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Reactions of 1-Alkyl-2-chloro-1*H*-indole-3-carbaldehyde with 4-Amino-5-alkyl(aryl)-4*H*-triazole-3-thioles

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Abstract—The reaction of 1-alkyl-2-chloro-1*H*-indole-3-carbaldehydes with 4-amino-5-alkyl(aryl)-4*H*-1,2,4-triazole-3-thiols afforded new heterocyclic compounds, namely triazolo(thiadiazepino)indoles. Structure of the latter was established by single crystal X-ray diffraction method. This heterocyclic system is formed upon cyclization of the intermediate 5-alkyl-4-[indol-3-yl(methylideneamino)]-4*H*-1,2,4-triazole-3-thiols.

Keywords: chloroindolecarbaldehyde, aminotriazolethiol, cyclization, triazolo(thiadiazepino)indole

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The indole system is unique among natural compounds in the distribution and importance of the biological role. Indole derivatives include hormones, neurotransmitters, phytoalexins, proteins, alkaloids, and plant pigments. Based on indole, plant protection products, electronic materials, dyes [1,2], as well as antibacterial [3], antiviral [4], antifungal [5], anti-inflammatory [6], and anti-tuberculosis drugs [7] have been created. Sulfur-containing polycyclic derivatives of indole are widely used in pharmaceuticals and materials science [8–10].

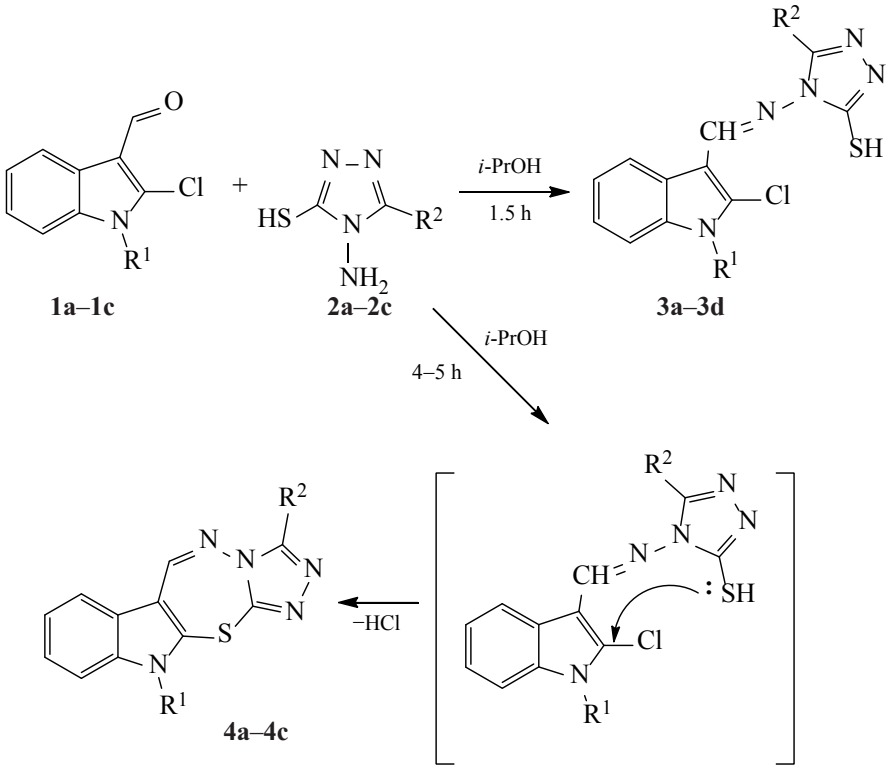
In order to synthesize new sulfur-containing indole polyheterocycles, we studied the reactions of 1-alkyl-2-chloro-1*H*-indole-3-carbaldehydes **1** with 5-alkyl-4-amino-4*H*-1,2,4-triazole-3-thiols 2 (Scheme 1).

4-[Indol-3-yl(methylidenamino)]-4*H*-1,2,4-triazole-3-thiols **3a**–**3d** were obtained with a yield of 44–73% (Scheme 1). Composition and structure of the obtained compounds were confirmed by elemental analysis, IR, ^{1}H and ^{13}C NMR spectroscopy methods.

The IR spectra of compounds **3a**–**3d** contain stretching vibration bands of C=N bonds in 1574–1601 cm^{-1} region and C=C bonds at 1463–1523 cm^{-1}. The ^{1}H NMR spectra (DMSO-d_6) of thiols **3a**–**3d** contain signals of the SH group in the range of 13.43–14.07 ppm, as well as singlets of the CH=N group at 9.72–10.31 ppm. Aromatic protons are recorded in the region of 7.23–8.32 ppm. In addition to these signals, signals of aliphatic protons of substituents in the strong field region are also recorded. The ^{13}C NMR spectra of thiols **3a**–**3d** contain signals of all the structural fragments.

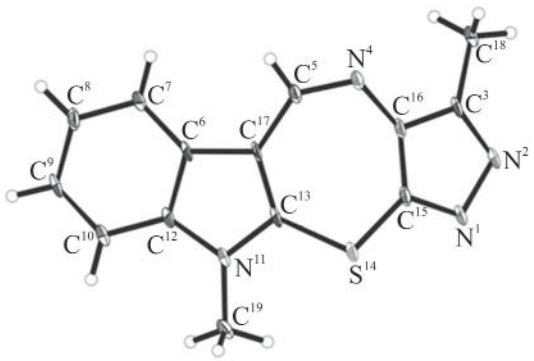
When performing the reaction for a longer time (4–5 h), we were able to obtain triazolo(thiodiazepino)indoles **4a**–**4c**, which are likely to result from cyclization of intermediates **3a**–**3d** with the elimination of the hydrogen chloride molecule. The triazolo(thiodiazepino)indole system has not been previously described. Structure of polyheterocycles **4a**–**4c** was proved using IR, ^{1}H and ^{13}C NMR spectroscopy, mass spectrometry, and single crystal X-ray diffraction data for compound **4a**. In the IR spectra of compounds **4a**–**4c**, absorption bands of the C=N bond stretching were registered at 1586–1623 cm^{-1}, as well as bands at 1456–1504 cm^{-1} due to vibrations of the C=C bonds. The strongest peak of the molecular ion (*m*/z 269) is present in the mass spectrum of compound **4a**. In the ^{1}H NMR spectra of compounds **4a**–**4c**, there are no SH group signals in the low-field region (12–14 ppm). The proton signals of the CH=N group are shifted to the high-field region (compared to signals of the CH=N group in the spectra of thiols **3a**–**3d**) and are recorded as singlets in the region of 8.77–9.16 ppm. The ^{1}H NMR spectra contain signals of aromatic protons in the range of 6.73–7.83 ppm and aliphatic protons at 1.40–3.87 ppm.

Scheme 1.



R^1 = Me (**1a**, **3a**, **4a**, **4b**), Et (**1b**, **3b**, **4c**), $CH_2CH(OEt)_2$ (**1c**, **3c**, **3d**);
R^2 = Me (**2a**, **3a**–**3c**, **4a**), Et (**2b**, **4b**, **4c**), 4-MeC_6H_4 (**2c**, **3d**).

Structure of compound **4a** was confirmed by single X-ray diffraction analysis (see figure). The nearest intermolecular contacts of two neighboring molecules in the crystal are 2.55 ($N^1\cdots H^{5'}$) and 2.57 Å ($N^1\cdots H^{7'}$). The left side of the molecule, including C^5, C^{17}, C^{13}, N^{11}, and C^{19} atoms, is located in the same plane (with an accuracy of 0.013, maximum deviation 0.024 Å); the sulfur atom S^{14} is out-off-plane by 0.29 Å. The seven-membered ring can be characterized by three planes: $S^{14}C^{13}C^{17}C^5$, $C^5N^4S^{14}$, and $S^{14}C^{15}N^{16}N^4$. In the latter, C^3, C^{18}, N^1, N^2 atoms are located with an accuracy of 0.03 Å. The two outside planes deviate from the central one at angles of 19.5 and 25.7°. The main bond lengths and bond angles in the molecule of **4a** are given in the table.



General view of the molecule of compound **4a** in the crystal (CCDC 1909252).

In conclusion, the reaction of 2-chloroindole-3-carbaldehydes with 4-aminotriazole-3-thiols when boiling in propan-2-ol leads to the formation of indole-1,2,4-triazole-3-thiols, which subsequently cyclize to triazole(thiadiazepino)indoles.

EXPERIMENTAL

The starting indole-3-carbaldehydes **1a**–**1c** were obtained according to the procedure reported in [11]. Aminotriazolethiols **2a**–**2c** were synthesized by the reaction of thiocarbohydrazide with the corresponding acids [12].

Elemental analysis was performed on a PerkinElmer 240C instrument. ^{1}H NMR spectra were recorded on a Bruker DPX-250 and Bruker DRX-600 spectrometers at 25°C. IR spectra were recorded on a Varian 3100 FT-IR

Main bonds lengths and valence angles in the molecule of 3,11-dimethyl-11*H*-1,2,4-triazolo[3',4':2,3][1,3,4]thiadiazepino[7,6-*b*]-indole **4a**

Bond	*d*, Å	Bond	*d*, Å	Angle	φ, deg	Angle	φ, deg
N^1–C^{15}	1.301(4)	C^7–C^8	1.386(4)	$C^{15}N^1N^2$	107.3(2)	$C^{12}N^{11}C^{19}$	125.1(2)
N^1–N^2	1.397(3)	C^8–C^9	1.399(4)	$C^3N^2N^1$	107.3(2)	$N^{11}C^{12}C^{10}$	129.6(3)
N^2–C^3	1.306(4)	C^9–C^{10}	1.373(4)	$N^2C^3N^{16}$	110.1(3)	$N^{11}C^{12}C^6$	108.6(2)
C^3–N^{16}	1.376(3)	C^{10}–C^{12}	1.394(4)	$N^2C^3C^{18}$	127.1(3)	$C^{10}C^{12}C^6$	121.8(3)
C^3–C^{18}	1.476(4)	N^{11}–C^{13}	1.369(3)	$N^{16}C^3C^{18}$	122.8(3)	$N^{11}C^{13}C^{17}$	110.6(3)
N^4–C^5	1.293(4)	N^{11}–C^{12}	1.381(4)	$C^5N^4N^{16}$	119.1(3)	$N^{11}C^{13}S^{14}$	120.5(2)
N^4–N^{16}	1.407(4)	N^{11}–C^{19}	1.452(4)	$N^4C^5C^{17}$	132.1(3)	$C^{17}C^{13}S^{14}$	128.3(2)
C^5–C^{17}	1.436(4)	C^{13}–C^{17}	1.375(4)	$C^7C^6C^{12}$	119.9(3)	$C^{15}S^{14}C^{13}$	98.55(14)
C^6–C^7	1.394(4)	C^{13}–S^{14}	1.750(3)	$C^7C^6C^{17}$	133.5(3)	$N^1C^{15}N^{16}$	110.6(3)
C^6–C^{12}	1.403(4)	S^{14}–C^{15}	1.743(3)	$C^{12}C^6C^{17}$	106.7(3)	$N^1C^{15}S^{14}$	122.1(2)
C^6–C^{17}	1.441(4)	C^{15}–N^{16}	1.369(4)	$C^8C^7C^6$	118.0(3)	$N^{16}C^{15}S^{14}$	127.1(2)
				$C^7C^8C^9$	121.6(3)	$C^{15}N^{16}C^3$	104.7(2)
				$C^{10}C^9C^8$	121.0(3)	$C^{15}N^{16}N^4$	136.1(2)
				$C^9C^{10}C^{12}$	117.8(3)	$C^3N^{16}N^4$	118.6(2)
				$C^{13}N^{11}C^{12}$	108.0(2)	$C^{13}C^{17}C^5$	129.2(3)
				$C^{13}N^{11}C^{19}$	126.9(3)	$C^{13}C^{17}C^6$	106.1(2)

instrument. The mass spectrum was obtained by direct input on a Finnigan MAT INCOS 50 mass spectrometer.

Single crystal X-ray diffraction analysis. The unit cell parameters of the crystal of compound **4a** and a three-dimensional set of intensities were obtained at 100 K on a Xcalibur Eos automated diffractometer (MoK_α radiation, graphite monochromator). The crystals of the molecule of compound **4a** are colorless, rhombic, $C_{13}H_{11}N_5S$, *M* 269.33, the unit cell parameters: *a* = 13.7343(12) Å, *b* = 7.2958(6) Å, *c* = 23.8287(15) Å, *V* = 2387.7(3) Å^3, *Z* = 8, d_{calc} = 1.498 г/см^3, μ(MoK_α) = 0.263 mm^{-1}, space group *Pbca*. The intensities of 7557 reflections were measured in the range of angles 2θ ≤ 58.4° by the ω-scanning method from a single crystal with dimensions of 0.20×0.11×0.05 mm. An empirical account of the absorption was carried out according to the Multiscan procedure. After eliminating systematically quenched reflexes and averaging intensities of equivalent reflections, the working array of measured $F^2(hkl)$ and σ(F^2) values amounted to 2319 independent reflections, of which 1774 with $F^2 > 2\sigma(F^2)$. The structure was solved by the direct method and refined by the full-matrix least squares method according to F^2 using the SHELXTL program in the anisotropic approximation for non-hydrogen atoms. In the crystal of compound **4a**, most H atoms are localized in the Fourier synthesis of difference electron density, the coordinates and isotropic thermal parameters of all the H atoms were calculated in the least-squares method using the rider model [13], in the last cycle of the full-matrix refinement, the absolute shifts of all 183 variable structure parameters are less than 0.001σ, the final value of R_1 factor is 0.0603.

5-Methyl-4-[(1-methyl-2-chloro-1*H*-indol-3-yl)-methylidenamino]-4*H*-1,2,4-triazole-3-thiol (3a). A mixture of 0.19 g (0.001 mol) of 1-methyl-2-chloro-1*H*-indole-3-carbaldehyde **1a** and 0.15 g (0.001 mol) of 4-amino-5-methyl-4*H*-1,2,4-triazole-3-thiol **2a** in 10 mL of propan-2-ol was boiled for 1.5 h. After cooling, the precipitate was filtered off, washed with propan-2-ol and petroleum ether, and then dried. Yield 0.18 g (60%), colorless crystals, mp 243–245°C (butan-1-ol). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 2.30 s (3H, CH_3), 3.73 s (3H, CH_3), 7.20–7.31 m (2H, H_{Ar}), 7.52 d (1H, H_{Ar}, *J* = 7.5), 9.98 s (1H, CH=N), 13.60 s (1H,

SH). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 11.25 (CH_3), 30.93 (NCH_3), 106.10 (C^2_{indole}), 111.22 (C_{indole}), 121.64 (C_{indole}), 123.13(C_{indole}), 123.60 (C^3_{indole}), 124.20 (C_{indole}), 123.12 (C_{indole}), 134.02 (C_{ndole}), 136.82 (C_{indole}), 148.88 ($C^3_{triazole}$), 157.42 (CH=N), 161.31 ($C^5_{triazole}$). Found, %: C 49.22; H 4.12; N 28.79; Cl 11.94; S 11.01. $C_{13}H_{12}ClN_5S$. Calculated, %: C 49.07; H 4.09; N 28.85; Cl 12.09; S 10.91.

5-Methyl-4-[(2-chloro-1-ethyl-1*H*-indol-3-yl)-methylidenamino]-4*H*-1,2,4-triazole-3-thiol (3b) was prepared similarly from aldehyde **1b** and triazole-3-thiol **2a**. Yield 0.18 g (44%), colorless crystals, mp 201–203°C (butan-1-ol). IR spectrum, ν, cm^{-1}: 1456, 1463, 1518 (C=C), 1574, 1599 (C=N). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 1.33 t (3H, CH_2CH_3), 2.39 s (3H, CH_3), 4.37 q (2H, CH_2CH_3), 7.30–7.42 m (2H, H_{Ar}), 7.69 d (1H, H_{Ar}, *J* = 7.5), 8.18 d (1H, H_{Ar}, *J* = 7.5), 10.09 s (1H, CH=N), 13.66 s (1H, SH). Found, %: C 52.62; H 4.43; N 22.03; Cl 11.01; S 10.29. $C_{14}H_{14}ClN_5S$. Calculated, %: C 52.58; H 4.38; N 21.91; Cl 11.10; S 10.03.

4-({2-Chloro-1-[2,2-(diethoxyethyl)-1*H*-indol-3-yl]methylene}amino)-5-methyl-4*H*-1,2,4-triazole-3-thiol (3c) was prepared similarly from aldehyde **1c** and triazole-3-thiol **2a**. Yield 0.22 g (56%), yellow crystals, mp 226–228°C. IR spectrum, ν, cm^{-1}: 1459, 1486, 1523 (C=C), 1574, 1601 (C=N). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 1.13 t (6H, OCH_2CH_3), 2.38 s (3H, Me), 3.77 q (4H, OCH_2CH_3), 4.55 s (2H, CH_2), 4.94 s (1H, CH), 7.47 m (2H, H_{Ar}), 7.81 d (1H, H_{Ar}, *J* = 5.0), 8.31 d (1H, H_{Ar}, *J* = 5.0), 10.31 s (1H, CH=N), 13.81 s (1H, SH). Found, %: C 57.47; H 5.93; N 18.11; Cl 9.31; S 8.61. $C_{18}H_{22}ClN_5S$. Calculated, %: C 57.53; H 5.85; N 18.16; Cl 9.45; S 8.53.

4-({2-Chloro-[2,2-(diethoxyethyl)-1*H*-indol-3-yl]-methylene}amino)-5-(*p*-tolyl)-4*H*-1,2,4-triazole-3-thiol (3d) was prepared similarly from aldehyde **1c** and triazole-3-thiol **2c**. Yield 0.35 g (73%), colorless powder, mp 228–230°C. IR spectrum, ν, cm^{-1}: 1463, 1486, 1510 (C=C), 1574, 1589 (C=N). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 1.21 t (6H, OCH_2CH_3), 2.37 s (3H, CH_3), 3.64 q (4H, OCH_2CH_3), 4.45 d (2H, CH_2, *J* = 2.5), 4.82 t (1H, CH, *J* = 2.5), 7.23–7.39 m (4H, H_{Ar}), 7.69 d (1H, H_{Ar}, *J* = 7.5), 7.86 d (2H, H_{Ar}, *J* = 10.0), 8.02 d (1H, H_{Ar}, *J* = 7.5), 9.80 s (1H, CH=N), 14.07 s (1H, SH). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 15.52 ($2OCH_2CH_3$), 21.40 (CH_3), 47.33 (CH), 63.68 ($2OCH_2CH_3$), 100.62 (C_{Ar}), 106.48 (C^i_{Ar}), 112.19 (C_{Ar}), 121.21 (C_{Ar}), 123.22 (C_{Ar}), 123.25 (C^i_{Ar}), 123.68 (C^i_{Ar}), 124.17 (C_{Ar}), 128.66 ($2C_{Ar}$), 129.59 ($2C_{Ar}$), 134.65 (C^i_{Ar}), 136.92 (C^i_{Ar}), 141.09 (C^i_{Ar}), 149.07 (C^i_{Ar}), 160.85 (CH=N), 162.53 (C^i_{Ar}). Found, %: C 63.87; H 5.83; N 15.31; Cl 7.62; S 7.01. $C_{24}H_{26}ClN_5S$. Calculated, %: C 63.79; H 5.75; N 15.50; Cl 7.86; S 7.08.

3,11-Dimethyl-11*H*-1,2,4-triazolo[3',4':2,3][1,3,4]-thiadiazepino[7,6-*b*]indole (4a). A mixture of 0.19 g (0.001 mol) of 1-methyl-2-chloro-1*H*-indole-3-carbaldehyde **1a** and 0.15 g (0.001 mol) of triazole-3-thiol **2a** in 10 mL of propan-2-ol was boiled for 4 h, and then stirred at room temperature for 6 h. The precipitate was filtered off, washed with propan-2-ol and petroleum ether, and then dried. Yield 0.25 g (45%), colorless powder, mp 230–232°C. IR spectrum, ν, cm^{-1}: 1377, 1463, 1505, 1537 (C=C), 1573, 1592, 1623 (C=N). ^{1}H NMR spectrum (DMSO-d_6), δ, ppm (*J*, Hz): 2.38 s (3H, CH_3), 3.86 s (3H, CH_3), 7.23–7.36 m (2H, H_{Ar}), 7.60 d (1H, H_{Ar}, *J* = 7.5), 7.81 d (1H, H_{Ar}, *J* = 7.5), 8.79 s (1H, CH=N). ^{13}C NMR spectrum (DMSO-d_6), δ_C, ppm: 10.99 (CH_3), 31.54 (NCH_3), 109.94(C^i_{indole}), 111.85 (C_{indole}), 118.32 (C_{indole}), 122.79 (C_{indole}), 124.23 (C_{indole}), 126.28 (C^i_{indole}), 133.13 (C^i_{indole}), 138.17 (C^i_{indole}), 141.69 ($C^i_{triazole}$), 151.59 (CH=N), 154.50 ($C^i_{triazole}$). Mass spectrum, *m/z* (I_{rel}, %): 269 (100) $[M]^+$, 241 (18.1), 236 (75), 199 (24.13), 183 (12), 173 (15.51), 168 (13.79), 155 (19.82), 140 (18.1), 128 (17.24), 114 (21.55), 102 (12.9), 89 (13.79), 75 (15.51), 69 (13.79), 59 (11.2), 51 (13.79), 42 (24.13), 27 (16.37), 15 (28.44).

3-Ethyl-11-methyl-11*H*-1,2,4-triazolo[3',4':2,3]-[1,3,4]thiadiazepino[7,6-*b*]indole (4b) was prepared similarly from aldehyde **1a** and triazole **2b**. Yield 35%, colorless powder, mp 336–338°C. IR spectrum, ν, cm^{-1}: 1419, 1463, 1469, 1493, 1513 (C=C), 1571, 1586, 1671 (C=N). ^{1}H NMR spectrum (CF_3COOH-d_1), δ, ppm (*J*, Hz): 1.43 t (2H, CH_2CH_3, *J* = 7.5), 3.12 q (2H, CH_2CH_3, *J* = 7.5), 3.74 s (3H, NCH_3), 6.79 d (1H, H_{Ar}, *J* = 7.5), 6.98 d (1H, H_{Ar}, *J* = 7.5), 7.26–7.40 m (2H, H_{Ar}), 9.14 s (1H, CH=N). Found, %: C 59.27; H 4.61; N 24.51; S 11.21. $C_{14}H_{13}N_5S$. Calculated, %: C 59.36; H 4.59; N 24.73; S 11.32.

3,11-Diethyl-11*H*-1,2,4-triazolo[3',4':2,3][1,3,4]-thiadiazepino[7,6-*b*]indole (4c) was prepared similarly from aldehyde **1b** and triazole **2b**. Yield 57%, colorless crystals, mp 212–214°C. IR spectrum, ν, cm^{-1}: 1422, 1456, 1466, 1491, 1519 (C=C), 1573, 1588 (C=N). ^{1}H NMR spectrum (CF_3COOH-d_1), δ, ppm (*J*, Hz): 1.28 t (3H, CH_2CH_3, *J* = 7.5), 1.40 t (3H, NCH_2CH_3, *J* = 7.5), 3.07 q (2H, CH_2CH_3, *J* = 7.5), 4.19 q (2H, NCH_2CH_3,

J = 7.5), 6.73–6.79 m (1H, H_{Ar}), 6.99 d (1H, H_{Ar}, J = 10.0), 7.26 m (1H, H_{Ar}), 7.41 d (1H, H_{Ar}, J = 10.0), 9.16 s (1H, CH=N). Found, %: C 60.23; H 4.97; N 23.78; S 10.58. $C_{15}H_{15}N_5S$. Calculated, %: C 60.60; H 5.05; N 23.57; S 10.79.

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CONFLICT OF INTEREST

No conflict of interest was declared by the authors.

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