Phosphorus pentachloride-induced transformation of (1,2,3-thiadiazol-5-yl)hydrazones of acetophenone*

*T. A. Kalinina, P. E. Prokhorova, T. V. Glukhareva, and Yu. Yu. Morzherin**

[a]*Ural Federal University named after the First President of Russia B. N. El´tsin , 19 ul. Mira, 620002 Ekaterinburg, Russian Federation. Fax: +7 (343) 375 4135. E-mail: morzherin@mail.ustu.ru*

Reactions of (4-ethoxycarbonyl-1,2,3-thiadiazol-5-yl)hydrazones of acetophenone with PCl_5 led to transformation of the 1,2,3-thiadiazole moiety involving four atoms of the side chain to give 6-aryl-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]thiadiazine-3-carbonyl chlorides. Reaction of the latter with alcohols and amines gave the corresponding esters and amides.

Key words: 1,2,3-thiadiazoles, 1,2,3-triazoles, Dimroth rearrangement, [1,2,3]triazolo-[5,1-*b*][1,3,4]thiadiazines, hydrazones, phosphorus pentachloride, heterocyclization.

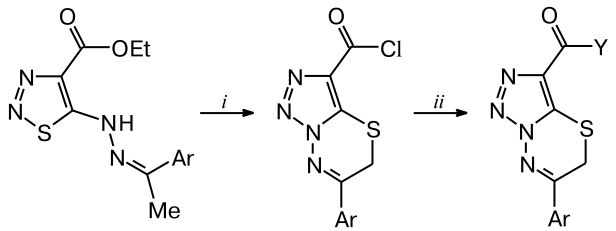
1,2,3-Thiadiazoles are the convenient objects for various rearrangements,[1] *e.g.*, the Dimroth,[2—4] the Cornforth,[5,6] and the L´abbé[7,8] rearrangements. Note that the Dimroth rearrangement of 1,2,3-thiadiazoles into 1,2,3-triazoles proceeds mainly in the basic media, only few examples of this rearrangement in the acidic conditions are documented.[1] It have previously been shown[9] that (1,2,3-thiadiazolyl)hydrazones under the action of PCl_5 underwent transformations to give 1,2,3-triazole derivatives; the directions of the reaction depended on the solvent used. The key step of the process is the Dimroth rearrangement of 1,2,3-thiadiazole ring into 1,2,3-triazole ring.

In continuation of our research on the rearrangements and transformations of 1,2,3-thiadiazoles, we carried out the reaction of (4-ethoxycarbonyl-1,2,3-thiadiazol-5-yl)hydrazones of acetophenone **1** with PCl_5 in benzene (Scheme 1). It was found that this reaction furnished 6-aryl-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]thiadiazine-3-carbonyl chlorides **2**.

It should be noted that this transformations is the third example[10,11] of the transformation of 1,2,3-thiadiazole ring involving four atoms of the side chain. Earlier,[11] similar transformation into 5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]-thiadiazines was observed on treatment of acetophenone (1,2,3-thiadiazol-5-yl)hydrazones with thionyl chloride with the exception that no conversion of ester group into acyl chloride one took place, however, the methylene group of thiadiazine ring was chlorinated.

Treatment of the resulting chlorides **2** with alcohols afforded triazolothiadiazine-3-carboxylates **3a—f** (see Scheme 1). Amides **3g—l** were synthesized without isolation of the intermediate chloride **2** by the addition of the corresponding amines in the reaction mixture.

Scheme 1



1a—c **2a,b** **3a—l**

i. PCl_5, C_6H_6; *ii*. HY.

Compound	Ar	Y
1a	Ph	—
1b	4-MeC_6H_4	—
1c	4-$MeOC_6H_4$	—
2a	Ph	—
2b	4-MeC_6H_4	—
3a	Ph	OEt
3b	4-MeC_6H_4	OEt
3c	Ph	$OCHMe_2$
3d	4-$MeOC_6H_4$	$OCHMe_2$
3e	Ph	OCH_2CHMe_2
3f	4-MeC_6H_4	$O(CH_2)_2CHMe_2$
3g	Ph	$NHCHMe_2$
3h	Ph	$NH(CH_2)_2OH$
3i	Ph	BnNH
3j	4-MeC_6H_4	BnNH
3k	Ph	4-$MeOC_6H_4NH$
3l	4-MeC_6H_4	4-$MeOC_6H_4NH$

In summary, one step procedure towards triazolothiadiazine chlorides *via* five successive reactions, namely, the

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Dimroth rearrangement, chlorination of the methyl group of the acetophenone fragment, ring closure involving nucleophilic nitrogen atom of the hydrazine group, and transformation of the ester group into chlorocarbonyl moiety, was suggested. This procedure serves for the significant increase in the yield of the fused heterocyclic compounds of the type **2**.

Experimental

IR spectra were recorded on a Bruker Alpha spectrometer utilizing attenuated total reflection. ^{1}H and ^{13}C NMR spectra were run on a Bruker AVANCE II 400 instrument (400 and 100 MHz, respectively) relative to Me_4Si (internal standard). Electrospray ionization mass spectra were recorded on a MicrOTOF-Q II instrument (Bruker Daltonics) equipped with six-port lock chamber using direct inlet kd Scientific (flow rate is 180 μL h^{-1}) operating in the positive ion mode, mass range is 50—800 Da. Mass spectrometry was monitored by micrOTOF control 2.3 patch 1 and HyStar 3.2 (Bruker Daltonics) software. Nominal mass resolution is *m/z* 17500. Electron impact mass spectra were obtained on a Varian MAT 3111A mass spectrometer (70 eV, direct inlet).

The starting thiadiazolylhydrazones of acetophenone **1a—c** were synthesized according to the known procedure[12]. Benzene (ZAO EKOS-1) was used as purchased.

6-Aryl-3-chlorocarbonyl-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]-thiadiazines 2 (general procedure). To a suspension of (1,2,3-thiadiazol-5-yl)hydrazone of acetophenone **1**[13] (1.40 mmol) in benzene (20 mL), PCl_5 (0.58 g, 2.8 mmol) was added and the mixture was refluxed for 3 h. Color of the reaction mixture was changed from yellow to orange. Then the reaction mixture was washed with water (3×20 mL), organic layer was dried with Na_2SO_4, the solvent was removed *in vacuo*, and the residue was triturated with EtOH. Brown precipitate that formed was filtered off and dried.

6-Phenyl-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]thiadiazine-3-carbonyl chloride (2a). Yield 0.22 g (56%), m.p. 173 °C. IR, ν/cm^{-1}: 1725 (C=O); 690 (C—Cl). ^{1}H NMR (DMSO-d$_6$), δ: 4.28 (s, 2 H, CH_2); 7.55—7.62 (m, 3 H, ArH); 8.09 (d, 2 H, ArH, *J* = 6.8 Hz). ^{13}C NMR (DMSO-d$_6$), δ: 21.65; 128.28; 128.96; 129.64; 133.04; 133.40; 133.64; 155.86; 161.63. MS (EI, 70 eV), *m/z* (*I*$_{rel}$ (%)): 278 [M(^{35}Cl)]$^+$ (7), 280 [M(^{37}Cl)]$^+$ (2). Found (%): C, 47.42; H, 2.52; N, 20.09; S, 11.53; Cl, 12.70. $C_{11}H_7ClN_4OS$. Calculated (%): C, 47.40; H, 2.53; N, 20.10; S, 11.50; Cl, 12.65.

6-(4-Methylphenyl)-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]thiadiazine-3-carbonyl chloride (2b). Yield 0.25 g (61%), m.p. 175—176 °C. IR, ν/cm^{-1}: 1725 (C=O); 690 (C—Cl). ^{1}H NMR (DMSO-d$_6$), δ: 2.45 (s, 3 H, CH_3); 4.24 (s, 2 H, CH_2); 7.36 (d, 2 H, ArH, *J* = 8.0 Hz); 8.00 (d, 2 H, ArH, *J* = 8.4 Hz). ^{13}C NMR (DMSO-d$_6$), δ: 21.50; 21.58; 128.28; 128.88; 130.23; 130.57; 133.61; 143.45; 155.73; 161.64. MS (ESI), *m/z*: 293.0211 [M(^{35}Cl) + H]$^+$, 295.0180 [M(^{37}Cl) + H]$^+$; calculated: 293.0258 [M(^{35}Cl) + H], 295.0229 [M(^{37}Cl) + H]. Found (%): C, 49.13; H, 3.14; N, 19.09; S, 11.00; Cl, 12.03. $C_{12}H_9ClN_4OS$. Calculated (%): C, 49.23; H, 3.10; N, 19.14; S, 10.95; Cl, 12.11.

6-(4-Methoxyphenyl)-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]-thiadiazine-3-carbonyl chloride (2c). Yield 0.28 g (65%), m.p. 179 °C. ^{1}H NMR (DMSO-d$_6$), δ: 3.89 (s, 3 H, OCH_3); 4.22 (s, 2 H, CH_2); 7.07 (d, 2 H, ArH, *J* = 9.2 Hz); 8.07 (d, 2 H, ArH, *J* = 8.8 Hz). ^{13}C NMR (DMSO-d$_6$), δ: 11.46; 21.34; 56.10; 115.09; 125.36; 128.67; 130.31; 133.58; 155.31; 161.66; 163.25. MS (EI, 70 eV), *m/z* (*I*$_{rel}$ (%)): 308 [M(^{35}Cl)]$^+$ (3), 310 [M(^{37}Cl)]$^+$ (1). Found (%): C, 46.69; H, 2.95; N, 18.14; S, 10.40. $C_{12}H_9ClN_4O_2S$. Calculated (%): C, 46.68; H, 2.94; N, 18.15; S, 10.39.

Alkyl 6-aryl-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]thiadiazine-3-carboxylates 3a—f (general procedure). To a suspension of (1,2,3-thiadiazol-5-yl)hydrazone of acetophenone (1.40 mmol) in benzene (20 mL), PCl_5 (0.58 g, 2.8 mmol) was added and the mixture was refluxed for 3 h. Then the reaction mixture was washed with water (3×20 mL). The organic layer was dried with Na_2SO_4, the solvent was removed *in vacuo*. The corresponding alcohol (10 mL) was added to the residue, and mixture was refluxed for 30 min. After cooling, the precipitate that formed was filtered off and dried.

Ethyl 6-phenyl-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]thiadiazine-3-carboxylate (3a). Yield 0.218 g (54%), m.p. 167 °C. IR, ν/cm^{-1}: 1720 (C=O). ^{1}H NMR (DMSO-d$_6$), δ: 1.40 (t, 3 H, $OCH_2C\underline{H}_3$, *J* = 7.0 Hz); 4.30 (s, 2 H, CH_2); 4.38 (q, 2 H, $OC\underline{H}_2CH_3$, *J* = 7.2 Hz); 7.55—7.63 (m, 3 H, ArH); 8.10 (d, 2 H, ArH, *J* = 7.2 Hz). ^{13}C NMR (DMSO-d$_6$), δ: 14.64; 21.65; 61.46; 128.32; 129.27; 129.66; 132.80; 133.11; 133.37; 156.08; 160.10. MS (ESI), *m/z*: 289.0716 [M + H]$^+$; calculated: 289.0754 [M + H]. Found (%): C, 52.15; H, 4.18; N, 19.44; S, 11.12. $C_{13}H_{12}N_4O_2S$. Calculated (%): C, 52.16; H, 4.20; N, 19.43; S, 11.12.

Ethyl 6-(4-methylphenyl)-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]-thiadiazine-3-carboxylate (3b). Yield 0.228 g (54%), m.p. 168 °C. IR, ν/cm^{-1}: 1720 (C=O). ^{1}H NMR (DMSO-d$_6$), δ: 1.40 (t, 3 H, $OCH_2C\underline{H}_3$, *J* = 7.0 Hz); 4.26 (s, 2 H, CH_2); 4.36 (q, 2 H, $OC\underline{H}_2CH_3$, *J* = 7.0 Hz); 7.37 (d, 2 H, ArH, *J* = 8.0 Hz); 7.99 (d, 2 H, ArH, *J* = 8.0 Hz). ^{13}C NMR (DMSO-d$_6$), δ: 14.64; 21.42; 21.68; 61.43; 128.28; 129.27; 130.66; 133.10; 133.80; 143.47; 156.08; 160.08. MS (ESI), *m/z*: 303.0870 [M + H]$^+$; calculated: 303.0910 [M + H]. Found (%): C, 55.65; H, 4.68; N, 18.59; S, 10.62. $C_{14}H_{14}N_4O_2S$. Calculated (%): C, 55.61; H, 4.67; N, 18.53; S, 10.61.

Isopropyl 6-phenyl-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]thiadiazine-3-carboxylate (3c). Yield 0.157 g (52%), m.p. 174 °C. IR, ν/cm^{-1}: 1720 (C=O). ^{1}H NMR (DMSO-d$_6$), δ: 1.39 (d, 6 H, $OCH(C\underline{H}_3)_2$, *J* = 6.0 Hz); 4.28 (s, 2 H, CH_2); 5.16—5.23 (m, 1 H, $OC\underline{H}(CH_3)_2$); 7.55—7.62 (m, 3 H, ArH); 8.10 (d, 2 H, ArH, *J* = 7.2 Hz). ^{13}C NMR (DMSO-d$_6$), δ: 21.64; 22.19; 69.25; 128.31; 129.12; 129.66; 133.00; 133.10; 133.39; 156.04; 159.61. MS (ESI), *m/z*: 303.0868 [M + H]$^+$; calculated: 303.0910 [M + H]. Found (%): C, 55.62; H, 4.71; N, 18.51; S, 10.60. $C_{14}H_{14}N_4O_2S$. Calculated (%): C, 55.61; H, 4.67; N, 18.53; S, 10.61.

Isopropyl 6-(4-methoxyphenyl)-5*H*-[1,2,3]triazolo[5,1-*b*]-[1,3,4]thiadiazine-3-carboxylate (3d). Yield 0.183 g (55%), m.p. 178—179 °C. IR, ν/cm^{-1}: 1720 (C=O). ^{1}H NMR (DMSO-d$_6$), δ: 1.39 (d, 6 H, $OCH(C\underline{H}_3)_2$, *J* = 6.0 Hz); 3.89 (s, 3 H, CH_3); 4.22 (s, 2 H, CH_2); 5.16—5.22 (m, 1 H, $OC\underline{H}(CH_3)_2$); 7.07 (d, 2 H, ArH, *J* = 8.8 Hz); 8.07 (d, 2 H, ArH, *J* = 8.8 Hz). ^{13}C NMR (DMSO-d$_6$), δ: 21.33; 22.19; 56.11; 69.18; 115.11; 125.30; 128.90; 130.32; 132.95; 155.48; 159.65; 163.30. MS (ESI), *m/z*: 333.0973 [M + H]$^+$; calculated: 333.1016 [M + H]. Found (%): C, 54.22; H, 4.89; N, 16.82; S, 9.68. $C_{15}H_{16}N_4O_3S$. Calculated (%): C, 54.20; H, 4.85; N, 16.86; S, 9.65.

Isobutyl 6-phenyl-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]thiadiazine-3-carboxylate (3e). Yield 0.199 g (45%), m.p. 182 °C. IR, ν/cm^{-1}: 1730 (C=O). ^{1}H NMR (DMSO-d$_6$), δ: 1.04 (d, 6 H, $OCH_2CH(C\underline{H}_3)_2$, J = 6.4 Hz); 2.04—2.11 (m, 1 H, $OCH_2C\underline{H}(CH_3)_2$); 4.10 (d, 2 H, $OC\underline{H}_2CH(CH_3)_2$, J = 6.4 Hz); 4.30 (s, 2 H, CH_2); 7.55—7.62 (m, 3 H, ArH); 8.10 (d, 2 H, ArH, J = 7.2 Hz). ^{13}C NMR (DMSO-d$_6$), δ: 19.36; 21.72; 27.83; 71.05; 128.31; 129.10; 129.66; 132.77; 133.11; 133.36; 156.07; 160.00. MS (ESI), *m/z*: 317.1026 [M + H]$^+$; calculated: 317.1067 [M + H]. Found (%): C, 56.92; H, 5.13; N, 17.72; S, 10.20. $C_{15}H_{16}N_4O_2S$. Calculated (%): C, 56.94; H, 5.10; N, 17.71; S, 10.14.

Isoamyl 6-(4-methylphenyl)-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]-thiadiazine-3-carboxylate (3f). Yield 0.203 g (42%), m.p. 190 °C. IR, ν/cm^{-1}: 1725 (C=O). ^{1}H NMR (DMSO-d$_6$), δ: 0.98 (d, 6 H, $OCH_2CH_2CH(C\underline{H}_3)_2$, J = 6.4 Hz); 1.65 (q, 2 H, $OCH_2C\underline{H}_2CH(CH_3)_2$, J = 6.8 Hz); 1.80—1.86 (m, 1 H, $OCH_2CH_2C\underline{H}(CH_3)_2$); 2.45 (s, 3 H, CH_3); 4.25 (s, 2 H, CH_2); 4.33 (t, 2 H, $OC\underline{H}_2CH_2CH(CH_3)_2$, J = 6.4 Hz); 7.37 (d, 3 H, ArH, J = 8.0 Hz); 7.99 (d, 2 H, ArH, J = 8.4 Hz). ^{13}C NMR (DMSO-d$_6$), δ: 21.58; 22.74; 24.98; 34.17; 37.33; 69.46; 128.30; 129.10; 130.25; 130.50; 132.76; 143.54; 155.93; 160.06. MS (ESI), *m/z*: 345.1327 [M + H]$^+$; calculated: 345.1380 [M + H]. Found (%): C, 59.30; H, 5.82; N, 16.29; S, 9.28. $C_{17}H_{20}N_4O_2S$. Calculated (%): C, 59.28; H, 5.85; N, 16.27; S, 9.31.

6-Aryl-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]thiadiazine-3-carboxamides 3g—l (general procedure). To a suspension of (1,2,3-thiadiazol-5-yl)hydrazone of acetophenone (0.2 g, 0.7 mmol) in benzene (20 mL), PCl_5 (0.29 g, 1.4 mmol) was added, the mixture was refluxed for 3 h, and washed with water (20 mL). Then the corresponding amine (1.4 mmol) was added and reflux was continued for 2 h, the mixture was washed with water (3×20 mL). The organic layer was dried with Na_2SO_4 and the solvent was removed *in vacuo*. The product was precipitated with hexane from chloroform solution. The precipitate that formed was filtered off and dried.

3-(*N*-Isopropylcarbamoyl)-6-phenyl-5*H*-[1,2,3]triazolo-[5,1-*b*][1,3,4]thiadiazine (3g). Yield 0.091 g (43%), m.p. 205 °C. IR, ν/cm^{-1}: 3390 (NH); 1650 (C=O). ^{1}H NMR (DMSO-d$_6$), δ: 1.22 (d, 6 H, 2 CH_3); 4.10—4.14 (m, 1 H, CH); 4.22 (s, 2 H, CH_2); 7.54—7.61 (m, 3 H, ArH); 8.08 (d, 2 H, ArH, J = 8.4 Hz); 8.19 (d, 1 H, NH, J = 8.4 Hz). ^{13}C NMR (DMSO-d$_6$), δ: 21.41; 22.67; 104.21; 126.15; 128.23; 129.63; 132.94; 133.61; 136.30; 155.93; 158.79. MS (ESI), *m/z*: 302.1033 [M + H]$^+$; calculated: 302.1070 [M + H]. Found (%): C, 55.82; H, 5.05; N, 23.23; S, 10.60. $C_{14}H_{15}N_5OS$. Calculated (%): C, 55.80; H, 5.02; N, 23.24; S, 10.64.

3-[*N*-(2-Hydroxyethyl)carbamoyl]-6-(4-methylphenyl)-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]thiadiazine (3h). Yield 0.107 g (48%), m.p. 176 °C. ^{1}H NMR (DMSO-d$_6$), δ: 2.45 (s 3 H, CH_3); 3.35 (q, 2 H, $C\underline{H}_2CH_2OH$, J = 4.2 Hz); 3.53 (t, 2 H, $CH_2C\underline{H}_2OH$, J = 5.2 Hz); 4.19 (s, 2 H, CH_2); 4.58 (br.s, 1 H, OH); 7.36 (d, 2 H, ArH, J = 8.4 Hz); 7.98 (d, 2 H, ArH, J = 8.4 Hz); 8.27 (t, 1 H, NH, J = 6.4 Hz). ^{13}C (DMSO-d$_6$), δ: 21.6; 21.58; 60.11; 99.99; 126.05; 128.23; 130.04; 130.23; 136.07; 143.36; 155.83; 159.79. MS (ESI), *m/z*: 318.0980 [M + H]$^+$; calculated: 318.1019 [M + H]. Found (%): C, 52.93; H, 4.78; N, 22.09; S, 10.10. $C_{14}H_{15}N_5O_2S$. Calculated (%): C, 52.98; H, 4.76; N, 22.07; S, 10.10.

3-(*N*-Benzylcarbamoyl)-6-phenyl-5*H*-[1,2,3]triazolo[5,1-*b*]-[1,3,4]thiadiazine (3i). Yield 0.127 g (52%), m.p. 211 °C. IR, ν/cm^{-1}: 3330 (NH); 1645 (C=O). ^{1}H NMR (DMSO-d$_6$), δ: 4.24 (s, 2 H, CH_2); 4.45 (d, 2 H, $NHC\underline{H}_2$, J = 6.4 Hz); 7.27—7.34 (m, 5 H, $CH_2\underline{Ph}$); 7.55—7.62 (m, 3 H, ArH); 8.08 (d, 2 H, ArH, J = 7.2 Hz); 9.13 (t, 1 H, NH, J = 6.4 Hz). ^{13}C NMR (DMSO-d$_6$), δ: 21.26; 46.73; 127.13; 128.28; 128.79; 129.65; 131.90; 132.80; 133.57; 136.32; 140.77; 143.13; 156.84; 160.27. MS (ESI), *m/z*: 350.1011 [M + H]$^+$; calculated: 350.1070 [M + H]. Found (%): C, 61.85; H, 4.35; N, 20.09; S, 9.17. $C_{18}H_{15}N_5OS$. Calculated (%): C, 61.87; H, 4.33; N, 20.05; S, 9.18.

3-(*N*-Benzylcarbamoyl)-6-(4-methylphenyl)-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]thiadiazine (3j). Yield 0.140 g (55%), m.p. 213 °C. IR, ν/cm^{-1}: 3330 (NH); 1645 (C=O). ^{1}H NMR (DMSO-d$_6$), δ: 2.45 (s, 3 H, CH_3); 4.19 (s, 2 H, CH_2); 4.46 (d, 2 H, $NHC\underline{H}_2$, J = 6.4 Hz); 7.29—7.37 (m, 7 H, ArH); 7.98 (d, 2 H, ArH, J = 8.0 Hz); 9.07 (t, 1 H, NH, J = 5.6 Hz). ^{13}C NMR (DMSO-d$_6$), δ: 21.26; 21.58; 42.43; 126.31; 127.24; 127.81; 128.24; 128.72; 130.23; 130.72; 135.99; 139.97; 143.37; 155.85; 159.77. MS (ESI), *m/z*: 364.1172 [M + H]$^+$; calculated: 364.1227 [M + H]. Found (%): C, 62.80; H, 4.71; N, 19.22; S, 8.87. $C_{19}H_{17}N_5OS$. Calculated (%): C, 62.79; H, 4.71; N, 19.27; S, 8.82.

3-[*N*-(4-Methoxyphenyl)carbamoyl]-6-phenyl-5*H*-[1,2,3]-triazolo[5,1-*b*][1,3,4]thiadiazine (3k). Yield 0.089 g (46%), m.p. 211 °C. IR, ν/cm^{-1}: 3370 (NH); 1670 (C=O). ^{1}H NMR (DMSO-d$_6$), δ: 3.77 (s, 3 H, OCH_3); 4.26 (s, 2 H, CH_2); 6.84 (d, 2 H, ArH, J = 8.8 Hz); 7.55—7.64 (m, 3 H, ArH); 7.74 (d, 2 H, ArH, J = 8.8 Hz); 8.10 (d, 2 H, ArH, J = 6.8 Hz); 10.33 (s, 1 H, NH). ^{13}C NMR (DMSO-d$_6$), δ: 21.47; 55.65; 114.21; 122.38; 127.13; 128.28; 128.79; 129.65; 131.90; 132.80; 133.02; 133.57; 136.32; 156.08; 156.15; 158.01. Found (%): C, 59.15; H, 4.13; N, 19.17; S, 8.77. $C_{18}H_{15}N_5O_2S$. Calculated (%): C, 59.16; H, 4.14; N, 19.17; S, 8.78.

6-(4-Methylphenyl)-3-[*N*-(4-methoxyphenyl)carbamoyl]-5*H*-[1,2,3]triazolo[5,1-*b*][1,3,4]thiadiazine (3l). Yield 0.122 g (48%), m.p. 213—214 °C. IR, ν/cm^{-1}: 3370 (NH); 1670 (C=O). ^{1}H NMR (DMSO-d$_6$), δ: 2.45 (s, 3 H, CH_3); 3.77 (s, 3 H, OCH_3); 4.23 (s, 2 H, CH_2); 6.83 (d, 2 H, ArH, J = 9.2 Hz); 7.37 (d, 2 H, ArH, J = 8.0 Hz); 7.74 (d, 2 H, ArH, J = 9.2 Hz); 8.00 (d, 2 H, ArH, J = 8.4 Hz); 10.31 (s, 1 H, NH). ^{13}C NMR (DMSO-d$_6$), δ: 21.31; 21.58; 55.64; 114.20; 122.36; 127.03; 128.26; 130.24; 130.90; 131.91; 136.30; 143.43; 155.93; 156.14; 158.02. MS (ESI), *m/z*: 380.1111 [M + H]$^+$; calculated: 380.1176 [M + H]. Found (%): C, 60.16; H, 4.53; N, 18.45; S, 8.43. $C_{19}H_{17}N_5O_2S$. Calculated (%): C, 60.14; H, 4.52; N, 18.46; S, 8.45.

References

1. Yu. Yu. Morzherin, T. V. Glukhareva, V. A. Bakulev, *Khim. Geterotsikl. Soedin.*, 2003, 803 [*Chem. Heterocycl. Compd.* (*Engl. Transl.*), 2003, **39**, 679].
2. T. Kindt-Larsen, C. Pedersen, *Acta Chem. Scand.*, 1985, **16**, 3871.
3. G. L´abbé, *J. Heterocycl. Chem.*, 1984, **21**, 627.
4. V. A. Bakulev, Yu. M. Shafran, V. A. Shevyrin, M. Yu. Kolobov, *Khim. Geterotsikl. Soedin.*, 1993, 840 [*Chem. Heterocycl. Compd.* (*Engl. Transl.*), 1993, **29**, 724].
5. G. L´abbé, E. Vanderstede, W. Dehaen, S. Toppet, *J. Chem. Soc., Perkin Trans. 1*, 1993, 1719.

6. V. A. Bakulev, E. V. Tarasov, Yu. Yu. Morzherin, S. Toppet, V. Dehaen, *Bull. Soc. Chim. Belg.*, 1997, **106**, 643.
7. G. L´abbé, *Bull. Soc. Chim. Belg.*, 1990, **99**, 281.
8. Yu. Yu. Morzherin, V. A. Bakulev, E. F. Dankova, V. S. Mokrushin, *Khim. Geterotsikl. Soedin.*, 1994, 548 [*Chem. Heterocycl. Compd.* (*Engl. Transl.*), 1994, **30**, 483].
9. T. V. Glukhareva, L. V. Dyudya, Yu. Yu. Morzherin, A. V. Tkachev, V. A. Bakulev, *Khim. Geterotsikl. Soedin.*, 2003, 134 [*Chem. Heterocycl. Compd.* (*Engl. Transl.*), 2003, **39**, 126].
10. P. E. Kropotina, L. V. Dyudya, T. V. Glukhareva, Yu. Yu. Morzherin, V. A. Bakulev, K. Van Hecke, L. Van Meervelt, W. Dehaen, *Mendeleev Commun.*, 2006, **16**, 76.
11. T. V. Glukhareva, Y. Y. Morzherin, I. N. Slepukhina, V. S. Mokrushin, A. V. Tkachev, V. A. Bakulev, *Mendeleev Commun.*, 2000, **10**, 19.
12. Yu. Yu. Morzherin, E. V. Tarasov, V. A. Bakulev, *Khim. Geterotsikl. Soedin.*, 1994, 554 [*Chem. Heterocycl. Compd.* (*Engl. Transl.*), 1994, **30**, 489].
13. G. L´abbé, E. Vanderstede, *J. Heterocycl. Chem.*, 1989, **26**, 1811.

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