Table 3 summarizes the results of two series of experiments. One of these was carried out at a constant overall concentration of catalysts **I** and **II** with variation of $[\text{AcX}]$, and the other, at constant concentrations of AcX and **II** with variation of $[\text{I}]_0$. In the first series experiments we observed a linear relation between the values of k and substrate concentrations $[\text{AcX}]$ since the k_1 value calculated by Eq. (11) remained constant (Table 3). The second series of experiments was characterized by a poorer reproducibility of the results, presumably because of high apparent reaction rates. Nevertheless, the value of k_1 calculated from these data was comparable with that obtained in the first series. The average k_1 value is $(7.9 \pm 3.0) \times 10^{-3} \text{ l mol}^{-1} \text{ s}^{-1}$.

Insofar as the linear parts of the kinetic plots for sulfonylation of dinitrophenol **IIIc** are fairly short (up to ~30% conversion; see Fig. 2, plot 1), they were treated according to Eq. (12) which takes into account change of the rate-determining stage during the process. Under the constraint $\alpha_{\text{II}} \rightarrow 1$ the expression for the apparent pseudofirst-order rate constant k_{ap} is as follows [5]:

$$k_{\text{ap}} = k_1 \frac{k_2}{k_{-1}} [\text{AcX}]_0 [\text{I}]_0 - \frac{k_2}{k_{-1}} \frac{[\text{B}]_0 - [\text{B}]}{\tau}. \quad (12)$$

Figure 3 gives examples of graphical representation of Eq. (12). The k_1 and k_2/k_{-1} values for various concentrations of the components ($\alpha_{\text{II}} \rightarrow 1$) are summarized in Table 4. The rate constant k_1 was thus estimated at $(3.7 \pm 1.6) \times 10^{-3} \text{ l mol}^{-1} \text{ s}^{-1}$. With regard to the error in determination of this quantity, the values of k_1 obtained for phenols **IIIb** and **IIIc** with different acidities are fairly similar, which is consistent with the postulated change of the rate-determining stage during sulfonylation of dinitrophenols **IIIb** and **IIIc** in the presence of catalyst mixture **I/II**.

From Eq. (12) we can calculate the ratio k_2/k_{-1} and the rate constant $k_4 = k_2 K_{\text{eq}}$. As applied to the sulfonylation of dinitrophenol **IIIc**, the data for the entire kinetic curves were used in the calculation (Table 4),

Table 3. Rate constants k and k_1 of the reaction of 4-toluenesulfonyl chloride (AcX) with 2,5-dinitrophenol (**IIIb**) in the presence of the catalytic system pyridine *N*-oxide (**I**)–triethylamine (**II**); $[\mathbf{II}]_0 = 0.0302$ M, $\alpha_{\mathbf{II}} = 0.79$,^a dioxane, 25°C

$[\text{AcX}]_0 \times 10^3$, M	$[\mathbf{I}]_0 \times 10^3$, M	$k \times 10^6$, mol l ⁻¹ s ⁻¹	$k_1 \times 10^3$, l mol ⁻¹ s ⁻¹	$(k_2/k_{-1}) \times 10^{-4}$, ^b l/mol
17.7	11.6	1.62	9.95	5.04
35.5	11.6	3.21	9.84	4.44
53.2	11.6	4.42	9.03	3.38
70.9	11.6	6.22	9.53	4.37
88.7	11.6	6.49	7.96	3.92
$k_1 = (9.2 \pm 0.9) \times 10^{-3}$, $k_2/k_{-1} = (4.2 \pm 0.8) \times 10^4$				
17.7	5.75	0.843	9.68	5.10
17.7	11.5	1.28	7.94	3.23
17.7	35.0	2.28	4.66	3.23
17.7	46.7	3.89	5.95	4.28
17.7	58.8	3.46	4.21	
$k_1 = (6.5 \pm 2.8) \times 10^{-3}$, $k_2/k_{-1} = (3.5 \pm 1.3) \times 10^4$				

^a Calculated by Eq. (8) using K_1 [5] and K_2 values (Table 1) determined by spectrophotometry.^b Estimated by Eq. (12) from the data corresponding to the final parts of the kinetic curves (more than ~80% conversion of complex **B**).**Table 4.** Values of k_1 and k_2/k_{-1} for the reaction of 4-toluenesulfonyl chloride (AcX) with 2,4-dinitrophenol (**IIIc**) in the presence of the catalytic system pyridine *N*-oxide (**I**)–triethylamine (**II**); $[\mathbf{II}]_0 = 0.0328$ M, $\alpha_{\mathbf{II}} \rightarrow 1$,^a dioxane, 25°C

$[\text{AcX}]_0 \times 10^3$, M	$[\mathbf{I}]_0 \times 10^3$, mol/l	$k_1 \times 10^3$, l mol ⁻¹ s ⁻¹	$(k_2/k_{-1}) \times 10^{-4}$, l/mol	$[\text{AcX}]_0 \times 10^3$, M	$[\mathbf{I}]_0 \times 10^3$, mol/l	$k_1 \times 10^3$, l mol ⁻¹ s ⁻¹	$(k_2/k_{-1}) \times 10^{-4}$, l/mol
17.3	24.8	3.09	10.8	21.6	9.95	3.46	9.7
30.3	24.8	2.92	11.5	21.6	19.9	4.32	8.5
43.3	24.8	3.08	11.0	21.6	34.0	2.98	9.4
65.0	24.8	5.40	9.8	21.6	49.7	3.23	9.2
82.3	24.8	5.80	–	21.6	64.7	3.83	–
21.6	4.98	3.14	11.0	21.6	74.5	4.05	–
$k_1 = (3.7 \pm 1.6) \times 10^{-3}$, $k_2/k_{-1} = (10.1 \pm 1.2) \times 10^4$							

^a Calculated using K_1 [5] and K_2 values (Table 1) determined by spectrophotometry.

whereas in the case of dinitrophenol **IIIb** a rough estimation was performed on the basis of the data corresponding to the “ends” of the kinetic plots (i.e., after ~80% conversion of complex **B**; Table 3). The average results of all calculations are collected in Table 2. For comparison, the data for analogous reactions with diethyl chlorophosphate as AcX reagent are also given. Although 4-toluenesulfonyl chloride is approximately an order of magnitude less reactive than diethyl chlorophosphate (cf. k_1 in Table 2), the kinetic relations holding in the examined catalytic

systems are qualitatively similar: change of the rate-determining stage in nucleophilic catalysis is observed with the same dinitrophenols **IIIb** and **IIIc**.

EXPERIMENTAL

The electron absorption spectra were measured on a Specord UV-Vis spectrophotometer. The IR spectra were obtained on a Perkin–Elmer FIR-180 instrument. The reaction kinetics was monitored by spectrophotometry, following the disappearance of the nucleophile

(nitrophenol **IIIa** and acid **IV**) or its complex with triethylamine (**II**) (dinitrophenols **IIIb** and **IIIc**); the procedure for kinetic measurements was described previously [5].

The complex formation constants K_2 for phenols **IIIa–IIIc** and pyridine *N*-oxide (**I**) were determined by IR spectroscopy from the change in the intensity of OH stretching vibration band (the corresponding values of ϵ and ν_{OH} are given in Table 1), according to the procedure described in [14]. In all cases, the fulfillment of the Bouguer–Lambert–Beer law was checked.

The pseudofirst-order rate constants k_{ap} (s^{-1}) were calculated by the formula

$$k_{ap} \tau = \ln[(D_{\infty} - D_0)/(D_{\infty} - D_{\tau})], \quad (13)$$

where D_0 , D_{τ} , and D_{∞} are the optical densities at the initial moment ($\tau = 0$), at time τ , and by the end of the reaction. The linear dependences were treated by the least-squares procedure.

Dioxane [15], pyridine *N*-oxide [16], and triethylamine [17] were purified by known procedures. 4-Toluenesulfonyl chloride was twice recrystallized from petroleum ether. Phenols **IIIa–IIIc** were purified by the procedure reported in [5]. 2-(4-Dimethylaminophenylazo)benzoic acid (**IV**) was recrystallized from toluene.

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