

A simple and efficient procedure for synthesis of optically active 1,2-bis(*s*-triazolo (Bis (*S*-triazolo[3,4-*b*][1,3,4]thiadiazole-3-yl) alkane derivatives containing L-amino acid moieties

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A series of 1,2-bis(*s*-triazolo (Bis (*S*-triazolo[3,4-*b*][1,3,4]thiadiazolo-3-yl)alkanes **3a–3k** were prepared by condensation reaction of 1,2-bis(4-amino-5-mercapto-*S*-triazol-3-yl)alkanes **1a–1c** with *N*-phetaloyl-L-amino acids **2a–2d** in the presence of the phosphoroxy chloride (POCl₃) as an anhydrous reagent.



Keywords: N-phetaloyl-L-amino acid; triazole; triazolothiadiazole; phosphoroxy chloride; optically active

1. Introduction

Bisheterocyclics are interesting compounds because of their various biological activities (1, 2). It is well documented that heterocycles bearing a triazole or 1,3,4-thiadiazole moiety exhibit many biological properties such as antiinflammatory (3), antinociceptive (4) and antitumor (5) activity.

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Additionally, N-bridged heterocyclic compounds derived from 1,2,4-triazoles have found several applications in the field of medicine, agriculture and industry (6). 1,3,4-Thiadiazoles display a wide range of biological activities, possibly due to the presence of the toxophoric N–C–S linkage (7). They are also known to have applications such as antibacterial, antitumor and antiinflammatory agents, pesticides, herbicides, dyes, lubricants and analytical reagents (8). [1,2,4]-Triazolo-[3,4-b]-[1,3,4]-thiadiazole derivatives, produced by fusion of the biolibale [1,2,4]-triazole and [1,3,4]-thiadiazole rings, are reported to possess pharmacological activities (9). Preparation of these compounds has extensively been studied during the past few years, and the most convenient and general method for the synthesis of [1,2,4]-triazolo-[3,4-b]-[1,3,4]-thiadiazole derivatives involves the reaction of 5-substituted 4-amino-(4H)-1,2,4-triazole-3-thioles with carboxylic acids (10–12). Nevertheless, to the best of our knowledge, there is no report for preparation of optically active bis-1,2,4-triazolo-[3,4-b]-1,3,4-thiadiazole ring systems containing L-amino acids.

On the basis of these reports and also in continuation of our research program on the synthesis of heterocyclic compounds containing 1,2,4-triazole and optically active compounds (13-18), we wish to report the synthesis of some new 3,6-disubstituted 1,2,4-triazolo[3,4-*b*]-1,3,4-thiadiazoles containing L-amino acid moiety, with the hope to improve their biological activities, because chirality is an important factor of the bioactive molecules and a recognition phenomenon associated with these molecules.

2. Results and discussion

This paper describes a facile one-step synthesis of bis-triazolothiadiazole derivatives **3** via reaction between 1 mol of bis-amino triazole with 2 mol of aliphatic acid. *N*-phthaloyl-L-amino acids **2a–2d** were synthesized according to the procedure described in literature (*19*). The starting 1,2-bis(4-amino-5-mercapto-4*H*-1,2,4-triazol-3-yl)alkanes (**1a–1c**) were prepared by heating an aliphatic diacid with 2 equiv. of carbonothioic dihydrazide in an oil bath at 170°C (*20*). The resultant bis-5,5'-(alkane-1,2-diyl)bis(4-amino-4*H*-1,2,4-triazole-3-thiol) compounds **1a–1c** were further converted to 1,2,4-triazolo-[3,4-*b*]-1,3,4-thiadiazoles **3a–3j** through a one-pot reaction by condensation with *N*-phthaloyl-L-amino acids **2a–2d** in the presence of phosphoroxy chloride (POCl₃) as outlined in Scheme 1.



Scheme 1. Synthesis of compounds **3a–3k**.

Phosphorus oxychloride was necessary for this condensation, which activates the carboxyl group of amino acids and increases its electrophilicity. This procedure afforded various

triazolothiadiazoles in 52–80% yields. All compounds were structurally characterized by using IR, ¹H NMR and ¹³C NMR data. The IR spectra of the bis(4-amino-5-mercapto-4*H*-1,2,4-triazol-3-yl)alkanes (**1a–1c**) showed absorption bands at 3161 and 3286 cm⁻¹ due to the NH₂ groups, which were absent in the IR spectra of the aza crown compounds **3a–3k**. Similarly, the ¹H NMR spectra of the bistriazoles **1a–1c** showed a broad signal at $\delta = 5.56$ ppm attributed to the resonance of NH₂ groups, which was not present in the spectra of compounds **3a–3k**. In addition, the substituted bis(4-amino-4*H*-1,2,4-triazole-3-thiol) derivatives showed a peak around 13.8 ppm indicating the existence of the thiol group, which clearly disappeared in the title compounds confirms their condensed structure.

3. Conclusion

We have been able to synthesize some N-phetaloyl-L-amino acids having a free terminal carboxyl function, which can react with bis-amino triazole. This reaction may be useful for combinational synthesis of type **3** compounds having various R and Y substitutions with a view to test for biological activities.

4. Experimental

4.1. General

Melting points were determined using an electrothermal digital apparatus and are corrected. Purity of the compound was checked by thin-layer chromatography using EtOH/*n*-hexane (1:1, v/v) as an eluent. IR spectra were prepared on a galaxy series FT-IR 5000 spectrophotometer using KBr discs. NMR spectra were recorded on a Bruker spectrophotometer (300 MHz) in DMSO- d_6 using TMS as an internal standard. Specific rotations were measured by using a Perkin Elmer 341 polarimeter.

4.2. General procedure for preparation of compounds 3a–3k

A mixture of 1,2-bis(4-amino-5-mercapto-4H-1,2,4-triazol-3-yl)alkanes (**1a**-**1c**) (1 mmol) and corresponding *N*-phthaloyl-L-amino acids **2a**-**2d** (2 mmol) in POCl₃ (7 ml) was refluxed for 16 h. The reaction mixture was slowly poured onto crushed ice with stirring and neutralized with solid potassium bicarbonate. The mixture was allowed to stand overnight and the obtained solid was filtered and washed with cold water. The compound so obtained was dried and crystallized from ethanol to give the pure products **3a**-**3k**.

4.3. 2,2'-(3,3'-(*Ethane-1,2-diyl*)*bis*([1,2,4]*triazolo*[3,4-*b*][1,3,4]*thiadiazole-6,3-diyl*)*bis*(*methylene*)*diisoindoline-1,3-dione* (3*a*)

Yield: 52%; m.p. 294–296°C; IR (KBr) (ν , cm⁻¹): 3043 (aromatic CH stretch), 2933 (aliphatic CH stretch), 1770, 1712 (C=O), 1600 (C=N), 1518, 1467 (C=C); ¹H NMR (DMSO- d_6 , δ , ppm): 7.94 (d, 4H, J = 5.1 Hz, H_{arom.}), 7.89 (d, 4H, J = 5.0 Hz, H_{arom.}), 5.13 (s, 4H, 2× N-CH₂), 3.49 (s, 4H, 2× CH₂); ¹³C NMR (DMSO- d_6 , δ , ppm): 22.0, 38.0, 124.0, 131.8, 135.4, 146.1, 153.9, 165.5, 167.5. Anal. Calcd. for C₂₆H₁₆N₁₀O₄S₂: C, 52.34; H, 2.70, N, 23.48%. Found: C, 52.51; H, 2.55, N, 23.31%.

4.4. 2,2'-(3,3'-(Butane-1,4-diyl)bis([1,2,4]triazolo[3,4-b][1,3,4]thiadiazole-6,3diyl))bis(methylene)diisoindoline-1,3-dione (3b)

Yield: 52%; m.p. 282–284°C; IR (KBr): 3032 (aromatic CH stretch), 2928 (aliphatic CH stretch), 1772, 1720 (C=O), 1521, 1469 (C=C) cm⁻¹; ¹H NMR (DMSO- d_6 , δ , ppm): 7.88 (s, 8H, H_{arom}.), 5.81 (s, 4H, 2× N-CH₂), 2.95 (br, 4H, 2× CH₂), 1.77 (br, 4H, 2× CH₂); ¹³C NMR (DMSO- d_6 , δ , ppm): 24.1, 25.6, 38.0, 124.0, 131.6, 135.3, 147.5, 153.4, 165.0, 167.4. Anal. Calcd. for C₂₈H₂₀N₁₀O₄S₂: C, 53.84; H, 3.23, N, 22.42%. Found: C, 53.71; H, 3.37, N, 22.65%.

4.5. 2,2'-(1,1'-(3,3'-(Ethane-1,2-diyl)bis([1,2,4]triazolo[3,4-b][1,3,4]thiadiazole-6,3diyl))bis(ethane-1,1-diyl))diisoindoline-1,3-dione (3c)

Yield: 70%; m.p. 290–293°C; $[\alpha]_D^{22} = -40$ (c = 0.01, DMSO); IR (KBr) (ν , cm⁻¹): 3040 (aromatic CH stretch), 1774, 1710 (C=O), 1518, 1467 (C=C); ¹H NMR (DMSO- d_6 , δ , ppm): 7.90 (s, 8H, H_{arom}), 5.80 (s, 2H, 2× N-CH), 3.52 (s, 4H, 2× CH₂), 1.85 (br, 6H, 2× CH₃); ¹³C NMR (DMSO- d_6 , δ , ppm): 17.0, 22.1, 46.7, 124.0, 131.7, 135.4, 146.2, 154.0, 167.3, 170.0. Anal. Calcd. for C₂₈H₂₀N₁₀O₄S₂: C, 53.84; H, 3.23, N, 22.42%. Found: C, 53.98; H, 3.30, N, 22.53%.

4.6. 2,2'-(1,1'-(3,3'-(Propane-1,3-diyl)bis([1,2,4]triazolo[3,4-b][1,3,4]thiadiazole-6,3diyl))bis(ethane-1,1-diyl))diisoindoline-1,3-dione (3d)

Yield: 83%; m.p. 243–246°C; $[\alpha]_D^{22} = -10$ (c = 0.01, DMSO); IR (KBr) (ν , cm⁻¹): 3030 (aromatic CH stretch), 2941 (aliphatic CH stretch), 1778, 1714 (C=O), 1608 (C=N); ¹H NMR (DMSO- d_6 , δ , ppm): 8.00 (s, 8H, H_{arom}), 5.82 (q, 2H, J = 7.1 Hz, 2× N-CH), 3.13 (br, 4H, 2× CH₂), 2.29 (br, 2H, CH₂), 1.87 (br, 6H, 2× CH₃); ¹³C NMR (DMSO- d_6 , δ , ppm): 16.7, 23.2, 24.0, 46.8, 123.8, 131.6, 135.4, 147.2, 153.5, 167.3, 169.7. Anal. Calcd. for C₂₉H₂₂N₁₀O₄S₂: C, 54.54; H, 3.47, N, 21.93%. Found: C, 54.38; H, 3.51, N, 22.14%.

4.7. 2,2'-(1,1'-(3,3'-(Butane-1,4-diyl)bis([1,2,4]triazolo[3,4-b][1,3,4]thiadiazole-6,3diyl))bis(ethane-1,1-diyl))diisoindoline-1,3-dione (3e)

Yield: 70%; m.p. 243–245°C; $[\alpha]_D^{22} = -10$ (c = 0.01, DMSO); IR (KBr) (ν , cm⁻¹): 3041 (aromatic CH stretch), 2964, 2874 (aliphatic CH stretch), 1780, 1720 (C=O), 1610 (C=N), 1516, 1467 (C=C) cm⁻¹; ¹H NMR (DMSO- d_6 , δ , ppm): 7.88 (s, 8H, H_{arom.}), 5.81 (br, 2× N-CH), 3.00 (br, 4H, 2× CH₂), 1.77–1.85 (m, 10H, 2× CH₃, 2× CH₂); ¹³C NMR (DMSO- d_6 , δ , ppm): 17.0, 24.1, 25.6, 46.8, 124.0, 131.7, 135.4, 147.5, 153.4, 167.3, 169.7. Anal. Calcd. for C₃₀H₂₄N₁₀O₄S₂: C, 55.20; H, 3.71, N, 21.46%. Found: C, 55.43; H, 3.79, N, 21.31%.

4.8. 2,2'-(1,1'-(3,3'-(Ethane-1,2-diyl)bis([1,2,4]triazolo[3,4-b][1,3,4]thiadiazole-6,3diyl))bis(2-methyl propane-1,1-diyl))diisoindoline-1,3-dione (3f)

Yield: 80%; m.p. 243–245°C; $[\alpha]_D^{22} = -40$ (c = 0.01, DMSO); IR (KBr): 3035 (aromatic CH stretch), 2968, 2876 (aliphatic CH stretch), 1782, 1716 (C–O), 1612 (C–N), 1518, 1469 (C–C) cm⁻¹; ¹H NMR (DMSO- d_6 , δ , ppm): 7.90 (s, 8H, H_{arom}.), 5.13 (d, 2H, J = 10.1 Hz, $2 \times$ N-CH), 3.53 (s, 4H, $2 \times$ CH₂), 2.90 (br, 2H, $2 \times$ CH), 0.98 (d, 6H, J = 6.3 Hz, $2 \times$ CH₃), 0.90 (d, 6H, J = 6.2 Hz, $2 \times$ CH₃); ¹³C NMR (DMSO- d_6 , δ , ppm): 19.4, 20.0, 22.3, 29.6, 57.9, 124.1, 131.2, 135.5, 146.1, 154.2, 166.9, 167.7. Anal. Calcd. for C₃₂H₂₈N₁₀O₄S₂: C, 56.46; H, 4.15, N, 20.58%. Found: C, 56.28; H, 4.01, N, 20.75%.

4.9. 2,2'-(1,1'-(3,3'-(Propane-1,3-diyl)bis([1,2,4]triazolo[3,4-b][1,3,4]thiadiazole-6,3diyl))bis(2-methyl propane-1,1-diyl))diisoindoline-1,3-dione (3g)

Yield: 76%; m.p. 226–228°C; $[\alpha]_D^{22} = -80$ (c = 0.01, DMSO); IR (KBr) cm⁻¹: 3050 (aromatic CH stretch), 2966, 2874 (aliphatic CH stretch), 1782, 1718 (C–O), 1612 (C=N), 1518, 1469 (C=C); ¹H NMR (DMSO- d_6 , δ , ppm): 7.90 (s, 8H, H_{arom.}), 5.22 (d, 2H, J = 9.9 Hz, 2× N-CH), 3.15 (br, 4H, 2× CH₂), 2.93 (br, 2H, 2× CH), 2.29 (br, 2H, CH₂), 1.01 (br, 6H, 2× CH₃), 0.93 (br, 6H, 2× CH₃); ¹³C NMR (DMSO- d_6 , δ , ppm): 19.4, 20.0, 23.4, 24.0, 29.5, 57.9, 124.1, 131.2, 135.5, 147.1, 154.0, 166.8, 167.7. Anal. Calcd. for C₃₃H₃₀N₁₀O₄S₂: C, 57.05; H, 4.35, N, 20.16%. Found: C, 57.20; H, 4.30, N, 20.27%.

4.10. 2,2'-(1,1'-(3,3'-(Butane-1,4-diyl)bis([1,2,4]triazolo[3,4-b][1,3,4]thiadiazole-6,3diyl))bis(2-methyl propane-1,1-diyl))diisoindoline-1,3-dione (3h)

Yield: 85%; m.p. 226–228°C; $[\alpha]_D^{22} = -80$ (c = 0.01, DMSO); IR (KBr): 3036 (aromatic CH stretch), 2928 (aliphatic CH stretch), 1776, 1712 (C=O), 1610 (C=N), 1518, 1462 (C=C) cm⁻¹; ¹H NMR (DMSO- d_6 , δ , ppm): 7.88 (s, 8H, H_{arom.}), 5.18 (br, 2H, 2× N-CH), 2.89–3.02 (m, 6H, 2× CH₂, 2× CH), 1.84 (br, 4H, 2× CH₂), 0.97 (br, 6H, 2× CH₃), 0.90 (br, 6H, 2× CH₃); ¹³C NMR (DMSO- d_6 , δ , ppm): 19.4, 20.0, 24.2, 25.7, 29.5, 57.9, 124.0, 131.2, 135.5, 147.4, 153.9, 166.7, 167.7. Anal. Calcd. for C₃₄H₃₂N₁₀O₄S₂: C, 57.61; H, 4.55, N, 19.76%. Found: C, 57.50; H, 4.49, N, 19.56%.

4.11. 2,2'-(1,1'-(3,3'-(*Ethane-1,2-diyl*)*bis*([1,2,4]*triazolo*[3,4-*b*][1,3,4]*thiadiazole-6,3-diyl*)*bis*(2-*phenyl ethane-1,1-diyl*)*)diisoindoline-1,3-dione* (3*i*)

Yield: 80%; m.p. 238–240°C; $[\alpha]_D^{22} = -40$ (c = 0.01, DMSO); IR (KBr): 3030 (aromatic CH stretch), 2930 (aliphatic CH stretch), 1778, 1718 (C=O), 1612 (C=N), 1520, 1469 (C=C) cm⁻¹; ¹H NMR (DMSO- d_6 , δ , ppm): 7.88 (s, 8H, H_{arom.}), 7.20 (br, 10H, H_{arom.}), 5.98–6.00 (m, 2H, 2× N-CH), 3.58–3.68 (m, 4H, 2× PhCH₂), 3.55 (s, 4H, 2× CH₂); ¹³C NMR (DMSO- d_6 , δ , ppm): 22.2, 36.0, 52.3, 124.1, 127.6, 128.9, 129.3, 130.9, 135.6, 136.0, 146.3, 153.7, 167.3, 168.3. Anal. Calcd. for C₄₀H₂₈N₁₀O₄S₂: C, 61.84; H, 3.63, N, 18.03%. Found: C, 61.71; H, 3.75, N, 18.11%.

4.12. 2,2'-(1,1'-(3,3'-(Propane-1,3-diyl)bis([1,2,4]triazolo[3,4-b][1,3,4]thiadiazole-6,3-diyl))bis(2-phenyl ethane-1,1-diyl))diisoindoline-1,3-dione (3j)

Yield: 85%; m.p. 235–237°C; $[\alpha]_D^{22} = -10$ (c = 0.01, DMSO); IR (KBr): 3035 (aromatic CH stretch), 2930 (aliphatic CH stretch), 1776, 1714 (C=O), 1605 (C=N) cm⁻¹; ¹H NMR (DMSO- d_6 , δ , ppm): 7.82 (s, 8H, H_{arom.}), 7.13–7.18 (m, 10H, H_{arom.}), 6.04–6.15 (m, 2H, 2× N-CH), 3.97–4.15 (m, 4H, 2× PhCH₂), 3.12 (br, 4H, 2× CH₂), 2.30 (br, 2H, CH₂); ¹³C NMR (DMSO- d_6 , δ , ppm) 23.2, 23.9, 36.0, 52.3, 124.1, 127.6, 128.9, 129.4, 130.8, 135.6, 136.0, 147.2, 153.6, 167.4, 168.1. Anal. Calcd. for C₄₁H₃₀N₁₀O₄S₂: C, 62.27; H, 3.82, N, 17.71%. Found: C, 62.13; H, 3.89, N, 17.53%.

4.13. 2,2'-(1,1'-(3,3'-(Butane-1,4-diyl)bis([1,2,4]triazolo[3,4-b][1,3,4]thiadiazole-6,3-diyl))bis(2-phenyl ethane-1,1-diyl))diisoindoline-1,3-dione (3k)

Yield: 85%; m.p. 232–234°C; $[\alpha]_D^{22} = -80$ (c = 0.01, DMSO); IR (KBr): 3050 (aromatic CH stretch), 2942 (aliphatic CH stretch), 1776, 1712 (C=O), 1610 (C=N) cm⁻¹; ¹H NMR (DMSO- d_6 , δ , ppm): 7.84 (s, 8H, H_{arom.}), 7.21 (br, 10H, H_{arom.}), 6.01 (br, 2H, 2× N-CH), 3.68 (br, 4H,

 $2 \times$ PhCH₂), 3.01 (br, 4H, $2 \times$ CH₂), 1.80 (br, 4H, $2 \times$ CH₂); ¹³C NMR (DMSO-*d*₆, δ , ppm) 24.1, 25.7, 36.1, 52.4, 124.0, 127.6, 128.9, 129.4, 130.9, 135.5, 136.1, 147.5, 153.5, 167.3, 167.9. Anal. Calcd. for C₄₂H₃₂N₁₀O₄S₂: C, 62.67; H, 4.01, N, 17.40%. Found: C, 62.85; H, 3.96, N, 17.56%.

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