Russian Journal of General Chemistry, Vol. 74, No. 5, 2004, pp. 731–737. Translated from Zhurnal Obshchei Khimii, Vol. 74, No. 5, 2004, pp. 797–803.

Tautomerism and Stereodynamics of Indophenols, Amidines, and Their Derivatives and Analogs: XVI.[1] Synthesis of Tetrahydroquinoxalines Having Spiro-Fused Oxoindole and Cyclohepta[*c*]furan Fragments

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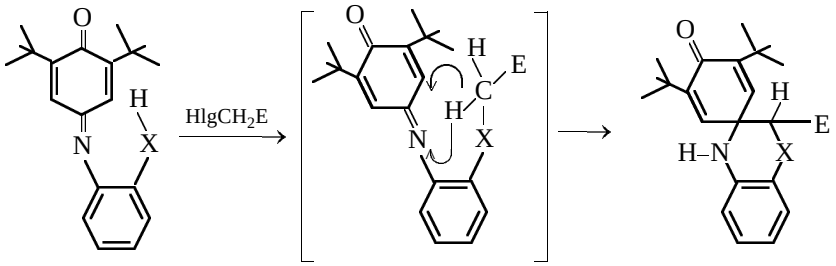
Received April 18, 2002

Abstract — Alkylation of *o*-(*N*-sulfonylamino)phenylimino derivatives of indol-2-one and cyclohepta[*c*]furan with phenacyl bromides is accompanied by cyclization to previously unknown tetrahydroquinoxalines having spiro-fused oxoindole and cyclohepta[*c*]furan fragments. The structure of the latter includes a nitrogen–oxygen triangular system typical of most natural cytotoxic compounds.

In the preceding communications we described cyclization of compounds **II** derived from *o*-indophenols [2, 3] and *o*-indoanilines **I** [4] to spiro-fused benzoxazines and quinoxalines **III**. Unlike known methods [5] for building up analogous ring systems, the proposed procedure involves closure of C–C rather than C–O or C–N bond in the final stage (Scheme 1).

Scheme 1.



I **II** **III**

X = O, NSO_2R; E = $CH{=}CH_2$, Ar, COAr.

The general character of this rearrangement was demonstrated with Schiff bases **IV** as examples; it was shown that the six-membered heteroring in **V** can be formed with participation of a carbon atom not included in the quinonimine ring [1]. It was important to elucidate whether such spiro systems could be obtained when the six-membered quinonimine fragment of *o*-indophenols or *o*-indoanilines **I** is replaced by a five- or seven-membered fragment, e.g., oxoindole (**VI**) or furotropone (**VII**) (Scheme 2).

The present communication reports on the synthesis of tetrahydroquinoxalines having spiro-fused oxoindole and furotropone fragments. The latter constitute a part of the structure of many natural compounds [6, 7] possessing various kinds of biological activity [8], including antitumor; some spiro derivatives of oxoindole were found to exhibit antileucemic activity [9].

Spirocyclic compounds **X** were synthesized by treatment with phenacyl bromides of thallium salts **VIII** instead of parent 1,5-dimethyl-3-(*o*-R-sulfonylaminophenylimino)indol-2-ones (Scheme 3). Reac-

[1] For communication XV, see [1].

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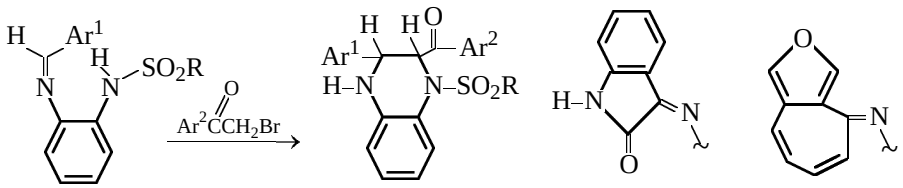
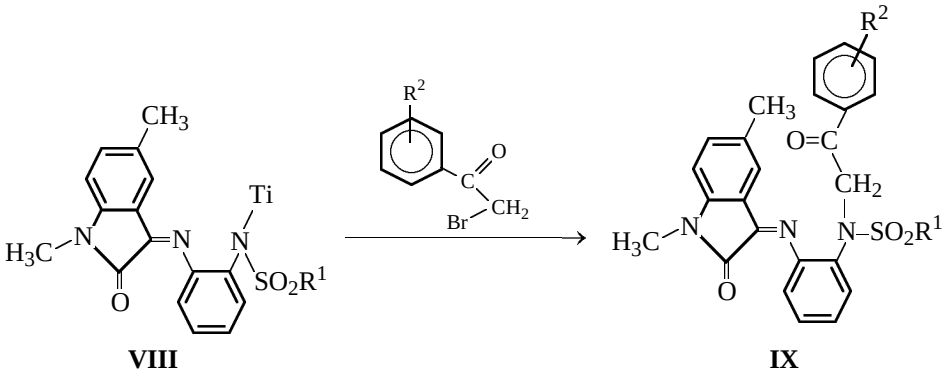
Scheme 2.

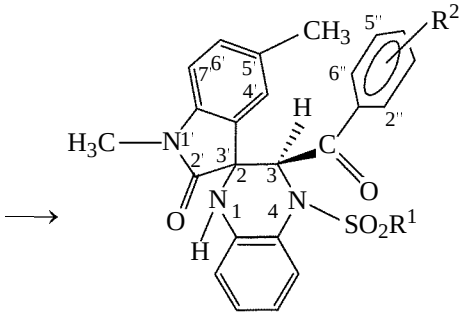


IV **V** **VI** **VII**

Scheme 3.



VIII **IX**



X

VIII, R^1 = CH_2Ph (**a**), 4-$CH_3C_6H_4$ (**b**); **IX, X**, R^1 = CH_2Ph, R^2 = 4-Br (**a**); R^1 = 4-$CH_3C_6H_4$, R^2 = 4-Br (**b**); R^1 = CH_2Ph, R^2 = 3-NO_2 (**c**); R^1 = 4-$CH_3C_6H_4$, R^2 = 4-NO_2 (**d**).

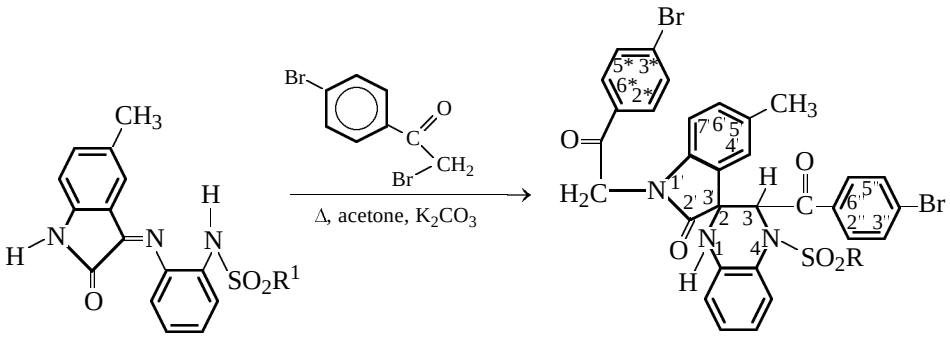
tions of the latter with phenacyl bromides on heating in acetone in the presence of K_2CO_3 [1] resulted in poor yields and strong contamination of the target products. The transformation of intermediate phenacyl derivatives **IX** into spiroquinoxalines **X** followed from the appearance in the ^1H NMR spectra of two one-proton signals from the N^1H and C^3H protons instead of one two-proton signal from the CH_2–C=O group. In addition, the spectrum of **Xa** contained an *AB* quartet from the CH_2SO_2 group, indicating the presence of a chiral center.

Scheme 4 illustrates the reaction of *p*-bromophenacyl bromide with iminoisatin **XI** which has no methyl group on the indole nitrogen atom. Even in reactions with insufficient (less than equimolar) amount of *p*-bromophenacyl bromide we isolated only spiroquinoxaline **XII** substituted at N1 by the phenacyl group.

Also, the presence of a labile NH proton in molecule **XI** hampered selective synthesis of the corresponding thallium salt at the sulfonamide nitrogen atom. In the ^1H NMR spectrum of **XII** we observed a characteristic *AB* quartet with a low $\Delta\nu/J$ ratio due to remoteness of the diastereotopic NCH_2CO group from the chiral center.

Unlike Schiff bases derived from isatin, which are readily obtained by reaction with anilines [9], we failed to synthesize in such a way furotroponimine

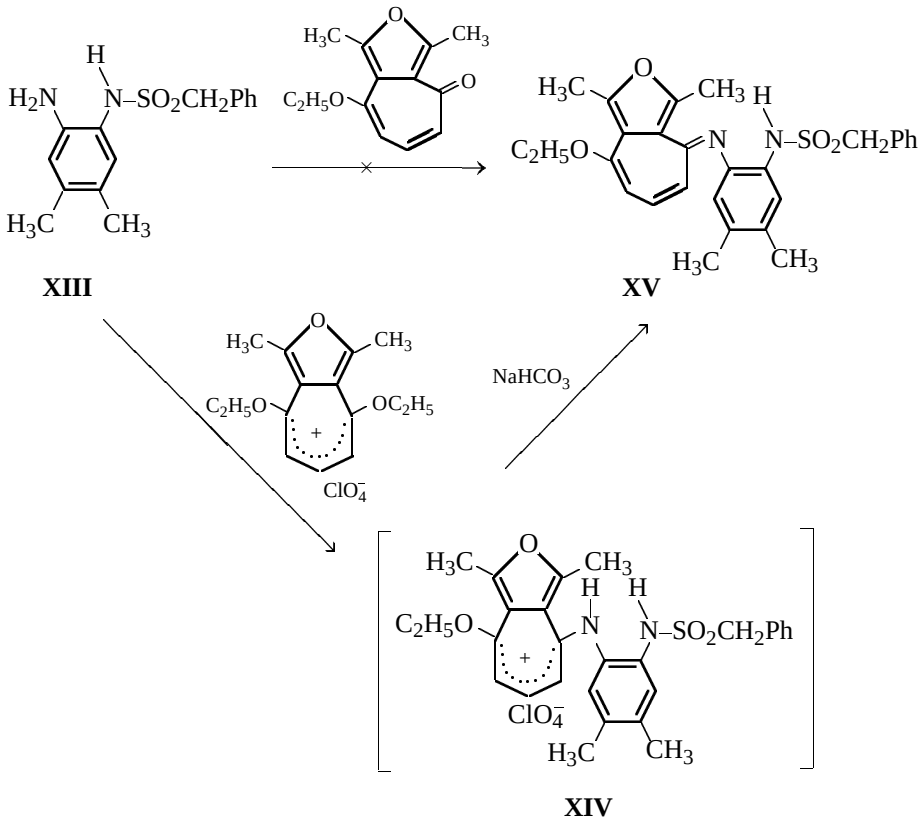
Scheme 4.



XI **XII**

XI, XII, R = 4-$CH_3C_6H_4$.

Scheme 5.



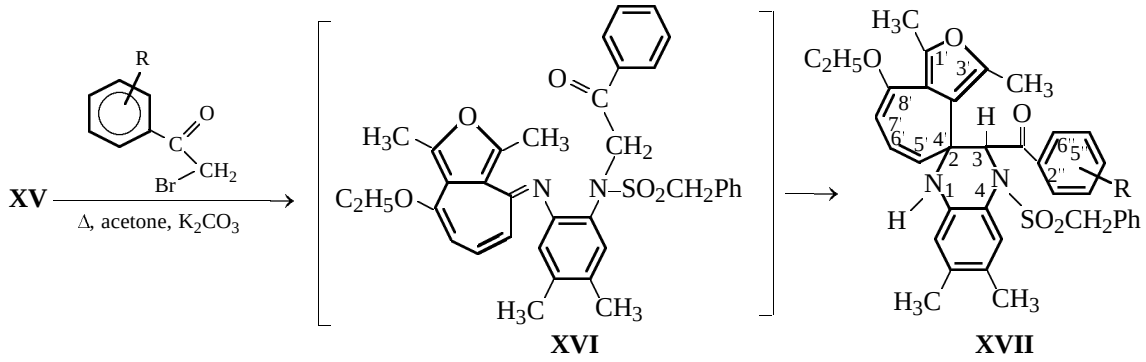
XIII **XV** **XIV**

XV even when the nucleophilicity of the amino group was enhanced via introduction of two methyl groups into the benzene ring (compound **XIII**; Scheme 5). Schiff base **XV** was obtained by nucleophilic substitution of the ethoxy group in furotropylium cation [10] and subsequent treatment of intermediate **XIV** with sodium hydrogen carbonate. In this reaction, we also used *N*-benzylsulfonyl-4,5-dimethylbenzene-1,2-diamine (**XIII**), for the transformation **XIII** → **XIV** successfully occurred only in chloroform and required high solubility of the reactants (which was achieved via introduction of methyl groups).

The reaction of furotroponimine **XV** with phenacyl bromides under the conditions described in [1] afforded spiroquinoxalines **XVIIa**–**XVIIc** (Scheme 6). As in the alkylation of Schiff bases derived from isatin, we failed to isolate even traces of open-chain isomers **XVI**.

Figure 1 shows the ^{1}H NMR spectra of tropon-

Scheme 6.



R = 4-Br (**a**), 3-NO_2 (**b**), 4-NO_2 (**c**).

imine **XV** and spiran **XVIIa**. The cyclic structure of the latter is confirmed by the presence of singlets from the CH (δ 6.40 ppm) and NH protons (δ 5.68 ppm) and an *AB* quartet (δ 4.55 ppm) from the CH_2SO_2 group.

Previous studies of the structure of about two hundred natural cytotoxic compounds [11] revealed that most of these contains a "pharmacophoric triangle" with its sides of 3.1±0.2, 6.4±0.3, and 7.5±0.4 Å, whose vertices are occupied by oxygen or nitrogen

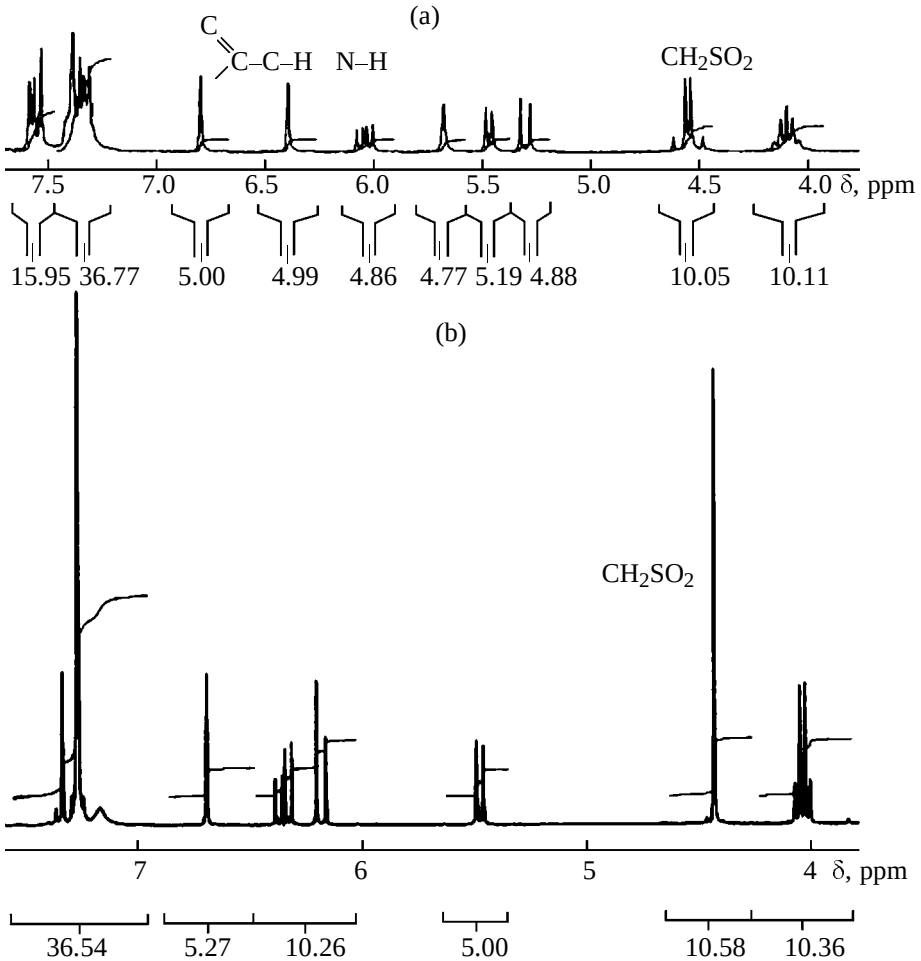


Fig. 1. ^{1}H NMR spectra of compounds (a) **XVIIa** (Bruker DPX-250, 250 MHz) and (b) **XV** (Varian Unity-300, 300 MHz) in acetone-d_6.

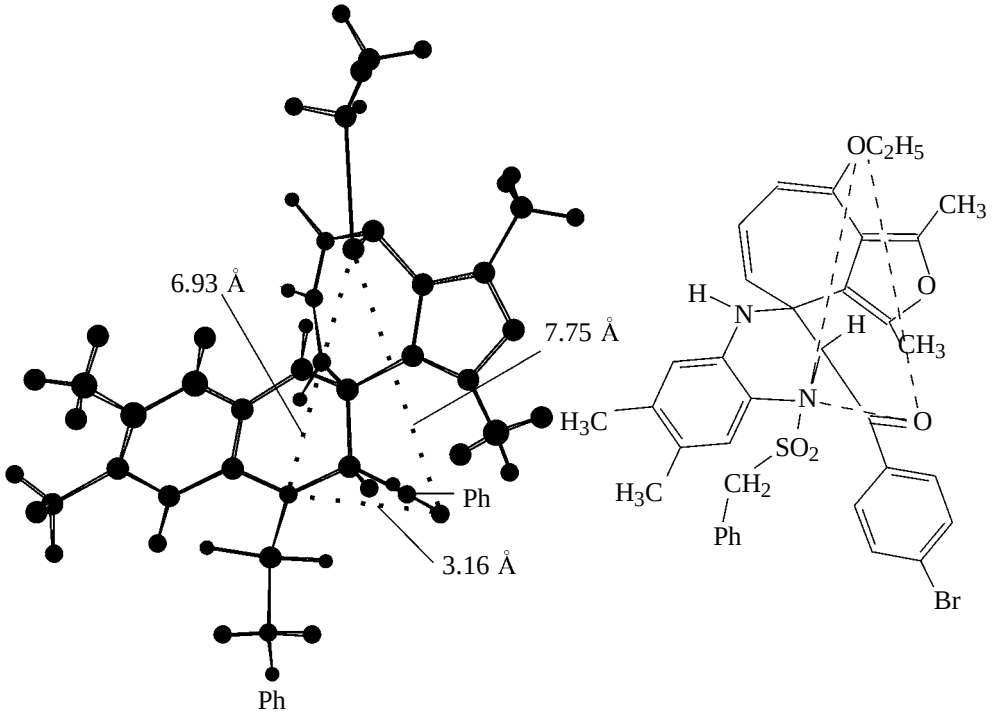


Fig. 2. Structure of compound **XVIIa** according to the MM2 and PM3 calculations.

atoms in any combination. The structure of compound **XVIIa** according to the MM2 and PM3 calculations is shown in Fig. 2. The carbonyl and ether oxygen atoms and the sulfonamide nitrogen atoms give rise to a triangle with its sides equal to 3.16, 6.93, and 7.75 Å.

Thus, the proposed procedure for building up quinoxaline ring opens new prospects in the synthesis of a large number of spiro structures which are difficult or impossible to obtain by other methods and are potential pharmacologically active compounds.

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on Bruker DPX-250 (250 MHz) and Varian Unity-300 (300 MHz) spectrometers at 25°C. The elemental compositions of the synthesized compounds were in agreement with the calculated values.

***N*-[2-(1,5-Dimethyl-2-oxo-1,2-dihydroindol-3-ylideneamino)phenyl]-*p*-toluenesulfonamide (VIa).** A mixture of 1.9 mmol of *N*-(2-aminophenyl)-*p*-toluenesulfonamide [1], 1.9 mmol of 1,5-dimethyl-1*H*-indole-2,3-dione [12], and 0.002 g of *p*-toluenesulfonic acid in 10 ml of toluene was heated for 2 h under reflux. The mixture was evaporated, and the dry residue was recrystallized from methanol. Yield 85%. Orange crystals, mp 115°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.02 s (3H, CH_3), 2.07 s (3H, CH_3), 3.25 s (3H, NCH_3), 6.43 s (1H, NH), 6.71 d (1H, 6-H, *J* = 7.9 Hz), 6.85 d (2H, 3''-H, 5''-H, *J* = 7.9 Hz), 7.04–7.27 m (5H, 4-H, 5-H, 4'-H, 6'-H, 7'-H), 7.54 d (2H, 2''-H, 6''-H, *J* = 7.8 Hz), 7.70 d (1H, 3-H, *J* = 7.4 Hz).

***N*-[2-(1,5-Dimethyl-2-oxo-1,2-dihydroindol-3-yideneamino)phenyl]phenylmethanesulfonamide (VIb)** was synthesized in a similar way. Yield 87%. Red crystals, mp 125°C.

***N*-[2-(5-Methyl-2-oxo-1,2-dihydroindol-3-ylideneamino)phenyl]-*p*-toluenesulfonamide (XI)** was synthesized in a similar way. Yield 65%. Yellow–orange crystals, mp 160°C.

***N*-[2-(1,5-Dimethyl-2-oxo-1,2-dihydroindol-3-ylideneamino)phenyl]-*p*-toluenesulfonamide thallium salt (VIIIa).** A solution of 0.7 mmol of KOH in 1 ml of MeOH and a solution of 0.7 mmol of thallium acetate in 5 ml of MeOH were added to a solution of 0.7 mmol of *N*-[2-(1,5-dimethyl-2-oxo-1,2-dihydroindol-3-ylideneamino)phenyl]-*p*-toluenesulfonamide (**VIa**) in 5 ml of MeOH. The mixture was carefully heated to the boiling point and cooled, and the precipitate was filtered off and washed with hexane. Yield 70%. Violet–black crystals, mp 240°C.

***N*-[2-(1,5-Dimethyl-2-oxo-1,2-dihydroindol-3-ylideneamino)phenyl]phenylmethanesulfonamide**

thallium salt (VIIIb) was synthesized in a similar way. Yield 78%. Violet–black crystals, mp 255°C.

***N*-[2-(5-Methyl-2-oxo-1,2-dihydroindol-3-ylideneamino)phenyl]-*p*-toluenesulfonamide thallium salt (VIIIc)** was synthesized in a similar way. Yield 70%. Violet–black crystals, mp 230°C.

Quinoxalines Xa–Xd *(general procedure).* A mixture of 0.3 mmol of thallium salt **VIIIa–VIIIc** and 0.3 mmol of the corresponding phenacyl bromide in 5 ml of acetonitrile was refluxed for 15 min. The mixture was filtered, the filtrate was evaporated, and the dry residue was recrystallized from isobutyl alcohol.

4-Benzylsulfonyl-3-*p*-bromobenzoyl-1',5'-dimethyl-2'-oxo-1,2,2',3,3',4-hexahydroquinoxaline-2-spiro-3'-indole (Xa). Yield 50%. Orange–pink crystals, mp 180°C. ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 2.21 s (3H, CH_3), 3.01 s (3H, NCH_3), 4.60 d (1H, CH, J = 14.1 Hz), 4.70 d (1H, CH, J = 14.1 Hz), 5.68 s (1H, CH), 6.84 d (1H, 8-H, J = 7.9 Hz), 6.88–6.94 m (2H, 7-H, 4'-H), 6.99 s (1H, NH), 7.05 d.d (1H, 6-H), 7.12–7.23 m (5H, 2'''-H–6'''-H), 7.30 d (2H, 6'-H, 7'-H, J = 6.6 Hz), 7.49 d (2H, 2''-H, 6''-H, J = 8.8 Hz), 7.57 d (2H, 3''-H, 5''-H, J = 8.7 Hz), 7.75 d (1H, 5'-H, J = 8.2 Hz).

1',5'-Dimethyl-3-*p*-nitrobenzoyl-2'-oxo-4-*p*-tolylsulfonyl-1,2,2',3,3'4-hexahydroquinoxaline-2-spiro-3'-indole (Xb). Yield 65%. Light yellow crystals, mp 230°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.26 s (3H, CH_3), 2.34 s (3H, CH_3), 3.06 s (3H, NCH_3), 4.48 s (1H, NH), 6.02 s (1H, CH), 6.77–6.87 m (3H, 6-H, 7-H, 8-H), 7.00 m (2H, 6'-H, 7'-H), 7.15 m (3H, 4'-H, 3'''-H, 5'''-H), 7.33 d (1H, 5-H, J = 8.1 Hz), 7.53 m (4H, 2''-H, 6''-H, 2'''-H, 6'''-H), 7.69 d (2H, 3''-H, 5''-H, J = 8.4 Hz).

4-Benzylsulfonyl-1',5'-dimethyl-3-*m*-nitrobenzoyl-2'-oxo-1,2,2',3,3',4-hexahydroquinoxaline-2-spiro-3'-indole (Xc). Yield 55%. Yellow crystals, mp 170°C. ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 2.21 s (3H, CH_3), 2.98 s (3H, NCH_3), 4.47 s (2H, CH_2), 5.70 s (1H, CH), 5.78 s (1H, NH), 6.81 d (1H, 8-H, J = 7.9 Hz), 6.92 m (3H, 6-H, 7-H, 4'-H), 7.04–7.19 m (5H, 2'''-H–6'''-H), 7.30 d (2H, 6'-H, 7'-H, J = 7.4 Hz), 7.67 d.d (1H, 5''-H), 7.80 d (1H, 5-H, J = 8.0 Hz), 7.93 d (1H, 6''-H, J = 7.7 Hz), 8.27 s (1H, 2''-H), 8.34 d (1H, 4''-H, J = 6.1 Hz).

1',5'-Dimethyl-3'-*p*-nitrobenzoyl-2'-oxo-4-*p*-tolylsulfonyl-1,2,2',3,3',4-hexahydroquinoxaline-2-spiro-3'indole (Xd). Yield 50%. Dark orange crystals, mp 135°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 2.21 s (3H, CH_3), 2.35 s (3H, CH_3), 3.05 s (3H, NCH_3), 4.38 s (1H, NH), 6.01 s (1H, CH), 6.79 m (2H, 5-H, 6'-H), 6.89 m (2H, 6-H, 7'-H), 7.00 d.d (1H, 7-H), 7.20 m (3H, 4'-H, 3'''-H, 5'''-H), 7.38 d (1H, 8-H), 7.53 d (2H, 2'''-H, 6'''-H), 7.99 d (2H, 2''-H, 6''-H), 8.24 d (2H, 3''-H, 5''-H).

3-*p*-Bromobenzoyl-1'-*p*-bromophenacyl-5'-methyl-2'-oxo-4-*p*-tolylsulfonyl-1,2,2',3,3',4-hexahydroquinoxaline-2-spiro-3'-indole (XII). Freshly calcined potassium carbonate, 2 g, and *p*-bromophenacyl bromide, 0.27 g, were added to a solution of 0.2 g of *N*-[2-(5-methyl-2-oxo-1,2-dihydroindol-3-ylideneamino)phenyl]-*p*-toluenesulfonamide in 10 ml of acetone. The mixture was heated for 30 min under reflux and filtered, the filtrate was evaporated, and the dry residue was recrystallized from isopentyl alcohol. Yield 0.28 g (70%). Gray–yellow crystals, mp 250°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 2.11 s (3H, CH_3), 2.34 s (3H, CH_3), 5.28 br.s (2H, CH_2), 6.06 s (1H, CH), 6.63–6.78 m (3H, 7-H, 8-H, NH), 6.89–7.00 m (2H, 5-H, 6-H), 7.10 m (2H, 6'-H, 7'-H), 7.31 m (3H, 4'-H, 2*-H, 6*-H), 7.54 d (2H, 3*-H, 5*-H, J = 8.2 Hz), 7.63 d (2H, 2''-H, 6''-H, J = 8.3 Hz), 7.75–7.84 m (4H, 3''-H, 5''-H, 3'''-H, 5'''-H), 8.03 d (2H, 2'''-H, 6'''-H, J = 8.2 Hz).

***N*-(2-Amino-4,5-dimethylphenyl)phenylmethanesulfonamide (XIII).** Phenylmethanesulfonyl chloride, 2.5 g, was added to a solution of 1.79 g of 4,5-dimethyl-1,2-phenylenediamine and 1.9 ml of pyridine in 20 ml of THF. The mixture was stirred for 2 h at room temperature, 100 ml of water was added, the mixture was heated to the boiling point and cooled, and the precipitate was filtered off and recrystallized from methanol. Yield 1.64 g (50%). Beige fine crystals, mp 158–160°C.

4-(2-Benzylsulfonylamino-4,5-dimethylphenylimino)-8-ethoxy-1,3-dimethyl-4*H*-cyclohepta[*c*]-furan (XV). A solution of 0.33 g of 1,3-dimethyl-4,8-cyclohepta[*c*]furylium perchlorate [10] and 0.27 g of *N*-(2-amino-4,5-dimethylphenyl)phenylmethanesulfonamide in 10 ml of dry chloroform was heated for 1.5 h under reflux. The mixture was cooled and poured into 100 ml of a 5% solution of NaHCO3, and (after stirring) the organic phase was separated. The solvent was removed, and the residue was recrystallized from methanol to obtain orange crystals of 4-(2-benzylsulfonylamino-4,5-dimethylphenylimino)-8-ethoxy-1,3-dimethyl-4*H*-cyclohepta[*c*]furan. Yield 0.28 g (60%), mp 170–173°C. ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 1.45 d.d (3H, CH_3), 2.25 t (6H, $2CH_3$), 2.40 s (3H, CH_3), 2.59 s (3H, CH_3), 4.05 q (2H, OCH_2, J = 6.7 Hz), 4.45 s (2H, CH_2), 5.48 d (1H, 6'-H), 6.09 d (1H, 4'-H), 6.35 d.d (1H, 5'-H, $J_{4',5'}$ = 12.6 Hz, $J_{5',6'}$ = 9.3 Hz), 6.70 s (1H, 6-H), 7.45–7.10 m (7H, 3-H, Ph, NH).

Quinoxalines XVIIa–XVIIc *(general procedure).* Freshly calcined potassium carbonate, 2 g, and the corresponding phenacyl bromide, 0.41 mmol, were added to a solution of 0.41 mmol of compound **XV** in 10 ml of acetone. The mixture was heated for 15 min under reflux, cooled, and filtered, the filtrate was evaporated, and the residue was recrystallized from isopentyl alcohol.

4-Benzylsulfonyl-3-*p*-bromobenzoyl-8'-ethoxy-1',3',6,7-tetramethyl-1,2,3,4-tetrahydro-4'*H*-quinoxaline-2-spiro-4'-cyclohepta[*c*]furan (XVIIa). Yield 65%. Yellow–gray crystals, mp 175°C. ^{1}H NMR spectrum (acetone-d_6), δ, ppm: 1.54 d.d (3H, CH_3), 2.08 s (3H, CH_3), 2.21 s (3H, CH_3), 2.23 s (3H, CH_3), 2.25 s (3H, CH_3), 4.11 q (2H, OCH_2), 4.50 d (1H, CH, J = 13.8 Hz), 4.60 d (1H, CH, J = 13.8 Hz), 5.30 d (1H, 7'-H, J = 11.3 Hz), 5.46 d (1H, 5'-H, J = 7.2 Hz), 5.69 s (1H, NH), 6.04 d.d (1H, 6'-H, J = 7.2 Hz), 6.40 s (1H, CH), 6.80 s (1H, 8-H), 7.30–7.43 m (7H, 2'''-H–6'''-H, 2''-H, 6''-H), 7.54–7.59 m (3H, 5-H, 3''-H, 5''-H).

4-Benzylsulfonyl-8'-ethoxy-1',3',6,7-tetramethyl-3-*p*-nitrobenzoyl-1,2,3,4-tetrahydro-4'*H*-quinoxaline-2-spiro-4'-cyclohepta[*c*]furan (XVIIb). Yield 60%. Dark orange crystals, mp 150°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.46 m (3H, CH_3), 1.92 s (3H, CH_3), 2.18 s (3H, CH_3), 2.20 s (3H, CH_3), 2.23 s (3H, CH_3), 4.01 m (2H, OCH_2), 4.10 s (1H, NH), 4.45 m (2H, CH_2), 5.30 m (2H, 7'-H, 5'-H), 5.97 d.d (1H, 6'-H), 6.29 s (1H, CH), 6.61 s (1H, 8-H), 7.73–7.45 m (8H, 5-H, 2'''-H–6'''-H, 2''-H, 6''-H), 8.04 d (2H, 3''-H, 5''-H).

4-Benzylsulfonyl-8'-ethoxy-1',3',6,7-tetramethyl-3-*m*-nitrobenzoyl-1,2,3,4-tetrahydro-4'*H*-quinoxaline-2-spiro-4'-cyclohepta[*c*]furan (XVIIc). Yield 60%. Dark yellow crystals, mp 160°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 1.35 m (3H, CH_3), 1.90 s (3H, CH_3), 2.18 s (3H, CH_3), 2.20 s (3H, CH_3), 2.24 s (3H, CH_3), 3.96 m (2H, OCH2), 4.09 s (1H, NH), 4.41 d (1H, CH, J = 13.9 Hz), 4.50 d (1H, CH, J = 13.9 Hz), 5.23 d (1H, 7'-H, J = 11.3 Hz), 5.29 d (1H, 5'-H, J = 7.4 Hz), 5.99 d.d (1H, 6'-H, J = 7.2 Hz), 6.23 s (1H, CH), 6.61 s (1H, 8-H), 6.95– 7.39 m (7H, 2'''-H–6'''-H, 5-H, 5''-H), 7.54 d (1H, 6''-H, J = 7.6 Hz), 8.06 s (1H, 2''-H), 8.20 d (1H, 4''-H, J = 7.7 Hz).

ACKNOWLEDGMENTS

This study was financially supported by the Russian Foundation for Basic Research (project nos. 01-03-32549 and 99-03-33496).

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