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1,4-DIHYDROPYRIDINE-3,5-DI- AND 2-METHYL-4-ARYL-5-OXO-4,5-DIHYDRO-
1H-INDENO[1,2-b]PYRIDINE-3-CARBOTHIONIC ACID ETHYL ESTERS

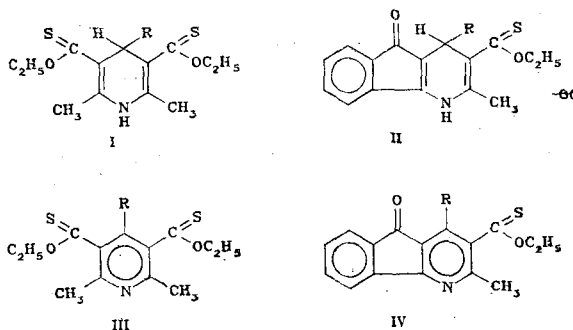
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Methods for the synthesis of 1,4-dihydropyridine-3,5-di- and 4-aryl-5-oxo-4,5-dihydro-1H-indeno[1,2-b]pyridine-3-carbothionic acid ethyl esters were developed. A comparative analysis of the physicochemical characteristics of this series of substances is given. Their reactivities in electrochemical and chemical oxidation reactions were studied. The electrochemical oxidation potentials of the thionic acid esters are found in a lower anodic range as compared with their oxygen analogs. According to the ionization constants, the thionic acid esters of 4,5-dihydroindenopyridines are stronger acids than the carbonyl esters; this is explained by participation of the free 3d orbitals of the sulfur atom in stabilization of the anion.

Highly effective coronary dilators [1, 2] and hypotensive agents [3] have been found among 4-aryl- and 4-hetaryl-1,4-dihydropyridines (1,4-DHP). 1-Unsubstituted 1,4-DHP are of practical interest as potential antioxidants [4].

Our previous communications [5, 6] were devoted to the results of a study of mono- and polycyclic 1,4-DHP with (alkylthio)carbonyl substituents in the β positions. In the present research we studied the effect of an ethoxythiocarbonyl substituent as compared with an ethoxycarbonyl substituent on the properties of 1,4-DHP (acidities and behavior in electrochemical and chemical oxidation).



4-Unsubstituted thionic ester I (R = H) is obtained by condensation of acetothioacetic O-ethyl ester (V) with urotropin.

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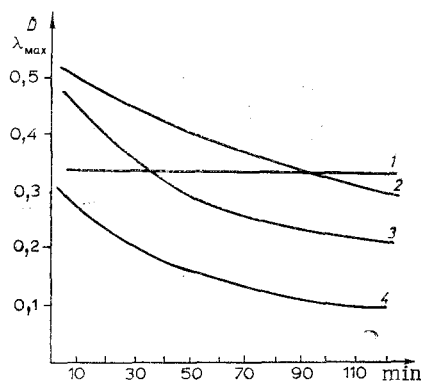
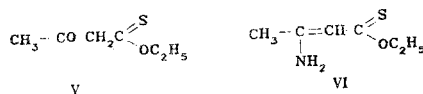


Fig. 1. Kinetic curves of the self-oxidation of 2,6-dimethyl-3,5-diethoxycarbonyl-1,4-dihydropyridine at $\lambda = 372$ nm (1) and of I (R = H) (2) at $\lambda = 480$ nm in $0.5 \cdot 10^{-4}$ molar solutions in ethanol in daylight and of the oxidation by chlor-anil in a molar ratio of 1:1 of I (R = H) (3) at $\lambda = 455$ nm and of 2,6-dimethyl-3,5-diethoxycarbonyl-1,4-dihydropyridine (4) at $\lambda = 360$ nm in $0.5 \cdot 10^{-4}$ molar solutions in benzene.



Ester V was condensed with the corresponding aldehydes and ammonium acetate by heating in acetic acid to synthesize 4-substituted 1,4-DHP (R = aryl, hetaryl). Acetaldehyde ammonia (1-aminoethanol) was used as the aldehyde component and the source of ammonia in the preparation of I (R = CH₃). The key compound — acetothioacetic O-ethyl ester (V) — is known [7, 8]; however, in order to increase the yield and simplify the method of synthesis we developed a fundamentally new method for its synthesis that consists in condensation of acetone with O,S-diethyl dithiocarbonate in 1,2-dimethoxyethane in the presence of sodium hydride.

β -Aminothioprotonic acid O-ethyl ester (VI) was obtained from ester V by the action of ammonia in a dry solvent at low temperature; primarily the oxygen ester is formed at room temperature. The reaction of VI with 2-arylideneindan-1,3-diones leads to ethyl 4-aryl-5-oxo-4,5-dihydro-1H-indeno[1,2-b]pyridine-3-carbothionates (II).

Thionic esters I and II are oxidized by nitrogen oxides to give pyridine derivatives III and indenopyridine derivatives IV (Table 2).

The UV, IR, and PMR spectroscopic data confirm the structures of I-IV (Tables 1 and 2). The absorption at 1120-1130 and 1220-1230 cm^{-1} can be assigned to the thiocarbonyl group of an ethoxythiocarbonyl substituent. In addition, the IR spectra of indeno-4,5-dihydropyridines of the II type contain two absorption maxima at 1630 and 1670 cm^{-1} , which characterize the benzoylene fragment of the molecule. Absorption of N-H groups is absent in the spectra of oxidized forms III and IV.

The UV spectra of I contain three absorption bands that are characteristic for monocyclic 1,4-DHP [9]; a 60-nm (average) bathochromic shift as compared with the carbonyl analogs and a 20-40-nm (average) bathochromic shift as compared with thiol esters [5] of the medium and long-wave maxima are observed for them. An \sim 30-nm hypsochromic shift of the long-wave maximum arises on passing from 4-unsubstituted dihydropyridine (R = H) to the 4-methyl derivative (I, R = CH₃), whereas both the 4-aryl and 4-hetaryl derivatives have 40-60-nm hypsochromic shifts of the long-wave maxima (Table 1). A similar effect of the introduction of substituents in the 4 position is also observed for oxygen esters [9].

TABLE 1. 1,4-Dihydropyridine-3,5-di- and 4-Aryl-5-oxo-4,5-dihydro-1H-indeno[1,2-b]pyridine-3-carbo-
thionic Acid Ethyl Esters (I and II)

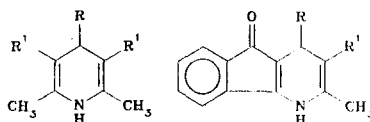
Com- pound	R	mp, °C	UV spectrum, λ_{max} , nm	IR spectrum at 1500- 1800 and 3000-3600 cm ⁻¹	PMR spectrum in deuteriochloro- form, δ , ppm	Found, %			Calculated, %			Yield, %		
						C	H	N	S	C	H		N	S
I	H	149	205, 250 sh 302, 480	1620, 3360	1.44 (t 6H, 2CH ₃); 2.20 (s 6H, 2,6-CH ₃); 3.77 (s 2H, 4-CH ₂); 4.64 (q 4H, 2OCH ₂); 5.53 (s NH)	55.1	16.8	4.8	22.8	54.7	16.7	4.9	22.5	33
I	CH ₃	97-99	205, 305, 450	1600, 3320, 1590 (m)	0.96 (d 3H, 4-CH ₃); 1.31 (t 6H, 2CH ₃); 2.24 (s 6H, 2,6-CH ₃); 4.49 (q 4H, 2OCH ₂); 4.76 (q, 1H, 4-CH); 5.69 (s NH)	56.1	17.0	4.7	21.8	56.1	17.1	4.7	21.4	30
I	C ₆ H ₅	128	205, 265 sh 307, 437	1600, 3380	1.33 (t 6H, 2CH ₃); 2.33 (s 6H, 2,6-CH ₃); 4.44 (q 4H, 2OCH ₂); 5.76 (s NH); 6.16 (s 1H, 4-CH); 7.16 (s 5H, C ₆ H ₅)	62.8	6.2	4.2	17.2	63.1	6.4	3.9	17.8	35
I	C ₆ H ₄ NO ₂ (4')	149- 151	205, 305, 437	1610, 3380	1.31 (t 6H, 2CH ₃); 2.33 (s 6H, 2,6-CH ₃); 4.44 (q 4H, 2OCH ₂); 5.82 (s NH); 6.22 (s 1H, 4-CH); 7.33-8.07 (m, aromatic protons)	56.0	5.5	7.4	16.2	56.1	5.5	6.9	15.8	39
I	C ₆ H ₄ N(3')	238	205, 270 sh 304, 423	1590, 3420	1.31 (t 6H, 2CH ₃); 2.33 (s 6H, 2,6-CH ₃); 4.44 (q 4H, 2OCH ₂); 6.09 (s 1H, 4-CH); 6.22 (s NH); 7.6-8.5 (m, aromatic protons)	59.0	5.9	7.2	17.3	59.6	6.1	7.7	17.7	51
II*	C ₆ H ₅	253	203, 237, 357, 508	1670, 3260, 1600	1.22 (t 3H, CH ₃); 2.38 (s 3H, 2-CH ₃); 4.33 (q 2H, OCH ₂); 5.24 (s 1H, 4-CH); 6.87 (s, aromatic protons); 10.16 (s NH)	72.8	5.2	3.8	9.00	73.1	5.3	3.9	8.9	41
II	C ₆ H ₄ NO ₂ (4')	256	203, 227, 265 345, 505	1670, 3300, 1600, 1635	1.26 (t 3H, CH ₃); 2.56 (s 3H, 2-CH ₃); 4.36 (q 2H, OCH ₂); 5.39 (s 1H, 4-CH); 7.07-8.16 (m, aromatic protons); 10.38 (s 1H, NH)	64.9	4.5	6.7	8.3	65.0	4.5	6.9	7.9	58
II	C ₆ H ₄ OCH ₃ (4')	245	203, 237, 270, 354, 508	1600, 1630, 1670, 3270	1.31 (t 3H, CH ₃); 2.44 (s 3H, 2-CH ₃); 3.64 (s 3H, OCH ₃); 4.42 (q 2H, OCH ₂); 5.24 (t 1H, 4-CH); 6.67-7.60 (m, aromatic protons); 10.13 (s, NH)	71.1	5.3	3.8	8.9	70.6	5.4	3.6	8.2	45
II	C ₆ H ₄ Br(4')	233	203, 336, 270, 355, 508	1600, 1630, 1670, 3280	1.29 (t 3H, CH ₃); 2.40 (s 3H, 2-CH ₃); 4.35 (s 2H, OCH ₂); 5.32 (s 1H, 4-CH); 6.80-7.20 (m, aromatic protons); 10.20 (s NH)	59.1	4.3	3.5	7.6	58.8	4.0	3.2	7.3	51

*The PMR spectra of II were obtained from solutions in dimethyl sulfoxide (DMSO).

TABLE 2. Pyridine-3,5-di- and 4-Aryl-5-oxo-5H-indeno[1,2-b]-3-carbothionic Acid Ethyl Esters (III and IV)

Yield, %	Calculated, %				Empirical formula	Found, %				PMR spectrum in deuteriochloroform, δ , ppm	mp, °C	R
	C	H	N	S		C	H	N	S			
80	55,10	6,1	4,9	22,6	$C_{13}H_{17}NO_2S_2$	55,10	6,1	4,9	22,6	1,47 (t 6H, 2CH ₃); 2,80 (s 6H, 2,6-CH ₃); 4,69 (q 4H, 2OCH ₂); 8,67 (s, 1H, 4-CH)	75-77	H
34	56,5	6,4	4,7	21,6	$C_{14}H_{19}NO_2S_2$	56,3	5,9	4,5	20,9	0,85 (s 3H, 4-CH ₃); 1,12 (t 6H, 2CH ₃); 2,40 (s 6H, 2,6-CH ₃); 4,29 (q 4H, 2OCH ₂)	45-48	CH ₃
61	63,5	6,0	3,9	17,8	$C_{19}H_{21}NO_2S_2$	63,2	6,0	3,6	17,2	1,07 (t 6H, 2CH ₃); 2,49 (s 6H, 2,6-CH ₃); 4,24 (q 4H, 2-OCH ₂); 7,17 (s 5H, C ₆ H ₅)	68-70	C ₆ H ₅
85	56,4	5,0	6,9	15,9	$C_{19}H_{20}N_2O_4S_2$	56,3	4,9	7,1	15,5	1,13 (t 6H, 2-CH ₃); 2,49 (s 6H, 2,6-CH ₃); 4,26 (q 4H, 2-OCH ₂ -); 7,27-8,07 (m, aromatic protons)	146-147	C ₆ H ₄ NO ₂ (4')
86	73,5	4,8	3,9	8,9	$C_{22}H_{17}NO_2S$	72,8	5,0	4,2	9,1	1,10 (t 3H, CH ₃); 2,50 (s 3H, 2-CH ₃); 4,26 (q 2H, OCH ₂); 7,20 (s, 5H, C ₆ H ₅)	153	C ₆ H ₅
80	70,9	4,9	3,6	8,2	$C_{23}H_{19}NO_3S$	71,1	5,3	3,8	8,7	1,27 (t 3H, CH ₃); 2,64 (s 3H, 2-CH ₃); 3,84 (s 3H, OCH ₃); 4,44 (q 2H, -OCH ₂); 6,84-7,91 (m, aromatic protons)	139-141	C ₆ H ₄ OCH ₃ (4')
74	60,3	3,7	3,2	7,3	$C_{22}H_{18}BrNO_2S$	59,8	3,9	3,2	7,6	1,15 (t 3H, CH ₃); 2,45 (s 3H, 2-CH ₃); 4,28 (q 2H, OCH ₂); 7,10-7,92 (m, aromatic protons)	209	C ₆ H ₄ Br (4')
79	65,3	4,0	6,9	7,9	$C_{22}H_{18}N_2O_4S$	65,6	3,7	6,6	8,1	1,13 (t 3H, CH ₃); 2,49 (s 3H, 2-CH ₃); 4,26 (q 2H, -OCH ₂); 7,27-8,08 (m, aromatic protons)	158	C ₆ H ₄ NO ₂ (4')

TABLE 3. Potentials of the Peaks and Half Waves in the Electrochemical Oxidation of Ethyl Esters of Derivatives of the 1,4-Dihydropyridine and 5 Oxindeno-4,5-dihydropyridine Series



R	R'	E_n, V	$E_{1/2}, V$	R	R'	$E_{1/2}, V$
H	COOC ₂ H ₅	0,97	0,89	C ₆ H ₅	COOC ₂ H ₅	1,10
H	CSOC ₂ H ₅	0,93	0,83	C ₆ H ₅	CSOC ₂ H ₅	1,10
CH ₃	COOC ₂ H ₅	1,04	0,96	C ₆ H ₄ NO ₂ (4')	COOC ₂ H ₅	1,23
CH ₃	CSOC ₂ H ₅	1,01	0,93	C ₆ H ₄ NO ₂ (4')	CSOC ₂ H ₅	1,16
C ₆ H ₅	COOC ₂ H ₅	1,12	1,08	C ₆ H ₄ OCH ₃ (4')	COOC ₂ H ₅	1,15
C ₆ H ₅	CSOC ₂ H ₅	1,06	0,98	C ₆ H ₄ OCH ₃ (4')	CSOC ₂ H ₅	1,05
C ₆ H ₄ NO ₂ (4')	COOC ₂ H ₅	1,22	1,18			
C ₆ H ₄ NO ₂ (4')	CSOC ₂ H ₅	1,14	1,06			

A shift of the signals of the methylene and methyl protons of the ethoxythiocarbonyl substituent to weak field as compared with carbonyl esters is observed in the PMR spectra of I-IV (Tables 1 and 2). A weak-field shift also occurs for the C-H protons adjacent to the thiocarbonyl group in the spectra of thioketones and thioamides [10-12]. One also observes a certain paramagnetic shift of the protons attached to the carbon atom in the 4 position and of the N-H protons as compared with the corresponding oxygen analogs [13]; this shift is difficult to interpret unambiguously.

Replacement of the oxo group of the ethoxycarbonyl substituent in the β position of the 1,4-dihydropyridine ring by a thiocarbonyl function gives rise to a 0.04-0.10 V (average) decrease in the electrochemical oxidation (EO) potentials, i.e., thionic esters of the I and II type are oxidized more readily than the carbonyl analogs, which indicates the less

pronounced electron-acceptor effect of the $-\overset{\text{S}}{\text{C}}\begin{matrix} \diagup \\ \diagdown \end{matrix} \text{OC}_2\text{H}_5$ grouping as compared with the ethoxycarbonyl substituent [14] if one assumes that they have approximately identical spatial orientations.

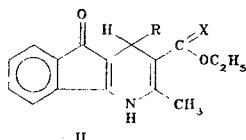
The rate constants for the oxidation of I (R = H) and its oxygen analog by chloranil in benzene are approximately the same, viz., 320-370 and 320-400 $\text{K} \cdot 10^2$ units (liters per mole per second) respectively (an oxidation rate constant of 450 $\text{K} \cdot 10^2$ units is given in [15] for the oxygen analog).

Much faster oxidation is observed for DHP I (R = H) as compared with the oxygen ester in the case of self-oxidation by air oxygen in light in DMSO or ethanol (Fig. 1). The mechanism of self-oxidative transformations has not been elucidated, but it may be assumed that it has the character of a photochemical radical reaction. The formation of pyridine III (R = H) in this process was rigorously proved by the distinct appearance of a signal of a proton attached to the carbon atom in the 4 position (δ 8.27 ppm) and the detection of other signals that are characteristic for oxidized form III (R = H) (Table 2).

The introduction of substituents in the 4 position of I leads to a 0.1-0.15 V increase in the EO potentials; this is in agreement with the regularities observed in the oxygen ester series [14].

The determination of the ionization constants of ethoxythiocarbonyl derivatives of 4,5-dihydro-1H-indenopyridine series II (Table 4) by spectrophotometry [16] makes it possible to draw the following conclusions. First, the introduction of an electron-acceptor substituent in the γ -phenyl residue increases the acidity of the compound, whereas the presence of an electron-donor methoxy group in it decreases the acidity somewhat as compared with an unsubstituted phenyl ring. Second, the pK_a values of thionic acid esters II are 0.2-0.3 units lower than in the case of the previously investigated oxygen analogs [6], i.e., the sulfur-containing esters are stronger N-H acids. The reason for this probably

TABLE 4. Ionization Constants (pK_a) of II and Their Oxygen Analogs in 50% Ethanol



R	X	pK_a
C_6H_5	O	$12,74 \pm 0,05$
C_6H_5	S	$12,52 \pm 0,05$
$C_6H_4NO_2$ (4')	O	$12,15 \pm 0,03$
$C_6H_4NO_2$ (4')	S	$11,87 \pm 0,03$
$C_6H_4OCH_3$ (4')	O	$12,84 \pm 0,05$
$C_6H_4OCH_3$ (4')	S	$12,63 \pm 0,05$
C_6H_4Br (4')	O	$12,40 \pm 0,05$
C_6H_4Br (4')	S	$12,15 \pm 0,05$

lies in the fact that the ethoxythiocarbonyl substituent, owing to the presence of free 3d orbitals of the sulfur atom, is capable of participating in stabilization of the anion through the enamine conjugation chain.

The effect of participation of the free 3d orbitals of the sulfur atom in stabilization of carbanions or the participation of the vacant d orbitals of the sulfur atom in conjugation is well known in compounds with sulfonyl, sulfide, or thioamide groupings [11, 12, 17-20].

EXPERIMENTAL

The IR spectra of suspensions of the compounds in mineral oil or hexachlorobutadiene were recorded with a UR-20 spectrometer. The UV spectra of $4 \cdot 10^{-5}$ molar solutions in ethanol were obtained with a Specord UV-vis spectrophotometer. The PMR spectra were obtained with Perkin-Elmer R12A (60 MHz) and Bruker WH90/DS (90 MHz) spectrometers with hexamethyldisiloxane or tetramethylsilane as the internal standard. The electrochemical oxidation (EO) potentials were determined by the previously described method with an LP-60 recording polarograph in acetonitrile with a rotating platinum microelectrode [21]. The rate constants for oxidation by chloranil in benzene at $37^\circ C$ were determined spectrophotometrically from the disappearance of the long-wave absorption maximum at 453 nm by the method in [14].

Acetothioacetic Acid O-Ethyl Ester (V). An 11.6-g (0.2 mole) sample of acetone was added slowly with stirring at room temperature in an argon atmosphere to a suspension of 9.6 g (0.4 mole) of sodium hydride in 200 ml of absolute 1,2-dimethoxyethane. After 30 min, 45 g (0.25 mole) of O,S-diethyl dithiocarbonate was added dropwise, and the mixture was stirred at room temperature for 1 h and refluxed for 4 h. The resulting solution was cooled and poured over ice, and the aqueous mixture was acidified with cold 1 N hydrochloric acid and extracted with methylene chloride. The extract was dried with magnesium sulfate and distilled *in vacuo* to give 23.3 g (80%) of a yellow oil with bp $89-90^\circ C$ (20 mm), the spectral characteristics of which were in agreement with the data in [8].

β -Aminothioprotonic Acid O-Ethyl Ester (VI). A 14.6-g (0.1 mole) sample of ester V was dissolved in 50 ml of absolute ether, and the solution was cooled to $0^\circ C$ and saturated with dry ammonia for 2 h. The mixture was then dried with anhydrous magnesium sulfate for 1 h, after which the ether was removed immediately *in vacuo*, and the residue was distilled at $96-100^\circ C$ (10 mm) to give 4.8 g (33%) of product. Found: C 48.9; H 7.4; N 9.1; S 21.5%. $C_6H_{11}NOS$. Calculated: C 49.6; H 7.6; N 9.6; S 22.1%.

2,6-Dimethyl-3,5-diethoxythiocarbonyl-1,4-dihydropyridine (I, R = H). A 2.92-g (0.02 mole) sample of ester V was refluxed with 0.70 g of urotropin and 0.53 g of ammonium acetate in 2 ml of absolute ethanol for 10 min, after which the mixture was cooled, and the orange crystals were separated and crystallized from methanol. The properties of this substance are presented in Table 1.

4-Substituted 2,6-Dimethyl-3,5-diethoxythiocarbonyl-1,4-dihydropyridines (I, Table 1). A 2.96-g (0.02 mole) sample of ester V and 0.01 mole of the corresponding aldehyde were refluxed with 2.4 g of ammonium acetate in 5 ml of glacial acetic acid for 30 min, after which the mixture was poured into water. The aqueous mixture was extracted with ether (three 30-ml portions), and the extract was dried over anhydrous sodium sulfate. The ether was removed, and the residue was chromatographed with a column packed with aluminum oxide [elution with benzene-ethyl acetate (20:1) or chloroform-hexane-acetone (9:7:1)] to give the corresponding DHP I in the form of orange crystals after crystallization from methanol.

2-Methyl-3-ethoxythiocarbonyl-4-aryl-5-oxo-4,5-dihydro-1H-indeno[1,2-b]pyridines (II, Table 1). A 0.01-mole sample of the corresponding 2-arylideneindan-1,3-dione was dissolved in 5 ml of glacial acetic acid, 3 g (0.02 mole) of amino ester VI was added, and the mixture was refluxed for 5 min. It was then cooled, and the precipitated dark-red crystals were recrystallized from acetic acid.

4-Substituted 2,6-Dimethyl-3,5-diethoxythiocarbonylpyridines (III, Table 2). A 1-mmole sample of the corresponding DHP I was dissolved in excess 6 N nitric acid by heating to 60°C, after which the solution was cooled to precipitate yellowish crystals of pyridine III, which were crystallized from dilute methanol.

2-Methyl-3-ethoxythiocarbonyl-4-aryl-5-oxo-5H-indeno[1,2-b]pyridines (IV, Table 2). A 1-mmole sample of the corresponding DHP II was dissolved in 2 ml of glacial acetic acid, an excess amount of sodium nitrite was added, and the mixture was diluted with water after 10 min. The precipitated yellow crystals were crystallized from ethanol.

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