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CONDENSED PYRIDINE BASES SYNTHESIS OF SOME BENZO[b]FURO[2,3-c]-, BENZO[b]- THIENO[2,3-c]-, AND BENZO[b]SELENOPHENO[2,3-c]-QUINOLINES

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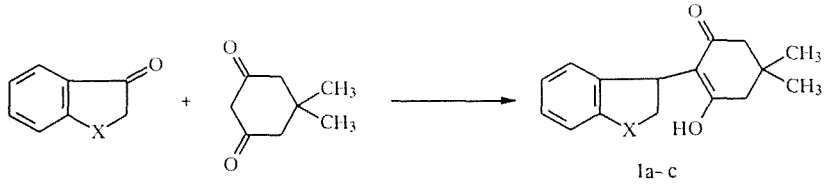
Condensation of benzo[b]furan-3(2H-one, benzo[b]thiophen-3(2H)-one and benzo[b]selenophen-3(2H)-one with dimedone gives 2-(3-heteryl)dimedones. Acylation of the latter leads to the corresponding tetracyclic pyrylium salts, from which condensed quinolines are obtained. Some condensed quinoline derivatives are obtained by reaction of 1-oxo-1,2,3,4-tetrahydroheterene[2,3-c]quinolines with sodium borohydride, hydrazine, and hydroxylamine.

Pharmacological research on a large number of β-carboline derivatives that were synthesized in our laboratory has revealed that the most active of them is 1-oxo-3,3,6-trimethyl-1,2,3,4-tetrahydroindolo[2,3-c]quinoline hydrochloride [1-3]. The broad spectrum of pharmacological action of this compound (tranquilier, nootrope, antishock agent) and its high activity have stimulated us to synthesize its isosteres — the oxygen, sulfur, and selenium analogs. The isomeric benzofuroquinolines have previously been investigated as mutagens and antitumor agents [4, 5].

Pyrylium salts are suitable intermediates for the synthesis of these analogs. To obtain the latter we introduced 2-(3-benzo[b]furyl)-, 2-(3-benzo[b]thienyl)- and 2-(3-benzo[b]selenyl)-dimedone (1a-c respectively) in an acid-catalyzed heterocyclization reaction. We have demonstrated the possibility of obtaining compounds Ia-c by condensing benzo[b]furan-3(2H)-one, benzo[b]thiophen-3(2H)-one, and benzo[b]selenophen-3(2H)-one with dimedone. The reaction takes place readily on heating a mixture of the reactants in acetic acid in the presence of triethylamine (cf. [6]). According to the IR spectroscopic data, products 1a-c exist in the enol form.



Ia-c

I a X = O, b X = S, c X = Se

Acylation of compounds Ia and Ib with aliphatic anhydrides in the presence of 70% perchloric acid leads to 1-oxo-3,3-dimethyl-6-R-1,2,3,4-tetrahydrobenzo[b]furo[2,3-c]pyrylium (IIa-c) and 1-oxo-3,3-dimethyl-6-R-1,2,3,4-tetrahydrobenzo[b]thieno[2,3-c]pyrylium (IIIa-c) perchlorates in high yield. Under these conditions 2-(3-benzo[b]selenyl)dimedone Ic undergoes degradation with elimination of H_2Se (the degradation of Ic frequently occurs explosively). Acylation of the substituted dimedone Ic occurs successfully in the presence of BF_3 etherate and leads to salt IV, which was not isolated in a pure form but was converted to the corresponding quinoline by the action of alcoholic ammonia.

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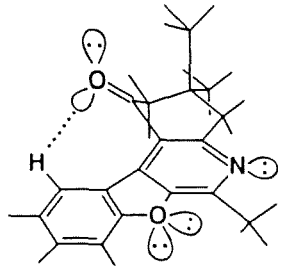
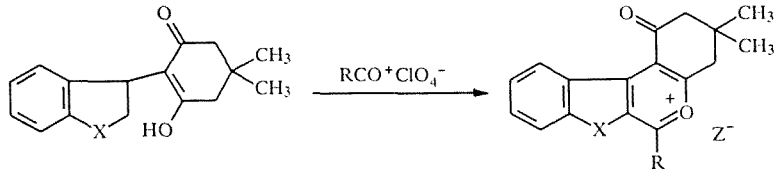


Fig. 1. Energetically most favorable conformation of 1-oxo-3,3,6-trimethyl-1,2,3,4-tetrahydrobenzo[b]furo[2,3-c]quinoline from the results of molecular mechanical calculations.

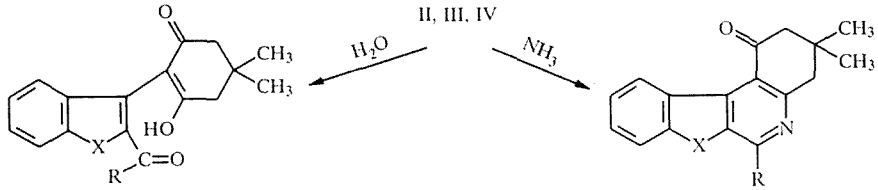
In the IR spectra of salts II and III there are absorption bands due to a carbonyl group at 1710 cm^{-1} and a ClO_4^- anion at 1100 cm^{-1}.



Ia-c IIa-c, IIIa-в, IV

II X = O, III X = S, IV X = Se; II, III Z = ClO_4, a R = CH_3, b R = C_2H_5, c R = C_3H_7, Z = BF_4

Absorption bands due to the C=C bonds of the pyrylium cation in salts II occur at 1645 cm^{-1} whereas in salts III they occur at 1590 cm^{-1}. In contrast to tricyclic benzo[b]thieno[2,3-c]- and benzo[b]furo[2,3-c]-pyrylium salts [7, 8], perchlorates II and III and tetrafluoroborate IV are converted in aqueous alcoholic solution to the tricarbonyl compounds Va-c, VIa-c, and VII respectively.



Va-в, VIa-в, VII VIIIa-в, IXa-в, X

V, VIII X = O, a R = CH_3, b R = C_2H_5, c R = C_3H_7; VI, IX X = S, a R = CH_3, b R = C_2H_5, c R = C_3H_7; VII, X X = Se, R = CH_3

The IR spectra of compounds V-VII contain a set of absorption bands due to the C=C and C=O bonds of the enol form in the region of 1615-1650 cm^{-1}. The RCO group in compounds V occurs at 1685 cm^{-1}, while in compounds VI and VII the absorption occurs at 1665 cm^{-1}.

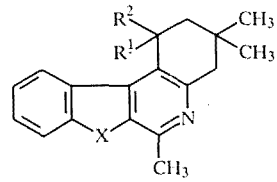
The desired compounds 1-oxo-3,3-dimethyl-6-R-1,2,3,4-tetrahydro-benzo[b]furo[2,3-c]quinolines, -benzo[b]thieno-[2,3-c]quinolines and -benzo[b]seleno[2,3-c]quinolines (VIII-X) have been synthesized by treatment of salts II-IV or the tricarbonyl compounds V-VII with an alcoholic solution of ammonia. It should be noted that, as in the case of the pyrylium cation, quinolines VIII-X exhibit an unusually large downfield shift of the 11-H aromatic proton signal in their PMR spectrum (see Tables 2 and 3). To identify the possible causes of this phenomenon, we carried out a theoretical study of the structure and intramolecular interaction energies of compounds VIII-X using molecular mechanical methods (MMKh 88 force field) and SCF MO LCO (see Table 3). The energetically most favorable conformation for X = O is shown in Fig. 1. On changing from O to S the distance between the carbonyl oxygen and the 11-H proton decreases appreciably, while at the same time their Van der Waals repulsion energy (E_{vdw}) increases by a factor of approximately 1.5 while their coulombic attraction (E_q) increases slightly. The magnitude of the latter energy suggests the existence of weak hydrogen bonding. However, it is quite clear that this hydrogen bonding could not lead to such a large downfield shift of the 11-H proton signal (by more than 2 ppm). The most

TABLE 1. Properties of Compounds I-XIV

Com pound	Empirical formula	mp, °C (T_{dec}, °C)	Yield, %	Com-pound	Empirical formula	mp, °C	Yield, %
Ia	$C_{16}H_{16}O_3$	158...159	64	VIIIb	$C_{19}H_{19}NO_2$	117...118	89
Ib	$C_{16}H_{16}O_2S$	206...207	68	VIIIc	$C_{21}H_{21}NO_2$	89...90	90
Ic	$C_{16}H_{16}O_2Se$	197...198	62	IXa	$C_{18}H_{17}NOS$	106...107	91
IIa	$C_{18}H_{17}ClO_7$	(240)	81	IXb	$C_{19}H_{19}NOS$	124...125	92
IIb	$C_{19}H_{19}ClO_7$	(217)	81	IXc	$C_{20}H_{21}NOS$	99...100	90
IIc	$C_{20}H_{21}ClO_7$	(167)	84	X	$C_{18}H_{17}NOSe$	97...98	87
IIIa	$C_{18}H_{17}ClO_6S$	(242)	80	XIa	$C_{18}H_{18}NO_2$	121...122	84
IIIb	$C_{19}H_{19}ClO_6S$	(210)	81	XIb	$C_{18}H_{18}NOS$	166...167	84
IIIc	$C_{20}H_{21}ClO_6S$	(160)	83	XIc	$C_{18}H_{18}NOSe$	161...162	84
IV	$C_{18}H_{17}BF_4O_2Se$	—	—	XIIa	$C_{18}H_{19}N_3O$	151...153	82
Va	$C_{18}H_{18}O_4$	(190)	91	XIIb	$C_{18}H_{19}N_3S$	155...156	79
Vb	$C_{19}H_{20}O_4$	(102)	90	XIIc	$C_{18}H_{18}NOSe$	164...165	79
Vc	$C_{20}H_{22}O_4$	(98)	93	XIIIa	$C_{18}H_{19}NO$	103...104	87
VIa	$C_{18}H_{18}O_3S$	(178)	90	XIIIb	$C_{18}H_{19}NS$	131...132	72
VIb	$C_{19}H_{20}O_3S$	(164)	93	XIIIc	$C_{18}H_{19}NSe$	132...133	54
VIc	$C_{20}H_{22}O_3S$	(152)	92	XIVa	$C_{18}H_{18}N_2O_2$	277...278	65
VII	$C_{18}H_{18}O_3Se$	(144)	90	XIVb	$C_{18}H_{18}N_2OS$	217...218	76
VIIIa	$C_{18}H_{17}NO_2$	144...145	91	XIVc	$C_{18}H_{18}N_2OSe$	215...216	77

likely origin of this phenomenon is a coordinated effect from the coulombic and Van der Waals interactions, with the latter predominating. A structural characteristic of quinolines VIII-X is the projection of the carbonyl group from the plane of the aromatic system. In such a conformation one unpaired oxygen pair participates in weak hydrogen bonding with the 11-H proton while the other remains free and can form a hydrogen bridge with proton donors (alcohols, acids, etc.).

The low reactivity of quinolines VIII-X stems from their structural features. Thus, their hydride reduction to form the corresponding carbinols XIa-c occurs relatively slowly while the reaction with a tenfold excess of hydrazine hydrate proceeds satisfactorily only with prolonged (15 h) heating of the reactants and leads to hydrazine XIIa-c. The latter are converted to compounds XIIIa-c by heating with KOH in ethylene glycol. The nature of the solvent significantly affects the rate of reaction between quinolines VIII-X and hydrazine hydrate. In methanol the reaction occurs much more rapidly than in isopropanol, while in dioxane it hardly takes place at all. This difference is obviously due to the general acid catalysis by the proton-containing solvents of the nucleophilic reactions of the carbonyl group [9]. Thus, in an aprotic solvent such as dioxane, where electrophilic solvation of the carbonyl oxygen is impossible, reaction does not occur. At the same time addition of a small quantity of acetic acid to the reaction medium causes a rapid acceleration of the process (the reaction is complete after 2-3 h). A similar pattern occurs in the reaction between compounds VIII-X and hydroxylamine. The use of traditional methods for preparing oximes [10] requires prolonged boiling of the initial reactants. In our case good yields are achieved by boiling the hydrochlorides of VIII-X with hydroxylamine hydrochloride in ethanol and also by "lethargic" oximation [11], which lead to oximes XIVa-c. The properties of compounds I-XIV are given in Table 1.



XIa-c - XIVa-c

XIa-c $R^1 = OH, R^2 = H$; XIIa-c $R^1 + R^2 = N—NH_2$; XIIIa-c $R^1 = R^2 = H$;
XIVa-c $R^1 + R^2 = N—OH$; a X=O, b X=S, c X=Se

Pharmacological studies on the quinolines VIII-X synthesized and their conversion products XI-XIV were carried out by the Department of Pharmacology at the Donetsk Medical Institute. It was shown that benzothienoquinolines have high axiolytic activity (the conflict situation method). Comparison of the axiolytic activity of compounds IXa and XIIIb established the role of the oxo group at the 1-position. Compounds in which it is absent do not exhibit any anticonflict activity up to 10 mg/kg dose levels.

TABLE 2. Spectroscopic Properties of Compounds II, III, VIII-XIV

Compound	PMR spectrum, δ, ppm; spin–spin coupling constant (J), Hz
IIa	1,37 (6H, s, 3-CH_3); 3,10 (2H, s, 2-CH_2); 3,37 (3H, s, 6-CH_3); 3,60 (2H, s, 4-CH_2); 7,63...8,20 (3H, m, 8-, 9-, 10-H); 9,33 (1H, d, J = 8, 11-H)
IIb	1,33 (6H, s, 3-CH_3); 1,66 (3H, t, 6-CH_3); 3,00 (2H, s, 2-CH_2); 3,53 (2H, s, 4-CH_2); 3,67 (2H, q, 6-CH_2); 7,60...8,20 (3H, m, 8-, 9-, 10-H); 9,33 (1H, d, J = 8, 11-H)
IIIa	1,26 (6H, s, 3-CH_3); 3,00 (2H, s, 2-CH_2); 3,26 (3H, s, 6-CH_3); 3,46 (2H, s, 4-CH_2); 7,67...8,07 (3H, m, 8-, 9-, 10-H); 9,27 (1H, d, J = 8, 11-H)
IIIb	1,26 (6H, s, 3-CH_3); 1,60 (3H, t, 6-CH_3); 3,00 (2H, s, 2-CH_2); 3,46 (2H, s, 4-CH_2); 3,60 (2H, q, 6-CH_2); 7,60...8,00 (3H, m, 8-, 9-, 10-H); 9,27 (1H, d, J = 8, 11-H)
VIIIa	1,14 (6H, s, 3-CH_3); 2,71 (2H, s, 2-CH_2); 2,86 (3H, s, 6-CH_3); 3,28 (2H, s, 4-CH_2); 7,47 (1H, t, 10-H); 7,68 (1H, t, 9-H); 7,75 (1H, d, J = 8, 8-H); 9,57 (1H, d, J = 8, 11-H)
IXa	1,08 (6H, s, 3-CH_3); 2,78 (3H, c, 6-CH_3); 2,80 (2H, s, 2-CH_2); 3,27 (2H, s, 4-CH_2); 7,52 (1H, t, 10-H); 7,61 (1H, t, 9-H); 8,04 (1H, d, J = 8, 8-H); 9,56 (1H, d, J = 8,2, 11-H)
X	1,08 (6H, s, 3-CH_3); 2,66 (3H, c, 6-CH_3); 2,80 (2H, s, 2-CH_2); 3,25 (2H, s, 4-CH_2); 7,43 (2H, t, 10-, 9-H); 8,05 (1H, d, J = 8, 8-H); 9,18 (1H, d, J = 8,2, 11-H)
XIb	1,13 (3H, s, 3-CH_3); 1,35 (3H, s, 3-CH_3); 2,28 (2H, m, 2-CH_2); 2,75 (3H, s, 6-CH_3); 3,00 (1H, d, J = 15, 4-CH); 3,23 (1H, d, J = 15, 4-CH); 5,87 (1H, m, 1-CH); 6,74 (1H, d, J = 6, 1-OH); 7,52 (2H, m, 9-, 10-H); 8,04 (1H, d, J = 8, 8-H); 9,26 (1H, d, J = 8,5, 11-H)
XIc	1,13 (3H, s, 3-CH_3); 1,35 (3H, s, 3-CH_3); 2,28 (2H, m, 2-CH_2); 2,75 (3H, s, 6-CH_3); 3,00 (1H, d, J = 15, 2-CH); 3,23 (1H, d, J = 15, 2-CH); 5,87 (1H, m, 1-CH); 6,74 (1H, d, J = 6, 1-OH); 7,50 (2H, m, 9-, 10-H); 8,13 (1H, d, J = 8, 8-H); 9,27 (1H, d, J = 8, 11-H)
XIIb	1,05 (6H, c, 3-CH_3); 2,70 (2H, c, 2-CH_2); 2,77 (3H, c, 6-CH_3); 3,02 (2H, c, 4-CH_2); 7,22 (2H, c, NH_2); 7,41 (1H, t, 10-H); 7,52 (1H, t, 9-H); 7,98 (1H, d, J = 7,4, 8-H); 9,48 (1H, d, J = 8,8, 11-H)
XIIIb	1,03 (6H, c, 3-CH_3); 1,68 (2H, t, J = 6,6, 2-CH_2); 2,78 (3H, c, 6-CH_3); 3,00 (2H, c, 4-CH_2); 3,24 (2H, t, J = 6,6, 1-CH_2); 7,58 (2H, m, 9-, 10-H); 8,06 (1H, d, J = 9, 8-H); 8,39 (1H, d, J = 10, 11-H)
XIVc	1,10 (6H, c, 3-CH_3); 2,74 (3H, c, 6-CH_3); 3,05 (2H, c, 2-CH_2); 3,16 (2H, c, 4-CH_2); 3,63 (1H, c, N—OH); 7,20 (2H, m, 9-, 10-H); 8,14 (1H, d, J = 8,2, 8-H); 9,50 (1H, d, J = 8,2, 11-H)

TABLE 3. Results of Theoretical Calculations on Quinolines VIII-X

Hetero atom X	Van der Waals radius of X, Å	11-H chemical shift, ppm	11-H...O=C< distance, Å	E_q, kcal/mole	E_{vdw}, kcal/mole	E, kcal/mole
O	1,40	9,54	2,397	-0,846	0,543	-0,303
N	1,50	9,88	2,374	-0,869	0,620	-0,249
S	1,85	9,58	2,3404	-0,863	0,751	-0,112
Se	2.00	9,15	—	—	—	—

EXPERIMENTAL

The IR spectra were recorded on a UR-20 instrument in petrolatum oil; the PMR spectra were recorded on Tesla BS-467 (60 MHz) and Gemini-200 (200 MHz) instruments, with CF_3COOH as solvent for the pyrylium salts and C_5D_5N for the pyridine bases, and TMS used as the internal standard. The theoretical calculations using the molecular mechanical and SCF MO LCAO methods were carried out on an IBM PC/AT 286/287 using the PCMODEL program, version 3.2 (Serena Software).

The elemental analysis data for C, H, N, Cl, and S in the synthesized compounds corresponded to the calculated values.

2-(3-Heteryl)dimedones (Ia-c). A mixture of 0.1 mole of the respective benzo[b]heteren-3(2H)-one, 0.1 mole of dimedone, 0.1 mole of triethylamine, and 200 ml of acetic acid was refluxed for 8 h. The acetic acid was removed under vacuum. The residue was dissolved in a 5% solution of $NaHCO_3$. The resulting solution was filtered and acidified. The precipitate that formed was filtered off and recrystallized from isopropanol.

1-Oxo-3,3-dimethyl-6-R-1,2,3,4-tetrahydrobenzo[b]furo[2,3-c]pyrylium and -benzo[b]thieno[2,3-c]pyrylium Perchlorates (IIa-c, IIIa-c). To a solution of 0.01 mole of compound Ia or Ib in 15 ml of the respective carboxylic anhydride

at room temperature and with agitation was added 1 ml of 70% perchloric acid. The mixture was agitated for 1 h at 35-40°C and for 2 h at room temperature; it was then cooled, 40 ml of dry ether was added, and the precipitate was filtered off, washed with dry ether, dried, and recrystallized from glacial acetic acid.

1-Oxo-3,3,6-trimethyl-1,2,3,4-tetrahydrobenzo[b]selenopheno[2,3-c]pyrylium Borofluoride (IV). This was obtained in a similar manner to compounds II and III by using boron trifluoride etherate (3 ml). The mixture was kept for 24 h at room temperature. The salt was precipitated by the addition of 100 ml of ether and was used without further purification.

2-(2-Acylbenzo[b]-3-furyl)-, (2-(2-Acylbenzo[b]-3-thienyl-)- and 2-(2-Acetylbenzo[b]-3-selenyl)-dimedones (Va-c, VIa-c, and VII). The respective salt IIa-c, III-c or IV (0.01 mole) was dissolved in 50 ml of ethanol, 5 ml of water was added, and the mixture was heated to boiling. After cooling, 10 ml of water was added, and the precipitate that formed was filtered off and crystallized from aqueous ethanol.

1-Oxo-3,3-dimethyl-6-R-1,2,3,4-tetrahydrobenzo[b]furo(thieno or selenopheno)[2,3-c]quinolines (VIIIa-c, IXa-c, X). Gaseous ammonia was passed through a solution of 0.01 mole of salt IIa-c, IIIa-c or IV in 50 ml of ethanol for 20 min, and the mixture was cooled and diluted with 100 ml of water. The precipitate that formed was filtered off and crystallized from aqueous ethanol. Quinolines VIII-X may also be obtained in a similar manner from the tricarbonyl compounds V-VII.

1-Hydroxy-3,3,6-trimethyl-1,2,3,4-tetrahydrobenzo[b]furo(thieno or selenopheno)[2,3-c]quinolines (XIa-c). A. To a suspension of 0.01 mole of $LiAlH_4$ in 200 ml of dry ether was added in small portions 0.01 ml of quinoline VIIIa, IXa, or X. The mixture was refluxed for 4 h, cooled, and treated in succession with ethanol, water, and acetic acid. The ether layer was separated, washed with water, and dried over Na_2SO_4. After removal of the ether, the residue was crystallized from toluene. **B.** To a solution of 0.01 mole of VIIa, IXa, or X in 60 ml of methanol was added 0.048 mole of $NaBH_4$ in small portions. The solution was left for 24 h at room temperature. The methanol was then removed, water was added to the residue, and the precipitate of product was filtered off.

1-Oxo-3,3,6-trimethyl-1,2,3,4-tetrahydrobenzo[b]furo(thieno or selenopheno) [2,3-c]quinoline Hydrazones (XIIa-c). A mixture of 0.01 mole of the respective quinoline VIIIa, IXa, or X, 10 ml of 65% hydrazine hydrate, 100 ml of ethanol (50 ml) was distilled off, the residue was poured into water, and the precipitate that formed was filtered off and crystallized from aqueous ethanol.

3,3,6-Trimethyl-1,2,3,4-tetrahydrobenzo[b]furo(thieno or selenopheno)[2,3-c]quinolines (XIIIa-c). A mixture of 0.01 mole of XIIa-c, 100 ml of ethylene glycol, and 5 g of KOH was maintained at 180°C for 4 h. After cooling, 200 ml of water was added, and the precipitate was filtered off and crystallized from aqueous ethanol.

1-Oxo-3,3,6-trimethyl-1,2,3,4-tetrahydrobenzo[b]furo(thieno or selenopheno)[2,3-c]quinoline Oximes (XIVa-c). A. To potassium tert-amylate obtained from 2 g of potassium and 50 ml of tert-amyl alcohol was added 2 g (0.03 mole) of hydroxylamine hydrochloride and a solution of 0.01 mole of VIIIa, IXa, or X in 30 ml of tetrahydrofuran. The resulting mixture was agitated for 40 h at room temperature, then poured into 200 ml of water, and neutralized with 3 ml of acetic acid. The resulting precipitate was filtered off and crystallized from methanol. **B.** A mixture of 0.01 mole of the hydrochloride of VIIa, IXa, or X and 1.38 g (0.02 mole) of hydroxylamine hydrochloride in 100 ml of ethanol was refluxed for 6 h. The ethanol was concentrated down to 50 ml, the mixture was cooled, and the precipitate that formed was filtered off.

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