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Synthesis of novel isoxazolyl bis-thiazolo[3,2-*a*]pyrimidines

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Abstract

A new synthetic strategy for the synthesis of novel 3-(3-(3-methyl-4-nitroisoxazol-5-yl)-2-phenyl-1-(5,7-diaryl-7*H*-thiazolo[3,2-*a*]pyrimidin-3-yl)propyl)-5,7-diaryl-7*H*-thiazolo[3,2-*a*] pyrimidines (**7a–i**) analogues is described. Reaction of 3-(2-(3-methyl-4-nitroisoxazole-5-yl)-1-phenylethyl)pentane-2,4-dione (**3**) with two moles of thiourea in presence of iodine and CuO afforded 4-(1-(2-aminothiazol-4-yl)-3-(3-methyl-4-nitroisoxazol-5-yl)-2-aryl propyl-thiazol-2-amine (**5**). Compound **5** on reaction with two moles of chalcone (**6**) furnished novel 3-(3-(3-methyl-4-nitroisoxazol-5-yl)-2-phenyl-1-(5,7-diaryl-7*H*-thiazolo[3,2-*a*]pyrimidin-3-yl)propyl)-5,7-diaryl-7*H*-thiazolo[3,2-*a*] pyrimidines (**7a–i**).

Keywords: Isoxazolyl bis-thiazoles; Isoxazolyl bis-thiazolo[3,2-*a*]pyrimidines; New synthetic strategy

Thiazolopyrimidines [TAPM] have been received particular attention and widely recognized as biologically useful systems, since they are important structural components of purine bases. Apparently, the thiazolopyrimidine scaffold is not very frequently used in drug discovery programs. However, biological activities of certain thiazolopyrimidines have been reported. Thaizolo[4,5-*b*]pyrimidine (a guanosine analogue) exhibited *in vitro* activity against a variety RNA and DNA viruses [1] and also had antitumor and antimetastatic properties [2]. 2-Aminothiazolo[4,5-*b*]pyrimidine acts as CXCR2 receptor antagonist [3]. Thaizolo[4,5-*b*]pyrimidine-5,7-dione analogues have been reported to possess anti-inflammatory activities due to TNF inhibition [4]. Thiazolopyrimidines are also have been found to exhibit a wide range of biological activities, such as antiviral, antibacterial, anticancer, androgenic, anabolic and anti-human cytomegalovirus (HCMV) activities [5–9]. In addition, thiazolopyrimidines also can be utilized as templates which have been additionally functionalized to achieve selective receptors or enzyme interactions [10]. Biological activity of substituted isoxazoles has made them a focus of medicinal chemistry over years. Isoxazoles are potent analgesic, anti-inflammatory [11], antimicrobial [12], COX-2 inhibitory [13], anti-tubercular [14], anticonvulsant [15] and anticancer agents [16]. Inspired with the biological profile of thiazolopyrimidine and isoxazole nuclei and their increasing importance in pharmaceutical and biological fields, and in connection with our search on the design and synthesis of biologically active and pharmacologically important new heterocycles linked to isoxazoles [17], it was thought worthwhile to synthesize the title compounds with a view to obtain certain chemical entities with active pharmachophores in a single molecular framework.

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1. Experimental

All the melting points were determined on a Cintex melting point apparatus and are uncorrected. Analytical TLC was performed on Merck precoated 60 F_{254} silica gel plates. Visualization was done by exposing to iodine vapor. IR spectra (KBr pellet) were recorded on a Perkin Elmer BX series FT-IR spectrometer. ^{1}H NMR spectra were recorded on a Varian Gemini 300 MHz spectrometer. ^{13}C NMR spectra were recorded on a Bruker 75 MHz spectrometer. Chemical shift values are given in δ ppm with tetramethyl silane as an internal standard. Mass spectral measurements were carried out by EI method on a Jeol JMC-300 spectrometer at 70 eV. Elemental analyses were performed on a Carlo Erba 106 and Perkin-Elmer model 240 analyzers.

1.1. Synthesis of 1-(2-aminothiazol-4-yl)-3-(3-methyl-4-nitroisoxazol-5-yl)-2-arylpropyl-thiazol-2-amine ***5***

A mixture of 3-(2-(3-methyl-4-nitroisoxazole-5-yl)-1-phenylethyl) pentane-2,4-dione (**3**) (0.01 mol), thiourea (0.02 mol), iodine (0.01 mol) and CuO (0.01 mol) were taken in ethanol (15 mL). The reaction mixture was refluxed for about 8 h. After completion of the reaction (monitored by TLC), the solvent was removed under pressure and added 30 mL water to the residue, then extracted with ethyl acetate and the residue was purified by recrystallization from methanol to produce **5** in high yield.

1.2. Synthesis of 3-(3-(3-methyl-4-nitroisoxazol-5-yl)-2-phenyl-1-(5,7-diaryl-7H-thiazolo[3,2-a]pyrimidin-3-yl)propyl)-5,7-diaryl-7H-thiazolo[3,2-a]pyrimidines ***7***

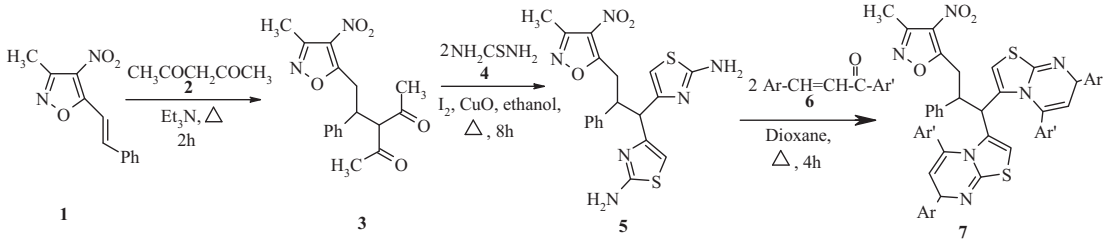
To a solution of 1-(2-aminothiazol-4-yl)-3-(3-methyl-4-nitroisoxazol-5-yl)-2-arylpropylthiazol-2-amine (**5**) (0.01 mol) in dioxane (20 mL) and chalcone (0.02 mol) was added and reaction mixture was refluxed for 4 h. After the completion of the reaction (monitored by TLC), the reaction mixture was poured onto crushed ice, separated solid was filtered off and recrystallized from ethyl acetate to produce (**7a**–**i**) in good yields.

2. Results and discussion

The synthesis of title compounds **7** was accomplished by a new synthetic strategy and is shown in Scheme 1. The key intermediate 3-(2-(3-methyl-4-nitroisoxazole-5-yl)-1-phenylethyl)pentane-2,4-dione **3**, required for synthesis of target compounds was obtained by Michael addition of 3-methyl-4-nitrostyrylisoxazole **1** with acetyl acetone in refluxing triethyl amine [18]. Compound **3** on cyclocondensation reaction with two moles of thiourea under reflux in presence of iodine and CuO in ethanol gave 4-(1-(2-aminothiazol-4-yl)-3-(3-methyl-4-nitroisoxazol-5-yl)-2-arylpropyl-thiazol-2-amine **5** in excellent yields [19]. This is in accordance with earlier observation [20].

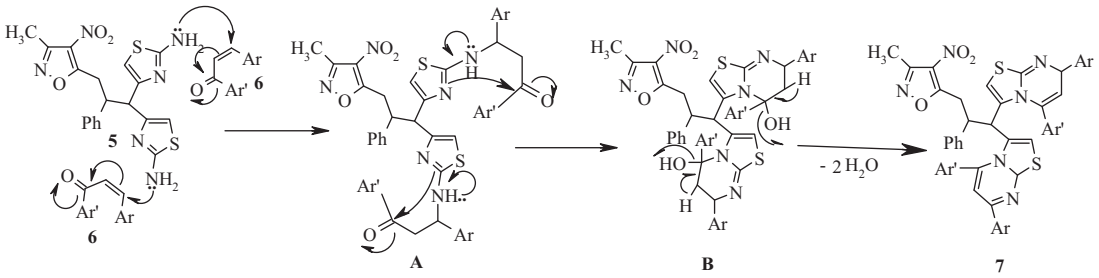
Compound **5** on reaction with two moles of chalcone (having α,β-unsaturated carbonyl system) in dioxane under reflux for 4 h. resulted in the formation of 3-(3-(3-methyl-4-nitroisoxazol-5-yl)-2-phenyl-1-(5,7-diaryl-7*H*-thiazolo[3,2-*a*]pyrimidin-3-yl)propyl)-5,7-diaryl-7*H*-thiazolo[3,2-*a*]pyrimidines **7** in excellent yields [21].

The pyrimidine ring is formed as the amino group of thiazole makes a nucleophilic attack on the β-carbon of the chalcone which is electron deficient due to electron withdrawing influence of carbonyl group, leading to the formation



7a-i: Ar = C_6H_5, Ar' = C_6H_5; Ar = C_6H_5, Ar' = 4-ClC_6H_4; Ar = C_6H_5, Ar'= 4-$CH_3C_6H_4$; Ar = C_6H_5, Ar'= 4-$CH_3OC_6H_4$; Ar = C_6H_5, Ar' = 2-OHC_6H_4; Ar = 4-$CH_3C_6H_4$, Ar' = C_6H_5; Ar = 4-$CH_3OC_6H_4$, Ar' = C_6H_5; Ar = 2-ClC_6H_4, Ar' = C_6H_5; Ar = 4-ClC_6H_4, Ar' = C_6H_5

Scheme 1. **7a**–**i**: Ar = C_6H_5, Ar′ = C_6H_5; Ar = C_6H_5, Ar′ = 4-ClC_6H_4; Ar = C_6H_5, Ar′ = 4-$CH_3C_6H_4$; Ar = C_6H_5, Ar′ = 4-$CH_3OC_6H_4$; Ar = C_6H_5, Ar′ = 2-OHC_6H_4; Ar = 4-$CH_3C_6H_4$, Ar′ = C_6H_5; Ar = 4-$CH_3OC_6H_4$, Ar′ = C_6H_5; Ar = 2-ClC_6H_4, Ar′ = C_6H_5; Ar = 4-ClC_6H_4, Ar′ = C_6H_5.



Scheme 2. Plausible mechanism for pyrimidine ring formation.

of Michael type addition product **A** which is not isolated. Then, imino group of thiazole influenced by secondary amino group attacks the carbonyl group resulting cyclocondensation product **B**, which spontaneously lose water to give the title compound **7** (Scheme 2). A new synthetic strategy has been utilized for the synthesis of title compounds involving the reaction between thiazole amine and chalcone. The structures of the products **5** and **7** have been elucidated on the basis of (IR, ^{1}H NMR, ^{13}C NMR and MS) and micro analytical data.

In conclusion, we report a new synthetic strategy for the synthesis of title compounds with potential biological activity using inexpensive and commercially available materials.

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[19] Analytical data for compound **5**: pale yellow; yield 80%, mp 156–158 °C; IR (KBr, cm^{-1}): 3350, 3325 (NH_2), 1615 (C═N), 1560, 1365 (NO_2); ^{1}H NMR (300 MHz, $CDCl_3$): δ 2.32 (s, 3H, CH_3), 3.83 (m, 2H, CH_2), 4.10 (s, 4H, $2NH_2$), 4.81 (m, 1H, Ar–CH), 5.25 (d, 1H, CH), 7.13 (s, 1H, thiazole-CH), 7.20–8.00 (m, 5H, ArH). EI-MS $[M]^+$ *m/z* 442. ^{13}C NMR (75 MHz, $CDCl_3$): δ 11.41, 34.26, 48.75, 50.13, 105.36, 115.26, 124.13, 124.75, 125.35, 126.13, 126.42, 140.36, 150.38, 152.45, 159.96, 162.36. Anal. Calcd. for $C_{19}H_{18}N_6O_3S_2$: C, 51.57; H, 4.10; N, 18.99. Found: C, 51.52; H, 4.17; N, 19.03%.
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[21] Analytical data for compounds: **7a**: pale yellow; yield 82%, mp 165–167 °C; IR (KBr, cm^{-1}): 1625 (C═N), 1555, 1360 (NO_2); ^1H NMR (300 MHz, $CDCl_3$): δ 2.33 (s, 3H, CH_3), 3.72 (m, 2H, CH_2), 4.14 (m, 1H, *J* = 4 Hz, benzylic-H), 4.80 (m, 1H, CH), 5.23 (d, 2H, Ar–CH), 5.53 (d, 2H, *J* = 4 Hz, ═CH), 7.12 (s, 2H, thiazole-CH), 7.28–7.84 (m, 25H, ArH). EI-MS $[M]^+$ *m/z* 822. ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.01, 34.56, 47.98, 50.48, 61.43, 94.28, 105.36, 115.28, 124.13, 124.21, 124.75, 125.35, 126.03, 126.13, 126.49, 126.67, 126.81, 127.01, 127.13, 127.45, 127.98, 128.11, 128.34, 135.68, 136.73, 140.36, 143.69, 150.58, 152.40, 159.78, 162.42. Anal. Calcd. for $C_{49}H_{38}N_6O_3S_2$: C, 71.51; H, 4.65; N, 10.21. Found: C, 71.58; H, 4.58; N, 10.18%. **7b**: pale yellow; yield 80%, mp 168–170 °C; IR (KBr, cm^{-1}): 1620 (C═N), 1542, 1358 (NO_2); ^1H NMR (300 MHz, $CDCl_3$): δ 2.28 (s, 3H, CH_3), 3.68 (m, 2H, CH_2), 4.10 (m, 1H, *J* = 4 Hz, benzylic-H), 4.78 (m, 1H, CH), 5.38 (d, 2H, Ar–CH), 5.50 (d, 2H, *J* = 4 Hz, ═CH), 7.00 (s, 2H, thiazole-CH), 7.12–7.92 (m, 23H, ArH). EI-MS $[M]^+$ *m/z* 890. ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.14, 35.56, 47.08, 51.08, 61.72, 94.18, 105.31, 115.15, 124.10, 124.28, 124.62, 125.32, 126.01, 126.10, 126.42, 126.54, 126.79, 127.11, 127.23, 127.37, 127.65, 128.14, 130.13, 134.98, 136.52, 140.13, 143.40, 150.28, 152.31, 159.16, 162.38. Anal. Calcd. for $C_{49}H_{36}N_6Cl_2O_3S_2$: C, 65.99; H, 4.07; N, 9.42. Found: C, 65.95; H, 4.11; N, 9.38%. **7c**: pale yellow; yield 84%, mp 164–166 °C; IR (KBr, cm^{-1}): 1630 (C═N), 1545, 1351 (NO_2); ^1H NMR (300 MHz, $CDCl_3$): δ 2.29 (s, 3H, CH_3), 2.81 (s, 6H, Ar–CH_3), 3.52 (m, 2H, CH_2), 4.06 (m, 2H, *J* = 4 Hz, benzylic-H), 4.63(m, 1H, CH), 5.28 (d, 2H, Ar–CH), 5.41 (d, 2H, ═CH, *J* = 4 Hz), 7.03 (s, 2H, thiazole-CH), 7.14–8.03 (m, 23H, ArH). EI-MS $[M]^+$ *m/z* 850. ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.57, 28.46, 35.16, 48.01, 51.28, 67.53, 91.38, 104.86, 117.11, 124.41, 124.63, 124.82, 125.97, 126.23, 126.41, 126.53, 126.48, 126.73, 127.11, 127.46, 127.88, 128.34, 128.56, 135.41, 136.63, 138.45, 141.06, 142.89, 151.08, 152.68, 159.67, 161.81. Anal. Calcd. for $C_{51}H_{42}N_6O_3S_2$: C, 71.98; H, 4.97; N, 9.87. Found: C, 71.92; H, 4.99; N, 9.91%. **7d**: pale yellow; yield 81%, mp 171–173 °C; IR (KBr, cm^{-1}): 1620 (C═N), 1543, 1349 (NO_2); ^1H NMR (300 MHz, $CDCl_3$): δ 2.21 (s, 3H, CH_3), 3.41 (m, 2H, CH_2), 3.61 (s, 6H, OCH_3), 4.11 (m, 1H, *J* = 4 Hz, benzylic-H), 4.54(m, 1H, CH), 5.31 (d, 2H, Ar–CH), 5.46 (d, 2H, *J* = 4 Hz, ═CH), 7.18 (s, 1H, thiazole-CH), 7.21–8.22 (m, 23H, ArH). EI-MS $[M]^+$ *m/z* 882. ^{13}C NMR (75 MHz, $CDCl_3$): δ 11.86, 33.97, 47.51, 50.36, 58.13, 61.43, 92.07, 106.11, 115.37, 124.26, 124.37, 124.67, 126.16, 126.34, 126.67, 126.71, 126.79, 127.11, 127.18, 127.54, 127.81, 128.00, 128.37, 135.59, 136.41, 140.71, 143.73, 150.42, 152.57, 159.81, 161.45, 162.42. Anal. Calcd. for $C_{51}H_{42}N_6O_5S_2$: C, 69.37; H, 4.79; N, 9.52. Found: C, 69.41; H, 4.77; N, 9.48%. **7e**: pale yellow; yield 79%, mp 155–157 °C; IR (KBr, cm^{-1}): 1615 (C═N), 1540, 1357 (NO_2); ^1H NMR (300 MHz, $CDCl_3$): δ 2.23 (s, 3H, CH_3), 3.33 (m, 2H, CH_2), 4.20 (m, 1H, *J* = 4 Hz, benzylic-H), 4.43(m, 1H, CH), 5.25 (d, 2H, Ar–CH), 5.47 (d, 2H, *J* = 4 Hz, ═CH), 7.18 (s, 2H, thiazole-CH), 7.20–8.31 (m, 23H, ArH), 9.01 (bs, 2H, OH, D_2O, exchangeable). EI-MS $[M]^+$ *m/z* 854. ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.23, 35.41, 48.03, 50.12, 61.72, 94.10, 105.46, 115.56, 123.98, 124.10, 124.47, 125.91, 126.01, 126.23, 126.77, 126.92, 127.12, 127.29, 127.61, 127.83, 128.03, 128.54, 135.72, 136.54, 140.36, 143.51, 150.51, 152.57, 158.11, 159.67, 162.81. Anal. Calcd. for $C_{49}H_{38}N_6O_5S_2$: C, 68.83; H, 4.48; N, 9.83. Found: C, 68.89; H, 4.51; N, 9.79%. **7f**: pale yellow; yield 82%, mp 173–175 °C; IR (KBr, cm^{-1}): 1635 (C═N), 1535, 1341 (NO_2); ^1H NMR (300 MHz, $CDCl_3$) δ: 2.30 (s, 3H, CH_3), 2.83 (s, 6H, Ar–CH_3), 3.50 (m, 2H, CH_2), 4.16 (m, 1H, *J* = 4 Hz, benzylic-H), 4.58(m, 1H, CH), 5.18 (m, 2H, Ar–CH), 5.39 (d, 2H, *J* = 4 Hz, ═CH), 7.11 (s, 2H, thiazole-CH), 7.23–7.91 (m, 23H, ArH). EI-MS $[M]^+$ *m/z* 850. ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.13, 29.01, 35.14, 48.41, 51.38, 67.51, 91.32, 104.86, 117.13, 124.51, 124.68, 124.73, 125.71, 126.11, 126.29, 126.42, 126.61, 126.79, 127.09, 127.25, 127.58, 128.21, 128.38, 135.35, 136.28, 138.15, 141.11, 142.68, 151.31, 152.52, 159.45, 161.69. Anal. Calcd. for $C_{51}H_{42}N_6O_3S_2$: C, 71.98; H, 4.97; N, 9.87. Found: C, 71.94; H, 4.91; N, 9.81%. **7g**: pale yellow; yield 85%, mp 169–171 °C; IR (KBr, cm^{-1}): 1618 (C═N), 1531, 1339 (NO_2); ^1H NMR (300 MHz, $CDCl_3$) δ 2.25 (s, 3H, CH_3), 3.43 (m, 2H, CH_2), 3.63 (s, 6H, OCH_3), 4.21 (m, 1H, *J* = 4 Hz, benzylic-H), 4.64 (m, 1H, CH), 5.38 (m, 2H, Ar–CH), 5.41 (d, 2H, *J* = 4 Hz, ═CH), 7.01 (s, 2H, thiazole-CH), 7.10–8.23 (m, 23H, ArH). EI-MS $[M]^+$ *m/z* 882. ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.16, 34.17, 47.45, 50.21, 58.14, 61.31, 92.16, 106.23, 115.41, 124.11, 124.21, 124.46, 126.31, 126.42, 126.54, 126.63, 126.85, 127.30, 127.45, 127.58, 127.74, 128.07, 128.24, 135.28, 136.38, 140.51, 143.58, 150.27, 152.37, 159.69, 161.23, 161.81 Anal. Calcd. for $C_{51}H_{42}N_6O_5S_2$: C, 69.37; H, 4.79; N, 9.52. Found: C, 69.32; H, 4.81; N, 9.47%. **7h**: pale yellow; yield 80%, mp 174–176 °C; IR (KBr, cm^{-1}): 1627 (C═N), 1538, 1338 (NO_2); ^1H NMR (300 MHz, $CDCl_3$): δ 2.30 (s, 3H, CH_3), 3.52 (m, 2H, CH_2), 4.21 (m, 1H, *J* = 4 Hz, benzylic-H), 4.61 (m, 1H, CH), 5.27 (m, 2H, Ar–CH), 5.47 (d, 2H, *J* = 4 Hz, ═CH), 7.08 (s, 2H, thiazole-CH), 7.13–7.82 (m, 23H, ArH). EI-MS $[M]^+$ *m/z* 890. ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.23, 34.89, 46.88, 51.26, 61.41, 94.05, 105.10, 115.24, 124.28, 124.34, 124.57, 125.41, 126.27, 126.36, 126.58, 126.68, 126.77, 127.19, 127.31, 127.49, 127.58, 128.37, 130.31, 134.69, 136.71, 140.21, 143.38, 150.36, 152.27, 159.23, 162.46. Anal. Calcd. for $C_{49}H_{36}N_6Cl_2O_3S_2$: C, 65.99; H, 4.07; N, 9.42. Found: C, 65.92; H, 4.02; N, 9.39%. **7i**: pale yellow; yield 80%, mp 170–172 °C; IR (KBr, cm^{-1}): 1631 (C═N), 1528, 1315 (NO_2); ^1H NMR (300 MHz, $CDCl_3$): δ 2.26 (s, 3H, CH_3), 3.41 (m, 2H, CH_2), 4.25 (m, 1H, *J* = 4 Hz, benzylic-H), 4.66 (m, 1H, CH), 5.20 (m, 2H, Ar–CH), 5.41 (d, 2H, *J* = 4 Hz, ═CH), 7.11(s, 2H, thiazole-CH), 7.20–7.91(m, 23H, ArH). EI-MS $[M]^+$ *m/z* 890. ^{13}C NMR (75 MHz, $CDCl_3$): δ 12.31, 35.31, 47.45, 51.46, 61.51, 94.27, 105.11, 115.38, 124.22, 124.31, 124.60, 125.41, 126.06, 126.23, 126.53, 126.61, 126.81, 127.00, 127.11, 127.41, 127.53, 128.23, 130.22, 134.73, 136.45, 140.17, 143.26, 150.31, 152.38, 159.47, 162.56. Anal. Calcd. for $C_{49}H_{36}N_6Cl_2O_3S_2$: C, 65.99; H, 4.07; N, 9.42. Found: C, 66.02; H, 4.10; N, 9.47%.