UNSATURATED HYDANTOIN DERIVATIVES.

19.* EFFECT OF TEMPERATURE ON THE RATE OF ALKALINE HYDROLYSIS OF 2-THIOXO-5-ARYLIDENEHYDANTOINS AND SOME OF THEIR DERIVATIVES

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The effect of temperature on the rate of alkaline hydrolysis of the monoanions of 2-thioxo-5-arylidenehydantoins was studied. The investigated series obeys an isokinetic law with isokinetic temperature β = 321 ± 10°K. The kinetic data obtained constitute evidence that the mechanisms of the hydrolysis of 2-thioxo-5-arylidenehydantoins and their oxygen analogs are identical. A change in the solvation of the starting and transition states is the principal reason for the change in the reactivities of 5-arylidene-2-thioxohydantoins under the influence of the substituent and the temperature as compared with 5-arylidenehydantoins, the N- and S-methyl derivatives, and 4-thioxo-5-benzylidenehydantoin.

We have previously shown that the alkaline hydrolysis of 2-thioxo-5-arylidenehydantoins [1] and their 2-oxo analogs [2, 3] is a bimolecular reaction. Depending on the alkali concentration in solution, the reactive forms of the substrate are nonionized molecules or the corresponding monoanions. Regardless of the form in which the hydantoin reacts, the rate-determining step is attack on the C_4=O group of the hydantoin ring by hydroxide ion. During a study of the effect of the temperature on the rate of alkaline hydrolysis of the monoanions of 5-arylidenehydantoins it was observed [3] that an isokinetic dependence with isokinetic temperature β = 320 ± 10°K is satisfied. The reason for the change in the reactivities of these compounds as the temperature is varied is the change in the ratio of the enthalpy and entropy contributions to the change in the free energy of activation due primarily to solvation processes. Up until now, the differences in the rates and mechanisms of hydrolysis of amides and their thioxo analogs, including cyclic compounds [4], and, in particular, hydantoins and thioxohydantoins [5] have been the subject of debate. In this connection, we undertook a study of the effect of the temperature on the rate of alkaline hydrolysis of the monoanions of 2-thioxo-5-arylidenehydantoins I-V, 3-mono- and 1,3-dimethyl-2-thioxo-5-

TABLE 1. Rate Constants for the Alkaline Hydrolysis of the Monoanions of 5-Arylidenethioxohydantoins I-VII, X, and Their Derivatives VIII and IX

Compound	R	λ_{max}, nm	T, K											
			368	363	358	353	343	338	335	328	323	313	303	293
			$k \cdot 10^4$, liters/mole • sec											
I	NO_2	457	37,20	25,00	17,10	11,52	4,68	2,80	1,83	1,06	—	—	—	—
II	Br	392	18,10	13,12	9,38	6,55	3,32	2,14	1,48	0,97	—	—	—	—
III	H	380	13,65	11,10	7,39	5,33	2,79	1,91	1,36	0,92	—	—	—	—
IV	CH_3	386	10,92	8,23	6,28	4,76	2,67	1,95	1,28	0,90	—	—	—	—
V	OCH_3	382	8,00	6,76	5,13	3,95	2,33	1,64	1,17	0,86	—	—	—	—
VI	H	360	—	7,41	—	3,19	1,66	—	0,69	—	0,32	—	—	—
VII	H	364	—	—	—	—	2340,00	—	1270,00	—	687,0	289,0	—	74,0
VIII	H	230	—	625,00	422,00	260,00	109,00	73,00	51,20	—	182,10	—	167,0	—
IX	H	361	—	—	—	—	1900,00	—	1290,00	—	832,00	533,0	—	204,0
X	H		14,03	9,98	7,16	4,62	2,38	1,44	1,00	0,66	—	—	341,0	—

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*See [1] for communication 18.

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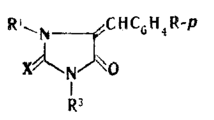
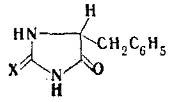
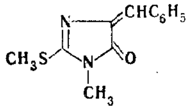
TABLE 2. Equations ($\log K = \log A - 10^3 \cdot BT^{-1}$) of the Dependence of the Rate of Alkaline Hydrolysis of the Monoanions of 5-Arylidenethioxohydantoins I-VII and X and Their Derivatives VIII and IX on the Temperature

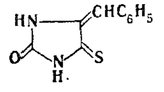
Eq.	Compound	R	lg A	±Δ lg A	B	±Δ B	n	r	s
1a	I	NO_2	10,150	0,060	4,623	0,021	8	0,9997	0,014
1b	II	Br	7,595	0,047	3,801	0,016	8	0,9998	0,012
1c	III	H	6,632	0,031	3,493	0,011	8	0,9999	0,014
1d	IV	Me	5,884	0,033	3,253	0,011	8	0,9998	0,010
1e	V	OMe	5,246	0,029	3,059	0,010	8	0,9997	0,010
1f	VI	H	7,774	0,100	3,967	0,030	5	0,9984	0,031
1g	VII	H	8,302	0,127	3,060	0,080	6	0,9992	0,034
1h	VIII	H	10,950	0,230	4,420	0,092	7	0,9972	0,045
1i	IX	H	4,932	0,080	1,939	0,025	6	0,9998	0,013
1j	X	H	8,001	0;170	3,996	0,070	8	0,9997	0,015

TABLE 3. Activation Parameters for the Alkaline Hydrolysis of the Monoanions of 5-Arylidenethioxohydantoins I-VII and X, Their Derivatives VIII and IX, and Their Oxygen Analogs Ia-VIIIa


I-VII, I-VII a


VIII, VIII a


IX


X

I-V, I-V a $R^1 = R^3 = H$; VI, VI a $R^1 = H$, $R^3 = CH_3$; VII, VII a $R^1 = R^3 = CH_3$; I-VIII X=S, a X=O

Compound	X	R=p-	$\Delta H^{\neq}$, kJ/mole	$-T\Delta S^{\neq}$, kJ/mole	$\Delta G^{\neq}$, kJ/mole
I	S	NO_2	85,8	20,9	106,7
II	S	Br	69,9	38,1	108,0
III	S	H	64,0	44,8	108,8
IV	S	Me	59,4	49,4	108,8
V	S	OMe	55,6	53,6	109,2
VI	S	H	73,2	36,0	109,2
VII	S	H	55,6	40,6	96,2
VIII	S	H	81,6	15,5	97,1
IX	—	—	34,3	55,7	90,0
X	—	H	73,3	35,1	108,4
Ia	O	NO_2	72,8	33,0	105,8
IIa	O	Br	56,1	50,6	106,7
IIIa	O	H	53,6	53,6	107,2
Va	O	OMe	47,3	60,7	108,0
VIa	O	H	72,0	37,2	109,2
VIIIa	O	H	58,6	45,6	104,2

Note. The error in the determination of $\Delta H^{\neq}$, $T\Delta S^{\neq}$, and $\Delta G^{\neq}$ does not exceed 2 kJ/mole, and the parameters were calculated for 75°C. The activation parameters of the alkaline hydrolysis of hydantoins Ia-VIIIa were taken from [3].

benzylidenehydantoins VI and VII, 5-benzylhydantoin VIII, 2-methylthio-3-methyl-5-benzylidene-4-imidazolone (IX), and 4-thioxo-5-benzylidenehydantoin (X). The temperature was varied from 55 to 95°C. The alkali concentration was selected from the ionization constants of I-V [6] and the limiting values of the concentrations of solutions of alkalis in which the linear dependences $\log k_1 = f(\log C_{KOH})$ [1] are satisfied under the assumption that all of the investigated compounds are capable of ionization and exist in solution only in the monoanionic form. The spectrophotometrically measured [7] second-order rate constants are presented in Table 1. The change in the rate of hydrolysis of the monoanions of I-VI, VIII, and X and nonionized VII and IX follow the Eyring—Arrhenius equation over the entire investigated range of temperatures [Fig. 1 and Table 2, Eqs. (1a-j)]. The activation parameters of the alkaline hydrolysis of the indicated compounds calculated from the well-known relationships are presented in Table 3.

TABLE 4. Equations [Y = AX + B (2 — 6)] of the Interactions of the Kinetic and Activation Parameters of the Alkaline Hydrolysis of the Monoanions of 5-(4-R-Benzylidene)thioxohydantoins

Eq.	X	Y	T, °C	A	±ΔA	B	±ΔB	n	r	s
2a	$\lg k_{70}$	$\lg k_T$	95	2,014	0,019	4,312	0,019	5	0,999	0,011
2b			85	1,629	0,011	2,671	0,021	5	0,999	0,012
2c			80	1,488	0,016	2,039	0,030	5	0,999	0,010
3a	$\Delta H^{\neq}$	$\lg k_T$	95	0,086	0,004	−4,194	0,066	5	0,999	0,012
3b			80	0,064	0,003	−4,243	0,044	5	0,999	0,012
3c			70	0,043	0,002	−4,223	0,038	5	0,999	0,014
3d			65	0,033	0,003	−4,232	0,046	5	0,997	0,017
3e			55	0,014	0,004	−4,260	0,066	5	0,989	0,027
4	$\Delta S^{\neq}$	$\Delta H^{\neq}$		322,8	2,1	105,18	0,26	5	0,999	0,46
4a				318,0	3,5	103,14	0,62	6	0,999	1,00
5a	σ_n	$\lg k_T$	95	0,553	0,083	−2,876	0,032	5	0,992	0,033
5b			85	0,452	0,048	−3,137	0,018	5	0,994	0,026
5c			80	0,397	0,042	−3,276	0,017	5	0,996	0,017
5d			70	0,285	0,074	−3,561	0,029	5	0,987	0,030
5e			65	0,225	0,039	−3,713	0,028	5	0,993	0,040
5f			55	0,101	0,072	−4,023	0,057	5	0,981	0,058
6	$1000/T$	ρ	95—55	−1,135	0,008	4,231	0,024	6	0,999	0,008
6a			95—20	−1,197	0,112	3,767	0,335	5	0,996	0,036

Note. Eqs. (4a) and (6a) pertain to the alkaline hydrolysis of 5-benzylidenehydantoins and were taken from [3].

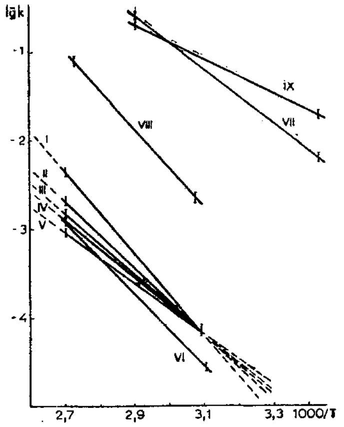


Fig. 1

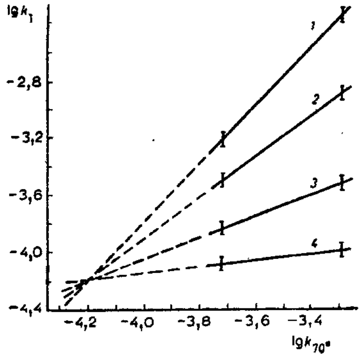


Fig. 2

Fig. 1. Dependence of the rate constants (k, liters·mole^{-1}·sec^{-1}) for the alkaline hydrolysis of the monoanions of 2-thioxo-5-arylidenehydantoins I-VI and their derivatives VIII-X on the temperature. The numbers of the lines correspond to the numbers of the compounds. See Table 2 for the log k = log A — 1000BT^{-1} equations.

Fig. 2. Logarithmic dependence of the rate constants (k, liters·mole^{-1}·sec^{-1}) for the alkaline hydrolysis of the monoanions of 2-thioxo-5-arylidenehydantoins I-VI at 95 (1), 80 (2), 65 (3), and 55°C (4) on the hydrolysis rate constants at 70°C (see Table 4 for the equations of the lines).

The investigated reaction series, viz., I-V, obeys an isokinetic law [8-10], as evidenced by a number of facts. The Arrhenius lines intersect at one point (Fig. 1). The dependences of the logarithms of the rate constants for the hydrolysis of I-V at various temperatures on the logarithms of the rate constants at 70°C are rectilinear and intersect at one point [Fig. 2 and Table 4, Eqs. (2a-c)]. The dependences of the rate constants at several temperatures on the enthalpies and energies of activation [Table 4, Eqs. (3a-e)], of the enthalpies

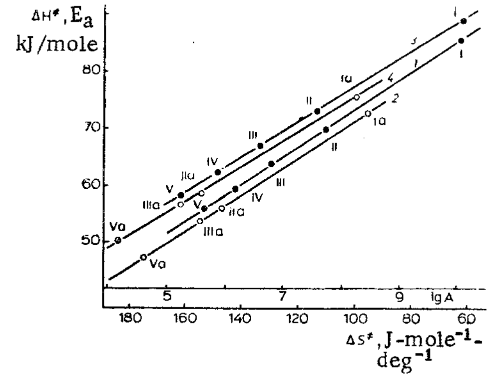


Fig. 3. Interdependence of the parameters [of the enthalpy on the entropy (1, 2) and of the energy of activation on the preexponential factor (3, 4)] of the alkaline hydrolysis of 2-thioxo-5-arylidenehydantoins I-V (1, 3) and their oxygen analogs Ia-Va (2, 4).

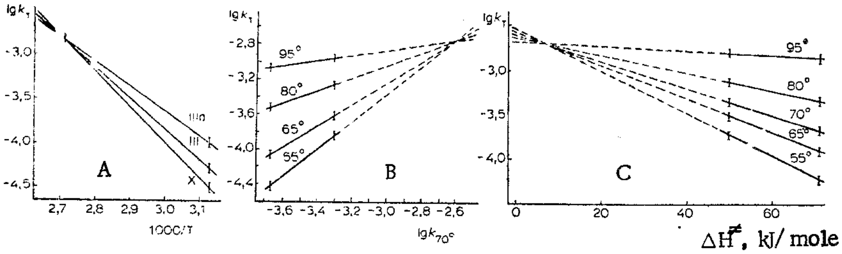


Fig. 4. Dependence of the logarithms of the rate constants (k, liters·mole^{-1}·sec^{-1}) of the alkaline hydrolysis of the monoanions of 5-benzylidenehydantoin IIIa and its 2- and 4-thioxo analogs III and X at various temperatures on the "reciprocal" of the temperature (A), logarithms of the rate constants at 70°C (B), and enthalpies of activation (C).

of activation on the entropies of activation [Table 4, Eq. (4)], and of the logarithm of the preexponential factor in the Arrhenius equation on the energy of activation (Fig. 3) are also linear. The Hammett equation is satisfied for all of the experimentally investigated temperatures. The logarithms of the rate constants for the alkaline hydrolysis of the monoanions of 2-thioxo-5-arylidenehydantoins I-V correlate satisfactorily with the MacDaniel – Brown σ constants [Table 4, Eq. (6)], and the ρ values depend linearly on the reciprocal of the temperature [Table 4, Eq. (6)]. The isokinetic temperatures calculated by various methods [8-10] average out to 321 ± 10°K and coincide with one another and with the isokinetic temperature of the alkaline hydrolysis of the monoanions of 5-arylidenehydantoins Ia-Va [3].

The reliability of the satisfaction of the isokinetic expression is confirmed by two tests [11, 12]. First, the slope (κ) of the lines in coordinates of log k_{T_1} and log k_{T_2} differs from unity [6, 7]. In fact, the $(\kappa - 1)/s_\kappa$ ratio for the case under discussion varies between two and three for $\Delta T > 15°C$. Second, the range of change in the rate constants in the case of variation of the substituent at a single temperature is rather large in the case under investigation. The error in the determination of the rate constants by the method used does not exceed 5%, and the difference between the extreme values of the logarithms of the constants for reliable discussion of the isokinetic temperature consequently should be no less than 20% of the average value, i.e., the condition $\log k - 3s_k > 0$ should be satisfied; this is actually observed when T is 20°C or more higher than β.

These facts make it possible to assume that the mechanisms of the alkaline hydrolysis of the monoanions of 2-thioxo-5-arylidenehydantoins I-V are identical. Moreover, the mechanism of the hydrolysis of these compounds is the same as in the case of the hydrolysis of the monoanions of 5-arylidenehydantoins [3]. In fact, the changes in the reactivities under the influence of the substituent and the temperature are the same in both series. In particular, one is persuaded of this by the identical character of the $\rho = f(1000/T)$ and $\Delta H^{\neq} = f(\Delta S^{\neq})$ dependences [see Eqs. (4) and (4a), and (6) and (6a) in Table 4]. As in the case of 5-arylidenehydantoins [3], an increase in the electron-donor capacity of the substituent leads to an increase in the free energy of activation and, correspondingly, to a decrease in the rate of hydrolysis; however, both the enthalpy and entropy of activation decrease in this case (Fig. 3 and Table 3). Consequently, it may be assumed that, just as in the case of their oxygen analogs [3] and 5-carboxymethylidenehydantoins [12], the most important factor in the change in the reactivities of 2-thioxohydantoins I-V is the change in the solvation of the starting and transition states under the influence of the substituent and the temperature. This also explains the high negative entropies of activation and the increase in the enthalpies and entropies of activation as the electron-acceptor capacity of the substituent increases (see [3, 12]). As in the case of 5-arylidenehydantoins, the $\delta\Delta H^{\neq}$ values in the investigated series do not change over the entire investigated temperature range (Fig. 1), and all of the changes in the reactivities are due to a change in the contribution of $T\Delta S^{\neq}$ to the free energy of activation. In this connection, the entropy control of the reaction under the investigated conditions — at experimental temperatures above the isokinetic temperature (T_{exp} = 328-368°K > β = 321°K) — becomes understandable.

2-Thioxo-5-arylidenehydantoins I-V are hydrolyzed more slowly than the corresponding oxygen analogs Ia-Va (compare the data in Table 1 and in [3]). In each pair of identically substituted compounds both the enthalpy and entropy of activation of hydrolysis of the thioxo derivatives are 8.3-12.6 kJ and 20-33.5 J/mole·deg higher; these differences increase as the electron-acceptor capacity of the substituent increases vis-a-vis an approximately constant difference in the change in the free energy of activation (within the limits of the errors in the measurements). In other words, replacement of the oxygen atom by a sulfur atom gives rise to the same (in direction) but smaller changes in the reactivities of the thioxo derivatives as compared with arylidenehydantoins, as does an increase in the electron-acceptor capacity of the substituent in the 4 position of the benzene ring within each series. It is evident that these differences can be explained by a difference in the solvation of the starting monoanions and transition states during the hydrolysis of thioxohydantoins I-V and their oxygen analogs Ia-Va.

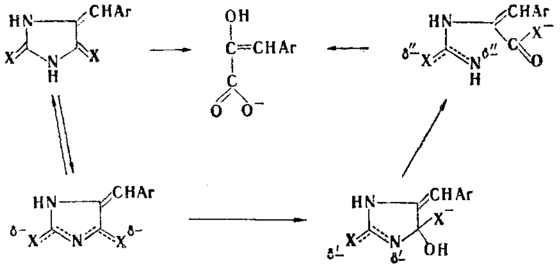
The rates of the reaction of 2-thioxo-5-benzylidenehydantoin III and 4-thioxo-5-benzylidenehydantoin X are extremely close (the difference in the hydrolysis rate constants increases somewhat as the temperature is lowered). One should probably expect this, assuming that the attack by the hydroxide ion is directed perpendicularly to the plane of the hydantoin ring. A comparison of the activation parameters of the hydrolysis of these compounds provides evidence for appreciable differences in the polarities of the starting monoanions and the corresponding transition states. This also evidently serves as the reason for the fact that 4-thioxohydantoin X is not included in a single isokinetic expression with 2-thioxohydantoins I-V, despite the fact that the principal steps of the mechanisms of the hydrolysis of these compounds are identical. Replacement of the oxygen atom of the $C_4{=}O$ group by a sulfur atom gives rise to appreciably greater, although identically directed, changes in the activation parameters as compared with replacement of $C_2{=}O$ by $C_2{=}S$. Judging from the available data, the change in the reactivity on passing from 5-benzylidenehydantoin IIIa to its 2- and 4-thioxo analogs is described by an isokinetic expression. Evidence for this is provided by at least four tests: intersection at one point of the lines of the $\log k = f(100/T)$, $\log k_T = f(\log k_{70})$, $\log k_T = f(\Delta H^{\neq})$, $\Delta H^{\neq} = f(\Delta S^{\neq})$, and $E_a = f(\log A)$ dependences (Fig. 4). Unfortunately, the data obtained are as yet insufficient for the formation of a strict judgment regarding the magnitude of the isokinetic temperature and degree of reliability of satisfaction of the isokinetic expression. However, the set of data presented above and in earlier studies makes it possible to state something regarding the identical character of the hydrolysis mechanisms and the factors that are reasonable for the change in the reactivities of 5-benzylidenehydantoins and their 2-thioxo analogs.

Methylated derivatives VI, VII, and IX and hydrogenated analog VIII are not included in a single reaction series either with hydantoins I-V or with unsubstituted IIIa, III, and X. The change in the reaction rates of these compounds as the temperature is varied is described by the Eyring and Arrhenius equations, but, as one should have expected, the kinetic and activation parameters are not included in correlation dependences (2-6) (Table 4). In all likelihood, in the case of the dimethyl derivatives this is explained by the difference in the nature of the reacting particles and the structure and solvation of the starting and transition states as compared with unsubstituted III. It is only natural that the most substantial difference — the increase in the reaction rate by a factor of $\sim$1000 — is due to the pronounced decrease in the electrostatic repulsion of the hydroxide ion and the substrate of the nonionized VII and IX as compared with the anion of hydantoin III (see Tables 1 and 2 and Fig. 1). This is also reflected in the activation parameters: the free energy of activation undergoes a decrease of 21 kJ/mole. At the same time, the differences in the activation parameters of VII and IX are probably due not so much to the difference in the charge on C_4 as to the solvation characteristics.

The rate of hydrolysis of 3-methyl-2-thioxohydantoin VI as the temperature changes also varies in conformity with the Eyring and Arrhenius equations; the reaction rate is somewhat decreased as compared with the rate in the case of unsubstituted III, and, as expected, the kinetic and activation parameters of the N-methyl derivative are not included in correlation dependences (2-6). The differences in the structures of the starting anions and the transition states and their solvation evidently serve as the only explanation for this.

In contrast to 5-benzylhydantoin VIIIa, which is hydrolyzed at approximately the same rate [3] as 5-benzylidenehydantoin IIIa, the hydrogenated analog (VIII) of parent compound III reacts appreciably more rapidly than hydantoin III itself (see Tables 1 and 2 and Fig. 1). The reasons for this behavior of VIII are as yet unclear. One should only point out that the rate of hydrolysis of this compound at various temperatures correlates satisfactorily with the rate of hydrolysis of other "saturated" hydantoins [e.g., the $\log k_{T_2} = f(\log k_{T_1})$ dependence is linear with a high degree of reliability], particularly 5-benzylhydantoin VIIIa and hydantoin-5-acetic acid.

Thus it may be assumed that under the investigated conditions the mechanisms of the alkaline hydrolysis of the monoanions of substituted 5-benzylidenehydantoins and their 2- and 4-thioxo analogs are identical and can be represented by the scheme



X=O, S; Ar=C_6H_5R-*p*, R-*p*=NO_2, Br, H, CH_3, OCH_3, $NAlk_2$

This scheme includes attack on the C_4 atom of the ionized carbonyl group as the rate-determining step, whereas the steps involving proton transfer, cleavage of the C_4-N_3 bond, and subsequent hydrolysis of the intermediate acyclic products to give arylpyruvic acids proceed rapidly. The important, if not decisive, factor in the change in the reactivities as the temperature and the substituent in the 4 position are varied or the oxygen atoms attached to the C_2 and C_4 atoms of the hydantoin ring are replaced by sulfur is the corresponding change in the solvation of the starting and transition states in the rate-determining step. Similar conclusions can also be drawn with respect to the alkaline hydrolysis of the nonionized compounds and their methylated analogs. In the latter case deviations from this mechanism that are associated with a change in the rate-determining step and participation of the hydroxide ion rather than a water molecule in the construction of the transition complex during proton transfer in the tetrahedral intermediate product of addition of hydroxide ion to $C_4{=}O$ are also possible.

EXPERIMENTAL

The investigated I-X were obtained by known methods [3, 6, 13-16] and were purified by recrystallization until they had constant melting points and extinction coefficients in their UV spectra, after which they were dried in vacuo (2-5 mm) at 100-130°C. The individuality of the compounds was monitored by means of paper and thin-layer chromatography (TLC).

The KOH solutions of the required concentration that were necessary for the kinetic measurements were prepared [1] by dilution of a saturated solution of deoxygenated distilled water. The concentrations of these solutions were selected in such a way that the compounds that were capable of undergoing ionization were present in solution in only the monoanionic form [1, 6]. The starting solutions of I-X were prepared by dissolving weighed samples of the substances in purified ethanol, and their concentrations were selected in the same way as in [1].

The kinetics of the alkaline hydrolysis of I-X were investigated by the spectrophotometric method described in [1-3, 7] with a no less than tenfold molar excess of alkali. The temperature during the kinetic measurements was maintained with an accuracy of ±0.05°C by means of a U-10 thermostat; temperature control was accomplished directly in the cuvette of the spectrophotometer, which was the reaction vessel. The analytical wavelengths were established beforehand for all of the compounds (Table 1) [1]. The degree of conversion of the starting compounds was no less than 75% in all of the experiments.

The rate constants were measured and calculated in the same way as in [1-3, 7]. Each rate constant was determined at least three times, and the average value was used in the subsequent calculations. The error in the determinations did not exceed 5%. The processing of the results of the kinetic measurements by the method of least squares and the calculation of the constants and other calculations were accomplished with a computer (the Student t distribution with a predesignated reliability of 95% was used).

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