

Reaction of Salt Ia with Methylamine Sulfite. A 5.3-ml (40 mmole) sample of a 25% aqueous solution of methylamine and 3.8 ml (25 mmole) of methylamine bisulfite, obtained by the general method, were added to 5 mmole of Ia, and the mixture was heated in a sealed ampul at 180°C for 15 h. Aniline IVa was isolated by the general method. A 0.37-g (69%) sample of Va was obtained. The results of analysis by GLC and IR and PMR spectroscopy of Va were in agreement with the data for a genuine sample.

In the case of a 12-fold excess of free methylamine in the reaction medium the yield of Va was 71%.

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#### ELECTROCHEMICAL TRANSFORMATIONS OF 4-(NITROARYL)-1,2- AND -1,4-DIHYDROPYRIDINES IN ACETONITRILE

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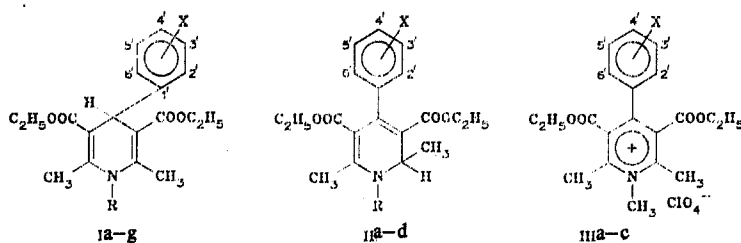
The potentials of the electrolytic oxidation and reduction of 3,5-diethoxycarbonyl-1,2- and -1,4-dihydropyridines with a nitroaryl substituent in the 4 position and their oxidized forms were determined by the method of a rotating disk electrode with a ring. The mechanism of the electrochemical oxidation of the starting dihydropyridines in acetonitrile on a graphite electrode was ascertained. The first step in the electrolytic reduction of the starting dihydropyridines is the addition of an electron to the nitro group to give anion radicals, the fine electronic structures and the hyperfine structure (hfs) constants of which were determined by EPR spectroscopy. The pyridinium fragment in the electrolytic oxidation products is reduced more readily than the nitro group in the aryl substituent.

In a continuation of our systematic study of the mechanism of the electrochemical oxidation (EO) of derivatives of 1,4- and 1,2-dihydropyridine (DHP) we directed our attention to compounds that contain a nitro group in the 4-aryl substituent of the dihydropyridine ring. A number of compounds of this type (for example, nifedipine or fenigidine) have pronounced biological activity and have found application in medical practice [1]. From the point of view of the redox transformations these compounds are characterized by the fact that they contain two electrolytically active centers, one of which is capable of electrolytic reduction (the nitro group), the other of which is capable of electrolytic oxidation (the dihydropyridine fragment). This makes it necessary to ascertain the ranges of the potentials of the corresponding transformations and the character of the interaction both between the individual groupings in the starting molecule and between the intermediates in the electrochemical transformations.

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In the present research we used a rotating disk electrode with a ring on graphite and platinum electrodes and EPR spectroscopy to study the primary electrochemical transformations of derivatives of the I and II type in acetonitrile (also see [2, 3]). For comparison, we drew model compounds of the III type - the hypothetical final products of the electrolytic oxidation of Ia-d and II - which were synthesized by the chemical oxidation of Ia-d and II.



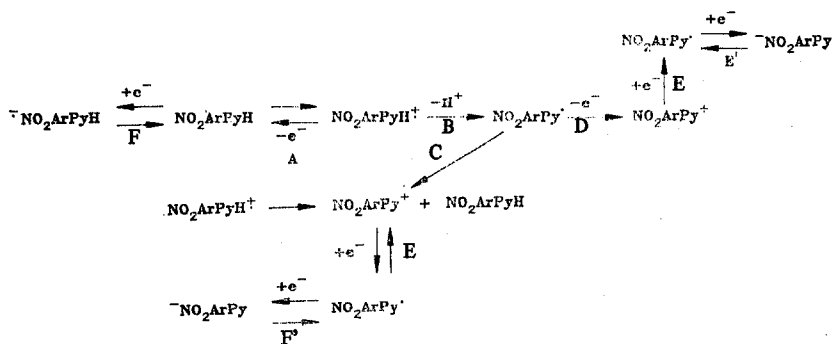
I-III a-c R=CH<sub>3</sub>, d R=C<sub>6</sub>H<sub>5</sub>, e-g R=H; a, e X=o-NO<sub>2</sub>, b, f X=m-NO<sub>2</sub>, c, d, g X=p-NO<sub>2</sub>

### Electrolytic Oxidation of I and II

It is apparent from Table 1, in which the potentials of the electrolytic oxidation and reduction of I-III in acetonitrile are presented, that the dihydropyridine grouping of I is oxidized on the disk electrode over the range of potentials ( $E_{1/2}^d$ ) from +0.67 V to +0.84 V (relative to the Ag/AgNO<sub>3</sub> reference electrode), whereas that of II is oxidized over the range from +0.46 V to +0.48 V.

A comparison of the data obtained with the potentials presented in [3] for the EO of the corresponding DHP that do not contain a nitro group in the 4-aryl ring provides evidence that the introduction of a nitro group into the 4-aryl substituent in the ortho, meta, or para position relative to the DHP ring does not have a substantial effect on the electrolytic oxidation potentials and hinders oxidation by only ~100 mV as compared with the corresponding 4-(methyl)aryl derivatives [3]. This shift is understandable if one bears in mind the relatively pronounced electron-acceptor effect of the nitro group.

The mechanism of the EO of I and II fits into the schemes proposed in [2, 3]. The corresponding scheme was defined concretely for the electrolytic oxidation of Ia-d and IIa-d.



The first step (A) in the electrolytic oxidation of nitro derivatives of 1,2-DHP (IIa-d) NO<sub>2</sub>ArPyH (wave 1, Fig. 1), in view of the relative stability of the corresponding cation radical NO<sub>2</sub>ArPyH<sup>+</sup>, is a one-electron and partially reversible (at room temperature) process as evidenced by the presence of anode-cathode wave 1, a (Fig. 1), recorded on the ring electrode. Under the given conditions only some of the cation radicals have time to undergo deprotonation in the zone of the disk electrode (process B, wave 2, Fig. 1, reduction of the split out protons on the ring electrode), whereas the resulting radical undergoes rapid disproportionation with the adjacent cation radical (process C) to give one pyridinium cation NO<sub>2</sub>ArPy<sup>+</sup> and one molecule of starting II.

In the case of the EO of 1,4-DHP (Ia-g) the cation radical has a much shorter lifetime (because of which the latter cannot be detected by the method of a rotating disk electrode with a ring), and the dominating process becomes process D, i.e., the step involving the transfer of a second electron, which proceeds at the same potentials as the first step, as a

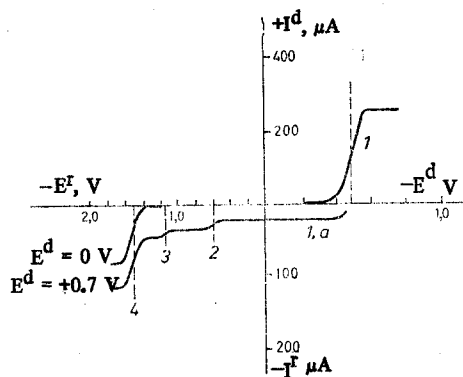


Fig. 1

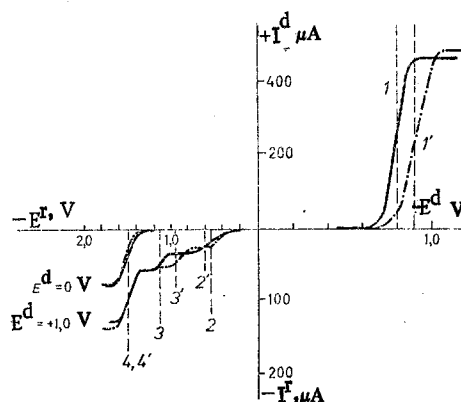


Fig. 2

Fig. 1. Volt-ampere curves of the electrolytic oxidation of IIa-c on a disk electrode (wave 1) and the electrolytic reduction of the intermediates (waves 1a and 2) and final products (wave 3) on a ring electrode ( $E^d$  and  $I^d$  are, respectively, the potential and current of the disk electrode, and  $E^r$  and  $I^r$  are, respectively, the potential and current of the ring electrode).

Fig. 2. Volt-ampere curves of the electrolytic oxidation of Ia-c (—) and IIe-g (---) on a disk electrode (waves 1 and 1') and of the electrolytic reduction of the intermediates (waves 2 and 2') and the final products (waves 3 and 3') on a ring electrode.

result of which one observes the formation of one coalesced wave 1 (Fig. 2), the height of which is close to a two-electron wave. Like the analogous wave described in [2], this wave decreases to the 1.3-1.5 electron level in the presence of dissolved molecular oxygen.

Thus, in the case of both 1,2- and N-substituted 1,4-DHP (IIa-c) and (Ia-c) the EO process terminates with the formation of a pyridinium cation, which, in turn, can be detected in the wave of its electrolytic reduction (process E) on a ring electrode (waves 3 and 3', Fig. 2). This is confirmed by the identical character of the potential of process E (which takes place over the range from -1.14 V to -1.32 V) for I and II and the potentials of the electrolytic reduction (ER) of the corresponding model compound III (wave 3, Fig. 3).

The final product of the EO of N-unsubstituted 1,4-DHP (Ie-g) is most likely the corresponding pyridine, which is protonated in solution either by the protons previously split out during the EO or by the proton donors present in the solution. The wave of the reduction (wave 3, Fig. 2) of the resulting pyridinium cation (~-1.0 V) coincides with the wave of the ER of the corresponding substituted pyridine in acetonitrile with added proton donors (in an aprotic medium the corresponding pyridine is not reduced at up to -2.0 V).

### Electrolytic Reduction of I-III

Dihydropyridines that have structures I and II but do not contain nitro groups are not reduced on graphite and platinum electrodes in acetonitrile over the range of potentials up to -2.0 V (see [4] for the reduction of such compounds on a mercury electrode). However, model compounds IIIa-c over the range of potentials from -1.15 V to -1.30 V (see Table 1) give an ER wave due to electrolytic reduction of the positively charged pyridinium part of the molecule rather than the nitro group. For the III that we investigated these potentials are, on the average, 0.2 V more positive than for 4-phenyl- or 4-tolyl-substituted pyridinium salts that do not contain a nitro group (for example, in the case of para isomers with a  $\text{CH}_3$  group the ER potential of the cation is -1.47 V, whereas it is -1.15 V in the case of the  $\text{NO}_2$  group); this is explained by the electron-acceptor effect of the nitro group. However, the orientation of the nitro group (ortho, meta, or para) does affect the potential of this wave.

It is apparent from Table 1 that the potential of this wave is 50-160 mV more negative if the nitro group is in the meta position as compared with the potentials of compounds that contain a nitro group in the para and ortho positions, in which the nitro group is in significant conjugation with the pyridinium ring. What we stated above regarding the ER wave of model compounds III also pertains to the ER wave on ring electrode of the final products of EO of Ia-c and IIa-c. A preliminary examination of the experimental data on the

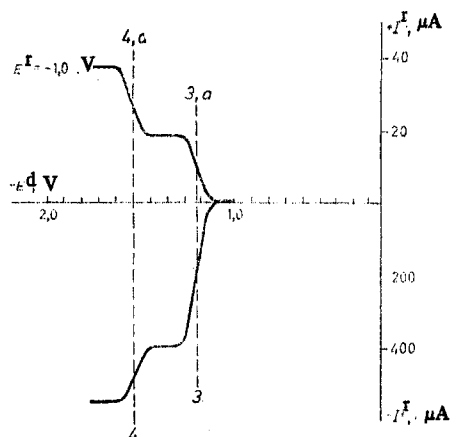


Fig. 3. Volt-ampere curves of the electrolytic reduction of model compounds IIIa-c on a disk electrode (waves 3 and 4) and of the reverse electrolytic oxidation of the products of the primary process (waves 3, a and 4, a).

electrolytic reduction of the pyridine fragment shows that this process does not consist simply in the addition of one electron to the starting particle but rather is complicated by several steps involving chemical transformations.

Reduction of the nitro group (processes F and F') both in the case of the starting DHP, viz., I and II (wave 4 in Figs. 1 and 2) and in the reduction of model compounds III (wave 4, Fig. 3) occurs at more negative potentials (at  $-1.49$  V to  $-1.70$  V). In the first case the first step of this process takes place with the addition of one electron and proceeds with greatest difficulty if the nitro group is in the ortho position of the 4-aryl substituent (see Table 1). This can evidently be explained by purely steric factors — in this position the approach of the nitro group to the electrode is hindered to the greatest extent, since it is shielded to a greater degree by the substituents of the dihydropyridine ring. In the case of the ER of model compounds III the reduction of the nitro group is complicated by chemical transformations because of prior reduction of the pyridinium part (wave 3, Fig. 3). For example, if the nitro group is in the para position of the 4-aryl substituent, the ER of the pyridinium cations (process E) is partially reversible and proceeds with the addition of 1.5 electrons (wave 3, Fig. 3), and the subsequent ER of the nitro group (wave 4, Fig. 3) takes place with the transfer of  $\sim 0.5$  of an electron. However, if the nitro group is in the meta or ortho position, both waves correspond to the one-electron level, and the ER of the pyridinium cation is completely irreversible only for the ortho isomer — wave 3, a in Fig. 3 is absent (at electrode rotation rates from 500 to 6000 rpm).

To identify the step involving ER of the nitro group and to distinguish it from the step involving the reduction of the pyridinium fragment I-III were compared with nitrobenzene, which was subjected to ER under the same experimental conditions. In all cases the nitro group is reduced in two steps. The first step is observed when  $E_{1/2} = -1.5$  V, and the second step is observed over the range from  $-1.7$  V to  $-2.0$  V (the wave of the second step is not shown in the figures).

The results of a study of the mechanism and the corresponding intermediates and final products of the various steps in the ER of cations with a pyridinium structure (including the electrolytically active centers) will be published separately.

Here, however, let us emphasize only the fundamental principle: the pyridinium part in III (or in the products of electrolytic oxidation of I and II) undergoes reduction more rapidly (at more positive potentials) than the nitro group does through the addition of the first electron. At the same time, the products of one-electron addition to the nitro group are more stable and can be detected by EPR spectroscopy in the form of free anion radicals even at room temperature; the products formed at the potentials of the addition of an electron to the pyridinium cation cannot be reliably detected under these conditions. The primary products (cation radicals) of the EO of I and II also cannot be detected at room temperature by EPR spectroscopy.

TABLE 1. Parameters of the Electrochemical Transformations of Ia-g, IIa-d, and IIIa-c on a Rotating Graphite Disk Electrode with a Ring in Acetonitrile [inert electrolyte 0.1 M tetrabutylammonium perchlorate, depolarizer concentration  $5 \cdot 10^{-4}$  mole/liter, reference electrode Ag/AgNO<sub>3</sub> (0.02 N), electrode rotation rate 2000 rpm, T = 293°K\*]

Compound	EO of the starting DHP on the disk electrode		ER of the cations of the pyridinium fragment on the ring electrode		ER of the nitro group on the ring electrode	
	E <sub>1/2</sub> , V	No. of electrons	E <sub>1/2</sub> , V	No. of electrons	E <sub>1/2</sub> , V	No. of electrons
Ia	+0,67	1,77	-1,22		-1,66	
Ib	+0,73	1,75	-1,31		-1,55	
Ic	+0,75	1,82	-1,14		-1,50	
Id	+0,79	1,78	-1,13		-1,51	
Ie	+0,81	1,87	-1,00		-1,70	
If	+0,84	1,91	-0,96		-1,51	
Ig	+0,83	1,94	-0,95		-1,51	
IIa	+0,46	0,94	-1,16		-1,66	
IIb	+0,46	0,97	-1,32		-1,54	
IIc	+0,48	0,98	-1,20		-1,49	
II d	+0,58	1,08	-1,12		-1,48	
IIIa†	—	—	-1,20	1,05	-1,62	1,02
IIIb	—	—	-1,30	1,02	-1,51	0,95
IIIc	—	—	-1,15	1,45	-1,49	0,46

\*The parameters of the wave (wave 2 in Figs. 1, 2, and 3 over the range of potentials from -0.4 V to -0.7 V) of the electrochemical reduction (ER) of the protons split out in the course of the electrochemical oxidation (EO) of compounds of the I and II type are not presented in the table.

†The potentials and heights of the corresponding waves for the investigated IIIa-c are identical if a disk or a ring electrode (with allowance for the dimensions of the electrodes).

#### Electrochemical Generation of Free Anion Radicals in the ER of the Nitro Group

In the course of the electrochemical generation (ECG) of free radicals from acetonitrile solutions of I-III at the potentials of the EO of the nitro group EPR spectra with well defined hyperfine structures (hfs) that are amenable to interpretation were recorded. The character of the splitting of the EPR spectra and the hfs constants are presented in Table 2.

It is apparent from the data presented in Table 2 that radicals similar to the anion radicals of substituted nitrobenzene are obtained, since the unpaired electron is delocalized only over the nitrobenzene fragment. In the case of I and II these are primary anion radicals, i.e., radicals that have the structure of starting I or II with one unpaired electron; however, this cannot be asserted unequivocally in the case of IIIa-c, since electrolytic reduction of the pyridinium part of the molecule precedes electrolytic reduction of the nitro group. The magnitudes of the hfs constants constitute evidence that the distribution of the unpaired electron over the nitrobenzene fragment depends on both the properties of the substituent, viz., the nitrogen-containing heteroring, and on its orientation with respect to the nitro group. As compared with the radicals obtained from substituted 1,4-DHP Ia-g, a decrease in the hfs constants of the nitrogen atom of the nitro group is characteristic for the radicals obtained from the pyridinium cations of III. This is evidently due to partial delocalization of the unpaired electron from the nitrobenzene fragment to the pyridyl fragment. For 1,2-DHP II one also might have expected an effect of conjugation with the  $\pi$ -electron system of nitrobenzene; however, in this case the direction of the change in the delocalization of the unpaired electron depends on the orientation of the nitro group. Thus in the case of substitution of nitrobenzene by a 1,2-dihydropyridine ring in the ortho or meta position the density of the unpaired electron is shifted to the nitrobenzene ring, and this density becomes greater on the atoms of the latter than for the radical of the corresponding 1,4-DHP; however, in the case of substitution in the para position the density is shifted in the reverse direction.

TABLE 2. Character of the Splitting and hfs Constants (a) of the EPR Spectra of the Anion Radicals Obtained from I-III in Acetonitrile and of the Anion Radicals of o-, m-, and p-Nitrotoluenes [5]

Compound	Character of the splitting	hfs constants, G					
		$a_{1'}$ *	$a_2$	$a_3$	$a_4$	$a_5$	$a_6$
Ia	$3N \cdot 2H \cdot 2H \cdot 2H \cdot 3H$	1,45	9,90	2,90	1,05	3,55	1,05
Ib	$3N \cdot 2H \cdot 3H \cdot 2H \cdot 2H$	0,60	3,15	9,95	3,15	1,05	3,65
Ic	$3N \cdot 3H \cdot 2H \cdot 3H$	1,70	1,10	3,05	9,85	3,05	1,10
Id	$3N \cdot 3H \cdot 2H \cdot 3H$	1,45	1,15	3,20	10,05	3,20	1,15
Ie	$3N \cdot 2H \cdot 2H \cdot 4H$	1,10	10,50	3,15	1,10	3,80	1,10
If	$3N \cdot 2H \cdot 3H \cdot 2H \cdot 2H$	0,45	3,25	10,50	3,25	1,05	4,00
Ig	$3N \cdot 3H \cdot 4H$	1,10	1,10	3,20	10,30	3,20	1,10
IIa	$3N \cdot 2H \cdot 2H \cdot 3H$	—	10,27	3,50	1,10	4,10	1,10
IIb	$3N \cdot 2H \cdot 3H \cdot 2H$	—	3,35	10,15	3,35	1,00	3,68
IIc	$3N \cdot 3H \cdot 3H$	—	1,05	3,20	9,25	3,20	1,05
IId	$3N \cdot 3H \cdot 3H$	—	0,95	3,05	8,95	3,05	0,95
IIIa	$3N \cdot 2H \cdot 2H \cdot 3H$	—	9,60	3,35	1,10	4,00	1,10
IIIb	$3N \cdot 2H \cdot 3H \cdot 2H$	—	3,25	9,75	3,25	1,05	3,75
IIIc	$3N \cdot 3H \cdot 3H$	—	0,95	3,00	8,95	3,00	0,95
o-Nitrotoluene	$3N \cdot 2H \cdot 5H \cdot 3H$	3,32	11,30	3,32	1,04	3,74	1,04
m-Nitrotoluene	$3N \cdot 2H \cdot 3H \cdot 5H$	1,09	3,39	10,70	3,39	1,09	3,84
p-Nitrotoluene	$3N \cdot 4H \cdot 3H \cdot 3H$	3,90	1,14	3,31	11,00	3,31	1,14

\*The numbering of the atoms pertains to the aryl substituent.

Coupling of the unpaired electron with the paramagnetic nuclei of not only the nitrobenzene fragment but also with the proton of the substituent of the alkyl type. It is apparent from Table 2 that the hfs constants due to the nuclei of the atoms of the nitrobenzene fragment of the free radicals of the examined 1,4-DHP I are close to the constants of the anion radicals of the corresponding nitrotoluenes [5]. This constitutes evidence that the distribution of the unpaired electron in the investigated free radicals depends only slightly on the type of alkyl substituents under consideration (the  $CH_3$  group or the 1,4-DHP residue). However, the hfs constants from the protons of the alkyl substituent ( $a_{1'}$ ), which are due to hyperconjugation, differ for nitrotoluene and the corresponding 4-(nitro)aryl-1,4-DHP I. This difference in the hfs constants may be due to different orientations of the CH bond in the  $CH_3$  group and 1,4-DHP I with respect to the direction of the  $\rho_\pi$  orbital in the benzene ring.

In addition to the radicals presented above, the formation of free radicals with different spectrometric parameters is observed at higher ECG potentials. The character of the splitting of the EPR spectra of these radicals is the same as for the examined radicals; however, the hfs constants differ somewhat.

#### EXPERIMENTAL

The electrochemical investigations by means of the method of a rotating disk electrode with a ring and cyclical voltammetry were carried out with the apparatus described in [6]. The disk of the electrode was prepared from glass graphite with a working area of  $0.47 \text{ cm}^2$ , and the ring was prepared from platinum with an area of  $0.062 \text{ cm}^2$ . The experimental coefficient of efficiency of the electrode  $\eta_e = 0.16$ . Prior to each recording the electrode was polished successively with water-resistant sandpaper and filter paper and washed with acetone and acetonitrile. All of the potentials were measured relative to an Ag/AgNO<sub>3</sub> reference electrode (0.2 N, in acetonitrile with 0.1 M tetrabutylammonium perchlorate). The depolarizer concentration in all cases was  $5 \cdot 10^{-4}$  mole/liter, and 0.1 tetrabutylammonium perchlorate prepared by the method in [7] was used as the inert electrolyte. The acetonitrile was purified by the method in [8]. Under the experimental conditions the investigated solution contained from 1 to 10 mmole/liter of water as an impurity. The number of electrons transferred in electrochemical reactions and the coefficients of diffusion of the corresponding compounds were determined as in [2].

For the ECG of the free radicals we used  $10^{-3}$  to  $5 \cdot 10^{-4}$  M solutions of the investigated compounds in acetonitrile. The solutions contained tetrabutylammonium hexafluorophosphate

in a concentration of  $10^{-1}$  M. The free radicals were generated under steady-state conditions on a platinum cathode in the ECG cell placed in the rectangular resonator ( $H_{102}$ ) of an ER-9 EPR spectrometer (Karl Zeiss, Jena, East Germany). The spectra were recorded at a magnetic-field sweep rate of 0.04 G/sec with a degree of high-frequency (100 kHz) modulation of the magnetic field of 0.05-0.9 G and a recording time constant of 0.45 sec. The magnetic-field sweep was calibrated from the EPR spectrum of the nitrobenzene anion radical [9]. Theoretical verification of the interpretation of the EPR spectrum was accomplished with an HP 2116C minicomputer connected on line with the spectrometer [10].

The 1,2-DHP derivatives IIa, c, d were obtained by the method in [11] by reduction of the pyridinium perchlorates with sodium borohydride. 1,2,6-Trimethyl-3,5-diethoxycarbonyl-4-(3'-nitrophenyl)-1,2-dihydropyridine (IIb) was synthesized for the first time and had mp 107-109°C (from methanol). IR spectrum (in mineral oil): 1690 (C=O) and 1665  $\text{cm}^{-1}$  (C=C). UV spectrum (in ethanol),  $\lambda_{\text{max}}$  (log  $\epsilon$ ): 260 (4.25), 289 (4.21), and 385 nm (3.92). PMR spectrum ( $\text{CDCl}_3$ ): 0.66 (t, 3H, 3-OCH<sub>2</sub>CH<sub>3</sub>), 0.82 (t, 3H, 5-OCH<sub>2</sub>CH<sub>3</sub>), 1.26 (d, 3H, 2-CH<sub>3</sub>), 2.49 (s, 3H, 6-CH<sub>3</sub>), 3.26 (s, 3H, 1-CH<sub>3</sub>), 3.62 (q, 2H, 3-OCH<sub>2</sub>CH<sub>3</sub>), 3.84 (q, 2H, 5-OCH<sub>2</sub>CH<sub>3</sub>), 4.61 (q, 1H, 2-H),  $J_{\text{CH-CH}_3} = 6.5$  Hz, 7.29-7.53 (m, 2H), and 8.00-8.21 ppm (m, 2H, aromatic protons). Found: C 61.8; H 6.2; N 7.6%.  $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_6$ . Calculated: C 61.8; H 6.2; N 7.2%.

The synthesis and physicochemical characteristics of the remaining 1,2- and 1,4-DHP and the pyridinium perchlorates are presented in [3, 11].

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