Synthesis of bis-spirocoupled pyrrolidinooxindoles derived from isatinylidene derivatives of thiazolotriazinones by [3+2] dipolar cycloaddition

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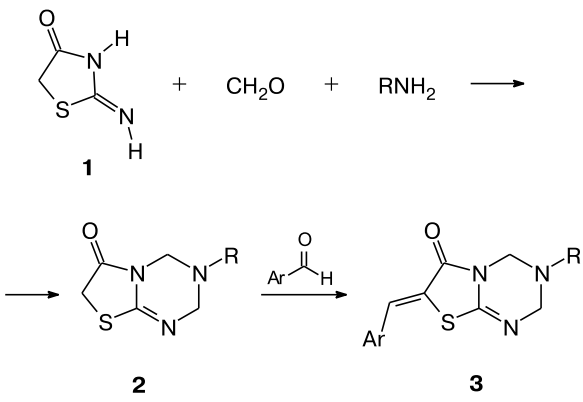
Isatines react with 3,4-dihydro-2*H*-[1,3]thiazolo[3,2-*a*][1,3,5]triazin-6(7*H*)-ones to give their isatinylidene derivatives, which add azomethinylide, generated *in situ* from paraformaldehyde and sarcosine, to yield bis-spirocoupled derivatives of pyrrolidine. The [3+2] dipolar cycloaddition reactions studied proceed absolutely diastereoselectively.

Key words: isatine, spiroheterocycle, spiropyrrolidinooxindole, azomethinylide, [3+2] dipolar cycloaddition.

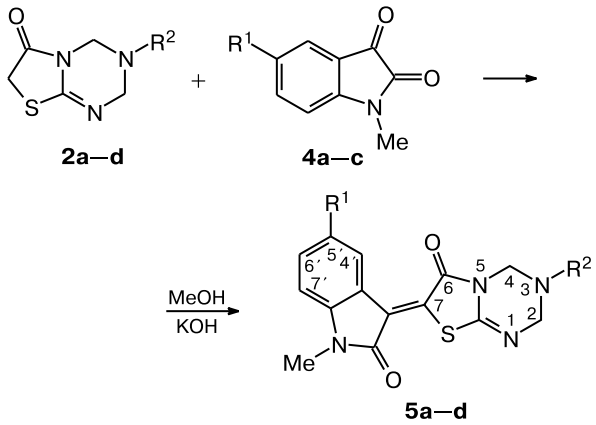
It is known[1—3] that pseudothiohidantoin **1** reacts with primary aliphatic and aromatic amines in the presence of formaldehyde to form 3,4-dihydro-2*H*-[1,3]thiazolo-[3,2-*a*][1,3,5]triazin-6(7*H*)-ones **2**, which, possessing an active methylene group, readily form with aromatic aldehydes arylidene derivatives **3** (Scheme 1).

Scheme 1



We have studied reactions of 3-aryl-3,4-dihydro-2*H*-[1,3]thiazolo[3,2-*a*][1,3,5]triazin-6(7*H*)-ones **2** with *N*-methylisatine derivatives **4a—c** (Scheme 2). The condensation was carried out in methanol in the presence of 40% aqueous KOH in catalytic amounts to give (7*Z*)-3-aryl-7-(2-oxo-1,2-dihydro-3*H*-indol-3-ylidene)-3,4-dihydro-2*H*-[1,3]thiazolo[3,2-*a*][1,3,5]triazin-6(7*H*)-ones **5a—d** in good yields.

Scheme 2



2: R^2 = 3-BrC_6H_4 (**a**), 3,4-$(OMe)_2C_6H_3$ (**b**), 4-F-C_6H_4 (**c**), 3,5-$(Me)_2C_6H_3$ (**d**); **4:** R^1 = Me (**a**), H (**b**), Br (**c**); **5:** R^1 = Me, R^2 = 3-BrC_6H_4 (**a**); R^1 = H, R^2 = 3,4-$(OMe)_2C_6H_3$ (**b**), 4-F-C_6H_4 (**c**); R^1 = Br, R^2 = 3,5-$(Me)_2C_6H_3$ (**d**).

The study of compounds obtained by ^{1}H NMR spectroscopy showed that the reactions result in the exclusive formation of *Z*-isomers, that is confirmed, first, by the absence of signals for possible *E*-isomers in the ^{1}H NMR spectra and, second, by the presence of significantly downfield shift for the proton H(4′) found in the region δ 8.7—9.0, which is due to the influence of the carbonyl group C(6)O of the [1,3]thiazolo[3,2-*a*][1,3,5]triazin-6(7*H*)-one fragment (usually, chemical shifts of the signals for the proton H(4) in isatines are found in the region δ 7.5—7.6).[4]

The thus formed carbon-carbon double bond, bound to the electron-withdrawing groups, is a good dipolaro-

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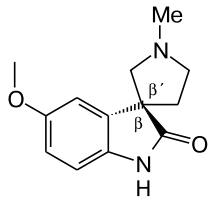
phile, and, therefore, isatinylidenes **5** can serve as convenient synthons for the synthesis of spiroheterocyclic skeletons with β,β′-spirocoupled oxindole and pyrrolidine rings. Such a β,β′-spirocoupled fragment is a part of many natural alkaloids possessing pronounced biological activity, for example, (–)-horsphiline **6**, spirotryprostatin A **7**, pteropodine **8** (see Ref. 5).



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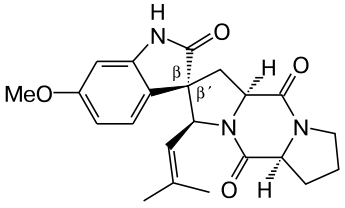

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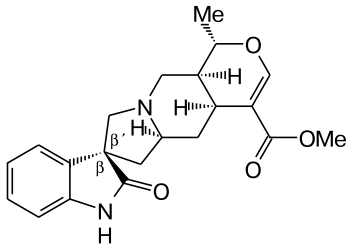

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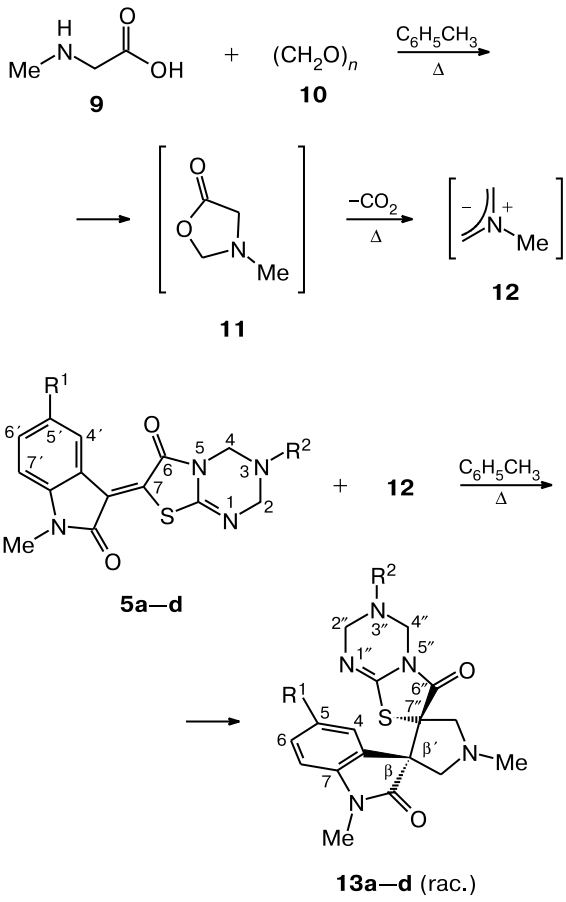
The 1,3-dipolar cycloaddition reactions of isatinylidenes **5a—d** leading to the bis-spirocoupled systems are given in Scheme 3. The *in situ* decarboxylation of the condensation product of sarcosine **9** and paraformaldehide **10**, *i.e.*, 5-oxazolidinone **11** (see Ref. 6), resulted in the generation of isomethinylide **12**, serving as a dipole in the [3+2] cycloaddition process.

The [3+2] dipolar cycloaddition reaction of **5a—d** and **12** proceeds in high yields and leads to bis-spirocycloadducts **13a—d**.

The cycloaddition results in developing two chiral centers and this means that four stereoisomers **13** can be formally formed. However, due to the fact that addition of azomethinylide **12** proceeds with equal probability from "above" or "below" of the enantiotopic plane of dipolarophile **5** with simultaneous formation of spiro-nodes, only two stereoisomers, *i.e.*, enantiomers, are formed, which are separated from the reaction mixture as a racemate.

The absence even trace amounts of other possible stereoisomers in the ^{1}H and ^{13}C NMR spectra confirms complete diastereoselectivity of the cycloaddition process. In the ^{1}H NMR spectra of compounds **13a—d**, the pairs of the diastereotopic groups CH_2(2′) and CH_2(5′) of the pyrrolidine, as well as CH_2(2″) and CH_2(4″) of the [1,3]thiazolo[3,2-*a*][1,3,5]triazin-6(7*H*)-one rings are found as AB-quartets in the regions δ 3.13—3.85 and 4.60—5.25, respectively, that confirms the presence of stereogenic tetracoordinated carbon atoms C(3′) and C(4′) in the molecules of compounds **13a—d**. The signal for the hydrogen atom H(4) is considerably upfield shifted to the region

Scheme 3



5, **13**	R^1	R^2
a	Me	3‴-BrC_6H_4
b	H	3‴,4‴-$(OMe)_2C_6H_3$
c	H	4‴-F-C_6H_4
d	Br	3‴,5‴-$Me_2C_6H_3$

δ 6.98—7.28, that indicates the absence of the double bond between the indolone and thiazolone fragments and influence of the carbonyl group of the thiazolone fragment. The carbon atoms involving into the formation of the spiro-nodes have characteristic chemical shifts in the ^{13}C NMR spectra: C(3′), in the region δ 61.9—62.11 and C(4′), about δ 60.6.

Variation of substituents both at the nitrogen atoms and in the oxindole core allows us to use this method for the synthesis of a wide range of potentially biologically active spiroheterocycles.

Experimental

^{1}H and ^{13}C NMR spectra were recorded on a Bruker DPX-250 spectrometer (250 MHz for ^{1}H and 62.5 MHz for ^{13}C, respectively) in DMSO-d_6 and $CDCl_3$ using Me_4Si as an internal standard. Mass spectra were recorded on a FINNIGAN LCQ DECA

XP MAX instrument (ESI, 1.8 kV) at 180 °C. 3-Aryl-3,4-dihydro-2*H*-[1,3]thiazolo[3,2-*a*][1,3,5]triazin-6(7*H*)-ones were obtained according to the known procedure,[1] isatine, 5-methylisatine, and 5-bromoisatine were methylated according to the described procedure.[7]

Synthesis of (7*Z*)-3-(3-aryl)-7-(1-methyl-2-oxo-1,2-dihydro-3*H*-indol-3-ylidene)-3,4-dihydro-2*H*-[1,3]thiazolo[3,2-*a*]-[1,3,5]triazin-6(7*H*)-ones 5a—d (general procedure). A 40% aqueous solution of KOH (0.001 mL) was added to a solution of equimolar amounts of the corresponding isatine **4a—c** (0.01 mol) and 3-aryl-3,4-dihydro-2*H*-[1,3]thiazolo[3,2-*a*][1,3,5]triazin-6(7*H*)-one **3a—d** (0.01 mol) in methanol (50 mL) at room temperature. After 20 min, a precipitate formed was filtered off and washed with methanol.

(7*Z*)-3-(3-Bromophenyl)-7-(1,5-dimethyl-2-oxo-1,2-dihydro-3*H*-indol-3-ylidene)-3,4-dihydro-2*H*-[1,3]thiazolo[3,2-*a*]-[1,3,5]triazin-6(7*H*)-one (5a). The yield was 97%. Dark orange crystals, m.p. 320—323 °C. Found (%): C, 53.77; H, 3.71; Br, 17.05; N, 11.89. $C_{21}H_{17}BrN_4O_2S$. Calculated (%): C, 53.74; H, 3.65; Br, 17.02; N, 11.94. ^{1}H NMR (DMSO-d_6), δ: 2.35 (s, 3 H, C(5′)Me); 3.25 (s, 3 H, N(1′)Me); 5.05 (s, 2 H, C(4)H_2); 5.39 (s, 2 H, C(2)H_2); 6.40 (d, 1 H, H(4), Ar, *J* = 7.9 Hz); 6.75—6.90 (m, 3 H, H(2), H(4), H(5), Ar); 6.96 (d, 1 H, H(7′), *J* = 8.4 Hz); 7.43 (dd, 1 H, H(6′), *J* = 8.4 Hz, *J* = 2.1 Hz); 8.65 (d, 1 H, H(4′), *J* = 2.1 Hz).

(7*Z*)-3-(3,4-Dimethoxyphenyl)-7-(1-methyl-2-oxo-1,2-dihydro-3*H*-indol-3-ylidene)-3,4-dihydro-2*H*-[1,3]thiazolo[3,2-*a*]-[1,3,5]triazin-6(7*H*)-one (5b). The yield was 71%. Dark orange crystals, m.p. 248 °C. Found (%): C, 60.60; H, 4.56; N, 12.89. $C_{22}H_20N_4O_4S$. Calculated (%): C, 60.54; H, 4.62; N, 12.84. ^{1}H NMR (DMSO-d_6), δ: 3.22 (s, 3 H, N(1′)Me); 3.64 (s, 3 H, OMe); 3.72 (s, 3 H, OMe); 4.99 (s, 2 H, C(4)H_2); 5.36 (s, 2 H, C(2)H_2); 6.49 (dd, 1 H, H(7′), *J* = 8.7 Hz, *J* = 2.7 Hz); 6.75—6.87 (m, 2 H, H(2), H(5), Ar); 7.02—7.15 (m, 2 H, H(6), Ar, H(6′)); 7.40 (dd, 1 H, H(5′), *J* = 8.6 Hz, *J* = 8.6 Hz); 8.77 (dd, 1 H, H(4′), *J* = 8.6 Hz, *J* = 1.3 Hz).

(7*Z*)-3-(4-Fluorophenyl)-7-(1-methyl-2-oxo-1,2-dihydro-3*H*-indol-3-ylidene)-3,4-dihydro-2*H*-[1,3]thiazolo[3,2-*a*][1,3,5]-triazin-6(7*H*)-one (5c). The yield was 66%. Dark orange crystals, m.p. 292 °C. Found (%): C, 60.99; H, 3.93; F, 4.88; N, 14.14. $C_{20}H_{15}FN_4O_2S$. Calculated (%): C, 60.90; H, 3.83; F, 4.82; N, 14.20. ^{1}H NMR (DMSO-d_6), δ: 3.24 (s, 3 H, N(1′)Me); 5.02 (s, 2 H, C(4)H_2); 5.39 (s, 2 H, C(2)H_2); 7.05—7.20 (m, 6 H, Ar, H(7′), H(6′)); 7.44 (dd, 1 H, H(5′), *J* = 7.9 Hz, *J* = 7.9 Hz); 8.80 (dd, 1 H, H(4′), *J* = 7.9 Hz, *J* = 1.2 Hz).

(7*Z*)-7-(5-Bromo-1-methyl-2-oxo-1,2-dihydro-3*H*-indol-3-ylidene)-3-(3,5-dimethylphenyl)-3,4-dihydro-2*H*-[1,3]thiazolo[3,2-*a*][1,3,5]triazin-6(7*H*)-one (5d). The yield was 86%. Dark orange crystals, m.p. 274 °C. Found (%): C, 54.68; H, 4.05; Br, 16.42; N, 11.67. $C_{22}H_{19}BrN_4O_2S$. Calculated (%): C, 54.66; H, 3.96; Br, 16.53; N, 11.59. ^{1}H NMR (DMSO-d_6), δ: 2.19 (s, 6 H, 5-Me, 3-Me, Ar); 3.21 (s, 3 H, N(1′)Me); 5.00 (s, 2 H, C(4)H_2); 5.39 (s, 2 H, C(2)H_2); 6.40—6.70 (m, 3 H, Ar); 6.98 (d, 1 H, H(7′), *J* = 8.2 Hz); 7.51 (dd, 1 H, H(6′), *J* = 8.2 Hz, *J* = 1.9 Hz); 8.97 (d, 1 H, H(4′), *J* = 1.9 Hz).

Synthesis of 3″-(3-aryl)-1,1′-dimethyl-3″,4″-dihydro-2″*H*-dispiro[indole-3,3′-pyrrolidine-4′,7″-[1,3]thiazolo[3,2-*a*][1,3,5]-triazine]-2,6″(1*H*)-diones 13a—d (general procedure). The corresponding (7*Z*)-3-(3-aryl)-7-(1-methyl-2-oxo-1,2-dihydro-3*H*-indol-3-ylidene)-3,4-dihydro-2*H*-[1,3]thiazolo[3,2-*a*][1,3,5]-triazin-6(7*H*)-one **5a—d** (0.001 mol), sarcosine **9** (0.004 mol), and paraformaldehide **10** (0.004 mol) were suspended in toluene (40 mL). The reaction mixture was refluxed for 5—6 h. The reaction progress was monitored by TLC on Silufol-254 plate in AcOEt with visualization in iodine vapors. After the reaction reached completion, the reaction mixture was cooled, toluene was evaporated at reduced pressure, the product was isolated by column chromatography (Merck Silicagel-60 (60—200 μm), eluent: AcOEt).

3″-(3‴-Bromophenyl)-1,1′,5-trimethyl-3″,4″-dihydro-2″*H*-dispiro[indole-3,3′-pyrrolidine-4′,7″-[1,3]thiazolo[3,2-*a*]-[1,3,5]triazine]-2,6″(1*H*)-dione (13a). The yield was 55%. Colorless crystals, m.p. 136 °C. Found (%): C, 54.63; H, 4.55; Br, 15.07; N, 13.41. $C_{24}H_{24}BrN_5O_2S$. Calculated (%): C, 54.76; H, 4.60; Br, 15.18; N, 13.30. ^{1}H NMR ($CDCl_3$), δ: 2.13 (s, 3 H, C(5)Me); 2.60 (s, 3 H, N(1′)Me); 3.13 (d, 1 H, C(2′)H_2, *J* = 10.4 Hz); 3.17 (s, 3 H, N(1)Me); 3.42 (d, 1 H, C(5′)H_2, *J* = 10.6 Hz); 3.59 (d, 1 H, C(2′)H_2, *J* = 10.4 Hz); 3.82 (d, 1 H, C(5′)H_2, *J* = 10.6 Hz); 4.60 (d, 1 H, C(4″)H_2, *J* = 15.8 Hz); 4.75 (d, 1 H, C(4″)H_2, *J* = 15.8 Hz); 4.88 (d, 1 H, C(2″)H_2, *J* = 12.7 Hz); 5.13 (d, 1 H, C(2″)H_2, *J* = 12.7 Hz); 6.60—6.75 (d, m, 2 H, H(7), H_{Ar}(6‴), *J* = 7.9 Hz); 6.93—7.07 (m, 4 H, H(6), H(4), H_{Ar}(2‴) + H_{Ar}(4‴)); 7.08—7.15 (m, 1 H, H_{Ar}(5‴)). ^{13}C NMR ($CDCl_3$), δ: 21.3 (C(5)$\underline{C}H_3$), 26.3 (N(1)$\underline{C}H_3$), 42.7 (N(1′)$\underline{C}H_3$), 60.6 (C(4′)), 61.7 (C(5′)), 61.9 (C(3′)), 62.4 (C(2′)), 63.6 (C(4″)), 65.6 (C(2″)), 108.1 (C(7)), 117.9 (C_{Ar}(6‴)), 123.2 (C_{Ar}(2‴), C_{Ar}(4‴)), 123.7 (C_{Ar}(3‴)), 125.1 (C(4)), 126.2 (C(6)), 130.0 (C(3a)), 130.6 (C_{Ar}(5‴)), 132.3 (C(5)), 142.3 (C(7a)), 148.9 (C_{Ar}(1‴)), 150.4 (C(8a″)), 171.8 (C(2)), 177.2 (C(6″)). MS (ESI), *m/z*: 527 $[M + 1]^+$.

3″-(3‴,4‴-Dimethoxyphenyl)-1,1′-dimethyl-3″,4″-dihydro-2″*H*-dispiro[indole-3,3′-pyrrolidine-4′,7″-[1,3]thiazolo-[3,2-*a*][1,3,5]triazine]-2,6″(1*H*)-dione (13b). The yield was 74%. Colorless crystals, m.p. 134 °C. Found (%): C, 60.86; H, 5.61; N, 14.12. $C_{25}H_{27}N_5O_4S$. Calculated (%): C, 60.84; H, 5.51; N, 14.19. ^{1}H NMR ($CDCl_3$), δ: 2.59 (s, 1 H, N(1′)Me); 3.11 (d, 1 H, C(2′)H_2, *J* = 10.4 Hz); 3.17 (s, 3 H, N(1)Me); 3.39 (d, 1 H, C(5′)H_2, *J* = 10.7 Hz); 3.60 (d, 1 H, C(2′)H_2, *J* = 10.4 Hz); 3.73 (s, 3 H, OMe); 3.85 (s, d, 4 H, C(5′)H_2, OMe, *J* = = 10.7 Hz); 4.64 (d, 1 H, C(4″)H_2, *J* = 15.8 Hz); 4.69 (d, 1 H, C(4″)H_2, *J* = 15.8 Hz); 4.97 (d, 1 H, C(2″)H_2, *J* = 12.8 Hz); 5.03 (d, 1 H, C(2″)H_2, *J* = 12.8 Hz); 6.28 (dd, 1 H, H_{Ar}(6‴), *J* = 8.6 Hz, *J* = 2.6 Hz); 6.35 (d, 1 H, H_{Ar}(2‴), *J* = 2.6 Hz); 6.57 (m, 2 H, H_{Ar}(5‴); H(6)); 6.73 (d, 1 H, H(7), *J* = 7.7 Hz); 6.98 (d, 1 H, H(4), *J* = 7.7 Hz); 7.16 (dd, 1 H, H(5), *J* = 7.7 Hz, *J* = 7.7 Hz). ^{13}C NMR ($CDCl_3$), δ: 26.2 (N(1)$\underline{C}H_3$), 42.7 (N(1′)$\underline{C}H_3$), 55.7 (C(3)O$\underline{C}H_3$), 56.0 (C(4)O$\underline{C}H_3$), 60.5 (C(4′)), 61.7 (C(5′)), 61.9 (C(3′)), 63.1 (C(2′)), 63.4 (C(4″)), 66.2 (C(2″)), 104.6 (C_{Ar}(2‴)), 108.1 (C(7)), 111.4 (C_{Ar}(5‴)), 111.5 (C_{Ar}(6‴)), 122.7 (C(5)), 123.3 (C(3a)), 124.1 (C(4)), 129.4 (C(6)), 141.3 (C(7a)), 144.5 (C_{Ar}(4‴)), 145.3 (C_{Ar}(1‴)), 149.3 (C_{Ar}(3‴)), 150.2 (C(8a″)), 172.0 (C(2)), 177.2 (C(6″)). MS (ESI), *m/z*: 493 $[M + 1]^+$.

3″-(4‴-Fluorophenyl)-1,1′-dimethyl-3″,4″-dihydro-2″*H*-dispiro[indole-3,3′-pyrrolidine-4′,7″-[1,3]thiazolo[3,2-*a*]-[1,3,5]triazine]-2,6″(1*H*)-dione (13c). The yield was 64%. Colorless crystals, m.p. 184 °C. Found (%): C, 61.22; H, 4.89; F, 4.15; N, 15.62. $C_{23}H_{22}FN_5O_2S$. Calculated (%): C, 61.18; H, 4.91; F, 4.21; N, 15.51. ^{1}H NMR ($CDCl_3$), δ: 2.60 (s, 1 H, N(1′)Me); 3.12 (d, 1 H, CH_2(2′), *J* = 10.4 Hz); 3.19 (s, 3 H, N(1)Me); 3.41 (d, 1 H, C(5′)H_2, *J* = 10.6 Hz); 3.62 (d, 1 H, C(2′)H_2, *J* = 10.4 Hz); 3.85 (d, 1 H, C(5′)H_2, *J* = 10.6 Hz); 4.61 (d, 1 H, C(4″)H_2, *J* = 15.8 Hz); 4.70 (d, 1 H, C(4″)H_2, *J* = 15.8 Hz); 4.98 (d, 1 H,

C(2″)H_2, J = 13.0 Hz); 5.04 (d, 1 H, C(2″)H_2, J = 13.0 Hz); 6.63 (dd, 1 H, H(6), J = 7.6 Hz, J = 7.6 Hz); 6.66—6.90 (m, 5 H, H(7), Ar); 7.03 (d, 1 H, H(4), J = 7.6 Hz); 7.21 (dd, 1 H, H(5), J = 7.6 Hz, J = 7.6 Hz). ^{13}C NMR ($CDCl_3$), δ: 26.2 (N(1)$\underline{C}H_3$), 42.7 (N(1′)$\underline{C}H_3$), 60.5 (C(4′)), 61.8 (C(5′)), 62.1 (C(2′)), 62.6 (C(3′)), 63.2 (C(4″)), 66.1 (C(2″)), 108.3 (C(7)), 116.0 (d, C_{Ar}(3‴), C_{Ar}(5‴), J = 22.5 Hz); 121.4 (d, C_{Ar}(2‴), C_{Ar}(6‴), J = 8.1 Hz); 122.8 (C(5)), 123.4 (C(3a)), 124.2 (C(4)), 129.5 (C(6)), 143.6 (d, C_{Ar}(1‴), J = 2.6 Hz), 144.6 (C(7a)), 150.3 (C(8a″)), 158.0 (d, C_{Ar}(4‴), J = 242.1 Hz), 172.0 (C(2)), 177.2 (C(6″)). MS (ESI), m/z: 451 $[M + 1]^+$.

5-Bromo-3″-(3‴,5‴-dimethylphenyl)-1,1′-dimethyl-3″,4″-dihydro-2″*H*-dispiro[indole-3,3′-pyrrolidine-4′,7″-[1,3]thiazolo-[3,2-*a*][1,3,5]triazine]-2,6″(1*H*)-dione (13d). The yield was 53%. Colorless crystals, m.p. 175 °C. Found (%): C, 55.63; H, 4.91; Br, 14.62; N, 13.06. $C_{25}H_{26}BrN_5O_2S$. Calculated (%): C, 55.56; H, 4.85; Br, 14.78; N, 12.96. ^{1}H NMR ($CDCl_3$), δ: 2.23 (s, 6 H, C(3‴)Me, C(5‴)Me, Ar); 2.59 (s, 3 H, N(1′)Me); 3.13 (d, 1 H, C(5′)H_2, J = 10.4 Hz); 3.17 (s, 3 H, N(1)Me); 3.38 (d, 1 H, C(2′)H_2, J = 10.7 Hz); 3.58 (d, 1 H, C(5′)H_2, J = 10.4 Hz); 3.79 (d, 1 H, C(2′)H_2, J = 10.7 Hz); 4.56 (d, 1 H, C(4″)H_2, J = 15.8 Hz); 4.72—4.90 (m, 2 H, C(4″)H_2, C(2″)H_2); 5.25 (d, 1 H, C(2″)H_2, J = 12.6 Hz); 6.49 (s, 2 H, H_{Ar}(6‴), H_{Ar}(2‴)); 6.60—6.72 (m, 2 H, H_{Ar}(4‴), H(7)); 7.28 (d, 1 H, H(4), J = 1.6 Hz); 7.33 (dd, 1 H, H(5), J = 8.2 Hz, J = 1.9 Hz). ^{13}C NMR ($CDCl_3$), δ: 21.5 (2 C, C(5)$\underline{C}H_3$, C(3)$\underline{C}H_3$, Ar), 26.3 (N(1)$\underline{C}H_3$), 42.6 (N(1′)$\underline{C}H_3$), 60.9 (C(4′)), 61.8 (C(5′)), 62.1 (C(3′)), 62.8 (C(2′)), 63.3 (C(4″)), 62.6 (C(2″)), 109.6 (C(7)), 115.4 (C(5)), 117.3 (2 C, C_{Ar}(2‴), C_{Ar}(6‴)), 125.1 (C(4)), 125.8 (C(3a)), 127.7 (C_{Ar}(4‴)), 132.4 (C(6)), 139.1 (2 C, C_{Ar}(3‴), C_{Ar}(5‴)), 143.8 (C(7a)), 147.2 (C_{Ar}(1‴)), 149.9 (C(8″a)), 171.6 (C(2)), 176.6 (C(6″)). MS (ESI), m/z: 541 $[M + 1]^+$.

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