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Chemistry of Heterocyclic Compounds **2019**, *55*(9), 823–826

Dioxothietanylation of heterocycles

2*. Imidazoles and benzimidazoles

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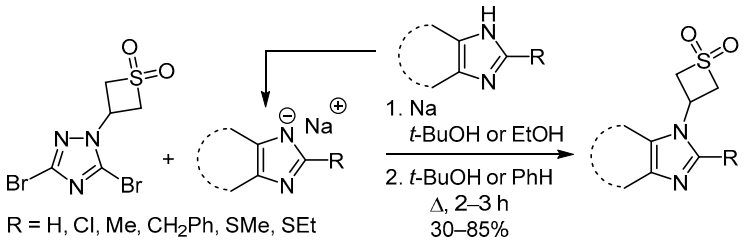


1-(1,1-Dioxidothietan-3-yl)-1*H*-imidazoles and 1-(1,1-dioxidothietan-3-yl)-1*H*-benzimidazoles were synthesized in the reaction of 3,5-dibromo-1-(1,1-dioxidothietan-3-yl)-1*H*-1,2,4-triazole with the sodium salts of imidazoles and benzimidazoles. The reaction involves addition of azoles to thiete 1,1-dioxide formed *in situ*, which acts as a Michael acceptor. The effect of pK_a values of imidazoles and benzimidazoles on their reactivity is shown.

Keywords: benzimidazole, imidazole, thietane 1,1-dioxide, 1,2,4-triazole, aza-Michael reaction, dioxothietanylation.

One of the common methods for the synthesis of compounds containing the 1,1-dioxidothietane ring is the oxidation of the corresponding thietanes.[2] This method requires preliminary introduction of a thietane ring into the initial molecule and has a number of drawbacks. For example, the thietanylation of compounds is often accompanied by the formation of polymers and leads to low yields of thietanyl derivatives,[3] and the oxidation of the latter leads to the formation of byproducts, which negatively affects the purity and reduces the yields of the target thietane 1,1-dioxides.

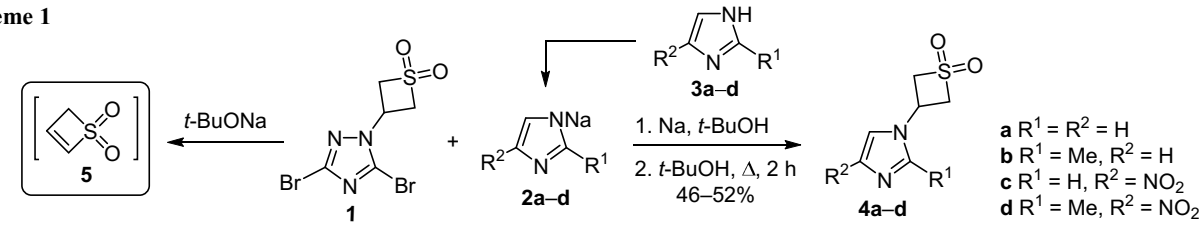
Previously, we developed a new method for the synthesis of 3-alkoxy-[4] and 3-aryloxythietane 1,1-dioxides[5] and 3,5-disubstituted 1-(1,1-dioxidothietan-3-yl)-1*H*-1,2,4-triazoles,[6] based on the use of 3,5-dibromo-1-(1,1-dioxidothietan-3-yl)-1*H*-1,2,4-triazole as the dioxothietanylating agent. In continuation of these studies, the reactions of this 1,2,4-triazole with imidazoles and benzimidazoles containing electron-withdrawing (Cl, Br, I, NO_2, CO_2Me, SO_2Me) and electron-donating (Ph, Me, CH_2Ph, SAlk) substituents and significantly differing in nucleophilicity and acidity were studied. An increase in the acidity of the azole forming the dioxothietanylating agent and a decrease in its nucleophilicity contribute to the retro-Michael reaction with the formation of thiete 1,1-dioxide and 3,5-dibromo-1*H*-1,2,4-triazole (pK_a 5.17).[7]

The reaction of 3,5-dibromo-1-(1,1-dioxidothietan-3-yl)-1*H*-1,2,4-triazole (**1**) with substituted imidazoles was carried out by heating under reflux sodium salts of imidazoles **2a**–**d**, obtained *in situ* from corresponding imidazoles **3a**–**d**, in *t*-BuOH. The choice of *t*-BuOH as the solvent was due to its low ability to compete with nucleophiles as a Michael donor because of steric hindrance. As a result, 1-(1,1-dioxidothietan-3-yl)-1*H*-imidazoles **4a**–**d** were isolated in moderate yields. The proposed reaction mechanism involves the generation of thiete 1,1-dioxide (**5**), which is involved in the aza-Michael reaction (Scheme 1).

It has been established that imidazoles with NH acidity in the pK_a range 9.30–15.1 (imidazole (**3a**) pK_a 14.4, 2-methylimidazole (**3b**) pK_a 15.1, 4(5)-nitroimidazole (**3c**) pK_a 9.30, and 2-methyl-4(5)-nitroimidazole (**3d**) pK_a 9.75) enter the reaction.[7] The use of asymmetric 4(5)-nitro- and 2-methyl-4(5)-nitroimidazoles **3c**,**d** can lead to the formation of two isomers (Fig. 1). The ^1H–^{13}C HMBC and HSQC spectra of compounds **4c**,**d**, as well as the ^1H–^{15}N HMBC spectrum of compound **4c**, indicate that only 4-nitroimidazoles **4c**,**d** are formed in the studied reaction, which is consistent with published data.[8]

*For Communication 1, see[1].

Scheme 1



With the aid of a $^{1}H–^{15}N$ HMBC experiment for compound **4c**, a correlation was found between the signals of the protons H-2,5 of the imidazole ring and protons of the 2,4-CH_2 group of the 1,1-dioxidothietane ring with the signals of the imidazole N-1,3 atoms (Fig. 1). The most informative are the cross peaks H-2/N-1, H-2/N-3, and H-5/N-1. The absence in the spectrum of the cross peak H-4/N-3 indicates the formation of 1-(1,1-dioxidothietan-3-yl)-4-nitro-1*H*-imidazole (**4c**). According to published data,[9] in the ^{1}H NMR spectra of 1,4-disubstituted imidazoles, the value of the spin-spin coupling constant (SSCC) $J_{H\text{-}2,H\text{-}5}$ is usually 1.1–1.5 Hz and in the spectra of 1,5-disubstituted imidazoles, the values of $J_{H\text{-}2,H\text{-}4}$ are in the range of 0.9–1.0 Hz. The SSCC values of 1.4 and 1.5 Hz obtained for the protons of the product of the reaction of 4(5)-nitroimidazole (**3c**) with 1-(1,1-dioxidothietan-3-yl)-1*H*-1,2,4-triazole **1** also confirm the proposed structure of compound **4c**. The $^{1}H–^{13}C$ HMBC spectra of compounds **4c**,**d** showed a correlation of the signals of the H-2.5 protons of the imidazole ring with the signals of the C-3 atoms of the 1,1-dioxidothietane ring and imidazole C-2,4 signals (Fig. 2).

In the case of 2,4,5-tribromoimidazole (pK_a 6.9) and 2,4,5-triiodimidazole (pK_a 8.0),[10] both possessing high acidity and low nucleophilicity, *N*-dioxothietanylation products are not formed. Dimethyl 4,5-imidazole dicarboxylate (pK_a 9.26)[11] and 4,5-diphenylimidazole (pK_a 12.80)[7] were also inactive in this reaction. This is apparently due to steric hindrances introduced by substituents at positions 4 and 5 of the imidazole ring.

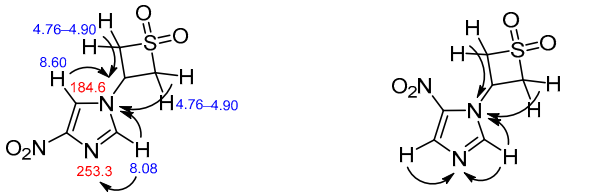


Figure 1. Major correlations in $^{1}H–^{15}N$ HMBC spectrum for compound **4c** and the expected correlations for 1-(1,1-dioxidothietan-3-yl)-5-nitro-1*H*-imidazole.

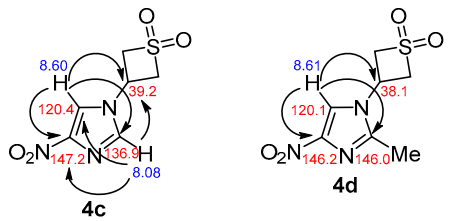
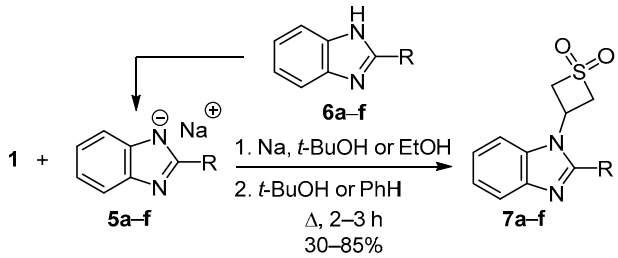


Figure 2. Major correlations in $^{1}H–^{13}C$ HMBC spectra for compounds **4c**,**d**.

A similar reaction of 3,5-dibromo-1-(1,1-dioxidothietan-3-yl)-1*H*-1,2,4-triazole (**1**) with sodium salts of benzimidazoles **5a–f**, obtained *in situ* from the corresponding benzimidazoles **6a–f**, was carried out in *t*-BuOH (for compounds **7a**,**b**,**d**,**e**) or in PhH (for compounds **7c**,**f**) with the formation of 1-(1,1-dioxidothietan-3-yl)-1*H*-benzimidazoles **7a–f** with 30–85% yields (Scheme 2).

Scheme 2



It was found that benzimidazoles with NH acidity in the pK_a range of 9.60–13.80 (benzimidazole (**6a**) pK_a 12.86, 2-chlorobenzimidazole (**6b**) pK_a 9.60, 2-methylbenzimidazole (**6c**) pK_a 13.18, 2-benzylbenzimidazole (**6d**) pK_a 12.7, 2-(methylsulfanyl)benzimidazole (**6e**) pK_a 11.8,[7] 2-(ethylsulfanyl)benzimidazole (**6f**) pK_a 11.67[11]) take part in the reaction. In the case of 2-(methylsulfonyl)benzimidazole (pK_a 6.59),[11] the corresponding product could not be obtained. Also, 2-isopropoxybenzimidazole (pK_a 13.28) and 2-(*N*-benzylamino)benzimidazole (pK_a 15.44)[11] did not enter the reaction, which is apparently explained by steric barriers introduced by substituents at position 2 of benzimidazole.

The IR spectra of compounds **4b–d** and **7b–f** contain intense absorption bands of stretching vibrations of the S=O bonds of the sulfonyl group in the ranges 1313–1346 and 1134–1147 cm^{-1}, which confirms the presence of the 1,1-dioxidothietane ring. Compound **7b** does not lower the melting temperature of the sample when mixed with the compound obtained by the known method,[12] and their IR spectra perfectly match. In the ^{1}H NMR spectra of compounds **4b–d** and **7b–f**, proton signals of the 2,4-CH_2 and 3-CH groups of the 1,1-dioxidothietane ring are observed in the ranges of 4.47–5.04 and 5.12–5.85 ppm, respectively. Signals of the aromatic protons in the downfield region confirm the presence of azole rings. The ^{13}C NMR spectra of compounds **4b–d** and **7b–f** contain the signals of the C-2,4 and C-3 atoms of the 1,1-dioxidothietane ring in the ranges of 70.2–72.0 and 35.1–39.2 ppm, respectively.

To conclude, an effective method for a one-step synthesis of 1-(1,1-dioxidothietan-3-yl)-1*H*-imidazoles and 1-(1,1-dioxidothietan-3-yl)-1*H*-benzimidazoles based on the reactions of 3,5-dibromo-1-(1,1-dioxidothietan-3-yl)-1*H*-1,2,4-triazole with the sodium salts of imidazoles and benzimidazoles. It was established that imidazoles and benzimidazoles with NH acidity in the pK_a ranges of 9.30–15.1 and 9.60–13.80, respectively, enter the reaction.

Experimental

IR spectra were registered on an Infralum FT-02 FT-IR spectrometer in KBr pellets. ^{1}H and ^{13}C NMR spectra (500 and 125 MHz, respectively), ^{1}H–^{13}C HMBC and HSQC (compounds **4c**,**d**) and ^{1}H–^{15}N HMBC (compound **4c**) spectra were acquired on a Bruker Avance 500 spectrometer in DMSO-d_6. The residual solvent signals (2.50 ppm for ^{1}H nuclei, 39.5 ppm for ^{13}C nuclei) or TMS (for compound **4c**) were used as internal standard. ^{13}C NMR spectra were additionally registered in DEPT mode. Mass spectra were recorded on an Agilent 5973A GC/MSD mass spectrometer, 6890B series, EI ionization (70 eV), scan range 15–600 Da, scan rate 2.5 scan/s. Temperature program of the chromatography column: starting temperature 80°C, 2 min isotherm, ramp to 260°C at 20°C/min, injector temperature 250°C. Elemental analysis was performed on a Hekatech Euro3000 Elemental analyzer. Melting points were determined on a Stuart SMP30 apparatus. Monitoring of the reaction progress and assessment of the purity of synthesized compounds were done by TLC on Sorbfil P-A-UV plates, visualization with UV or in an iodine chamber.

3,5-Dibromo-1-(1,1-dioxidothietan-3-yl)-1*H*-1,2,4-triazole (**1**) was synthesized according to a literature method.[4] Synthesis according to the proposed method and analytical data for 1-(1,1-dioxidothietan-3-yl)-1*H*-imidazole (**4a**) and 1-(1,1-dioxidothietan-3-yl)-1*H*-benzimidazole (**7a**) were published earlier.[13]

Synthesis of 1-(1,1-dioxidothietan-3-yl)-1*H*-imidazoles 4b–d and 1-(1,1-dioxidothietan-3-yl)-1*H*-benzimidazoles 7b,d,e (General method). Metallic Na (76 mg, 3.3 mmol) was added to *t*-BuOH (30 ml), and the mixture was heated under reflux until effervescence ceased. Then azole **3b**–**d** or **6b**,**d**,**e** (3 mmol) and 3,5-dibromo-1-(1,1-dioxidothietan-3-yl)-1*H*-1,2,4-triazole (**1**) (0.99 g, 3 mmol) were added. The reaction mixture was heated under reflux for 2 h (for compounds **4b**–**d**, **7d**,**e**) or 3 h (for compound **7b**). The solvent was evaporated under reduced pressure, and H_2O (15 ml) was added to the residue. The precipitate was filtered off, dried at 60°C, and recrystallized from a suitable solvent.

1-(1,1-Dioxidothietan-3-yl)-2-methyl-1*H*-imidazole (4b). After evaporation of the solvent, the residue was washed with $CHCl_3$ (20 ml). Yield 0.27 g (49%), white powder, mp 188–190°C (PhH–hexane, 1:5). IR spectrum, ν, cm^{-1}: 1430 (C=N, C=C), 1313 (S=O), 1225, 1144 (S=O). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.27 (3H, s, CH_3); 4.47–4.55 (2H, m) and 4.76–4.84 (2H, m, 2,4-CH_2); 5.12–5.20 (1H, m, 3-CH); 6.80 (1H, d, *J* = 1.0, H-4); 7.40 (1H, d, *J* = 1.1, H-5). ^{13}C NMR spectrum, δ, ppm: 13.4 (CH_3); 36.2 (CH); 72.0 (2CH_2); 116.8 (C Ar); 127.6 (C Ar); 145.1 (C Ar). Found, %: C 45.09; H 5.48; N 15.09. $C_7H_{10}N_2O_2S$. Calculated, %: C 45.15; H 5.41; N 15.04.

1-(1,1-Dioxidothietan-3-yl)-4-nitro-1*H*-imidazole (4c). After filtration, the precipitate was washed with Me_2CO (15 ml). Yield 0.30 g (46%), white powder, mp 235–237°C (EtOH). IR spectrum, ν, cm^{-1}: 1549 (NO_2), 1495 (C=N, C=C), 1388, 1346 (S=O), 1224, 1146 (S=O). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.76–4.90 (4H, m, 2,4-CH_2); 5.34–5.41 (1H, m, 3-CH); 8.08 (1H, d, *J* = 1.4, H-2); 8.60 (1H, d, *J* = 1.5, H-5). ^{13}C NMR spectrum, δ, ppm: 39.2 (CH); 71.0 (2CH_2); 120.4 (C-5); 136.9 (C-2); 147.2 (C-4). Found, %: C 33.23; H 3.31; N 19.54. $C_6H_7N_3O_4S$. Calculated, %: C 33.18; H 3.25; N 19.35.

1-(1,1-Dioxidothietan-3-yl)-2-methyl-4-nitro-1*H*-imidazole (4d). Yield 0.36 g (52%), white powder, mp 188–190°C (EtOH). IR spectrum, ν, cm^{-1}: 1533 (NO_2), 1495 (C=N, C=C), 1394, 1317 (S=O), 1222, 1147 (S=O). ^{1}H NMR spectrum, δ, ppm: 2.35 (3H, s, CH_3); 4.77–4.88 (4H, m, 2,4-CH_2); 5.22–5.29 (1H, m, 3-CH); 8.61 (1H, s, H-5). ^{13}C NMR spectrum, δ, ppm: 13.5 (CH_3); 38.1 (CH); 71.2 (2CH_2); 120.1 (C-5); 146.0 (C-2); 146.2 (C-4). Found, %: C 36.26; H 3.84; N 18.11. $C_7H_9N_3O_4S$. Calculated, %: C 36.36; H 3.92; N 18.17.

2-Chloro-1-(1,1-dioxidothietan-3-yl)-1*H*-benzimidazole (7b). Yield 0.45 g (58%), white powder, mp 187–189°C (*n*-BuOH). IR spectrum, ν, cm^{-1}: 1479 (C=N, C=C), 1327 (S=O), 1222, 1143 (S=O). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.84–5.04 (4H, m, 2,4-CH_2); 5.75–5.85 (1H, m, 3-CH); 7.27–7.43 (2H, m, H-5,6); 7.66 (1H, d, *J* = 8.0, H-7); 7.91 (1H, d, *J* = 8.2, H-4). ^{13}C NMR spectrum, δ, ppm: 36.0 (CH); 70.6 (2CH_2); 111.6 (C Ar); 119.8 (C Ar); 123.5 (C Ar); 124.0 (C Ar); 133.6 (C Ar); 140.6 (C Ar); 141.8 (C Ar). Mass spectrum, *m*/*z* (I_{rel}, %): 258 $[M]^+$ (12), 256 $[M]^+$ (32), 180 (33), 178 (100). Found, %: C 46.70; H 3.48; N 11.11. $C_{10}H_9ClN_2O_2S$. Calculated, %: C 46.79; H 3.53; N 10.91.

2-Benzyl-1-(1,1-dioxidothietan-3-yl)-1*H*-benzimidazole (7d). After filtration, the precipitate was washed with *n*-BuOH (20 ml). Yield 0.30 g (32%), white powder, mp 195–197°C (EtOH). IR spectrum, ν, cm^{-1}: 1458 (C=N, C=C), 1332 (S=O), 1213, 1134 (S=O). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 4.31 (2H, s, CH_2Ph); 4.48–4.56 (2H, m) and 4.83–4.91 (2H, m, 2,4-CH_2); 5.67–5.76 (1H, m, 3-CH); 7.20–7.34 (7H, m, H-5,6, H Ph); 7.65 (1H, d, *J* = 7.9, H-7); 7.84 (1H, d, *J* = 8.0, H-4). ^{13}C NMR spectrum, δ, ppm: 33.6 ($\underline{C}H_2$Ph); 35.2 (CH); 70.2 (2CH_2); 111.4 (C Ar); 120.1 (C Ar); 122.6 (C Ar); 123.0 (C Ar); 127.2 (C Ph); 129.0 (2C Ph); 129.1 (2C Ph); 132.7 (C Ar); 137.1 (C Ph); 143.5 (C Ar); 154.8 (C Ar). Found, %: C 65.11; H 5.23; N 8.91. $C_{17}H_{16}N_2O_2S$. Calculated, %: C 65.36; H 5.16; N 8.97.

1-(1,1-Dioxidothietan-3-yl)-2-(methylsulfanyl)-1*H*-benzimidazole (7e). Yield 0.66 g (82%), white powder, mp 201–202°C (*i*-BuOH). IR spectrum, ν, cm^{-1}: 1447 (C=N, C=C), 1323 (S=O), 1275, 1142 (S=O). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.83 (3H, s, CH_3); 4.84–5.00 (4H, m, 2,4-CH_2); 5.52–5.62 (1H, m, 3-CH); 7.18–7.29 (2H, m, H-5,6); 7.60 (1H, d, *J* = 7.7, H-7); 7.82 (1H, d, *J* = 7.8, H-4). ^{13}C NMR spectrum, δ, ppm: 15.4 (CH_3); 35.7 (CH); 70.3 (2CH_2);

110.9 (C Ar); 118.8 (C Ar); 122.5 (C Ar); 122.6 (C Ar); 134.5 (C Ar); 144.0 (C Ar); 153.4 (C Ar). Mass spectrum, *m*/*z* (I_{rel}, %): 268 $[M]^+$ (100), 253 $[M–CH_3]^+$ (18), 189 (99), 175 (50), 171 (52), 157 (50), 131 (34). Found, %: C 49.47; H 4.50; N 10.32. $C_{11}H_{12}N_2O_2S_2$. Calculated, %: C 49.23; H 4.51; N 10.44.

Synthesis of 1-(1,1-dioxidothietan-3-yl)-1*H*-benzimidazoles 7c,f (General method). Metallic Na (76 mg, 3.3 mmol) was added to anhydrous EtOH (20 ml), and the mixture was heated under reflux until effervescence ceased. Benzimidazole **6c**,**f**, (3 mmol) was then added, and the mixture was heated under reflux for 5 min. The solvent was evaporated under reduced pressure. Then PhH (30 ml) and 3,5-dibromo-1-(1,1-dioxidothietan-3-yl)-1*H*-1,2,4-triazole (**1**) (0.99 g, 3 mmol) were added to the residue. The reaction mixture was heated under reflux for 2 h. The solvent was evaporated under reduced pressure, and H_2O (20 ml) was added to the residue. The precipitate was filtered off, dried at 60°C, and recrystallized from EtOH.

1-(1,1-Dioxidothietan-3-yl)-2-methyl-1*H*-benzimidazole (7c). Yield 0.21 g (30%), white powder, mp 227–228°C. IR spectrum, ν, cm^{-1}: 1466 (C=N, C=C), 1327 (S=O), 1229, 1135 (S=O). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 2.59 (3H, s, CH_3); 4.75–5.00 (4H, m, 2,4-CH_2); 5.58–5.70 (1H, m, 3-CH); 7.15–7.30 (2H, m, H-5,6); 7.58 (1H, d, *J* = 7.8, H-7); 7.80 (1H, d, *J* = 7.9, H-4). ^{13}C NMR spectrum, δ, ppm: 14.7 (CH_3); 35.1 (CH); 70.3 ($2CH_2$); 111.0 (C Ar); 120.0 (C Ar); 122.3 (C Ar); 122.6 (C Ar); 132.9 (C Ar); 143.4 (C Ar); 153.0 (C Ar). Mass spectrum, *m*/*z* (I_{rel}, %): 236 $[M]^+$ (36), 158 (100), 157 (27). Found, %: C 55.98; H 5.01; N 11.73. $C_{11}H_{12}N_2O_2S$. Calculated, %: C 55.92; H 5.12; N 11.86.

1-(1,1-Dioxidothietan-3-yl)-2-(ethylsulfanyl)-1*H*-benzimidazole (7f). Yield 0.72 g (85%), white powder, mp 134–135°C. IR spectrum, ν, cm^{-1}: 1450 (C=N, C=C), 1319 (S=O), 1276, 1143 (S=O). ^{1}H NMR spectrum, δ, ppm (*J*, Hz): 1.38 (3H, t, *J* = 7.3, $SCH_2C\underline{H}_3$); 3.32 (2H, q, *J* = 7.3, $SC\underline{H}_2CH_3$); 4.84–4.99 (4H, m, 2,4-CH_2); 5.54–5.62 (1H, m, 3-CH); 7.19–7.27 (2H, m, H-5,6); 7.61 (1H, d, *J* = 7.8, H-7); 7.83 (1H, d, *J* = 7.7, H-4). ^{13}C NMR spectrum, δ, ppm: 15.3 ($SCH_2\underline{C}H_3$); 27.3 ($S\underline{C}H_2CH_3$); 35.6 (CH); 70.3 ($2CH_2$); 111.0 (C Ar); 118.9 (C Ar); 122.6 (2C Ar); 134.2 (C Ar); 144.0 (C Ar); 152.4 (C Ar). Found, %: C 51.11; H 4.92; N 9.98. $C_{12}H_{14}N_2O_2S_2$. Calculated, %: C 51.04; H 5.00; N 9.92.

Supplementary information file containing ^{1}H and ^{13}C NMR spectra for compounds **4b**–**d** and **7b**–**f**, ^{1}H–^{13}C HMBC and HSQC 2D NMR spectra for compounds **4c**,**d**, and ^{1}H–^{15}N HMBC spectrum for compound **4c**, as well as mass spectra for compounds **7b**,**c**,**e** is available at the journal website at http://link.springer.com/journal/10593.

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