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SYNTHESIS OF 3,4-DIHYDRO-2*H*-THIOPYRANS AND THIOPYRANO[3,4-*c*]CHROMENES HAVING A 1,2,3-TRIAZOLE SUBSTITUENT BY USING THIONYLATION – HETERO-DIELS–ALDER DOMINO REACTION

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We have developed a thionylation – hetero-Diels–Alder domino reaction using α,β-unsaturated ketones with 1,2,3-triazole substituents, namely, 3-[2-(allyloxy)phenyl]-1-(1-aryl-5-methyl-1H-1,2,3-triazol-4-yl)-prop-2-en-1-ones, leading to 3,4-dihydro-2H-thiopyranes and thiopyrano[3,4-c]chromenones. Variants of intra- and intermolecular cycloaddition were studied, as well the stereo- and regioselectivity of such reactions was assessed.

Keywords: thiopyran, thiopyrano[3,4-*c*]chromene, 1,2,3-triazole, domino reaction, hetero-Diels–Alder reaction, thionylation.

The properties of thiopyrans and their condensed derivatives, as well as the methods for preparation of these compounds attract an increasing interest of researchers [1-4]. 3,4-Dihydro-2*H*-thiopyrans and thiopyrano[3,4-*c*]chromenones have been well studied, due to the easy preparation of these compounds by Knoevenagel – hetero-Diels–Alder domino reaction [5-7]. This method of creating heterocyclic systems with a thiopyran ring has been quite productive and several variants have been developed [8-13]. Another method has been studied less and involves thionylating unsaturated ketones with Lawesson's reagent [14-17] or treating amides with phosphorus pentasulfide in pyridine [18] in order to obtain the heterodiene fragment C=C–C=S used in cycloaddition. The effects of electron-deficient heterocycles on the course of such reactions have not been investigated.

In the current work, we studied the possibility of performing this synthesis with 4-acetyl-1-aryl-5-methyl-1,2,3-triazoles obtained by reacting aryl azides with acetylacetone [19]. Our developed method provides access to similar substituted triazoles, which can be used as reagents. The acetyltriazoles **1a**-**c** reacted with the aldehydes **2a**-**c**, giving high yields of the α,β-unsaturated ketones **3a**-**e**. Our attempts to use these chalcones in a hetero-Diels–Alder reaction showed that the heterodiene system C=C–C=O was insufficiently active for cycloaddition with acrylates, but an intermolecular variant of thionylation – a hetero-Diels–Alder domino reaction was successfully accomplished with the ketone **3a** and methyl acrylate (**4**) in the presence of

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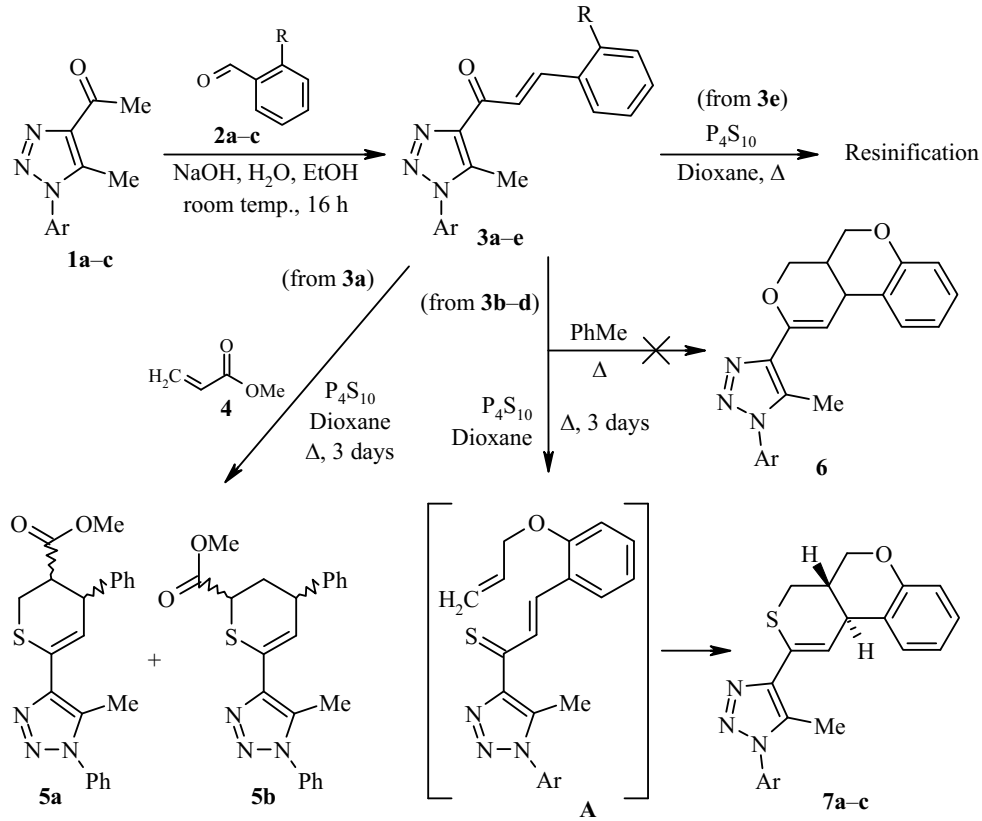
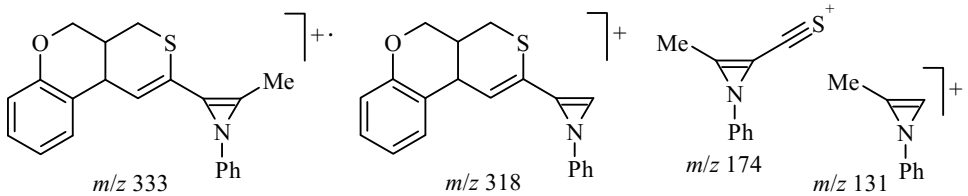
phosphorus pentasulfide. The resulting mixture of regioisomers **5a** and **5b** exhibited a double set of ^{1}H NMR signals. According to LC/MS data, the ratio of the regioisomers **5a**:**5b** was 5:1, and this reaction could not be performed regioselectively. The chalcones **3b-e**, containing both the diene and the dienophile fragment in one molecule, would prevent the formation of regioisomers in the case of a successful intramolecular (2+4) cycloaddition. It was determined that no cycloaddition occurred during refluxing compounds **3b-d** in toluene, and compound **6** was not obtained. In this case, we used thionylation of a keto group to obtain the diene system C=C–C=S. Substituting the oxygen atom with a sulfur atom increased the energy of HOMO and decreased the energy of LUMO of the diene molecule, i.e., reduced the energy barrier between the frontier orbitals and increased the heterodiene reactivity in (2+4) cycloaddition reactions. It was found that heating the ketones **3b-d** with phosphorus pentasulfide in dioxane resulted in thionylation with a subsequent hetero-Diels–Alder reaction, producing compounds **7a-c**, which contained the little known heterocyclic system of dihydro-4*H*,5*H*-thiopyrano[3,4-*c*]chromene.



1, **7 a** Ar = Ph, **b** Ar = 4-MeC_6H_4, **c** Ar = 4-ClC_6H_4; **2 a** R = H, **b** R = $OCH_2CH{=}CH_2$, **c** R = $OCH_2C{\equiv}CH$; **3 a** Ar = Ph, R = H, **b** Ar = Ph, R = $OCH_2CH{=}CH_2$, **c** Ar = 4-MeC_6H_4, R = $OCH_2CH{=}CH_2$, **d** Ar = 4-ClC_6H_4, R = $OCH_2CH{=}CH_2$, **e** Ar = Ph, R = $OCH_2C{\equiv}CH$

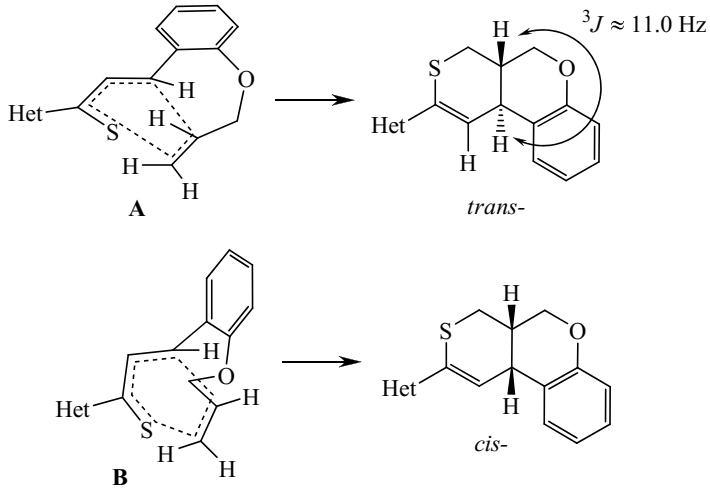
This type of domino reaction was performed by us for the first time. This method enabled the preparation of chromatographically pure products. It should be noted that compound **3e**, containing a propargyl fragment instead of the allyl group, did not undergo cycloaddition under such conditions, and gave only intractable resin.



The mass spectrum of compound **7a** contained a weak molecular ion peak (*m*/*z* 361), a strong signal of the fragment ion $[M\text{-}N_2]^{+\cdot}$ (*m*/*z* 333), as well as characteristic peaks with *m*/*z* 318, 174, and 131, which were formed by fragmentation of the $[M\text{-}N_2]^{+\cdot}$ daughter ion.

The presence of sulfur in compound **7a** was evident from the strong signals with *m*/*z* 32 and 44.

We should note that the (2+4) cycloaddition occurred stereoselectively, with *trans* fusion of the pyran and thiopyran rings, which was evidenced by the single set of NMR signals and the high value of coupling constant for the protons common to both rings ($^3J \approx 11$ Hz). Such a stereochemical result may be explained from the geometry of the transition states **A** and **B**, which lead to the *trans* or *cis* products. The protons common to both rings approach each other in the transition state **B**, thus increasing the energy of this transition state. The favored transition state is **A**, where the protons common to both rings are located at the opposite sides of the fused rings.



Het = 1-aryl-5-methyl-1*H*-1,2,3-triazol-4-yl

Thus, we have developed a thionylation – hetero-Diels–Alder domino reaction using α,β-unsaturated ketones of 1,2,3-triazole series, enabling the preparation of new 3,4-dihydro-2*H*-thiopyrans and thiopyrano[3,4-*c*]-chromenones.

EXPERIMENTAL

^{1}H NMR spectra of compounds **7b**,**c** were acquired on a Bruker Avance 500 instrument (500 MHz), those of the other compounds – on a Varian Unity 400 instrument (400 MHz). ^{13}C NMR spectra were acquired on a Bruker Avance 500 instrument (500 MHz). The solvent for all NMR spectra was DMSO-d_6, with TMS as internal standard. Mass spectra were recorded on an Agilent 1100 LC/MSD chromato-mass spectrometer, with chemical ionization at the ambient pressure. Additionally, EI mass spectrum (at 70 eV) was recorded for compound **7a** on a Finnigan MAT INKOS-50 spectrometer. Elemental analysis was performed on a Carlo Erba 1106 instrument. Melting points were determined on a Boetius apparatus. The starting ketones **1a**-**c** were obtained according to a published method [19].

Synthesis of the Chalcones 3a-e (General Method). The ketone **1a**-**c** (7.5 mmol) was dissolved in a minimum amount of EtOH and added to a cold (0°C) 10% NaOH solution (4 ml). The aldehyde **2a**-**c** (7.5 mmol) was added dropwise, and the reaction mixture was left overnight at room temperature. The precipitate was filtered off, washed with water, and recrystallized from a mixture of EtOH–DMF.

(2*E*)-1-(5-Methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)-3-phenylprop-2-en-1-one (3a). Yield 88%. White crystals. Mp 154-155°C (mp 123-125°C [20]). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.60 (3H, s, CH_3); 7.47-7.50

(3H, m, H-3,4,5 Ph); 7.65-7.69 (5H, m, H NPh); 7.82-7.85 (2H, m, H-2,6 Ph); 7.87 (1H, d, *J* = 16.1) and 8.04 (1H, d, *J* = 16.1, COCH=CH). ^{13}C NMR spectrum, δ, ppm: 10.4 (CH_3); 123.4 (CH=); 125.7 (C-2,6 Ph); 126.0 (C-2,6 NPh); 130.2 (C-3,5 NPh); 130.3 (C-3,5 Ph); 130.6 (C-4 Ph); 130.7 (C-4 PhN); 131.3 (C-5); 135.3 (C-1 Ph); 135.6 (C-1 PhN); 139.5 (CH=); 143.6 (C-4); 183.9 (CO). Mass spectrum, *m*/*z*: 290 $[M+H]^+$. Found, %: C 74.64; H 5.06; N 14.30. $C_{18}H_{15}N_3O$. Calculated, %: C 74.72; H 5.23; N 14.52.

(2*E*)-3-[2-(Allyloxy)phenyl]-1-(5-methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)prop-2-en-1-one (3b). Yield 87%. White crystals. Mp 106-107°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.65 (3H, s, CH_3); 4.72 (2H, d, *J* = 4.9, OCH_2); 5.35 (1H, d, *J* = 10.4) and 5.53 (1H, d, *J* = 17.2, $=CH_2$); 6.09-6.21 (1H, m, C<u>H</u>=CH_2); 7.03 (1H, t, *J* = 7.6, H-5 Ar); 7.06 (1H, d, *J* = 8.0, H-6 Ar); 7.39 (1H, t, *J* = 7.8, H-4 Ar); 7.59-7.66 (5H, m, H Ph); 7.80 (1H, d, *J* = 7.4, H-3 Ar); 8.05 (1H, d, *J* = 16.2) and 8.17 (1H, d, *J* = 16.2, COCH=CH). Mass spectrum, *m/z*: 346 $[M+H]^+$. Found, %: C 73.17; H 5.40; N 12.07. $C_{21}H_{19}N_3O_2$. Calculated, %: C 73.03; H 5.54; N 12.17.

(2*E*)-3-[2-(Allyloxy)phenyl]-1-[5-methyl-1-(4-methylphenyl)-1*H*-1,2,3-triazol-4-yl]prop-2-en-1-one (3c). Yield 85%. Mp 102-103°C. White crystals. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.44 (3H, s, ArC<u>H</u>$_3$); 2.59 (3H, s, 5-CH_3); 4.74 (2H, d, *J* = 4.7, OCH_2); 5.35 (1H, d, *J* = 10.4) and 5.52 (1H, d, *J* = 16.8, $=CH_2$); 6.15 (1H, ddd, *J* = 4.7, *J* = 10.4, *J* = 16.8, C<u>H</u>=CH_2); 7.07 (1H, t, *J* = 7.4, H-5 Ar); 7.16 (1H, d, *J* = 8.4, H-6 Ar); 7.42-7.50 (3H, m, H-4 Ar, H-3,5 NAr); 7.55 (2H, d, *J* = 8.1, H-2,6 NAr); 7.85 (1H, d, *J* = 7.5, H-3 Ar); 8.10 (1H, d, *J* = 16.2) and 8.18 (1H, d, *J* = 16.2, COCH=CH). ^{13}C NMR spectrum, δ, ppm: 10.4 (CH_3); 21.3 (CH_3); 69.3 (OCH_2); 113.6 (C-6 Ar); 118.1 ($=CH_2$); 121.6 (C-4 Ar); 123.6 (C-2 Ar); 123.7 (<u>C</u>H=CH_2); 125.7 (C-2,6 NAr); 129.4 (C-3 Ar); 130.6 (C-3,5 NAr); 132.7 (C-1 NAr); 133.1 (C-5); 133.8 (CH=); 138.4 (CH=); 139.3 (C-5 Ar); 140.5 (C-4 NAr); 143.6 (C-4); 157.9 (C-1 Ar); 184.2 (CO). Mass spectrum, *m*/*z*: 360 $[M+H]^+$. Found, %: C 73.60; H 5.71; N 11.54. $C_{22}H_{21}N_3O_2$. Calculated, %: C 73.52; H 5.89; N 11.69.

(2*E*)-3-[2-(Allyloxy)phenyl]-1-[1-(4-chlorophenyl)-5-methyl-1*H*-1,2,3-triazol-4-yl]prop-2-en-1-one (3d). Yield 89%. White crystals. Mp 108-109°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.60 (3H, s, CH_3); 4.73 (2H, d, *J* = 5.0, OCH_2); 5.34 (1H, dd, *J* = 1.4, *J* = 10.6) and 5.51 (1H, dd, *J* = 1.4, *J* = 17.3, $=CH_2$); 6.09-6.19 (1H, m, C<u>H</u>=CH_2); 7.06 (1H, t, *J* = 7.5, H-5 Ar); 7.15 (1H, d, *J* = 8.4, H-6 Ar); 7.46 (1H, t, *J* = 7.8, H-4 Ar); 7.70 (2H, d, *J* = 8.9, H-2,6 NAr); 7.75 (2H, d, *J* = 8.9, H-3,5 NAr); 7.84 (1H, d, *J* = 7.7, H-3 Ar); 8.08 (1H, d, *J* = 16.1) and 8.16 (1H, d, *J* = 16.1, COCH=CH). Mass spectrum (^{35}Cl isotope), *m*/*z*: 378 $[M+H]^+$. Found, %: C 66.58; H 4.64; N 11.19. $C_{21}H_{18}ClN_3O_2$. Calculated, %: C 66.40; H 4.78; N 11.06.

(2*E*)-1-(5-Methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)-3-[2-(prop-2-yn-1-yloxy)phenyl]prop-2-en-1-one (3e). Yield 82%. White crystals. Mp 137-138°C. ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.61 (3H, s, CH_3); 3.65 (1H, t, *J* = 2.2, ≡CH); 4.99 (2H, d, *J* = 2.2, OCH_2); 7.11 (1H, t, *J* = 7.5, H-5 Ar); 7.22 (1H, d, *J* = 8.4, H-6 Ar); 7.49 (1H, t, *J* = 8.5, H-4 Ar); 7.65-7.70 (5H, m, H Ph); 7.88 (1H, d, *J* = 7.6, H-3 Ar); 8.05 (1H, d, *J* = 16.1) and 8.12 (1H, d, *J* = 16.1, COCH=CH). ^{13}C NMR spectrum, δ, ppm: 10.4 (CH_3); 56.7 (OCH_2); 79.1 (≡CH); 79.4 (<u>C</u>≡CH); 114.0 (C-6 Ar); 122.2 (C-4 Ar); 123.8 (CH=); 123.9 (C-2 Ar); 126.0 (C-2,6 Ph); 129.1 (C-3 Ar); 130.2 (C-3,5 Ph); 130.6 (C-4 Ph); 132.6 (C-5); 135.6 (C-1 Ph); 138.0 (CH=); 139.4 (C-5 Ar); 143.7 (C-4); 156.9 (C-1 Ar); 184.1 (CO). Mass spectrum, *m/z*: 344 $[M+H]^+$. Found, %: C 73.20; H 5.18; N 12.08. $C_{21}H_{17}N_3O_2$. Calculated, %: C 73.45; H 4.99; N 12.24.

Synthesis of 3,4-Dihydro-2*H*-thiopyrans 5a,b and Thiopyrano[3,4-*c*]chromenones 7a-c (General Method). The chalcone **3a-d** (5.0 mmol) was dissolved with heating in anhydrous dioxane (10 ml), and P_4S_{10} (0.6 g, 1.3 mmol) was added to the solution. In the case of the chalcone **3a**, methyl acrylate (0.45 ml, 5.0 mmol) was also added to the solution. The reaction mixture was refluxed for 3 days in a flask equipped with a calcium chloride drying tube, then cooled to room temperature, and filtered through alumina. The filtrate was poured on ice. The precipitate that formed was filtered off, washed with water, and recrystallized from aqueous EtOH. In the cases when the product was a thick liquid, it was extracted with hot hexane, then the extract was evaporated under reduced pressure at room temperature.

Methyl 6-(5-methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)-4-phenyl-3,4-dihydro-2*H*-thiopyran-3-carboxylate (5a) and methyl 6-(5-methyl-1-phenyl-1*H*-1,2,3-triazol-4-yl)-4-phenyl-3,4-dihydro-2*H*-thiopyran-2-carboxylate (5b) were isolated with the total yield of 52% (the isomer ratio of **5a**:**5b** ≈ 5:1). White powder. Mp

137-138°C (EtOH). ^{1}H NMR spectrum of compound **5a**, δ, ppm (*J*, Hz): 2.38 (3H, s, 5'-CH_3); 3.03 (2H, d, *J* = 6.5, 2-CH_2); 3.20-3.24 (1H, m, 3-CH); 3.56 (3H, s, $COOCH_3$); 4.23 (1H, t, *J* = 5.8, 4-CH); 6.24 (1H, d, *J* = 6.2, H-5); 7.18 (2H, d, *J* = 6.2, H-2,6 Ph); 7.27 (1H, t, *J* = 6.1, H-4 Ph); 7.74-7.59 (7H, m, H Ph). ^{1}H NMR spectrum of compound **5b**, δ, ppm (*J*, Hz): 2.34 (3H, s, 5'-CH_3); 2.75 (2H, dd, *J* = 5.2, *J* = 10.7, 3-CH_2); 3.17 (1H, d, *J* = 5.2, 2-CH); 3.76 (3H, s, $COOCH_3$); 4.06 (1H, dd, *J* = 3.9, *J* = 10.7, 4-CH); 6.09 (1H, d, *J* = 3.9, H-5); 7.18 (2H, d, *J* = 6.2, H Ph); 7.25-7.39 (1H, m, H Ph); 7.59-7.74 (7H, m, H Ph). Mass spectrum, *m*/*z*: 392 $[M+H]^+$. Found, %: C 67.21; H 5.18; N 10.60. $C_{22}H_{21}N_3O_2S$. Calculated, %: C 67.50; H 5.41; N 10.73.

4-(4a,10b-Dihydro-4*H*,5*H*-thiopyrano[3,4-*c*]chromen-2-yl)-5-methyl-1-phenyl-1*H*-1,2,3-triazole (7a). Yield 69%. White powder. Mp 158-159°C (EtOH). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.17-2.29 (1H, m, 4a-CH); 2.39 (3H, s, CH_3); 3.02 (1H, t, *J* = 11.7) and 3.08 (1H, dd, *J* = 3.4, *J* = 11.7, 4-CH_2); 3.70 (1H, d, *J* = 10.9, H-10b); 3.92 (1H, t, *J* = 10.8) and 4.44 (1H, dd, *J* = 3.4, *J* = 10.8, 5-CH_2); 6.54 (1H, s, H-1); 6.82 (1H, d, *J* = 7.8, H-7); 6.94 (1H, t, *J* = 7.4, H-9); 7.14 (1H, t, *J* = 7.6, H-8); 7.45 (1H, d, *J* = 7.7, H-10); 7.55-7.69 (5H, m, H Ph). ^{13}C NMR spectrum, δ, ppm: 10.4 (CH_3); 28.0 (C-4); 34.0 (C-10b); 37.3 (C-4a); 69.7 (C-5); 117.0 (C-7); 121.0 (C-8); 122.2 (C-9); 123.9 (C-10a); 125.5 (C-2); 125.7 (C-2,6 Ph); 126.6 (C-1); 128.1 (C-10); 130.1 (C-3,5 Ph); 130.2 (C-4 Ph); 131.2 (C-5 triazole); 136.4 (C-1 Ph); 143.3 (C-4 triazole); 154.4 (C-6a). Mass spectrum (CI), *m/z*: 362 $[M+H]^+$. Mass spectrum (EI), *m/z* (I_{rel}, %): 361 $[M]^+$ (16), 333 $[M-N_2]^+$ (66), 318 (10), 174 (13), 131 (23). Found, %: C 69.91; H 5.11; N 11.58. $C_{21}H_{19}N_3OS$. Calculated, %: C 69.78; H 5.30; N 11.62.

4-(4a,10b-Dihydro-4*H*,5*H*-thiopyrano[3,4-*c*]chromen-2-yl)-5-methyl-1-(4-methylphenyl)-1*H*-1,2,3-triazole (7b). Yield 61%. White powder. Mp 165-166°C (EtOH). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.07-2.20 (1H, m, 4a-CH); 2.38 (3H, s, 5'-CH_3); 2.43 (3H, s, Ar$C\underline{H}_3$); 2.97-3.13 (2H, m, 4-CH_2); 3.70 (1H, d, *J* = 11.1, 10b-CH); 3.92 (1H, t, *J* = 10.6) and 4.45 (1H, dd, *J* = 1.7, *J* = 10.6, 5-CH_2); 6.54 (1H, s, H-1); 6.83 (1H, d, *J* = 7.5, H-7); 6.94 (1H, t, *J* = 7.3, H-9); 7.15 (1H, t, *J* = 7.6, H-8); 7.42-7.46 (3H, m, H-10, H-2,6 Ar); 7.49 (2H, d, *J* = 7.2, H-3,5 Ar). Mass spectrum, *m*/*z*: 376 $[M+H]^+$. Found, %: C 70.20; H 5.89, N 11.01. $C_{22}H_{21}N_3OS$. Calculated, %: C 70.37; H 5.64, N 11.19.

1-(4-Chlorophenyl)-4-(4a,10b-dihydro-4*H*,5*H*-thiopyrano[3,4-*c*]chromen-2-yl)-5-methyl-1*H*-1,2,3-triazole (7c). Yield 64%. White powder. Mp 184-185°C (EtOH). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.10-2.20 (1H, m, 4a-CH); 2.41 (3H, s, CH_3); 3.03 (1H, t, *J* = 11.8) and 3.09 (1H, dd, *J* = 3.0, *J* = 11.8, 4-CH_2); 3.71 (1H, d, *J* = 10.9, 10b-CH); 3.93 (1H, t, *J* = 10.8) and 4.45 (1H, dd, *J* = 3.1, *J* = 10.8, 5-CH_2); 6.54 (1H, s, H-1); 6.83 (1H, dd, *J* = 0.9, *J* = 7.8, H-7); 6.95 (1H, t, *J* = 7.7, H-9); 7.15 (1H, t, *J* = 7.8, H-8); 7.45 (1H, d, *J* = 7.7, H-10); 7.67 (2H, d, *J* = 8.8, H-2,6 Ar); 7.72 (2H, d, *J* = 8.8, H-3,5 Ar). ^{13}C NMR spectrum, δ, ppm: 10.4 (CH_3); 27.9 (C-4); 33.9 (C-10b); 37.2 (C-4a); 69.6 (C-5); 117.0 (C-7); 121.0 (C-8); 122.3 (C-9); 123.9 (C-10a); 125.4 (C-2); 126.5 (C-1); 127.5 (C-2,6 Ar); 128.1 (C-10); 130.2 (C-3,5 Ar); 131.4 (C-5 triazole); 134.8 (C-4 Ar); 135.2 (C-1 Ar); 143.4 (C-4 triazole); 154.4 (C-6a). Mass spectrum (^{35}Cl isotope), *m*/*z*: 397 $[M+H]^+$. Found, %: C 63.88; H 4.45; N 10.49. $C_{21}H_{18}ClN_3OS$. Calculated, %: C 63.71; H 4.58; N 10.61.

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