CYCLIZATION OF N-ALKYLAZINIUM CATIONS WITH BIFUNCTIONAL NUCLEOPHILES.

29.* SYNTHESIS OF A NOVEL 1,3,4-THIADIAZOLO-[2,3-*a*]QUINOXALINO[2,3-d]PYRROLE HETEROCYCLIC SYSTEM

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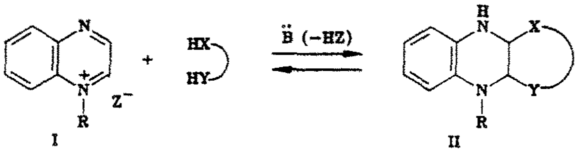
1,4,4a,5,10,10a-Hexahydro-1,3,4-thiadiazino[5,6-b]quinoxalines react with acetylacetone and acetoacetate esters upon heating in ethanol to give derivatives of a novel heterocyclic system, hexahydro-1,3,4-thiadiazolo[2,3-a]quinoxalino[2,3-d]pyrrole.

Diaddition reactions of bifunctional nucleophiles to 1,4-diazinium cations I, which lead to the formation of cyclic adducts II, appear to be reversible processes. Their equilibria can be shifted to the right, as expected, in the presence of any type of base (such as di- and triethylamine, sodium ethoxide, etc.), and the resulting hydrogenated condensed 1,4-diazine derivatives can then be isolated in high yields [2, 3].



I

II

Compounds of type II are relatively stable in the crystalline state, but addition of equimolar amounts of mineral acids to their solutions results in complete dissociation of the cyclic adducts II to the 1,4-diazinium cation precursors and the corresponding dinucleophiles [2, 3]. In the presence of catalytic amounts of acids, or upon heating in protic solvents, the cyclization products dissociate to a small degree, which provides a basis for different types of isomerization reactions or other reactions, leading to changes in the regio- and stereoorientation of the annelated ring, and even to changes in the actual nature of the annelated ring [2, 3]. The direction or pathway of the interconversion reactions of condensed tetrahydropyrazines upon treatment with catalytic amounts of acids is determined by the relative bond strengths in the corresponding hydrogenated heterocycle systems. Thus, according to mass spectrometric data [4], annelation of N,S-bifunctional reagents to 1,4-diazines results in the formation of less stable rings than reactions with C,N-, N,N-, and other nucleophiles. At the same time, thiazolo[4,6-b]- and thiazino[5,6-b]annelated tetrahydroquinoxalines are particularly susceptible to acid-catalyzed regioisomerization and other types of reactions [5, 6].

In the present paper we have investigated the reactions of 1,3,4-thiadiazino[5,6-b]quinoxaline III upon treatment with CH-acids such as acetylacetone and acetoacetate esters IVa–c. Considering the possibility of dissociation of thiadiazinoquinoxaline III to quinoxalinium cation V and thiobenzhydrazide VI, we would expect the most likely reaction products to include furo[2,3-b]quinoxaline derivatives VII, from cation V reacting with dicarbonyl compounds IV [7], or pyridazino[3,4-b]quinoxalines VIII, in the case that hydrazone IX, formed from thiobenzhydrazide VI and β-dicarbonyl compounds, can react as a 1,4-N,C-dinucleophile in a subsequent cyclization reaction [8].

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*For Communication 28, see [1].

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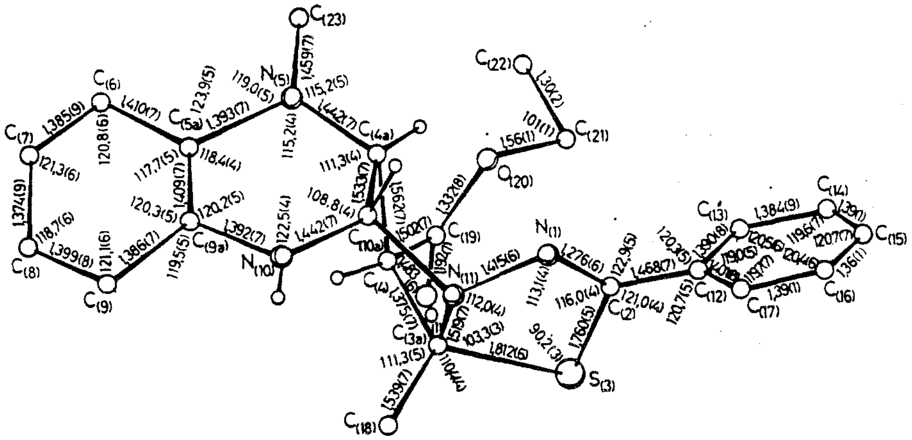


Fig. 1. Molecular structure of the ethyl ester of acid XIc.

Our experiments revealed that reactions of thiadiazino[5,6-b]quinoxaline III with β-dicarbonyl compounds proceed irreversibly in ethanol at 60°C, resulting in the formation of colorless crystalline substances XIa–c, with melting points higher than the tetrahydroquinoxaline starting material III. Mass spectrometric data and the results of elemental analysis for compound XIb, which was isolated from the reaction with methyl acetoacetate ester IVb, indicated that its empirical composition corresponded to that of an adduct of thiadiazinoquinoxaline III with one molecule of dicarbonyl compound. Adducts having the same general composition are also formed upon reaction of thiadiazinoquinoxaline III with acetylacetone and ethyl acetoacetate (see Experimental).

The PMR spectra of compounds XIa–c exhibit signals for all of the same fragments as the starting materials, although there are significant changes in the chemical shift values of the signals, their multiplicity, and coupling constants (SSCC) for the tetrahydropyrazine ring protons, relative to the analogous parameters in thiadiazinoquinoxaline III; these changes suggest extensive changes in the molecular skeleton. At the same time, however, these changes do not make it possible to establish the nature of the combination or annelation of individual fragments. It is also impossible to establish the structures of these compounds according to their ^{13}C-NMR spectral data, judging from compound XIa; the structures of compound XIa-c were therefore solved unequivocally by carrying out an x-ray structural analysis on XIc.

The x-ray structural analytical data demonstrated that compounds XIa-c correspond in structure to a hexahydro-1,3,4-thiadiazolo[2,3-*a*]quinoxalino-[2,3-d]pyrrole derivative (see Fig. 1). The molecular structure of XIc consists of five cyclic ring systems, four of which are bound together with one another (benzene, pyrazine, pyrrolidine, and thiadiazole rings). A phenyl substituent $C_{(12)}$–$C_{(17)}$ is connected via a C–C bond to the $C_{(2)}$ atom in the thiadiazole ring. The tetrahydroquinoxaline system, comprised of a benzene ring condensed with a pyrazine ring, is almost planar (only atom $C_{(4a)}$ deviates greatly from the average plane of the remaining atoms). cis-Annelation of the pyrrolidine ring with the pyrazine ring relative to the $C_{(4a)}$–$C_{(10a)}$ bond (the $H_{(10a)}C_{(10a)}C_{(4a)}H_{4a)}$ torsional angle is 45.2°) gives it an envelope conformation (atom $C_{(4a)}$ deviates from the plane of the other atoms by 0.54 Å). The five-membered thiadiazole ring annelated with the former via an $N_{(11a)}$–$C_{(3a)}$ bond exists in a gauche conformation (the $N_{(11a)}$ and $C_{(3a)}$ atoms are oriented in different directions from the plane passing through the three remaining atoms in the thiadiazole ring: –0.10 and 0.29 Å, respectively). The angle of rotation of the phenyl group around the $C_{(2)}$–$C_{(12)}$ bond is equal to 21.7°. The presence of methyl and hydroxyethyl substituents in the pyrrolidine ring and of the two cyclic systems annelated with it raises the possibility that compounds XIa–c can exist in the form of several configurational isomers. For example, if in the pyrrolidine ring bond $C_{(4a)}$–$N_{(5)}$ occupies a pseudoequatorial position, and bond $C_{(10a)}$–$N_{(10)}$ a pseudoaxial position, then a conformer is possible with these bonds oriented in different or opposite directions.

The principal geometric parameters in the molecular structure of XI (bond lengths and bond angles) are similar to standard values [9], with the exception of the parameters for the hydroxyethyl group (significant elongation of the $C_{(20)}$–$C_{(21)}$ bond and contraction of the $C_{(21)}$–$C_{(22)}$ bond). The $C_{(19)}O_{(20)}C_{(21)}$ and $O_{(20)}C_{(21)}C_{(22)}$ bond angles also differ substantially from normal tetrahedral values; this is probably due to their disorder, i.e., to the possibility of rotation about the $O_{(20)}$–$C_{(21)}$ single bond. The difference in the $S_{(3)}$–$C_{(3a)}$ [1.812(6) Å] and $S_{(3)}$–$C_{(2)}$ [1.760(5) Å] bond lengths is consistent with different atomic radia for $C_{(3a)}$ (sp^3) and $C_{(2)}$ (sp^2). The length of the N–C double bond also depends on the hybridization state of the carbon atom. For example, the $C_{(9a)}$–$N_{(10)}$ (sp^2 hybridized carbon atom) bond length is equal to 1.392(7) Å, which is quite a bit different from the value of the $N_{(10)}$–$C_{(10a)}$ (sp^3 hybridized carbon) atom bond length of 1.442(7) Å. The $N_{(11)}$ atom, in contrast to all

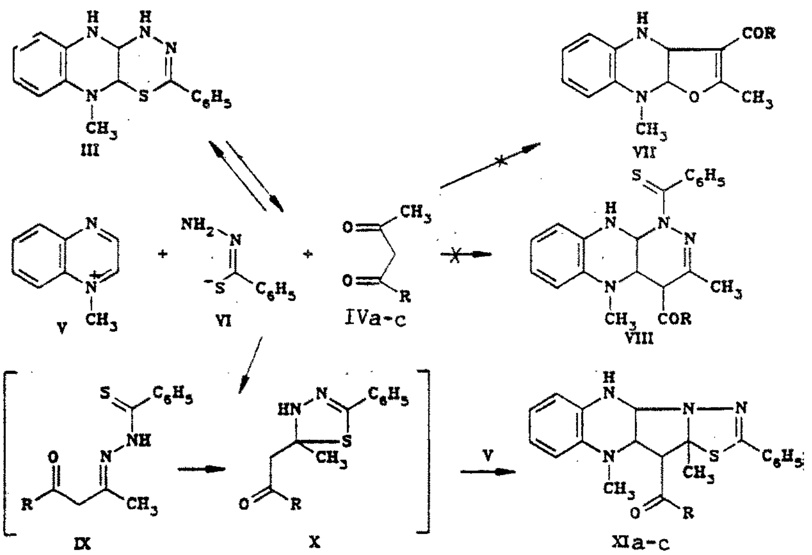
TABLE 1. Coordinates for the Nonhydrogen (× 10^4) and Hydrogen Atoms (× 10^3)

Atom	x	y	z	Atom	x	y	z
$N_{(1)}$	3671(1)	1274(4)	−374(5)	$C_{(18)}$	−606(9)	2305(6)	1033(6)
$N_{(5)}$	4922(7)	1893(4)	3247(4)	$C_{(19)}$	4106(8)	4035(4)	2800(4)
$N_{(10)}$	1384(7)	588(4)	1585(4)	$C_{(21)}$	7246(17)	5235(8)	2950(15)
$N_{(11)}$	2191(8)	1267(4)	302(5)	$C_{(22)}$	8494(24)	5516(11)	3946(20)
$S_{(3)}$	2361(3)	3096(1)	206(1)	$C_{(23)}$	6678(6)	2696(3)	4131(4)
$O_{(1)}$	3309(8)	4811(4)	3254(5)	$H_{(4)}$	190	287	284
$O_{(20)}$	6040(8)	4101(4)	2664(6)	$H_{(4a)}$	581	221	197
$C_{(2)}$	3849(11)	2113(6)	−521(7)	$H_{(5)}$	376	23	70
$C_{(3a)}$	1697(8)	2401(4)	1021(4)	$H_{(6)}$	486	226	542
$C_{(4)}$	3028(10)	2870(5)	2309(5)	$H_{(7)}$	193	161	614
$C_{(4a)}$	4378(9)	1996(5)	2198(5)	$H_{(8)}$	−153	54	460
$C_{(5a)}$	3278(9)	1567(4)	3632(5)	$H_{(9)}$	−158	4	235
$C_{(6)}$	3321(10)	1828(5)	3822(5)	$H_{(10)}$	32	−4	102
$C_{(7)}$	1649(13)	1447(5)	5137(6)	$H_{(13)}$	517	402	−46
$C_{(8)}$	−108(11)	817(5)	4307(6)	$H_{(14)}$	535	57	−218
$C_{(9)}$	−182(10)	555(5)	3123(5)	$H_{(15)}$	744	62	−352
$C_{(9a)}$	1491(8)	898(4)	2779(4)	$H_{(16)}$	874	271	−316
$C_{(10a)}$	3051(8)	932(4)	1167(4)	$H_{(17)}$	776	419	−176
$C_{(12)}$	5866(8)	3303(4)	−1108(8)	$H_{(18-1)}$	−109	299	141
$C_{(13)}$	5167(10)	2256(5)	−1271(5)	$H_{(18-2)}$	−154	197	22
$C_{(14)}$	5766(12)	1353(6)	−2145(7)	$H_{(18-3)}$	−95	185	160
$C_{(15)}$	7065(11)	1487(7)	−2827(7)	$H_{(23-1)}$	714	249	472
$C_{(16)}$	7740(11)	2529(7)	−2652(7)	$H_{(23-2)}$	619	331	469
$C_{(17)}$	7132(13)	3419(7)	−1822(8)	$H_{(23-3)}$	782	280	376

of the other nitrogen atoms in the molecular structure of XIc, exists in a pyramidal configuration, namely, the bond angles around $N_{(11)}$ are close to tetrahedral.

In the crystalline state molecules of XI, due to the presence of an unshared pair of electrons on $N_{(11)}$ and a hydrogen atom at $N_{(10)}$, are engaged in intermolecular hydrogen-bond formation of the type $N_{(10)}–H_{(10)}$ ... $N_{(11)}$, with bond lengths equal to 3.13 Å; the existence of this type of hydrogen bonding leading in turn to the formation of centrosymmetric dimers.

In accord with the established structures for these reaction products, the reaction of thiadiazinoquinoxaline III with β-dicarbonyl compounds IVa-c can be represented by means of the following scheme:



IV, VII—XI a R=CH_3, b R=OCH_3, c R=OC_2H_5

The reaction process is initiated by dissociation of the cycloadduct III starting material to give quinoxalinium ion V and thiobenzhydrazide VI; the latter in turn reacts with a β-dicarbonyl compound to form hydrazone IX, which under the reaction conditions undergoes intramolecular cyclization to the 1,3,4-thiadiazole X [10]. Compound X then reacts further as a 1,3-

TABLE 2. Bond Angles in the Pyrrolidine Ring $C_{(10a)}C_{(4a)}C_{(4)}C_{(3a)}N_{(11)}$

Angle	ω°	Angle	ω°
$C_{(10a)}C_{(4a)}C_{(4)}$	102,5(4)	$O_{(20)}C_{(19)}O_{(1)}$	123,9(6)
$C_{(4a)}C_{(4)}C_{(3a)}$	104,9(4)	$N_{(11)}C_{(3a)}C_{(18)}$	110,0(5)
$C_{(4)}C_{(3a)}N_{(11)}$	104,9(4)	$C_{(10a)}N_{(11)}N_{(1)}$	106,9(4)
$C_{(3a)}N_{(11)}C_{(10a)}$	106,9(4)	$N_{(11)}C_{(10a)}H_{(10a)}$	108
$N_{(11)}C_{(10a)}C_{(4a)}$	103,9(4)	$C_{(19)}C_{(4)}H_{(4)}$	108
$S_{(3)}C_{(3a)}C_{(4)}$	116,3(4)	$C_{(4a)}C_{(4)}H_{(4)}$	110
$N_{(10)}C_{(10a)}N_{(11)}$	108,8(4)	$C_{(3a)}C_{(4)}H_{(4)}$	104
$N_{(5)}C_{(4a)}C_{(4)}$	116,0(4)	$N_{(5)}C_{(4a)}H_{(4a)}$	107
$C_{(3a)}C_{(4)}C_{(19)}$	111,7(5)	$C_{(10a)}C_{(4a)}H_{(4a)}$	108
$C_{(4a)}C_{(4)}C_{(19)}$	117,6(5)	$C_{(4)}C_{(4a)}H_{(4a)}$	108
$C_{(4)}C_{(19)}O_{(1)}$	123,5(6)	$N_{(10)}C_{(10a)}H_{(10a)}$	108
$C_{(4)}C_{(19)}O_{(20)}$	112,5(5)	$C_{(4a)}C_{(10a)}H_{(10a)}$	119

TABLE 3. Coefficients for the Plane Equations of the Form Ax + By + Cz - D = 0, for Various Planar Molecular Fragments

Plane	*A*	*B*	*C*	*D*
1	5,948	4,587	−6,396	1,572
2	3,997	0,564	7,274	1,270
3	4,689	−4,341	7,865	0,439
4	−3,297	12,660	−2,809	−0,137

*The following dihedral angles are formed: pl. 1 and pl. 2 ∠65.5°, pl. 2 and pl. 3 ∠21.7°, pl. 2 and pl. 4 ∠94.7°, pl. 1 and pl. 4 ∠96.2°.

C,N-nucleophile and as a result of cyclization with quinoxalinium cation V is converted to a hexahydro-1,3,4-thiadiazolo[2,3-*a*]quinoxalino[2,3-d]pyrrole XIa–c.

The 4-H, 4*a*-H, and 10*a*-H signals in the PMR spectra of compounds XIa–c are readily identified based on their multiplicity patterns. Proton 10*a*-H exhibits spin-spin coupling with $NH_{(10H)}$ and appears as broadened doublet in the PMR spectra, while its chemical shift values are consistent with the fact that atom $C_{(10a)}$ is attached to two nitrogen atoms. The chemical shifts for 4*a*-H (4.2–4.4 ppm), as well as the presence of two vicinal $^3J_{4a10a}$ and $^3J_{44a}$ coupling constants, point unequivocally to a bond between atom $C_{(4a)}$ and the CH-fragment in the carbonyl compound (see Experimental). The values of the $^3J_{4a10a}$ vicinal coupling constants, on the other hand, suggest cis-annealation of the pyrrole ring with the tetrahydropyrazine ring.

In conclusion, we have succeeded, based on these studies, in synthesizing derivatives of a novel heterocyclic system which has been identified according to x-ray structural analysis as 1,3,4-thiadiazolo[2,3-*a*]quinoxalino[2,3-d]pyrrole. This novel heterocyclic system is obtained due to the propensity of thiadiazinoquinoxalines to dissociate upon treatment with CH acids; this facility may be exploited further in the preparation of derivatives of other cyclic systems as well.

EXPERIMENTAL

PMR spectra (in DMSO-D_6) were recorded in a Perkin–Elmer R12B (60 MHz) spectrometer versus TMS as internal standard. ^{13}C NMR spectra (in DMSO-D_6) were recorded on a Bruker WP80 (20.13 MHz) spectrometer. ^{13}C Chemical shifts were measured relative to solvent signal (39.6 ppm).

Mass spectra were obtained on a Varian MAT-311A spectrometer at an accelerating voltage of 3 kV, cathode emission current of 1 mA, and an ionizing electron energy of 70 eV; direct sample injection into the spectrometer was used.

X-ray structural analysis was performed on a Syntex P1 diffractometer. Crystals of XIc, obtained via recrystallization from acetone in the form of colorless plates, are triclinic, at 20°C: $a = 6.707(2)$, $b = 14.035(3)$, and $c = 13.016(2)$ Å, $\alpha = 118.04(2)$, $\beta = 97.72(2)$, $\gamma = 95.80(2)°$, $V = 1052.6$ Å^3, $Z = 2$, P1 space group. The structure was solved by direct methods

TABLE 4. Atomic Deviations from Planes 1–4

Atom	Deviation Å	Atom	Deviation Å	Atom	Deviation Å	Atom	Deviation Å
Plane 1		Plane 2		Plane 3		Plane 4	
$C_{(3a)}$	−0,11	$C_{(2)}$	0,00	$C_{(12)}$	0,00	$C_{(5a)}$	0,02
$C_{(4)}$	0,07	$C_{(13)}$	0,00	$C_{(13)}$	0,00	$C_{(9a)}$	0,00
$C_{(4a)}$	0,54*	$C_{(3a)}$	0,29*	$C_{(14)}$	−0,01	$C_{(9)}$	0,02
$C_{(5)}$	−0,08	$N_{(11)}$	−0,10*	$C_{(15)}$	0,00	$C_{(8)}$	0,00
$C_{(19)}$	0,93*	$N_{(1)}$	0,00	$C_{(16)}$	0,01	$C_{(7)}$	−0,02
$N_{(5)}$	0,15*	$C_{(3)}$	0,00	$C_{(17)}$	−0,01	$C_{(6)}$	0,00
$N_{(10)}$	−1,49*	—	—	—	—	$C_{(10a)}$	−0,02*
$N_{(11)}$	0,12	—	—	—	—	$C_{(4a)}$	0,60*
$H_{(4)}$	−0,94*	—	—	—	—	$N_{(5)}$	0,00
$H_{(4a)}$	1,64*	—	—	—	—	$N_{(10)}$	−0,02
$H_{(10a)}$	0,32*	—	—	—	—		

*These atoms are not involved in the calculations for the corresponding planes.

and was refined by full matrix least squares with anisotropic thermal approximations, to give final R = 0.075, R_w = 0.089 for 1970 reflections with $F^2 \geq 3\sigma$ ($\lambda_{Cu\,\alpha}$), Ni filter, $\theta/2\theta$ scanning, $3 \leq 2\theta \leq 120°$).

The atomic coordinates and bond angles are listed in Tables 1 and 2. The coordinates and thermal parameters for the hydrogen atoms, which were visualized in a difference Fourier synthesis, were not refined. The position of the hydrogen atom in the hydroxyethyl group did not appear, due to disorder.

3*a*,5-Dimethyl-4-acetyl-2-phenyl-3*a*,4,4*a*,5,10,10*a*-hexahydro-1,3,4-thiadiazolo[2,3-*a*]quinoxalino[2,3-*d*]pyrrole (XIa, $C_{21}H_{22}N_4OS$). To a suspension of 0.59 g (2 mmoles) thiadiazinoquinoxaline III in 10 ml ethanol was added 0.2 g acetylacetone. The reaction mixture was heated to 60-70°C and maintained at this temperature for 30 min, then cooled to room temperature and allowed to stand overnight. The resulting precipitate of XIa was removed by filtration and recrystallized from ethanol, mp 161-162°C. Yield 0.64 g (85%). PMR spectrum: 2.03 (3H, s, CH_3); 2.19 (3H, s, $COCH_3$), 2.66 (3H, s, N–CH_3); 3.20 (1H, d, J_{44a} = 9.5 Hz, 4-H); 4.28 (1H, d.d, J_{4a10a} = 5.0 Hz, 4*a*-H); 5.09 (1H, br.d, 10*a*-H); 6.4-6.7 (5H, m, benzene ring and NH-protons); 7.3-7.7 (5H, m, C_6H_5). ^{13}C NMR spectrum: 32.1 ($COCH_3$); 35.5 (CH_3); 39.0 (N–CH_3); 62.9 (C_{4a}, $^1J_{CH}$ = 145 Hz); 65.3 ($C_{(4)}$); 72.5 ($C_{(10a)}$); $^1J_{CH}$ = 163 Hz), 88.5 ($C_{(3a)}$); 113.0, 113.5, 118.9, 119.5, 125.8, 129.9, 131.2, 134.4, 132.0, 134.1, 147.4 ($C_{(2)}$); 205.7 ($COCH_3$).

3*a*,5-Dimethyl-2-phenyl-3*a*,4,4*a*,5,10,10*a*-hexahydro-1,3,4-thiadiazolo-[2,3-*a*]quinoxalino[2,3-*d*]pyrrole-4-carboxylic Acid, Methyl Ester (XIb, $C_{21}H_{22}N_4O_2S$). To a suspension of 0.59 g (2 mmoles) thiadiazinoquinoxaline III in 10 ml ethanol was added 0.23 g (2 mmoles) methyl acetoacetate IVb. The reaction mixture was heated at 60–70°C for 30 min, then allowed to stand overnight at room temperature. The resulting precipitate of compound XIb was removed by filtration and recrystallized from ethanol, mp 143–144°C. Yield 0.5 g (63%). PMR spectrum: 1.96 (3H, s, CH_3); 2.79 (3H, s, N–CH_3); 3.00 (1H, d, $J_{(4,4A)}$ = 9.5 Hz, 4-H); 3.70 (3H, s, $COCH_3$); 4.25 (1H, d.d, J_{4a10a} = 5.0 Hz, 4*a*-H); 5.07 (1H, br.d, 10*a*-H); 6.40 (1H, br.s, 10-H); 6.5–6.7 (4H, m, CH benzene ring); 7.3–7.7 ppm (5H, m, C_6H_5). Mass spectrum (I ≥ 10%), m/e: 76 (10.4), 77 (15.2), 103 (15.1), 121 (15.6), 130 (10.9), 131 (18.8), 133 (10.9), 145 (100), 146 (23.3), 177 (34.7), 394 (16.7 M^+).

3*a*,5-Dimethyl-2-phenyl-3*a*,4,4*a*,5,10,10*a*-hexahydro-1,3,4-thiadiazolo[2,3-*a*]quinoxalino[2,3-*d*]pyrrole-4-carboxylic Acid, Ethyl Ester (XIc, $C_{22}H_{24}N_4O_2S$). This was obtained in an analogous manner to compound XIb, from thiadiazinoquinoxaline III and ethyl acetoacetate IVc, mp 145–146°C. Yield 73%. PMR spectrum: 1.20 (3H, t, CH_2CH_3); 1.95 (3H, s, CH_3); 2.79 (3H, s, N–CH_3); 2.79 (3H, s, N–CH_3); 2.98 (1H, d, J_{44a} = 9.5 Hz, 4-H); 4.17 (3H, m, CH_2CH_3 and 4*a*-H); 5.07 (1H, br.d, J_{4a10a} = 5.0 Hz , 10*a*-H); 6.40 (1H, br.s, 10-H); 6.5–6.7 (4H, m, CH benzene ring); 7.3–7.7 ppm (5H, m, C_6H_5).

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