ESR AND POLAROGRAPHY OF NITROAZOLES.

4.* POLAROGRAPHIC STUDY OF NITROBENZIMIDAZOLES

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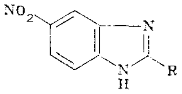
The electrochemical reduction of 2-substituted 5(6)-benzimidazoles has been studied with the aid of classical polarography and cyclic voltammetry in acetonitrile. The influence of the substituents in position 2 on the magnitudes of the half-wave potentials of the first stage is exerted by induction and resonance mechanisms with approximately the same contributions, and that on the magnitudes of the half-wave potentials of the second stage predominantly by the resonance mechanism. The possibilities of taking the radical-stabilizing factor $\sigma^{\cdot}$ into account in the correlations are discussed. In order to study the 2-substituted 5(6)-nitrobenzimidazole series, their pK_a values in acetonitrile have been determined by potentiometric titration.

Continuing an investigation of nitroazoles [1-3], we have studied the polarographic reduction of 2-substituted 5(6)-nitrobenzimidazoles (I-XI) in acetonitrile. There is practically no information on the polarographic behavior of these compounds in the literature. There has been only a report of the reduction of 5(6)-nitrobenzimidazoles in aqueous solutions in which the high absorbability of the polarizer on the cathode was mentioned [4].



I – XI

All the compounds are reduced at a dropping mercury electrode and each has two waves on its polarogram (Table 1). As can be seen from Table 1, a substituent in position 2 exerts a substantial influence on the magnitudes of the reduction potential, $E_{1/2}$ being more sensitive to the influence of a substituent than $E''_{1/2}$.

The first and second waves have a diffusion nature, as is shown by a graph of the linear dependence of the limiting currents of the first and second half-waves (i'_{lim} and i''_{lim}, respectively) on the height of the mercury column ($\sqrt{h_{Hg}}$) (Fig. 1). The two waves are practically identical in height and correspond to the transfer of approximately one electron. This is shown both by the figure for the number of electrons (n) (Table 1) calculated by means of the Ilković equation and by a comparison of the waves of the compounds under investigation with the first reduction wave of nitrobenzene observed under practical conditions.

The cyclic volt-ampere curves (Fig. 2) show that, under the conditions of the experiment (rate of scanning 0.1-1 V/sec), the first wave was practically irreversible and the second reversible.

The results presented permit the assumption that at the potential of the first half-wave an unstable anion radical is formed, but we were unable to record it by the ESR method [2]. At the potential of the second half-wave, a fairly stable dianion radical was formed [2, 5], the well-resolved ESR spectrum of which indicates the presence of the maximum density of the unshared electron on the nitro group.

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*For communication 3, see [1].

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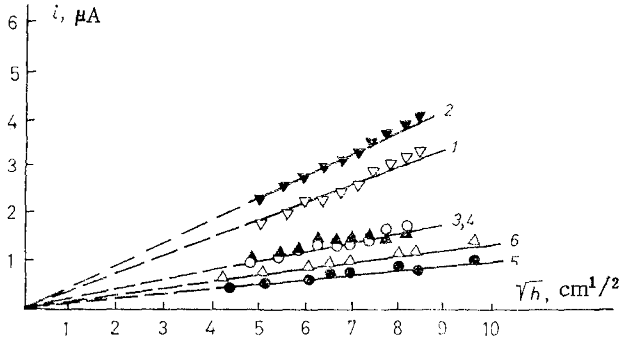

Fig. 1. Dependence of the limiting currents of the first and second half-waves on the height of the mercury column ($\sqrt{h_{Hg}}$) for 2-amino-5(6)-nitrobenzimidazole: 1) $i'_{\lim}$; 2) $i''_{\lim}$; 5(6)-nitrobenzimidazole: 3) ○ – $i'_{\lim}$; 4) ▲ – $i''_{\lim}$; and 2-trifluoromethyl-5(6)-nitrobenzimidazole: 5) $i'_{\lim}$; 6) $i''_{\lim}$.

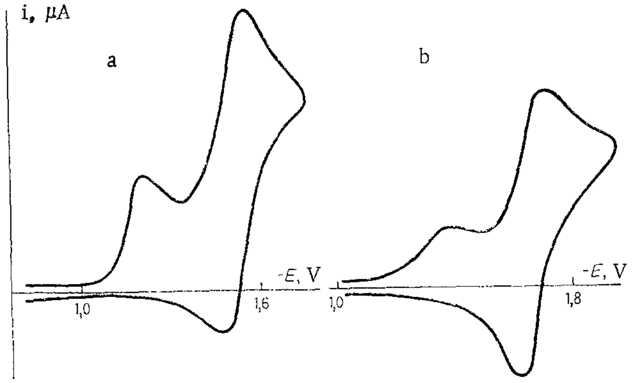

Fig. 2. Cyclic volt–ampere curves for 5(6)-nitrobenzimidazole (a) and 2-amino-5(6)-nitrobenzimidazole (b).

TABLE 1. Characteristics of the Electrochemical Reduction and pK_a Values of 2-Substituted 5(6)-Nitrobenzimidazoles in Acetonitrile

Compound	R	$-E'_{1/2}$, V	$-E''_{1/2}$, V	$i'_{\lim}$, μA	$i''_{\lim}$, μA	$i'_{\lim}/i''_{\lim}$	n'	n''	pK_a
I	$N(CH_3)_2$	1,24	1,64	0,60	0,60	1,00	1,00	1,00	23,16
II	NH_2	1,24	1,62	0,47	0,53	0,89	0,78	0,88	22,48
III	OCH_3	1,16	1,62	0,69	0,79	0,87	1,15	1,30	21,38
IV	OC_2H_5	1,17	1,65	0,60	0,60	1,00	1,00	1,00	21,72
V	CH_3	1,14	1,47	0,60	0,60	1,00	1,00	1,00	23,40
VI	H	1,13	1,47	0,54	0,45	1,20	0,90	0,75	22,31
VII	Cl	0,95	1,43	0,50	0,70	0,71	0,83	1,25	18,40
VIII	$COOCH_3$	0,91	1,34	0,48	0,60	0,80	0,80	1,00	20,24
IX	$COCH_3$	0,92	1,32	0,44	0,57	0,77	0,73	0,95	20,78
X	CF_3	0,88	1,33	0,48	0,60	0,80	0,80	1,00	17,90
XI	CN	0,75	1,30	0,60	0,70	0,86	1,00	1,17	16,63

The applicability of the LFE principles to both reversible and irreversible electrical processes [6] permits the use of the correlation equations for studying, in particular, the sensitivity of the potentials of electrochemical reduction to substituent effects.

The results of a two-parameter correlation of $\Delta E'_{1/2}$ and $\Delta E''_{1/2}$ ($\Delta E_{1/2} = E_{1/2}^X - E_{1/2}^H$) with the substituent constants $\sigma_I\sigma_R$, $\sigma_I\sigma_R^\circ$, FR, and $\sigma_I\sigma_R^+$ are given in Table 2. The influence of substituents on the first half-wave potential is exerted both by the induction and by the resonance mechanism, the ratio of the contributions of these effects being approximately the same and being practically independent on the choice of the parameters of the substituents. The results of the correlation of the magnitudes of the second half-wave poten-

TABLE 2. Parameters of the Correlation Equation $\Delta E_{1/2} = ax + by + d$

$\Delta E_{1/2}$	xy	a	b	d	r	s	n	b, %*
$\Delta E'_{1/2}$	$\sigma_I\sigma_R$	0,49±0,06	0,26±0,03	0,03±0,01	0,984	0,03	11	54±4
	$\sigma_I\sigma_R^0$	0,49±0,05	0,38±0,03	0,02±0,01	0,990	0,02	11	55±3
	FR	0,29±0,06	0,25±0,05	0,03±0,01	0,964	0,04	11	54±7
	$\sigma_I\sigma_R^+$	0,48±0,09	0,14±0,02	0,03±0,00	0,966	0,04	11	53±6
$\Delta E''_{1/2}$	$\sigma_I\sigma_R$	0,15±0,07	0,31±0,03	0,04±0,00	0,968	0,03	11	82±7
	$\sigma_I\sigma_R^0$	0,16±0,05	0,45±0,04	0,03±0,00	0,981	0,03	11	82±5
	FR	0,06±0,07	0,31±0,05	0,04±0,00	0,941	0,05	11	87±13
	$\sigma_I\sigma_R^+$	0,15±0,10	0,16±0,03	0,04±0,01	0,931	0,05	11	81±11

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*Here and below, b represents the resonance contribution.

TABLE 3. Parameters of the Correlation Equation $\Delta E_{1/2} = a\sigma^* + b\sigma^r + d$

$\Delta E_{1/2}$	a	b	d	r	s	n	b, %*	r_{12}*
$\Delta E'_{1/2}$	0,14±0,03	0,16±0,02	0,03±0,01	0,977	0,03	11	71±5	0,394
$\Delta E''_{1/2}$	0,01±0,03	0,16±0,01	0,03±0,01	0,976	0,03	11	97±8	0,394

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*r is the correlation coefficient between the parameters σ^* and σ^r (partial correlation coefficient).

TABLE 4. Parameters of the Correlation Equation $\Delta E_{1/2} = ax + by + cz + d$

$\Delta E_{1/2}$	xyz	a	b	c	d	r	s	n	a, %*	b, %	c, %†
$\Delta E'_{1/2}$	$\sigma_I\sigma_R\sigma^{\cdot}$	0,47±0,03	0,33±0,02	0,15±0,03	−0,01±0,01	0,995	0,02	11	34±2	53±2	13±2
	$\sigma_I\sigma_R^0\sigma^{\cdot}$	0,49±0,04	0,41±0,03	0,06±0,03	0,01±0,01	0,992	0,02	11	41±3	54±3	6±3
	$FR\sigma^{\cdot}$	0,25±0,04	0,36±0,05	0,19±0,07	−0,01±0,01	0,979	0,03	11	29±4	54±5	17±5
	$\sigma_I\sigma_R^+\sigma^{\cdot}$	0,44±0,04	0,21±0,02	0,25±0,05	−0,02±0,03	0,989	0,02	11	28±3	53±4	20±4
$\Delta E''_{1/2}$	$\sigma_I\sigma_R\sigma^{\cdot}$	0,13±0,05	0,37±0,03	0,12±0,05	0,01±0,02	0,979	0,02	11	12±4	74±6	14±5
	$\sigma_I\sigma_R^0\sigma^{\cdot}$	0,15±0,04	0,46±0,04	0,03±0,04	0,02±0,01	0,982	0,03	11	17±4	80±6	4±6
	$FR\sigma^{\cdot}$	0,03±0,05	0,41±0,06	0,17±0,08	−0,00±0,02	0,959	0,04	11	4±7	77±9	19±8
	$\sigma_I\sigma_R^+\sigma^{\cdot}$	0,10±0,07	0,23±0,03	0,22±0,09	−0,00±0,01	0,957	0,04	11	8±5	71±8	21±7

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*a is the contribution of the induction component.
†c is the contribution of the radical-stabilizing factor.

tial show that the transmission of the substituent effects takes place mainly through the resonance mechanism (approximately 80%).

In the anions of the 2-substituted benzimidazoles, the effects of substituents are transmitted exclusively by the resonance mechanism [7]. It is quite possible that this is connected with the fact that the polarization of the π-electrons of the benzimidazole ring in the anion is considerably higher than in the neutral molecule.

In addition to these constants that are widely used in correlation analysis, it appeared to us to be interesting to make two-parameter correlations of the half-wave potentials with the constants σ^* and σ^r (where σ^* is Taft's inductive constant and σ^r is the resonance constant determined from ESR measurements) since they can be applied successfully to free-radical reactions [8-10]. It can be seen (Table 3) that the correlation coefficients when these constants are used are practically unchanged from those obtained with the constants used previously (Table 2), but the contribution of the resonance component in them rises somewhat.

Table 4 gives the characteristics of the correlation equation taking into account the parameter $\sigma^{\cdot}$ (the radical-stabilizing factor) characterizing the stabilization of the radical state [11-13]. The coefficients of multiple correlation rise but the relationship between

TABLE 5. Parameters of the Correlation Equation $\Delta pK_a = ax + by + d$

xy	a	b	d	r	s	n	b, %
$\sigma_I\sigma_R$	$-9{,}95\pm0{,}98$	$-1{,}44\pm0{,}48$	$0{,}60\pm0{,}13$	0,975	0,48	11	24 ± 6
$\sigma_I\sigma_R^0$	$-10{,}01\pm0{,}98$	$-2{,}02\pm0{,}69$	$0{,}65\pm0{,}15$	0,974	0,49	11	24 ± 7
$\sigma_I\sigma_R^+$	$-9{,}89\pm1{,}03$	$-0{,}78\pm0{,}28$	$0{,}55\pm0{,}16$	0,973	0,50	11	24 ± 7
FR	$-6{,}06\pm1{,}03$	$-1{,}37\pm0{,}80$	$0{,}36\pm0{,}07$	0,947	0,70	11	23 ± 11

the resonance parameters and $\sigma^\cdot$ ($r_{23} \simeq 0.7$) does not permit a reliable judgment of the contributions of the radical-stabilizing factor to the overall substituent effect.

It is known that the addition of an electron in the process of electrochemical reduction may be considered as the occupancy by it of the lowest unoccupied molecular orbital (LUMO) and that a linear relationship exists between $E_{1/2}^{red}$ and E_{LUMO} [14]. We made a correlation between $E'_{1/2}$ and E_{LUMO} calculated in the INDO approximation [15] and found a linear relationship between these magnitudes:

$$-E'_{1/2} = (-0.62\pm0.04)E_{LUMO}+0.06;\ r=0.987;\ s=0.02;\ n=7$$

Since nitrobenzimidazoles possess acidic properties, it appeared of interest to determine whether the "acid" properties of the proton of the NH group affected the polarographic parameters. With this aim, we carried out a potentiometric titration of the 2-substituted 5(6)-nitrobenzimidazoles in acetonitrile and determined the values of pK_a accordingly (Table 1). The acid properties of these compounds should primarily affect the value of the current i'_{lim} (or i'_{lim}/i''_{lim}) and, consequently, n' (Table 1), as for the case of the nitroimidazoles [16].

We carried out a correlation of the values of pK_a with i'_{lim} with i'_{lim}/i''_{lim}, and with n' and found no interrelationship whatever between these magnitudes, i.e., the 2-substituted 5(6)-nitrobenzimidazoles either do not dissociate in acetonitrile or dissociate in such a small degree that the influence of dissociation on the polarographic parameters is below the limits of accuracy of their determination.

The influence of substituents in position 2 on the values of pK_a (Table 5) is exerted by a mechanism different from that on the half-wave reduction potential. The induction effect amounts to about 80% of the total effect of a substituent, which is a consequence of differences in the nature and positions of the centers of reduction and of dissociation.

EXPERIMENTAL

The polarographic investigation was performed in anhydrous acetonitrile purified by a published procedure [17], with tetrabutylammonium perchlorate as supporting electrolyte. The number of electrons n consumed in the reduction of molecule of depolarizer was calculated from the Ilkovič equation and was also estimated by comparing i_{lim} of the compound under study and i_{lim} of a standard substance (nitrobenzene). The polarograms were recorded on PAR-170 (USA) and OH-102 (Hungary) polarograms in a thermostated cell (25 ± 1°C). A dropping mercury electrode with forced detachment of the drop (m = 0.7 mg/sec, t = 0.22 sec) was used as the cathode, and a saturated calomel electrode as the comparison electrode. The half-wave potentials were determined with an accuracy of ± 0.01 V, and the limiting currents with an accuracy of ±0.05 μA. The cyclic volt–ampere curves were recorded on the PAR-170 polarograph at a stationary mercury electrode (PAR-9323 type). The rate of scanning of the potential was varied between 1 and 0.1 V/sec.

The dissociation constants pK_a were determined by the method of potentiometric titration at 20°C using glass (ESL 43-07) and silver chloride (ÉVL-1MZ) electrodes. The titrant was 0.1 N tetrabutylammonium hydroxide in benzene–ethanol (9:1), and the standard was benzoic acid, the pK_a value of which in acetonitrile is 20.7 [18]. The error in the determination of the pK_a values was ±0.05.

The statistical treatment of the experimental results was performed by means of a standard program on a BÉSM-6 computer. The percentage contribution of the resonance component was calculated by means of a program drawn up by the method of Swain and Lupton [19]. The values of the parameters of σ_I, σ_R, σ_R°, and σ_R^+ were taken from the literature [20], those of F and R from [19], and σ^* and σ^r from [21]. The values of the radical-stabilizing factor $\sigma^\cdot$ were either taken from [13] or calculated by means of the formula proposed in the same paper.

The compounds studied were obtained by methods described in the literature: (I) — [22], (II) — [23], (V) and (VI) — [24], (VII) — [25], (IX) — [26], and (X) — [27].

2-Methoxy-5(6)-nitrobenzimidazole (III). A mixture of 7.6 ml of nitric acid (d = 1.42) and 11 ml of concentrated sulfuric acid was added dropwise to a solution of 14.8 g (0.1 mole) of 2-methoxybenzimidazole [28] in 45 ml of concentrated sulfuric acid at 0-5°C. The reaction mixture was kept at 0-5°C for 40 min and was then cooled and poured onto 200 g of ice and, after neutralization with ammonia to pH ≈ 6, the precipitate formed was separated off. Yield, 82%, mp 226-227°C (from toluene or methanol). Found, %: C 50.0, H 3.8, N 21.8. $C_8H_7N_3O_3$. Calculated, %: C 49.7, H 3.6, N 21.8.

2-Ethoxy-5(6)-nitrobenzimidazole (IV) was obtained similarly from 2-ethoxybenzimidazole [28]. Yield 87%, mp 153°C (from toluene). Found, %: C 51.8, H 4.4, N 20.8. $C_9H_9N_3O_3$. Calculated, %: C 52.2, H 4.4, N 20.3.

5,6-Nitrobenzimidazole-2-carbonitrile (XI). With stirring and cooling, 2.8 g (0.01 mole) of 2-trichloromethyl-5(6)-nitrobenzimidazole was added to 15 ml of 30% aqueous ammonia [29]. The precipitate of 5(6)-nitrobenzimidazole-2-carbonamide was filtered off, and the filtrate was acidified to pH ≈ 5 with concentrated hydrochloric acid. The yield of the nitrile (XI) was 34%, mp 292°C (from methanol). Found, %: C 50.6, H 2.4, N 30.1. $C_8H_4N_4O_2$. Calculated, %: C 51.1, H 2.1, N 29.8.

2-Methoxycarbonyl-5(6)-nitrobenzimidazole (VIII) was obtained by the nitration of 2-methoxycarbonylbenzimidazole [30] by Buchel's method [31].

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ACETALS OF LACTAMS AND AMIDES.

42.* REACTIONS OF ENAMINO AMIDES AND ENAMINO ESTERS WITH SOME FORMYLATING AGENTS. SYNTHESIS OF DERIVATIVES OF 2-PYRIDONE AND OF PYRIMIDIN-4- AND -6-ONES

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The reactions of enamino esters and enamino amides with the diethyl acetal of dimethylformamide and with orthoformic ester have been investigated and it has been shown that the process takes place in two directions — with the formation of pyrimidone or 2-pyridone derivatives and with the replacement of an aminopropylene (or N,N-dimethylaminopropylene) fragment of the enamine by a dimethylaminomethylene (or ethoxymethylene) group. Some conditions favoring the occurrence of one or other of these processes have been determined.

In the reaction of the benzylamide of β-amino-α-cyanocrotonic acid (Ia) with the diethyl acetal of dimethylformamide (II) 1-benzyl-5-cyano-4-β-dimethylaminovinylpyridin-6-one (IIIa) is formed with good yield [2]. Continuing investigations relating to the synthesis of pyrimidine derivatives from enamino amides, we have similarly converted the β-amino-α-cyanocrotonanilide (Ib) into the 1-phenylpyrimidin-6-one (IIIb). In order to synthesize N-substituted pyrimidin-6-ones not having a β-dimethylamino group in position 4, we have made an attempt to cyclize compound (Ia) with orthoformic ester in acetic anhydride. However, instead of the expected 1-benzyl-5-cyano-4-methylpyrimidin-6-one (IVa), we isolated a compound (Va) with a molecular weight of 230 (mass spectrum). Its PMR spectrum in DMSO-D_6 contained signals at (ppm): 1.26 (t) and 4.37 (q), belonging to an OC_2H_5 group; 4.34 (d), which is characteristic for the methylene protons of a $C_6H_5CH_2NH$ group; 7.66 (m, C_6H_5); 8.00 (s, CH); and 8.20 (t, NH). These facts, and also the results of microanalysis, permitted the assumption that the compound obtained was the benzylamide of ethoxymethylenecyanoacetic acid (Va).† The structure of compound (Va) was shown by its independent synthesis from N-benzylcyanoacetamide (VIa) and $CH(OEt)_3$. By means of the analogous reaction of the N-phenylamide (Ib) with $CH(OEt)_3$ we succeeded in isolating 5-cyano-4-methyl-1-phenylpyrimidin-6-one (IVb). An investigation of the mother solutions by chromato-mass spectrometry showed the presence in them of the ethoxymethylene derivative (Vb), its mass spectrum being identical with that of the substance obtained by independent synthesis from the amide (VIb) and $CH(OEt)_3$.

The results obtained induced us to make a more detailed study of the reaction of compounds (Ia, b) with the acetal (II). An investigation by the chromato-mass-spectrometric method of the mother solution obtained after the pyrimidinone (IIIb) had been filtered off showed the presence of N-phenyl-α-cyano-β-dimethylaminoacrylamide (VIIb), the mass spectrum of which coincided with the spectrum of the authentic substance obtained by independent synthe-

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*For communication 41, see [1].
†We have briefly described this reaction and others of the same type previously [3].

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