Facile and efficient one-pot procedure for thieno[2,3-e][1,2,3]triazolo[1,5-a]pyrimidines preparation

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“Supplementary Information”

5-Oxo-4,5-dihydrothieno[2,3-*e*][1,2,3]triazolo[1,5-*a*]pyrimidine-3-carbonitrile

7a: Yield: 83 %; m.p.: >300°C; ¹H NMR (400 MHz, DMSO-*d*₆+CCl₄) δ 8.05 (d, *J* = 4.9 Hz, 1H, H_{Th}-5), 7.20 (d, *J* = 5.2 Hz, 1H, H_{Th}-4). ¹³C-NMR (125 MHz, CDCl₃): δ 162.8 (CO), 152.7 (C_{Th}-3), 148.2 (C_{Tr}-5), 136.3 (CH_{Th}-5), 124.6 (C_{Th}-4), 114.5 (CN), 107.8 (C_{Th}-2), 103.2 (C_{Tr}-4). MS (m/z): 218 (M⁺+1); anal. calcd. for C₈H₃N₅OS: C 44.24, H 1.39, N 32.24; found: C 44.06, H 1.26, N 32.38.

5-Oxo-4,5-dihydrothieno[2,3-*e*][1,2,3]triazolo[1,5-*a*]pyrimidine-3-carboxamide

7b: Yield: 87 %; m.p.: >300°C; ¹H NMR (500 MHz, DMSO-*d*₆): δ 8.04 (d, *J* = 5.3 Hz, 1H, H_{Th}-5), 7.90 (br.s, 1H, NH), 7.27 (d, *J* = 5.5 Hz, 1H, H_{Th}-4), 7.26 (br.s, 2H, NH₂). ¹³C-NMR (125 MHz, CDCl₃): δ 163.4 (CO), 160.3 (CONH₂), 152.7 (C_{Th}-3), 147.2 (C_{Tr}-5), 135.5(CH_{Th}-5), 124.5 (CH_{Th}-4+C_{Tr}-4), 107.9 (C_{Th}-2). MS (m/z): 236 (M⁺+1); anal. calcd. for C₈H₅N₅O₂S: C 40.85, H 2.14, N 29.77; found: C 40.90, H 2.03, N 29.59.

3-(1,3-Benzothiazol-2-yl)thieno[2,3-*e*][1,2,3]triazolo[1,5-*a*]pyrimidin-5(4*H*)-one

7c: Yield: 89 %; m.p.: >300°C; ¹H NMR (400 MHz, DMSO-*d*₆+CCl₄) δ 8.10 – 8.04 (m, 2H, H_{Bth}-4+ H_{Th}-5), 8.00 (d, *J* = 7.9 Hz, 1H, H_{Bth}-7), 7.48 (t, *J* = 7.4 Hz, 1H, H_{Bth}-6), 7.40 – 7.28 (m, 2H, H_{Bth}-5+H_{Th}-4). ¹³C-NMR (125 MHz, CDCl₃): δ 164.3 (CO), 160.2 (C_{Bth}-2), 154.6 (C_{Bth}-3a), 153.4 (C_{Th}-3), 148.3 (C_{Tr}-5), 135.5(CH_{Th}-5), 134.1 (C_{Bth}-7a), 126.5 (CH_{Bth}-5), 125.5 (C_{Tr}-4), 125.4 (CH_{Bth}-6), 124.5 (CH_{Th}-4), 124.3 (CH_{Bth}-4), 122.1 (CH_{Bth}-7), 107.7(C_{Th}-2).MS (m/z): 326 (M⁺+1); anal. calcd. for C₁₄H₇N₅OS₂: C 51.68, H 2.17, N 21.52; found: C 51.74, H 2.03, N 21.40.

3-[(1-Methyl-1*H*-pyrrol-2-yl)carbonyl]thieno[2,3-*e*][1,2,3]triazolo[1,5-

***a*]pyrimidin-5(4*H*)-one 7d:** Yield: 77 %; m.p.: >300°C; ¹H NMR (400 MHz, DMSO-*d*₆+CCl₄) δ 8.01 (d, *J* = 5.3 Hz, 1H, H_{Th}-5), 7.63 (br.s, 1H, H_{Pyr}-4), 7.31 (d, *J* = 5.3 Hz, 1H, H_{Th}-4), 7.08 (br.s, 1H, H_{Pyr}-3), 6.15 (br.s, 1H, H_{Pyr}-5), 3.98 (s, 3H, CH₃). ¹³C-NMR (125 MHz, CDCl₃): δ 176.4 (CO_{Pyr}), 163.42 (CO), 152.7 (C_{Th}-3), 147.2 (C_{Tr}-5), 135.4 (CH_{Th}-5), 131.8 (C_{Pyr}-2), 129.4 (CH_{Pyr}-5), 127.2 (C_{Tr}-4), 124.4 (CH_{Th}-4), 121.5 (CH_{Pyr}-4), 108.5 (CH_{Pyr}-3), 107.90 (C_{Th}-2), 37.9 (Me). MS (m/z): 300 (M⁺+1); anal. calcd. for C₁₃H₉N₅O₂S: C 52.17, H 3.03, N 23.40; found: C 52.24, H 3.11, N 23.27.

5-Oxo-4,5,7,8,9,10-hexahydrobenzo[4,5]thieno[2,3-*e*][1,2,3]triazolo[1,5-

***a*]pyrimidine-3-carbonitrile 7e:** Yield: 80 %; m.p.: >300°C; ¹H NMR (500 MHz, DMSO-*d*₆): δ 2.81 (s, 2H, CH₂), 2.65 (s, 2H, CH₂), 1.86 (s, 2H, CH₂), 1.81 (s, 2H, CH₂). ¹³C-NMR (125 MHz, CDCl₃): δ 162.8 (CO), 151.6 (C_{Th}-3), 148.1 (C_{Tr}-5), 135.4 (C_{Th}-5), 123.5 (C_{Th}-4), 114.5 (CN), 107.8 (C_{Th}-2), 103.3 (C_{Tr}-5), 26.0 (CH₂), 24.9 (CH₂), 22.6 (CH₂), 22.2 (CH₂). MS (m/z): 272 (M⁺+1); anal. calcd. for C₁₂H₉N₅OS: C 53.13, H 3.34, N 25.81; found: C 53.07, H 3.48, N 25.69.

5-Oxo-4,5,7,8,9,10-hexahydrobenzo[4,5]thieno[2,3-*e*][1,2,3]triazolo[1,5-

***a*]pyrimidine-3-carboxamide 7f:** Yield: 88 %; m.p.: >300°C; ¹H NMR (500 MHz, DMSO-*d*₆): δ 8.01 (s, 1H, NH), 7.19 (s, 1H, NH₂), 2.82 (s, 2H, CH₂), 2.66 (s, 2H, CH₂), 1.87 (s, 2H, CH₂), 1.83 (s, 2H, CH₂). ¹³C-NMR (125 MHz, CDCl₃): δ 163.7 (CO), 160.6 (CONH₂), 151.7 (C_{Th}-3), 147.1 (C_{Tr}-5), 135.4 (C_{Th}-5), 124.2 (C_{Tr}-5), 123.5 (C_{Th}-4), 107.8 (C_{Th}-2), 26.1 (CH₂), 24.9 (CH₂), 22.6 (CH₂), 22.2 (CH₂). MS

(m/z): 290($M^+ + 1$); anal. calcd. for $C_{12}H_{11}N_5O_2S$: C 49.82, H 3.83, N 24.21; found: C 49.76, H 3.94, N 24.07.

Methyl 5-oxo-7-phenyl-4,5-dihydrothieno[2,3-*e*][1,2,3]triazolo[1,5-*a*]pyrimidine-3-carbimide 7g: Yield: 81 %; m.p.: $>300^\circ\text{C}$; ^1H NMR (500 MHz, DMSO- d_6): δ 8.73 (s, 1H, NH), 7.86 (t, $J = 7.2$ Hz, 2H, $H_{\text{Ph-3,5}}$), 7.74 (s, 1H, H_{Th}), 7.52 (t, $J = 6.5$ Hz, 2H, $H_{\text{Ph-2,6}}$), 7.46 (q, $J = 7.3$ Hz, 1H, $H_{\text{Ph-4}}$), 3.85 (s, 3H, MeO). ^{13}C -NMR (125 MHz, DMSO- d_6): δ 162.7 (CO), 160.7 (C=NH), 152.2 ($C_{\text{Th-3}}$), 150.2 ($C_{\text{Th-5}}$), 147.7 ($C_{\text{Tr-5}}$), 134.0 ($C_{\text{Ph-1}}$), 129.7 (2x $\text{CH}_{\text{Ph-2,6}}$), 126.5 ($C_{\text{Tr-4}}$), 126.5 (2x $\text{CH}_{\text{Ph-3,5}}$), 121.9 ($C_{\text{Th-4}}$), 120.7 ($\text{CH}_{\text{Ph-4}}$), 107.1 ($C_{\text{Th-2}}$), 51.9 (MeO). MS (m/z): 326($M^+ + 1$); anal. calcd. for $C_{15}H_{11}N_5O_2S$: C 55.38, H 3.41, N 21.53; found: C 55.24, H 3.32, N 21.41.

5-Oxo-7-phenyl-4,5-dihydrothieno[2,3-*e*][1,2,3]triazolo[1,5-*a*]pyrimidine-3-carboxamide 7h: Yield: 79 %; m.p.: $>300^\circ\text{C}$; ^1H NMR (400 MHz, DMSO- d_6 + CCl_4): δ 7.91 (s, 1H, NH), 7.83 (d, $J = 7.4$ Hz, 2H, $H_{\text{Ph-3,5}}$), 7.72 (s, 1H, H_{Th}), 7.51 (t, $J = 6.9$ Hz, 2H, $H_{\text{Ph-2,6}}$), 7.47 – 7.40 (m, 1H, $H_{\text{Ph-4}}$), 7.27 (s, 1H, NH). ^{13}C -NMR (125 MHz, DMSO- d_6): δ 163.5 (CO), 160.1 (CONH $_2$), 152.6 ($C_{\text{Th-3}}$), 150.1 ($C_{\text{Th-5}}$), 147.4 ($C_{\text{Tr-5}}$), 133.9 ($C_{\text{Ph-1}}$), 129.7 (2x $\text{CH}_{\text{Ph-2,6}}$), 126.5 (2x $\text{CH}_{\text{Ph-3,5}}$), 124.4 ($C_{\text{Tr-4}}$), 121.9 ($C_{\text{Th-4}}$), 120.7 ($\text{CH}_{\text{Ph-4}}$), 107.2 ($C_{\text{Th-2}}$). MS (m/z): 312 ($M^+ + 1$); anal. calcd. for $C_{14}H_9N_5O_2S$: C 54.01, H 2.91, N 22.50; found: C 54.14, H 2.73, N 22.71.