Multicomponent facile synthesis of highly substituted [1,2,4]triazolo[1,5-*a*] pyrimidines

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A simple and convenient method for one-pot synthesis of a series of potentially biologically active [1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide derivatives has been developed in solvent-free conditions using maltose as a commercially available, cheap and eco-friendly catalyst. The salient features of the present protocol are mild reaction conditions, good to excellent yields, high atom economy, benign environmental conditions, easy isolation of products without column chromatography and clean reaction profiles.

Keywords: [1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide, multicomponent reaction, maltose, solvent-free conditions

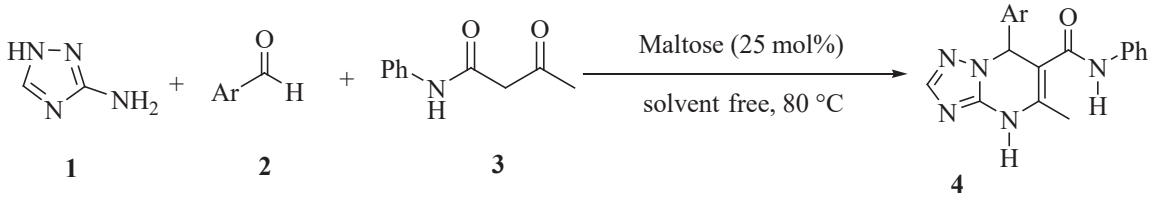
Multicomponent reactions (MCRs) have proven to be useful in organic chemistry, medicinal chemistry and other industries.[1–4] Such protocols can be used for drug design and diversity-oriented synthesis of heterocycles because of their simplicity, efficiency and high selectivity.[5] This environmentally friendly process can avoid a waste of time as well as energy consumption, and often permits a reduction in the number of steps. MCRs under solvent-free conditions have attracted much interest from chemists, particularly from the viewpoint of green chemistry. The possibility of performing MCR reactions under solvent-free conditions with a catalyst could enhance their efficiency from an economic as well as an ecological point of view.[6–10] Fused triazole and pyrimidine ring systems have been studied for several years because of their medicinal and agricultural significance.[11–13] Among their important effects, triazolopyrimidine derivatives are used as blood pressure regulators,[14] antibacterial agents,[15] selective serotonin 5-HT6 receptor antagonists[16] and cardiovascular vasodilators.[17] In addition, several triazolopyrimidine-2-sulfonamide derivatives with herbicidal activity, such as florasulam, flumetsulam and metosulam, are produced commercially.[18] Some important structures containing fused triazole and pyrimidine scaffolds have biological activities, such as anti-appetite, anticonvulsant and anticancer activities.[19–21]

The use of organic catalysts has received considerable attention in organic synthesis owing to their important advantages, such as the possibility of performing reactions with acid-sensitive substrates, milder reaction conditions and selectivity.[22–24] Maltose has received considerable attention in organic synthesis owing to its advantages, such as low cost and ease of handling and isolation. Encouraged by the remarkable results obtained from the above conditions, and also in continuation of our ongoing green chemistry programme that utilises homogeneous systems[25–27] in various organic transformations, we report a simple, mild and convenient procedure for effecting the one-pot, three-component reaction of aryl aldehydes, acetoacetanilide and 3-amino-1,2,4-triazole for preparation of [1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide derivatives using maltose as a homogeneous catalyst under solvent-free conditions (Scheme 1).

Results and discussion

Initially, a mixture containing 3-amino-1,2,4-triazole **1** (1.0 mmol), benzaldehyde **2** (1.0 mmol) and acetoacetanilide **3** (1.0 mmol) was reacted in the absence of any catalyst (Table 1, entry 1) and solvent at 80 °C, but the reaction did not occur. When the reaction was conducted in the presence of a catalytic amount of maltose, a satisfactory result was obtained. Use of 25 mol% of maltose (Table 1, entry 3) was found to be sufficient for obtaining optimum yields of the desired [1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide. Further decrease or increase in the amount of catalyst did not improve the yield of the product. To identify the optimal temperature for this reaction in solvent-free conditions, the reaction was repeated at room temperature (25 °C), 60, 80 and 100 °C for 2 h. As indicated in Table 1, the reaction did not occur at room temperature; however, the yield at 80 °C was 95% after 20 min.

To explore the scope of this reaction, various aldehydes **2** were employed under the optimised reaction conditions. Various aryl aldehydes with different donor and acceptor substituents produced the corresponding products cleanly, as shown in Table 2, and no undesirable side reactions were observed. Higher yields and short reaction times were noticed with electron-deficient aldehydes. All the new compounds were characterised from their elemental analysis and IR, ^{1}H NMR and ^{13}C NMR spectra, and the structure of the products was confirmed by comparison with the literature[13]. For example, the ^{1}H NMR spectrum of **4a** exhibited a singlet for the methyl group ($\delta = 2.19$) and a singlet at $\delta = 3.67$ for the methoxy group. One singlet at 6.55 ppm was attributed to a methine group (CHN). Ten aromatic hydrogens gave rise to characteristic signals in the aromatic region of the spectrum. The spectrum also displayed a



1 **2** **3** **4**

Scheme 1 Maltose-catalysed synthesis of [1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide derivatives.

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Table 1 Optimisation of the catalyst for the synthesis of [1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamides[a]

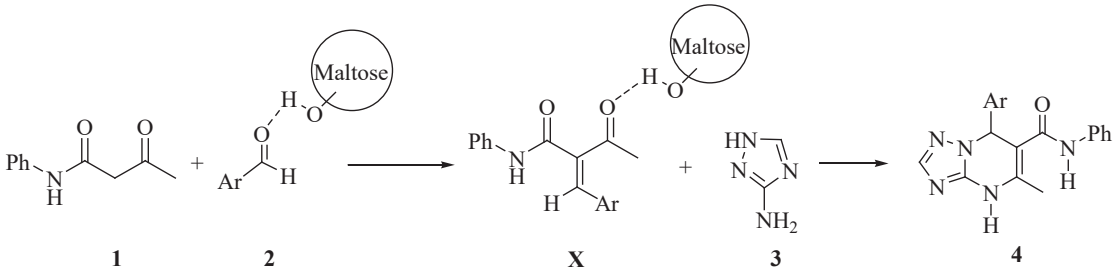
Entry	Catalyst	Mol%	Temperature/°C	Time/min	Yield/%[b]
1	Maltose	-	80	120	–
2	Maltose	20	80	25	84
3	Maltose	25	80	20	95
4	Maltose	30	80	18	87
5	Maltose	25	25 (r.t.)	120	–
6	Maltose	25	60	40	45
7	Maltose	25	100	18	89

[a]Reaction conditions: 3-amino-1,2,4-triazole, acetoacetanilide and benzaldehyde in the presence of catalyst.
[b]Isolated yield.

Table 2 Synthesis of [1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide derivatives

Entry	Ar	Time/min	Yield/%[a]	Product	M.p. (lit. m.p.)/°C
1	3-OMe–C_6H_4	20	92	**4a**	244 (246)
2	2,4-$Cl_2C_6H_3$	18	94	**4b**	246 (248)
3	2-Cl–C_6H_4	22	91	**4c**	252 (254)
4	4-OMe–C_6H_4	18	94	**4d**	245 (247)
5	4-F–C_6H_4	14	95	**4e**	277 (279)
6	Ph	25	91	**4f**	251 (253)
7	2-Br–C_6H_4	22	92	**4g**	241 (243)
8	2-OMe–C_6H_4	20	94	**4h**	226 (228)
9	4-Br–C_6H_4	16	95	**4i**	238 (240)
10	4-NO_2–C_6H_4	25	90	**4j**	258 (260)

[a]Isolated yield.



Scheme 2 Suggested mechanism for the synthesis of [1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide derivatives.

broad singlet (δ = 9.78*)* attributed to the NHCO group, and a singlet at 10.26 ppm was recorded for the NH group. The ^{1}H-decoupled ^{13}C NMR spectrum of **4a** showed 20 distinct carbon signals in agreement with the expected product.

A plausible mechanism for the formation of products **4a** is shown in Scheme 2. Initially, the intermediate X is formed *in situ* from the reaction of acetoacetanilide with an aldehyde. Then, nucleophilic attack of 3-amino-1,2,4-triazole in the presence of the catalyst upon cyclisation afforded the desired product **4a**.[28–30]

Conclusion

In conclusion, we have developed an efficient method for the synthesis of *N*,7-diaryl-5-methyl-4,7-dihydro-1,2,4-triazolo[1,5-*a*]-pyrimidine-6-carboxamides *via* a one-pot, three-component reaction under solvent-free conditions. The environmentally benign and inexpensive catalyst maltose was used as a catalyst in the reaction process.

Experimental

Melting points and IR spectra of all compounds were measured on an Electrothermal 9100 apparatus and a JASCO FTIR 460 Plus spectrometer, respectively. The ^{1}H NMR and ^{13}C NMR spectra were recorded on Bruker DRX-300 Avance instrument with DMSO as a solvent. All reagents and solvents obtained from Fluka and Merck were used without further purification.

Synthesis of N*,7-diaryl-5-methyl-4,7-dihydro-1,2,4-triazolo[1,5-*a*]-pyrimidine-6-carboxamides* (**4a**–**j**)*; general procedure*

Maltose (25 mol%, 0.096 g) was added into a mixture of benzaldehyde (1.0 mmol), 3-amino-1,2,4-triazole (1.0 mmol) and acetoacetanilide (1.0 mmol), then the reaction mixture was heated to 80 °C and stirred for the appropriate time (Table 1). After completion of the reaction (monitored by TLC), the reaction mixture was washed with H_2O (3 × 10 mL). The catalyst is soluble in water and was removed from the reaction mixture. Then, the residue was recrystallised from EtOH.

4,7-Dihydro-7-(3-methoxyphenyl)-5-methyl-N-*phenyl-[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide* (**4a**): White solid; yield 92%; m.p. 244–246 °C; IR (KBr, cm^{-1}) 1596 (C=C), 1672 (CON), 3279 (NH); ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.19 (s, 3H, CH_3), 3.67 (s, 3H, OCH_3), 6.53 (s, 1H, $H_{benzylic}$), 6.74 (d, 1H, *J* = 2.1 Hz, $H_{aromatic}$), 6.78 (d, 1H, *J* = 7.8 Hz, $H_{aromatic}$), 6.84 (dd, 1H, *J* = 7.8 Hz, *J* = 2.4 Hz, $H_{aromatic}$), 7.02 (t, 1H, *J* = 7.5 Hz, $H_{aromatic}$), 7.53 (d, 1H, *J* = 8.4 Hz, $H_{aromatic}$), 7.74 (d, 1H, *J* = 8.6 Hz, $H_{aromatic}$), 7.28 (d, 1H, *J* = 8.1 Hz, $H_{aromatic}$), 7.53 (d, 1H, *J* = 0.9 Hz, $H_{aromatic}$), 7.56 (d, 1H, *J* = 1.8 Hz, $H_{aromatic}$), 7.68 (s, 1H, $H_{aromatic}$), 9.78 (s, 1H, NH), 10.26 (s, 1H, NH); ^{13}C NMR (300 MHz, DMSO-d_6): δ 17.8 (CH_3), 55.4 (OCH_3), 60.5 ($C_{benzylic}$), 103.9, 113.3, 113.5, 119.6, 119.9, 120.0, 123.7, 129.0, 130.1, 137.0, 139.3, 142.7, 148.2, 150.4, 159.6, 165.2 (C=O). Anal. calcd for $C_{20}H_{19}N_5O_2$: C, 66.47; H, 5.30; N, 19.38; found: C, 66.95; H, 5.51; N, 19.66.

7-(2,4-Dichlorophenyl)-4,7-dihydro-5-methyl-N-*phenyl-[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide* (**4b**): Brown solid; yield 94%; m.p. 246–248 °C; IR (KBr, cm^{-1}): 1600 (C=C), 1670 (CON), 3304 (NH); ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.17 (s, 3H, CH_3), 6.92 (s, 1H, $H_{benzylic}$), 7.03 (t, 1H, *J* = 7.5 Hz, $H_{aromatic}$), 7.27 (t, 2H, *J* = 7.8 Hz, $H_{aromatic}$), 7.35 (d, 1H, *J* = 8.4 Hz, $H_{aromatic}$), 7.42 (dd, 1H, *J* = 8.4 Hz, *J* = 1.8 Hz, $H_{aromatic}$), 7.50 (d, 2H, *J* = 8.1 Hz, $H_{aromatic}$), 7.56 (d, 1H, *J* = 1.8 Hz, $H_{aromatic}$), 7.66 (s, 1H, $H_{aromatic}$), 9.88 (s, 1H, NH), 10.39 (s, 1H, NH); ^{13}C NMR (300 MHz, DMSO-d_6): δ 17.6 (CH_3), 57.8 ($C_{benzylic}$), 102.7, 119.8, 119.9, 123.8, 128.3, 129.1, 129.5, 131.9, 133.6, 134.0, 137.2, 137.4, 139.2, 139.3, 148.2, 150.6, 164.8 (C=O). Anal. calcd for $C_{19}H_{15}C_{12}N_5O$: C, 57.01; H, 3.78; N, 17.50; found: C, 57.39; H, 4.02; N, 17.81.

7-(2-Chlorophenyl)-4,7-dihydro-5-methyl-N-*phenyl-[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide* (**4c**): White solid; yield 91%; m.p. 252–254 °C; IR (KBr, cm^{-1}): 1595 (C=C), 1671 (CON), 3297 (NH); ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.17 (s, 3H, CH_3), 6.95 (s, 1H, $H_{benzylic}$), 7.01 (t, 1H, *J* = 7.5 Hz, $H_{aromatic}$), 7.22–7.39 (m, 6H, $H_{aromatic}$), 7.49 (d, 1H, *J* = 8.1 Hz, $H_{aromatic}$), 7.65 (s, 1H, $H_{aromatic}$), 9.86 (s, 1H, NH), 10.33 (s, 1H, NH); ^{13}C NMR (300 MHz, DMSO-d_6): δ 17.5 (CH_3), 58.2 ($C_{benzylic}$), 103.1, 119.8, 119.9, 123.7, 128.0, 129.0, 130.1, 130.3, 130.6, 132.6, 137.2, 138.1, 139.2, 139.3, 148.2, 150.4, 164.9 (C=O). Anal. calcd for $C_{19}H_{16}ClN_5O$: C, 62.38; H, 4.41; N, 19.14; found: C, 62.56; H, 4.32; N, 19.25.

4,7-Dihydro-7-(4-methoxyphenyl)-5-methyl-N-*phenyl-[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide* (**4d**): White solid; yield 94%; m.p. 245–247 °C; IR (KBr, cm^{-1}): 1596 (C=C), 1672 (CON), 3297 (NH),

^{1}H NMR (300 MHz, DMSO-d_6): δ 2.18 (s, 3H, CH_3), 3.70 (s, 3H, OCH_3), 6.50 (s, 1H, $H_{benzylic}$), 6.86 (d, 2H, J = 8.7 Hz, $H_{aromatic}$), 7.01 (t, 1H, J = 7.5 Hz, $H_{aromatic}$), 7.17 (d, 2H, J = 8.4 Hz, $H_{aromatic}$), 7.26 (t, 2H, J = 7.5 Hz, $H_{aromatic}$), 7.53 (d, 2H, J = 7.5 Hz, $H_{aromatic}$), 7.64 (s, 1H, $H_{aromatic}$), 9.74 (s, 1H, NH), 10.20 (s, 1H, NH); ^{13}C NMR (300 MHz, DMSO-d_6): δ 17.7 (CH_3), 55.5 (OCH_3), 60.1 ($C_{benzylic}$), 104.1, 114.2, 119.8, 119.9, 123.7, 128.8, 129.0, 133.3, 136.8, 139.4, 148.0, 150.2, 159.4, 165.3 (C=O). Anal. calcd for $C_{20}H_{19}N_5O_2$: C, 66.47; H, 5.30; N, 19.38; found: C, 66.69; H, 5.44; N, 19.60.

7-(4-Fluorophenyl)-4,7-dihydro-5-methyl-N-*phenyl-[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide* (**4e**): White solid; yield 95%; m.p. 277–279 °C; IR (KBr, cm^{-1}): 1596 (C=C), 1664 (CON), 3271 (NH); ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.19 (s, 3H, CH_3), 6.56 (s, 1H, $H_{benzylic}$), 6.99–7.04 (m, 1H, $H_{aromatic}$), 7.12–7.18 (m, 2H, $H_{aromatic}$), 7.23–1.32 (m, 4H, $H_{aromatic}$), 7.49–7.52 (m, 2H, $H_{aromatic}$), 9.78 (s, 1H, NH), 10.29 (s, 1H, NH); ^{13}C NMR (300 MHz, DMSO-d_6): δ 17.7 (CH_3), 60.0 ($C_{benzylic}$), 103.8, 115.6, 115.9, 119.9, 120.0, 123.8, 129.0, 129.6, 129.7, 137.0, 137.1, 139.2, 139.3, 148.2, 150.4, 160.6, 163.8, 165.3 (C=O). Anal. calcd for $C_{19}H_{16}FN_5O$: C, 65.32; H, 4.62; N, 20.05; found: C, 65.91; H, 4.97; N, 20.34.

4,7-Dihydro-5-methyl-N,*7-diphenyl-[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide* (**4f**): White solid; yield 91%; m.p. 252–254 °C; IR (KBr, cm^{-1}): 1595 (C=C), 1661 (CON), 3295 (NH); ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.19 (s, 3H, CH_3), 6.55 (s, 1H, $H_{benzylic}$), 7.02 (t, 1H, J = 7.5 Hz, $H_{aromatic}$), 7.21–7.35 (m, 7H, $H_{aromatic}$), 7.50 (s, 1H, $H_{aromatic}$), 7.53 (d, 2H, J = 1.2 Hz, $H_{aromatic}$), 7.66 (s, 1H, $H_{aromatic}$), 9.78 (s, 1H, NH), 10.25 (s, 1H, NH); ^{13}C NMR (300 MHz, DMSO-d_6): δ 17.7 (CH_3), 60.7 ($C_{benzylic}$), 104.0, 104.0, 119.9, 120.0, 123.7, 127.4, 128.5, 128.9, 129.0, 136.9, 137.0, 139.3, 139.4, 141.2, 148.2, 150.4, 165.2 (C=O). Anal. calcd for $C_{19}H_{17}N_5O$: C, 68.87; H, 5.17; N, 21.13; found: C, 69.04; H, 5.09; N, 21.03.

7-(2-Bromophenyl)-4,7-dihydro-5-methyl-N-*phenyl-[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide* (**4g**): White solid; yield 92%; m.p. 241–243 °C; IR (KBr, cm^{-1}): 1594 (C=C), 1661 (CON), 3312 (NH); ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.17 (s, 3H, CH_3), 6.93 (s, 1H, $H_{benzylic}$), 7.01 (t, 1H, J = 7.5 Hz, $H_{aromatic}$), 7.17 (d, 1H, J = 2.7 Hz, $H_{aromatic}$), 7.19–7.27 (m, 2H, $H_{aromatic}$), 7.30–7.38 (m, 2H, $H_{aromatic}$), 7.48 (d, 1H, J = 0.9 Hz, $H_{aromatic}$), 7.50 (d, 1H, J = 1.2 Hz, $H_{aromatic}$), 7.54 (d, 1H, J = 7.5 Hz, $H_{aromatic}$), 7.64 (s, 1H, $H_{aromatic}$), 9.87 (s, 1H, NH), 10.33 (s, 1H, NH); ^{13}C NMR (300 MHz, DMSO-d_6): δ 17.6 (CH_3), 60.4 ($C_{benzylic}$), 103.5, 119.8, 119.9, 122.8, 123.7, 128.6, 129.0, 130.6, 133.3, 136.9, 137.0, 139.2, 139.3, 139.8, 148.2, 150.4, 165.0 (C=O). Anal. calcd for $C_{19}H_{16}BrN_5O$: C, 55.62; H, 3.93; N, 17.07; found: C, 56.11; H, 4.15; N, 17.44.

4,7-Dihydro-7-(2-methoxyphenyl)-5-methyl-N-*phenyl-[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide* (**4h**): White solid; yield 94%; m.p. 226–228 °C; IR (KBr, cm^{-1}): 1596 (C=C), 1672 (CON), 3296 (NH); ^{1}H NMR (300 MHz, DMSO-d_6): δ = 2.13 (s, 3H, CH_3), 3.63 (s, 3H, OCH_3), 6.85 (s, 1H, $H_{benzylic}$), 6.87 (d, 1H, J = 7.5 Hz, $H_{aromatic}$), 6.94 (d, 1H, J = 7.8 Hz, $H_{aromatic}$), 7.01 (t, 2H, J = 7.2 Hz, $H_{aromatic}$), 7.18–7.28 (m, 3H, $H_{aromatic}$), 7.52–7.55 (m, 2H, $H_{aromatic}$), 7.60 (s, 1H, $H_{aromatic}$), 9.77 (s, 1H, NH), 10.12 (s, 1H, NH); ^{13}C NMR (300 MHz, DMSO-d_6): δ 17.5 (CH_3), 55.6 (OCH_3), 56.2 ($C_{benzylic}$), 103.9, 112.2, 119.7, 119.8, 120.9, 123.5, 128.6, 129.0, 129.4, 129.8, 136.8, 139.6, 148.9, 150.0, 151.0, 157.0, 165.3 (C=O). Anal. calcd for $C_{20}H_{19}N_5O_2$: C, 66.47; H, 5.30; N, 19.38; found: C, 66.70; H, 5.51; N, 19.30.

4,7-Dihydro-7-(4-bromophenyl)-5-methyl-N-*phenyl-[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide* (**4i**): White solid; yield 95%; m.p. 238–240 °C; IR (KBr, cm^{-1}): 1594 (C=C), 1661 (CON), 3312 (NH); ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.13 (s, 3H, CH_3), 3.63 (s, 3H, OCH_3), 6.85 (s, 1H, $H_{benzylic}$), 6.87 (d, 1H, J = 7.5 Hz, $H_{aromatic}$), 6.94 (d, 1H, J = 7.8 Hz, $H_{aromatic}$), 7.01 (t, 2H, J = 7.2 Hz, $H_{aromatic}$), 7.18–7.28 (m, 3H, $H_{aromatic}$), 7.52–7.55 (m, 2H, $H_{aromatic}$), 7.60 (s, 1H, $H_{aromatic}$), 9.77 (s, 1H, NH), 10.12 (s, 1H, NH); ^{13}C NMR (300 MHz, DMSO-d_6): δ 17.8 (CH_3), 60.1 ($C_{benzylic}$), 103.5, 119.9, 120.0, 121, 8, 123.8, 129.0, 129.7, 131.7, 131.9, 132.8, 137.2, 137.3, 139.2, 140.4, 148.1, 150.5, 165.2 (C=O). Anal. calcd for $C_{19}H_{16}BrN_5O$: C, 55.62; H, 3.93; N, 17.07; found: C, 55.94; H, 3.99; N, 17.33.

4,7-Dihydro-5-methyl-7-(4-nitrophenyl)-N-*phenyl-[1,2,4]triazolo[1,5-*a*]pyrimidine-6-carboxamide* (**4j**): Light brown solid; yield 90%; m.p. 258–260 °C; IR (KBr, cm^{-1}): 1600 (C=C), 1672 (CON), 3260 (NH); ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.20 (s, 3H, CH_3), 6.70 (s, 1H, $H_{benzylic}$), 7.02 (t, 1H, J = 7.5 Hz, $H_{aromatic}$), 7.26 (t, 1H, J = 7.8 Hz, $H_{aromatic}$), 7.50 (d, 4H, J = 8.7 Hz, $H_{aromatic}$), 7.71 (s, 1H, $H_{aromatic}$), 8.20 (d, 2H, J = 8.7 Hz, $H_{aromatic}$), 9.83 (s, 1H, NH), 10.44 (s, 1H, NH). Anal. calcd for $C_{19}H_{16}N_6O_3$: C, 60.63; H, 4.28; N, 22.33; found: C, 60.99; H, 4.53; N, 22.65.

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